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Short term variability of soil aggregate stability and associated physico-chemical processes

Baptiste Algayer

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ÉCOLE DOCTORALE SCIENCES ET TECHNOLOGIES

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Unité de Recherche de Science du Sol, centre d'Orléans

THÈSE

présentée par : **Baptiste ALGAYER**

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Variabilité à court terme de la stabilité structurale des sols et processus physico-chimiques associés

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« La gratitude peut transformer votre routine en jour de fête »

William Arthur Ward

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Introduction générale

1. Contexte et problématique

1.1. L'érosion hydrique, une menace pour la durabilité de la ressource sol

Le sol est une ressource non renouvelable à l'échelle de temps de nos sociétés. Cette ressource est soumise à différents processus de dégradation qui menacent la durabilité de son fonctionnement et les services éco-systémiques qui y sont liés. L'érosion hydrique est l'un de ces processus.

Selon la FAO (1998) « 26 millions d'hectares seraient affectés par l'érosion hydrique des sols dans l'Union Européenne ». En France, selon le rapport du Gis Sol (2011), « près de 18% des sols de France métropolitaine présentent un aléa d'érosion moyen à très fort ». En contexte agricole, l'érosion des sols cultivés engendre des pertes en fertilité du sol, des baisses de rendement à la levée des plantes et, dans certains cas, la destruction des semis ou la formation de ravines qui constituent un obstacle pour les opérations culturales. Le transport vers l'aval des particules minérales et des fertilisants prélevés par l'érosion constitue une source de pollution pour la qualité des eaux de surface (perturbation des écosystèmes) et des dommages importants pour les collectivités (salissement et destruction de chaussées, coulées boueuses...). Ainsi, l'érosion hydrique des sols cultivés représente une menace pour la durabilité de la ressource sol et génère une charge économique importante. L'optimisation des pratiques agricoles et la mise en place d'aménagements afin de lutter contre l'érosion apparaît donc comme un enjeu crucial.

D'un point de vue scientifique, l'érosion hydrique des sols est définie comme l'ensemble des processus de détachement, de transport et de dépôt de particules issues de la surface du sol sous l'action de l'eau. L'érosion hydrique fait intervenir deux processus de dégradation des sols qui interagissent : le détachement de particules et la formation de croûtes de battance. Sous l'effet des pluies, les mottes et agrégats présents à la surface du sol se fragmentent. Ses fragments peuvent être mobilisés par le splash ou le ruissellement, et être alors transportés

vers l'aval. La concentration de fragments de petites tailles à la surface peut conduire à la formation d'une croûte qui engendre la fermeture progressive de la porosité ouverte du sol. La présence de croûtes de battance réduit considérablement l'infiltration de l'eau dans le sol et, de ce fait, augmente la quantité de ruissellement. Le ruissellement ainsi généré est le moteur du détachement et du transport des particules sur de longues distances.

1.2. L'érosion hydrique est un processus difficile à modéliser

La modélisation numérique de l'érosion hydrique constitue un outil indispensable pour optimiser la lutte contre l'érosion des sols. Les modèles d'érosion des sols permettent d'estimer les risques d'érosion, de quantifier les flux d'eau et de particules générés et d'évaluer l'impact des mesures de lutte contre l'érosion. La plupart des modèles considèrent deux paramètres clefs dans les processus d'érosion hydrique des sols : l'érosivité et l'érodibilité. L'érosivité de la pluie ou du ruissellement correspond à la capacité des agents érosifs à détacher et à transporter les particules de sol. L'érodibilité du sol correspond à la sensibilité du sol face à ces agents érosifs. Ce paramètre réfère donc aux propriétés du sol. On distingue généralement l'érodibilité des rigoles (« rill erodibility ») qui renvoie à la sensibilité du sol aux processus de cisaillement lié à l'érosion concentrée, et l'érodibilité inter-rigoles (« interrill erodibility ») qui renvoie aux processus de l'érosion diffuse.

Actuellement, si les modèles d'érosion sont indispensables, les prédictions de l'érosion données par ces modèles présentent globalement de gros problèmes de précision (Jetten et al., 1999, Jetten et al., 2003 ; Boardman, 2006 ; Guimere et al., 2009). Les difficultés de prédiction de l'érosion par les modèles sont en partie liées à des difficultés dans l'estimation de l'érodibilité du sol (Cheviron et al., 2011). En effet, l'érodibilité est une notion complexe englobant différentes propriétés du sol influencées par de nombreux facteurs interagissant entre eux. Si de nombreuses études concernant l'influence des propriétés du sol sur les processus d'érosion ont permis d'améliorer nos connaissances sur l'érodibilité (e.g.

Wishmeier & Mannering, 1969; Bryan et al., 1989; Lal, 1991; Bryan, 2000 ; Bajracharya et al., 1998, Salvator Sanchis et al., 2008), ce paramètre reste difficile à estimer et à quantifier (Borseli et al., 2012).

Il existe différentes méthodes pour estimer l'érodibilité inter-rigolle d'un sol. Des mesures directes sur le terrain ou sous simulateur de pluie peuvent être réalisées. Ces mesures consomment beaucoup de temps, nécessitent des investissements lourds et donc représentent un poids financier important. L'érodibilité peut aussi être estimée par des fonctions statistiques intégrant des propriétés du sol comme la texture ou la teneur en matières organiques (e. g. Alberts et al., 1995; Renard et al., 1997). Largement utilisée par les modèles d'érosion (e.g. RUSLE, WEPP) cette méthode nécessite une connaissance approfondie des propriétés du sol influençant l'érodibilité, ce qui n'est pas le cas actuellement. Une méthode intermédiaire consiste à mesurer la stabilité structurale des agrégats comme proxy de l'érodibilité inter-rigolle (Bajracharya et al., 1992 ; Le Bissonnais, 1996 ; Barthès & Roose, 2002).

1.3. La prédiction de la stabilité structurale : un enjeu pour améliorer les prédictions de l'érodibilité du sol

La structure du sol correspond à l'arrangement des solides et des vides au sein de la matrice du sol. À travers différents processus, les particules minérales et les substances organiques ou inorganiques présentes dans le sol peuvent s'agglomérer entre elles pour former un agrégat possédant une certaine cohésion mécanique. La stabilité des agrégats (ou stabilité structurale) correspond à la capacité d'un agrégat à conserver sa structure lorsqu'il est soumis à un stress exogène comme par exemple une humectation. Il s'agit d'une propriété intrinsèque du sol que l'on mesure de façon empirique. La stabilité structurale influence les flux d'eau et de gaz au sein du sol, l'activité biologique, et le développement des plantes (Amézketa, 1999). La stabilité structurale influence également la sensibilité du sol à l'érosion

inter-rigole et à l'encroûtement (Le Bissonnais, 1996 ; Bajracharya et al., 1998 ; Barthès & Roose, 2002). Les agents érosifs tendent à fragmenter les agrégats présents à la surface du sol. Il en résulte la présence de fragments de petites tailles. Plus leur taille sera petite, plus ces fragments seront susceptibles d'être mobilisés par les agents de transport. De plus, la fragmentation de ces agrégats est à l'origine de la formation des croûtes de battance. Une stabilité structurale élevée à la surface du sol limite donc la fragmentation des agrégats par les agents érosifs et, donc, la libération de particules facilement mobilisables ainsi que la formation de croûte. Ainsi, plus la stabilité structurale est élevée, moins le sol est sensible à l'érosion et à l'encroûtement. Ainsi, en améliorant nos connaissances sur les facteurs contrôlant la stabilité structurale, on pourra mieux estimer le paramètre « érodibilité inter-rigoles » des modèles et donc mieux prédire l'érosion des sols.

De nombreuses études se sont intéressées aux facteurs de la stabilité structurale du sol. Cependant, les connaissances concernant la variation de la stabilité structurale pour un même type de sol restent limitées. Des suivis de terrain à pas de temps mensuel ont permis de mettre en valeur une forte saisonnalité de cette propriété en relation avec le climat et l'activité biologique (e.g. Bullock et al., 1988 ; Blackman, 1992, Chan et al., 1994 ; Dimoyiannis, 2009). Des suivis menés en laboratoires ont montré que la stabilité structurale d'un sol donné variait à pas de temps plus court (quelques jours à quelques semaines) en relation avec les cycles d'humidité et la stimulation de l'activité biologique (e.g. Deneff et al., 2001 ; Cosentino et al., 2006). En revanche, les connaissances concernant les variations de stabilité structurale à pas de temps court sans stimulation de l'activité biologique restent méconnues. Bien évidemment, il en va de même pour les facteurs contrôlant ces variations. À ce jour, s'il n'existe pas de fonction pédo-transfert capable de prévoir les variations de la stabilité structurale, de nombreuses études ont permis d'identifier des facteurs contrôlant la stabilité structurale. Ces facteurs ont été répertoriés à travers différentes synthèses bibliographiques

(e.g. Amézketa, 1999; Brownick & Lal, 2005; Six et al., 2006). Amézketa (1999) et Bronick & Lal (2005) ont distingué des facteurs internes incluant les propriétés du sol (teneur en matières organiques, électrolytes, texture, minéralogie des argiles,...) et des facteurs externes (climat, activité biologique, pratique culturale,...). Six et al. (2004) ont séparé les agents organiques des agents inorganiques, les deux types étant dépendants de variables environnementales (e.g. cycle d'humidité, température). Ces facteurs sont impliqués dans les variations de la stabilité structurale au travers de processus biotiques et physico-chimiques. Les processus biotiques ont bénéficié de nombreuses études récentes qui ont notamment permis de souligner l'influence de l'activité biologique et de la dynamique des matières organiques dans les variations de la stabilité structurale (Chenu et al., 2000 ; Cosentino et al., 2006 ; Abiven et al., 2007 ; Leguillou et al., 2012). Cependant, ces études n'ont pas permis de prédire les variations de la stabilité structurale de façon satisfaisante notamment lorsque l'activité biologique n'est pas stimulée ou lorsque la teneur en matières organiques du sol est faible (<5%) (Chan et al. 1994; Bajracharya et al., 1998; Dimoyiannis 2009). Dans ces cas, les processus incriminés dans la variation de la stabilité structurale sont des processus abiotiques liés au climat et notamment aux cycles d'humidité. Historiquement, les processus physico-chimiques ont été les premiers étudiés (e.g. Yoder, 1936; Hénin, 1939; Emerson, 1967; Utomo & Dexter, 1981; Kemper & Rosenau, 1984 ; Dexter et al., 1988). Certains processus ont été clairement observés tandis que d'autres restent des conjectures et nécessitent d'être vérifiés. Ainsi par exemple le processus d'éclatement (slaking) ou celui de flocculation-dispersion ont été étudié en profondeur à travers des expérimentations reproductibles, tandis que d'autres processus comme le réarrangement particulaire intra-agrégat ou le durcissement thixotropique reposent sur des hypothèses. Mieux prédire les variations de stabilité structurale d'un sol donné implique de mieux connaître ces processus.

2. Objectifs et plan de la thèse

Cette thèse a été réalisée avec pour objectif général d'améliorer les connaissances concernant les processus physico-chimiques de variation de la stabilité structurale en climat tempéré pour des sols cultivés. L'approche adoptée est basée sur des mesures de la stabilité structurale à pas de temps court en se focalisant sur les variations d'humidité du sol, qui sont le moteur des processus physico-chimiques cités dans la littérature. Le premier objectif est de quantifier — à pas de temps court et sur le terrain — les variations de stabilité structurale, et d'identifier les facteurs contrôlant ces variations. Le second objectif est — sur la base d'un inventaire des processus physico-chimiques de variation de la stabilité structurale cités dans la littérature — d'identifier les processus méconnus et de les étudier à travers des expérimentations en laboratoire.

Le présent manuscrit s'articule autour de trois parties, chacune divisée en deux chapitres rédigés sous forme d'articles. La première partie présente le contexte des travaux. Le chapitre 1, soumis à la revue *European Journal of Soil Science*, correspond à une étude de terrain qui met en valeur les variations spatiales de stabilité structurale pour un même type de sol encroûté, et les difficultés de prédictions de la stabilité structurale à partir des propriétés usuellement mesurées sur un sol. Ce chapitre souligne les difficultés d'une bonne estimation de l'érodibilité dans les modèles d'érosion et met en évidence la nécessité d'une mesure de la stabilité structurale à pas de temps court afin d'identifier clairement les facteurs contrôlant sa variabilité. Le chapitre 2 est une synthèse bibliographique des processus physico-chimiques cités dans la littérature comme étant à l'origine de variation de la stabilité structurale. Ce chapitre souligne l'influence majeure des cycles humectation-dessiccation sur ces processus et identifie les processus qui restent méconnus (dont le réarrangement particulaire intra agrégat).

La deuxième partie rapporte les résultats d'un suivi de terrain réalisé sur des sols de Beauce dans l'objectif de quantifier les variations de stabilité structurale à pas de temps court et d'identifier les facteurs contrôlant ces variations. Les deux chapitres qui le constituent feront l'objet d'une soumission à la revue *Soil Science Society of America Journal*. Le chapitre 3 met en valeur d'importantes variations de la stabilité structurale à pas de temps court et souligne l'effet de l'historique des précipitations comme facteur de ces variations. Le chapitre 4 détaille cette relation et identifie l'histoire hydrique du sol comme le facteur dominant des variations de la stabilité structurale à pas de temps court (expliquant jusqu'à 60% des variations observées).

La troisième partie porte sur l'identification des processus influençant les variations de stabilité structurale liées aux cycles d'humidité. Il s'agit de deux expérimentations menées en laboratoire. Le chapitre 5 met en évidence des variations de pression interne, à l'échelle millimétrique, entre les particules d'un massif d'agrégat soumis à des cycles d'humidité contrôlés. Le chapitre 6 présente les résultats d'une expérimentation visant à valider le processus de réarrangement particulaire comme étant à l'origine de variations de la stabilité structurale. L'imagerie par micro-tomographie X suggère que si le processus de réarrangement particulaire est la cause de variations de la stabilité structurale, il ne s'agit pas d'un réarrangement global de la structure mais de modifications localisées.

Première partie

Variabilité de la stabilité structurale sur le terrain. Evaluation des processus physico-chimiques associés

Introduction

L'érodibilité inter-rigole correspond à la sensibilité d'un sol au détachement de particules et à leur transport par les agents érosifs que sont la pluie et le ruissellement. Il s'agit donc d'un paramètre clef dans la modélisation de l'érosion. Ce paramètre ne peut pas être mesuré directement. Il réfère à des processus interagissant, contrôlés par de nombreux facteurs liés aux propriétés du sol (texture, structure, teneur en matières organiques, etc.). Aussi, l'érodibilité du sol reste difficile à estimer. De ce fait, la plupart des modèles d'érosion sont contraints de considérer le paramètre « érodibilité » d'un sol donné comme une constante dans l'espace et le temps (Jetten et al., 2003 ; Gumiere et al., 2009). Au laboratoire, les tests de stabilité structurale constitue une méthode d'estimation de l'érodibilité, une forte stabilité structurale correspondant à une faible érodibilité (Le Bissonnais, 1996 ; Barthès & Roose, 2002). Du fait de cette relation, la prédiction de la stabilité structurale apparait comme un enjeu pour l'amélioration de la paramétrisation des modèles d'érosion.

Malgré de nombreuses études concernant les facteurs contrôlant la stabilité structurale, cette propriété du sol reste actuellement difficile à prédire (Chan et al. 1994; Bajracharya et al., 1998; Dimoyiannis 2009). Différents processus sont impliqués dans les variations de la stabilité structurale. Ces processus sont biotiques (liés à l'activité biologique et à la dynamique des matières organiques) ou abiotiques (physico-chimiques), liés notamment au climat mais aussi, en contexte agricole, à l'irrigation et aux amendement minéraux (chaulage). En lien avec la dynamique du carbone, les processus biotiques ont fait l'objet de nombreuses études récentes (Chenu et al., 2000 ; Cosentino et al., 2006 ; Abiven et al., 2007 ; Leguillou et al., 2012) mais n'ont pas permis de prédire les variations de la stabilité structurale de façon satisfaisante notamment lorsque l'activité biologique n'est pas stimulée ou lorsque la teneur en matière organique du sol est faible (<5%) (Chan et al. 1994; Bajracharya et al., 1998; Dimoyiannis 2009). Dans ces cas, les études sur les processus biotiques renvoient aux

processus abiotiques pour expliquer les variations de la stabilité structurale. . Ces processus abiotiques ont été étudiés dans un passé plus lointain (e.g. Yoder, 1936; Hénin, 1939; Emerson, 1967), certains processus ont été clairement observés tandis que d'autres restent basés sur des conjectures et nécessitent donc toujours d'être vérifiés.

La première partie de ce manuscrit a été construite sur 2 chapitres avec pour objectif de poser les bases contextuelles de la thèse. Le chapitre 1 correspond à une étude de terrain réalisée dans la région du Plateau de Loess (Chine), à l'automne 2009. Les objectifs étaient de caractériser la variabilité de l'érodibilité d'un sol à travers des mesures de stabilité structurale réalisées sur des états encrouvés, et de relier les résultats obtenus à la stabilité structurale du matériau sous-jacent et aux propriétés standards du sol (texture, teneur en matières organiques, etc...) Ce chapitre montre que l'érodibilité d'un même type de sol peut varier fortement même pour un secteur de faible dimension. Les résultats soulignent la nécessité de mesurer l'érodibilité du sol sur le matériau de surface et non pas dans l'horizon labouré comme c'est usuellement le cas. Enfin, cette étude montre que les propriétés du sol généralement mesurées ne permettent pas de prédire les variations de stabilité structurale. Rédigée sous la forme d'une article, cette étude a été soumise à la revue *European Journal of Soil Science* et est actuellement en cours de relecture.

Le chapitre 2 est une synthèse bibliographique dressant l'état de l'art sur les processus physico-chimiques de variation de la stabilité structurale. Il dresse un inventaire exhaustif des processus physico-chimiques cités dans la littérature comme étant à l'origine de variation de la stabilité structurale et pointe ceux qui sont méconnus. C'est notamment le cas du réarrangement particulière intra-agrégat qui demandera une étude approfondie pour vérifier son implication dans les variations de stabilité structurale.

Chapitre 1 :

Evaluation de l'érodibilité pour un sol encrouté et conséquences pour la modélisation de l'érosion.

Erodibility of a crusted soil: assessment of controlling factors and consequences for erosion modeling:

An example from the Loess Plateau of China

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Abstract

Soil interrill erodibility is a key parameter in soil erosion models. However, because soil interrill erodibility is still difficult to predict accurately, current erosion models use a constant erodibility value for a given soil. Moreover, when using aggregate stability to assess soil erodibility, samples are usually collected from the plow layer, while soil erosion occurs at the soil surface. Hence, the potential changes in erodibility caused by crusting are ignored. This study was conducted to assess the variability of aggregate stability for a crusted soil and to relate this variability to the aggregate stability of the underlying material and to the standard soil properties. A field study was conducted in a limited area of the Loess Plateau (China). The crusts and the underlying materials were sampled. The soil aggregate stability was measured as a proxy of soil erodibility. Standard soil properties (organic matter content, sand content, silt content, clay content, CEC, and pH) were measured as potential explanatory factors of erodibility. The results showed a large variability in aggregate stability among the sites, although the sites were very homogeneous based on the standard soil properties. Even if some correlations existed, none of the standard soil properties was able to predict aggregate stability accurately. The aggregate stability of the crusts was significantly higher than that of the underlying material. The large differences in aggregate stability imply large differences in soil interrill erodibility. Because a unique soil type was investigated, this finding proves that erodibility can vary greatly spatially even for a given soil. Soil erodibility should be estimated based on the exact material actually exposed to erosive forces, i.e., the soil surface material. Using the underlying material would have led to greatly overestimated erodibility and thus to a notable bias in the erosion model prediction.

Résumé

L'érodibilité du sol est un paramètre clef des modèles d'érosion. Cependant, comme l'érodibilité reste encore difficile à prédire avec précision, les modèles d'érosion utilisent actuellement une érodibilité constante pour un type de sol donné. En conséquence, ils ne tiennent pas compte des variations d'érodibilité au sein d'un type de sol donné. De plus, lorsqu'elle est évaluée par des tests de stabilité structurale, l'érodibilité est habituellement évaluée sur des échantillons prélevés dans l'ensemble de la couche de surface ou de l'horizon labouré, alors que l'érosion a lieu à la surface du sol. Ainsi, les changements potentiels d'érodibilité causés par la formation de croûtes sont ignorés, ce qui pourrait conduire à des biais dans l'évaluation de l'érodibilité. Cette étude a été conduite pour évaluer la variabilité de l'érodibilité sur un sol encroûté, et pour relier cette variabilité à la stabilité structurale du matériau sous-jacent et aux propriétés standards du sol. Une étude de terrain a été réalisée sur un secteur limité du Plateau de Loess (Chine). Pour différentes occupations du sol, des échantillons provenant de la croûte et de la sous-croûte ont été collectés. La stabilité structurale a été mesurée comme un proxy de l'érodibilité. Les propriétés standards (teneur en matières organiques ; teneurs en sable, limon et argile ; CEC et pH), connues pour être reliées à la stabilité structurale, ont été mesurées en tant que facteurs explicatifs potentiels de l'érodibilité. Les résultats ont montré une grande variabilité de la stabilité structurale entre les différents sites tandis que ces derniers étaient très homogènes vis-à-vis des propriétés standards du sol. Bien que la stabilité structurale et les propriétés standards du sol présentaient des corrélations, aucune de ces propriétés n'a permis de prédire précisément la stabilité structurale. La stabilité structurale de la croûte était significativement supérieure à celle du matériau sous-jacent pour la plupart des sites. La différence de stabilité structurale entre croûte et sous-croûte était très variable et aucune des propriétés standards mesurées n'a permis de prédire cette différence avec précision. Les grandes différences de stabilité

structurale mesurées impliquent des érodibilités très contrastées. Puisqu'un seul type de sol a été étudié, ce résultat prouve que l'érodibilité peut être très variable spatialement pour un type de sol donné. L'érodibilité du sol devrait être mesurée sur le matériau exact qui subi l'érosion, c'est-à-dire le matériau de surface. L'utilisation du matériau sous-jacent aurait engendré une forte surestimation de l'érodibilité et donc un biais important dans les prédictions des modèles d'érosion.

3. Introduction

In the context of soil erosion by water, interrill erodibility corresponds to the sensitivity of the surface material to detachment and transport by the raindrop impacts and by the sheet flow. Accordingly, interrill erodibility is a key parameter in soil erosion models. Notwithstanding that no unified way of defining erodibility currently exists in the erosion models, researchers also have no widely-recognized method to assess erodibility. Indeed, several assessment methods are used, and erodibility can be estimated through standard soil properties (e.g., soil texture, carbon content) using statistical functions (Alberts *et al.*, 1995; Renard *et al.*, 1997). Although quick, such estimations postulate that samples with similar standard soil properties have similar erodibilities. While these estimates rely on other measurements, their validity scopes are often not considered. Field plot monitoring or rainfall simulations are the most time-consuming methods of erodibility estimation (e.g., Siegrist *et al.*, 1998). An intermediary approach measures aggregate stability in the laboratory (Bajracharya *et al.*, 1992; Le Bissonnais, 1996; Barthès & Roose, 2002). Whatever the estimation method, erosion models typically use the same erodibility value for a given soil, hence postulating a low heterogeneity of erodibility (Renard *et al.*, 1997; Jetten *et al.*, 2003).

The properties of a given soil change over a period of a few weeks or months due to crust development (Poesen, 1981; Bryan *et al.*, 1989). In an agricultural context, the soil surface

evolves from a seedbed (loose surface layer composed of clods and macro-aggregates) to successive stages of crusting that correspond to different types of crust (Bresson & Boiffin, 1990), such as the structural crust (i.e., a thin surface layer where the micro-aggregates resulting from the breakdown of surface clods are sealed together) and the sedimentary crust (i.e., a thick and compact surface layer where the surface pores and micro-depressions are filled by small fragments resulting from the erosion and sedimentation processes). The presence of a crust can induce notable differences between the plow layer properties and the soil surface properties. Numerous studies show that the infiltration capacities can be very different between the crust and the underlying material (e.g., Hairsine & Hook, 1994; Morin & Van Winkel, 1996). However, few studies have addressed the effect of a crust on erodibility (McIntyre, 1958; Poesen, 1981; Kuhn & Bryan, 2004; Darboux & Le Bissonnais, 2007).

In fact, most of the studies using aggregate stability to assess erodibility are performed on samples collected inside the plow layer (e.g., Bullock *et al.*, 1988; Bajracharya & Lal, 1998; Barthès & Roose, 2002; Legout *et al.*, 2005), notwithstanding that interrill erosion occurs at the soil surface and thus depends directly on the erodibility of the crust and not on the erodibility of the plow layer material. For a clay loam soil, Darboux and Le Bissonnais (2007) demonstrated notable differences in aggregate stability between a sedimentary crust and a seedbed material (without crust). This finding led these researchers to conclude that estimations of erodibility performed on material collected from the plow layer may be invalid for the crust, resulting in potential bias in the estimated erodibility. However, the conclusions of this laboratory experiment had a limited scope. Moreover, the study did not attempt to assess the factors responsible for the differences in aggregate stability, while numerous factors have previously been identified as affecting aggregate stability (Amézqueta, 1999). For example, aggregate stability increases with the soil clay content and decreases with the soil

silt content (Wischmeier & Mannering, 1969). Organic matter content is recognized to have a positive effect on aggregate stability (Tisdall & Oades, 1982; Piccolo & Mbagwu, 1999). The soil CEC, linked to soil organic matter content and mineralogy, is positively correlated with aggregate stability (Zhang & Horn, 2001).

For the present study, a field study was conducted in a limited area of the Loess Plateau (China). The crusts and the underlying materials were sampled in areas designated for different land uses. Aggregate stability was measured as a proxy of soil erodibility. The standard soil properties known to be related to aggregate stability were also measured. The goals were 1) to assess the variability of aggregate stability for crusted soils in a limited area and 2) to relate this variability to the aggregate stability of the underlying material and to the standard soil properties. The consequences for erodibility assessment and erosion modeling are discussed.

4. Material and methods

4.1. Sampling sites

The Chinese Loess Plateau (northwest China) is recognized as the largest deposit of loess in the world. Silt particles resulting from the wind erosion of the Tibetan Plateau accumulated during the Quaternary glacial periods (from 2.5 million years ago) to an average thickness of 150 meters. The silt loam soils that developed on this substrate are very homogeneous in both texture and chemical properties and are recognized to be very sensitive to erosion processes (Zheng, 2005). Soil samples from seven field sites with different land uses were collected in the Ziwuling area in the hilly-gully region of the Loess Plateau (Table 1). The Ziwuling site altitude varies between 1100 and 1300 meters. The average annual temperature is 9°C, and the average annual precipitation is 577 mm. Precipitation occurs mainly from June to

September, which accounts for 60% to 70% of the total annual precipitation (Zheng *et al.*, 1994).

The studied sites were geographically close together. Sites A, B, C and D were located in a 7.5 km radius. Sites D1, D2, D3 and D4 were located along the erosion sequence of a 200-meter-long hillslope. The sampling was performed in September 2009 over a period of three consecutive days, beginning four days after the last rain event.

Table 1: Site localizations and land uses

Site	Geographic location (latitude; longitude)	Land-use and slope position	Slope gradient
A	36°03.888' N; 109°12.621' E	Cultivated corn field, upslope	5° - 10°
B	36°03.874' N; 109°12.675' E	Apple orchard, shoulder of a terrace	5° - 30°
C	36°04.227' N; 109°11.226' E	Cultivated radish crop, middle slope, sampling in ridges and furrows	5° - 13°
D1	36°05.149' N; 109°8.958' E	Ziwuling experimental station, bare soil, upslope, interrill area	5° - 10°
D2	36°05.431' N; 109°8.951' E	Ziwuling experimental station, bare soil, mid-slope, rill area	30° - 35°
D3	36°05.450' N; 109°8.947' E	Ziwuling experimental station, bare soil, 20 m from foot slope, ephemeral gully area	25° - 35°
D4	36°05.460' N; 109°8.884' E	Ziwuling experimental station, bare soil, 10 m from foot slope, gully area	35° - 40°

4.2. Sampling method

For each site, five plots (one square meter each) were defined to collect samples. For site D, samples were collected on the gully and rill sides, not in the gully and rill centers. Prior to sampling, the soil surface was described, and the crust type was identified (Bresson & Boiffin, 1990; Belnap *et al.*, 2008). The soil surfaces did not present mosses or lichens and had a light color, indicating a low level of cyanobacterial development (Belnap *et al.*, 2008).

Paired samples (crust and underlying material) were collected from each plot. The crusts were collected separately from the underlying material (hereafter referred to as “under-crust”). All of the sites had a structural crust, but only site C had a sedimentary crust. Therefore, only structural crusts are considered hereafter. Because the lower limit of the structural crusts was indefinite, a thickness of approximately 5 mm was considered to be the limit. The under-crust was defined as the soil material between -1 cm and -5 cm (from the

initial soil surface). In all cases, large pieces of the sampled material were collected using a sharp knife to cut through the material without affecting its structure.

4.3. Measurements

4.3.1. Aggregate stability

Aggregate stability was measured using a slightly-modified version of Le Bissonnais' method (Le Bissonnais, 1996; ISO/DIS 10930, 2012). Specifically, air-dried samples were cut into 2-5 mm fragments.

The three stability tests of Le Bissonnais (1996) (fast wetting, slow wetting and stirring) were designed to reproduce the processes involved in crust formation and interrill erosion (slaking, differential clay swelling and mechanical breakdown). The 5 g sub-samples were dried at 40°C for 24 h before application of a test, and each test was replicated twice. After the tests, the resulting fragments were sieved in ethanol. The results are presented using the mean weighted diameter (MWD). Each MWD value corresponds to one of five classes of stability: MWD above 2 mm corresponds to very stable material (very low erodibility), between 2 and 1.3 mm corresponds to stable material (low erodibility), between 1.3 and 0.8 mm corresponds to median stability (median erodibility), between 0.8 and 0.4 mm corresponds to unstable material (high erodibility), and lower than 0.4 mm corresponds to very low stability (very high erodibility) (Le Bissonnais, 1996).

4.3.2. Standard soil properties

The standard soil properties were measured to explain the stability differences between the sites and between the crust and under-crust. The soil properties that are assessed on a regular basis by soil scientists and known to be related to aggregate stability were measured: soil texture (using laser diffraction granulometry, Mastersizer 2000, Malvern Instruments Ltd.), soil organic matter content (Walkey & Black, 1934), cation-exchange capacity (CEC)

(Ammonium rapid method; Mackenzie, 1951) and pH (1:2.5 soil:water ratio, using a pH meter).

4.4. Statistical analysis

Statistical analyses were performed using version 2.9.2 of software R (R Development Core Team, 2011). To identify the differences between the sites and between the sampled materials, paired comparisons between the materials were performed with the Wilcoxon method on the MWD and the standard soil properties. We considered a threshold of 5% to be significant. The variability of the soil properties was quantified using the coefficient of variation, which is a normalized measure of dispersion. Linear correlation analyses (Pearson's coefficient) were performed to identify the standard soil properties related with aggregate stability. If the Pearson's coefficient was above 0.33 or below -0.33, we considered the relationship to be significant. The standard soil properties found to be significantly related to the MWD were combined through step-up multiple regression analyses to predict the MWD.

5. Results

5.1. Variability of the aggregate stability

Considering the mean of the three stability tests, the MWD of the crust among the sites varied between 0.33 mm (site C) and 2.04 mm (site D1), with a variation coefficient of 37% (Table 2a). Of all the stability tests on the crust samples, sites A (cultivated corn field) and D1 (Ziwuling exp. Station, interrill area) presented the highest MWD, whereas site C (cultivated radish) presented the lowest MWD (Figure 1).

Table 2: Variability of the mean weighted diameter (a) among the sampling sites (inter-site variability) for all stability tests, and (b) for each site (intra-site variability) for the mean of the 3 stability tests

(a)

Stability test	MWD of the crust					MWD of the under-crust				
	Min. (mm)	Max. (mm)	Mean (mm)	σ (mm)	CV	Min. (mm)	Max. (mm)	Mean (mm)	σ (mm)	CV
Fast wetting	0.20	1.62	0.98	0.41	0.42	0.13	0.95	0.36	0.18	0.51
Slow wetting	0.41	2.22	1.47	0.52	0.36	0.22	1.93	0.97	0.52	0.54
Stirring	0.29	1.77	1.14	0.41	0.39	0.23	1.23	0.69	0.27	0.39
Mean of the 3 tests	0.33	2.04	1.20	0.44	0.37	0.23	1.42	0.68	0.32	0.47

σ : standard deviation; CV: coefficient of variation.

(b)

Site	MWD of the crust					MWD of the under-crust				
	Min. (mm)	Max. (mm)	Mean (mm)	σ (mm)	CV	Min. (mm)	Max. (mm)	Mean (mm)	σ (mm)	CV
A	1.51	1.76	1.66	0.09	0.05	0.74	0.98	0.86	0.09	0.11
B	0.85	1.74	1.23	0.34	0.26	0.40	0.56	0.47	0.07	0.15
C	0.33	0.50	0.37	0.08	0.22	0.18	0.23	0.21	0.02	0.12
D1	1.43	2.04	1.63	0.29	0.18	0.79	1.42	1.11	0.23	0.21
D2	0.91	1.38	1.23	0.19	0.15	0.70	1.39	0.92	0.30	0.33
D3	0.85	1.03	0.93	0.07	0.08	0.63	0.83	0.75	0.10	0.13
D4	1.20	1.49	1.35	0.12	0.09	0.31	0.49	0.42	0.07	0.17

σ : standard deviation; CV: coefficient of variation.

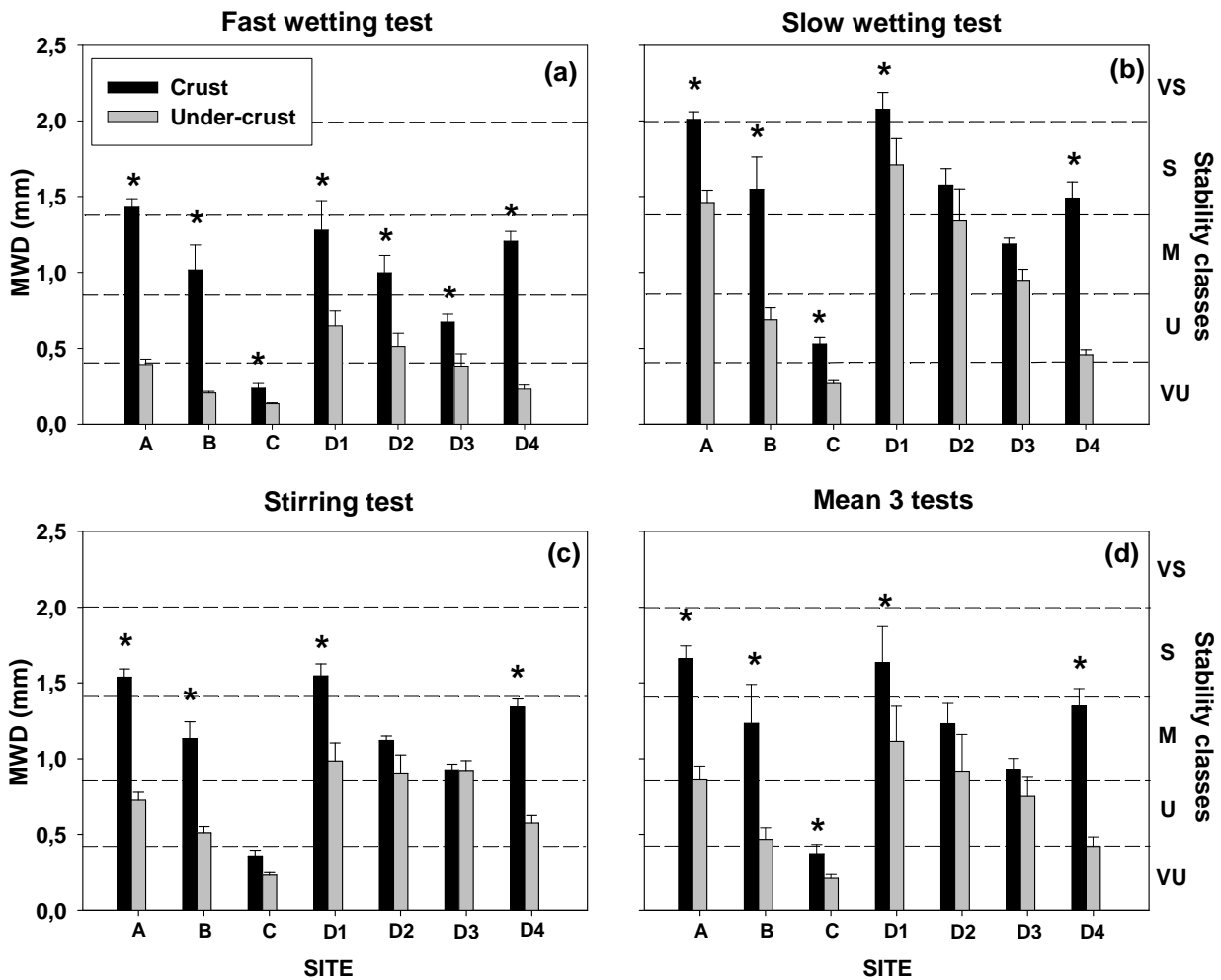


Figure 1: Aggregate stability of crusts and under-crusts for (a) fast wetting, (b) slow wetting, (c) stirring tests and (d) the mean of the 3 tests for all sites.

Each MWD corresponds to the mean of 5 plots with 2 replicates each, $n=10$. Bars represent standard errors.

*: difference statistically significant at $p < 0.05$ between crust and under-crust (Wilcoxon test, $\alpha=5\%$).

VS: very stable; S: stable; M: medium; U: unstable; VU: very unstable (Le Bissonnais, 1996).

Considering the mean of the three stability tests, the MWD of the under-crust samples varied between 0.23 mm (site C) and 1.42 mm (site D1), with a variation coefficient of 47% (Table 2a). Of all the stability tests on the under-crust samples, site D1 (Ziwuling exp. Station, interrill area) presented the highest MWD, whereas site C (cultivated radish) presented the lowest MWD (Figure 1).

For each site, samples were collected from 5 plots to consider intra-site variability. Considering the mean of the three stability tests, the intra-site variation coefficients were larger for the under-crust samples than for the crust samples for sites A, D1, D2, D3 and D4 (Table 2b). Among the sites, the variation coefficients were larger for the under-crust samples than for the crust samples, except for the stirring test (Table 2a).

5.2. Comparison of aggregate stability for paired crust and under-crust samples

For most of the paired samples, the aggregate stability of the crust was larger than the aggregate stability of the corresponding under-crust, and the under-crust samples were never more stable than the corresponding crust (Figure 1). A correlation analysis was performed to study the relationships between the MWD of the crust and the MWD of the under-crust material. An analysis was performed for each aggregate stability test and for the mean of the three tests. The highest correlation coefficient ($r=0.69$) was found between the MWD of the crust and the MWD of the under-crust for the slow wetting test. For the other tests, the correlation coefficients were 0.43 (fast wetting test), 0.48 (stirring test) and 0.59 (mean of the three tests). However, these correlation coefficients were greatly influenced by the very low MWD of site C, hence making site C a hot spot. Without site C, the correlation coefficients were only 0.52 (slow wetting), 0.20 (fast wetting), -0.06 (stirring) and 0.28 (mean of the three tests).

While the difference in aggregate stability between a crust and its under-crust was always in the same direction, the amplitude of this difference varied greatly both for a given site and among the sites. For example, for the mean of the three tests, the inter-site coefficient of variation was 0.60 (Table 3a), while the intra-site coefficients of variation ranged from 0.16 (site D4) to 0.90 (site D1) (Table 3b).

Table 3: Variability of the difference in mean weighted diameter between crust and under-crust (a) among the sampling sites (inter-site variability) for all stability tests, and (b) for each site (intra-site variability) for the mean of the 3 stability tests

(a)

Stability test	Difference in MWD between crust and under-crust				
	Min. (mm)	Max. (mm)	Mean (mm)	σ (mm)	CV
Fast wetting	0.10	1.04	0.62	0.35	0.56
Slow wetting	0.24	1.03	0.50	0.30	0.60
Stirring	0.00	0.81	0.45	0.32	0.71
Mean of the 3 tests	0.16	0.93	0.46	0.28	0.60

σ : standard deviation; CV: coefficient of variation.

(b)

Site	Difference in MWD between crust and under-crust				
	Min. (mm)	Max. (mm)	Mean (mm)	σ (mm)	CV
A	0.61	0.90	0.80	0.12	0.22
B	0.44	1.24	0.77	0.30	0.39
C	0.09	0.27	0.16	0.07	0.45
D1	0.08	1.25	0.52	0.47	0.90
D2	-0.08	0.59	0.31	0.27	0.87
D3	0.05	0.40	0.18	0.14	0.76
D4	0.83	1.13	0.93	0.15	0.16

σ : standard deviation; CV: coefficient of variation.

5.3. Variability of standard soil properties

The samples showed silt content between 65.5% and 73.1% and clay content between 10.0% and 14.4% (Figure 2) and thus belonged to the silt loam texture class (Soil Survey Division Staff, 1993). Clay, silt and sand contents did not differ significantly between the sampling sites. The organic matter content varied between 0.7% (site D4) and 2.2% (site B) (Figure 3a). The CEC varied between 15.6 mEq/100 g (site C) and 30.7 mEq/100 g (site A) (Figure 3b). The organic matter content and the CEC differed significantly between the sites.

The pH, with values in the range of 8.3 to 8.6, did not differ significantly between the sites (Figure 3c).

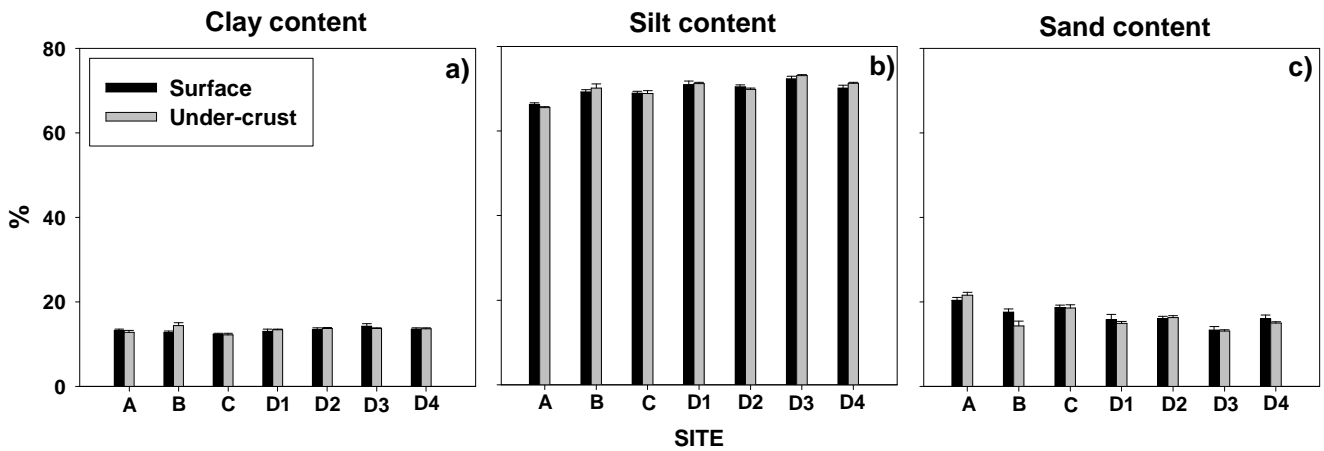


Figure 2: Crust and under-crust contents in (a) clay, (b) silt, and (c) sand for all sites.

The data from each site correspond to the mean of 5 plots with 2 replicates each, $n=10$. Bars represent standard errors.

*: difference statistically significant at $p<0.05$ between crust and under-crust (Wilcoxon test, $\alpha=5\%$).

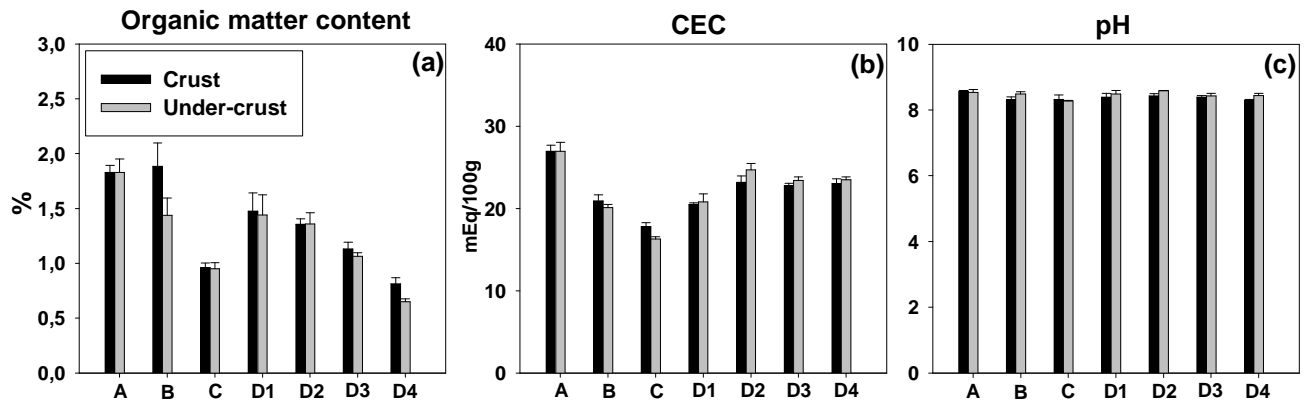


Figure 3: Crust and under-crust values for (a) organic matter content, (b) CEC and (c) pH, for all sites.

The data from each site correspond to the mean of 5 plots with 2 replicates each, $n=10$. Bars represent standard errors.

*: difference statistically significant at $p<0.05$ between crust and under-crust (Wilcoxon test, $\alpha=5\%$).

Finally, the percentages of clay, silt and sand (Figure 2), organic matter content, CEC and pH (Figure 3) did not differ significantly between a structural crust and its under-crust. The ranges of the standard soil properties were low compared to the variability of the aggregate stability.

5.4. Relationship between standard soil properties and aggregate stability

A correlation analysis was performed between the aggregate stability (MWD) and the standard soil properties taken as potential explanatory factors (Table 3). This analysis was performed on the crusts (Table 4a) and under-crusts (Table 4b) separately. In both cases, the highest correlation coefficients were found between the MWD of the slow wetting test and the organic matter content (0.57 and 0.56, respectively). In all cases, the CEC was significantly correlated with the MWD. For the crusts (Table 4a), pH was not significantly correlated with any of the MWD. For the under-crusts (Table 4b), pH was positively correlated with the MWD of the slow wetting test, the stirring test and the mean of the 3 stability tests. Finally, both for the crusts and under-crusts, the clay, silt and sand contents were not significantly correlated with any of the MWD.

A step-up multiple regression analysis was performed using the standard soil properties found to be significantly correlated to aggregate stability: organic matter content, CEC, and pH. For the crust, among all the tested combinations, the best regression was found for the mean MWD of the three tests as the dependent variable and for the organic matter content and CEC as the explanatory variables (moreover, the organic matter content and CEC are not independent from one another): $MWD_{\text{mean}}(\text{mm}) = 0.39(\pm 0.15) \times \text{OM}(\%) + 0.06(\pm 0.02) \times \text{CEC} - 0.66(\pm 0.47)$. The coefficient of determination (r^2) was 0.38, meaning that this model explained 38% of the variation of the mean of the three aggregate stability

tests. The residual standard error for the estimated MWD was 0.36 mm at the 95% confidence interval.

For the under-crust, among all the tested combinations, the most statistically meaningful regression was found for the MWD of the slow wetting test as the dependent variable and the organic matter content and pH as the explanatory variables: $MWD_{sw}(mm) = 0.69(\pm 0.17) \times OM(\%) + 1.15 (\pm 0.44) \times pH - 9.62(\pm 3.70)$. The coefficient of determination (r^2) was 0.40, meaning that this model explained 40% of the variation of the slow wetting test. The residual standard error for the estimated MWD was 0.43 mm at the 95% confidence interval.

Table 4: Correlations (Pearson's coefficient) between aggregate stability and standard soil properties (a) for the crusts and (b) for the under-crusts.

(a)

MWD	Organic matter	CEC	pH	Clay content	Silt content	Sand content
Fast wetting	0.50	0.50	0.11	0.08	-0.31	0.21
Slow wetting	0.57	0.46	0.22	0.09	-0.18	0.11
Stirring	0.42	0.56	0.20	0.14	-0.16	0.06
Mean of the 3 tests	0.52	0.52	0.18	-0.10	-0.22	0.13

$N = 35; \alpha = 5\%; r = 0.32$

(b)

MWD	Organic matter	CEC	pH	Clay content	Silt content	Sand content
Fast wetting	0.51	0.44	0.19	0.12	0.09	-0.07
Slow wetting	0.56	0.44	0.41	0.04	-0.12	0.11
Stirring	0.22	0.46	0.47	0.28	0.29	-0.29
Mean of the 3 tests	0.49	0.48	0.41	0.13	0.03	-0.04

$N = 35; \alpha = 5\%; r = 0.32$

5.5. Relationship between standard soil properties and the difference in aggregate stability between crust and under-crust

A linear correlation analysis was performed to further attempt to link the differences in MWD between the crust and the under-crust materials to the standard soil properties (Table 5). Potential explanatory factors were the standard soil properties (as before) but also the difference between the crust and the under-crust for a given soil property. The differences in stability between the crust and the under-crust materials for the fast wetting test were positively correlated with (a) the crust organic matter content, (b) the crust and the under-crust CEC, and (c) the crust sand content but were negatively correlated with the crust silt content. For the slow wetting test, the MWD difference between the crust and under-crust materials was positively correlated with the differences in organic matter and sand content between the crust and under-crust but negatively correlated with the difference in clay content between the crust and under-crust. For the stirring test, the MWD difference between the crust and the under-crust materials was positively correlated with the crust organic matter, the crust CEC and the crust sand content but negatively correlated with the crust silt content. Considering the mean of the three stability tests, the MWD difference between the crust and the under-crust materials was positively correlated with the crust organic matter, the sand content and the difference in organic matter between the crust and under-crust but negatively correlated with the crust silt content.

A step-up multiple regression analysis was performed using the difference in aggregate stability between the crust and under-crust materials as the dependent variable and the standard soil properties and the differences between each property for the crust and under-crust as the explanatory variables. No statistically meaningful relationship was found.

Table 5: Correlations (Pearson's coefficient) between the differences in aggregate stability between crust and under-crust and the standard soil properties.

Difference in MWD	Organic matter			CEC			pH			Clay content			Silt Content			Sand content		
	C	U	C-U	C	U	C-U	C	U	C-U	C	U	C-U	C	U	C-U	C	U	C-U
Fast wetting	0.40	0.27	0.29	0.45	0.45	-0.16	0.04	0.16	0.09	-0.07	0.20	-0.21	-0.46	-0.26	-0.23	0.37	0.16	0.23
Slow wetting	0.18	-0.01	0.42	0.12	0.15	-0.10	-0.19	-0.07	-0.11	-0.21	0.25	-0.35	-0.22	-0.04	-0.25	0.23	-0.06	0.36
Stirring	0.40	0.29	0.29	0.35	0.33	-0.07	-0.01	-0.28	-0.06	-0.19	0.09	-0.21	-0.48	-0.32	-0.16	0.42	0.23	0.19
Mean of the 3tests	0.34	0.19	0.37	0.33	0.33	-0.12	-0.06	0.05	-0.10	-0.17	0.20	-0.28	-0.41	0.22	-0.23	0.36	0.11	0.28

C=crust; U=under-crust; C-U=difference in soil property value between the crust and the under-crust. N= 35; $\alpha=5\%$; $r = 0.32$

6. Discussion

6.1. The aggregate stability of a crust is different from the aggregate stability of its under-crust material

For a given site, large differences in aggregate stability were found between the crust and the under-crust. The differences in aggregate stability between the crust and under-crust were always in the same direction; the crust aggregate stability was always larger than or equal to the aggregate stability of its under-crust. No under-crust sample was more stable than its corresponding crust sample. Differences in aggregate stability as a function of the crusting stage were drawn by Darboux and Le Bissonnais (2007). Through a lab experiment, they measured the aggregate stability of a seedbed (non-crust, initial material), a structural crust and a sedimentary crust. The data of Darboux and Le Bissonnais (2007) showed that sedimentary crusts tended to be more stable than their original material, while the stability of structural crusts remained quite similar to the stability of the seedbed. In the present study, the

structural crusts were generally more stable than the underlying material, irrespective of the sampling site and the stability test. The differences between the results of these two studies may lie in the experimental settings: the study of Darboux and Le Bissonnais (2007) was performed in a laboratory (thus using well-controlled experimental conditions) and used a soil with a different texture (11% clay, 58% silt and 31% sand).

The variability of the aggregate stability measured in the crust samples was lower than the variability measured in the under-crust samples. This situation was consistently encountered in the inter-site comparison and often observed in the intra-site comparison. This finding may mean that the development of the crust decreased the spatial variability of the aggregate stability.

6.2. Standard soil properties do not adequately predict aggregate stability

We looked for relationships between the aggregate stability and the standard soil properties (organic matter content, clay content, silt content, sand content, CEC and pH) because 1) these variables are measured routinely for soils and 2) relationships with aggregate stability have frequently been reported in the literature (Wischmeier and Mannering, 1969; Tisdall and Oades, 1982; Amézketa, 1999; Zhang and Horn, 2001). Hence, these variables could be assumed to be suitable explanatory factors for the differences in aggregate stabilities between the crust and under-crust materials of a given site and also between sites.

The test area was very homogeneous based on the standard soil properties; the soil texture and pH were almost constant among the sites, while the organic matter content and CEC did not change much. Accordingly, one may have expected that the homogeneity of standard soil properties in the test area would have led to homogeneity in the aggregate stability, but this was clearly not the case.

For a given site, the standard soil properties could not explain the differences in aggregate stability between a crust and its under-crust. Indeed, the standard soil properties were very similar between the crust and under-crust materials, while the stability of the crust tended to be very different from the stability of its under-crust.

Among the sites, even if the standard soil properties showed a low heterogeneity, correlations existed among the aggregate stabilities. However, none of these standard soil properties (or combination of standard soil properties) was able to satisfactorily predict the aggregate stability of the crusts or under-crusts; the statistical relationships explained 40% of the variations at most, and with a residual standard error of approximately 0.4 mm, the predicted MWD values could be flawed by as much as two stability classes (out of five stability classes) (Figure 1). Consequently, these relationships had no practical capacity of prediction, and using these relationships in the predictions would likely lead to large flaws in the interpretations.

Land use may have caused the large differences in stability among the sites without affecting the standard soil properties. The results strongly indicate that other variables should be considered. In future studies, additional variables may need to be examined, such as the organic matter quality, microbial activity, wetting-drying cycles and crust formation processes. These variables are known to be related to aggregate stability (Amézqueta, 1999) but are not commonly measured. Because the crust and under-crust originate from the same initial material, the resulting differences in stability had necessarily accumulated over time. This finding advocates for a time-monitoring of the aggregate stability and the aforementioned variables in both the crust and under-crust.

6.3. Consequences for erodibility assessment and erosion modeling

Aggregate stability is one of the methods used to assess soil erodibility (Bajracharya *et al.*, 1992; Barthès & Roose, 2002), with a low aggregate stability corresponding to a high erodibility and vice versa. When used for erodibility assessment, aggregate stability is usually measured in the under-crust material (e.g., Bullock *et al.*, 1988; Bajracharya and Lal, 1998; Barthès & Roose, 2002; Legout *et al.*, 2005). In the present study, large differences in aggregate stability were found between the crust and under-crust materials of a given site. From the results, we can infer the existence of large differences in erodibility for a given site, with the crust being generally less erodible than the under-crust. This finding strongly suggests that erodibility should be assessed on the exact material actually exposed to erosive forces: the soil surface. In the present case, the common practice of using the underlying material – instead of the crust – would cause an overestimation by at least one erodibility class in 60% of cases and by at least two erodibility classes in 30% of cases (Figure 1). Accordingly, assessing the erodibility on the underlying material leads to a large bias, and this bias is clearly an overestimation of the soil erodibility.

In current erosion models, erodibility is assessed for a given soil type (Gumiere *et al.*, 2009). Because a single soil type was investigated in the present study, a similar erodibility could have been expected. This outcome was clearly not the case: Erodibility can vary greatly in space, even for a given soil. This finding questions our general ability to adequately assess erodibility. Currently, parameterization of erosion models sets a unique erodibility value for a given soil and thus does not consider the variability of erodibility within a given soil. This oversimplification could explain part of the large inaccuracy in the predicted results of erosion models (Jetten *et al.*, 2003; Boardman, 2006; Vigiak *et al.*, 2006).

Comparisons between the sites showed that the variability of the crust was lower than the variability of the under-crust. Using crust samples for erodibility assessment, instead of under-crust samples, would decrease the heterogeneity of the mapped erodibility (although this heterogeneity would remain large).

Based on the current knowledge, erodibility assessment should be performed with a very high density of measurements, which could be impractical, leaving the construction of a sound erodibility map currently impractical. Indeed, the large variability of erodibility appears to be a difficult problem to confront because the variability of erodibility could not be explained by the variability of the standard soil properties. New variables should be investigated to accurately predict erodibility. Factors that affect the erodibility of the soil surface should be better understood so that reliable erodibility maps can be produced from a reasonably small set of measurements.

7. Conclusions

Crusts showed an aggregate stability that was generally higher than their underlying material. This finding emphasizes the importance of estimating soil erodibility on the exact material that is exposed to erosive forces, i.e., the soil surface material. On a crusted soil, the use of material collected from the plow layer may lead to greatly mis-estimated erodibility and thus bias the results of the erosion models. The large variability in aggregate stability among sites proves that erodibility can be greatly variable in space, even when considering a small test area and a single soil type with homogeneous standard soil properties. These standard soil properties were not able to accurately predict the observed differences in aggregate stability. Clarifying the causes of aggregate stability variability and improving the parameterization of erodibility in soil erosion models will require further research to account for other controlling factors, such as 1) the organic matter quality and the biological activity,

2) the soil hydric history linked to local climatic conditions, and 3) the physical processes involved in crust formation.

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Chapitre 2 :

Synthèse bibliographique des processus physico-chimiques affectant la stabilité structurale

**Physico-chemical processes affecting soil aggregate stability:
a review**

Baptiste ALGAYER *et al.*

In preparation

Abstract

Aggregate stability corresponds to the ability of an aggregate to keep its size and not break up into smaller particles when it is submitted to wetting. It is considered as a key property that affects the movement and storage of water, biological activity, plant development, and the soil sensitivity to erosion. Aggregate stability was found to be temporally dynamic by several studies, incriminating various processes in the observed variations. The processes implied in aggregate stability variation are related to biological activity but are also abiotic processes related to soil water content variations. If the processes linked to biological activity have been widely studied, predictions of aggregate stability still remain inaccurate, and abiotic processes are often incriminated to precise the predictions. A bibliographical review has been performed with the aim to inventory the physical chemical processes involve in aggregate stability variation cited in the literature. The objectives were to assess the state of art concerning physical chemical processes of aggregate stability variation, and give directions of further investigations. Abiotic processes act at two scales. The identified processes acting at the macro-aggregate scale were slaking, raindrop impact, freezing-thawing, and differential swelling of the clays. Processes acting at the micro-aggregate scale were: particle rearrangement and clay redistribution, friction, clay dispersion/flocculation, crystallization/dissolution and thixotropic age hardening. Among those abiotic processes, particle rearrangement, crystallization/dissolution and thixotropic age hardening were identified to need more researches and knowledge. Moreover, the identified processes interact closely, and the effects of those interactions still remain unknown. Understanding the mechanical aspects of such processes separately would be useful to understand the interaction between abiotic and biotic processes and finally permit to predict aggregate stability variations accurately.

1. Introduction

1.1. Soil structure and aggregate stability

Soil is a complex and heterogeneous material composed of four phases that interact closely (mineral, organic, liquid and gaseous). Soil structure refers to the size, the shape and the arrangement of solids and voids in the soil matrix (Lal, 1991). The most common element of soil structure is the aggregate. An aggregate is a secondary particle (cluster) formed by the combination of mineral particles with organic and inorganic substances in such a way that the strength of the forces combining the particles within the aggregate exceeds external forces applied from the environment in which the aggregate exists (Farres, 1980; Kemper & Rosenau, 1986).

Several models have been proposed to describe the way in which individual mineral particles are held together to form aggregates (Edwards & Bremner, 1967; Tisdall & Oades, 1982; Amézkéta, 1999; Bronick et al., 2005; Six *et al.*, 2006). In those models, aggregates are grouped by sizes, differing in properties such as the nature and mechanisms of the binding agents and the resistance of the bonds. The most cited model has been proposed by Tisdall & Oades (1982). Such model can be applied to soils where organic matter is the main bonding agent, and can be summarized by the following description. Size of the soil mineral particles, soil biota and hierarchical levels of aggregation are presented in table 1. The lowest hierarchical order concerns particles with size lower than 2 μm such as flocs of clay particles. These < 2 μm nano-aggregates consist of organic molecules (OM) attached to clay particle (Cl) and polyvalent cations (P): (Cl – P – OM) (Edwards & Bremner, 1967; Tisdall & Oades, 1982), and of plate particles are held together by Van der Waals forces, hydrogen bonding, and coulombic attractions (Edwards & Bremner, 1967; Tisdall & Oades, 1982; Bronick & Lal, 2005). Microbial biomass acts at this scale with bacterial populations developing on clay platelets (Tisdall & Oades, 1982). These (Cl – P – OM) nano-aggregates are joined together to

form the higher hierarchical stage of aggregation: [(Cl – P – OM)_x]_y. This stage of aggregation consists in aggregates between 2 and 250µm diameter composed of particles bonded together by various cements including persistent organic materials and crystalline oxides. Silt and sand particles can be joined to each other by micro-aggregates to form macro-aggregates > 250 µm diameter (Edwards & Bremner, 1967; Tisdall & Oades, 1992). Macro-aggregates (> 250 µm) are very porous and are composed of micro-aggregates (< 250 µm) joined together by clay bridges or enmeshed by a fine network of roots and hyphae (Tisdall & Oades, 1982). Bonds within micro-aggregates are stronger than bonds within macro-aggregates (Edwards & Bremner, 1967), and if a lower level of aggregate is destroyed, the higher levels are also destroyed.

Table 1: size of the soil mineral particles, soil biota and hierarchical levels of aggregation.

Scale	Mineral particles	Biota	Aggregation
0,1 nm	Atoms		
1 nm	<i>Clay platelet</i>	<i>organic molecule</i>	
10 nm	Clays	<i>polysaccharides</i>	Clay domain Nano-aggregates
0,1 µm		<i>Bacteria</i>	
1 µm	Silt	<i>Fungal hyphae</i>	2 µm Micro aggregates
10 µm		<i>Root hair</i>	
100 µm	Sand	<i>Meso fauna</i>	250 µm Macro aggregates
1 mm		<i>Worms</i> <i>Insects</i>	
	Gravels		
1 cm	Stones		Clods

Aggregate stability corresponds to the capacity of a soil aggregate to keep its size and not break up into smaller fragments when it is submitted to an exogenous stress such as wetting. Stability of aggregates is a function of whether the cohesive forces between particles withstand the applied disruptive force (Kemper & Rosenau, 1986). The stability of aggregates and the pores between them affects the movement and storage of water, soil carbon sequestration and biological activity (Six *et al.*, 2000), plant growth and developments by influencing plant emergence and root penetration (Gallardo-Carrera *et al.*, 2007). Finally, it also affects soil sensitivity to erosion and crusting (Le Bissonnais, 1996; Bajracharya *et al.*, 1998; Barthès & Roose, 2002).

1.2. Factors and processes of aggregate stability variation

Many studies showed that aggregate stability changed with time (e.g. Bullock *et al.*, 1988; Chan *et al.*, 1994; Bajracharya *et al.*, 1998; Deneff *et al.*, 2001; Cosentino *et al.*, 2006; Dimoyiannis, 2009). These temporal variations were observed by field monitoring and laboratory experiments. Field monitoring performed at monthly time step identified seasonal trends of aggregate stability variation following a nearly cyclic pattern with the greatest values during summer and the lowest values during winter (Bullock *et al.*, 1988; Chan *et al.*, 1994; Dimoyiannis, 2009). Laboratory experiments allowed identifying temporal variation of aggregate stability at shorter time step. Temporal variations of aggregate stability have been shown at weekly time step in relationship with wetting and drying cycles (e.g. Utomo & Dexter, 1982; Deneff *et al.*, 2001) and biological activity stimulation (e.g. Deneff *et al.*, 2001; Cosentino *et al.*, 2006; Abiven *et al.*, 2007). If such studies allowed identifying factors and trends of aggregate stability variations, there are still difficulties to predict aggregate stability variations suggesting that aggregate stability is a complex function.

Numerous factors are involved in aggregate stability variation (Amézketa, 1999; Bronick & Lal, 2005). Several bibliographic reviews list the factors of aggregate stability

variation and group them into different types (e. g. Kay, 1990; Amézketa 1999; Six *et al.*, 2004; Bronick & Lal, 2005). Amézketa (1999) and Bronick & Lal (2005) distinguished internal factors including soil properties (electrolyte, clay mineralogy, organic matter, (hydr)oxydes) and external factors (climate, time, biological factor, and agricultural management). Six *et al.* (2004) opposed the organic binding agents to the inorganic binding agents, both exposed to environmental variables (wetting-drying cycles, freezing-thawing cycles). Factors of aggregate stability are involved through different processes. We can distinguish biotic processes, linked to biological activity involving organic binding/fragmentation agents, and abiotic processes, linked to climate and involving inorganic binding/fragmentation agents. From 1970 to the beginning of the 90's, the proportion of studies treating about biotic processes of aggregate stability variation have increased drastically in comparison with studies about abiotic processes of aggregate stability variation (figure 1). Studies about biotic factors showed that biological activity and organic matter dynamics affect aggregate stability through different processes (Bronick & Lal, 2005). Fungal hyphae and root hair enmeshed particles at the macro-aggregate scale (Degens *et al.*, 1994). The decay of organic matter by biological activity produces polysaccharides that bind clay particles and stabilize the structure at the micro-aggregate scale (Tisdall & Oades, 1982; Chenu & Guérif, 1991). Organic hydrophobic coatings on mineral particles decrease the wetting rate and the negative effect of wetting on aggregate stability (Cosentino *et al.*, 2006; Goebel *et al.*, 2012). Such researches were based on stimulations of biological activity by organic amendments and showed that organic amendments had positive effect on aggregate stability. However, they could not accurately predict aggregate stability variation for a given soil, especially when organic matter content was low (<5%) and when microbial activity was not stimulated by organic amendments. Field experiments showed mild correlations between aggregate stability and the temporal variation of the organic matter content or biological

activity indices, explaining at best 50% of the aggregate stability variation (Chan *et al.* 1994; Bajracharya *et al.*, 1998; Dimoyiannis 2009). Better aggregate stability prediction requires more detailed researches on biological processes or incriminated abiotic processes.

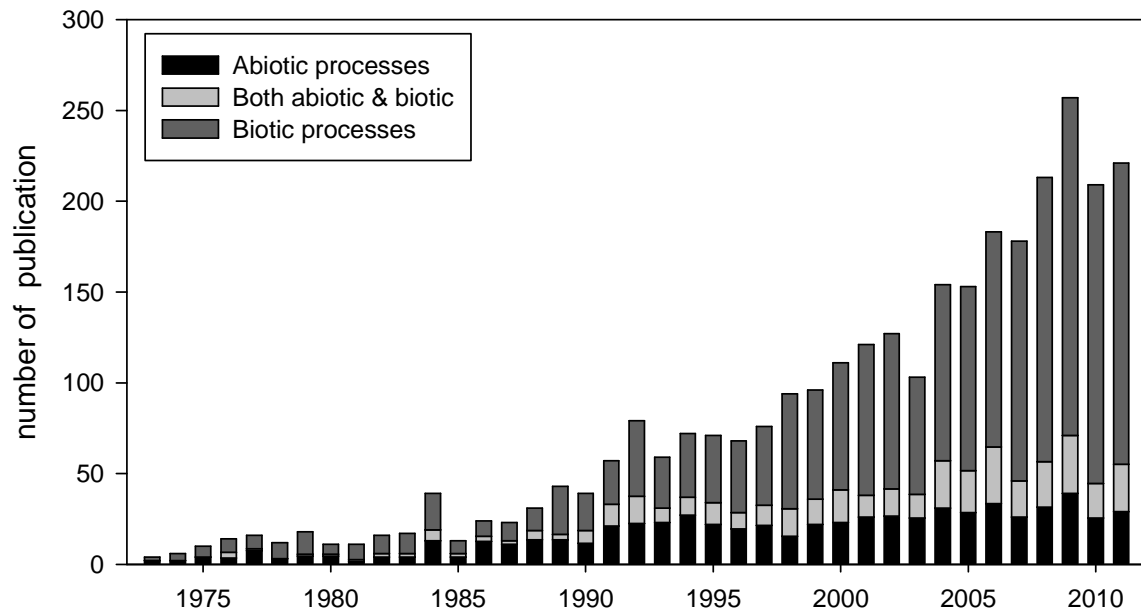


Figure 1: Evolution of the number of publications citing the processes of aggregate stability variation. Comparison between citations occurrences of the biotic and abiotic processes incriminated in aggregate stability variation (between 1970 and 2011). Bibliometric study performed on October 1st 2012, using the platform Web of knowledge editor database. The number of publication corresponds to the number of publication citing the processes in their abstract or keywords.

Abiotic (or physico-chemical) processes of aggregate stability variation have been studied since the beginning of the last century (e.g. Yoder, 1936; Hénin, 1939). Those studies allowed understanding the influence of water on aggregate stability through various physico-chemical processes acting at different levels such as clay dispersion and slaking. Many processes have been incriminated and theorized. Some of those processes have been concretely observed and understood, while others have only been hypothesized.

This bibliographical review aims at inventorying the physical and chemical processes involved in aggregate stability variation and the method used to characterize them. The objectives of the present work were 1) to assess the state of the art concerning physico-chemical processes of aggregate stability variation, 2) to point the need in knowledge and 3) to give directions for further investigations. First, we present the inventory of the abiotic processes found in the literature to affect aggregate stability. Secondly, we present the results of a bibliometric study that aimed assessing the state of the art and give directions for further investigations.

2. Physical and chemical processes involve in aggregate stability

The present review inventories the abiotic processes cited in literature as processes of aggregate stability variation. According to the mainly cited models of aggregation (Tisdall & Oades, 1982; Amézketa, 1999), we distinguished two scales in which processes affect aggregate stability: the macro-aggregate scale ($>250\ \mu\text{m}$) and the micro-aggregate scale ($<250\ \mu\text{m}$). Each process was described and the occurrence conditions are presented.

2.1. Macro-aggregate scale

The following processes affect the aggregate stability at the macro-aggregate scale ($>250\ \mu\text{m}$). Such processes affect the bonds within macro-aggregates and can lead to their breakdown into micro-aggregates.

2.1.1. Slaking

Slaking is caused by compression of air entrapped inside the aggregate during wetting (Yoder 1936; Hénin, 1939; Panabokke and Quirk, 1957; Hénin, 1958; Emerson, 1967; Le Bissonnais, 1996; Zaher *et al.*, 2005; Goebel *et al.*, 2012). When an aggregate is rapidly wetted, water enters the capillary pores that are filled with air. The inter particle water menisci only allowed part of the air contained within the pores to escape through the few unobstructed

(non wetted) capillaries. Most of the air remains trapped within the aggregate and is compressed by the incoming water. The rupture of the bonds between the particles occurs when the resulting internal pressure is great enough to overcome the aggregate cohesion (Yoder, 1936; Hénin, 1958; Grant and Dexter, 1990; Zaher *et al.* 2005). By an experimental study, Zaher *et al.* (2005) measured and modeled the internal air pressure variation, and the air release from aggregates submitted to immersion. The maximum measured pressure varied between 0.5 kPa and 1.3 kPa depending on the treatment, leading to the breakdown of aggregate within the first 8 seconds following immersion. This study allowed to observe directly the process of slaking and allowed to understand precisely its conditions of occurrence.

Slaking occurrence is controlled by the volume of air inside the aggregate and by the rate of wetting. The first condition for slaking occurrence is the presence of air within the pore system of the aggregate. This condition means an initial dried state of the aggregate. Many studies showed that humid aggregates were less sensitive to slaking than dried aggregate (e. g. Panabokke & Quirk, 1957; Perfect *et al.*, 1990). Humid aggregates contain less air volume to be compressed by the water entry, and thus, less pressure build-up to break the inter-particles bonds within the aggregate. Moreover, the wetting rate has to be high enough to prevent the air to escape from the aggregate (Panabokke & Quirk, 1957; Zaher *et al.*, 2005). The presence of organic hydrophobic substances that decrease wettability of soil aggregate was found to limit the slaking occurrence (Piccolo & Mbagwu, 1999; Chenu *et al.* 2000; Goebel *et al.* 2012). Slaking requires a state of quasi immersion of dry aggregates. This condition might limit the occurrence of slaking in the field. Indeed, the wetting rate has to be high and uniform on all the aggregate surface, and the surface aggregates have to be initially dry. Only important rain events during dry season can gather both conditions.

Slaking has been widely studied and controlling factors are identified. Recent studies allowed predicting its occurrence by modelisation (Zaher et al., 2005), and it can be measured by several tests (Le Bissonnais, 1996).

2.1.2. Raindrop impact

When soil is submitted to rain event or sprinkler irrigation, the water drop beats the surface aggregates. The impact can break the inter-particle bonds leading to a decrease of aggregate stability or to the aggregate breakdown. This is a totally physical process: the chemical properties of both soil and the applied water are not involved. Rain drop impact only affects aggregates at the soil surface.

Decrease in aggregate stability by the raindrop impact is controlled by the kinetic energy of the water drops. Aggregate stability decreases as droplet kinetic energy increases (Levy *et al.*, 1986; Lehrsch & Kincaid 2006). Kinetic energy of a water drop depends on the size of the drop and on the water drop speed when it impacts the soil surface. The higher are the size and the speed of the drop, the higher is the kinetic energy. Only rain with high kinetic energy can induce aggregate stability decreased by rain drop impact. Through a field experiment, Lehrsch & Kincaid (2006) tested different kinetic energy of water drops on soil aggregate stability. They showed that, for a silt loam, droplet energies superior to 8 J kg^{-1} decreased aggregate stability significantly. Farres (1980) also showed that kinetic energy was relevant factor of aggregate breakdown. However, he showed that frequency of impacts independent of the character of each impact was the best single component at predicting breakdown occurrence, and that the volume of water involved in impacts was as important as the energy of impacts for explaining the aggregate breakdown. Mechanical breakdown of aggregate by raindrop impact usually occurs in combination with other processes related to soil wetting (Imeson & Vis, 1984; Le Bissonnais, 1996; Ramos *et al.*, 2003).

Raindrop impact has been widely studied and controlling factors are identified. The resistance of aggregate structure against raindrop impact can be measured through laboratory experiments such as the water drop test (Low, 1965; Imeson & Vis, 1984) and the stirring test (Le Bissonnais, 1996).

2.1.3. Freezing- thawing

When a soil is frozen, the expansion of ice crystals induced by the freezing of water present in pores breaks particle to particle bonds leading to the decrease of aggregate stability with thawing (Bullock *et al.*, 1988; Lehrsch *et al.*, 1991). Numerous studies showed that aggregate stability was inversely proportional to soil water content at the time of freezing (Bullock *et al.*, 1988; Lehrsch *et al.*, 1991; Staricka & Benoit, 1995; Lehrsch, 1998; Oztas & Fayetorbay, 2003; Kvaerno & Oygarden, 2006). Aggregate stability was not found to be affected when aggregates were air dried before freezing (Bullock *et al.*, 1988; Oztas & Fayetorbay, 2003). At low water content, the ice crystals completed their growth in the pores without applying disruptive force on inter-particle bonds (Bullock *et al.*, 1988). Bullock *et al.* (1988) showed that aggregate stability decreased significantly when aggregates were frozen at water content greater than 0.20 kg.kg^{-1} . This threshold of water content was observed for a silt loam soil and a loam soil (Bullock *et al.*, 1988).

Freezing-thawing has been widely studied and controlling factors are identified. The resistance of aggregate structure against freezing-thawing can be measured through laboratory experiments using freezers (Bullock *et al.*, 1988; Staricka & Benoit, 1995; Lehrsch, 1998; Oztas & Fayetorbay, 2003).

2.1.4. Differential swelling of clay

Soil wetting is not a homogeneous process in time. During wetting, an aggregate present wetted and dry regions. Part of the clay minerals have the property to swell when

hydrated (2:1 clay minerals). Considering an aggregate containing 2:1 clay minerals, in the wetted regions of the aggregate, clay particles will swell appreciably while dry regions will keep the same volume. This process, called differential swelling, causes mechanical stresses between the dry and the wetted portions of the aggregate, causing the development of shear planes on the wetting front, breaking many of the bonds between particles (Kemper & Rosenau, 1986; Quirk & Murray, 1991). Differential swelling of clay during wetting and drying cycles result in micro-cracking of aggregates (Kheyrabi & Monnier, 1968).

The first condition for differential swelling occurrence is the amount of swelling clay. Aggregate breakdown by differential swelling was found to increase with increasing clay content (Le Bissonnais, 1996; Ruiz-Vera & Wu, 2006). Zhang & Horn (2001) found that differential swelling depended on clay mineralogy and its properties such as cation size, valence, ionic concentration and composition of the soil solution. More than clay content, clay mineralogy plays a preponderant role in differential swelling occurrence. Emerson (1977) calculated crystalline swelling of smectite (2:1 clay minerals) to be 25 times that of kaolinite (1:1 clay minerals). Ruiz-Vera & Wu (2006) observed that differential swelling was not the dominant process for aggregate stability decrease in soil where kaolinite was the main clay minerals. The presence of swelling clays is the necessary factor of differential swelling occurrence. Differential swelling can occur at low wetting rate, but is emphasized at high wetting rate (Le Bissonnais, 1996). Indeed, if an aggregate is quickly wetted, the hydration and expansion of swelling clay minerals happen quicker and the differential in swelling between the wetted and the dry regions of the aggregate is larger (Kemper & Rosenau, 1986). Mechanical stresses induced by differential swelling are more important than stresses induced by differential shrinkage during drying. Indeed, drying is a more progressive and homogeneous process at the aggregate scale, and thus, the differential in shrinkage is lower than the differential swelling induced by wetting process.

Differential swelling of clay has not been directly observed yet. This process has been deduced through experimental studies based on the comparison of swelling soils and non swelling soils during wetting and drying cycles. If micro-cracking has been observed by microscopy, there relation with differential swelling process still has to be proved.

2.2. Micro-aggregate scale

In order to better understand the following processes, it is necessary to remind the forces involved in inter-particle cohesion within micro-aggregates. Three main sources of cohesion exist within a granular media: water tension, inter-particle bonds, and inter-particle friction.

Water tension (also termed capillary forces) is linked to the presence of inter-particle water bridges (or menisci) within a humid aggregate. The water tension intensity is directly linked to the morphology of the inter-particle menisci (Sheel *et al.*, 2008). Capillary forces are null when soil is totally dry. It increases while wetting, until reaching a maximum, and then decreased to be null when soil is saturated. Inter-particle bonding needs the intervention of bonding agents (clay particles, mineral or organic colloids). The intensity of bonding is linked to the amount of bonding agents, their distribution within the soil matrix and their fixation properties. Inter particle friction appears as the least important source of cohesion involved in aggregate stability. Friction involves the morphology of mineral particles and their surface roughness and requires a direct contact between particles.

2.2.1. Particle rearrangement during wetting drying cycles

As described by Kemper & Rosenau (1986) and Dexter *et al.* (1988), the rearrangement of particles within the soil aggregates in link with wetting and drying cycles includes two distinct processes which act at two different scales: the rearrangement of coarse particles and the redistribution of clays particles and colloids. Both processes interact closely.

The rearrangement of coarse particles (silt and sand grains) involves short distance displacements of the silt and sand particles and induces changes in the structure topology at the coarse grain scale. The redistribution of clay and colloid particles involves displacement of the colloids through longer distances and induces changes in the bonds between coarser particles.

Considering the coarser grain scale, soil wetting causes the decrease of the inter-particle water tension, leading to a decrease in inter particle cohesion (Sheel *et al.*, 2008). Water tension and so inter particle cohesion reach a minimum value when soil is saturated. Considering the clay grain scale, wetting phase can potentially cause the hydration, dispersion or dissolution of the clay particles and colloids, depending on their properties, leading to the weakening of the bonds between coarser particles. The combination of both inter-particle cohesion decrease and bond weakening leads to a decrease in aggregate stability during wetting.

As the soil dries the water phase recedes into capillary wedges surrounding particle to particle contacts and films between closely adjacent platelets. The internal cohesive tension pull adjacent particles together (Kemper & Rosenau, 1986). The tension in the retreating water menisci can generate enough force to produce direct contact between particles (Dexter *et al.*, 1988). Direct contact can induce friction forces that increase inter-particle cohesion. When soil is saturated, soluble compounds such as silica, carbonates, (hydr)oxydes and organic molecules are concentrated in the liquid phase. Considering the clay grain scale, as the soil dries the capillary wedges retreat toward the inter-particles contact point. While soil solution becomes saturated, the solute molecules and ions precipitate around these inter-particle contact points, cementing particles together (Kemper & Rosenau 1986; Dexter *et al.*, 1988). Such processes lead to the aggregate stability increasing during drying.

The succession of wetting and drying phases modify the silt clay fabric within the aggregate, leading to changes in aggregate stability (Attou *et al.*, 1998). The formation of the close matrix fabric with clay coatings and bridges required several wetting and drying cycles to reorganize the clay particle distribution within the soil matrix. The formation of clay bridges between coarser particles induced by successive wetting and drying cycles was observed by microscopic imaging (Singer *et al.*, 1992; Attou *et al.*, 1998).

The wetting and drying cycles is a required condition for the occurrence of both coarse particle rearrangement and redistribution of clay and colloids. Numerous studies showed that aggregate stability variation was affected by wetting and drying cycles and incriminated both particle rearrangement and clay redistribution. The results of the latter studies are still controversial (Amézketa, 1999; Bronick & Lal, 2005). The result of wetting and drying succession on aggregate stability depend on the counteracting effects of the negative impact of wetting and the positive impact of drying (Denef *et al.*, 2001).

The particle rearrangement during wetting and drying cycles theorized by Kemper & Rosenau (1984; 1986) and Dexter *et al.* (1988) have never been directly observed yet. However this process is widely incriminated aggregate stability variation linked to wetting and drying cycles.

2.2.2. *Interlocking: frictional effect*

As cited previously, soil wetting induces the decrease of inter-particle water tension and the development of water films around the mineral particles, increasing the distance between them. In such conditions, the friction force can become almost null. On the contrary, while drying, water tension increases, pulling mineral particles into direct contact with great forces (Dexter *et al.*, 1988). Even smooth-looking particles have an irregular and rough shape at the micro scale. When pulled into direct contact, particles can fit together in a more stable arrangement, thus providing an increase in cohesion, and thus in aggregate stability. Such a

process is called interlocking. Macro interlocking concerns the particles and micro interlocking concerns their surface roughness. The strength due to the interlocking is a function of the normal stress at the contacts and causes an increase in the frictional component of the strength.

The processes involving friction forces only concerns coarse textured soils with clay content lower than 15% (Lebert & Horn, 1992). The first condition for interlocking is the close contact between the coarse particles within the aggregate. The strength of an aggregate, defined by its angle of internal friction and its cohesion, depends on the number of contact points (Horn *et al.*, 1994). The number of such contact is increased by the coarse particle rearrangement while drying, as mentioned earlier. Rajaram & Eberbach (1998) showed that friction angle was affected by the drying rate. Friction angle of a silt loam tends to increase with the drying rate until reaching a 15% drying rate.

While interlocking have been observed and incriminated in the changes in mechanical soil properties such as the shear strength, its influence in aggregate stability seems to be limited. Indeed, interlocking involves slight forces compared to the other processes of aggregate stability variation.

2.2.3. Clay dispersion and flocculation

In order to understand the mechanisms of clay dispersion and flocculation, and the conditions of their occurrence, it is necessary to remind the physico-chemical properties of clay minerals and their role in aggregation. Clay minerals are characterized by a layered structure. Such structure has large exchangeable surface area and thus, large fixation capacity. Depending on their structure and induced physico chemical properties, clay minerals are generally divided in two main groups: 1:1 crystalline clays and 2:1 cristalline clays. 1:1 crystalline clays (e.g. kaolinite) are non expanding, have low cation exchange capacity and surface area, and thus have low fixation capacity. 2:1 clays, such as smectite, are expanding

and are associated with high cation exchange capacity, large surface areas and thus, high fixation capacity. Because of high fixation capacity, 2:1 clay particles act as a cementing agent, grouping in clay domains and forming mineral bonds between coarser particles (Shainberg *et al.*, 1992; Le Bissonnais, 1996). Smectitic clays are the most efficient in aggregation because of large specific surface area, high CEC, and thus high physio-chemical interaction capacity. Clay particles also have colloidal properties: they can be dispersed in solution or flocculate depending on their mineralogy and in the electrolyte concentration of the solution.

Physico-chemical clay dispersion results from the reduction of attractive forces between colloidal particles during wetting (Emerson, 1967). As water content increases, clay particles become separated into distinct individual entities and suspended in the soil solution (Kay & Dexter, 1990; Zhang & Horn, 2001). This process results in the reduction in the cohesive strength of all clay inter-particle bonds and clay domains that decreases aggregate stability, leading to the structure breakdown (Kay & Dexter, 1990). When attractive forces between colloidal increases, the colloids flocculate and clay particles can accumulate in domains and depose at points of contact between larger particles as drying proceeds. This process results in the agglomeration of clay particles in clay domains, the deposit of clay coatings at the interface of fractures and at inter-aggregate pores resulting in an increase in aggregate stability (Attou *et al.*, 1998; Kjaergaard *et al.*, 2004).

Physico chemical clay dispersion and flocculation are mainly controlled by water content dynamics, clay mineralogy, sodicity, pH, and electrolyte concentration of the soil solution (Van Olphen, 1977; Emerson, 1977; Rengasamy *et al.*, 1984; Amézketa, 1999; Bronick & Lal, 2005). Controlling factors of clay dispersion and flocculation interact closely. Chemical dispersion depends on clay mineralogy. It is generally recognized that aggregates dominated by 2:1 layer minerals are dispersed more readily than those dominated by 1:1

minerals because of a higher charge density (Shainberg & Letey, 1984, Kjaergaard *et al.*, 2004). Illite clays are the clays the most sensitive to dispersion (high flocculation values). This is due to smaller edge to face attraction forces in comparison to the other clay because of the irregular and terraced surfaces of the illite particles (Amézqueta, 1999). Smectite clays are also recognized to be very dispersible. Kaolinite clay minerals are less susceptible to dispersion (Zhang & Horn, 2001). It is important to note that the clay minerals the more susceptible to dispersion are also the most involved in aggregate stability (2:1 clay minerals).

The effect of clay dispersion and flocculation on aggregate stability is closely related to the dynamics of soil water content. Water presence is a necessary condition for clay dispersion occurrence. If the free water content is limited (unsaturated soil) the clay particle may not be able to disperse (Quirk, 1978; Rengasamy *et al.* 1984). Drying allows the clay particles to concentrate in the retreating water menisci and to deposit at the coarser particle interface, acting as clay bonds and increasing aggregate stability (Dexter *et al.*, 1988; Attou *et al.*, 1998). Soil water properties such as pH, electrolyte concentration or sodicity also influence clay dispersion and flocculation.

According to Van Olphen's theory (1977), repulsion and attraction between particles depend on the distance between them. It is well known that an increase of the concentration in the soil solution reduces the thickness of the electrical double layer around the particles and therefore reduces the repulsive forces between them. As, in contrast, the attractive Van der Waals forces remain constant independently of the electrolyte concentration, the combination of repulsive and attractive forces results in different net potential curves. Hence, a low electrolyte concentration causes predominantly repulsion whereas at a high electrolyte concentration no repulsion forces exists (Holthusen *et al.*, 2010). Electrolyte concentration and sodicity control the threshold of dispersion/flocculation (Rengasamy & Olsson, 1991). Low electrolyte concentration and high sodicity produce clay dispersion and consequently the

loss of soil structure (Rengasamy *et al.* 1984; Amézketa, 1999). High sodium concentration causes dispersion of clay particles while high salinity causes clay flocculation (Ruiz-Vera & Wu, 2006). A threshold concentration curve defined by the sodium adsorption ratio and the electrolyte concentration is very useful to define chemical conditions that destabilize soil structure. Clay dispersion increase with increase the soil pH (Shainberg & Olsson, 1991): the negative surface charge on clay particles increases with pH, increasing particle repulsion (Chorom *et al.*, 1994). Organic matter appears to protect soil aggregate from dispersion. In natural field soils, the effect of mineralogy is influenced by the presence of surface adsorbed organic carbon, which alters the clay behaviour. Organic coatings have been found to induce colloid stability (Kjaergaard *et al.*, 2004). Finally, iron and aluminium (hydr)oxides in solution acts as flocculant.

Dispersion-flocculation of the clay has been widely studied through laboratory experiments. Soil resistance against dispersion can be measured by water dispersive clay tests (Rasiah *et al.*, 1992; Kjaergaard *et al.*, 2004).

2.2.4. *Dissolution and crystallisation*

When a solid is immersed in a liquid there is a chemical reaction with the liquid that transfer a portion the solid phase to the aqueous phase. Solubility determines how much of the solid phase dissolves into the solution. If the dissolved material exceeds a certain amount, some of the aqueous material precipitates back into a solid phase. Hence, soil wetting can induce the dissolution of components as salts, silica, carbonates or (hydr)oxides. Such components acting as binding agents, their dissolution decreases aggregate stability. When a soil is saturated, soluble compounds are in the liquid phase. As the soil dries, soil solution retreats toward the inter-particles contact points. The solutes molecules and ions are concentrated in the water menisci, and thus precipitate at these inter-particle contact points, cementing coarser particles together (Kemper & Rosenau, 1986; Dexter *et al.*, 1988). Such

phenomenon was observed for glass beads in NaCl solution of different concentrations (Soulié *et al.*, 2007), but not directly for soil material, according to our knowledge. When crystallizing at the water-air interface, precipitation builds up much stronger links than cohesion, resulting in strength increase.

Crystallization (or precipitation) and dissolution processes affects the bonds between particles at different scales, and involves different type of bonding agents. Carbonates, silicates, iron and aluminium (hydr)oxides are the mostly cited (Amézqueta, 1999; Six *et al.*, 2004; Bronick & Lal, 2005). Carbonates, phosphates and silicates can form bridges between both coarse particles and clay particles (Muneer & Oades, 1989; Clough & Sklemstad, 2000; Fernandez-Ugalde *et al.*, 2011). Ca^{2+} and Mg^{2+} carbonates precipitate to form secondary carbonate coatings and bind primary particles together. Carbonates also play an important role in bond formation between mineral and organic components (Muneer & Oades, 1989; Baldock & Skjemstad, 2000). Recent studies showed that potassium crystallization/dissolution also played a role in aggregate stability (Hohlthusen *et al.*, 2012). (Hydr)oxides are mainly incriminated in bonds at clay particle scale.

Changes in soil water content is a required condition for the occurrence of precipitation-dissolution. As soil dries, the concentration in dissolved components in the solution increases until reaching the saturation that leads to precipitation. The amount of soluble components is also an important factor for the dissolution-precipitation occurrence. In soil rich in carbonates, the carbonate is the main binding agent, and thus, its dissolution and precipitation are the dominant processes of aggregate stability variation (Six *et al.*, 2004; Fernandez-Ugalde *et al.*, 2011). This is especially the case of arid or semi arid calcareous soils where the rain amount is not large enough to leach the carbonate ions. This process has been less studied for soils under temperate climate probably because carbonate content is lower and binding agents are dominated by the organic components. Metal ion solubility is

strongly influenced by the soil solution pH (Bronick & Lal, 2005). Finally, organic matter content also affects precipitation. Baldock & Skjemstad (2000) observed that precipitation of dissolved carbonates was more probable to happen when organic matter was present in the soil.

If dissolution-crystallization is often cited as a process of aggregate stability increase, there is no dedicated experimental setup able to measure the occurrence and influence of this process on aggregate stability. Moreover, the role of dissolution-crystallization in aggregate stability variation has been hardly studied under temperate climate.

2.2.5. Age Hardening & thixotropy

Age hardening refers to the regain in aggregate strength with time at constant conditions (temperature, density and water content) after disturbance (Mitchell, 1960; Blake & Gilman, 1970; Molope *et al.*, 1985; Dexter *et al.*, 1988). It has been observed for artificially compacted aggregates or clods. Such phenomenon is also named as “structural resiliency” (Kay, 1990). Because the cited processes involved in age hardening were found to depend on thixotropic properties of the soil at the clay fraction scale (Mitchell, 1960; Molope *et al.*, 1985; Dexter *et al.*, 1988), it is also termed “thixotropic age hardening” (Utomo & Dexter, 1981), “thixotropic hardening” (Molope *et al.*, 1985), or simply “thixotropy” (Mitchell, 1960; Dexter *et al.*, 1988).

The thixotropy is a physical property referring to rheology. According to Barnes (1997), thixotropy is a time dependant fluid behaviour in which the apparent viscosity decreases with the time of shearing and in which the viscosity tends to recovers its original value after the stirring ceased. It corresponds to a viscoelastic behaviour (reversible) involving local particle rearrangement at the microstructure scale (colloids) (Barnes, 1997). The driving force for micro structural change is the result of the competition between structure breakdown

due to flow stresses, build up due to inflow collision and Brownian motion. Brownian motion is the random thermal agitation of atoms and molecules that results in element of the microstructure being constantly removed, causing them to move to a different position where they could be attached again to the microstructure. Mitchell (1960) extends this property to soil behaviour in order to explain observed age hardening in soils. Mitchell (1960) defined thixotropy in soil as an isothermal, reversible, time-dependent process, taking place under conditions of constant composition and volume, whereby a material stiffens while at rest, and softens or liquefies when remoulded (Mitchell, 1960). While it never had been directly observed, such phenomenon has been widely cited as the main process controlling age hardening (Utomo & Dexter, 1981; Dexter *et al.*, 1988; Shainberg *et al.*, 1996; Six *et al.*, 2004). Such processes have been theorized by Mitchell (1960) without microscopic observations, and thus staid partly speculative.

Two stages have to be considered: the structural strength decrease when disturbance is applied and the subsequent strength regain when sample is at rest. The processes incriminated in both stages involve the clay particles rearrangement, physico-chemical dispersion and flocculation processes, and dissolution/precipitation processes. In Mitchell's (1960) hypothesis, soil straining results in reorientation of platy clay particles by externally applied shearing forces into a more uniformly parallel arrangement. Clay particles are moved from their former equilibrium positions of low free energy to positions of higher free energy (Dexter *et al.*, 1988) inducing a decrease in cohesion strength. According to Utomo & Dexter (1981), remoulding would also create clay particle dispersion that induces the drop of soil structure cohesion. Moreover, remoulding a soil could also destroy the bonds between coarser particles (Mitchell, 1960). When the shearing force is stopped and the system sets to rest, thermal oscillations (Brownian motion) tend to randomize the orientation of the clay platelets. The particles gradually rearrange their positions and orientations back towards a minimum

free energy configuration. The resulting disorder imparts rigidity to the system by creating new bonds between particles (Mitchell, 1960; Molope *et al.*, 1985; Dexter *et al.*, 1988). After the colloid rearrangement, clay particles get in closer contact and can flocculate (Utomo & Dexter, 1981). Chemical change such as time dependent formation of cementation bonds between particles, are also considered as a possible cause of thixotropic behaviour. Iron oxides, carbonates, organic matter, and amorphous silica and alumina may bond particles together (Mitchell, 1960). The stability increase could also be attributed to component ions, atoms and molecules migrating to lower energy positions where there are more effective in bonding clay particles together (Dexter *et al.*, 1988).

The amount and speed of these changes depend on the soil water content and properties of the colloid solute system (Dexter *et al.*, 1988). The loss of strength and subsequent recovery would be greater when clay in the failure zone is dispersible. At low water content, the cation concentration maintains the clay flocculated. Further rearrangement is not possible in this condition and so no age hardening is possible (Mitchell, 1960). At high water content, the soil colloidal particles may stay into stable suspensions, and age hardening would be negligible. At intermediate water content, colloids are able to rearrange and flocculate with consequent increases in soil strength. The optimum water content for age hardening would be around the plastic limit of the soils (Utomo & Dexter, 1981). The range in water content considered by Utomo & Dexter (1981) would result in electrolyte concentration changes by no more than a factor of 2, and would therefore require that the concentration of the pore fluids be close to the critical concentration for flocculation-dispersion to be sensitive to solute concentration.

Age hardening is an observed phenomenon that has been hypothetically attributed to clay particle and colloid rearrangement. Such process has been called thixotropy by comparison with rheological properties of material and fluids, but had never been directly

observed yet. Anyway, thixotropic age hardening is widely cited as a process of aggregate stability variation.

2.3. Interactions between the processes during wetting and drying cycles

Water content was found to be the dominant factor of each of the cited processes of aggregate stability variation whatever the scale of the process occurrence. Soil liquid phase can affect aggregate stability in different ways. At first, water content influences aggregate stability through the presence of water menisci and induced capillary forces at the interface between particles. The presence of water menisci in the aggregate porosity prevents the air to escape during rapid wetting and provides slaking. Capillary forces are involved in particle rearrangement and frictional interlocking. The localisation of water menisci at the interface between coarser particles allowed the formation of inter-particle bonds formed from flocculated clays and crystallized components. Secondly, the kinetic energy of water drops is can break inter-particle bonds within the aggregate and lead to aggregate breakdown. Finally, soil solution also influences aggregate stability through physico-chemical interactions between water and soil particles. Wetting can cause the differential swelling of clay, and clay dispersion and the inter-particle bonds dissolution, while drying can cause the flocculation of clay and crystallization of suspended components that can act as bonding agents. Water physico-chemical properties are also involved when constant in freezing-thawing and thixotropic hardening processes.

These processes involve different mechanisms and so, must be distinguished. However, because water content variation is a common condition of occurrence, during wetting and drying cycles, most of these processes interact at the same time.

During wetting, soil water tension and thus the inter-particle cohesion decrease. This decrease in cohesion affects the aggregate strength against raindrop impact (Le Bissonnais et al., 1995). Moreover, clay dispersion and dissolution of soluble components affect the bonds

between the particles, decreasing their resistance against fragmentation processes. Processes at the micro aggregate scale decrease the resistance against disruptive processes at the macro aggregate scale. On the other hand, the wetting induced slaking and differential swelling of clay occurring at the macro-aggregate scale creates planes of weakness and cracks that stimulate processes at the micro-aggregate scale. At the interface of those planes of weakness or micro cracks, new particles are directly exposed to the water phase and can be dispersed or dissolve depending on their properties (Le Bissonnais, 1989). The combination of these processes leads to a general aggregate stability decrease during wetting. According to our knowledge, wetting does not provide physico-chemical processes which increase aggregate stability.

During drying, three main processes are involved in aggregate stability variation: particle rearrangement, friction and creation of bonds. According to Kemper & Rosenau (1984), during wetting, the water tension increase induces the rearrangement of particles (coarser grains and clay particles) in a closer arrangement leading to a direct contact between them. For the reinforcement of inter particle bonds while drying, it is necessary for the coarser particles to be in contact initially. As the water tension pulls adjacent mineral particles into closer proximity, there are also more opportunities for bonds to develop (Kemper & Roseau, 1986; Dexter *et al.*, 1988). Inorganic stabilizing material and organic molecules will be capable of bonding clay particles and cementing larger mineral grains together at their points of contact. However, when grains are separated by greater distances, material of molecular scale will be largely ineffective (Kay, 1990). Moreover, water evaporation provides an increase in electrolyte concentration and thus, triggers the flocculation of the dispersed clay and the crystallization of soluble components (Holthusen *et al.*, 2010). Because the remaining water get concentrated in the inter-particle retreating menisci, the dispersed clay and soluble compounds will flocculate and precipitate at the particle interfaces, creating new inter-particle

bonds. The combination of those processes leads to a general positive effect of drying on aggregate stability. According to our knowledge, drying only provide physico-chemical processes which increase aggregate stability.

Even if processes involved in aggregate stability are very different, they all are related to water content. It is difficult to experimentally separate them since they can occur simultaneously. However, only a few studies took interest in the interaction between those processes. Further researches must be done in order to find experimental methods of measurements taking into account the effect of each process of aggregate stability separately, but also to study the interactions between those processes during wetting and drying cycles.

3. Summary of current knowledge and orientation of further investigations

In the previous part, we described each process involved in aggregate stability and detailed their conditions of occurrence. The present bibliometrical study allowed us to hold a global point of view on the state of the art concerning the abiotic processes affecting aggregate stability. Those processes have been differentially studied and cited in literature, as presented in table 2 and 3. We made a bibliographic study with the aim to assess the intensity of citation of each identified process (table 2). Bibliometric study was performed the first of October 2012, from the Web of knowledge editor platform. For each identified process, we presented the number of citing studies (A index), the number of citation occurrence (B index) and the citation intensity (B/A). The number of citing studies (A index) corresponds to the number of publication between 1970 and 2011 citing the identified processes in their abstract or keywords. It inventories the publications which directly studied the processes or directly implied such process in aggregate stability variation. Publications identified by the A index have been cited by latter studies. The B index corresponds to the total citation occurrence of the publications identified by the A index, without taking self citations into account. It inventoried the publication citing the processes without directly studying them. Hence,

processes are not necessarily cited in keywords, but their descriptions refer to publications which directly studied them. The intensity of citation (B/A) provides information about the knowledge status of each process. A high ratio means a process well cited but few studied and thus, that can need further researches. A low ratio corresponded to a process well studied but few cited, thus with a low importance in aggregate stability variation compared to other processes.

Table 3 summarizes the information detailed in part 2. For each process, qualitative information is given about their knowledge status. The “observed” processes have been well studied, recommended method of measurement are given. “Hypothesized” processes are based on theory but have never been observed directly as processes of aggregate stability variation, and need further researches.

Table 2: Results from the bibliometrical study: number of publications directly studying the process of aggregate stability variation (A), number of publication referring the previous studies (B), and ratio between both values (B/A).

A. corresponds to the number of publication citing the processes in their abstract or keywords. B. corresponds to the number of publication referring to A. Bibliometric study performed on October 1st 2012, using the platform Web of knowledge editor.

Process	A. Number of studies citing process	B. Number of citations of those studies	Intensity of citation (B/A)
Dispersion / flocculation	369	6055	16.4
Slaking	217	6193	28.5
Raindrop impact	96	1401	14.6
Freezing-thawing	93	1707	18.4
Crystallisation/dissolution	29	660	22.8
Friction	20	211	10.6
Particle rearrangement	11	436	39.6
Differential swelling of clays	8	388	48.5
Thixotropic age hardening	3	88	29.3
Mean	94	1904	25.4

Table 3: qualitative synthesis of the results from the bibliographical study: scale of occurrence, observation status and recommended method of measurement for each identified process.

Process	Scale	Observed or Conjectured	Measurement
Dispersion/flocculation	Micro aggregate/ Clay domains	Observed	Water dispersive clay tests (<i>Rasiah et al., Kjaergaard, 2004</i>)
Slaking	Macro aggregate	Observed	Fast wetting test, immersion (<i>Le Bissonnaux, 2005</i>) Pressure sensor (<i>Zaher, 2005</i>)
Rain drop impact	Macro aggregate	Observed	Water drop test (<i>Low, 1965; Imeson & Visser, 1996</i>) Stirring test (<i>Le Bissonnaux, 1996</i>)
Freezing-thawing	Macro aggregate	Observed	Freezer (<i>e.g. Bullock et al., 1988</i>)
Crystallisation/dissolution	Micro aggregate/ clay domains	Hypothetical	-
Friction	Micro aggregate	Observed	Friction angle measurement (<i>Rajaram & Arora, 1999</i>)
Particle rearrangement			
<i>Coarse particles</i>	Micro aggregate	Hypothetical	-
<i>Clay redistribution</i>	Micro aggregate	Hypothetical	-
Differential swelling	Macro aggregate	Hypothetical	-
Thixotropic age hardening	Clay domains	Hypothetical	-

3.1. Widely studied processes

At the macro aggregate scale, slaking, raindrop impact and freezing-thawing processes of aggregate stability variation are the most widely known. Since 1970, 217 publications cited slaking, 96 publications cited raindrop impact and 93 publications cited freezing-thawing as processes of aggregate stability variation in their abstract or keywords (Table 2). Intensity of citation were 28.5 for the slaking, 14.6 for the raindrop impact and 18.4 for the freezing-thawing processes. Those processes have been observed through repeated laboratory experiments, their conditions of occurrence have been identified and the involved forces quantified (air pressure, kinetic energy of the raindrop and expansion of ice crystals). Such processes have negative effects on aggregate stability. The aggregate strength against disruptive processes can be tested by various laboratory experiments (e.g. Le Bissonnais, 1996; Farres et al, 1980) (Table 3). The resistance against slaking can be easily measured by wet sieving after immersion of dry aggregates (Yoder, 1936; Le Bissonnais, 1996). The recent work of Zaher *et al.* (2005) permitted to quantify the pressure required for the aggregate breakdown by slaking. The water drop test (Low, 1965; Imeson & Vis, 1984) and the stirring test (Le Bissonnais, 1996) allow the assessment of the aggregate resistance against the raindrop impact. The effect of freezing on aggregate stability can be performed through laboratory experiments using freezer.

Differential swelling-shrinkage of the clays has not been observed directly yet. This process has been deduced through experimental studies based on the comparison of swelling clay soils and non swelling clay soil during wetting and drying cycles. Since 1970, only 8 publications cited this process in link with aggregate stability variations (table 2). Many studies considered differential swelling of the clay as part of the slaking process, and thus named it slaking. This simplification might biases the results of the bibliometric study.

Because of strong interactions with the other processes induced by wetting and drying cycles (slaking, clay dispersion and flocculation), the occurrence of differential swelling and shrinkage is hard to estimate. The slow wetting test from aggregate stability tests (Le Bissonnais, 1996) emphasizes the effect of differential swelling compare to slaking. Microscopic imaging allowed identifying micro-cracks within soil aggregate that are caused by differential swelling. Anyway, if micro-cracks have been observed as a consequence of differential swelling of the clays, consequences on aggregate stability remained qualitative and need further researches to be quantified.

At the micro aggregate scale, physico-chemical dispersion and flocculation of the clay has been widely studied. Since 1970, 369 publications cited dispersion and flocculation of the clay as a process of aggregate stability variation (Table 2). The intensity of publication was 16.4 for clay dispersion/flocculation which do not correspond to an extreme value. This process has been experimentally observed and aggregate resistance against dispersion can be measured by water dispersible clay tests (Rasiah *et al.*, 1992; Kjaergaard *et al.*, 2004).

Finally, friction is a process that is well known especially in material mechanic science. Friction effect between coarser particles can be assessed by friction angle measurements (Rajaram & Erbach, 1998). However, since 1970, friction processes have only been cited 20 times in relation with aggregate stability variation, and intensity of citation was the lowest among the studied processes (10.4). Such results may mean that friction is not considered as a dominant process in aggregate stability variation. Incriminated in phenomenon affecting soil structure at higher scale such as resistance against compaction, friction influence may be lower at the aggregate scale compare to other processes of aggregate stability variation.

3.2. Processes needing further researches

According to the present study, three processes of aggregate stability variation are based on hypotheses and have not been directly observed yet. Those processes remained unknown and thus need further researches.

The internal particle rearrangement during wetting and drying cycles theorized by Kemper & Rosenau (1984; 1986) and Dexter et al. (1988) has never been observed yet. Microscope image analysis allowed identifying clay bridges or microcracks within aggregate structure (e. g. Singer et al., 1992; Attou et al., 1998) but the evolution of internal aggregate structure linked with wetting and drying at both coarse grain scale and clay scale has not been observed yet as a process of aggregate stability variation. However, this process is widely incriminated in aggregate stability changes linked to wetting and drying cycles (e.g. Zhang & Horn, 2001; Denef et al., 2001; Six et al., 2004). If only 11 publications directly cited this process as a cause of aggregate stability variation since 1970, those publications have been cited 436 times, corresponding to an intensity of citations of 39.6 (table 2). Further researches must be performed in order to verify the validity of such process as a cause of aggregate stability variations, and if it's the case, to understand its occurrence conditions and interactions between other processes. New techniques development in imaging (e.g. X-ray 3D micro-tomography) could allow characterizing the evolution of the coarse grain structure within an aggregate during wetting and drying cycles.

Since 1970, dissolution-crystallization has been cited by 29 publications as a process of aggregate stability variation (Table 2). If crystallization is often cited as a process of aggregate stability increase, there is no dedicated experimental setup able to measure the occurrence and influence of this process on aggregate stability. If the effect of carbonates and (hydr)oxides on aggregate stability have been widely studied, this not the case of silicates or other soluble minerals. Moreover, the role of dissolution-crystallization in aggregate stability

variation has been hardly studied under temperate climate, probably because it is considered as secondary process compared to other processes of aggregate stability variation. Recently, studies from Hohlthusen *et al.* (2010; 2012) took interest on the role of potassium precipitation/dissolution on aggregate stability and rheological properties of German soils, indicating that new researches are taking this process into account. However, current knowledge does not allow studying separately the effect of ions on clay dispersion/flocculation processes and crystallisation/dissolution processes. Hence, further researches are needed to clarify the effect of dissolution-crystallization on aggregate stability variation in temperate climate.

Age hardening is an observed phenomenon at the macro aggregate scale. The strength regain after the remoulding has been hypothetically attributed to clay particles and colloids rearrangement. Such process has been called thixotropy because of a comparison with rheological properties of material and fluids, but had never been directly observed yet. Since 1970, only 3 publications have cited thixotropy in their abstract or keywords in relation with aggregate stability variation, however, such process is discussed in articles as a process of aggregate stability variation (e. g. Dexter *et al.*, 1988; Shainberg *et al.*, 1996; Six *et al.*, 2004). The occurrence of the processes described in Mitchell's (1960) theory should be checked by further researches. Moreover, the use of the term "thixotropy" in order to describe such hypothetical process should be deleted until further researches directly observe its occurrence.

These researches would allow a better understanding of the physio-chemical processes and the effect of wetting and drying on aggregate stability. Understanding the mechanical aspects of such processes separately would be useful to understand the interaction between abiotic and biotic processes and would enable to better predict aggregate stability variations for a given soil.

4. Conclusion

The present review allowed identifying the abiotic processes involved in aggregate stability variations. The soil water content dynamics affects each of the identified process. At the macro-aggregate scale, slaking, raindrop impact and freezing-thawing have been identified as processes of aggregate stability decreasing. Such processes have been observed and their conditions of occurrence condition are well known. At the micro-aggregate scale, particle rearrangement and clay redistribution, friction, clay dispersion/flocculation, dissolution/crystallization and thixotropic age hardening have been identify as processes of aggregate stability variation. While clay dispersion/flocculation is well known, other processes need further research in order to check their involvement with aggregate stability variation. Coarser particle rearrangement has been theorized but not observed yet. The influence of crystallization-dissolution on non carbonated soils remains mostly unknown even if recent studies aimed at assessing its effect on aggregate stability. Because soil is a complex media where many processes interact even at the aggregate scale, understanding the processes, conditions of occurrence and interactions between abiotic processes is a current challenge for aggregate stability predictions.

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Synthèse et conclusion

L'objectif de cette première partie était de poser les bases contextuelles de la thèse sur deux problématiques : l'estimation de l'érodibilité et les processus physico-chimique en jeu.

1) L'estimation de l'érodibilité est imprécise et est donc une source d'erreur dans les modèles d'érosion.

- *L'érodibilité varie fortement pour un même type de sol selon la localisation du site.*

Les résultats de l'étude de terrain présentés dans le chapitre 1 ont clairement montré que pour un même type de sol présentant des propriétés homogènes, l'érodibilité estimée par des mesures de stabilité structurale pouvait fortement varier selon la localisation du site. Ce résultat souligne les limites dans l'estimation du paramètre érodibilité par les modèles d'érosion. En effet, la plupart des modèles d'érosion considèrent l'érodibilité comme une constante pour un type de sol donné (Jetten et al., 2003 ; Guimere et al., 1999). Or, les résultats de la présente étude montrent que l'érodibilité d'un sol donné varie selon la localisation du site. Améliorer la précision des modèles d'érosion nécessite de déterminer la source de ces variations.

- *La stabilité structurale de la surface du sol peut être très différente de celle du matériau sous jacent*

Les résultats de l'étude de terrain ont également montré de fortes différences entre la stabilité structurale de la croute de surface et la stabilité structurale du matériau sous jacent. Même lorsque la stabilité structurale est utilisée comme un proxy de l'érodibilité, les mesures sont généralement réalisées dans l'horizon labouré du sol. En considérant les résultats de

l'étude, les mesures de stabilité structurale doivent portées sur le matériau au contact du processus érosif, c'est-à-dire sur le matériau de surface.

- *les propriétés standard du matériau ne permettent pas d'expliquer les variations de stabilité observées*

Enfin, les propriétés standard du matériau ne permettent pas d'expliquer la variabilité de stabilité structurale observée. D'autres variables sont donc à l'origine de ses variations. On suggère, par exemple, des variations dans le climat local entre les sites. Ses variations pourraient causer des différences d'histoire hydrique du sol pouvant être à l'origine des variations de stabilité structurale observées. Un suivi de terrain à pas de temps court associant des mesures de stabilité structurale, de variables biologiques et de variables climatiques est proposé pour identifier les facteurs de variation de la stabilité structurale. Une telle étude a été réalisée ; elle est présentée dans la partie suivante.

2) Les processus physico-chimiques à l'origine des variations de la stabilité structurale doivent être mieux connus.

La synthèse bibliographique présentée dans le chapitre 2 a permis de dresser un inventaire exhaustif des processus physico-chimiques cités dans la littérature comme étant à l'origine des variations de la stabilité structurale. Cette étude souligne que la dynamique de la teneur en eau du sol est le facteur commun de ces processus. A l'échelle du macro-agrégat, les processus identifiés (éclatement, impact des gouttes de pluies, gel-dégel) diminuent la stabilité structurale. Ces processus ont été observés et leurs conditions d'occurrence sont bien connues. A l'échelle du micro-agrégat, certains processus sont bien compris (telle la dispersion/floculation des argiles), mais, pour d'autres processus, des études approfondies validant leur existence et déterminant leur implication dans les variations de stabilité structurale restent à conduire. C'est le cas notamment du réarrangement particulaire intra-

agrégat lié aux cycles d'humidité. S'il a certes été théorisé, il n'a pas encore été directement observé. Aussi, deux études portant sur les changements de structure interne des agrégats ont été réalisées et sont présentées dans la troisième partie.

Deuxième partie

**Etude de terrain de la variabilité de la stabilité
structurale à pas de temps court.
Evaluation des facteurs explicatifs.**

Introduction

L'érodibilité du sol est un paramètre clef pour la prédiction de l'érosion par les modèles. Ce paramètre peut être estimé par des mesures de stabilité structurale. De ce fait, la prédiction de la stabilité structurale apparaît comme un enjeu majeur pour améliorer les prédictions de l'érosion. Dans le chapitre 1, nous avons pu voir que l'érodibilité du sol était très variable, et que, de ce fait, considérer le paramètre érodibilité comme constante dans l'espace pour un sol donné était une forte source d'imprécision. Les résultats présentés dans le chapitre 1 ont aussi montré qu'à un instant donné l'érodibilité d'un même type de sol pouvait varier entre le matériau de surface et le matériau immédiatement sous-jacent.

Les chapitres suivants s'intéressent à la variabilité temporelle de l'érodibilité estimée par des mesures de stabilité structurale. De nombreuses études ont montré que la stabilité structurale variait dans le temps (e.g., Bullock et al. 1988 ; Blackman, 1992 ; Chan et al. 1994 ; Bajracharya et al. 1998 ; Deneff et al., 2001 ; Cosentino et al., 2006 ; Dimoyiannis, 2009). Des suivis de terrain réalisés à pas de temps mensuels ont mis en valeur des variations saisonnières : la stabilité structurale est maximale à la fin de l'été et minimale à la fin de l'hiver (Bullock et al. 1988 ; Blackman, 1992 ; Chan et al. 1994 ; Dimoyiannis, 2009). Des suivis réalisés en laboratoire ont permis d'identifier des variations à pas de temps court (hebdomadaire à journalier) en relation avec les cycles d'humidité et la stimulation de l'activité biologique (Utomo & Dexter, 1982 ; Deneff et al. 2001 ; Cosentino et al. 2006). Cependant, de telles variations à pas de temps court n'ont, à notre connaissance, jamais été rapportées sur le terrain. Si les études précédemment citées ont permis d'identifier des facteurs de variation de la stabilité structurale, les prédictions de la stabilité structurale restent peu précises. De plus, il est actuellement difficile de relier les facteurs identifiés par des expérimentations en conditions contrôlées aux variations de stabilité structurale mesurées sur le terrain. Ces difficultés peuvent être expliquées par une résolution temporelle trop large des

suivis de terrain : des prélèvements à pas de temps mensuel ne peuvent pas prendre en compte l'ensemble de la variabilité de facteurs explicatifs tels que la teneur en eau et l'activité biologique. Un suivi de terrain à pas de temps court (un à deux jours) apparaît donc comme opportun pour préciser la relation entre la stabilité structurale et les facteurs considérés *a priori* comme explicatifs.

Un suivi de terrain à pas de temps court a été réalisé sur deux Luvisols localisés dans le sud du Bassin parisien. Les objectifs étaient de quantifier les variations de la stabilité structurale à pas de temps court et de relier ces variations à des facteurs explicatifs à travers des variables liées à l'activité biologique et au climat. Ce suivi de terrain s'est étalé sur une période de 6 mois, de mars à août 2011. Sur la base des résultats présentés dans le chapitre 1, la stabilité structurale du sol a été mesurée sur le matériau de surface et dans l'horizon labouré. Le sol a été maintenu sans végétation durant tout le suivi et l'activité biologique n'a pas été stimulée par des apports. Des variables liées à l'activité biologique et au climat ont été mesurés en tant que facteurs explicatifs potentiels de la stabilité structurale. Les résultats de cette étude de terrain sont présentés dans les chapitres 3 et 4. Ces deux chapitres ont été rédigés sous forme d'articles en vue d'une soumission à la revue *Soil Society of America Journal*.

Le chapitre 3 présente les variations de la stabilité structurale à pas de temps court et compare ces variations avec les modalités spatiales suivantes : deux sites présentant des types de sol similaires et une proximité géographique de quelques kilomètres ; deux placettes situées dans la même parcelle cultivée ; la surface et la sub-surface au sein d'une même placette. Les résultats montrent de fortes variations temporelles de la stabilité structurale à pas de temps court en lien avec l'historique des précipitations, ainsi que des comportements spatialement différenciés.

Le chapitre 4 porte sur les variables potentiellement explicatives des variations de la stabilité structurale. Ce chapitre montre que la teneur en eau et l'histoire hydrique du sol étaient les facteurs dominants de la variation de la stabilité structurale. Les variables liées à l'activité biologique n'ont pas influencé la stabilité structurale. Un modèle de régression incluant différents indices de l'histoire hydrique explique près de 60% des variations de stabilité observées. Cette étude met en valeur le caractère dominant des facteurs abiotiques sur la variation de la stabilité structurale à pas de temps court sans stimulation de l'activité biologique.

Chapitre 3 :

**Mesure de la variation de la stabilité structurale
à pas de temps court.
Conséquences pour l'estimation de l'érodibilité**

**Short-term dynamics of aggregate stability in the field,
consequences for erodibility assessment**

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Abstract

Aggregate stability can be used as a proxy for soil interrill erodibility. Because soil interrill erodibility is still difficult to predict accurately, current erosion models use only one erodibility value for a given soil type as a time constant. Many studies have shown that aggregate stability may change over time. Variations over short time steps have been observed through lab experiments, but have not yet been observed through field monitoring. We conducted a field monitoring experiment to assess variations in aggregate stability over short time steps and the influences of rainfall amounts on such variation. Variability in aggregate stability was measured for different spatial treatments. Rainfall occurrence was measured as a potential factor in aggregate soil stability variation. Aggregate stability varied greatly over short time steps and was differentially influenced by the studied treatments. The two sites, located on similar soil types in close proximity, presented with contrasting ranges for their aggregate stabilities, but they showed the same trends in variation. The influence of soil surface and subsurface on aggregate stability was less clear. Finally, rainfall, as assessed by the antecedent precipitation index, was negatively correlated with soil surface aggregate stability, regardless of treatment. Variations in aggregate stability correspond to contrasting erodibility values. Parameterizations of erosion models for a given soil type should consider these short time step variations.

1. Introduction

Aggregate stability corresponds to the ability of an aggregate to retain its structure when exposed to water. In the literature, many studies have shown that aggregate stability changes temporally (e.g., Bullock et al. 1988; Blackman, 1992; Chan et al. 1994; Bajracharya et al. 1998; Deneff et al., 2001; Cosentino et al., 2006; Dimoyiannis, 2009). Field monitoring, performed monthly, has identified seasonal trends in aggregate stability variation which

follow a nearly cyclical pattern, with the greatest values recorded during summer and the lowest values during winter (Bullock et al. 1988; Blackman, 1992; Chan et al. 1994; Dimoyiannis, 2009). These experiments permit the identification of explanatory climatic factors related to aggregate stability variation, such as the occurrence of rain (Blackman, 1992; Dimoyiannis, 2009). Lab experiments have allowed for the identification of temporal variations in aggregate stability over shorter time steps. Temporal variations in aggregate stability have been shown to be in line with weekly wetting-drying cycles and with the stimulation of biological activity (Utomo & Dexter, 1982; Deneff et al. 2001; Cosentino et al. 2006). Although lab experiments have shown that aggregate stability can vary at shorter time steps (from a few days to several weeks), such short time step variations still have not been observed through field monitoring.

In the context of water erosion, aggregate stability is recognized as a proxy for soil water interrill erodibility: the higher the aggregate stability, the lower the soil interrill erodibility (Le Bissonnais, 1996; Barthes & Roose, 2002; Gumiere et al. 2009). Soil water interrill erodibility is the sensitivity of soil material to detachment and transport by raindrop impact and sheet flow. Thus, interrill erodibility is considered to be a key parameter in soil erosion modeling and prediction. Currently, erosion models have great difficulty predicting water erosion accurately (Jetten *et al.*, 2003; Boardman, 2006). These difficulties may be linked to difficulties in estimating erodibility. First, when measured by aggregate stability tests, erodibility is often assessed in samples collected within the plough layer (e. g. Bullock *et al.*, 1988; Bajracharya et al., 1998; Barthès & Roose, 2002), despite the fact that interrill erosion occurs at the soil surface of crusted soils and, thus, depends directly upon the erodibility of the crust and not upon the erodibility of the subsurface material. Previous work has shown that the aggregate stability of the soil surface can be very different than the aggregate stability of the subsurface (Poesen, 1981; Kuhn & Bryan, 2004; Darboux & Le Bissonnais, 2007;

Algayer et al., under review). This finding has lead researchers to conclude that estimates of erodibility may be less accurate if performed on material collected from the plow layer than if they are performed on surface samples, resulting in a potential bias in the estimated erodibility. Moreover, parameterizations of erosion models currently set a unique erodibility value for a given soil and, thus, do not consider the temporal variability of the erodibility for a given soil (Alberts et al., 1995; Jetten *et al.*, 2003; Gumiere *et al.*, 2009). Finally, an erosion model typically uses one erodibility value for one soil type as a time constant or, at best, one erodibility value as a constant for one season. For example, in the Water Erosion Prediction Project model (WEPP), the soil interrill erodibility factor (K_i) is calculated as a function of primary soil properties (Alberts et al., 1995). K_i corresponds to the erodibility of freshly tilled soil and can be adjusted using correcting factors including canopy cover, roots and crusting. Such models do not consider the temporal variability in erodibility that occurs with a bare soil.

For the present study, we conducted field monitoring of aggregate stability variations over short time steps (of a few days) in two sites located in Luvisols, in the south of the Parisian basin (France). The objectives of the study were as follows: 1) to assess variations in aggregate stability at short time steps in the field; 2) to compare temporal variations in aggregate stability for different treatments for soil surface and subsurface measured on the same field plot, for two different plots located on the same crop field, and for two different fields presenting very close soil types; and 3) to assess the relationship between aggregate stability variation over short time steps and rain occurrence. The consequences for erodibility assessment and erosion modeling are discussed.

2. Materials and Methods

2.1. Sampling sites

Field monitoring was performed in two sites, Marcheville and La Gouëthière, both located in the south of the Parisian Basin, 15 kilometers to south-west from the city of Chartres. Sites were located on two silt loam cultivated Luvisols (Soil Survey Division Staff 1993) and were geographically very close together (5 kilometers). Soil surface (A horizon) properties of each site are presented in table 1. The Marcheville field was located (48°21'512"N; 1°16'0.55"E) on a typical Luvisol, cultivated by wheat and presented a slight slope (7%) oriented towards the North. The antecedent crop was corn. The La Gouëthière field was located (48°22'489"N; 1°12'100"E) on a degraded Luvisol, cultivated by pea and presented a with slight slope (5%) oriented towards the Southwest. The antecedent crop was wheat. Sampling was performed in the A horizon of the soil. The A horizon of the Marcheville soil was comprised of 16.4% clay and 2.16% organic matter, while the La Gouëthière A horizon was comprised of 11.3% clay and 1.37% organic matter (table 1). Other soil characteristics were very similar between the two sampling soils (table 1).

Table 1: Characteristics of the soil material from the two sampling sites

Site	Clay	Silt	Sand	OM	CEC	pH	Ca	Mg	Ka	Na
		g kg ⁻¹			Cmol kg ⁻¹			g kg ⁻¹		
Marcheville	164	798	38	21.6	9.1	6.7	8.8	0.5	0.6	0.03
La Gouëthière	113	848	39	13.7	6.7	6.7	6.6	0.4	0.2	0.04

2.2. Monitoring setup

For each site, two 50 m² plots (upslope and downslope) were defined within the crop field. The distance between the two plots was approximately 50 meters. Plots were kept bare with herbicide during the 6 months of monitoring to facilitate sampling and minimize the effects of soil vegetation on aggregate stability. Monitoring was conducted on four plots (two plots for each site) during five months in 2011. Monitoring started just after the seed bed

work, on 9 March for the Marcheville site and on 16 March for the La Gouëthière site, and it ended on 18 August.

Sampling was carried out at two time scales: monthly time steps and shorter time steps of a few days (two to three days) during the two weeks after an important rain event. For each monthly sample, three distinct samples were collected for each 50 m² plot to assess the spatial variability of the measured variables within the plot. Three periods of short time step sampling occurred during the monitoring. Seven samples were recorded at short time steps between 3 May and 18 May. Five samples were recorded between 7 June and 16 June and, again, between 8 August and 18 August. On each sampling date, samples were collected from a one square meter area within the plot. Each one square meter area was collected only once.

2.3. Sampling setup

Prior to sampling, the soil surface was described visually. If the soil surface presented crust, the crust type (structural crust or sedimentary crust) was identified (Bresson & Boiffin, 1990; Belnap *et al.*, 2008).

Paired samples (surface and subsurface material) were collected from each plot. The surface samples were collected separately from the subsurface samples. For the surface samples, material was collected from the first five millimeters of soil depth. After 5 May, soil surfaces at both sites presented a structural crust. This crust evolved into a sedimentary crust at the Marcheville site only, from 2 August onward. When the soil surface was crusted, large pieces of the crust material were collected using a sharp knife to cut through the material without affecting its structure. The subsurface material was defined as the soil material between -1 cm and -5 cm (from the initial soil surface).

2.4. Measurements

Aggregate stability was measured for each sample using the Le Bissonnais method (Le Bissonnais 1996, *ISO/DIS 10930, 2012*). The three stability tests of Le Bissonnais (1996) (fast wetting, slow wetting and stirring) were designed to reproduce the processes involved in crust formation and interrill erosion (slaking, differential clay swelling and mechanical breakdown). The 5 g sub-samples were dried at 40°C for 24 h before application of the test, and each test was replicated three times. After the tests, the resulting fragments were sieved in ethanol. The results are presented using the mean weighted diameter (MWD). Each MWD value corresponds to one of five classes of stability as follows: MWD above 2 mm corresponds to very stable material (very low erodibility), between 2 and 1.3 mm corresponds to stable material (low erodibility), between 1.3 and 0.8 mm corresponds to median stability (median erodibility), between 0.8 and 0.4 mm corresponds to unstable material (high erodibility) and lower than 0.4 mm corresponds to very low stability (very high erodibility) (Le Bissonnais, 1996).

Rainfall was measured hourly using two pluviometers (one for each site) during the entire monitoring period. The antecedent precipitation index (API) was calculated for 7 days prior to aggregate stability measurement as follows:

$$API = \sum_{i=0}^6 \frac{10-i}{10} * P_i$$

Where i is the i^{th} day before the sampling, and P_i (in millimeters) is the total precipitation height on the i^{th} day.

2.5. Statistical analysis

Statistical analyses were performed using R version 2.9.2 (R Development Core Team, 2011). To identify differences in MWD between the different factors (surface/subsurface; upslope/down slope; Marcheville/La Gouëthière), paired comparisons were performed with

the Wilcoxon signed rank test. Such non-parametric methods allow for comparisons of the variability of short datasets ($n < 30$) without testing the normality of the data dispersion. We considered a threshold of $p < .05$ to be significant. Linear correlation analyses (Pearson's coefficient) were performed to identify the relationships between MWD variations in the different treatments and between MWD values and the antecedent precipitation index (API).

3. Results

3.1. Temporal variation of aggregate stability

For temporal variations in aggregate stability, only the results of the slow wetting test are presented in detail. Moreover, in the present part, only results from the upslope plots are presented in detail.

3.1.1. Monthly variation of aggregate stability

For each monthly sample, three distinct samples were collected for each of the 50 m² plots to assess the spatial variability of the measured variables within the plot. In Figures 1 and 2, error bars of the monthly points include this spatial variability. This measurement was performed to take spatial variability into account. In the Marcheville site, the largest measured spatial variability was 9% for the soil surface and 12% for the soil subsurface. In the La Gouëthière site, the largest measured spatial variability was 10% for both the soil surface and subsurface (Figure 1). Short time step variations in aggregate stability superior to the maximum spatial variation coefficients were considered to be significant.

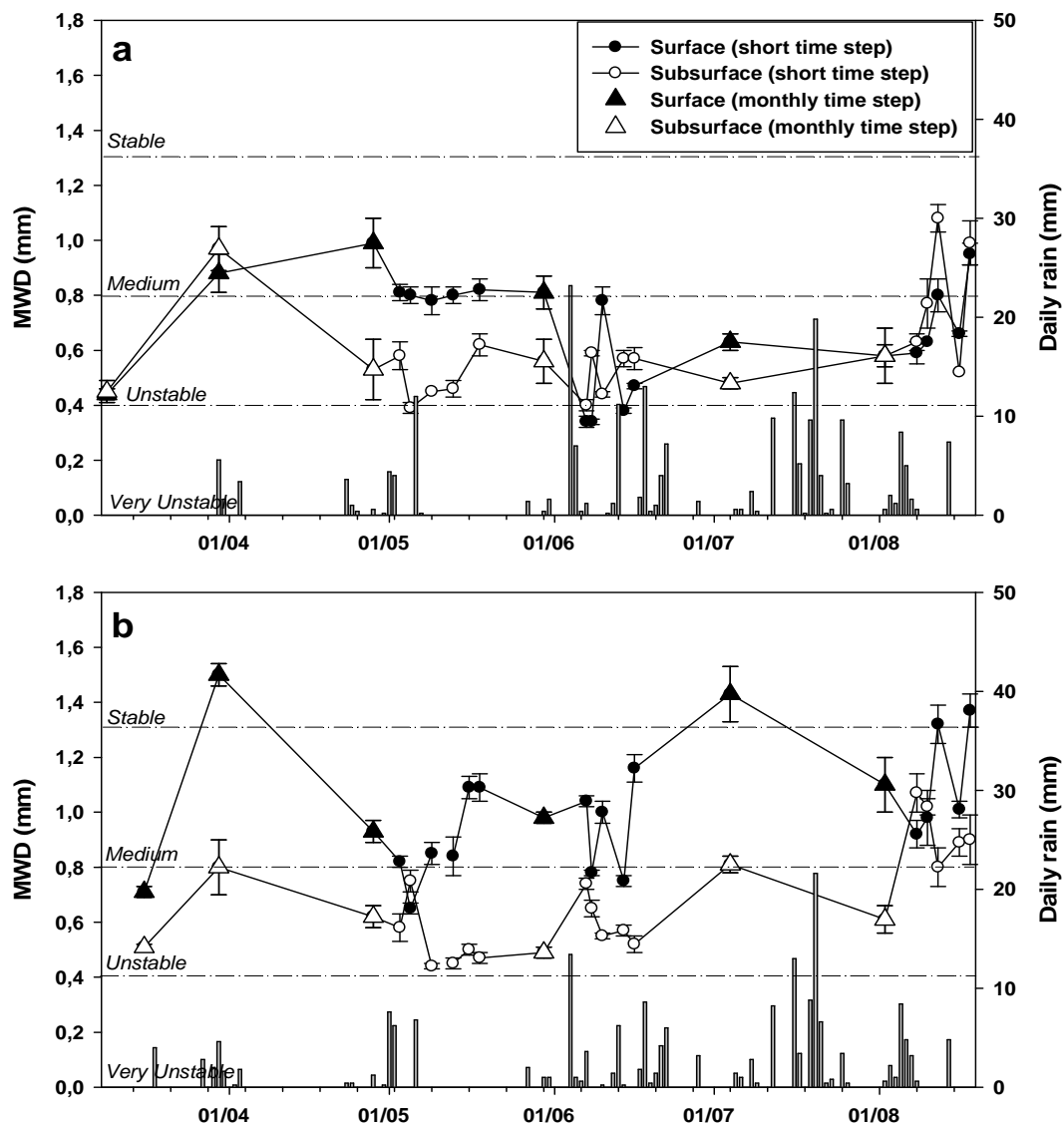


Figure 1: Temporal variation of aggregate stability for the slow wetting test of surface and subsurface for Marcheville site (a) and La Gouëthière site (b).

MWD monthly time step: each MWD corresponds to the mean of three samples located on the same plot (spatial variability) and 3 replicates for each measurements ($n=9$). Bars are standard error. MWD short time step: each MWD corresponds to the mean of three replicates ($n=3$). Bars are standard error.

Stable, Medium, Unstable, Very unstable, refers to the aggregate stability classes (Le Bissonnais, 1996).

3.1.2. Short time step variation of aggregate stability

For both sites, aggregate stability varied greatly during the monitoring, for both surface and subsurface. Such variations are considered to be significant because there are larger than the spatial variability as assessed during the monthly sampling (Figure 1 and 2).

At the Marcheville site, for the surface, MWD for the slow wetting test varied between 0.34 (very unstable) and 0.99 mm (medium stability), with a mean of 0.68 mm. For the subsurface, MWD varied between 0.39 (very unstable) and 1.08 mm (medium stability), with a mean of 0.60 mm (Figure 1). At the La Gouëthière site, for the surface, the mean MWD of the three stability tests varied between 0.65 (unstable) and 1.43 mm (stable), with a mean of 1.01 mm. For the subsurface, MWD varied between 0.44 (unstable) and 1.07 mm (medium stability), with a mean of 0.67 mm (Figure 1).

Short timestep sampling periods subsequent to rainfall events showed various trends in aggregate stability changes (Figure 2).

May:

May was characterized by two significant rain events. Rainfall 1 happened on 2 May and rainfall 2 happened on 7 May.

At the Marcheville site, rainfall 1 was characterized by a total rain height of 4 mm in 5 hours, with a maximum intensity of 2 mm/h (figure 2A). After rainfall 1, MWD of the surface dropped from 0.99 mm (28 Apr., prior to rainfall 1) to 0.81 mm (3 May). After this decrease, MWD did not change significantly at the next time step. MWD of the subsurface did not change significantly (Figure 2A) after rainfall 1. Rainfall 2 was characterized by a total rain height of 13.2 mm in 3 hours and a maximum rain intensity of 7 mm/h (figure 2A). After this rain event, MWD of the surface and the subsurface did not change significantly (Figure 2A).

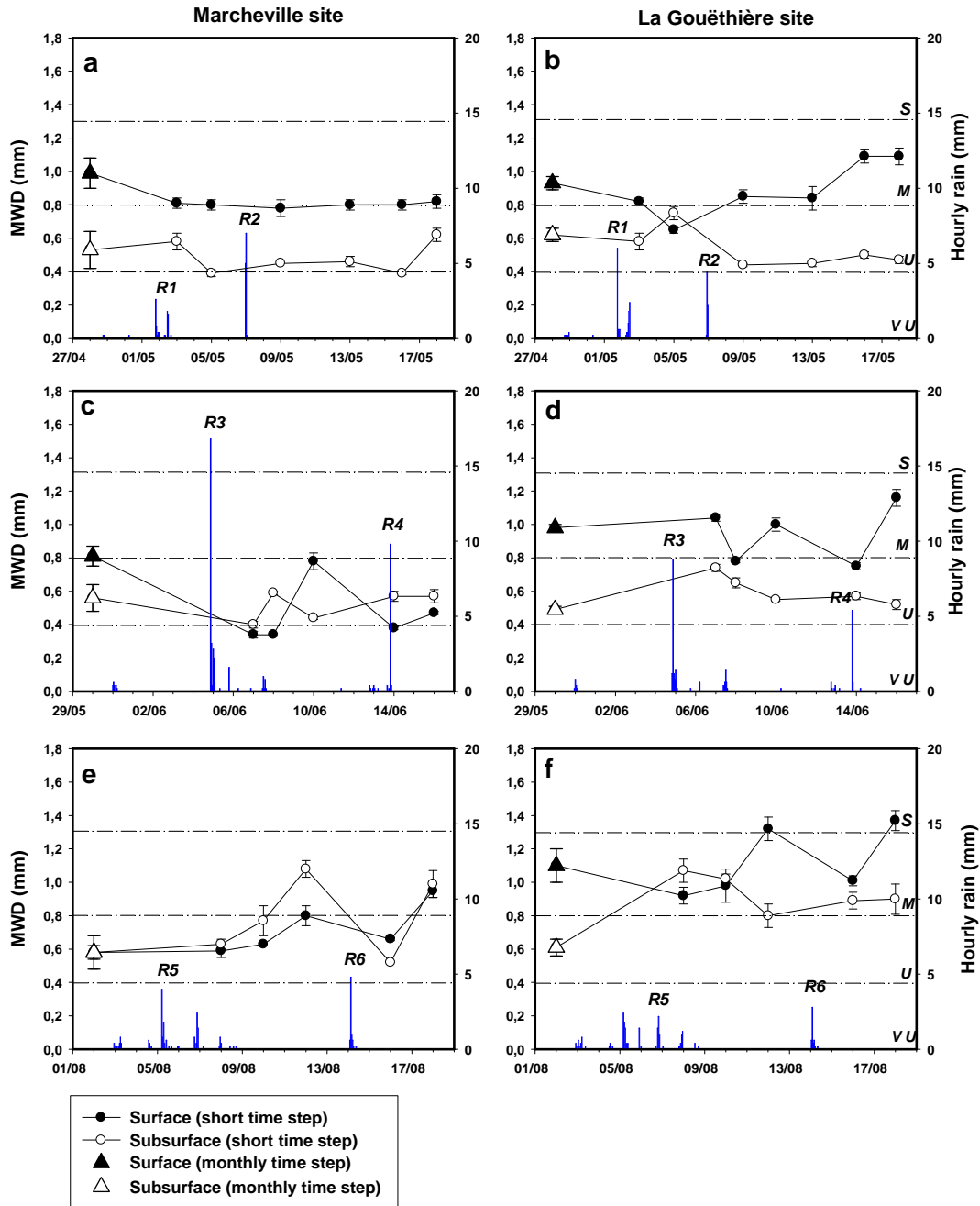


Figure 2: Temporal variation of aggregate stability for the slow wetting test at short time step for Marcheville site (a, c, e) and La Gouëthière site (b, d, f) for May period (a, b), June period (c, d) and August period (e, f). MWD monthly time step: each MWD corresponds to the mean of three samples located on the same plot (spatial variability) and 3 replicates for each measurements ($n=9$). Bars are standard error. MWD short time step: each MWD corresponds to the mean of three replicates ($n=3$). Bars are standard error.

R1, R2, R3, R4, R5, and R6 refer to the number of the rain event.

S=Stable, M=Medium, U=Unstable, VU=Very unstable (Le Bissonnais, 1996).

At the La Gouëthière site, rainfall 1 was characterized by a total rain height of 13.8 mm in 10 hours, with a maximum intensity of 6 mm/h. After this rain event, MWD values decreased from 0.93 mm (28 Apr., prior to rainfall 1) to 0.65 mm (5 May). MWD of the subsurface increased significantly from 0.62 mm (28 Apr., prior to rainfall 1) to 0.75 mm (5 May) three days after the rain event (Figure 2B). Rainfall 2 reached a total rain height of 6.6 mm in 3 hours, with a maximum intensity of 4.4 mm/h. After this rain event, MWD of the soil surface dropped from 0.65 mm (5 May, prior to rainfall 2) to 0.85 mm (9 May) and 1.09 mm (16 May). MWD of the subsurface decreased from 0.75 mm (5 May, prior to rainfall 2), to 0.44 mm (9 May) and remained stable until 18 May (Figure 2B).

June:

June was characterized by two significant rain events. Rainfall 3 happened on 4 June and rainfall 4 happened on 14 June.

At the Marcheville site, rainfall 3 reached a rain height of 26.2 mm in 7 hours, with a maximum rain intensity of 16.8 mm/h. After this rain event, MWD of the surface decreased from 0.81 mm (30 May, prior to rainfall 3) to 0.34 mm (7 June) and it then increased from 0.34 mm (8 June) to 0.78 mm (10 June). MWD of the subsurface decreased from 0.59 mm (30 May, prior to rainfall 3) to 0.44 mm (7 June) and then increased to 0.57 mm (8 June) (Figure 2C). Rainfall 4 reached a rain height of 9.8 mm in 1 hour. After this rain event, MWD of the surface decreased from 0.78 mm (10 June, prior rainfall 4) to 0.38 mm (14 June) and it then increased to 0.47 mm (16 June). MWD of the subsurface increased from 0.44 mm (10 June, prior the rain) to 0.57 mm (16 June) (Figure 2C).

At the La Gouëthière site, rainfall 3 reached a rain height of 14.2 mm in 6 hours, with a maximum rain intensity of 8.8 mm/h. After this rainfall event, MWD from the surface decreased from 0.98 mm (30 May, prior to rainfall 3) to 0.78 mm (8 June) and it then increased to 1.0 mm (10 June). MWD of the subsurface increased from 0.49 mm (30 May,

prior to rain 3) to 0.74 mm (7 June) and then decreased to 0.55 mm (10 June) (Figure 2D). Rainfall 4 reached a rain height of 6 mm in 2 hours, with a maximum rain intensity of 5.4 mm/h. After this rainfall event, MWD of the surface decreased from 1.0 mm (10 June, prior to rain 4) to 0.75 mm (June 14), and it then increased to 1.16 mm (16 June). MWD of the subsurface did not change significantly during this period (Figure 2D).

August:

August was characterized by two significant rain events. Rainfall 5 happened on 4 August, and rainfall 6 happened on 14 August.

At the Marcheville site, rainfall 5 reached a total rain height of 13.6 mm in 8 hours, with a maximum rain intensity of 4 mm/h. MWD of the surface remained stable between 2 August (0.58 mm) and 10 August (0.63 mm), and it then increased to 0.80 mm (Aug. 12). MWD of the subsurface increased from 0.58 mm (2 Aug., prior to rainfall 5) to 1.08 mm (Aug. 12) (Figure 2E). Rainfall 6 reached a total rain height of 7.4 mm in 5 hours, with a maximum intensity of 4.8 mm/h. MWD of the surface decreased from 0.80 mm (12 Aug., prior to rainfall 6) to 0.66 mm (16 Aug.), and it then increased to 0.95 mm (18 Aug.). MWD of the subsurface followed the same trend: decreasing from 1.08 mm (12 Aug., prior to rainfall 6) to 0.52 mm (16 Aug.) and then increasing to 0.99 mm (18 Aug.) (Figure 2E).

At the La Gouethière site, rainfall 5 reached a total rain height of 11.2 mm in 8 hours, with a maximum rain intensity of 2.4 mm/h. MWD of the surface decreased from 1.1 mm (2 Aug., prior to rain 5) to 0.92 mm (8 Aug.), and it then increased to 1.32 mm (12 Aug.). MWD of the subsurface followed the opposite trend: increasing from 0.61 mm (2 Aug., prior to rainfall 5) to 1.07 mm (8 Aug.) and then decreasing to 0.80 mm (12 Aug.) (Figure 2F). Rainfall 6 reached a total rain height of 4.6 mm, with a maximum intensity of 2.8 mm. Aggregate stability of the surface decreased from 1.32 mm (12 August, prior to rainfall 6) to 1.01 mm

(16 Aug.), and it then increased to 1.37 mm (18 Aug.). MWD of the subsurface did not change significantly during this period (Figure 2F).

3.2. Comparisons between aggregate stability values for the different treatments

Wilcoxon signed rank tests were performed to compare aggregate stability values as measured for the different treatments (surface/subsurface; upslope/down slope; Marcheville/La Gouëthière) (Table 2).

Table 2: statistical significance level for the effect of each treatment (surface/subsurface; upslope/down slope; Marcheville/La Gouëthière) on aggregate stability values for the three stability tests.

	Fast wetting	Slow wetting	Stirring	Mean of the 3 tests
Marcheville				
Upslope: Surface/Subsurface	**	NS	NS	NS
Down slope: Surface/Subsurface	NS	NS	NS	NS
Upslope/Down slope	NS	*	NS	NS
La Gouëthière				
Upslope: Surface/Subsurface	**	**	NS	**
Down slope: Surface/Subsurface	NS	*	*	*
Upslope/Down slope	NS	NS	**	NS
Marcheville/La Gouëthière				
Surface sample set for upslope	**	**	*	**
Subsurface sample set for upslope	*	NS	NS	NS
Surface sample set for down slope	*	*	**	**
Subsurface sample set for down slope	NS	NS	NS	NS

NS, not significant at the 10% level

. Significant at the 10% level with Wilcoxon's statistic.

** Significant at the 5% level with Wilcoxon's statistic.*

*** Significant at the 1% level with Wilcoxon's statistic.*

At the Marcheville site, for the upslope plot, the difference in MWD between the surface and the subsurface was significant at the 5% level for the fast wetting test. However, differences in MWD between the surface and subsurface were not significant for the slow wetting test, the stirring test or the mean of the three tests. For the downslope plot, the difference in MWD between the surface and subsurface was not significant. The difference in MWD between the upslope and the down slope was significant at the 5% level for the slow wetting test, but it was not significant for the fast wetting test, the stirring test or the mean of the three tests.

At the La Gouëthière site for the upslope plot, the difference in MWD between the surface and the subsurface was significant at the 5% level for the fast wetting test, the slow wetting test and the mean of the three tests, but it was not significant for the stirring test. For the downslope plot, the difference in MWD between the surface and subsurface was significant at the 5% level for the slow wetting test, the stirring test and the mean of the three tests, but it was not significant for the fast wetting test. The difference in MWD between the upslope and the downslope was significant at the 5% level for the stirring test, but it was not significant at the 5% level for the fast wetting test, the slow wetting test or the mean of the three tests.

Considering the upslope plots for the surface, the difference in MWD between the Marcheville and La Gouëthière sites was significant at the 5% level for the three stability tests. However, for the subsurface sample sets, the difference in aggregate stability between the Marcheville and La Gouëthière site was not significant at the 5% level for the slow wetting test, the stirring test or the mean of the three tests. Considering the downslope plots, the difference in MWD between the Marcheville and La Gouëthière sites was significant at the 5% level for the three stability tests for the surface, but it was not significant for the subsurface regardless of the stability test.

3.3. Relationships between aggregate stability variations for the different treatments

3.3.1. Relationship between MWD of the surface and MWD of the subsurface

A correlation analysis was performed between the aggregate stability (MWD) of the surface sample set and the subsurface sample set (Table 3). This analysis was conducted separately for the two sites and for the two plots of each sites (upslope and downslope), for the three stability tests and for the mean of the three tests. For the fast wetting test, MWD of the surface was significantly correlated with MWD of the subsurface, except for at the Marcheville downslope plot. For the slow wetting test, MWD of the surface did not correlate significantly with the aggregate stability of the subsurface regardless of site or plot. For the stirring test, MWD of the surface did not correlate significantly with MWD of the subsurface. The only significant relationship was found between the surface and the subsurface aggregate stability at the La Gouëthière downslope plot. Finally, the mean of the three tests of MWD at the surface, for both sites and plots, did not significantly correlate with the aggregate stability of the subsurface.

Table 3: coefficient correlation (Pearson's coefficient) between surface and subsurface samples for Marcheville and La Gouëthière sites (upslope and down slope plots).

	Marcheville		La Gouëthière	
	Upslope	Down slope	Upslope	Down slope
Fast wetting	0.48*	0.32 (NS)	0.52*	0.40(NS)
Slow wetting	0.31(NS)	0.39(NS)	0.28 (NS)	0.36(NS)
Stirring	0.37 (NS)	0.39(NS)	0.40 (NS)	0.77*
Mean of the 3 tests	0.25 (NS)	0.38 (NS)	0.34 (NS)	0.4 (NS)

Upslope dataset: N= 20; $\alpha=5\%$; $r=0.434$

Downslope dataset: N= 11; $\alpha=5\%$; $r=0.576$

** Significant at the 5% level*

3.3.2. Relationship between aggregate stability of two plots on the same site (upslope and downslope).

A correlation analysis was performed between the aggregate stability (MWD) of the upslope plot and the aggregate stability of the downslope plots located on the same field site (Table 4). This analysis was conducted for the two sites separately, for both surface and subsurface separately and for the three stability tests. For the two sampling sites, for both surface and subsurface sample sets and for each test, significant correlations were found between MWD from the two plots located in the same field, except for the MWD measured by the stirring test in the Marcheville subsurface.

Table 4: coefficient correlations (Pearson's coefficient) between upslope and down slope plots, for Marcheville and La Gouëthière sites, and for surface and subsurface samples.

	Marcheville		La Gouëthière	
	Surface	Subsurface	Surface	Subsurface
Fast wetting	0.84*	0.81*	0.63*	0.64*
Slow wetting	0.85*	0.74*	0.52*	0.67*
Stirring	0.74*	0.54(NS)	0.63*	0.76*
Mean of the 3 tests	0.85*	0.76*	0.75*	0.63*

Marcheville sample sets: N=11; $\alpha=5\%$; and $r=0.602$

La Gouëthière sample sets: N=17; $\alpha=5\%$; and $r=0.482$

** Significant at the 5% level*

3.3.3. Relationship between aggregate stability of the Marcheville site and the La Gouëthière site

A correlation analysis was performed between the aggregate stability (MWD) of the Marcheville site and the La Gouëthière site (Table 5). This analysis was conducted for the two plots at each site, for the surface and the subsurface separately and for the three stability tests. For the fast wetting test and for the stirring tests, the MWD of the Marcheville site was significantly correlated with the MWD of the La Gouëthière site. These significant

correlations were observed for both upslope and downslope plots and for both surface and subsurface. For the slow wetting test, the MWD of the Marcheville site did not correlate significantly with the MWD of the La Gouëthière site, for both the upslope and the downslope plots and the surface and the subsurface. Finally, considering the mean of the three stability tests, the MWD of the Marcheville site did not significantly correlate with the MWD of the La Gouëthière site for the surface sample set, but it did correlate significantly for the subsurface sample set.

Table 5: coefficient correlation (Pearson coefficients) between Marcheville soil and La Gouëthière site for upslope and down slope, and for surface and subsurface datasets.

	Upslope		Downslope	
	Surface	Subsurface	Surface	Subsurface
Fast wetting	0.58*	0.58*	0.73*	0.62*
Slow wetting	0.23 (NS)	0.43 (NS)	0.41(NS)	0.24(NS)
Stirring	0.71*	0.56*	0.73*	0.61*
Mean of the 3 tests	0.41 (NS)	0.49*	0.73*	0.76*

Upslope dataset: N= 20; $\alpha=5\%$; $r=0.434$

Downslope dataset: N= 11; $\alpha=5\%$; $r=0.576$

** Significant at the 5% level*

3.4. Relationship between aggregate stability and rain height

A correlation analysis was performed between the aggregate stability (MWD) and the antecedent precipitation index (Table 6). This analysis was conducted for both plots at the Marcheville and the La Gouëthière sites, separately for the surface sample set and the subsurface datasets and for the three stability tests. For both the Marcheville and the La Gouëthière sites, surface MWD was significantly negatively correlated with the antecedent precipitation index for the fast wetting test, the slow wetting test and the mean of the three tests. Subsurface MWD did not significantly correlate with the antecedent precipitation index

for either site or plot. MWD measured by the stirring test did not significantly correlate with the antecedent precipitation index for either sites, plot, or either surface or subsurface.

Table 6: coefficient correlation between aggregate stability and the antecedent precipitation index (API) for Marcheville and La Gouëthière soil (upslope and down slope plots), for surface and subsurface and for the three aggregate stability tests.

	Marcheville				La Gouëthière			
	Upslope		Down slope		Upslope		Down slope	
	Surface	Subsurface	Surface	Subsurface	Surface	Subsurface	Surface	Subsurface
Fast wetting	-0.63*	-0.19 (NS)	-0.76*	-0.37(NS)	-0.51*	0.19 (NS)	-0.61*	-0.24(NS)
Slow wetting	-0.65*	-0.25 (NS)	-0.64*	-0.15(NS)	-0.49*	0.33 (NS)	-0.31(NS)	0.04(NS)
Stirring	-0.27 (NS)	0.17 (NS)	-0.44(NS)	-0.33(NS)	-0.24 (NS)	-0.08 (NS)	-0.44(NS)	-0.15(NS)
Mean of the 3 tests	-0.52*	-0.18 (NS)	-0.68*	-0.34(NS)	-0.53*	0.22 (NS)	-0.58*	-0.12(NS)

Upslope dataset: N= 20; $\alpha=5\%$; $r=0.434$

Down slope dataset: N= 11; $\alpha=5\%$; $r=0.576$

**Significant at the 5% level*

4. Discussion

4.1. Aggregate stability varied at short time step

The present study demonstrated that the aggregate stability of a given soil varied greatly even at short time steps (of a few days). The temporal variability of the aggregate stability was larger than the spatial variability of the aggregate stability, as measured for a given field site. Indeed, for the duration of the monitoring, aggregate stabilities varied between 20% and 30%, depending upon treatment, while the spatial variability was only 10%. In some cases, variations in aggregate stability at short time steps induced changes of at least two stability classes (Le Bissonnais, 1996). These large changes in aggregate stability have been observed for both surface and subsurface materials and both plots located at each site. For example, in the Marcheville site upslope plot, the aggregate stability of the soil surface, as measured by

the slow wetting test, dropped from 0.81 mm to less than half of its value seven days later (0.34), and it then rebounded to 0.78 mm only two days later. This result corresponds to a variation from a “medium stable” soil to a “very unstable” soil and then back to a “medium stable” soil, according to the Le Bissonnais stability classes (1996).

Temporal variations in aggregate stability have already been observed at monthly time steps (Blackman, 1992; Bajracharya et al., 1998; Suwardji & Eberbach, 1998; Dimoyiannis, 2009). Such studies have shown that the temporal variability of the aggregate stability, as measured at monthly time steps over a year, varied between 20% and 30%, depending upon the soil type. The present study showed similar variability in aggregate stability (between 20% and 30%, depending upon the treatment), measured at short time steps (of a few days) over a 5 month period.

4.2. Influences of the different treatments on aggregate stability temporal variability

4.2.1. Difference between surface and subsurface samples aggregate stability

For the same field plot, the aggregate stability of the soil surface can be different from the aggregate stability of the soil subsurface. If differences are observed at a given time, they do not always remain the same. Moreover, the results differed depending upon the study site and the aggregate stability test used. At the Marcheville site, the aggregate stability average over the whole sampling dates of the field monitoring did not differ significantly between the surface and the subsurface sample sets. At the La Gouëthière site, the aggregate stability average over the whole sampling range differed significantly between surface and subsurface aggregate stability. These results suggest that the aggregate stability of the surface and the subsurface varied within the same range at the Marcheville site but within different ranges at the La Gouëthière site. Generally, analyses of the relationship between the aggregate stability of the surface and of the subsurface did not show significant correlations. These results

suggest that, for the two sites, aggregate stabilities of the surface and of the subsurface did not follow the same trends in variability. Such results explain how, at a given time, the aggregate stabilities of the surface can be very different than the aggregate stabilities of the subsurface. Moreover, these differences changed temporally. Surface material can be either more stable or less stable than the subsurface material, depending upon the date.

Differences in aggregate stability between the surface and the subsurface have already been observed in previous studies (Poesen, 1981; Kuhn & Bryan, 2004; Darboux & Le Bissonnais, 2007; Algayer et al., under review). Such studies have presented contradictory results. The aggregate stability of crusted material was found to be larger than the aggregate stability of its underlying material (Poesen, 1981; Kuhn & Bryan, 2004; Algayer et al., under review). However, through simulated rainfall experiments, Darboux and Le Bissonnais (2007) showed that the aggregate stability of structural crust was similar to the aggregate stability from its original uncrusted material. The present study showed that this difference can vary greatly with time and even at short time steps. The surface of a crusted soil can present with higher, similar or even smaller aggregate stabilities than its underlying material, depending upon the date. Further research must be performed to better understand the factors and processes controlling differences in aggregate stability between the surface and subsurface materials in crusted soils.

4.2.2. Difference between aggregate stability of two plots located on the same crop field

For each field site (Marcheville and La Gouëtère), sampling was conducted in two 50 m² plots located at different points in the field (upslope and downslope). Soil properties and crop management were the same for the two plots in each field. Considering the aggregate stability average over the whole sample, no significant differences were found between each of the two plots located in the same field. Analyses of the relationships between aggregate

stability showed significant correlations for the three stability tests. These relationships were observed for the two sites and for both surface and subsurface materials. These results suggest that, for two plots located in the same field, aggregate stabilities vary in the same range and follow nearly the same trends in variation.

4.2.3. Differences between the aggregate stability of two field sites located in similar soil types

The two studied field sites (Marcheville and La Gouëthière) presented very similar soil types (silt loam Luvisols). The Marcheville soil was a typical Luvisol, while the La Gouëthière soil was a degraded Luvisol. The Marcheville soil A horizon presented with slightly higher clay content and organic matter content than the La Gouëthière soil, although both soils had the same pH (6.7) and very close Ca, Mg, K and Na content. Both sites were located at very short distances (5 km) from each other, and they were exposed to similar climatic conditions in terms of rainfall amounts and temperatures. However, both sites presented with differences in slope orientations, which could induce differences in microclimate or hydric history. Considering cultural practices, the Marcheville site was located in a wheat crop field and the La Gouëthière site was located in a pea crop field; however, for both sites, the soil was well drained and the sampling plots were kept bare for the duration of monitoring.

Significant differences were found in the average aggregate stability of the whole sampling range between the Marcheville and the La Gouëthière sites for the surface material, but no significant differences were found between the two sites for the subsurface material. Significant correlations were found between aggregate stabilities from the Marcheville and the La Gouëthière sites for both the surface and the subsurface materials. These results mean that, in general, the aggregate stability of the soil surface varied between the two sites within contrasting ranges and followed similar trends in temporal variation. Aggregate stabilities of

the subsurface varied within the same range and followed the same trends in temporal variation.

It is widely recognized that temporal variation in aggregate stability is mainly controlled by climate and biological activity (Amézketa, 1999). The fact that both sites were exposed to the same climatic conditions explains why aggregate stabilities from both sites followed the same temporal trend. However, variability in the aggregate stabilities of the surface remained difficult to explain in the present case. Indeed, because the soil properties from both sites were so close and because the land use was similar, similar aggregate stability ranges are expected. However, this was not the case in our present study. More detailed research should be performed to explain differences in aggregate stability between both sites.

4.3. Relationship between aggregate stability variation and precipitation

For both sites, the aggregate stability of the soil surface was negatively correlated with the antecedent precipitation index. Such negative influences of rain on surface aggregate stability have already been observed in previous studies (e. g. Blackman, 1992; Shainberg et al. 2003; Dymoyiannis, 2009). Through monthly field monitoring, Blackman (1992) and Dimoyiannis (2009) showed negative correlations between the aggregate stability and the total monthly rainfall. The present study showed that this relationship also influenced the variability of the surface aggregate stability over short time steps (of a few days). The negative effects of the rain on the aggregate stability of the soil surface were clearly observed during the short sampling periods. Surface aggregate stability, measured from samples collected immediately after the rainfall event, tended to be inferior to those measured before the rainfall event. Further, any additional rain tended to increase the surface aggregate stability subsequent to the last rainfall. These significant relationships were not measured between the antecedent precipitation index and the aggregate stability of the soil subsurface. The lack of a significant relationship suggests that the negative effect of rainfall on aggregate

stability does not extend to the sub-surface. Indeed, the surface aggregates are directly submitted to raindrop impact, and they may protect the underlying material from the influences of the rain.

The influences of rainfall on aggregate stability can be linked to several processes. First, the kinetic energy from water drops impacting on the soil surface induces a disruptive effect for the inter-particle bonds within the aggregates. This, in turn, decreases aggregate stability (Shainberg et al. 2003; Lehrs & Kincaid, 2006). Moreover, the increase in soil water content induced by the rain affects the aggregate stability by slaking (differential swelling of the clay and clay dispersion; Perfect et al. 1990; Le Bissonnais, 1996; Zaher et al. 2005). Previous studies have shown that aggregate stability is negatively correlated with soil water content (Perfect et al., 1990; Caron et al., 1992; Chan & Mullins, 1994; Shainberg et al. 2003). On the other hand, soil drying after a rain event is recognized to increase aggregate stability (Kemper & Rosenau, 1984; Kemper et al. 1987; Dexter et al. 1988). Finally, soil moisture variations induced by rain can affect biological activity and indirectly influence the aggregate stability (Perfect et al. 1990; Cosentino et al. 2006).

Considering the results of the present study, the antecedent precipitation index is a dominant factor in soil surface aggregate stability variation over short time steps. Although factors predicting aggregate stability variation (and linked to rainfall) have already been identified, it is still difficult to predict aggregate stability variation accurately. The relationships between aggregate stability, biological activity and soil water content dynamics have to be studied further to better predict temporal variations in aggregate stability. Indeed, recent studies have shown that the influence of microbial activity on aggregate stability depends upon the structure of the microbial population (Leguillou et al., 2012). Interrelationships between variables linked to biological activity and climatic variables seem to be the key to aggregate stability prediction.

4.4. Consequences for erodibility assessment and erosion modeling

Aggregate stability is recognized as a proxy for soil erodibility (Bajracharya et al., 1998; Barthes & Roose, 2002; Gumiere et al., 2009). High aggregate stability corresponds to low erodibility. Currently, erosion models rarely predict water erosion accurately (Jetten *et al.*, 2003; Boardman, 2006). Biases in erodibility estimations may partially explain these difficulties. The results of the present study point to some possible sources of these biases in erodibility assessments and predictions.

When assessed by aggregate stability, soil erodibility is usually measured in samples collected from the plough layer (e. g. Bullock *et al.*, 1988; Bajracharya et al., 1998; Barthès & Roose, 2002). The present study demonstrates that erodibility from the soil surface can be very different from the erodibility of its underlying material. As erosion processes occur first at the soil surface, erodibility has to be assessed from samples from the soil surface. Measurements of erodibility in samples collected in the plough layer may be a large source of bias in erodibility assessments at any given time.

Our results also demonstrated that intra-field variability in erodibility was very low for the two studied fields, meaning that the assessment of the erodibility of a crop field can be performed on a single plot and extended to the whole field. This result is only valid for uniform crop fields with similar soil properties, and it must be tested for different soil types.

Erosion models currently calculate erodibility as a function of soil properties, assuming that one soil type corresponds to one erodibility value (Jetten *et al.*, 2003; Boardman, 2006; Gumiere *et al.*, 2009). This is the case for the widely used RUSLE model (Renard et al., 1997) and the WEPP model (Alberts et al., 1995). As the soil properties were very similar for our two sites and the land uses were similar (bare soils), we might have expected homogeneity of aggregate stability. This was not the case for surface aggregate stability, as the Marcheville and the La Gouëthière sites presented with contrasting ranges of

surface aggregate stability, and thus contrasting ranges in erodibility. In a previous study, Algayer et al. (under review) showed that the erodibility of 7 crusted soils with similar properties (particle size, organic matter, CEC and pH) could be very different. These results indicate that erosion models with fine temporal resolution should take into account the variability of erodibility within a given soil. The computation of a single erodibility value for a single soil type as a function of its soil properties has important limitations. Such a simplification of the erodibility assessment could explain, in part, the present difficulties in accurately modeling erosion (Jetten *et al.*, 2003; Boardman, 2006).

Moreover, current parameterizations of erosion models typically set a single erodibility value for a given soil over time. This is the case, for example, in the widely used WEPP model (Alberts et al., 1995). In the best case, the parameterization of the model sets an average erodibility value for a season. However, the present study showed that the temporal variability of the aggregate stability over short time steps was about the same as the temporal variability of the aggregate stability measured at monthly or seasonal time steps. For lack of a better solution, the erodibility values for a given soil should be computed as a range of values corresponding to its temporal variation in amplitude: one soil type corresponds to one range of erodibility values.

Based upon our current knowledge, and considering the latter points of our discussion, soil erodibility should be measured at a very high spatial and temporal resolution, which could be impractical. Thus, the current challenge is to accurately predict variations in aggregate stability over short time steps. The present study pointed to rainfall as a factor in aggregate stability variation over short time steps. Such relationships should be studied in depth in the future.

5. Conclusion

Aggregate stability varied greatly over short time steps. These results were observed regardless of the studied treatment. Such variations in aggregate stability correspond to contrasting erodibility values. Parameterizations of erosion models for a given soil type should consider these short time step variations by replacing single erodibility values (computed as a time constant), with ranges corresponding to the temporal variations in amplitude of the soil erodibility. Temporal variations in aggregate stability are differentially influenced by the studied treatments. The aggregate stability of the two sites, located in similar soil types, followed the same trends in variation but had contrasting ranges in value. The aggregate stability of two plots located on a same field site varied in the same range and followed the same trends in variation. The influence of the soil surface and the subsurface on the aggregate stability was less clear and depended upon the sampling site. Finally, rainfall, as assessed by the API, was negatively correlated with soil surface aggregate stability regardless of treatment. API can be considered to be a factor in aggregate stability variations over short time steps. These relationships need to be studied in depth by future researchers.

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Chapitre 4 :

Evaluation des facteurs explicatifs de la variation de la stabilité structurale à pas de temps court

Short-term dynamics of aggregate stability in the field.
Assessment of explanatory factors to improve erodibility
predictions

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Abstract

Predicting temporal variation in soil erodibility is a current challenge in improving model predictions of erosion. Soil interrill erodibility can be assessed using measurements of aggregate stability. However, even when the factors controlling seasonal variations in aggregate stability have been identified, it remains difficult to accurately predict aggregate stability variations in the field. These difficulties may reflect an inadequacy between the temporal resolution of the field monitorings (e.g., a monthly time step) and the hypothesized explanatory variables for aggregate stability; better predictions of aggregate stability variations in the field require short time step monitoring. Field monitoring was conducted in bare soil with objectives of 1) identifying factors of aggregate stability that varied over the short term and 2) predicting these variations using variables associated with biological activity and climate. Soil water content at the time of sampling and soil hydric history were found to be dominant factors influencing short time step aggregate stability variations for the soil surface. Variables associated with biological activity did not explain aggregate stability variations. These results highlight the dominant influence of abiotic factors such as water content dynamics on short time step aggregate stability variations in the field when biological activity is not stimulated with amendments. A regression model that included hydric history indices predicted up to 59% of surface aggregate stability. Because aggregate stability is a proxy for soil interrill erodibility, prediction of aggregate stability could improve the parameterization of erodibility in soil erosion models. Further research on the processes involved in aggregate stability variations associated with hydric history would also improve erodibility predictions.

1. Introduction

Soil erodibility corresponds to soil sensitivity to erosion and is therefore a key parameter for erosion modeling and prediction. Soil erodibility is known to vary seasonally (Coote *et al.*, 1988; Bajracharya *et al.*, 1998; Salvador Sanchis *et al.*, 2008) and at a short time step (Algayer *et al.*, to be submitted). Even when factors of erodibility variation have been identified, it remains difficult to accurately predict variations in erodibility (Salvador Sanchis *et al.*, 2008). Without better information, existing erosion models treat soil erodibility for a given soil as a constant in space and time, leading to bias in erosion predictions (Jetten *et al.*, 2003; Algayer *et al.*, under review). Thus, predicting erodibility variations is a current challenge in improving model predictions of erosion.

Aggregate stability describes the ability of an aggregate to retain its structure when exposed to water. In the context of water erosion, aggregate stability is recognized as a proxy for soil water interrill erodibility: the higher the aggregate stability, the lower the soil erodibility (Le Bissonnais, 1996; Barthes & Roose, 2002). Many studies have shown that the aggregate stability in a given soil changes with time (e. g. Bullock *et al.* 1988; Bajracharya *et al.* 1998; Cosentino *et al.* 2006; Dimoyiannis, 2009, Algayer *et al.*, to be submitted). Seasonal variations have been identified through field monitoring on a monthly time step (Bullock *et al.* 1988; Bajracharya *et al.* 1998; Dimoyiannis, 2009). Short-term variability (from several days to several weeks) in aggregate stability and factors thereof have been identified in field monitoring (Caron *et al.*, 1992; Algayer *et al.*, to be submitted) and laboratory experiments (e. g. Kemper *et al.* 1987; Cosentino *et al.* 2006).

The factors that influence aggregate stability variations are linked to biological activity and to climate. It is well established that microbial activity and organic matter content have a positive effect on aggregate stability (e.g., Tisdall & Oades, 1982; Chenu *et al.* 2000). Organic matter additions stimulate microbial activity and thus indirectly increase aggregate

stability (Tisdall & Oades, 1982, Le Guillou *et al.*, 2012). Microbial activity stabilizes aggregates through several processes, such as bacterial production of extracellular polysaccharides that bind mineral particles together (Tisdall & Oades, 1982) and the production of hydrophobic substances that decrease the wetting rate (Piccolo & Mbagwu 1999; Cosentino *et al.* 2006). The influence of climate on aggregate stability is much more complex. Soil temperature affects aggregate stability directly through the freezing process (Bullock *et al.* 1988) and indirectly through seasonal stimulation of microbial activity (Suwardji & Eberbach, 1998). Rain affects aggregate stability of the soil surface through several processes, including the kinetic energy of raindrop impact and slaking (Shainberg *et al.*, 2003; Lehrsch & Kincaid 2006). Soil water content at the time of sampling was found to correlate negatively with aggregate stability (Perfect *et al.* 1990; Caron *et al.*, 1992). Soil hydric history affects aggregate stability through physico-chemical processes (Utomo & Dexter, 1982; Kemper & Rosenau, 1984) and through its influence on microbial activity (Denef *et al.* 2001). Even when the factors influencing aggregate stability variation have been identified, it is difficult to accurately predict aggregate stability variations. Algayer *et al.* (under review) showed that standard soil properties did not allow for the prediction of aggregate stability and promoted monitoring of climatic variables and biological activity to improve predictions.

Moreover, factors controlling aggregate stability have been identified almost entirely through laboratory experiments. Linking these explanatory variables to aggregate stability variation in the field remains a challenge. These difficulties could be explained by inadequate temporal resolution of field monitoring (e.g., on a monthly time step). Indeed, one sample per month is insufficient to record the full range in the dynamics of those variables considered to control aggregate stability (e.g., soil water content and microbial biomass). Monitoring on a

shorter time step (a few days) is required to more precisely evaluate the relationships between aggregate stability and its controlling factors.

In a previous study, we found that aggregate stability varied greatly for both surface and subsurface material, even on a short time step (few days) (Algayer *et al.*, to be submitted). This study also revealed a close relationship between rainfall and aggregate stability at the surface (Algayer *et al.*, to be submitted): aggregate stability tended to decline after a rain event. The objectives of the present study were the following: 1) to identify those variables linked to biological activity and climate that control aggregate stability variations at a short time step; and 2) to predict short time step variations in aggregate stability in order to improve the accuracy of model predictions of erosion.

A field monitoring on a short time step (few days) was conducted on two Luvisols located in the southern portion of the Parisian basin (France). Aggregate stability, soil properties and climatic variables were measured, and hydric history was identified as the dominant factor controlling short time step aggregate stability variation. A multiple regression model combining hydric history indices predicted approximately 60% of variation in aggregate stability.

2. Material and method

2.1. Sampling sites

Field monitoring was performed on two sites, Marcheville (48°21'512"N; 1°16'0.55"E) and La Gouëthière (48°22'489"N; 1°12'100"E), located in the southern portion of the Parisian Basin, 15 kilometers southwest of the city of Chartres. The field sites were located on two cultivated Luvisols and were geographically very near one another (5 kilometers). Both soils were silt loam soils (Soil Survey Division Staff, 1993). The soil surface (A horizon) properties of each site are presented in Table 1. The Marcheville site was located on a typic

Luvisol with a gentle slope (7%) oriented to the north, and the La Gouthière site was located on a degraded Luvisol with a gentle slope (5%) oriented to the southwest. Sampling was performed in the A horizon of the soils. The Marcheville A horizon was 16% clay and 2.2% organic matter, while the La Gouëthière A horizon was 11% clay and 1.4% organic matter (table 1). Other characteristics of the two soils were very similar (table 1).

Table 1: Soil properties for the two sampling sites

	Culture (actual/ antecedent)	Clay (g/kg)	Silt (g/kg)	Sand (g/kg)	Organic matter (g/kg)	CEC (cmol/kg)	pH	Ca (g/kg)	Mg (g/kg)	Ka (g/kg)	Na (g/kg)
Marcheville	Wheat/Maize	164	798	38	21.6	9.1	6.7	8.8	0.5	0.6	0.03
La Gouëthière	Pea/Wheat	113	848	39	13.7	6.7	6.7	6.6	0.4	0.2	0.04

2.2. Monitoring and sampling setup

Within the agricultural field on each site, a 50 m² plot was defined. To minimize the influence of vegetation on changes in soil properties, plots were kept bare during the 6-month monitoring period using herbicides. Monitoring began just after seedbed preparation, on March 9 and 16, 2011, for the Marcheville and La Gouëthière sites, respectively, and ended on August 18, 2011. After May 5, the soil surfaces of both sites presented a structural crust, which, on the Marcheville site, developed into a sedimentary crust after August 2 (Bresson & Boiffin, 1990).

Samples were collected at short intervals, every 2 to 3 days during the 2 weeks subsequent to each of three significant rain events. Field sampling was conducted 7 times between May 3 and May 18, 5 times between June 7 and June 16 and 5 times between August 8 and August 18. On each sampling date, samples were collected from a one-m² subplot within each plot. Each subplot was sampled only once.

Paired samples of surface and subsurface material were collected separately from each subplot. For the surface samples, material was collected from the top 5 millimeters. When the

soil surface was crusted, large pieces of the crust material were collected using a sharp knife to cut through the material without affecting its structure. The subsurface material was defined as the material between 1 cm and 5 cm below the soil surface.

2.3. Measurements

2.3.1. Aggregate stability

Aggregate stability was measured using the Le Bissonnais method (Le Bissonnais 1996; ISO/DIS 10930, 2012). Two tests, the fast-wetting and slow-wetting tests, were performed (Le Bissonnais, 1996). These tests were designed to reproduce the slaking process and differential swelling of the clay which are involved in interrill erosion. 5 g sub-samples were dried at 40°C for 24 h prior to each test, and each test was replicated three times. Following each test, the resulting fragments were sieved in ethanol, and results are presented using the mean weighted diameter of the fragments (MWD) (Le Bissonnais, 1996).

2.3.2. Variables linked to biological activity

Variables associated with biological activity were measured on the samples collected from the soil surface and subsurface including organic matter content, microbial biomass and subcritical water repellency. Organic matter content was measured using the sulfochromique oxidation method (NF ISO 14235), microbial biomass using the fumigation method (NF ISO 14240-2, 1997), and subcritical water repellency using the intrinsic sorptivity method (Tillman *et al.* 1989). A water repellency index (R) was determined from the sorptivity measurements of two wetting liquids with different solid-liquid contact angles: water and ethanol (Tillman *et al.* 1989). R was evaluated with sorptivity measurements taken at -4 cm pressure head for both liquids, and measurements were performed using the experimental design described by Hallett & Young (1999). Measurements were performed on 1-cm-diameter aggregates collected from the soil surface only. When the soil surface was crusted,

measurements were made on 1 cm² crust fragments, on the top of the fragment. Samples were dried at 40°C during the 48 h prior to measurements. The R value presented for each sampling date corresponds to the mean of 10 replicates. An R index equal to 1.0 corresponds to a completely non-repellent soil, an R index between 1.0 and 1.95 corresponds to a non-repellent soil and an R index higher than 1.95 corresponds to a subcritical water repellent soil. For R indexes above 1.95, the greater the R index, the greater the water repellency.

2.3.3. *Variables associated with climate*

The measured variables associated with climate include air relative humidity and temperature, soil water content and temperature and rain height. Gravimetric water content was measured on the samples collected from the surface and subsurface. Volumetric soil water content was measured hourly during TDR monitoring (Decagon Devices, soil moisture sensor 5TE) at both depths (1 cm and 5 cm). This probe was also used to measure soil temperature. Volumetric water content and soil temperature were measured at two different points in each plot. Air relative humidity and temperature were measured hourly (Vaisala, HMP45C). Rain height was measured hourly using a pluviometer (Campbell Scientific, ARG 100) (one for each site). The antecedent precipitation index (API) was calculated for 7 days prior to aggregate stability measurement as:

$$API = \sum_{i=0}^6 \frac{10-i}{10} * P_i$$

where i is the i^{th} day before sampling and P_i (in millimeters) is the total precipitation height on the i^{th} day.

To characterize the hydric history of the soil, two indices were calculated from the water content data: the mean of hourly water content values for a given period prior to sampling (WC_t) and the difference in water content between the beginning and end of that period (ΔWC_t). Both indices were calculated for periods of varying duration prior to sampling.

Simple correlation coefficients (Pearson coefficients) were calculated between aggregate stability values (MWD), and the two hydric history indices calculated for durations between 4 and 200 hours before sampling.

2.4. Statistical analysis

Statistical analyses were completed using R software version 2.9.2 (R Development Core Team, 2011). Linear correlation analyses (Pearson's coefficient) were used to identify relationships between aggregate stability variation and the measured variables. This analysis was completed separately for each site (Marcheville and La Gouëthière) and for each surface and subsurface dataset. Only those correlation coefficients found to be significant at the 5% level were considered. To predict aggregate stability variations, simple and multiple regression analyses were completed for variables that were significantly correlated with aggregate stability.

3. Results

3.1. Aggregate stability

3.1.1. Marcheville site

On the Marcheville site, for the fast wetting test, MWD of the soil surface varied between 0.17 mm (June 8) and 0.47 mm (May 30), with a mean of 0.32 mm (Figure 1A). For the slow wetting test, MWD of the soil surface varied between 0.34 mm (June 8) and 0.99 mm (April 28), with a mean of 0.68 mm (Figure 1C). Variation coefficient of the surface MWD reached 27% for the fast wetting test, and 28% for the slow wetting test. On the subsurface for the fast wetting test, MWD varied between 0.20 mm (July 4) and 0.36 mm (May 30) with a mean of 0.26 mm (Figure 1A). For the slow wetting test, subsurface MWD varied between 0.39 mm (May 5) and 1.08 mm (August 12), with a mean of 0.59 mm (figure

1C). The variation coefficient of the surface MWD reached 17% for the fast wetting test, and 31% for the slow wetting test.

3.1.2. La Gouëthière site

On the La Gouëthière site, for the fast wetting test, MWD of the soil surface varied between 0.28 mm (June 8) and 0.61 mm (July 4), with a mean of 0.41 mm (Figure 1B). For the slow wetting test, MWD of the soil surface varied between 0.65 mm (May 5) and 1.43 mm (July 4), with a mean of 1.01 mm (Figure 1D). The variation coefficient of the surface

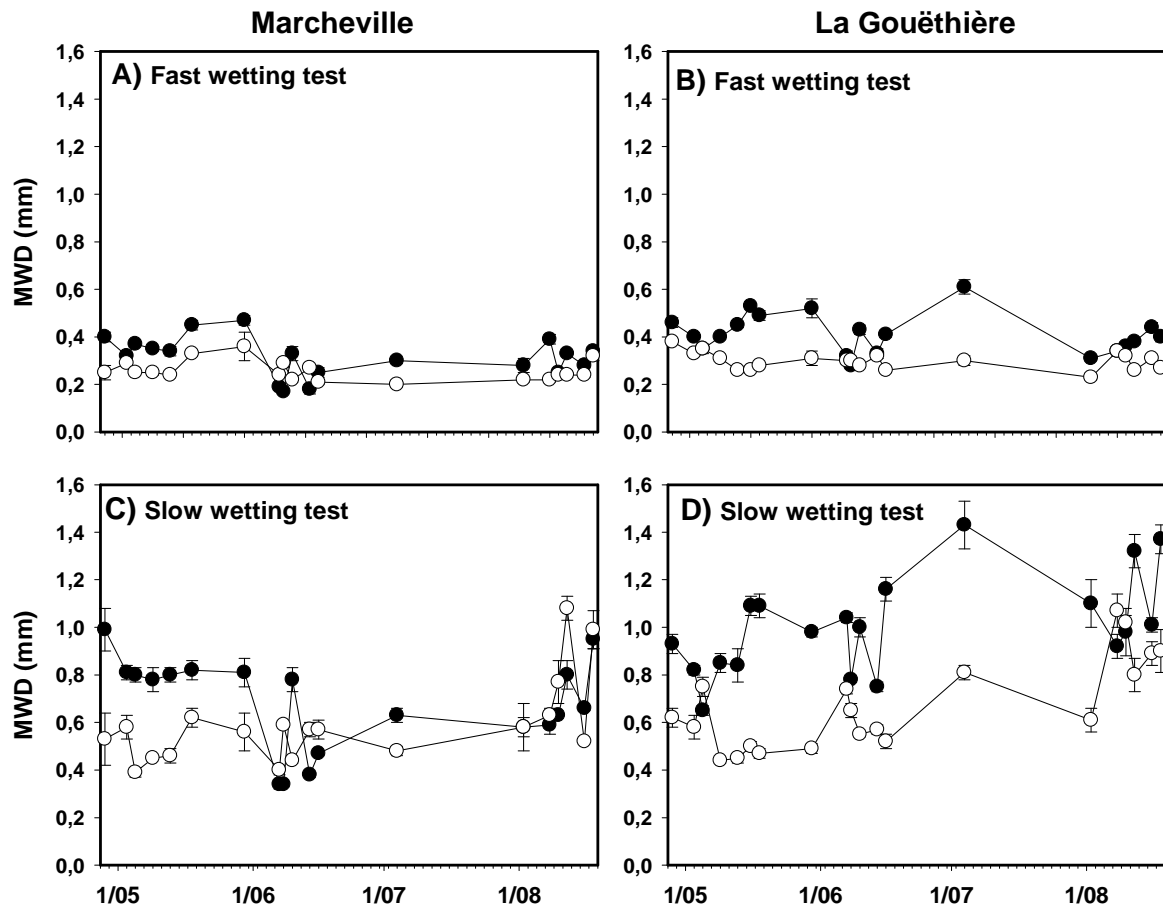


Figure 1: Temporal variation in aggregate stability measured by the MWD after fast wetting test (A and B) and slow wetting test (C and D) for the Marcheville (A and C) and La Gouëthière sites (B and D).

Each MWD value corresponds to the mean of three replicates; bars are standard errors.

MWD reached 20% for both the fast and slow wetting tests. On the subsurface, for the fast wetting test, MWD varied between 0.23 mm (August 2) and 0.38 mm (April 28) with a mean of 0.30 mm (Figure 1B). For the slow wetting test, subsurface MWD varied between 0.44 mm (May 9) and 1.07 mm (August 8), with a mean of 0.67 mm (Figure 1D). The variation coefficient of the surface MWD reached 12% for the fast wetting test and 28 % for the slow wetting test.

3.2. Explanatory variables

3.2.1. Variables linked to biological activity

Organic matter content:

On the Marcheville site, the organic matter content of the soil surface varied between 1.42% (August 12) and 1.79% (May 9), with a mean of 1.66% and a variation coefficient of 6% (Figure 2A). The organic matter content of the subsurface varied between 1.47% (August 18) and 1.82% (May 9), with a mean of 1.7% and a variation coefficient of 7% (Figure 2A). On the La Gouëthière site, the organic matter content of the soil surface varied between 1.3% (June 16) and 2.18% (June 8), with a mean of 1.66%, and a variation coefficient of 11% (Figure 2B). The organic matter content of the subsurface varied between 1.44% (May 13) and 1.92% (June 14), with a mean of 1.7% and a variation coefficient of 9% (Figure 2B).

Microbial biomass:

On the Marcheville site, the microbial biomass of the soil surface varied between 72.1 mg/kg (June 7) and 234.2 mg/kg (August 16), with a mean of 147.5 mg/kg and a variation coefficient of 26% (Figure 2C). The microbial biomass of the subsurface varied between 37.4 mg/kg (May 11) and 256.5 mg/kg (June 8), with a mean of 117.3 mg/kg and a variation coefficient of 46% (Figure 2C). On the La Gouëthière site, the microbial biomass of the soil surface varied between 75.4 mg/kg (April 28) and 304.13 mg/kg (June 4), with a mean of

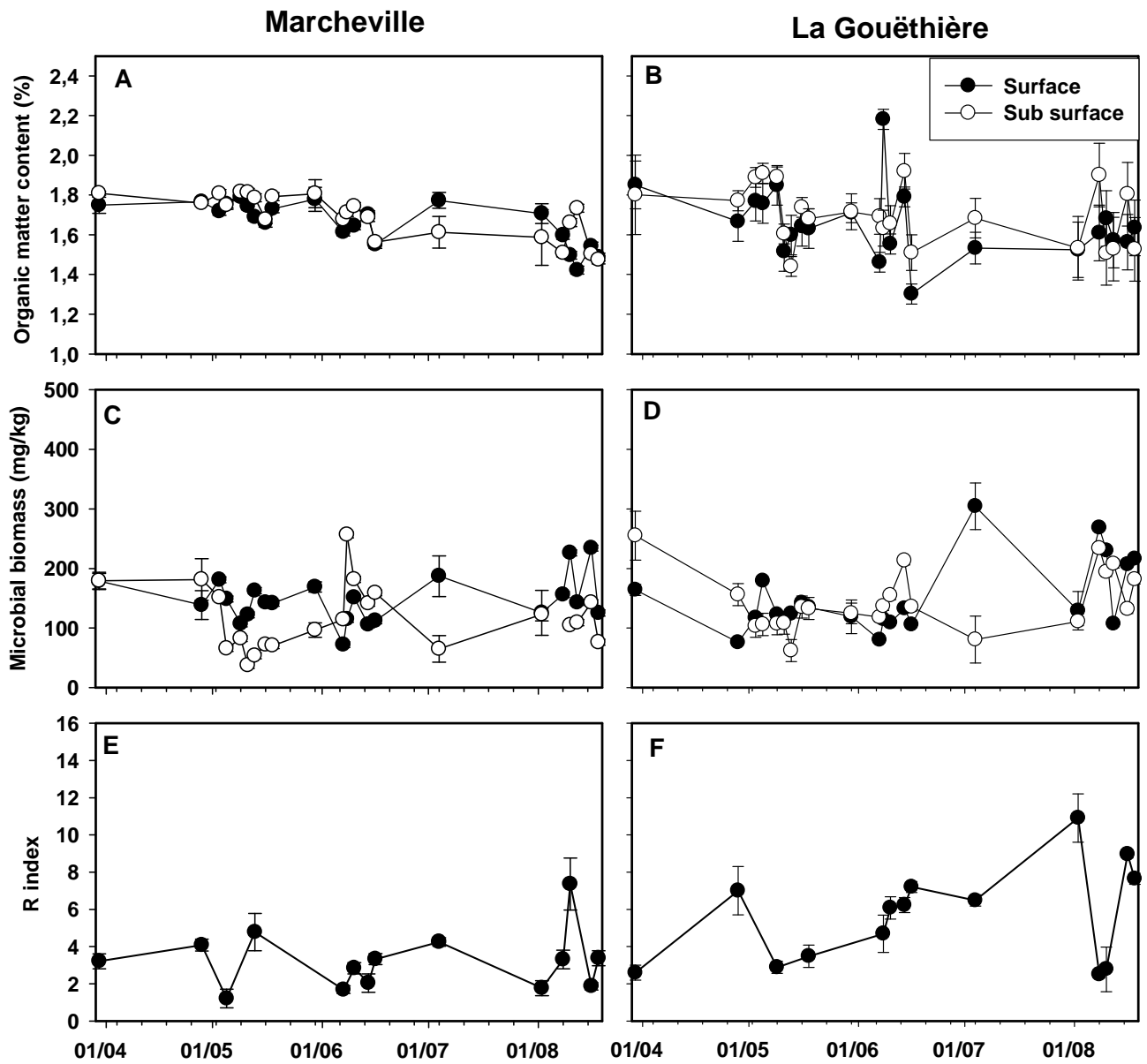


Figure 2: Temporal variation in organic matter content (A, B), microbial biomass (C, D) and water repellency (E, F) for the Marcheville (A, C, E) and La Gouëthière sites (B, D, F)

A, B, C, D: each point with error bar corresponds to the mean of three replicates; bars are standard errors.

E, F: each point corresponds to the mean of 10 replicates

149.7 mg/kg and a variation coefficient of 40% (Figure 2D). The microbial biomass of the subsurface varied between 62.2 mg/kg (May 13) and 255.2 mg/kg (March 30), with a mean of 140.4 mg/kg and a variation coefficient of 43% (Figure 2D).

Water repellency:

For the Marcheville site, the R index of the soil surface varied between 1.21 (May 5) and 7.36 (August 10) with a mean of 3.33 and a standard deviation of 1.66. For the La Gouëthière site, the R index varied between 2.5 (August 8) and 10.9 (August 2) with a mean of 5.7 and a standard deviation of 2.6. These results indicate that the studied soils exhibited subcritical hydrophobicity (Tillmann *et al.*, 1999).

3.2.2. Variables linked to climate

Atmospheric variables:

During monitoring on the Marcheville site, the hourly air temperature varied between 1.6°C and 36°C with a mean of 16.1°C and a standard deviation of 5.3°C (Figure 3A). The cumulative rain height reached 219 mm, and the mean air water content was 76.8% with a standard deviation of 20.5% (figure 3C). For the La Gouëthière site, the hourly air temperature varied between 1.6°C and 41.0°C with a mean of 15.7°C and a standard deviation of 5.1°C (Figure 3B). The cumulative rain height reached 181 mm, and the mean air water content was 70.7% with a standard deviation of 19.5% (figure 3D).

Soil variables:

For the Marcheville site, the temperature of the soil surface (-1 cm) varied between 4.8°C and 40.2°C with a mean of 19.1°C and a standard deviation of 6.2°C (Figure 4A). The temperature of the soil subsurface (-5 cm) varied between 9.7°C and 30.4°C, with a mean of 18.9°C and a standard deviation of 3.5°C (Figure 4C). The soil water content of the soil surface varied between 22.5% (July 21) and 6.1% (June 4) with a mean of 11.7% and a

variation coefficient of 28% (Figure 4E). For the subsurface, the water content was very stable, with a mean of 19.6% and a variation coefficient of 4.1% (Figure 4E). For the La Gouëthière site, the soil surface temperature varied between 4.3°C and 38.3°C with a mean of 18.9°C and a standard deviation of 3.2°C (Figure 4B). The soil subsurface temperature varied between 9.7°C and 29.5°C with a mean of 18.9°C and a standard deviation of 3.2°C (Figure 4D). The soil water content of the soil surface varied between 27.5% (July 21) and 1.4% (June 4) with a mean of 8.2% and a variation coefficient of 58% (Figure 4E). The mean subsurface water content was 21% with a variation coefficient of 4.4% (Figure 4E).

3.3. Relationships between aggregate stability and explanatory variables

3.3.1. Relationships between aggregate stability and biological variables

In the Marcheville site, for both surface and subsurface datasets, MWD was not significantly correlated with microbial biomass, organic matter or water repellency at the 5% level, regardless of the aggregate stability test used (table 2). In the La Gouëthière site, for the subsurface dataset, MWD was positively and significantly correlated with microbial biomass for both stability tests, and with organic matter content for the fast wetting test. For the surface dataset, none of the variables linked to biological activity were significantly correlated with MWD at the 5% significance level (table 2).

3.3.2. Relationships between aggregate stability and climatic variables

For both sites, regardless of the aggregate stability test used, surface and subsurface MWD did not correlate significantly with air temperature, soil temperature or air humidity (table 3). Significant correlations were found between aggregate stability and soil water content at the time of sampling (WC_0) (table 3). For the Marcheville site, for the soil surface, MWD was significantly and negatively correlated with soil water content at the time of sampling for every stability test. Subsurface MWD of was significantly and negatively correlated with soil

water content for the slow wetting test ($r=-0.57$) but was not significantly correlated with soil water content for the fast wetting test (table 3). In the La Gouëthière site, for the fast wetting test, surface MWD correlated significantly with soil water content ($r=-0.77$). However, for the slow wetting test, MWD was not significantly correlated with soil water content at the 5% level.

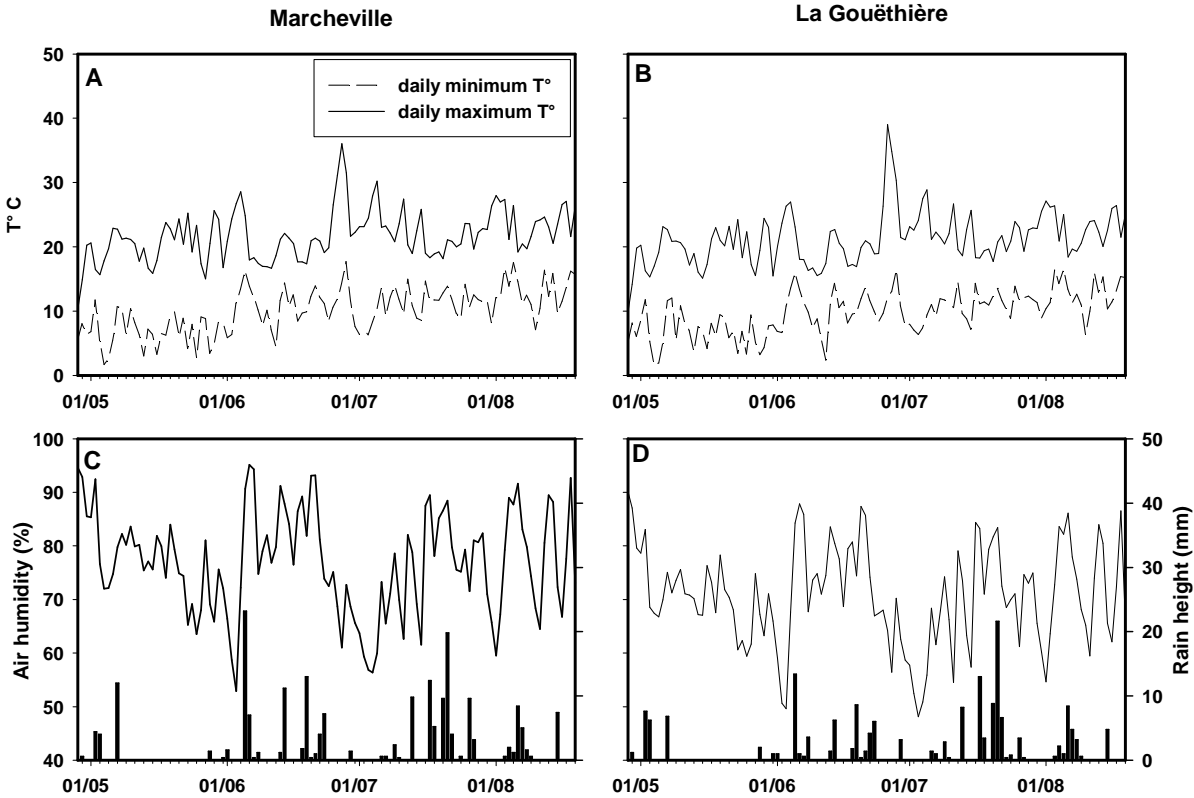


Figure 3: Temporal variation in daily air temperature (A, B), humidity and rain (C, D) for the Marcheville (A, C) and La Gouëthière sites (B, C).

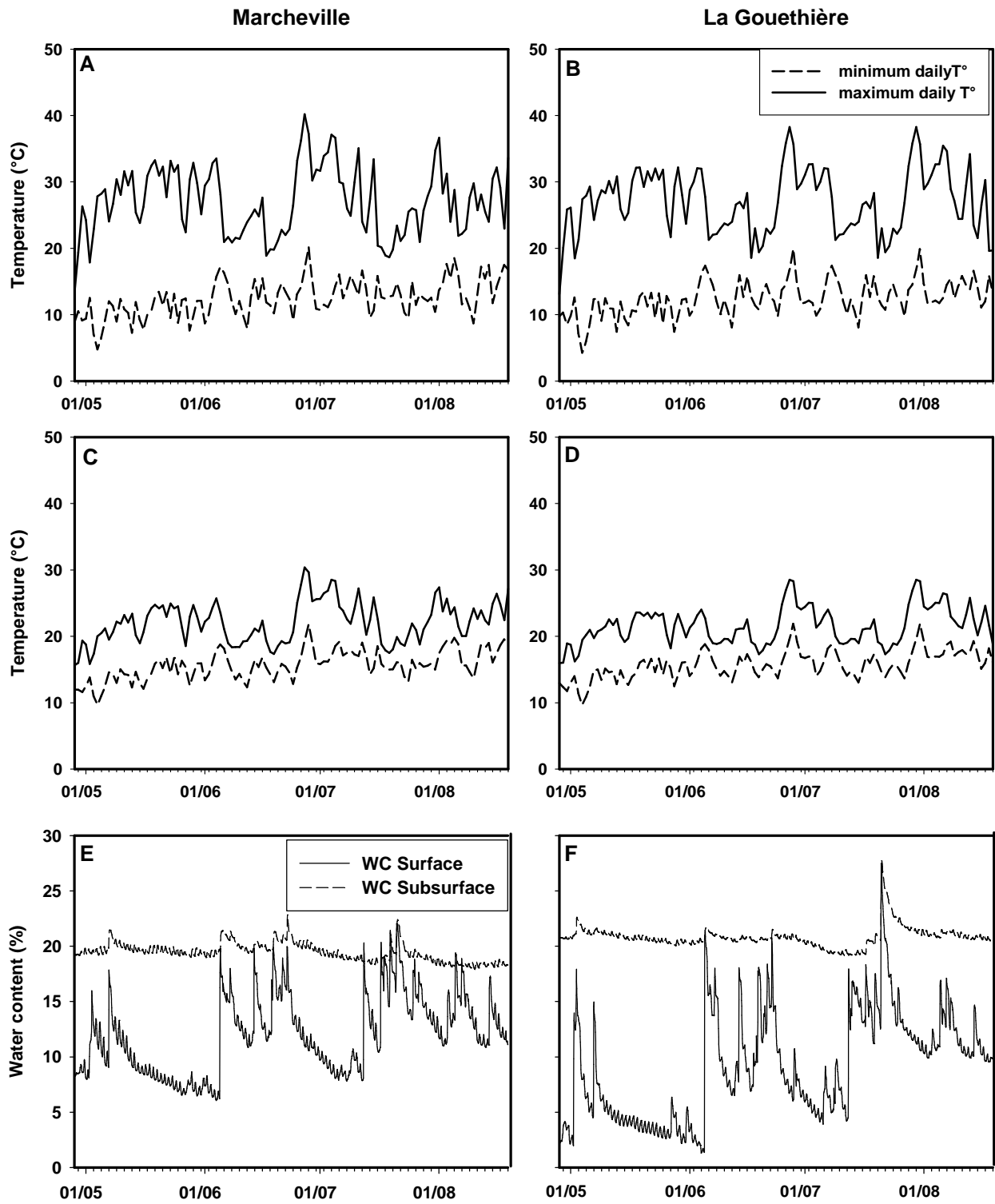


Figure 4: Temporal variation of soil surface temperature (A, B), soil subsurface temperature (C, D), soil water content and daily rain height (E, F) for the Marcheville site (A, C, E) and the La Gouethière site (B, D, F).

Curve values correspond to the mean of two replicates

Table 2: Correlations (Pearson's coefficient) between aggregate stability and variables linked to biological activity: organic matter content, microbial biomass and water repellency.

	Surface			Subsurface	
	Organic matter	Microbial biomass	Water repellency	Organic matter	Microbial biomass
Marcheville soil					
MWD fast wetting	0.33 (NS)	0.25 (NS)	0.12 (NS)	0.34 (NS)	-0.07 (NS)
MWD slow wetting	0.14 (NS)	0.32 (NS)	0.24 (NS)	-0.29 (NS)	-0.06 (NS)
La Gouëthière soil					
MWD fast wetting	-0.04 (NS)	0.20 (NS)	-0.27 (NS)	0.55*	0.47*
MWD slow wetting	-0.23 (NS)	0.30 (NS)	0.10 (NS)	-0.02 (NS)	0.51*

For the Marcheville dataset: $N=19$; $DF=17$; $\alpha=5\%$; $r=0.456$

For the La Gouëthière dataset: $N=20$; $DF=18$; $\alpha=5\%$; $r=0.444$

* Significant at the 5% level

NS=Not significant at the 5% level

For the subsurface, no significant correlations were found between MWD and variables linked to hydric history (API , WC_t and ΔWC_t), regardless of the aggregate stability test used. For the surface, MWD correlated significantly and negatively with all variables linked to hydric history except WC_t , which was not significantly correlated with MWD for the slow wetting test on the La Gouëthière site (table 4). The highest correlation coefficients between MWD and WC_t were observed for the mean water content calculated for a duration of approximately 0.5 day prior to sampling ($WC_{1/2}$) (figure 5). For the Marcheville site, correlation coefficients of -0.76 and -0.71 were observed for the fast and slow wetting tests, respectively (table 4). For the La Gouëthière site, the correlation coefficient reached -0.71 with respect to MWD for the fast wetting test (table 4). On both sites and for both stability tests, MWD for the surface was significantly and negatively correlated with ΔWC_t ; the highest correlation coefficient between MWD and ΔWC_t was found for the difference in soil water content approximately four days before sampling and at the time of sampling (ΔWC_4) (figure 5). For the Marcheville site, correlation coefficients of -0.54 and -0.70 were observed

for the fast and slow wetting tests, respectively, while for the La Gouëthière site, correlation coefficients for the fast and slow wetting tests were -0.51 and -0.50 (table 4).

Table 3: Correlations (Pearson's coefficient) between aggregate stability and variables linked to climate: soil water content (Soil WC), air water content (Air WC) and soil and air temperature.

	Surface				Subsurface			
	Air T°	Air WC	Soil T°	Soil WC ₀	Air T°	Air WC	Soil T°	Soil WC ₀
Marcheville								
MWD fast wetting	-0.01(NS)	-0.1(NS)	0.31(NS)	-0.73*	-0.04(NS)	0.11(NS)	0.10(NS)	0.14(NS)
MWD slow wetting	-0.12(NS)	-0.15(NS)	0.25(NS)	-0.69*	0.25(NS)	0.37(NS)	-0.11(NS)	-0.57*
La Gouëthière								
MWD fast wetting	0.20(NS)	-0.30(NS)	0.35(NS)	-0.77*	-0.30(NS)	0.10(NS)	-0.21(NS)	0.31(NS)
MWD slow wetting	0.38(NS)	0.08(NS)	0.40(NS)	-0.15(NS)	0.19(NS)	0.08(NS)	0.31(NS)	0.03(NS)

For the Marcheville dataset: N=19; DF=17; $\alpha=5\%$; $r=0.456$

For the La Gouëthière dataset: N=20; DF=18; $\alpha=5\%$; $r=0.444$

** Significant at the 5% level*

NS=Not significant at the 5% level

Table 4: Correlations (Pearson's coefficient) between MWD hydric history indices: WC_{1/2} and ΔWC_4

	Surface			Subsurface		
	API	WC ₁₂	ΔWC_{96}	API	WC ₁₂	ΔWC_{96}
Marcheville						
MWD fast wetting	-0.63*	-0.76*	-0.54*	-0.18(NS)	0.13(NS)	0.25(NS)
MWD slow wetting	-0.65*	-0.72*	-0.7*	-0.25(NS)	-0.37(NS)	-0.04(NS)
La Gouëthière						
MWD fast wetting	-0.65*	-0.77*	-0.51*	0.39(NS)	0.31(NS)	0.36(NS)
MWD slow wetting	-0.51*	-0.25(NS)	-0.5*	0.35(NS)	0.03(NS)	0.04(NS)

For the Marcheville dataset: N=19; DF=17; $\alpha=5\%$; $r=0.456$

For the La Gouëthière dataset: N=20; DF=18; $\alpha=5\%$; $r=0.444$

** significant at the 5% level*

NS=Not significant at the 5% level

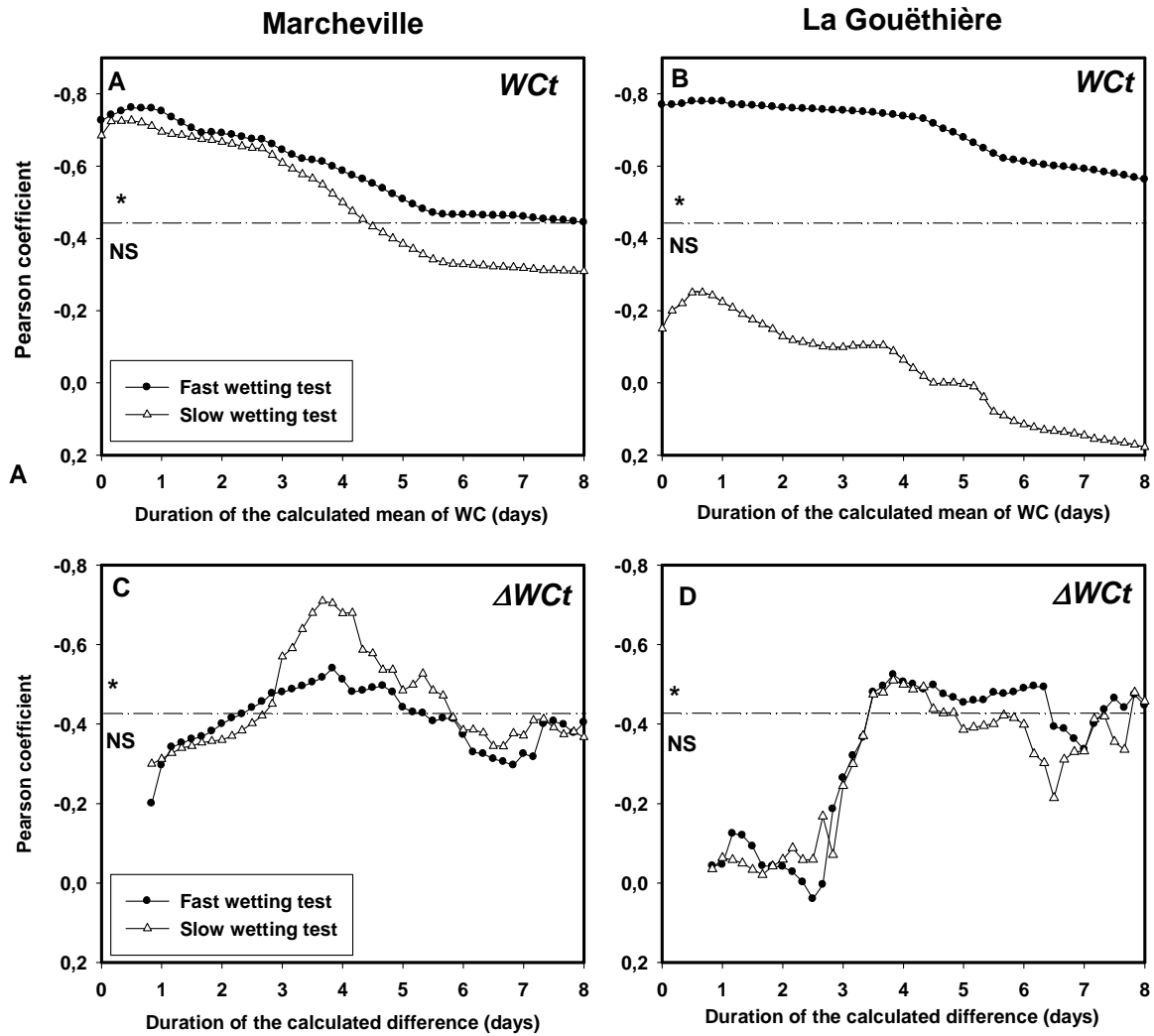


Figure 5: Correlations (Pearson's coefficient) between aggregate stability and hydric history indices: WCt (A, B) and ΔWCt (C, D), calculated for periods of varying duration prior to sampling (x axis), for the Marcheville site (A, C) and for the La Gouëthière site (B, D).

* Pearson coefficient is significant at the 5% level

NS Pearson coefficient is not significant at the 5% level

$N=20$; $DF=18$; $\alpha=5\%$; $r=0.444$

3.4. Prediction of aggregate stability variations

Regression analyses were performed using the variables found to be significantly correlated with MWD: WC_0 , $WC_{1/2}$, API and ΔWC_4 for the surface dataset and WC_0 , microbial biomass (BIOMI) and organic matter content (OM) for the subsurface dataset.

Initially, simple regression analysis was conducted separately for each site (table 5). For the subsurface dataset, none of the simple regression models tested were significant at the 5% level, regardless of site and the aggregate stability test considered. For the surface dataset from the Marcheville site for the fast wetting test, the best simple regression model included $WC_{1/2}$ and accounted for 54% of MWD variation. Models that included WC_0 and API were also significant at the 5% level. For the slow wetting test, models that included WC_0 , $WC_{1/2}$, API and ΔWC_4 were significant at the 5% level (table 5). For the La Gouëthière site, the best simple regression model included WC_0 and predicted 57% of the MWD variation for the fast wetting test. Models including $WC_{1/2}$ and API were also significant at the 5% level. For the slow wetting test, none of the simple regression models evaluated were significant at the 5% level. Simple regression models that included OM and BIOMI did not significantly explain aggregate stability variation for any combination of site and aggregate stability test.

For each site and for the surface and subsurface data sets, the variables found to be significant during simple regression analysis were combined in multiple regression models. Because WC_0 , $WC_{1/2}$, and API were significantly correlated, and thus not independent, these variables were not combined in multiple regression models. Among all tested combinations and for both sites, the only valid multiple regression models were found for the surface dataset for the Marcheville site. For the slow wetting test, $WC_{1/2}$ and ΔWC_4 together accounted for 59% of the MWD variation, while the combination of WC_0 and ΔWC_4 accounted for 57% of the MWD variation.

Table 5: Simple regression models for MWD variation

Datasets		Df	WC ₀		WC _{1/2}		API		ΔWC ₄		OM		BIOMI		
			R ²	level	R ²	level	R ²	level	R ²	level	R ²	level	R ²	level	
Surface	Marcheville	FW	17	0.51	**	0.54	**	0.37	*	0.25	.	0.01	NS	0.06	NS
		SW	17	0.44	*	0.50	**	0.39	*	0.47	**	0.05	NS	0	NS
	La Gouëthière	FW	18	0.57	**	0.55	**	0.39	*	0.22	.	0	NS	0.03	NS
		SW	18	0	NS	0.07	NS	0.22	.	0.21	.	0.04	NS	0.09	NS
Subsurface	Marcheville	FW	17	0	NS	0	NS	0	NS	0.01	NS	0	NS	0.06	NS
		SW	17	0.23	.	0.29	.	0.07	NS	0	NS	0	NS	0.03	NS
	La Gouëthière	FW	18	0.05	NS	0.05	NS	0.11	NS	0.16	.	0	NS	0.26	.
		SW	18	0	NS	0.09	NS	0.07	NS	0	NS	0.23	.	0	NS

Df= degrees of freedom; R²= adjusted r²

FW: Fast wetting test. SW: Slow wetting test.

** Model significant at the 1 % level

* Model significant at the 5% level

. Model significant at the 10 % level

NS Model not significant at the 10 % level

4. Discussion

Field monitoring permitted identification of explanatory variables of short time step aggregate stability variation. The relevance of each measured variable as an explanatory factor in aggregate stability variation is discussed, as are the relevance of the predictive models and implications for erodibility prediction.

4.1. Factors linked to biological activity

It is widely recognized in the scientific literature that biological activity is positively correlated with aggregate stability (e.g., Tisdal & Oades 1982; Chenu et al. 2000; Six et al. 2004). Organic matter addition, by stimulating microbial activity, is recognized to have a positive effect on aggregate stability (Tisdal & Oades 1982; Cosentino et al. 2006). In an incubation experiment, Cosentino et al. (2006) found that organic matter addition stimulated microbial activity and stabilized aggregates by increasing both their cohesion and water repellency. Other studies have shown that the exudation of extracellular polysaccharides by bacteria and fungi bond mineral particles together, increasing inter-particle cohesion within

the aggregate (Tisdall & Oades, 1982, Chenu & Guérif 1991). In addition, organic hydrophobic aggregate coatings decrease the rate of wetting and limit the destructive impact of slaking on aggregate stability (Piccolo & Mbagwu, 1999, Goebel et al. 2012).

Such studies of the influence of biological activity on aggregate stability have generally been based on either the external stimulation of biological activity by organic amendments, where amended soils were compared to non-amended soils (e.g., Cosentino et al., 2006; Abiven et al., 2007; Leguillou et al., 2012) or the comparison of soils with greatly contrasting organic matter content or management practices (e.g., Blackman 1992; Suwardji & Eberbach, 1998). In the present study, soils were kept bare for all monitoring, and no amendments were incorporated. Biological activity was measured by assessing organic matter content, microbial biomass and subcritical water repellency on each sampling date. For the surface dataset, no significant relationships between these variables and aggregate stability were found. In light of these results, it appears that the variables linked to biological activity were not dominant explanatory factors in aggregate stability variation on the soil surface. Similar results have also been observed for monthly and seasonal variations of aggregate stability (Chan et al., 1994; Dimoyiannis, 2009), and the present study confirms this conclusion for a short time step. For the subsurface dataset, significant positive correlations were identified between microbial biomass and aggregate stability for the fast wetting test and between organic matter content and aggregate stability for the slow wetting test. However, simple regression models that included microbial biomass and organic matter content could not explain a significant proportion of aggregate stability variation. These findings lead us to conclude that without stimulation of biological activity through amendments, biological factors do not adequately explain aggregate stability variation.

4.2. Factors linked to climate

Soil and air temperature

Several researchers have found a positive influence of air and soil temperatures on aggregate stability (Bullock *et al.* 1988; Blackman 1992; Dimoyiannis 2009). Soil and air temperatures affect aggregate stability directly through the freezing process (Bullock *et al.* 1988), and indirectly through seasonal stimulation of microbial activity (Suwardji & Eberbach, 1998). In the present study, as recorded temperatures were positive throughout the duration of monitoring, no frost occurred. Air and soil temperatures did not correlate significantly with aggregate stability for any stability test or soil depth. For the pedo-climatic conditions evaluated in this study, as would be expected, air and soil temperatures were not explanatory factors of aggregate stability variation for a short time step.

Air humidity

Air humidity affects aggregate stability indirectly through its influence on soil water content dynamics: low air humidity increases soil drying which promotes aggregate stability, such as through inter-particle cohesion within the aggregate (Kemper *et al.*, 2007). Combeau (1965) observed a negative relationship between aggregate stability variation at the monthly time step and air humidity. In the present study, air humidity was not a dominant factor in aggregate stability variation at a short time step.

Precipitation and soil water content

Temporal patterns in precipitation are considered an important factor in aggregate stability variation (Shainberg *et al.* 2003; Dimoyiannis 2009). Precipitation has a negative effect on aggregate stability through raindrop impact, which affects the structure of surface aggregates (Shainberg *et al.* 2003; Lehrs & Kincaid, 2006), and by increasing the soil water content. Soil water content is recognized as a key factor in aggregate stability variation (Utomo & Dexter, 1982; Perfect *et al.* 1990; Caron *et al.* 1992; Shainberg *et al.* 2003). Perfect *et al.*

(1990) found that aggregate stability measured on moist field samples decreased with increasing water content at the time of sampling. The same relationship was observed for air dried samples (Caron *et al.* 1992; Chan *et al.*, 1994). Secondly, aggregate stability was found to be affected by hydric history (Caron *et al.*, 1992). Soil wetting is recognized to decrease aggregate stability through several processes such as slaking (Zaher *et al.* 2005), the differential swelling of clays and clay dispersion (Le Bissonnais, 1996). However, drying is recognized as a process of aggregate stability increase (Kemper *et al.* 1987; Dexter *et al.* 1988). Two suggested processes by which drying may increase aggregate stability are the rearrangement of the mineral particles within the aggregates induced by retreating water menisci (Kemper and Rosenau, 1984; Dexter *et al.*, 1988) and the deposition of colloids and precipitation of slightly soluble minerals around the contact points between particles (Kemper *et al.* 1987). The balance between the counteracting processes induced by wetting and drying determines the level of aggregate stability at a given moment (Denef *et al.* 2001).

The antecedent precipitation index (API) characterizes the cumulative amount of water added to the soil by precipitation during the preceding six days. In a previous study conducted on the same soils, Algayer *et al.* (to be submitted) showed that API was negatively correlated with aggregate stability variation on a short time step.

In the present study, soil water content was measured both indirectly with API and directly with hourly measurements taken throughout the monitoring period. The high temporal resolution of this monitoring permitted the assessment of both water content at the time of sampling (WC_0) and the hydric history of the samples. Two indices of hydric history were calculated: WC_t and ΔWC_t . The mean water content (WC_t) provided information on the hydric status of the soil prior to sampling, while the difference in water content (ΔWC_t) described the trend in water content over a given period. A negative ΔWC_t indicates that soil was drying, while a positive ΔWC_t indicates that soil water content was increasing.

The results of the present study reveal a high temporal variability in surface water content and a low temporal variability of the subsurface water content for both sites. For the surface dataset, API and WC_0 were significantly and negatively correlated with aggregate stability: the higher the water content at the time of sampling, the lower the aggregate stability. Simple regression models that included the API explained 37% of aggregate stability variation for the Marcheville site and 39% of the aggregate stability variation for the La Gouëthière site (fast wetting test). Simple regression models with WC_0 explained 51% of aggregate stability variation for the Marcheville site and 57% of aggregate stability variation for the La Gouëthière site (fast wetting test). Previous studies have found negative correlations between water content and aggregate stability variation at the monthly time step. Blackman (1992) and Dymoyiannis (2009) found negative correlations between aggregate stability and total monthly rainfall. Other studies have found a negative correlation between soil water content at the time of sampling and aggregate stability (e.g., Perfect *et al.*, 1990; Caron *et al.*, 1992). The present study found similar relationships for a short time step (a few days) for surface aggregate stability only. API and WC_0 were dominant factors of surface aggregate stability variation at a short time step. For the subsurface dataset, API and WC_0 were generally not significantly correlated with aggregate stability. This result can be explained by almost constant water content of the subsurface soil in contrast with highly variable aggregate stability. Moreover, the surface aggregates directly exposed to raindrop impact protected the underlying material from this negative influence on soil structure.

WC_t and ΔWC_t were calculated for monitoring periods of varying duration, and correlation analyses between aggregate stability and each of the calculated indices of water content were conducted. For WC_t , the maximum correlation coefficient corresponded to a duration of approximately $\frac{1}{2}$ day prior to sampling ($WC_{1/2}$). For ΔWC_t , the maximum coefficient corresponded to a duration of approximately 4 days (ΔWC_4). This pattern was

observed in both the Marcheville and La Gouëthière study sites and for both the fast and slow wetting tests. Very similar soil properties and geographical proximity, and climatic conditions that remained very similar throughout the monitoring period explain the similarity in the results between study sites. Other soil types and climatic conditions would likely lead to different WC_t and ΔWC_t values.

$WC_{1/2}$ was negatively correlated with aggregate stability for the soil surface. For the Marcheville site, simple regression models that included $WC_{1/2}$ explained 51% and 54% of the aggregate stability variations for the fast and slow wetting tests, respectively. The same model explained 55% of the aggregate stability variation for the La Gouëthière site (fast wetting test). $WC_{1/2}$ was therefore a dominant explanatory factor in aggregate stability variation at a short time step. For the surface dataset, ΔWC_4 was negatively correlated with aggregate stability for both sites and for both aggregate stability tests. This indicates that aggregate stability increased when soil was in the drying phase and decreased when soil was in the wetting phase. This result is consistent with the results of previous laboratory studies (Kemper and Rosenau, 1984; Kemper *et al.*, 1987; Dexter *et al.*, 1988). The present study determined that this relationship also holds in a field setting and that ΔWC_t was a dominant explanatory factor in soil surface aggregate stability variation at a short time step. For the soil subsurface, none of the evaluated correlations were significant.

In summary, API, WC_0 , $WC_{1/2}$ and ΔWC_4 were the dominant explanatory variables for soil surface aggregate stability variation at a short time step. Each of these indices is linked to soil water content variation and involves physico-chemical processes of aggregate stability variation. The lack of significant relationships between aggregate stability and variables associated with biological activity (organic matter content, microbial biomass and water repellency) may illustrate the dominance of abiotic processes in short term aggregate stability

variation when biological activity is not stimulated with amendments. Subsurface aggregate stability variation could not be explained by the measured variables.

4.3. Prediction of aggregate stability and consequences for erosion predictions

Erodibility is a dynamic soil property and is considered a key parameter in erosion modeling. Because temporal variations in soil erodibility remain difficult to predict, most erosion models consider soil erodibility for a given soil to be constant in space and time, leading to bias in erosion predictions (Jetten *et al.*, 2003; Boardman, 2006; Algayer *et al.*, under review). Thus, predicting erodibility variations is a current challenge in improving erosion prediction. Soil interrill erodibility can be assessed using aggregate stability measurements (Le Bissonnais, 1996; Barthes & Roose, 2002).

The present study attempted to predict aggregate stability variations at a short time step using the identified explanatory factors WC_0 , $WC_{1/2}$, API and ΔWC_4 to improve erodibility prediction. These factors showed different precisions for aggregate stability predictions and their measurement in the field necessitated various practical needs. The calculation of API only requires rain data, which can be easily measured or calculated by models. However, the best model using API for aggregate stability variations only explained 39% of variation. The measurement of WC_0 required field sample collection. Models that included WC_0 predicted up to 57% of aggregate stability variations, while the best model containing $WC_{1/2}$ explained 59% of aggregate stability variation. However, the calculation of $WC_{1/2}$ required high temporal resolution monitoring of the soil water content that necessitated heavy instrumentation of the soil with expensive equipment. Even if measuring this variable was more convenient, the use of API for aggregate stability prediction was not efficient due to inaccuracy in the predictions. While models that included $WC_{1/2}$ were the most predictive, the use of WC_0 in place of $WC_{1/2}$ would permit more convenient measurements, with a loss of

only 2 or 3% of aggregate stability prediction. As a result, among all factors measured in the present study, the use of WC_0 is recommended for aggregate stability prediction.

Based on the results of the present study, soil hydric history was found to be the dominant factor controlling soil surface aggregate stability variations at a short time step. The best model predicted 59% of the variation in aggregate stability, a proxy for soil erodibility. Hydric history affects aggregate stability through several processes, and further research into these processes is needed to improve soil erodibility predictions.

5. Conclusion

In the studied conditions, short time step variations of aggregate stability were primarily controlled by water content dynamics. Water content at the time of sampling, hydric history indices ($WC_{1/2}$ and ΔWC_4) and the antecedent precipitation index were the dominant factors influencing surface aggregate stability variations. Variables associated with biological activity did not adequately explain aggregate stability variations. These results underscore the dominant effect of abiotic factors such as water content dynamics on aggregate stability variations at a short time step in the field in the absence of biological activity stimulation. A regression model that included hydric history indices predicted up to 59% of surface aggregate stability. Because aggregate stability is a proxy for soil interrill erodibility, better prediction of this variable could improve the parameterization of erodibility in soil erosion models. Further research on the processes involved in aggregate stability variations associated with hydric history would also be useful in improving predictions of erodibility.

Acknowledgements

The authors thank the Centre region (France) for the funding that permitted this study. This work was also supported by the SPATIOFLUX project (CPER 2007-2013) founded by the Centre region, the FEDER and the INRA. We also thank Mr. Carnis (Marcheville) and

Mr. Besnard (La Gouëthière), owners of the fields in which monitoring was conducted. Finally, we thank Guillaume Giot, David Colosse, Hervé Gaillard, Alexandre Roy, Olivier Josière, Bernard Nicoullaud, Christophe Lelay, Pierre Courtemanche and Lionel Cottenot for their skilled technical contributions.

Compléments à la partie 2 :

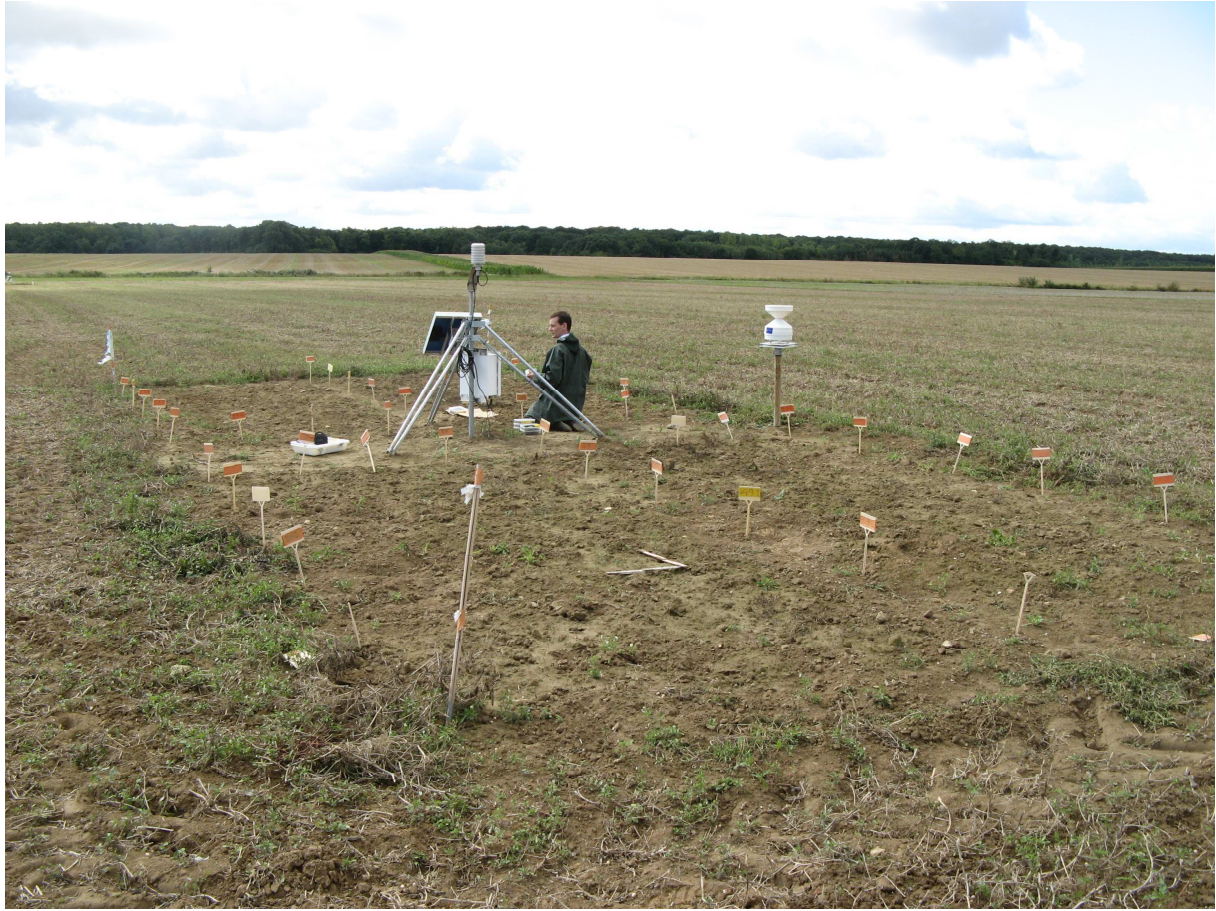


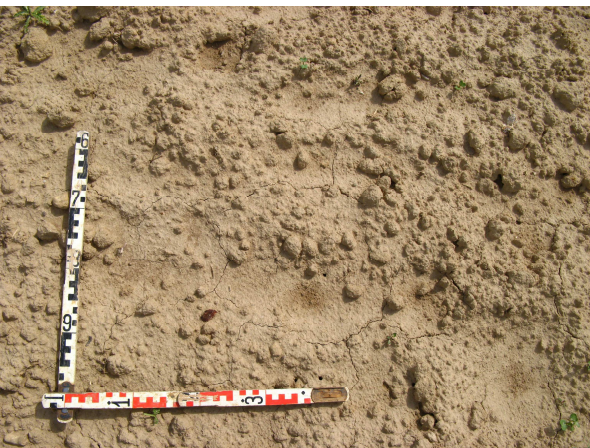
Figure 1 : Vue d'une placette expérimentale du site de La Gouëthière



28/04/2011 : lit de semence



9/05/2011 : croûte structurale en formation



16/06/2011 croûte structurale formée



18/08/2011 : croûte structurale formée

Figure 2 : Evolution de l'état de surface du sol sur le site La Gouêthière A.



Figure 3 : Evolution de l'état de surface du sol sur le site de Marcheville A.

Synthèse et conclusion

Les objectifs de cette étude de terrain étaient de mesurer les variations de la stabilité structurale à pas de temps court sur le terrain et d'identifier les facteurs contrôlant ces variations à travers des variables liés au climat et à l'activité biologique.

1) La stabilité structurale varie fortement à pas de temps court

Les résultats présentés dans le chapitre 3 montrent clairement que la stabilité structurale varie fortement à pas de temps court pour la surface du sol comme pour la sub-surface. Les variations observées à pas de temps court sont de la même amplitude que celles observées à pas de temps mensuel (variations de 20% à 30% selon les modalités). Ces variations de stabilité structurale correspondent à des érodibilités très contrastées.

Les variations temporelles de la stabilité structurale étaient différemment influencées par les modalités spatiales de l'étude. Pour les deux sites localisés à une distance de quelques kilomètres et présentant des types de sol similaires, la stabilité structurale a suivi les mêmes tendances de variations mais à varier dans des gammes contrastées. Pour deux placettes localisées à quelques dizaines de mètres d'écart au sein de la même parcelle, la stabilité structurale a présenté les mêmes tendances et la même gamme de variation. Sur une placette donnée, la surface et la sub-surface ont présenté des stabilités structurales très contrastées : ces différences n'étaient pas toujours dans le même sens, les deux matériaux suivant des dynamiques différentes. Ce dernier résultat souligne, comme pour l'étude présentée dans le chapitre 1, que la stabilité structurale utilisée comme proxy de l'érodibilité doit être mesurée sur le matériau de surface car c'est celui qui est directement soumis aux processus érosifs.

Actuellement, les modèles d'érosion ne prennent pas en compte cette variation temporelle de l'érodibilité. Dans les meilleurs cas, la paramétrisation des modèles intègre une

moyenne saisonnière de l'érodibilité (par exemple, RUSLE, WEPP). Si ces importantes variations temporelles de l'érodibilité du sol doivent être considérées par les modèles d'érosion, compte-tenu de nos connaissances actuelles, nous suggérons d'utiliser une gamme de variabilité de l'érodibilité pour un sol donné. Cela intégrerait à la fois les variabilités temporelle et spatiale et permettrait d'obtenir des prédictions de meilleure qualité.

2) Les variations de la stabilité structurale de la sub-surface n'ont pas pu être expliquées

Si la sub-surface du sol présentait de fortes variations de stabilité structurale, aucune des variables étudiées n'a permis d'expliquer ces variations. Les facteurs de variation de la stabilité structurale de ce matériau restent obscurs, et nécessitent donc des études spécifiques.

3) Pour la surface, les facteurs contrôlant les variations de la stabilité structurale sont liés à la teneur en eau et à l'histoire hydrique du sol.

Les résultats présentés dans le chapitre 4 montrent que, pour les conditions étudiées (sol nu, pas de stimulation de l'activité microbienne par des apports), les variations de la stabilité structurale de la surface du sol à pas de temps court étaient essentiellement influencées par la dynamique de la teneur en eau du sol. La teneur en eau du sol au prélèvement, l'histoire hydrique et l'histoire des précipitations sont apparus comme les facteurs dominants les variations de stabilité structurale en surface. Un modèle de régression incluant des indices de l'histoire hydrique du sol a permis de prédire jusqu'à 60% des variations de la stabilité structurale. En revanche, les variables liées à l'activité biologique n'ont pas permis d'expliquer les variations de stabilité, ni d'améliorer les prédictions des modèles testés. Ce résultat souligne le caractère dominant des facteurs abiotiques dans les variations de stabilité structurale à pas de temps court lorsque l'activité biologique n'est pas stimulée. Ces facteurs sont impliqués dans différents processus physico-chimiques liés aux

cycles d'humidité. Ces processus, présentés dans le chapitre 2, nécessitent d'être mieux connus afin de mieux prédire la stabilité structurale. La troisième partie de ce manuscrit présente les résultats de deux expérimentations de laboratoire dans l'objectif d'améliorer les connaissances des processus physico-chimiques de variation de la stabilité structurale.

Troisième partie

**Caractérisation du réarrangement particulaire
comme processus physico-chimique de variation
de la stabilité structurale lié aux cycles
d'humidité.
Etudes expérimentales**

Introduction

La stabilité structurale est un proxy de l'érodibilité du sol. Cette propriété intrinsèque reste difficile à prédire. Dans la partie précédente, nous avons vu que la stabilité structurale du sol variait à pas de temps court en lien avec des facteurs liés à la dynamique de la teneur en eau du sol.

En plein champ, le sol est soumis constamment à des cycles d'humidité sous l'action des facteurs climatiques. Les cycles d'humidité sont reconnus pour exercer une forte influence sur la stabilité structurale par le biais de processus physico-chimiques et biologiques (e.g. Utomo & Dexter, 1982 ; Deneff et al., 2001 ; Brownick & Lal, 2005 ; Cosentino et al., 2006). Si les relations entre cycles d'humidité, activité biologique et stabilité structurale ont bénéficié de l'intérêt d'études récentes (e.g. Deneff et al., 2001 ; Cosentino et al., 2006), certains processus physico-chimiques influençant la stabilité structurale lors des cycles d'humidité reste encore méconnus. Les processus abiotiques de variation de la stabilité structurale ont été inventoriés à travers la synthèse bibliographique présentée dans le chapitre 2. Certains ont été largement étudiés, observés par des expérimentations reproductibles et leur conditions d'occurrence sont connus. C'est le cas par exemple de l'éclatement ou encore de la dispersion/floculation des argiles. Cependant, d'autres processus, bien que reposant sur des bases théoriques, n'ont jamais été concrètement observés et directement reliés aux variations de la stabilité structurale. C'est le cas notamment du réarrangement particulaire intra-agrégat théorisé par Kemper et Rosenau (1984 ; 1986) et Dexter et al. (1988). Selon ces auteurs, les cycles d'humidité engendrent des modifications de la structure interne des agrégats, ce qui modifie sa stabilité structurale.

Plus précisément, et toujours selon ces auteurs, l'humectation d'un agrégat cause à la fois la diminution du nombre de contact inter-particule et la détérioration des liens entre ces particules conduisant à une diminution générale de la stabilité structurale. Lorsqu'un agrégat

sèche, la phase liquide se concentre dans les ménisques à l'interface entre les particules générant une tension qui conduit à rapprocher les particules concernées, pouvant amener à un contact. Ce contact peut induire des forces de friction qui augmentent la cohésion générale de l'agrégat. De plus, tandis que les ménisques se rétractent à l'interface entre les particules lors du séchage, l'eau qui les compose voit sa concentration en particules d'argile et composés solubles augmenter. Ces particules et composés peuvent alors flocculer ou cristalliser pour former de nouveaux liens entre les particules. Ces processus conduisent à une augmentation de la stabilité structurale lors du séchage. Bien que basé sur des théories référant notamment à la physique des milieux granulaires humides, ce processus et son lien avec la variation de la stabilité structurale restent des conjectures et n'ont jamais été directement observés. Cela n'empêche pas ce processus d'être largement cité dans la littérature et incriminé dans les variations de stabilité structurale en liens avec les cycles d'humidité (e.g. Zhang & Horn, 2001; Deneff et al., 2001; Six et al., 2004).

L'objectif de cette troisième partie de la thèse est de vérifier la survenue de ce processus à travers deux études expérimentales. Ces études ont été réalisées sur des échantillons de sol collectés sur l'un des sites présenté dans la deuxième partie. La stabilité structurale de ce sol s'était avérée sensible à l'histoire hydrique du sol.

Le chapitre 5 présente les résultats d'une expérimentation dont les objectifs étaient d'évaluer les processus physico-chimiques actifs lors de cycles d'humidité appliqués à un massif d'agrégats, de quantifier les variations de déformation globale et de pression interne entre les particules à l'aide de micro-capteurs de contrainte afin de vérifier l'occurrence du réarrangement particulaire théorisé par Kemper et Rosenau (1984 ; 1986). Cette étude a montré que la pression interne diminuait lors de l'humectation et augmentait lors de la dessiccation. Les déformations globales de l'échantillon montraient des variations concordantes. Avec le nombre de cycles d'humidité, le massif d'agrégats tendait à se

contracter et la pression interne tendait à augmenter, ce qui atteste d'un réarrangement de la structure de l'échantillon à l'échelle millimétrique. En revanche, ces variations n'ont pas pu être reliées à la stabilité structurale qui est restée quasi-constante pendant l'expérimentation.

Le chapitre 6 présente les résultats d'une expérimentation dont l'objectif était de caractériser un réarrangement particulière à une échelle infra-millimétrique par des mesures de micro-tomographie X. Des agrégats provenant du même type de sol et ayant une stabilité structurale initiale similaire ont été soumis à différents types de cycle d'humidité. Ces différents traitements ont permis d'obtenir des stabilités structurales contrastées. La structure interne d'agrégats ayant subis ces traitements a été caractérisée par micro-tomographie X. Dans un premier temps, les mesures devaient être réalisées par un micro-tomographe de laboratoire. Les problèmes techniques liés à une résolution inadaptée de l'appareil, puis à des défaillances de l'appareil lui-même, ont nécessité l'utilisation un autre tomographe. Finalement, des mesures de micro-tomographie synchrotron ont pu être réalisées à Shanghai en décembre 2011. Néanmoins, la qualité des images était relativement décevante, et questionnait leur utilité pour une analyse quantitative. Cependant, grâce à l'appui de Laurent Michot (LEM CNRS, Nancy) et de Pierre Levitz (PECSA, UPMC, Paris) nous avons pu réaliser des mesures quantitatives sur une partie de nos images, et finalement aller jusqu'au bout de notre démarche. Si les traitements appliqués ont engendré des changements dans la structure interne des agrégats, ces changements n'ont pas pu clairement être reliés à la stabilité structurale.

Chapitre 5 :

**Effets des cycles humectation-dessiccation sur la
contrainte interne, le réarrangement particulaire et
la stabilité structurale**

**Wetting-and-drying cycles effects on internal stress, particle
rearrangement and soil aggregate stability**

Baptiste ALGAYER *et al.*

In preparation.

1. Introduction

Soil structure refers to the size, the shape and the arrangement of solids and voids within the soil matrix (Lal, 1991). It is a key factor in soil functioning, its ability to support agriculture and to moderate environment quality (Brownick & Lal, 2005). Aggregate stability is the ability of an aggregate to retain its structure when exposed to exogenous stress such as wetting. The stability of aggregates affects the movement and storage of water, soil carbon sequestration and biological activity (Six et al., 2000), as well as plant growth and developments by influencing plant emergence and root penetration (Gallardo-Carrera et al., 2007). Finally, it also affects soil sensitivity to erosion and crusting (Le Bissonnais, 1996; Bajracharya et al., 1998; Barthès & Roose, 2002).

In the field, soil is submitted to wetting and drying cycles induced by rainfall occurrence. Wetting and drying cycles are recognized to affect soil aggregate stability through physical, chemical and biological processes (e.g. Utomo & Dexter, 1982; Amézketa, 1999; Deneff et al., 2001; Brownick & Lal, 2005; Cosentino et al., 2006). While the interactions between wetting and drying cycles and biological activity and their influence on aggregate stability have benefit from recent studies (e.g. Deneff et al., 2001; Cosentino et al., 2006), the physico-chemical processes affecting aggregate stability during wetting and drying cycles still remain unclear (Algayer et al., in preparation).

Previous studies suggested several physico-chemical processes of aggregate stability variation related to wetting and drying. Aggregate stability can be affected by the flocculation and dispersion of clay (e.g. Emerson, 1977; Dexter *et al.*, 1988) and by the crystallization/dissolution of soluble components (e.g. Kemper & Rosenau, 1986; Hohlthussen *et al.*, 2010) which create bonds between coarser particles. It was also suggested that aggregate stability could be affected by the particle rearrangement occurring during wetting and drying cycles (Kemper & Rosenau, 1984; 1986; Dexter et al., 1988).

Particle rearrangement refers to processes widely studied in physics of the granular media and related to the interaction between solid and liquid phases (e.g. Fisher & Israelachvili, 1981; Coussy, 1991; Alberts et al., 1997; Hornbacker et al., 1997; Bocquet et al., 2002; Sheel et al., 2008). In a humid granular media, inter-particle cohesion is related to the presence of water bridges (or menisci) at the interface between solid particles. Inter-particle cohesion is more influenced by the inter-particle menisci morphology than by the total water content in the porous space (Sheel et al., 2008). According to the Laplace law, a concave menisci shape is related to a pressure lower within the water menisci than within the atmosphere. This differential pressure results in attractive forces between particles bonded by the water menisci (Fisher & Israelachvili, 1981; Coussy, 1991; Alberts et al., 1997). The more concave the inter-particle menisci shape, the greater the inter-particle attractive force. Such force can induce the local displacement of the involved particles, leading to a more compact structure at larger scale (Coussy, 1991). Hence, the intensity of attractive forces is indirectly linked to the dynamic of water within the material. When a granular media is totally dry, there are no water bridges between the particles and thus, there is no inter-particle attractive force. At low water content, solid particles are bonded by water bridges, and the inter-particle attractive forces are related to the menisci numbers and morphology. With water content increase, water bridges coalesce into larger clusters inducing a decrease of the inter-particle attractive forces. With water content decrease, the water menisci retreats at the interface between particles into concave morphology, inducing an increase of the attractive forces. Such attractive forces can lead to the particle rearrangement into a more compacted structure with a greater number of inter-particle contacts. (Coussy, 1991; Hornbacker et al., 1997; Bocquet et al., 2002; Sheel et al., 2008).

Kemper & Rosenau (1984; 1986) and Dexter et al. (1988) used such theory to explain aggregate stability variations during wetting and drying cycles. They hypothesized that during

drying, particles within the aggregate rearranged in a more compact structure in such a way that aggregate stability increases. On the other hand, wetting would cause the decrease of the inter-particle cohesion and the weakening of the bonds between coarser particles leading to a decrease in aggregate stability. According to our knowledge, even if this hypothesis was based on solid theoretical background, the internal particle rearrangement during soil wetting and drying, and their influences on aggregate stability have never been observed yet. Further studies are required to verify its occurrence and if it is the case, quantify the involved forces and assess its influence on aggregate stability variation.

The aims of the work presented here are to 1) check the occurrence of particle rearrangement in relation with wetting and drying cycles by quantify the variation of soil internal stress 2) to assess the relationships between soil internal stress variation and aggregate stability.

2. Material and method

2.1. Soil aggregate cylinder preparation

The soil used in this study was a silt loam Luvisol collected from the Marcheville experimental site located in the south of the Parisian Basin, 15 kilometres in the south-west from the city of Chartres. Soil presented 16% clay, 80% silt, 3% sand and 1.2% organic carbon. The clay phase was mainly composed of illite, chlorite and very few swelling clay minerals. Soil was air dried at room temperature and sieved at 0.5 millimetres. Only the fraction < 0.5 mm was used.

Cylindrical aggregate blocks of 9.5 cm diameter and 4.0 cm height were built from this material. The air dried soil aggregates were carefully packed into a rigid PVC cylinder until reaching a bulk density close to 1.3 g/cm^3 . During the cylinder construction, 3 pressure sensors were placed at different positions at 2 cm height within the aggregate cylinder. The dry cylinder was wetted with deionized water using a vaporizer until reaching a 20%

gravimetric water content. Two micro-tensiometers were inserted into the cylinder at a 2 cm height and 2.5 cm from the cylinder side. The characteristics of the sensors will be mentioned later. The rigid PVC cylinder was carefully pulled out, resulting in a non-constrained and instrumented cylindrical soil sample.

3 cylinders were built, one instrumented (3 pressure sensors and 2 micro-tensiometers) and two non-instrumented to allow for sampling during the experimentation. The cylinders were placed on a hydrophilic to allow capillary wetting during the wetting phases. An initial wetting was applied near the saturation of the three cylinders.

2.2. Wetting-drying cycle parameters

The three cylinders were submitted to four successive wetting and drying cycles. Water tension of the instrumented cylinder was continuously measured in order to control the amplitude of drying phases. The experiment was done in a climatic room at a constant air temperature of 20°C and relative air humidity of 50%.

The drying phase durations were about 5 days. During the drying phases, samples were let to dry in the climatic controlled conditions until reaching a water tension of 5700 mm. When tensiometers reached the minimum value of 5700 millimetres, 90g of deionised and degassed water was applied at the base of the sample. Cylinders were capillary wetted up to near saturation. The wetting phase durations were about 4 hours. After each drying phase, half a non-instrumented cylinder was collected to measure aggregate stability. After this sampling, only half of the water amount was applied to the remaining half cylinder (45g of water) during the following wetting phase.

An initial first drying phase was applied at the beginning of the experiment. Then, four successive wetting and drying cycles were applied. For the drying phase of the fourth cycle cylinders were dried in constant air condition during about 19 days. The whole duration of one experiment was about 41 days. It was replicated three times.

2.3. Measurements

2.3.1. Sensors

All the measurements by sensors were done at a minute time step using a Campbell central. Water tension was measured using 2 micro tensiometres. Tensiometres were placed at opposite positions within the instrumented cylinder at a 2 cm height and 2.5 cm from the cylinder side.

The cylinder vertical strain was measured using laser sensor (Micro epsilon optoNCDT 1302) with a 10 μm resolution, positioned at 3 cm above the centre of the cylinder top.

Internal stress was measured by 3 pressure micro-transducers (Kyowa PS-05KC) with a 50 kPa capacity. Micro sensor had a disk shape with a 2 mm height and a 5 mm diameter. The 2 mm² sensitive area was located on one face of the disk. Pressure sensor was located on one face of the soil cylinder. Pressure micro sensors were placed within the soil cylinder at a 2 cm height and at 2.5 cm from the cylinder side. Each micro pressure sensor was positioned to a different orientation. Sensor 1 was oriented along a radial axis, sensor 2 was oriented along an axial axis and sensor 3 was located along an orthoradial axis (figure 1).

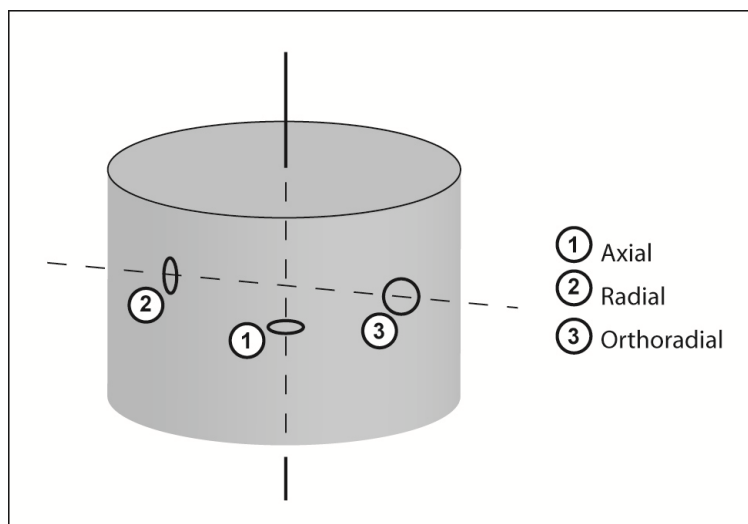


Figure 1: orientation of the pressure sensor

2.3.2. Aggregate stability

Aggregate stability measurements were done on samples collected from the non-instrumented cylinders at the end of each drying phases. Half cylinder samples were air dried at 40°C during 48 hours and cut into 3 to 5 mm pieces using a sharp knife (Darboux & Le Bissonnais, 2007). Aggregate stability was measured using Le Bissonnais method (Le Bissonnais 1996, *ISO/DIS 10930, 2012*). 5 g sub-samples were dried at 40°C for 24 h before application of one of the three tests (fast wetting, slow wetting, stirring), and each test was replicated three times. After the tests, the resulting fragments were sieved in ethanol. The results are presented using the mean weighted diameter (MWD).

3. Results

3.1. Water tension

At the beginning of the experiments, soil water tension was close to zero for the three replicates, indicating a saturated state of the soil sample (figure 2, 3, 4). As described in the previous part, the first drying phase was used to reach a similar state (5700 mm) before the cycle application. Hence, measurements are only considered from the first wetting phase. The tensiometres reached their limit capacity of measurement around 7000 mm. Hence, water tension could not be measured for the last 13 days of the final drying phase.

The water tension measurements showed similar pattern among the three replicates (figure 2A, 3A, 4A, table 1). The wetting phase duration was closely similar among the replicates, varying between 0.01 day (replicate 2, cycle 4) and 0.1 day (replicate 1, cycle 1 and 2). The drying phase duration varied between 5.7 days (replicate 1, cycle 1) and 4.5 days (replicate 3, cycle 1). The drying phase duration was found to decrease with the number of cycle for replicates 1 and 2. This phenomenon was not observed for replicate three.

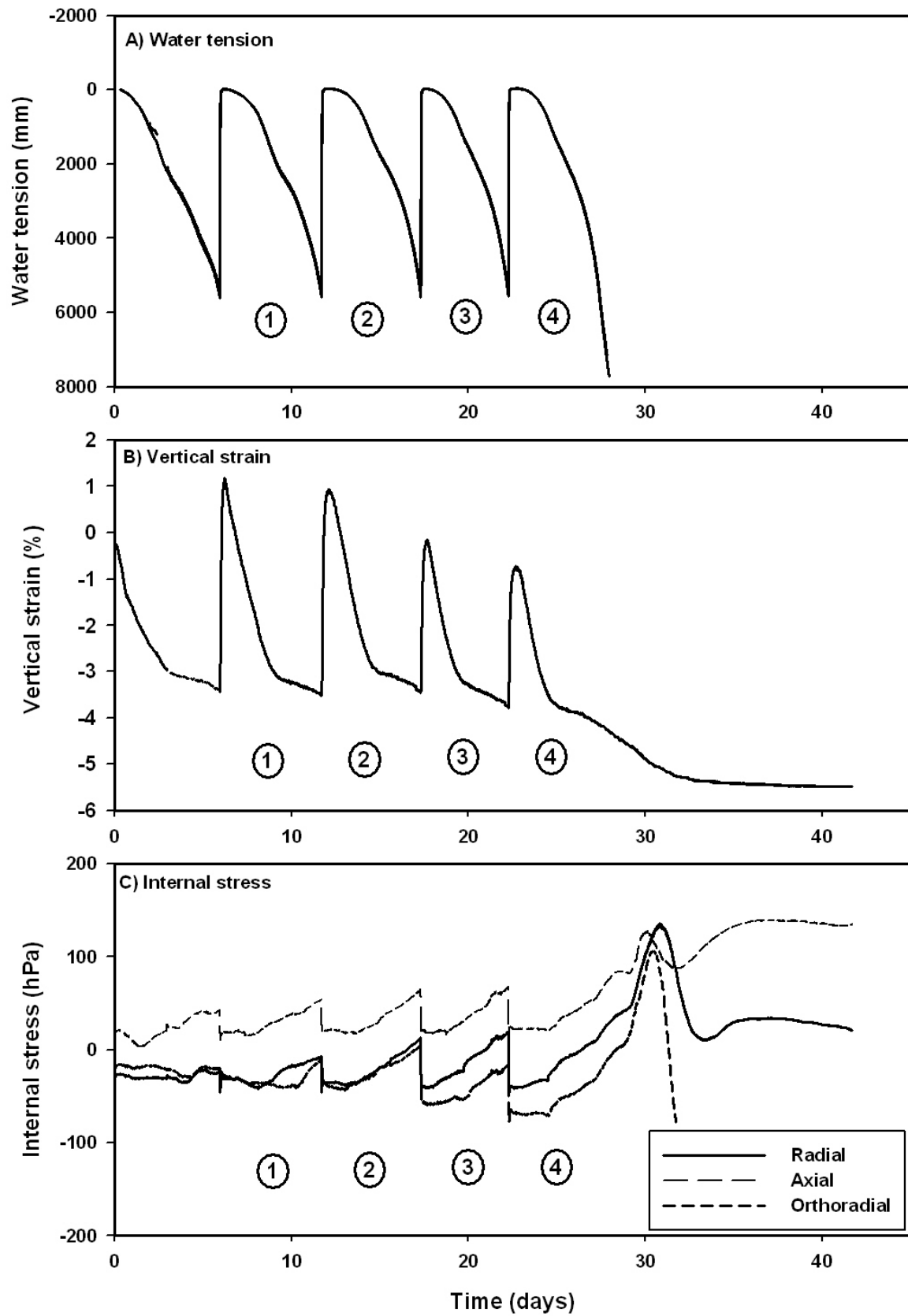


Figure 2: variation of water tension (A), vertical strain (B) and internal stress (C) in link with wetting and drying cycles, results of the replicate 1.

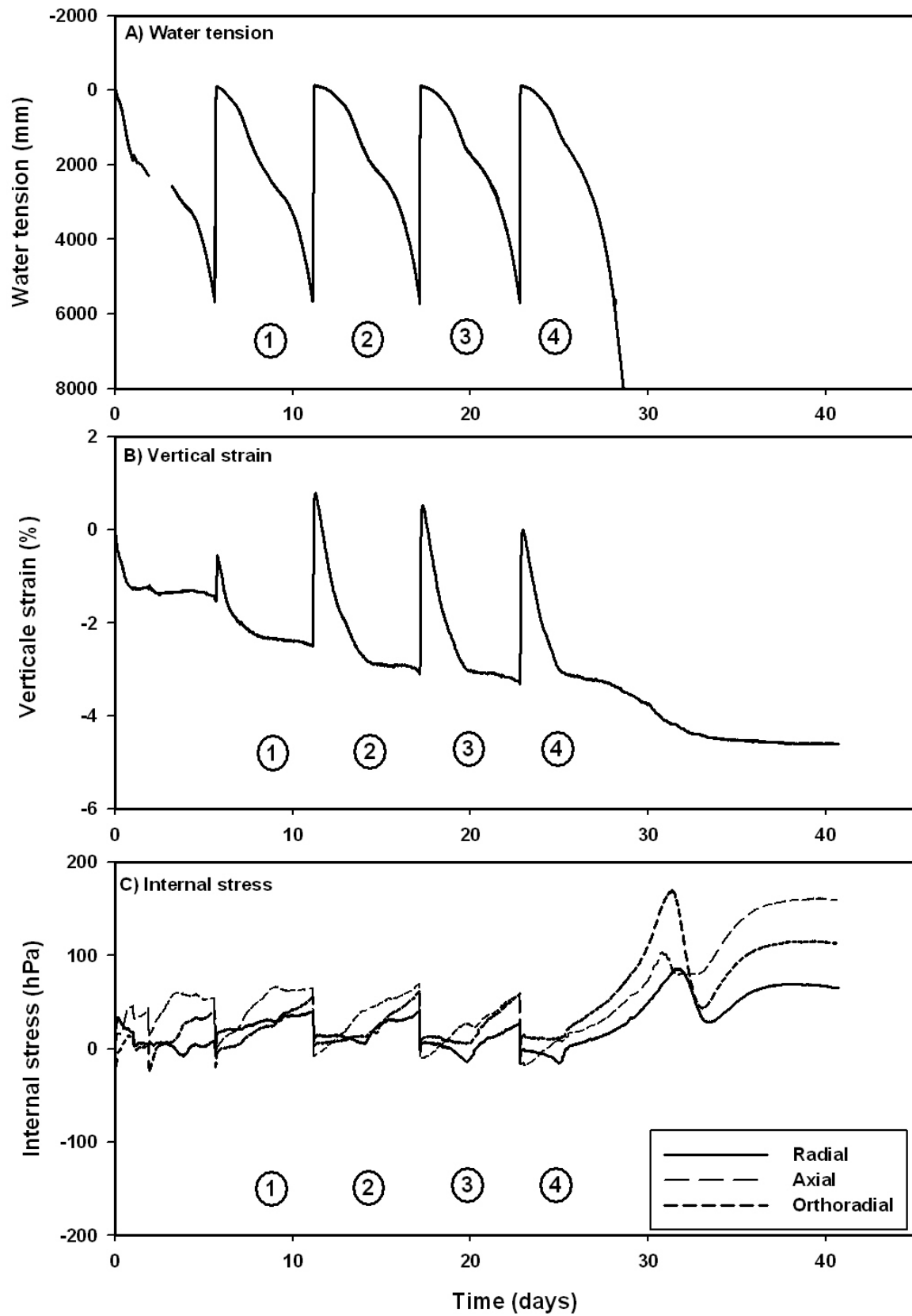


Figure 3: variation of water tension (A), vertical strain (B) and internal stress (C) in link with wetting and drying cycles, results of the replicate 2.

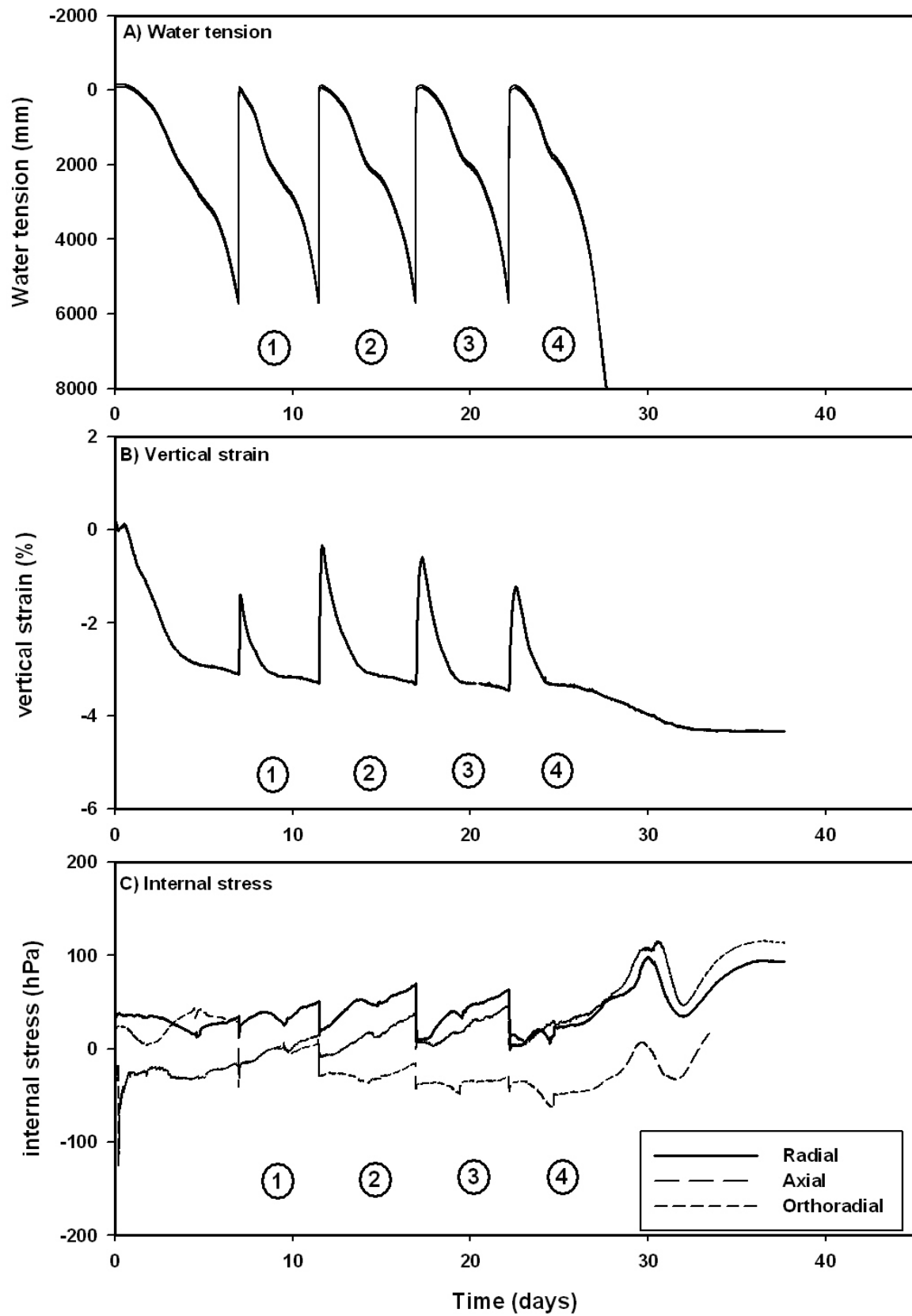


Figure 4: variation of water tension (A), vertical strain (B) and internal stress (C) in link with wetting and drying cycles, results of the replicate 3.

Table 1: Time duration (in days) of the wetting phases, saturation state (Sat.) and Drying phases for each cycle and for the 3 repetitions

	Cycle 1			Cycle 2			Cycle 3			Cycle 4	
	Wetting	Sat.	Drying	Wetting	Sat.	Drying	Wetting	Sat.	Drying	Wetting	Sat.
Replicate 1	0.12	0.38	5.74	0.12	0.74	4.75	0.09	0.50	4.36	0.09	0.80
Replicate 2	0.09	0.34	5.50	0.07	0.80	5.15	0.05	0.64	4.95	0.01	0.64
Replicate 3	0.05	0.09	4.51	0.06	0.44	4.95	0.07	0.64	4.54	0.10	0.54

3.2. Vertical strain

Vertical strain was quantified in percentage of the initial height of the soil cylinder (figure 2B, 3B and 4B). Table 2 presents the extreme values and amplitude of strain for each cycles of the three experimental replicates.

For the three replicates, the shape of the vertical strain curve followed the same pattern during the experimentation. The cylinder height increased quickly from the beginning of the wetting phase, until reaching a maximum value at the end of the wetting phase. During drying phases, the cylinder height firstly decreased rapidly and constantly. It was followed by a slow decrease period until reaching the minimum value corresponding to the maximum water tension (figure 2B, 3B and 4B). During final drying (cycle 4), cylinder height decreased quickly at first and then more slowly, and finally remained almost constant until reaching its minimum value during the last days of the experimentation (figure 2B, 3B and 4B).

Amplitudes of the vertical strains differed between experimental replicates (table 2). During drying-wetting cycles, vertical strain amplitude varied between 3% and 5%, corresponding to a height variation between 1.2 mm and 2 mm. For the cycle 1, the amplitude of vertical strain was the lowest (replicate 2 and 3) or the highest (replicate 1). For the following cycles, the amplitude of strains and the maximum cylinder height decreased with the number of cycles (Table 2; Figures 3B and 4B). Finally, the vertical strain amplitude was

larger for cycle 4 because sample was allowed to dry longer than for previous cycles (Table 2; Figure, 2B, 3B, 4B).

Table 2: Extreme values and amplitude of the vertical strain (% of initial sample height) for each cycle and for the three repetitions

	Cycle 1			Cycle 2			Cycle 3			Cycle 4		
	Max	Min	Ampl.	Max	Min	Ampl.	Max	Min	Ampl.	Max	Min	Ampl.
Replicate 1	1,13	-3,52	4,65	0,92	-3,42	4,34	-0,17	-3,78	3,61	-0,74	-5,49	4,75
Replicate 2	-0,54	-2,50	1,96	0,79	-3,12	3,91	0,52	-3,33	3,85	-0,02	-4,60	4,58
Replicate 3	-1,41	-3,31	1,90	-0,35	-3,32	2,97	-0,58	-3,46	2,88	-1,22	-4,30	3,08

3.3. Internal stress

For the three experimental replicates, internal stress during wetting and drying cycles followed closely the same pattern whatever the orientation of the sensor. During wetting phases, internal stress firstly dropped quickly (about fifteen minutes after the beginning of wetting) until reaching a minimum value. This period was followed by a slow increase until reaching a value that was stable while water tension stayed close to zero. This period of stability continued during the beginning of the following drying phase, and was followed by a progressive increase in internal stress until reaching a maximum value at the end of the drying phase (figure 2C; 3C; 4C).

During the three first drying phases, internal stress variation was irregular (figure 2C, 3C and 4C). Indeed, even when internal stress was globally increasing, short changes in the internal stress occurred for all the replicates and sensor orientations. These short time variations could occur simultaneously. This phenomenon was observed neither during the wetting phases nor during last days of the experiments (figure 2C, 3C and 4C).

Overall, during wetting-drying cycles, internal stress amplitude varied between 50 hPa and 170 hPa. Amplitudes of the internal stress variations differed with the wetting-drying cycles, with the orientation of the sensor and among the replicates (Table 3). Amplitude of internal stress also varied among successive drying-wetting cycles. Generally, amplitude of

internal stress increased during the three first cycles (table 3). Same results were found for maximum and minimum values of internal stress: generally, extreme values of internal stress increased during the first three cycles (table 2). During the fourth cycle, amplitude was generally larger than during previous cycles.

During the final drying phase, stress firstly increased progressively until reaching a maximum. Then, stress decreased rapidly and finally, it increased to reach a stable value during the last days of measurement. The three stress orientation curves varied quite simultaneously during the final drying phase. First peak was reached around the thirtieth day for the three experimental repetitions (figure 2C, 3C and 4C).

Table 3: extreme values and amplitude of the internal stress (kPa) for each cycle, for the different orientations and for the three repetitions.

	Cycle 1			Cycle 2			Cycle 3			Cycle 4		
	Max	Min	Ampl	Max	Min	Ampl	Max	Min	Ampl	Max	Min	Ampl
Replicate 1												
Axial	54,21	10,4	43,81	65,44	14,86	50,58	68,04	16,48	51,56	135,75	18,08	117,67
Radial	-7,33	-45,35	38,02	12,96	-45,67	58,63	19,88	-44,77	64,65	139,27	-46,08	185,35
Orthoradial	-9,94	-36,47	26,53	4,93	-45,45	50,38	-14,86	-59,63	44,77	106,73	-77,12	183,85
Replicate 2												
Axial	66,08	-0,81	66,89	69,87	-8,50	78,37	58,13	-11,43	69,56	102,63	-17,52	120,15
Radial	39,46	-7,02	46,48	41,86	11,55	30,31	26,84	-3,28	30,12	86,06	-13,63	99,69
Orthoradial	55,31	-19,30	74,61	63,56	2,88	60,68	59,18	-2,91	62,09	169,77	-2,09	171,86
Replicate 3												
Axial	7,42	-23,25	30,67	-14,79	-30,88	16,09	-29,66	-43,07	13,41	7,39	-45,76	53,15
Radial	50,97	12,25	38,72	69,87	13,81	56,06	63,73	8,96	54,77	97,75	-0,15	97,90
Orthoradial	14,88	-40,51	55,39	38,11	-18,09	56,20	46,74	0,44	46,3	107,44	0,79	106,65

3.4. Aggregate stability

Aggregate stability was measured after each cycle (figure 5). Generally, MWD values were very low and for each of the measurements. MWD varied between 0.09 mm and 0.20 mm corresponding to very low aggregate stability according to Le Bissonnais' classes (1996) whatever the test. Even if we observed a slight increase of MWD with the successive drying-wetting cycles, considering the low range of variation, MWD stayed closely constant for the three experimental repetitions (figure 5).

4. Discussion

4.1. Influence of wetting and drying cycles on aggregate stability

Samples submitted to wetting and drying cycles corresponded to cylinders of “re-formed” aggregates composed of aggregates with diameters lower than 0.5 mm. Aggregate stability measurements were performed after each drying phases, on re-formed aggregates with diameters between 3 and 5 mm. Hence, during the first wetting-drying cycle, particles composing the initial aggregates (<0.5 mm) rearranged to form larger aggregates. Internal structure of the sample was reorganised: new bonds were created between initial aggregates. The aggregate stability tests performed after each drying phases aimed at assessing the strength of those created bonds. Results showed very low aggregate stability whatever the experimental repetitions. MWD was above 0.2 mm, corresponding to very unstable structure according to Le Bissonnais (1996).

Several studies showed that aggregate stability was affected by wetting and drying cycles (e. g. Utomo & Dexter, 1982; Singer et al., 1992; Barzegar et al, 1995; Cosentino et al., 2006). Such studies showed opposite results in term of aggregate stability, depending mainly on the wetting and drying rates of the wetting and drying cycles. In the present study,

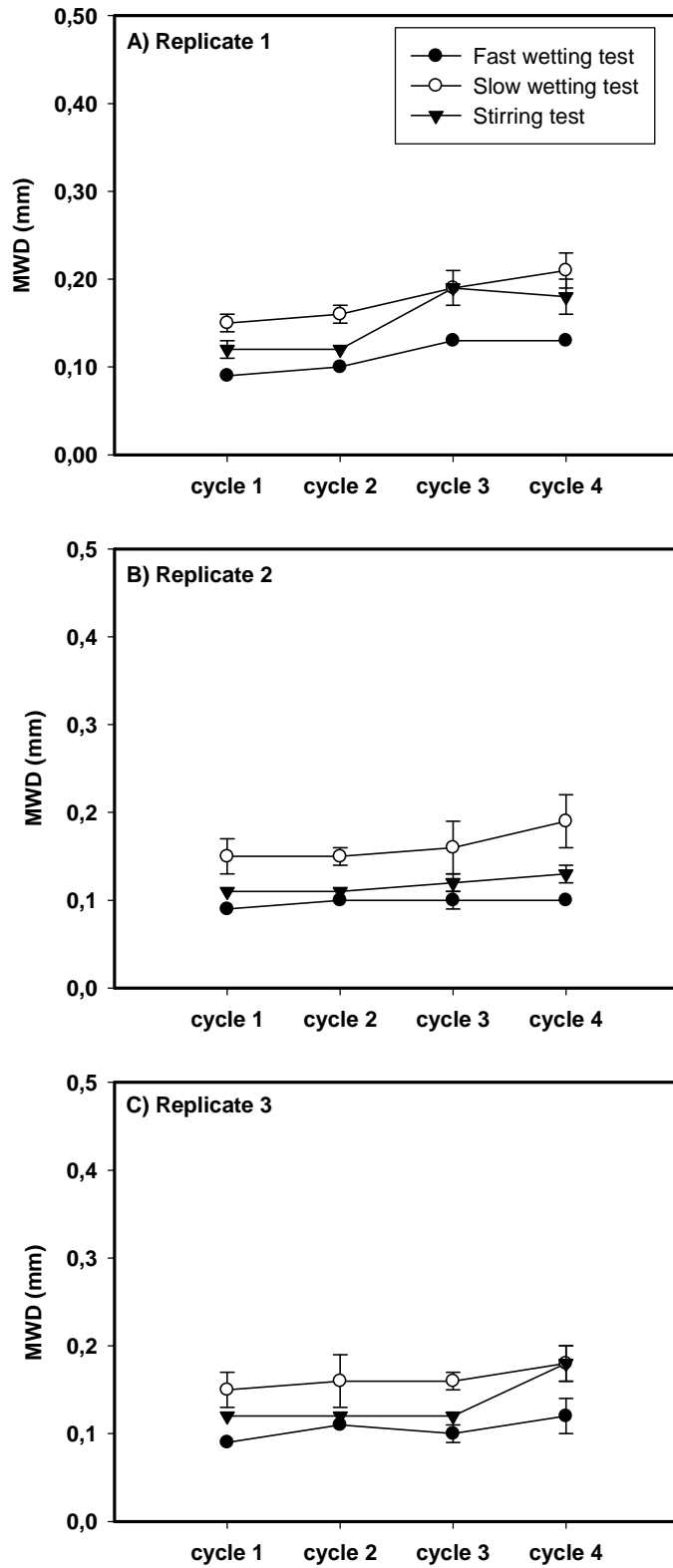


Figure 5: variation of aggregate stability with wetting and drying cycles for A) first experimental replicate, B) second replicate and C) third replicate.

aggregate stability did not change with the wetting and drying cycles. Such result can be explained by the experimental setups of the study. Wetting and drying cycles including fast wetting rate and slow drying rates tend to decrease aggregate stability, while cycles including slow wetting rates and fast drying rates tended to increase aggregate stability (Utomo & Dexter, 1982; Barzegar et al., 1995; Deneff et al., 2001). For the present study, the applied wetting and drying cycles corresponded to low amplitude of water tension. Moreover, both wetting and drying rates were slow. Indeed, we had to limit the water tension amplitude in order to stay in the measurement capacity of the tensiometers. Hence, lack of effect of wetting and drying on aggregate stability can be explained by the low amplitude of water tension induced by the wetting and drying cycles. After the final drying phase, aggregate stability slightly increased. Even if this increase was very low, it was observed for the three experimental repetitions. Such result underlined the influence of the drying amplitude on aggregate stability variation: wetting and drying cycles including a strong drying would probably have induced larger increases in aggregate stability. Hence, the challenge is now to reproduce the present experimental setup with larger amplitude of the wetting and drying cycles with the aim to investigate further the influence of water tension and internal stress variation on aggregate stability.

4.2. Quantification of the variables measured by the sensors

The three replicates showed similar results. During wetting and drying cycles, water tension varied a saturated state, and 5700 mm (about 55 kPa). It is important to note that during the three first wetting and drying cycles, sample never reached a full dry state. Such dry state was reached at the end of the final drying phase. The sample height increased during wetting phases and decreased during drying phases. Vertical strain amplitude varied between 3% and 6%, corresponding to variations height between 1.2 mm and 2.4 mm. Internal stress

decreased during wetting phases and increased during drying phases. Internal stress amplitude varied through a range between 50 hPa and 170 hPa.

The vertical strain and internal stress measurements aimed at assessing particle rearrangement in the sample structure at two scales: the whole sample (4 cm height) and millimetric scale (area of the sensitive surface of the internal stress sensor). Vertical strain and internal stress followed cyclic patterns corresponding to water tension cycles. While vertical strain and internal stress variations followed the variations of water tension, the relationship between them was quite complex and could not be described by a simple proportionality. In fact the rate of water tension increase was smaller at the beginning of drying and greater at the end of drying phase while exactly the inverse is true for internal stresses and vertical strain.

Vertical strain was measured at the whole sample scale (4 cm height) from variation of the sample height. The soil used for the experiment did not have swelling clays, and thus, the measured global swelling and shrinkage was not due to clay swelling and shrinking, but was controlled by the variations of the porous volume. The internal stress micro-sensors measured the pressure within the sample at a millimetric scale. As the result of internal stress variation did not present the same pattern than water tension variation, it means that the pressure micro-sensors did not measured the water tension. Those variations of stress corresponded to the pressure exerted by the particles near and at the contact with the sensor surface. The initial pressure measured at the beginning of the experiment may correspond mainly to the sample weight: particles composing the cylinder exerted a pressure on the sensor by their own weight. But, as it stayed constant during the experiment, the particle weight did not influence the variations of pressure measured during wetting and drying cycles. Internal stress variation was related to the attractive or repulsive forces between particles, controlled by the inter-particle water bridges dynamics.

4.3. Non reversible behaviour and particle rearrangement

For each cycle, the extreme values of water tension were controlled which explain the perfect repetition of water tension records whatever the cycle and replicate. Otherwise, the maximum sample height reached at the end of the wetting phases and the vertical strain amplitude tended to decrease with the number of cycles: the sample height reached at the end of a wetting phase was not reached again at the end of the following wetting phase. At the same time, maximum internal stress reached at the end of drying phases and internal stress amplitude tended to increase with the number of cycles.

This non reversible behavior corresponded to a global packing of the cylinder and an overall decrease of the pore space with the number of cycle. This phenomenon occurred at the whole sample scale as showed the decrease of maximum height of the cylinder. The shrinkage of the pore space induced by the drying phases was not reversible and the samples never found their initial heights during the swelling induced by the following wetting phase. This packing of the structure is also manifested at local scale with the progressive increase of the internal stress. While the total porous volume decreased, solid particles get into a closer contact to each other and the overall contact of grains with sensors increased as well as the number of water bridges between particles leading to a greater internal stress. From this point of view the stress and strain measurements were consistent to each other.

4.4. Processes induced by wetting and drying cycles

Both vertical strain and internal stress dynamics corresponded to two facets of the same process. Upon drying, an increasing part of the water evaporated, macro pores were emptied firstly, and the remaining water retreated in menisci at the interface between particles. While capillary forces increased, water menisci retreated with more concave shape and particles bonded by water bridges were submitted to greater attractive forces (Coussy, 1991; Alberts et al., 1997; Sheel et al., 2008) generating a shrinkage and an increase of the internal stress. This

may induced that the pressure of the solid particles on the sensor surface increased during drying phases. Upon wetting, the pores were progressively filled by the incoming water, and the remaining inter-particle menisci coalesced leading to a rapid decrease in the attractive forces between particles (Coussy, 1991; Alberts et al., 1997; Sheel et al., 2008). Such release induced a decrease of the internal stress. The progressive hydration of the porous phase led to the global swelling of the sample.

During the final drying phase, water tension increased until reaching the limit of the tensiometres measurement capacity: around 7000 mm (about 70 kPa). Water tension continued to increase during the last 13 days of the experiment but was not measured. During this final drying phase, sample height decreased continuously until equilibrating to its minimum value (between -4% and -5% from its initial height). Internal stress increased until reaching its maximum about 8 days after the beginning of the final drying phase. Such phenomenon was observed almost simultaneously for the three oriented sensors. This result can be explained by the increase of tension within inter-particle menisci as described previously. Indeed, because the sample was allowed to dry longer than for previous drying phases, internal stress increased to higher values, until reaching its maximum. After this peak, the internal stress decreased quickly. Such result may be explained by the disappearance of the remaining inter-particle menisci: the contacts between particles were almost dry at the end of the final drying phase, and thus, the attractive forces induced by the retreating menisci almost disappeared. Currently, the variations of internal stress following this decrease remained unexplained.

During drying phases, the increasing attractive forces induced by the inter-particle retreating menisci must be great enough to involve local particle displacement. Solid matrix rearranged in a more compact structure inducing closer contact between particles. This is confirmed by the overall shrinkage of the sample during the drying phases. While the shape of

the water tension and vertical strain curve showed a smooth and progressive pattern during drying phases, internal stress curve showed a less regular pattern. This phenomenon can be explained by local particle rearrangement. During drying, solid particles were submitted to two opposite forces: the attractive force induced by the increase of inter-particle capillary tension, and the resistance against this attraction induced by the friction of the irregular shapes of the involved particles. During drying, attractive forces between particles increased. When such attractive forces exceeded the frictional resistance, particles moved into a position inducing a shorter distance (and possibly a contact). Irregularities on the internal stress curve could be explained by such local rearrangements of particle at and near the sensor surface. Water tension and vertical strain measurements are integrative methods. Water tension was measured by micro-tensiometres with a spatial influence of a few cubic centimetres. Vertical strain was measured at the global sample scale, and corresponded to the sum of all the local particle rearrangements. Internal stress was measured by a less integrative method: the sensor sensitive area was 2 mm² only, explaining why only internal stress curve showed irregularities. Those irregularities were not observed at the end of the drying phase. This must mean that particle rearrangement did not occur any more at the end of the final drying. Indeed, as water bridges were evaporated, the particles were not any more submitted to the attractive forces induced by inter-particle capillary tension.

Hence, the results of the present study advocate for a rearrangement of the solid matrix of the sample during wetting and drying cycles. Such rearrangement involved local movement of the solid particles and are in accordance with Kemper and Rosenau (1984; 1986) theory. Such phenomenon was observed at the scale of the whole sample scale and at the stress sensor scale (few square millimetres). Measurements of particle rearrangement at microscopic scale are required to definitely confirm this hypothesis.

5. Conclusion

A cylinder composed of <0.5 mm aggregates from a silt-loam soil was submitted to controlled wetting and drying cycles. Aggregate stability measured on 3-5 mm “re-formed” aggregates from the cylinder was low and constant after each cycle. Such result can be explained by low applied drying rates. Larger amplitude of the wetting and drying cycles and faster drying rate would have been required to induce aggregate stability variation. The whole sample showed vertical strain: swelling during wetting phases and shrinking during drying phases. The amplitude of vertical strain reached 3 to 5 % from the initial sample height. Internal stress was measured within the sample. Internal stress decreased during wetting phases and increased during drying phases within a range of 50 to 170 hPa. Such phenomenon was probably related to the inter-particle water bridges dynamics, generating attractive or repulsive forces between particles. Cylinder tended to shrink with the succession of wetting and drying cycle while internal stress tended to increase, leading in a whole compaction of the sample. This result also advocates for a rearrangement of the solid matrix with wetting and drying cycles. Such rearrangement has been observed at a millimetric scale and has now to be confirmed by observation at smaller scales (micrometric).

Acknowledgements

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Chapitre 6 :

**Influence des cycles d'humectation-dessiccation sur
la stabilité structurale et la structure interne.
Approche expérimentale par micro-tomographie X.**

**Influence of wetting-and-drying cycles on aggregate stability and
internal structure.**

Baptiste ALGAYER *et al.*

In preparation.

1. Introduction

Aggregate stability corresponds to the ability of an aggregate to retain its structure when exposed to a stress such as wetting. The stability of aggregates affects the water movement and retention of water within the soil matrix, the soil carbon sequestration and biological activity (Six et al., 2000) and plant growth and developments by influencing plant emergence and root penetration (Gallardo-Carrera, 2007). Finally, it also affects soil sensitivity to erosion and crusting (Le Bissonnais, 1996; Barthès & Roose, 2002).

Aggregate stability is a dynamic property that changes with time in relation with biological activity and climate (Caron et al., 1992; Bajracharya *et al.*, 1998; Cosentino et al., 2006; Dimoyiannis, 2009; Algayer et al., to be submitted). It is well established that microbial activity and organic matter content have a positive effect on aggregate stability (e.g. Tisdall & Oades, 1982; Chenu *et al.* 2000). Climate may affect aggregate stability mainly by the water content dynamics (e.g. Caron et al., 1992; Bajracharya *et al.*, 1998; Algayer et al., to be submitted). If some factors influencing aggregate stability variation have been identified, there are still difficulties to predict aggregate stability, especially when biological activity was not stimulated by external amendments (Cosentino et al., 2006; Dimoyiannis, 2009; Algayer et al., to be submitted).

Table 1: effect of soil type, wetting rate and drying rate on aggregate stability variation during previous wetting and drying cycles experiments (without stimulation of biological activity).

WDC= wetting and drying cycles.

Reference	Soil type	Wetting rate	Drying rate	Effect on aggregate stability
Tisdall et al., 1978	Sandy loam (Sterilized)	Capillary wetted -4 kPa	21°C (24h)	Increase with WDC
Utomo & Dexter, 1982	Sandy loam (sterilized)	Capillary wetted -10kPa	-100 kPa	Increase after 2 WDC
Singer et al., 1992	Artificial aggregates (non sterilized)	Capillary wetted until saturation (5min)	40°C (3h)	Decreased with WDC
Barzegard et al., 1995	Clay soil (not amended)	Capillary wetted -10kPa	Room T° (3d) then 50°C 24h	Increased with WDC
Denef et al., 2001	Silt loam (with fungicide)	Capillary wetted field capacity	25°C 2d	No changes
Cosentino et al., 2006	Silt loam (not amended)	Capillary wetted to -3.1kPa (2d) an then, -10kPa (14h)	20°C (10h)	Slight increase after 2 WDC

It is well known that aggregate stability variation is affected by wetting and drying cycles (Amézketa, 1999; Brownick & Lal, 2005). However, there are uncertainties on the effect of successive wetting and drying on aggregate stability (table 1). Some studies found that wetting and drying cycles decreased aggregate stability (e.g. Tisdall et al., 1978, Singer et al., 1992) while others showed that wetting and drying cycles increased aggregate stability (e.g. Utomo & Dexter 1982; Barzegard et al., 1995) Those opposite results may arise from differences in the studied soil properties (texture, mineralogy, aggregation) and differences in experimental conditions such as the intensity of the wetting-drying cycles (rate of wetting, intensity of drying, number of cycles) (table 1). Wetting induces physico-chemical processes recognized to decrease aggregate stability (e.g. Kay and Dexter, 1990; Le Bissonnais, 1996; Zaher *et al.* 2005). Wetting provokes the decrease in water tension leading to a loss of inter-particle cohesion (Sheel et al., 2008). At the macro aggregate scale, soil wetting can cause air entrapment inside capillary pores that induces aggregate slaking while wetting. Non uniform or differential hydration and swelling of the clay fraction can cause micro-cracking. The soil

wetting can also cause the dispersion of clay and chemical dissolution of soluble components that acts as cement and form inter-particle bonds. The response of soil aggregate under wetting depends closely on the wetting rate. Highest wetting rate triggers the most disruptive processes that break inter-particle bonds, leading to aggregate stability decrease or even aggregate breakdown in smaller fragments. On the other hand, drying tends to increase aggregate stability (e.g. Kemper and Rosenau, 1984; Rajaram and Erbach, 1998; Deneff et al., 2001). The increase in water tension induced by drying increase the inter-particle cohesion (Kemper and Rosenau, 1984; Kemper et al., 1987; Dexter et al., 1988; Rajaram and Eberbach, 1998). The cited processes are the precipitation and flocculation of cements agents (clay, soluble components, (hydr)oxides) creating bonds at the inter-particle contacts and the changes within the aggregate micro-structure such as particle rearrangement (Kemper & Rosenau, 1984; 1986; Dexter et al., 1988). According to these authors, soil wetting would cause both a decrease of the inter-particle cohesion and a weakening of the bonds between particles. The combination of both processes leads to a decrease in aggregate stability while wetting. During drying, the water phase recedes into capillary wedges surrounding particle to particle contacts. The internal tension pulls adjacent particles together leading to much closer contact between particles (Kemper & Rosenau, 1986; Dexter et al., 1988). Direct contact between particles can induce friction forces that increase inter-particle cohesion, and provide the formation of bonds between particles due to flocculation of clays or precipitation of soluble components, increasing aggregate stability. Based on the literature review, the internal particle rearrangement during wetting and drying cycles theorized by Kemper & Rosenau (1984; 1986) and Dexter et al. (1988) have never been directly observed yet. However, this process is widely cited and incriminated in aggregate stability changes linked to wetting and drying cycles (e.g. Zhang & Horn, 2001; Deneff et al., 2001; Six et al., 2004).

Micro-tomography provides the opportunity to image and investigate the 3D structure of numerous materials at scales ranging from few nanometres to several millimetres. In recent years, the development of X-ray micro-tomography techniques has offered a great opportunity to explore the three-dimensional inner space of soil aggregates non-destructively (Young et al., 2001). It is therefore an excellent tool for studying soil aggregate microstructure (Peth et al., 2010). While study on pore characteristics (e.g. porosity, pore size distribution, pore length, pore shape) benefited from micro-tomography techniques (e. g. Peth et al., 2008; Zhou et al., 2012; Levitz et al., in press), It also offers an excellent opportunity to study the arrangement of solid particles within the aggregate at the micro-scale. Calculation of mass auto-correlation function and morphological analysis can be used to characterize the structure and properties of the material, while topology analysis can assess the connectivity of the solid phase within the sample. Hence, X-ray micro-tomography and associated tools of image analysis could be used to check the existence of the particle rearrangement as a process of aggregate stability variation in relation with wetting and drying cycles.

The objectives of the the work presented here are to 1) study the stability of aggregates submitted to different types of wetting-drying cycles, 2) check the existence of the particle rearrangement as a process of aggregate stability variation using observation by X-ray micro-tomography.

Two millimetres diameter aggregates from a silt clay soil were submitted to wetting and drying cycles. Aggregate stability decreased after the treatment including a fast wetting rates and a slow drying rate, while it increased with the treatment including a slow wetting rate and a fast drying rate. Image analysis of micro-tomography measurements showed very slight differences in the aggregate micro-structure questioning the occurrence of the particle rearrangement.

2. Material and method

2.1. Material

The aggregates used in this study were collected from the surface horizon of a silt loam Luvisol located at the Marcheville experimental site, South of the Parisian Basin, 15 kilometres in the south-west from the city of Chartres (48°21'512"N; 1°16'0.55"E). Soil presented 16% clay, 80% silt, 3% sand and 1.2% organic carbon. Soil was air dried at room temperature, clods were slightly fragmented by hand and sieved between 1 and 2 millimetres. Treatments

Before the application of wetting and drying cycles, a measurement of the initial aggregate stability was done (T0). Two treatments corresponding to different types of wetting and drying cycles were applied to aggregate samples.

Treatment T- was designed to decrease the aggregate stability of the sample. The wetting phase was done using a fast wetting rate, and the drying phase was done using a slow drying rate. 60 grams samples were capillary wetted on a suction table equilibrated at a -1 kPa matric potential (pF 0.4) during 10 minutes (until saturation). A 10 g subsample was collected for gravimetric water content measurement. After saturation, the sample was moved into a pressure cell to control the matric potential during the drying phase. Hydric potential was firstly set at -3.2 kPa (pF 1.5) for 3 hours, and then set at -10 kPa (pF 2) for 15 hours. A 10 g subsample was collected for gravimetric water content measurement. Then the remaining sample was oven dried at 40°C for 48 hours. T- included only one wetting and drying cycle. A 15 grams sample was collected for aggregate stability measurement. 15 grams sample remaining were kept for tomography measurements.

Treatment T+ was designed to increase the aggregate stability. The wetting phase involved a slow wetting rate and the drying phase involved a fast drying rate. A 120 g sample was capillary wetted on a suction table equilibrated at a -10 kPa matric potential (pF 2) during

4 hours. A 10 g subsample was collected for gravimetric water content measurement. After wetting, the sample was oven dried at 40°C during 24 hours. After this drying phase, a 10 g subsample was collected to measure gravimetric water content. The wetting and drying cycle was repeated 3 times. After each cycle, a 15 g subsample was collected for aggregate stability measurement. The 15 g sample remaining at the end of the third cycle were kept for tomography measurements.

A 50 g sample was kept in the dry state (oven, 40°C) for all the experiment duration and was used as a control.

2.2. Measurements

2.2.1. Aggregate stability

Aggregate stability was measured before the first wetting and drying cycle and after each cycle using a slightly modified version of slow wetting test from Le Bissonnais's method (Le Bissonnais 1996, *ISO/DIS 10930, 2012*). 5 g sub-samples were dried at 40°C for 24 h before the application of the test, and each test was replicated three times. Aggregates with diameters between 1 and 2 mm were capillary wetted on a tension table at a matric potential of -0.3kPa for 30 min. After the tests, the resulting fragments were sieved in ethanol. The results are presented using the mean weighted diameter (MWD).

2.2.2. Water content

Water content was measured gravimetrically after each wetting and drying phases for both treatments. 3 g were oven drying at 105°C for 48h. Each measurement was replicated 3 times.

2.3. Micro tomography and image analysis

2.3.1. *Micro-tomography measurements*

At the end of each treatment, the 1-2 mm diameter aggregates were dried and carefully cut into 0.8 mm diameter aggregates, using a sharp blade. Micro tomography measurements were performed on these individual aggregates. For each treatment (T-, T+, and T0), three aggregates were imaged by X-ray micro-tomography. Aggregates were scanned with a synchrotron-based micro computed tomography (m-CT) at beam line BL13W1 of the Shanghai Synchrotron Radiation facility (SSRF). Aggregates were placed into a plastic tube mounted on a rotary stage. The stage rotated from 0° to 180° and absorption radiographs of the samples were acquired at 0.1° interval. The distance between the sample and the detector was 10 cm. Scanning was conducted with a maximum X-ray energy of 28 keV. The “ctconstruct” software (developed by SSRF) was used to reconstruct the slices from the radiographs. 2048 slices with a size of 2048 * 2048 pixels for each slice were reconstructed for every sample. Every voxel had a volume of 0.74 μm * 0.74 μm * 0.74 μm, and the voxel associated attenuation coefficients were stored as values ranging from 0 (lowest attenuation) to 255 (highest attenuation).

2.3.2. *Image Analysis*

Calculation of mass auto-correlation function

The calculation of mass auto-correlation function is an indirect way to analyse structural correlation of a solid matrix, and thus, a useful tool for characterizing material structure which did not required the segmentation of the images (Brisard et al., 2012). Such method consists, for one pixel localized in the solid matrix, to calculate the probability to find another pixel at a given distance (r) in the solid matrix. Such method does not allow assessing precisely the sample microstructure but give statistical information (Brisard et al., 2012). Results were

presented by the $I(q)$ graph, where the scattered intensity $I(q)$ is the Fourier Transform of the correlation function of the electronic density $r(r)$, which corresponds to the probability to find a scatterer at position r in the sample if another scatterer is located at position 0 , and q is the momentum transfer or scattering vector. More detailed information on the small angle scattering analysis can be found in Boyard et al., (2005) and Levitz (2007). Measurements were performed from the non segmented 3D projections images, on the three samples for each treatment (T0, T-, T+). For each sample, 5 3D projection images were analyzed.

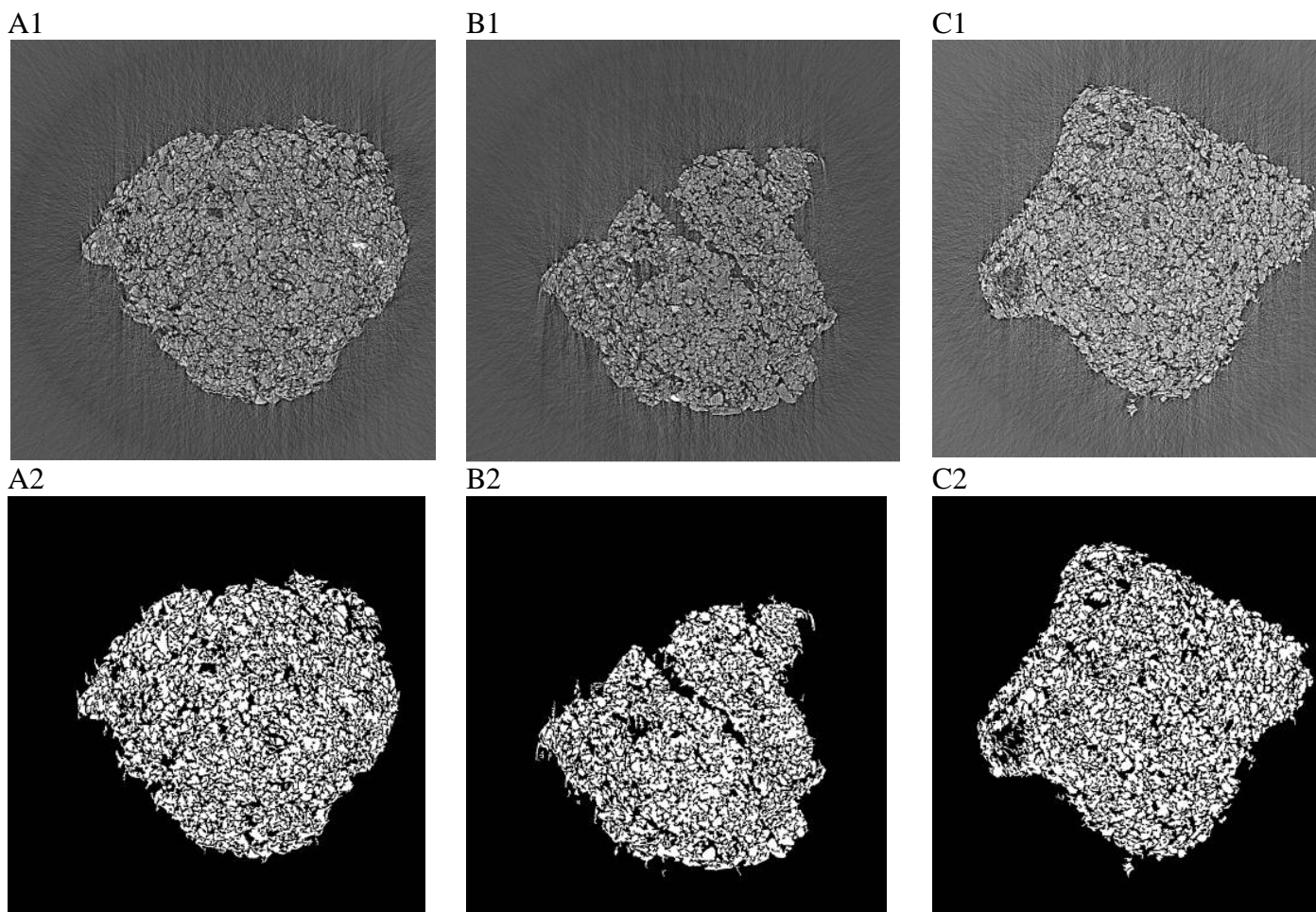


Figure 1: original slides from the reconstruction (A1, B1, B3) compared to segmented slides (A2, B2, C2).

Segmentation

Precise image quantification involves correctly segmenting the solid and porous phases. For the present study, the segmentation threshold was defined from the gray scale histogram of each slide. Segmentation was applied for each slide separately. Aberrant points and isolated clusters were deleted by erosion. Results of the segmentation of three slides from samples submitted to the three treatments are shown in figure 1.

Morphology

The concept of “chord-length distribution” introduced by Mering and Tchoubar (1968) is used to characterize two-phase system such as porous materials on stereological principles. A chord is defined as a segment which belongs either to the pore or to the solid and has both ends on the interface. Hence, chord distributions are stereological tools used to describe the interface between pore and solid phases (Levitz and Tchoubar, 1992; Rozenbaum et al., 2007). Chords are obtained by tracing random and homogeneously distributed straight lines (rays) through a section or a 3D structure. The chord length distribution function gives the probability of having a chord length between r and $r+dr$ belonging either to the pore network ($f_p(r)$) or to the solid matrix ($f_m(r)$). $\langle l_p \rangle$ and $\langle l_m \rangle$ are the average chord length in the pore network and in the solid matrix. They are considered as estimators of the mean size of the pore and solid particles (Cousin et al., 1996; Rozenbaum et al., 2007). 3D global parameters such as the relative volume of the solid matrix (ϕ_s) can be estimated from the mean chord length as:

$$\phi_s = \frac{\langle l_m \rangle}{\langle l_m \rangle + \langle l_p \rangle}$$

For each treatment (T0, T-, T+), chord distribution measurements were performed on 3D segmented sections of 100 voxels high, selected from the 3D segmented images. At the moment, only one sample from each treatment has been analysed. Repetitions have to be done

in order to confirm the results. Chord length distribution and relative volume of the solid matrix (ϕ_s) were calculated for each sample.

Topology

The topological properties of a network quantifies its connectivity. The topology of the solid matrix was assessed by building the skeleton graph of segmented 3D images and by calculating its connectivity index (C_T).

Skeleton graph is defined as the set of centres of all maximal spheres included in the solid network. It is obtain by progressively narrowing the solid space of segmented 3D images (Levitz et al., in press). Skeleton graph was used to quantify the number of vertexes (α_0) and branches (α_1) between them. In order to characterize in a simple way the topology of the 3D skeleton graph, the connectivity index (C_T) was calculated as follow (Levitz et al., in press):

$$C_T = \frac{\alpha_1 - \alpha_0}{\alpha_0}$$

The higher the connectivity index, the more connected is the solid matrix.

For each treatment (T0, T-, T+), skeleton graph and connectivity indice (C_T) were calculated on the same 3D segmented sections as the morphology characterization.

3. Results

3.1. Water content

Initial water content was 0.9 % \pm 0.1%. For the treatment T-, after the wetting phase, gravimetric water content reached 23.5 % \pm 0.2%. After 15 hours equilibration at -10 kPa, gravimetric water content was 19.5 % \pm 0.2%. At the end of the drying phase, water content was 0.7% \pm 0.1%. For the treatment T+, during the first cycle, water content varied between 12.9% \pm 0.7% (end of the wetting phase) and 0.8% \pm 0.1% (end of the drying phase). During the second cycle, water content varied between 12.4% \pm 0.9% at the end of the wetting phase

and $0.5\% \pm 0.1\%$ after the drying phase. For the third cycle, water content varied between $13.5\% \pm 1.2\%$ and $0.7\% \pm 0.2\%$.

3.2. Aggregate stability

Aggregate stability varied significantly for both treatments while it remained constant for the experiment duration for the control samples (Figure 2). Initial and final values of MWD of the samples were $0.67 (\pm 0.06)$ mm and $0.63 (\pm 0.05)$ respectively. After the treatment T-, MWD decreased to 0.39 mm, corresponding to a decrease of 42% (figure 2). For the treatment T+, after the first wetting and drying cycle, MWD decreased slightly up to 0.60 mm. After the third cycle, the MWD increased up to 0.85 mm corresponding to an increase of 27% (Table 2).

3.3. Microstructure measured by tomography

3.3.1. Mass auto-correlation function

Figure 3 presents the 3D projection images for each treatment (A1, B1 and C1) and the associated log-log scale plot of $I(q)$ versus q (A2, B2, C2). Considering the curves, no differences have been observed among the different repetitions for a same treatment (data not shown). Moreover, no difference was found between the treatments: curves exhibit a similar pattern for all the treatments (figure 3A2, 3B2, 3B3).

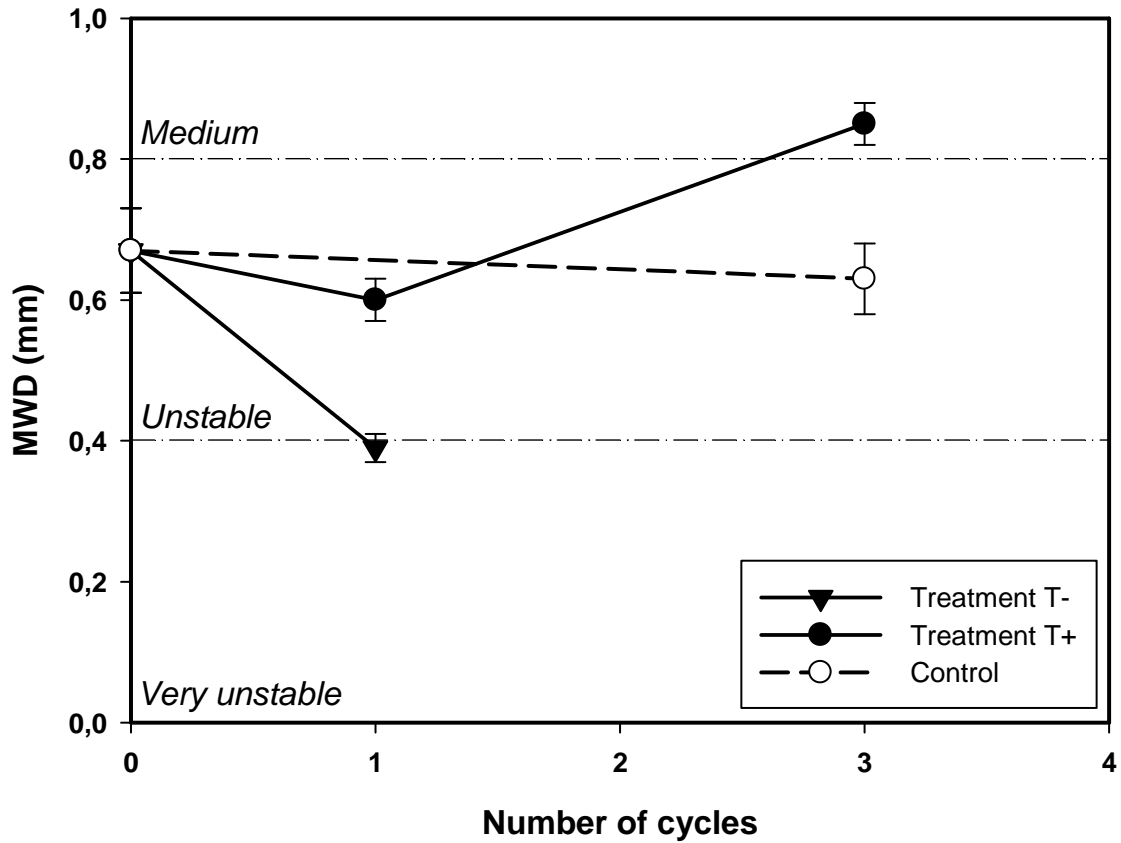


Figure 2: MWD (mm) variation in relation with wetting and drying cycles

Treatment + = slow wetting rate / fast drying rate

Treatment - = fast wetting rate / slow drying rate

Control = no wetting and drying cycles (continue dry state, 40°C)

Each MWD = mean of 3 repetition, $n=3$

Bars = standard errors

Aggregate stability classes from Le Bissonnais (1996)

Table 2: mean chord length for the pore fraction (l_p) and the solid matrix (l_m) (μm) and relative solid volume (ϕ_s) for the three treatments.

Sample	l_p	l_m	ϕ_s
Treatment T0	6.03	7.35	0.55
Treatment T-	5.81	6.76	0.53
Treatment T+	5.74	7.89	0.58

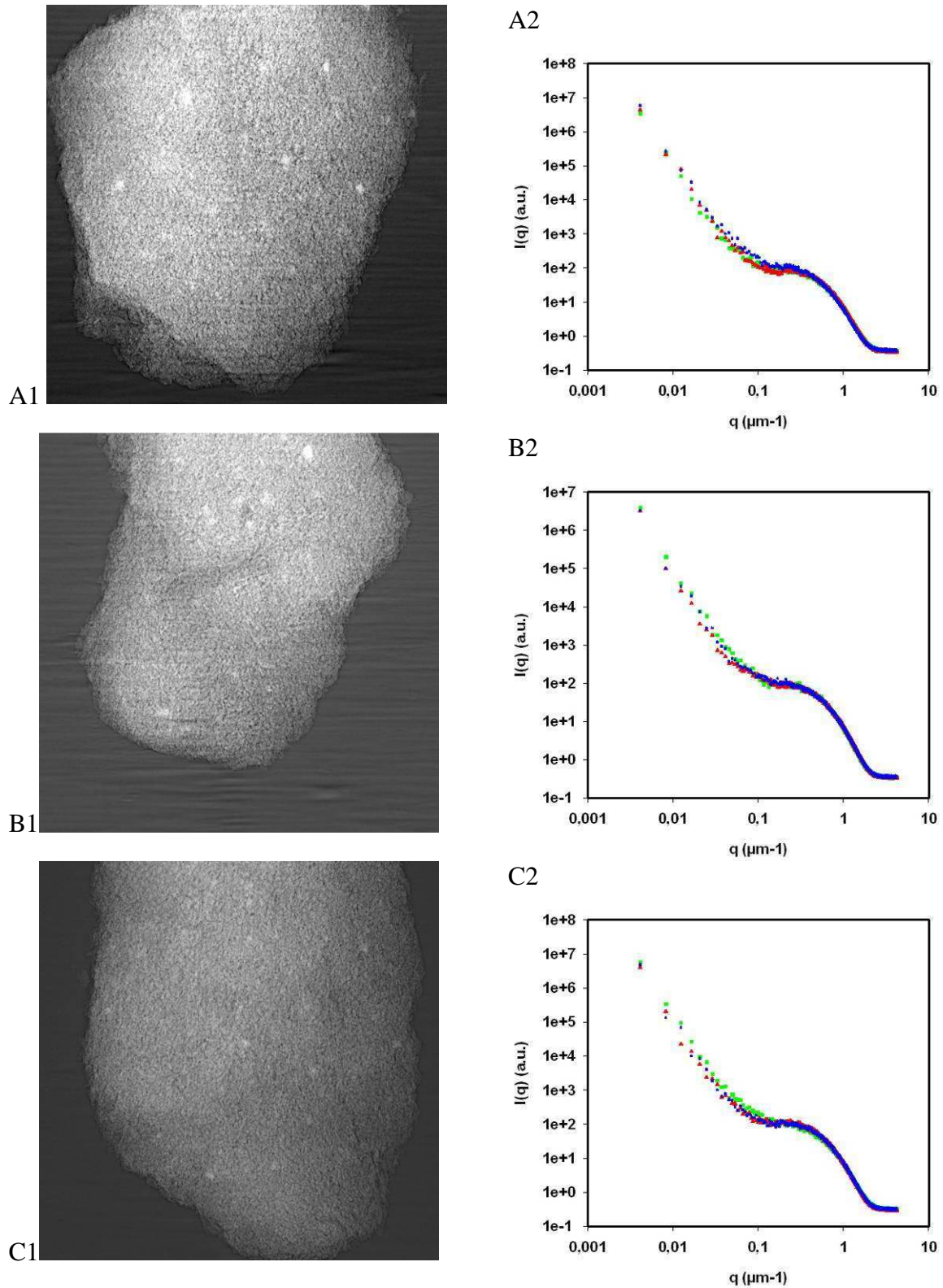


Figure 3: X-ray micro-tomography 3D projections images (A1, B1, C1) and corresponding to small-angle scattering curves (A2, B2, C2) for samples before treatment (T0) (A1, A2), after treatment - (B1, B2) and after treatment + (C1, C2).

Different colours means different replicates

Image size is 2048*2048 pixels with a pixel size of $0.74 \mu\text{m}$. a.u. = arbitrary unit

The curve had two slopes about -4, at high q values ($> 0.5\mu\text{m}^{-1}$) and at low q values ($< 0.05\mu\text{m}^{-1}$) (figure 3A2, 3B2, 3B3). Such slopes correspond to the Porod's law indicating a sharp interface between the solid and the porous phases of the image (Boyard et al., 2005). Large q values refer to the interface between the two media at the surface of the elementary particles composing the sample. Porod's law at high q values indicates a sharp interface between elementary particles and the porous phase. Low q values refer to the surface of the clusters composed of aggregated elementary particles. Porod's law at low q values indicates a sharp interface between clusters and the porous phase. The intermediate q values refer to the size and morphology of elementary particles.

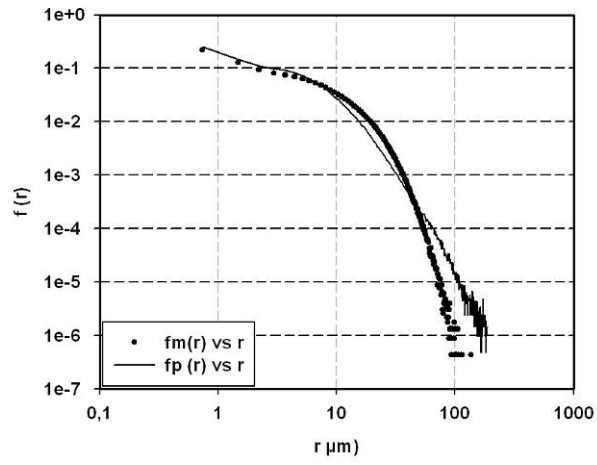
3.3.2. Morphology

Figure 4 present the chord distribution of the solid matrix ($f_m(r)$) and the porous phases ($f_m(p)$) for the three treatments. Very close patterns can be observed between the different treatments. Chord distribution in the solid matrix and porous phase presented an exponential distribution. Table 2 presents the mean chord length for the solid matrix and the porous phase and the relative solid volumes for the three treatments. Small differences can be observed between the different treatments. Initially (T0), ϕ_s was 0.55. It decreased slightly up to 0.53 for treatment T- and increased slightly up to 0.58 for treatment T+ corresponding to variations of about 5% (Table 1).

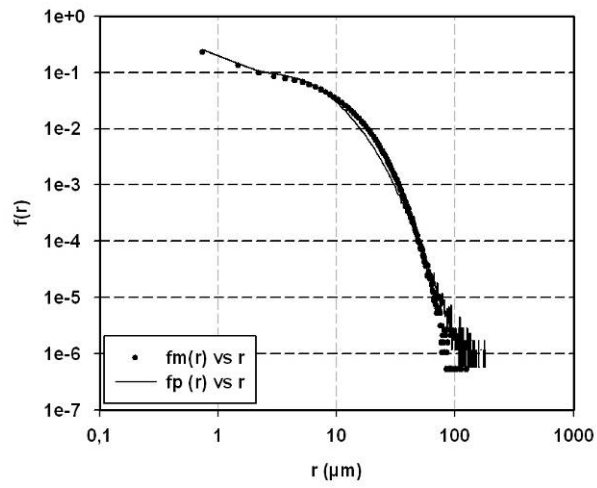
3.3.3. Topology

The connectivity index CT summarizes the information on the sample topology measured on the 3D skeleton graph. CT indices were very similar between treatment T0 and treatment T- (0.72 and 0.73, respectively). For treatment T+ the CT index decreased to 0.62 (about 15%) corresponding to a lower connectivity among the solid matrix (table 2).

A



B



C

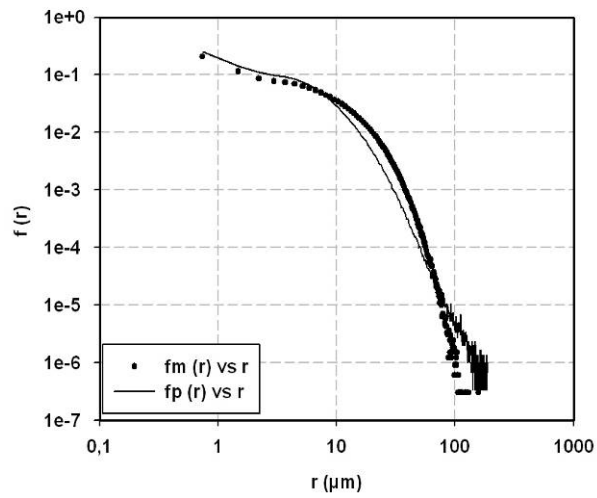


Figure 4: chord distribution for the three treatments T0 (A), T- (B) and T+ (C).

Image size is $2048 \times 2048 \times 100$ voxels with a voxel size of $0.74 \mu\text{m}$.

Figure 5 showed the 3D skeleton graphs of the section of 100 voxels height for each treatment. We can observe that the three considered samples presented very dense internal structure characterized by a high number of connections between particles. Such high connectivity is underlined by a high number of vertexes and branches (table 3).

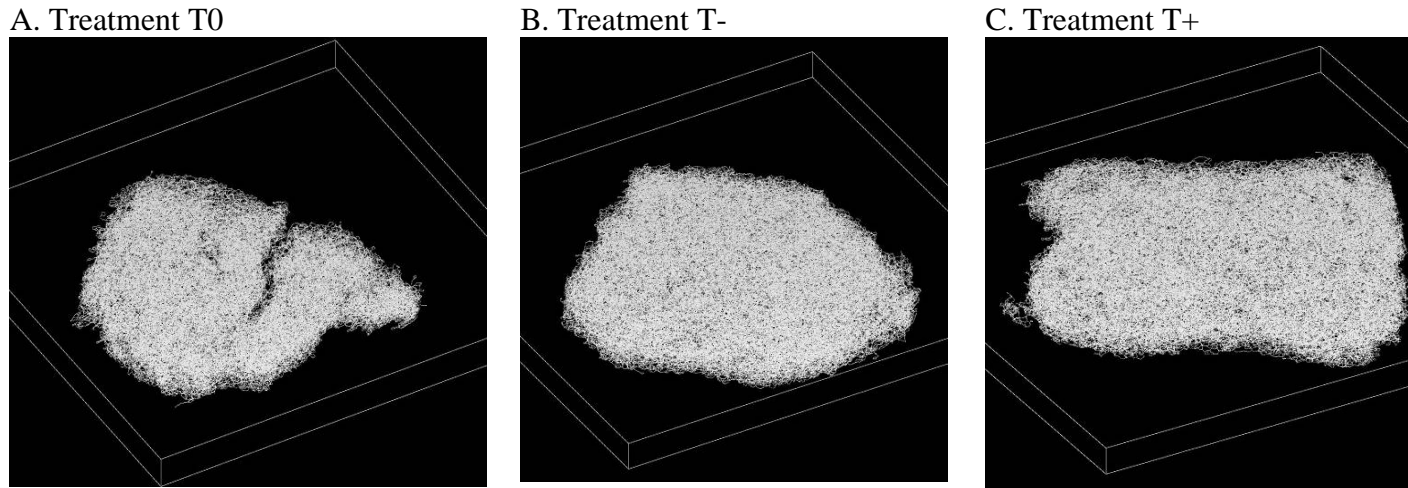


Figure 5: skeleton graph for the three treatments T0 (A), T- (B) and T+ (C).

Image size is 2048*2048*100 voxels with a voxel size of 0.74 μm .

Table 3: number of vertexes (α_0), branches (α_1) and connectivity index (C_T) for the three treatments.

Sample	α_0	α_1	C_T
Treatment T0	57717	99133	0.72
Treatment T-	76819	132590	0.73
Treatment T+	68797	117289	0.62

4. Discussion

4.1. Aggregate stability changes

The present study aimed at testing two different wetting and drying cycles treatments on a population of aggregates (1-2mm diameter). Treatment T- corresponded to a fast wetting rate followed by a slow drying rate, and so was expected to decrease aggregate stability. MWD decreased by -42% from its initial value after a single wetting-drying cycle. Treatment T+ corresponded to a slow wetting rate followed by a rapid drying, and so it was expected to increase aggregate stability. MWD stayed almost constant after the first cycle, and increased by 27% after three wetting and drying cycles.

Numerous studies showed that aggregate stability was affected by wetting and drying cycles even without stimulation of microbial activity (e.g. Tisdall et al., 1978; Utomo & Dexter, 1982; Barzegar et al., 1995; Cosentino et al., 2006). Such studies showed that the influence of wetting and drying cycles on the aggregate stability was mainly controlled by the balance between the applied wetting and drying rates. The results of the present study confirm this relationship.

4.2. Changes in the microstructure

The results of the present study showed similar mass auto-correlation function curves among the samples submitted to a given treatment. For different aggregates submitted to the same treatment, the organisation of the solid matrix within the aggregates was very similar. Such result showed that internal structure of the analysed aggregates was homogeneous. The mass auto-correlation function analysis was not affected by the different treatments: curves followed closely the same pattern whatever the applied treatment.

According to Kemper & Rosenau (1984; 1986), the variation of aggregate stability induced by wetting and drying cycles is controlled by the rearrangement of the aggregate

micro-structure. The present study aimed at relating aggregate stability variations to modifications within the aggregate micro-structure in order to verify the real occurrence of this process. According to Kemper & Rosenau theory (1984; 1986) one may have expected that the contrasted aggregate stability measured after the different treatments would have led to differences in the mass auto-correlation function curves between the samples but this was not the case. This result can be explained by the limitation of mass auto-correlation function measurements. Indeed, such method gives a statistical averaged information and does not allow assessing precisely the sample microstructure (Brisard et al., 2012). If changes in the microstructure would occur, they affected specific locations of the solid matrix. Hence, a more detailed analysis of the solid matrix morphology and topology is required.

Chord distribution in the solid matrix and in the porous phase showed very small differences between treatment T- and treatment T0. Treatment T- showed a small decrease in the relative solid volume, and thus a small increase of the porous volume compare to the initial sample (T0). Moreover, treatment T- did not affect the solid matrix topology assessed by connectivity index. Connectivity was very similar for the measurements before and after the treatment. Such result means that during treatment T-, the sample porous volume increased slightly, but the global connectivity of the solid matrix stayed closely the same. Those results did not allowed to validate the particle rearrangement induced by the fast wetting rate theorized by Kemper & Rosenau (1984, 1986). Even if aggregate stability decreased significantly after the treatment T-, such decrease did not correspond to observable internal micro-structure changes. Other physico-chemical processes may be involved. These processes could act at the macro-aggregate scale (such as differential swelling of the clay), or at the interface of coarse particles (such as clay dispersion or dissolution of the bonds components).

Morphology and topology measurements showed much contrasted results between the treatment T+ and the reference T0. Treatment T+ showed a small increase in the relative solid volume, and thus a small decrease of the porous volume compared to the initial sample T0. Such result corresponds to a small shrinkage of the whole sample. The connectivity index decreased by 14% from its initial value after the treatment T+. These two results may seem contradictory: the whole shrinkage of the sample (assessed by morphological analysis) may result in a more packed internal structure, but the topological analysis showed an overall decrease in the solid matrix connectivity. The decrease in connectivity can be interpreted as a decrease in the number of contacts between particles within the aggregate. Hence, it seems difficult to relate the modification in the internal micro-structure observed within the aggregate, to the increase in its stability induced by the treatment T+. We can suggest that the increase in aggregate stability was induced by the rearrangement of small particles. Indeed the resolution of the images (about $1\mu\text{m}^3$) did not allowed to consider smaller particles. New clusters composed of small elementary particles could have been formed during the wetting and drying cycles of treatment T+. Such hypothesis is sustained by the whole shrinkage of the aggregate but has not been observed directly. Because of the small size of the involved particles, such hypothetical clusters may have been considered as individual particles after the image segmentation, and thus as individual vertex in the skeleton graph. Hence, the measured decrease in connectivity may be biased by this hypothetical formation of new clusters: the connectivity assessed at the coarse particle scale decreased but the connectivity at the smaller particle size might increase. Anyway, the present results only concerned one sample per treatment and so have to be confirmed by analysing the replicates.

The results of this study showed that aggregate stability decrease linked to the treatment T- was not due to modifications in the internal micro-structure. The occurrence of the particle rearrangement theorized by Kemper & Rosenau (1984; 1986) was not observed

for treatment T-. Treatment T+ resulted in slight changes of the aggregate micro-structure but morphological and topological analyses showed contradictory results and thus, were difficult to relate to the observed increase in aggregate stability.

5. Conclusion

Aggregate stability was clearly affected by both wetting and drying treatments. Aggregate stability decreased after one single cycle of high wetting and slow drying rates, and increased after 3 cycles of slow wetting and fast drying rates. This confirms that aggregate stability variations may be closely related to the wetting and drying rates. Internal structure of the aggregates was characterized by X-ray micro-tomography. Mass auto-correlation function calculation showed no differences in overall micro-structure between the treatments. Morphology analysis showed slight differences in the relative solid volume between the treatments, and topology analysis showed differences in connectivity of about 15% between initial samples and treatment T+. The present results have to be confirmed with replicates analysis. The study showed that aggregate stability decrease linked to the treatment T- was not due to detectable changes in the internal micro-structure. Moreover, even if treatment T+ showed changes in micro-structure, it was difficult to relate these changes to the observed increase in aggregate stability. The occurrence of a particle rearrangement as a process of aggregate stability increase, theorized 30 years ago remains to be confirmed. With the current experimental setup, the images corresponding to the different treatments also corresponded to different aggregates. The current challenge would be to measure internal micro-structure changes on a single aggregate submitted to wetting and drying cycles. Such measurements may allow to observe variations of internal micro-structure linked to aggregate stability variations, and thus to verify the occurrence of particle rearrangement theorized by Kemper & Roseau (1984; 1986).

Synthèse et conclusion

L'objectif de cette troisième partie était d'évaluer les processus actifs au sein des agrégats lors des cycles d'humectation-dessiccation, et de vérifier l'occurrence du processus de réarrangement particulaire intra agrégat théorisé par Kemper & Rosenau (1984 ; 1986) et Dexter (1988). Nous aboutissons aux conclusions suivantes :

1) L'influence des cycles d'humidité sur la stabilité structurale est essentiellement contrôlée par l'intensité de l'humectation et de la dessiccation.

Les résultats présentés dans les chapitres 5 et 6 montrent que l'intensité de l'humectation et de la dessiccation jouent un rôle majeur dans les variations de stabilité structurale liées aux cycles d'humidité. Les deux type de cycles d'humidité testés dans le chapitre 6 ont montré des variations contrastées de la stabilité structurale pour des agrégats issus d'un même type de sol et présentant une même stabilité structurale initiale. Un cycle d'humidité composé d'une humectation rapide et d'une dessiccation lente a permis de diminuer la stabilité de plus de 40%. Trois cycles d'humidité successifs, composés d'une humectation lente et d'un séchage rapide ont permis d'augmenter la stabilité structurale de 27%. Le protocole expérimental présenté dans le chapitre 5 utilisait des cycles composés d'une humectation de faible amplitude (variation entre l'état saturé et -55kPa) et d'une humectation et d'une dessiccation lentes (environ 12 h pour l'humectation et 5 jours pour la dessiccation). De tels cycles d'humidité n'ont pas permis de faire varier la stabilité structurale. Ce résultat confirme notre bilan des résultats observés par les précédentes études (e.g. Tisdall et al., 1978 ; Utomo & Dexter, 1982 ; Barzegar et al., 1995 ; Cosentino et al., 2006) : l'influence des cycles d'humidité sur la stabilité structurale est essentiellement contrôlée par l'intensité des phases d'humectation et de dessiccation.

2) Les cycles d'humidité engendrent une variation de la pression inter-particulaire qui peut générer un déplacement des particules.

Pour des variations d'humidité entre état saturé et -55kPa, la déformation verticale globale du massif d'agrégats a varié de 3% à 5%, tandis que la pression interne entre les particules mesurée par les micro-capteurs a variée de 50 hPa à 170 hPa. De telles variations peuvent entraîner des déplacements locaux de particules et donc un réarrangement de la structure interne du matériau. Ces déplacements sont contrôlés par les variations de tension dans les ménisques à l'interface des particules. Un fort séchage (fin du dernier cycle) entraîne des variations de contraintes internes qui n'ont pu être reliées à la déformation du massif d'agrégats. Le processus sous-jacent reste à préciser.

3) Les expérimentations n'ont pas permis de relier l'occurrence du réarrangement particulaire à des variations de stabilité structurale.

- *Les cycles d'humidité peuvent engendrer des variations de la structure interne des échantillons.*

Les résultats présentés dans le chapitre 5 attestent d'un réarrangement de la structure interne du massif d'agrégat à l'échelle millimétrique (surface active du capteur de pression). Avec le nombre de cycles, l'amplitude des déformations tend à diminuer, et le massif d'agrégat tend globalement à se contracter. Dans le même temps, la pression interne entre les particules tend à augmenter. Ces résultats sont concordants avec un rapprochement global des particules qui composent l'échantillon, attestant ainsi d'un réarrangement de la structure interne du massif d'agrégats avec les cycles d'humidité.

Les résultats de l'expérimentation présentés dans le chapitre 6, ont également permis d'observer des variations de la structure interne des agrégats soumis aux différents

traitements. Cette étude a permis de caractériser la structure interne des agrégats à une échelle beaucoup plus fine (quelques microns). Le traitement T+ composé d'une humectation rapide et d'une dessiccation lente a engendré une diminution du volume de pore de 10% et une diminution de la connectivité de la matrice solide de 15%. En revanche, pour le traitement composé d'une humectation rapide et d'une dessiccation lente (T-), nous n'avons pas observé de variations de microstructure.

- *Ces variations dans la structure interne n'ont pas pu être reliées à la stabilité structurale.*

Si des variations de microstructure ont été observées ou fortement suggérées par les résultats de ces deux études, en revanche, leur relation avec la stabilité structurale n'a pas pu être clairement établie. Pour l'expérimentation présentée dans le chapitre 5, les résultats de pression interne et de déformation du massif ont fortement suggéré un réarrangement de la structure qui aurait dû engendrer l'augmentation de la stabilité structurale. Cependant la stabilité structurale de l'échantillon est restée constante. On peut expliquer cette constance par une amplitude et des intensités faibles dans les cycles d'humidités appliqués. Des cycles plus amples avec des phases d'humectation et de dessiccation plus intenses auraient permis de faire varier la stabilité structurale. On peut également souligner que pour cette étude, la stabilité structurale de l'échantillon est restée très faible durant l'expérimentation. Le massif d'agrégat était initialement composé d'agrégats de diamètres inférieurs à 0,5 mm. Ces agrégats se sont combinés en macro-agrégats lors de la première humectation, ce qui a permis au massif non contraint d'être cohésif. On peut donc dire que le premier cycle d'humectation-dessiccation a permis de créer de nouveaux liens entre les agrégats initiaux.

Pour l'expérimentation présentée dans le chapitre 6, si le traitement T+ a permis d'augmenter la stabilité structurale, les variations de microstructure identifiées n'étaient pas totalement cohérentes et de ce fait difficiles à interpréter. Le volume poral a diminué, mais

dans le même temps, la connectivité de la phase solide a diminué également, attestant d'une diminution du nombre de contact entre les particules au sein de l'agrégat. Ces résultats ne permettent pas de valider le processus de réarrangement particulaire comme étant à l'origine de la variation de stabilité structurale observée. En se basant sur la théorie de Kemper & Rosenau (1984 ; 1986), une augmentation de la stabilité structurale aurait dû être accompagnée d'une augmentation de la connectivité de la matrice solide à l'échelle microscopique. Or, une telle variation de la microstructure n'a pas été observée. Parallèlement, la diminution de stabilité observée lors du traitement T- n'a pas pu être reliée à des variations de la microstructure interne de l'agrégat. Si une augmentation faible (environ 5%) du volume poral a bien été observée, la connectivité de la phase solide est restée la même (alors qu'une diminution était attendue). De ce fait, les deux expérimentations n'ont pas pu valider l'occurrence du réarrangement particulaire comme processus physico-chimique de variation de la stabilité structurale lors des cycles d'humidité. Ce processus doit faire l'objet d'études plus approfondies. Les deux expérimentations réalisées ont toutefois permis de mettre en place les bases de protocoles solides utilisant des techniques innovantes (microtomographie X, micro capteurs de pression) afin d'observer les variations de la microstructure des échantillons aux échelles millimétrique et micrométrique, offrant de ce fait, des perspectives intéressantes pour de prochaines expérimentations.

Conclusions générales et perspectives

Actuellement, les prédictions des modèles d'érosion présentent globalement des problèmes de précision (Jetten et al., 2003 ; Boardman, 2006). Les difficultés de prédiction de l'érosion par les modèles sont en partie liées à des difficultés dans l'estimation de l'érodibilité du sol (Jetten et al., 2003 ; Guimere et al., 2009). En effet, l'érodibilité est une notion complexe englobant différentes propriétés du sol influencées par de nombreux facteurs interagissant entre eux. L'érodibilité peut être estimée par la stabilité structurale du sol. Cependant, les connaissances concernant la variation de la stabilité structurale pour un même type de sol restent limitées, ainsi que les facteurs et processus qui sont à l'origine de ces variations. De ce fait, l'amélioration des connaissances concernant les facteurs et processus contrôlant la stabilité structurale apparaît comme un enjeu important pour permettre de mieux estimer le paramètre « érodibilité inter-rigoles » dans les modèles et donc de mieux prédire l'érosion.

Ce travail a été réalisé dans l'objectif général d'améliorer les connaissances concernant les processus physico-chimiques de variation de la stabilité structurale en se basant sur l'étude de la variation de la stabilité structurale à pas de temps court en relation avec les cycles d'humidité. Il a combiné suivi de terrain et expérimentations de laboratoire.

Synthèse des résultats

1. L'érodibilité d'un sol est une propriété dynamique que les modèles d'érosion doivent considérer

Comme nous l'avons énoncé précédemment, la stabilité structurale est un proxy de l'érodibilité inter-rigole. Nos travaux ont montré que la stabilité structurale d'un sol donné était une propriété fortement dynamique et que les variations de stabilité structurale observées correspondaient à des érodibilités contrastées.

1.1. La stabilité structurale de la surface d'un sol donné peut être très différente de celle du matériau sous-jacent

Les deux études de terrain présentées dans les chapitres 1 et 3 ont montré des différences importantes de stabilité structurale entre la surface du sol (0-5mm) et le matériau sous-jacent (10-50 mm). L'étude présentée dans le chapitre 1 a montré que, à un instant donné, la stabilité de la surface d'un sol encroûté est plus importante que celle du matériau sous-jacent. Ce phénomène a été observé sur la quasi totalité des sites prélevés. Au début du suivi de terrain présenté dans le chapitre 3, un travail du sol avait été réalisé. Le sol présentait donc un matériau homogène dans les 10 premiers centimètres de profondeur, et la stabilité structurale était la même pour la surface et la sub-surface. Au cours du temps, la surface et la sub-surface ont vu leur stabilité structurale suivre des variations différentes. Aussi, à un temps donné, la surface et la sub-surface ont pu présenter des stabilités tantôt similaires, tantôt très contrastées. Les différences observées n'étaient pas toujours dans le même sens : la surface présentant tantôt une stabilité plus forte, tantôt une stabilité plus faible que la sub-surface.

Les différences de stabilité entre surface et sub-surface n'ont pas pu être expliquées par les propriétés standards du sol (chapitre 1), ni par l'histoire hydrique ou la variables biologiques (chapitre 3). On peut suggérer que les processus de formation des croûtes structurales et sédimentaires soient à l'origine des différences de stabilité observées. Cette hypothèse doit être vérifiée par d'autres études.

1.2. La stabilité structurale d'un sol donné varie avec la localisation du site

Les résultats de l'étude de terrain menée en Chine (présentés dans le chapitre 1) ont clairement montré que pour un matériau présentant des propriétés similaires (en termes de mesures habituellement effectuées sur des échantillons de sols), l'érodibilité estimée par des mesures de stabilité structurale pouvait fortement varier selon le site. Ces sites étaient séparés

par des distances de quelques kilomètres au maximum. Des résultats similaires ont été obtenus lors du suivi de terrain effectué en France (présenté dans le chapitre 3) : la stabilité structurale de la surface des deux sites localisés sur des types de sol très voisins et séparés par quelques kilomètres, variait dans des gammes contrastées, et donc présentait des érodibilités différentes. Ces deux études ont également montré que la stabilité structurale du sol était similaire pour des placettes localisées dans un même champ et séparées par quelques dizaines de mètres. L'homogénéité spatiale d'une parcelle agricole semble donc se retrouver dans la stabilité structurale.

Pour l'étude chinoise, l'effet du site influençait à la fois la stabilité structurale de la surface du sol encrouté et celle du matériau sous-jacent. Les différences d'occupation du sol entre sites pourraient expliquer au moins une partie des variations observées, mais cela reste à vérifier. Pour l'étude française, le site n'influencait que la stabilité structurale de la surface, la sub-surface des deux sites présentant des stabilités globalement similaires. Les différences de stabilité structurale observées entre les sites français n'ont pu être expliquées ni par les propriétés du sol (car similaires entre les sites), ni par le climat régional (étant donné les faibles distances qui les séparaient), ni par l'occupation du sol (les deux sites étant gardés sans végétation au cours du suivi). Les différences pourraient provenir des cultures antécédentes, hypothèse qu'il reste à tester. Selon une autre hypothèse, les différences de stabilité entre les sites (français comme chinois) seraient dues à des différences de micro-climat, impliquant des dynamiques de teneur en eau du sol différentes. Les résultats du suivi de terrain à pas de temps court montrent en effet de petites différences dans la dynamique de la teneur en eau entre les sites Marcheville et La Gouethière : globalement, le site de La Gouethière présentait une stabilité structurale généralement plus élevée et des variations de teneur en eau plus fortes que pour le site de Marcheville. Cette influence du micro-climat à l'échelle des sites d'étude reste à vérifier.

1.3. La stabilité structurale d'un sol donné varie dans le temps

Les résultats présentés dans le chapitre 3 montrent clairement que la stabilité structurale varie fortement à pas de temps court pour la surface du sol comme pour la sub-surface. Les variations observées à pas de temps court sont de la même amplitude que celles observées à pas de temps mensuel (variations entre 20% et 30% selon les modalités). Ces variations de stabilité structurale correspondent à des érodibilités du sol contrastées.

Les variations temporelles de la stabilité structurale étaient différentes selon l'échelle spatiale considérée. Pour les deux sites (localisés à quelques kilomètres de distance et présentant des types de sol proches), la stabilité structurale a suivi les mêmes tendances de variations. Pour deux placettes localisées à quelques dizaines de mètres d'écart, dans le même champ, la stabilité structurale a présenté des tendances et des gammes de variation très semblables. Les relations entre les dynamiques de surface et de sub-surface de la stabilité structurale sont apparues moins évidentes, les deux matériaux superposés suivant tantôt les mêmes tendances de variation, tantôt des tendances différentes.

1.4. L'estimation de l'érodibilité dans les modèles d'érosion

Ces différents résultats attestent d'un comportement fortement dynamique de l'érodibilité d'un sol donné et soulignent, de ce fait, les difficultés d'estimation du paramètre érodibilité par les modèles d'érosion. En effet, la plupart des modèles d'érosion considèrent l'érodibilité comme une constante pour un type de sol donné (Jetten et al., 2003 ; Gumiere et al., 2009) : à un type de sol ils font correspondre une valeur d'érodibilité constante dans l'espace et le temps. Or, les résultats de la présente étude montrent que l'érodibilité d'un sol donné varie à la fois selon la localisation du site et dans le temps (à pas de temps court). Actuellement, les modèles d'érosion ne prennent pas en compte cette variation spatiale et temporelle de l'érodibilité d'un sol donné. Dans les meilleurs cas, la paramétrisation des modèles intègre une moyenne saisonnière de l'érodibilité (par exemple, RUSLE, WEPP), et

ignore donc la variabilité temporelle à pas de temps court. Mieux prédire l'érosion nécessitent pourtant de considérer les fortes variations temporelles de l'érodibilité d'un sol. Dans l'état actuel des connaissances, et afin d'optimiser la qualité des prédictions, les modèles d'érosion devraient considérer une gamme d'érodibilité pour un sol donné (et non plus une valeur unique), gamme qui intégrerait à la fois la variabilité temporelle et spatiale.

En complément, une moindre hétérogénéité de la stabilité structurale a été constatée au sein d'une même parcelle agricole. Ce résultat facilite la paramétrisation des modèles d'érosion.

Enfin, lorsque l'érodibilité du sol est estimée par la stabilité structurale, les mesures sont généralement réalisées dans l'horizon labouré. Ainsi, la valeur d'érodibilité attribuée à un sol est celle mesurée dans en sub-surface et non en surface. C'est pourtant bien la surface du sol qui est soumise aux agents érosifs. Plusieurs résultats présentés dans cette thèse montrent que la stabilité structurale peut être très différente entre la surface et la sub-surface. Aussi, en estimant l'érodibilité par des mesures réalisées dans l'horizon labouré, on tend à biaiser les résultats, et ainsi attribuer au matériau subissant l'érosion une érodibilité qui ne lui correspond pas forcément. De ce fait, et pour éviter ce type de biais, lorsque l'érodibilité est estimée par des mesures de stabilité structurales, ces mesures doivent être réalisées sur le matériau de surface.

2. Hors amendements organiques, les facteurs contrôlant les variations temporelles de la stabilité structurale sont essentiellement liés à la teneur en eau du sol.

Nous avons identifié des variations fortes de la stabilité structurale à pas de temps court. Le suivi de terrain présenté dans le chapitre 4 a également permis d'identifier les facteurs contrôlant ces variations.

2.1. Les variations à pas de temps court de la stabilité structurale de la surface du sol sont influencées par la dynamique de la teneur en eau

Les résultats présentés dans le chapitre 4 montrent que, pour les conditions étudiées (sol nu, pas de stimulation de l'activité microbienne par des apports), les variations de la stabilité structurale de la surface du sol à pas de temps court étaient essentiellement influencées par la dynamique de la teneur en eau du sol. La teneur en eau du sol au prélèvement, l'histoire hydrique et l'histoire des précipitations sont apparus comme des facteurs dominants des variations de stabilité structurale observées pour la surface. Un modèle de régression incluant les indices de l'histoire hydrique du sol a permis de prédire jusqu'à 60% des variations de la stabilité structurale observées. En revanche, les variables liées à l'activité biologique n'ont pas permis d'expliquer les variations de stabilité, ni d'améliorer les prédictions des modèles testés. Ce résultat souligne le caractère dominant des facteurs abiotiques dans les variations de stabilité structurales à pas de temps court lorsque l'activité biologique n'est pas stimulée. Ces facteurs sont impliqués dans différents processus physico-chimiques liés aux cycles d'humidité. Ces processus physico-chimiques, présentés dans le chapitre 2, nécessitent d'être mieux connus afin de permettre de mieux prédire la stabilité structurale.

2.2. Les variations à pas de temps court de la stabilité structurale de la sub-surface ne sont pas expliquées

Si la sub-surface du sol présentait de fortes variations de stabilité structurale, aucune des variables étudiées n'a permis d'expliquer ces variations. L'identification des facteurs contrôlant les variations de stabilité de ce matériau nécessite des études dédiées.

3. Les facteurs abiotiques contrôlent la stabilité structurale au travers de processus physico-chimiques

Les facteurs de variation de la stabilité structurale à pas de temps court sont des variables abiotiques liées à la teneur en eau du sol. Ces facteurs sont impliqués dans des processus physico-chimiques.

3.1. Les processus physico-chimiques de variation de la stabilité structurale sont liés à la dynamique de l'eau

La synthèse bibliographique présentée dans le chapitre 2 a permis de dresser un inventaire exhaustif des processus physico-chimiques cités dans la littérature comme étant à l'origine de variations de stabilité structurale. Ces processus agissent à l'échelle du macro-agrégat et à l'échelle du micro-agrégat de façon très diversifiées. Cette synthèse souligne la dynamique de la teneur en eau du sol comme étant le facteur commun de ces processus, notamment à travers les cycles d'humidité.

Les résultats présentés dans les chapitres 5 et 6 ont montré que l'intensité de l'humectation et de la dessiccation jouait un rôle majeur dans les variations de stabilité structurale liées aux cycles d'humidité. Les deux types de cycles d'humidité testés dans le chapitre 6 ont montré des variations contrastées pour des agrégats issus d'un même type de sol et présentant une stabilité structurale initiale identique. Un cycle d'humidité composé d'une humectation rapide et d'une dessiccation lente a permis de diminuer la stabilité de plus de 40%. Trois cycles d'humidité successifs, composés d'une humectation lente et d'un séchage rapide ont permis d'augmenter la stabilité structurale de 27%. Le protocole expérimentale présenté dans le chapitre 5 utilisait des cycles d'humectation d'amplitude faible (variation entre état saturé et -55kPa) composés d'une humectation et d'une dessiccation lente

(environ 3 h pour l'humectation et 5 jours pour la dessiccation). De tels cycles d'humidité n'ont pas fait varier la stabilité structurale. Ce résultat confirme une tendance qui avait déjà été observée par de précédentes études (e.g. Tisdall et al., 1978; Utomo & Dexter, 1982; Barzegar et al., 1995; Cosentino et al., 2006) : l'influence des cycles d'humidité sur la stabilité structurale est essentiellement contrôlée par l'intensité des phases d'humectation et de dessiccation.

3.2. Un état des connaissances variable selon les processus

La synthèse bibliographique présentée dans le chapitre 2 a montré que l'état des connaissances concernant les processus physico-chimiques de variation de la stabilité structurale présentait des statuts variables.

A l'échelle du macro-agrégat, les processus identifiés (éclatement, impact des gouttes de pluies, gel-dégel, gonflement différentiel des argiles) ont été observés et leurs conditions d'occurrence sont largement connues. A l'échelle du micro-agrégat, certains processus sont bien maîtrisés (dispersion/floculation des argiles) mais d'autres nécessitent de nouvelles études pour valider leur occurrence et leur implication dans les variations de stabilité structurale. C'est le cas par exemple du réarrangement particulaire intra-agrégat, de la cristallisation/dissolution en climat tempéré ou encore du « durcissement thixotropique ». Cette synthèse a pointé le réarrangement particulaire intra-agrégat comme étant le plus important à vérifier étant donné le nombre de citations dont il fait l'objet. Ce processus repose sur des bases théoriques fondées, mais n'a jamais été directement observé. Son occurrence et son implication dans les variations de stabilité structurale en lien avec les cycles d'humidité demandaient donc à être vérifiées. Deux études expérimentales en laboratoire ont été réalisées dans le cadre de cette thèse.

3.3. La structure interne des agrégats est affectée pas les cycles d'humectation-dessiccation

Les résultats présentés dans le chapitre 5 attestent d'un réarrangement de la structure interne d'un massif d'agrégats à l'échelle millimétrique (surface du capteur de pression). Avec le nombre de cycles, l'amplitude des déformations tend à diminuer, et le massif d'agrégat tend globalement à se contracter. Dans le même temps, la pression interne entre les particules tend à augmenter. Ces résultats sont concordants et correspondent à un rapprochement global des particules de l'échantillon, attestant ainsi d'un réarrangement de la structure interne du massif d'agrégats avec les cycles d'humidité. Concernant cette même étude, pour des variations d'humidité entre un état saturé et -55kPa, la déformation verticale globale du massif d'agrégats a été comprise entre 3% et 5%, tandis que la pression interne mesurée par les micro-capteurs a varié entre 50 hPa à 170 hPa. De telles variations ont entraîné des déplacements locaux de particules et donc un réarrangement de la structure interne du matériau. Ces déplacements sont contrôlés par les variations de tension dans les ménisques à l'interface des particules.

Les résultats de l'expérimentation présentés dans le chapitre 6 ont également permis d'observer des variations de la structure interne entre les agrégats soumis aux différents traitements. Cette étude a permis de caractériser la structure interne des agrégats à une échelle beaucoup plus fine (quelques microns). Le traitement composé d'une humectation rapide et d'une dessiccation lente (T+) a engendré la diminution du volume de pore de 10% et la diminution de la connectivité de la matrice solide de 15%. En revanche, le traitement composé d'une humectation rapide et d'une dessiccation lente (T-) n'a pas engendré de variations dans la microstructure de l'agrégat.

3.4. Les expérimentations menées n'ont pas permis de relier les variations de structure à la stabilité structurale

Si des variations de microstructure ont été observées ou fortement suggérées par les résultats de ces deux études, en revanche, leur relation avec la stabilité structurale n'a pas pu être clairement établie. Pour l'expérimentation présentée dans le chapitre 5, les résultats de pression interne et de déformation du massif ont fortement suggéré un réarrangement de la structure qui aurait dû *a priori* engendrer une augmentation de la stabilité structurale. Cependant la stabilité structurale de l'échantillon est restée constante. On peut expliquer cette constance par une amplitude et des intensités faibles dans les cycles d'humidité. Des cycles plus amples avec des phases d'humectation et de dessiccation plus intenses auraient vraisemblablement permis de faire varier la stabilité structurale. Pour l'expérimentation présentée dans le chapitre 6, si le traitement T+ a permis d'augmenter la stabilité structurale, les variations de microstructure identifiées n'étaient pas totalement cohérentes et, de ce fait, difficiles à interpréter. Ces résultats n'ont pas permis de relier l'augmentation de stabilité observée au processus de réarrangement particulaire théorisé par Kemper & Rosenau (1984 ; 1986). Parallèlement, la diminution de stabilité observée lors du traitement T- n'a pas pu être reliée à des variations de la microstructure interne de l'agrégat. Si une augmentation faible (environ 5%) du volume poral a été observée, la connectivité de la phase solide est restée la même, alors qu'une diminution aurait été attendue. Ainsi, au vue de ce dernier résultat, nous pouvons penser que le réarrangement particulaire comme théorisé par Kemper & Rosenau (1984 ; 1986) n'intervient pas dans la diminution de la stabilité liées à des cycles d'humidité. D'autres processus impliquant des déplacements plus importants de particules, comme la dispersion des ciments argileux, ou actifs à des échelles plus grandes, comme le gonflement différentiel des argiles peuvent être suggérés dans ce cas. Les deux expérimentations n'ont

donc pas pu clairement valider l'occurrence du réarrangement particulaire comme processus physico-chimique de variation de la stabilité structurale lors des cycles d'humidité.

Perspectives

Les différentes études réalisées à travers cette thèse ont permis de progresser dans la compréhension de la stabilité structurale, de sa dynamique, des facteurs et des processus qui en sont à l'origine. Les résultats obtenus à travers ces études nous conduisent à proposer les perspectives suivantes pour de futures recherches.

Les variations de stabilité structurale étudiées lors des études de terrain ont été observées pour des types de sols limoneux ou limono-argileux. D'autres types de sols (plus argileux) pourraient présenter des variabilités temporelles et spatiales différentes. Il en va de même pour les facteurs qui contrôlent la variation de la stabilité structurale. Aussi, la réalisation d'un suivi de terrain à pas de temps court sur un autre type de sol constituerait un complément important dans l'objectif d'une meilleure paramétrisation de l'érodibilité par les modèles.

L'étude de terrain présentée dans le chapitre 1 suggère d'étudier l'influence des processus de formation de croûte de battance sur la stabilité structurale des croûtes elles-mêmes. En effet, si l'impact de la stabilité structurale sur la formation des croûtes de battance a été largement étudié (e.g. Hairsine & Hook, 1994 ; Kuhn et al., 2003), la relation inverse reste méconnue. Les résultats présentés dans les chapitre 1 et 3 montrent des stabilité différentes entre surface encroutée et sub-surface. Ces différences doivent être expliquées.

Si l'histoire hydrique est apparue comme un facteur dominant dans les variations de la stabilité structurale à la surface du sol, la grande inconnue qui résulte du suivi de terrain reste les facteurs et processus qui contrôlent la stabilité structurale de la sub-surface. D'autres études sont à réaliser pour éclaircir les causes de ces variations.

La synthèse bibliographique a mis en avant des processus physico-chimiques incriminés dans les variations de la stabilité structurale mais méconnus. C'est le cas notamment de la dissolution/cristallisation en climat tempéré pour des sols non carbonatés, ou encore le cas de la thixotropie impliqué dans le durcissement par vieillissement (« thixotropic age hardening »). Si ces processus ont été identifiés comme nécessitant des études complémentaires approfondies, de telles études n'ont pas pu être réalisées dans le cadre de la thèse, et reste à concevoir.

Les expérimentations présentées dans la troisième partie ont permis de mettre en place des protocoles solides dans l'objectif de caractériser les processus physico-chimiques de variation de la stabilité structurale en relation avec les cycles d'humidité. Ces protocoles offrent des perspectives intéressantes pour la réalisation d'expérimentations futures.

L'expérimentation présentée dans le chapitre 5 doit être reproduite en utilisant des cycles d'humidité plus amples et des phases de séchage plus intenses afin de permettre d'observer des variations de stabilité structurale et de les relier aux variations de pression interne. La mise en place de tels cycles d'humidité a pour l'instant été limitée par les gammes de mesure des micro-tensiomètres.

Enfin, la démarche expérimentale présentée dans le chapitre 6 pourrait être reproduite avec des mesures de tomographie réalisées sur un même agrégat. En effet, dans l'état présent du protocole, les mesures de tomographies sont réalisées sur des agrégats différents soumis à différents traitements. De telles mesures ne permettent pas de différencier la part de variabilité provenant des disparités entre les agrégats de celle provenant des traitements. Aussi, la réalisation des traitements et des mesures sur un seul et même agrégat limiterait ce biais et permettrait une étude optimale des changements de la micro-structure des agrégats.

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Baptiste ALGAYER

Variabilité à court terme de la stabilité structurale des sols et processus physico chimiques associés

Résumé :

La stabilité structurale du sol peut être utilisée pour estimer l'érodibilité du sol, paramètre clef des modèles d'érosion. Afin d'améliorer les connaissances sur les facteurs et processus contrôlant la stabilité structurale, cette thèse avait pour objectif d'étudier la variabilité de la stabilité structurale à pas de temps court (quelques jours) ainsi que les processus physico-chimiques qui en sont à l'origine. Des études de terrain ont permis de caractériser une dynamique rapide de la stabilité structurale pour un sol donné avec notamment : des différences importantes entre surface et sub-surface ; une forte variabilité selon la localisation du site (mais une faible variabilité pour un même site) ; et une forte variabilité à pas de temps court liée à la teneur en eau du sol. Les variations de stabilité structurale à pas de temps court apparaissent majoritairement contrôlées par des processus physico-chimiques fortement liés aux cycles d'humidité. Une synthèse bibliographique a montré que l'occurrence de certains de ces processus nécessitait d'être vérifiée, notamment pour le réarrangement particulaire. Des expérimentations en laboratoire ont montré que la structure interne des agrégats était affectée pas les cycles d'humidité à l'échelle millimétrique comme micrométrique. Cependant, les réarrangements de particules observés n'ont pas pu être strictement reliés aux variations de stabilité structurale. Les résultats de ces expérimentations confirment le suivi de terrain : la teneur en eau du sol a un rôle majeur dans les variations de stabilité structurale à pas de temps court. Dans la plupart des cas, les différences de stabilité mesurées correspondaient à des érodibilité du sol contrastées. Les modèles d'érosion devraient donc inclure cette variabilité pour améliorer leurs prédictions.

Mots clés : sol, stabilité structurale, érodibilité, agrégation, réarrangement particulaire, teneur en eau

Short time step variability of soil aggregate stability and physico-chemical processes involved

Abstract:

Soil aggregate stability can be used to estimate soil erodibility, a key parameter in erosion models. To improve the knowledge about the factors and processes controlling aggregate stability, this PhD thesis aimed at analyzing the variability of soil aggregate stability at short time step (a few days) and the physico-chemical processes controlling this variability. Field studies allowed characterizing a quick dynamic of aggregate stability for a given soil. Results showed: large differences between surface and sub-surface, a large variability among sites (but a low variability within a given site); and a large variability at short time step in relation with soil water content. Aggregate stability changes at short time step are mainly controlled by physico-chemical processes closely related to wetting-and-drying cycles. A literature review showed that the occurrence of some of these processes needed to be validated; especially for the particle rearrangement. Laboratory experiments showed that the internal structure of aggregates was affected by wetting-and-drying cycles at the millimeter and micrometer scales. However, the observed particle rearrangements could not be strictly related to aggregate stability changes. The results of these experiments confirm the field monitoring: soil water content plays a major role in the changes of aggregate stability at short time step. In most of the cases, the measured differences in aggregate stability corresponded to contrasted erodibilities. Hence, erosion models should include this variability to improve their predictions.

Keywords: soil, aggregate stability, erodibility, aggregation, particle rearrangement, water content



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