

# Couches polycristallines orientées d'aragonite biomimétique, synthétisées par voie électrochimique

Soutenance de thèse de Doctorat de  
l'Université de Caen

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École doctorale SIMEM

29 Septembre 2009

Daniel Chateigner

- Directeurs de thèse -

Otávio Gil

Contexte  
Dépôts Minéraux  
Dépôts Minéraux-Organiques  
Conclusions et Perspectives



## Contexte

- ▶ La Nacre naturelle
- ▶ La Nacre synthétique
- ▶ Les Techniques de synthèses et de caractérisation
- ▶ Exemples



La Nacre naturelle est composée de Carbonate de Calcium

# Le Carbonate de Calcium

3 formes allotropiques anhydres:

- ▶ Calcite:  $R\bar{3}c$  (rhomboédrique)  
forme stable
- ▶ Vaterite:  $P\frac{6_3}{m}mc$  (hexagonal)  
forme métastable
- ▶ Aragonite:  $Pm\bar{c}n$  (orthorhombique)  
forme métastable:  
 $\Delta G_{Cal \Rightarrow Ara}^\circ = 1 \text{ kJ} \cdot \text{mol}^{-1}$



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# Aragonite



Aragonite:  $Pm\bar{c}n$   
(orthorhombique)

forme métastable:

$$\Delta G_{Cal \Rightarrow Ara}^{\circ} = 1 \text{ kJ.mol}^{-1}$$

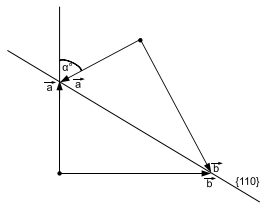


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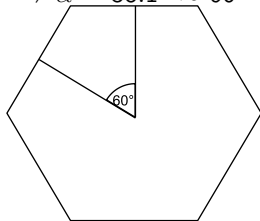


Aragonite:  $Pm\bar{c}n$   
(orthorhombique)  
forme métastable:

$$\Delta G_{CaI \Rightarrow Ara}^{\circ} = 1 \text{ kJ.mol}^{-1}$$



$$a = 4.96 \text{ \AA}, b = 7.96 \text{ \AA}$$
$$\Rightarrow \alpha = 58.1^{\circ} \approx 60^{\circ}$$



# Aragonite



Aragonite:  $Pm\bar{c}n$   
(orthorhombique)  
forme métastable:

$$\Delta G_{Cal \Rightarrow Ara}^{\circ} = 1 \text{ kJ} \cdot \text{mol}^{-1}$$

$$Z_{C-O_1} = (z_C - z_{O_1}) * c^1$$

▶  $Z_{C-O_1_{Calcite}} = 0$

▶  $Z_{C-O_1_{Aragonite}} = 0.0574 \text{ \AA}$

▶  $Z_{C-O_1} \Rightarrow$  influence  
organique

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<sup>1</sup>Pokroy, B. *et al* (2007). Atomic Structure of Biogenic Aragonite. *Chemistry of materials*, 19(13):3244–3251.

# Structure de Mollusques

- ▶ Couche prismatique (externe): Calcite
- ▶ Couche nacréée (interne): Aragonite

*Haliotis tuberculata tuberculata*

Gastéropode



*Pinctada maxima*

Bivalve



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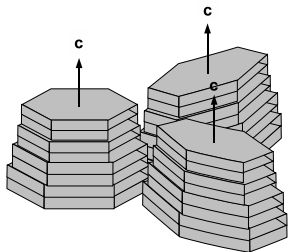
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Bivalve



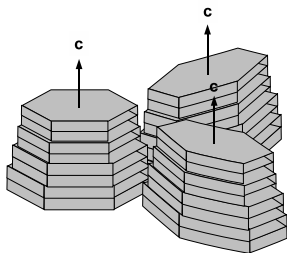
# Micro-structure de la Nacre

Nacre colonnaire:  
*Haliotis tuberculata*

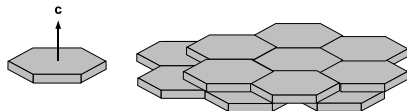


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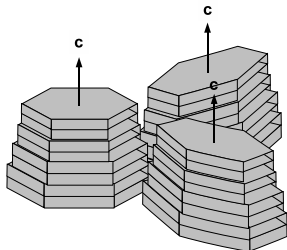


Nacre mur de briques:  
*Pinctada maxima*

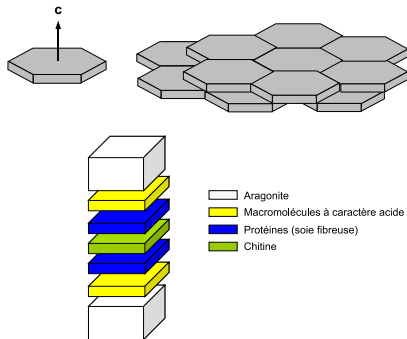


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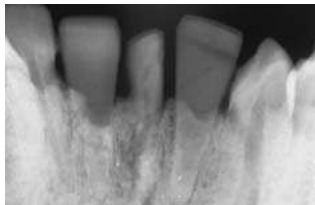
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# Problématique

Bobbio A. (1972): Prothèses dentaires datant de l'époque Maya<sup>1</sup>  
Biocompatibilité

Lopez E. (1992): Ostéoinduction importante de la nacre de *Pinctada maxima* in-vitro<sup>2</sup> et in-vivo



Loi européenne ⇒ La nacre synthétique:

- ▶ Massifs en nacre
- ▶ Recouvrement de massifs en titane: dépôt par voie électrochimique

Objectifs:

Réalisation de dépôts d'aragonite biomimétique  
Influence de molécules organiques

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<sup>1</sup>Bobbio, A. (1972). The first endosseous alloplastic implant in the history of man. *Bulletin of Historical Dentology*, 20(1):1–6.

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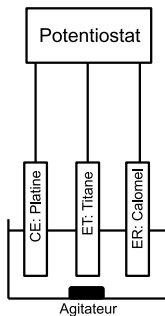
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# Montage Électrochimique

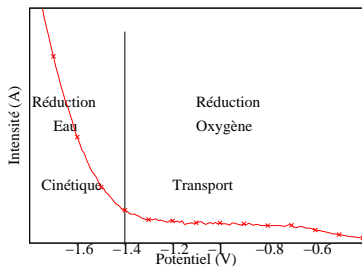
- ▶ Montage classique à 3 électrodes.
- ▶ Électrode de travail: plaque de titane grade 2 ( $2\text{cm}^2$ ).
- ▶ Électrolyte d'eau de mer synthétique (norme *ASTM – D1141*).

Sels	Concentrations ( $\text{mol.L}^{-1}$ )
<i>NaCl</i>	0.42
<i>MgCl<sub>2</sub>, 6H<sub>2</sub>O</i>	0.0546
<i>Na<sub>2</sub>SO<sub>4</sub></i>	0.0288
<i>CaCl<sub>2</sub>, 2H<sub>2</sub>O</i>	0.0105
<i>KCl</i>	0.0093
<i>NaHCO<sub>3</sub></i>	0.0028

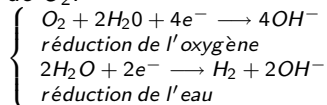


# Dépôt par Voie Électrochimique

Courbe Intensité-Potentiel

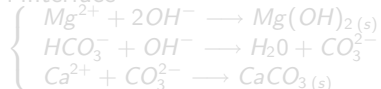


Réactions cathodiques de  $H_2O$  et de  $O_2$ :



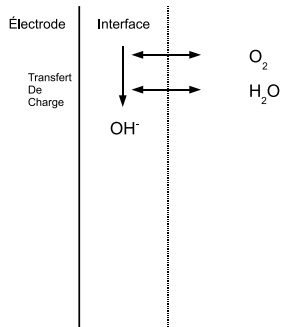
$pH_{interfacial}$  basique

Réactions chimiques: précipitation à l'interface<sup>1</sup>

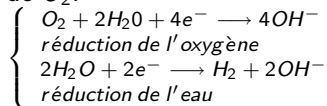


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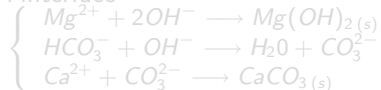


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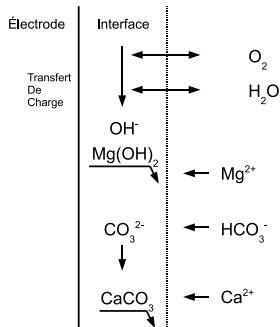
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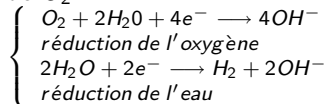


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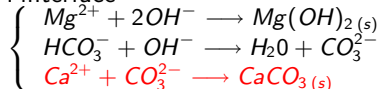


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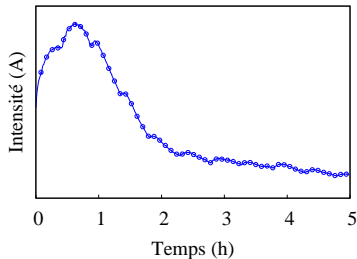
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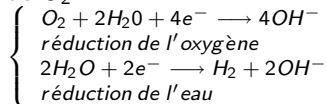
<sup>1</sup>Roche, M. et al (1990). *Chimie des solutions*

# Dépôt par Voie Électrochimique

Courbe Chronoampérométrique

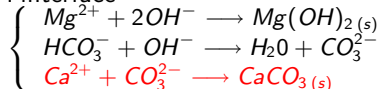


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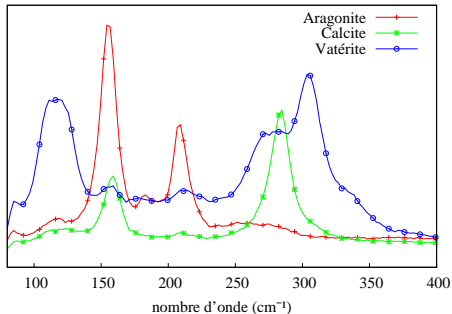


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# Caractérisation des dépôts

- ▶ Influence des paramètres expérimentaux
- ▶ Influence des molécules organiques (structure cristalline)

## $\mu$ -Spectroscopie RAMAN



## Imagerie MEB



# Analyse Combinée

- ▶ 936 diagrammes

- ▶ Affinement *Rietveld*:

$$y_{ic} = y_{ib} + \sum_{\Phi=1}^{N_{\Phi}} S_{\Phi} \sum_{k=K_1}^K j_{\Phi k} L_{p_{\Phi k}} P_{\Phi k} |F_{\Phi k}^2| \Omega_{i_{\Phi k}} A$$

- ▶  $y_{ib}$  bruit de fond
- ▶  $S_{\Phi}$  facteur d'échelle en volume de chaque phase  $\Phi$
- ▶  $j_{\Phi k}$  facteur de multiplicité de chaque pic pour une phase  $\Phi$
- ▶  $L_{p_{\Phi k}}$  facteur de Lorentz et/ou de polarisation
- ▶  $P_{\Phi k}$  facteur d'orientation préférentielle  $\Rightarrow$  ODF  $\Rightarrow$  figures de pôles
- ▶  $F_{\Phi k}^2$  facteur de structure  $\Rightarrow$  positions atomiques  $\Rightarrow Z_{C-O_1}$
- ▶  $\Omega_{i_{\Phi k}}$  fonction de profil  $\Rightarrow$  taille des cristallites
- ▶  $A$  facteur d'absorption  $\Rightarrow$  épaisseur

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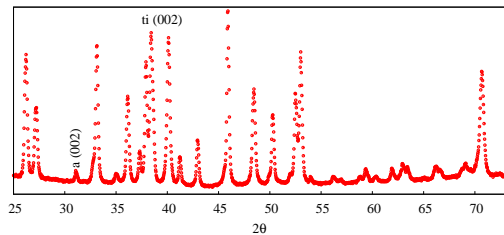
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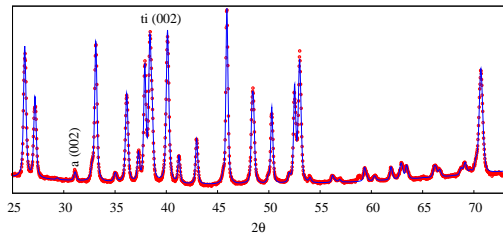
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# Exemple d'Affinement

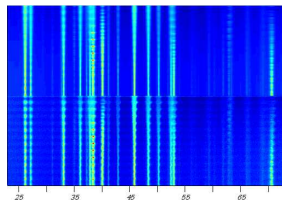
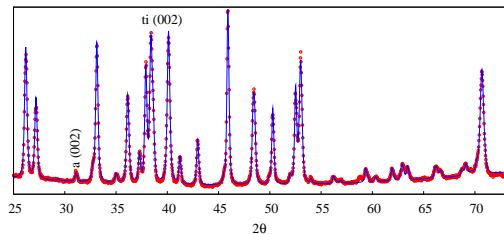


# Exemple d'Affinement



Affinement Rietveld:

# Exemple d'Affinement



Affinement Rietveld:

$$R_B(\%) = 16.4$$

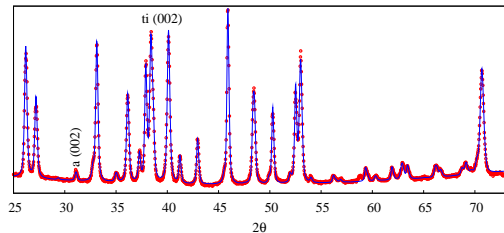
$$R_{WP}(\%) = 21.8$$

$$R_{Exp}(\%) = 9.8$$

$$\chi^2 = 2.21$$



# Exemple d'Affinement



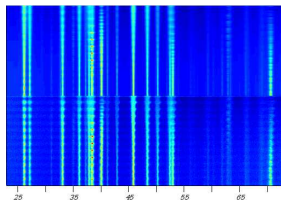
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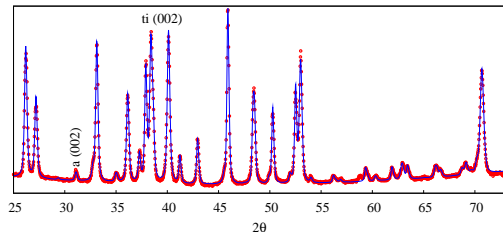


Affinement OD:

$$R_w(\%)=20.9$$

$$R_b(\%)=20.1$$

# Exemple d'Affinement



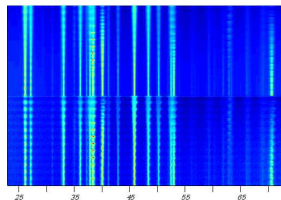
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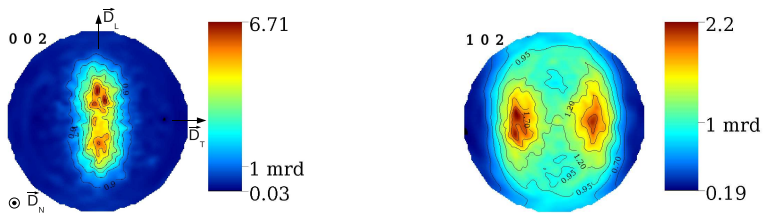
$$R_w(\%)=20.9$$

$$R_b(\%)=20.1$$

$$F^2=2.0\text{mrd}^2$$

$$ODF_{max}=33.4\text{mrd}$$

## Exemple de Figure de Pôles

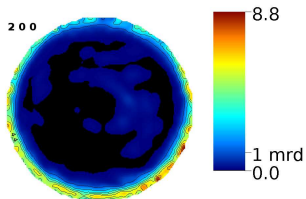
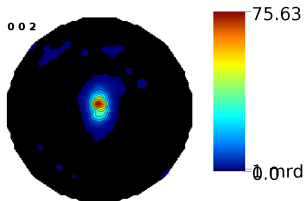


Titane laminé (hc):

- ▶ Plan de glissement et de maclage: (102)
- ▶ Dédoublément (002) et Renforcement direction de laminage

# Exemple de Texture de Nacres

## *Haliotis tuberculata*



texture de fibre

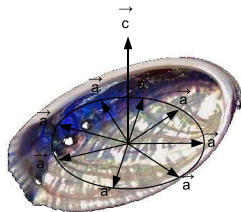
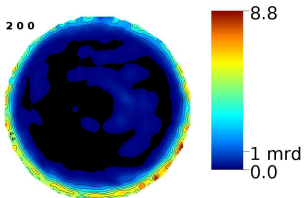
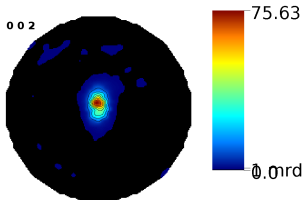
$$F^2 = 211.05 \text{mrd}^2$$

$$ODF_{max} = 1498.1 \text{mrd}$$

$$Z_{C-O_1} = 0.0895 \text{ \AA}$$

# Exemple de Texture de Nacres

## *Haliotis tuberculata*



texture de fibre

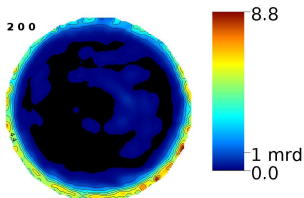
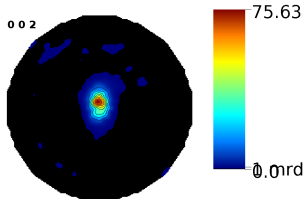
$$F^2 = 211.05 \text{ mrd}^2$$

$$ODF_{max} = 1498.1 \text{ mrd}$$

$$Z_{C-O_1} = 0.0895 \text{ \AA}$$

## Exemple de Texture de Nacres

*Haliotis tuberculata*



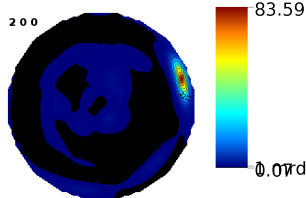
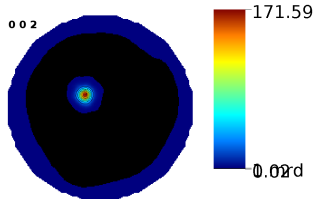
texture de fibre

$$F^2 = 211.05 \text{ mrd}^2$$

$$ODF_{max} = 1498.1 \text{ mrd}$$

$$Z_{C-O_1} = 0.0895 \text{ \AA}$$

*Pinctada maxima*



symétrie pseudo-hexagonale

$$F^2 = 625.66 \text{ mrd}^2$$

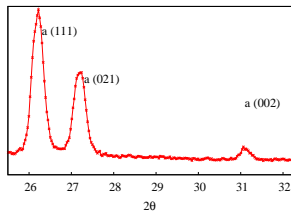
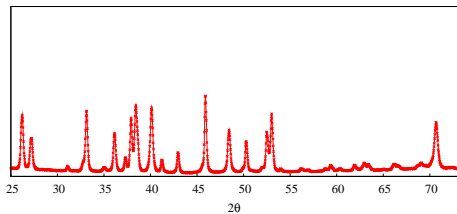
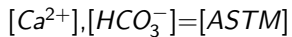
$$ODF_{max} = 1907.1 \text{ mrd}$$

$$Z_{C-O_1} = 0.0455 \text{ \AA}$$

## Dépôts Minéraux d'Aragonite

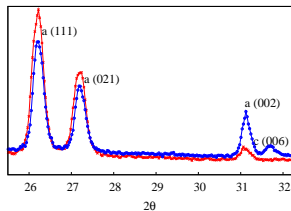
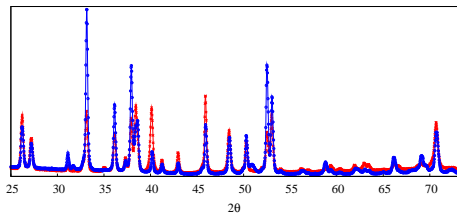
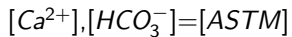
- ▶ Influence des paramètres expérimentaux
  - ▶ Concentration
  - ▶ Potentiel
  - ▶ Température
  - ▶ Agitation
- ▶ Mode et Stade de croissance

# Choix de l'Électrolyte

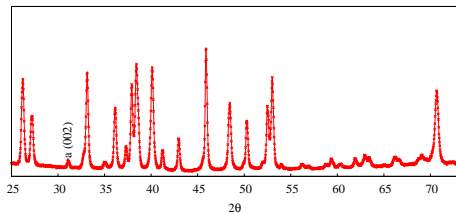
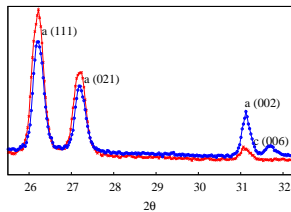
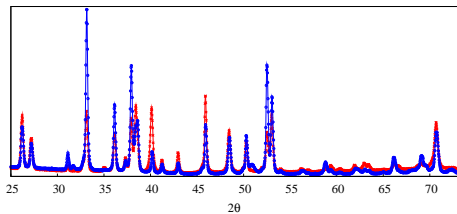
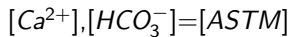




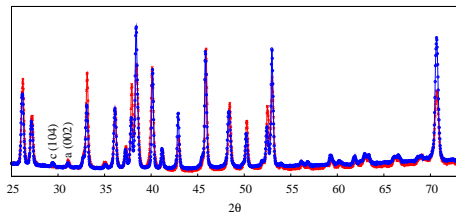
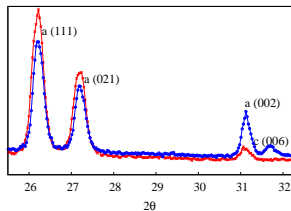
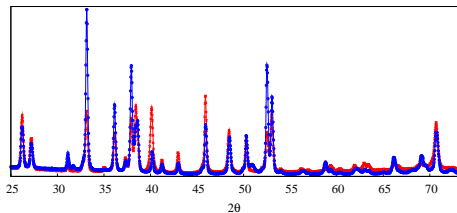
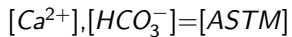
# Choix de l'Électrolyte



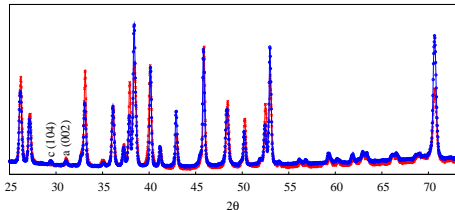
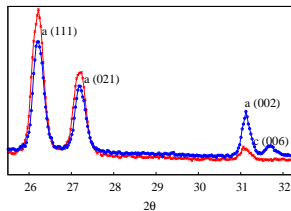
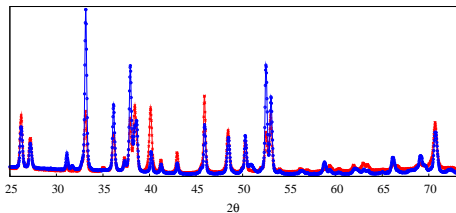
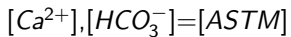
# Choix de l'Électrolyte



# Choix de l'Électrolyte

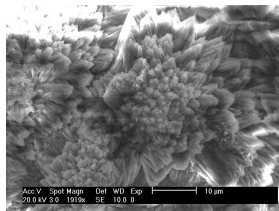
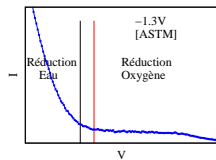


# Choix de l'Électrolyte



Electrolyte = [ASTM]

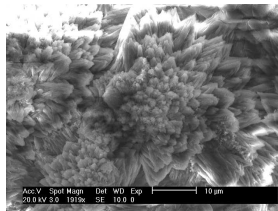
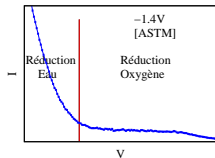
# Influence du Potentiel



Potentiel	Épaisseur (Å)
-1.3V	949(1)
-1.4V	12601(8)
-1.6V	7779(1)

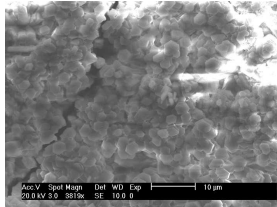
Potentiel = -1.4V

# Influence du Potentiel

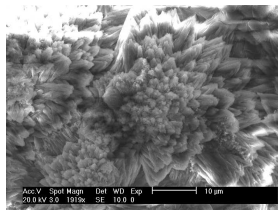
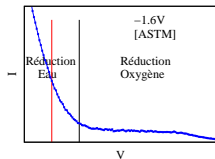


Potentiel	Épaisseur (Å)
-1.3V	949(1)
-1.4V	12601(8)
-1.6V	7779(1)

Potentiel = -1.4V

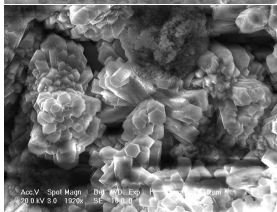
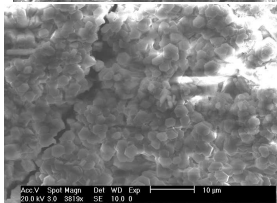


# Influence du Potentiel

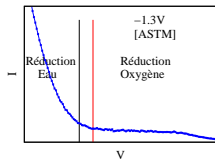


Potentiel	Épaisseur (Å)
-1.3V	949(1)
-1.4V	12601(8)
-1.6V	7779(1)

Potentiel = -1.4V

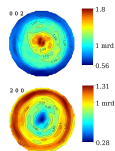
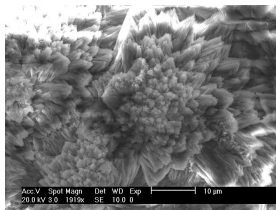


# Influence du Potentiel



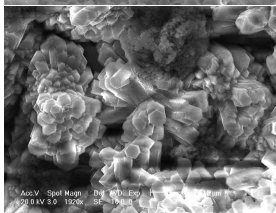
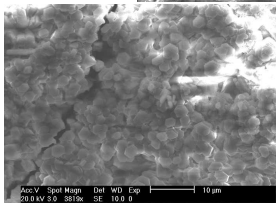
$$F^2 = 1.14 \text{ mrd}^2$$

$$ODF_{max} = 6.5 \text{ mrd}$$



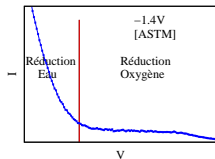
Potentiel	Épaisseur (Å)
-1.3V	949(1)
-1.4V	12601(8)
-1.6V	7779(1)

Potentiel = -1.4V





# Influence du Potentiel

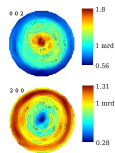
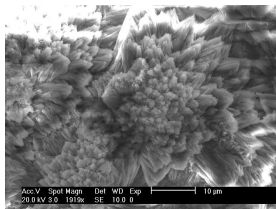


Potentiel	Épaisseur (Å)
-1.3V	949(1)
-1.4V	12601(8)
-1.6V	7779(1)

Potentiel = -1.4V

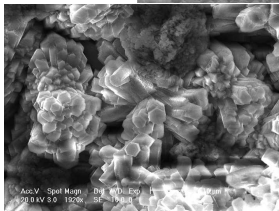
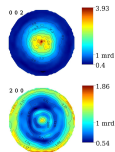
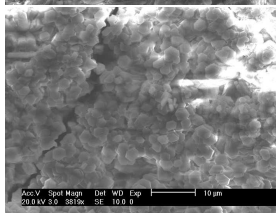
$$F^2 = 1.14 \text{ mrd}^2$$

$$ODF_{max} = 6.5 \text{ mrd}$$

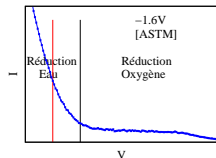


$$F^2 = 2.0 \text{ mrd}^2$$

$$ODF_{max} = 33.4 \text{ mrd}$$



# Influence du Potentiel

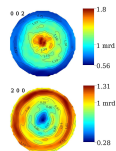
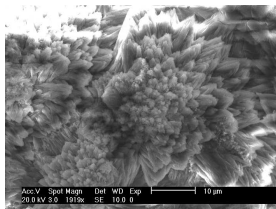


Potentiel	Épaisseur (Å)
-1.3V	949(1)
-1.4V	12601(8)
-1.6V	7779(1)

Potentiel = -1.4V

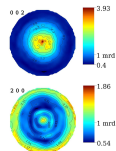
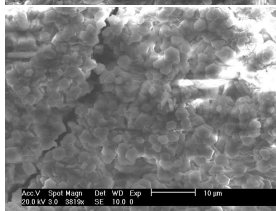
$$F^2 = 1.14 \text{ mrd}^2$$

$$ODF_{max} = 6.5 \text{ mrd}$$



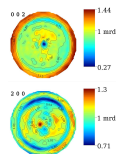
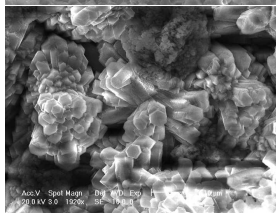
$$F^2 = 2.0 \text{ mrd}^2$$

$$ODF_{max} = 33.4 \text{ mrd}$$

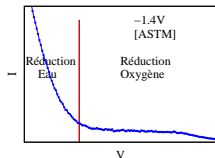


$$F^2 = 1.23 \text{ mrd}^2$$

$$ODF_{max} = 9.8 \text{ mrd}$$



# Influence du Potentiel

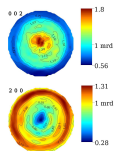
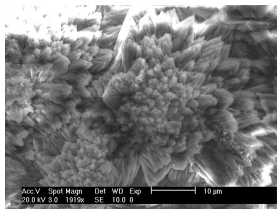


Potentiel	Épaisseur (Å)
-1.3V	949(1)
-1.4V	12601(8)
-1.6V	7779(1)

Potentiel =  $-1.4V$

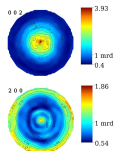
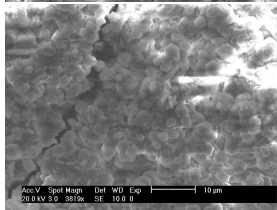
$$F^2 = 1.14 \text{ mrd}^2$$

$$ODF_{max} = 6.5 \text{ mrd}$$



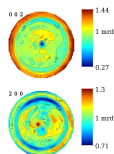
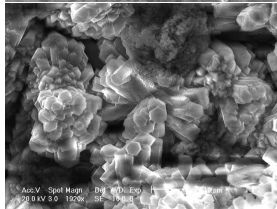
$$F^2 = 2.0 \text{ mrd}^2$$

$$ODF_{max} = 33.4 \text{ mrd}$$

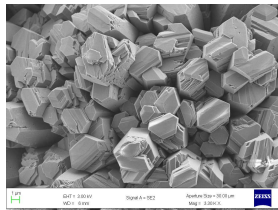
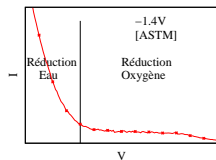


$$F^2 = 1.23 \text{ mrd}^2$$

$$ODF_{max} = 9.8 \text{ mrd}$$



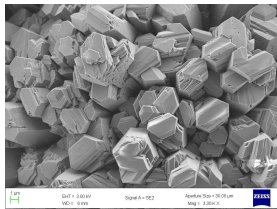
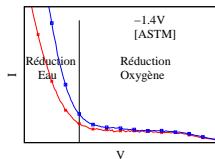
# Influence de la Température



Température	Épaisseur (Å)
40° C	12601(8)
60° C	1171(1)

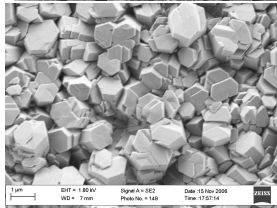
Température = 40° C

# Influence de la Température

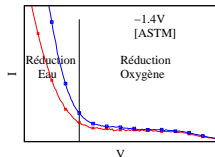


Température	Épaisseur (Å)
40° C	12601(8)
60° C	1171(1)

Température = 40° C

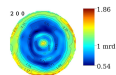
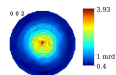
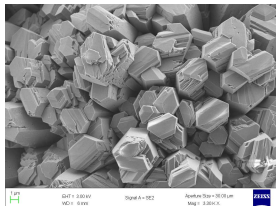


# Influence de la Température



$$F^2 = 2.0 \text{ mrd}^2$$

$$ODF_{max} = 33.4 \text{ mrd}$$

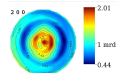
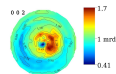
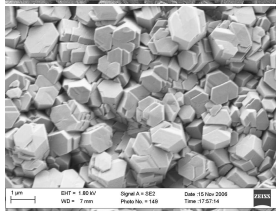


Température	Épaisseur (Å)
40° C	12601(8)
60° C	1171(1)

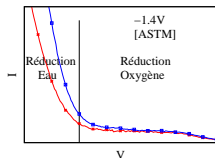
Température = 40° C

$$F^2 = 1.34 \text{ mrd}^2$$

$$ODF_{max} = 7.8 \text{ mrd}$$

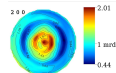
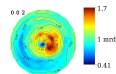
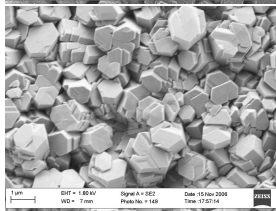
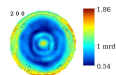
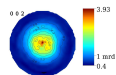
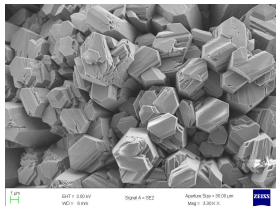


# Influence de la Température



$$F^2 = 2.0 \text{ mrd}^2$$

$$ODF_{max} = 33.4 \text{ mrd}$$



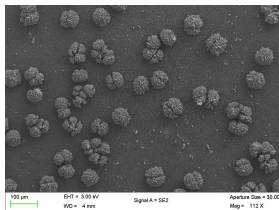
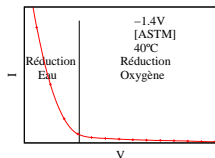
Température	Épaisseur (Å)
40° C	12601(8)
60° C	1171(1)

$$F^2 = 1.34 \text{ mrd}^2$$

$$ODF_{max} = 7.8 \text{ mrd}$$

Température = 40° C

# Influence de l'Agitation



Agitation	Épaisseur (Å)
faible	406(0.6)
moyenne	12601(8)
forte	25102(24)

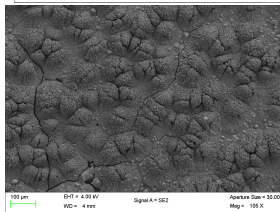
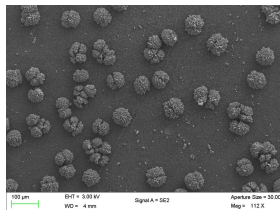
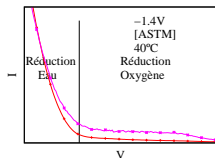
Agitation = moyenne

Taille des Cristallites:  
886.1(1) Å

$Z_{C-O_1} = 0.0868 \text{ Å}$



# Influence de l'Agitation



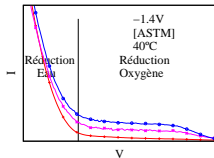
Agitation	Épaisseur (Å)
faible	406(0.6)
moyenne	12601(8)
forte	25102(24)

Agitation = moyenne

Taille des Cristallites:  
886.1(1) Å

$Z_{C-O_1} = 0.0868 \text{ Å}$

# Influence de l'Agitation

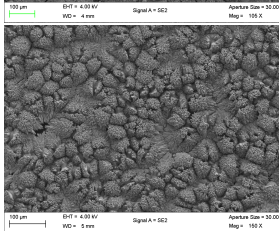
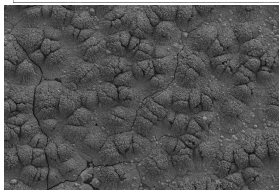
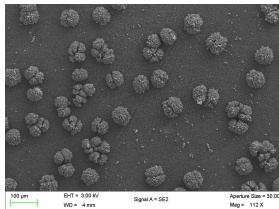


Agitation	Épaisseur (Å)
faible	406(0.6)
moyenne	12601(8)
forte	25102(24)

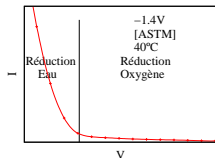
Agitation = moyenne

Taille des Cristallites:  
886.1(1) Å

$Z_{C-O_1} = 0.0868 \text{ Å}$

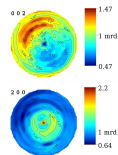
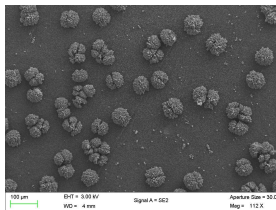


# Influence de l'Agitation



$$F^2 = 1.17 \text{mrd}^2$$

$$ODF_{max} = 3.5 \text{mrd}$$

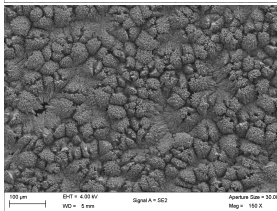
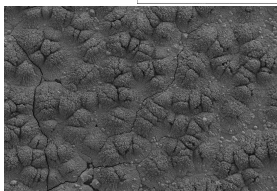


Agitation	Épaisseur (Å)
faible	406(0.6)
moyenne	12601(8)
forte	25102(24)

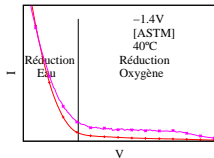
Agitation = moyenne

Taille des Cristallites:  
886.1(1) Å

$Z_{C-O_1} = 0.0868 \text{ Å}$

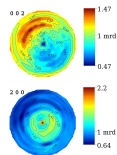
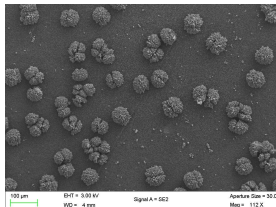


# Influence de l'Agitation



$$F^2 = 1.17 \text{ mrd}^2$$

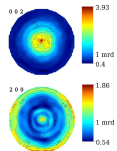
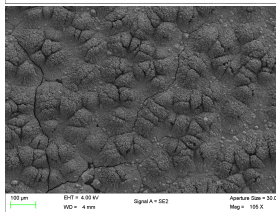
$$ODF_{max} = 3.5 \text{ mrd}$$



Agitation	Épaisseur (Å)
faible	406(0.6)
moyenne	12601(8)
forte	25102(24)

$$F^2 = 2.0 \text{ mrd}^2$$

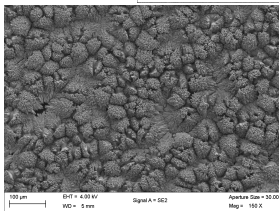
$$ODF_{max} = 33.4 \text{ mrd}$$



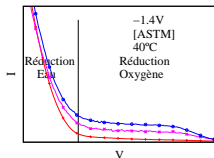
Agitation = moyenne

Taille des Cristallites:  
886.1(1) Å

$Z_{C-O_1} = 0.0868 \text{ Å}$



# Influence de l'Agitation



$$F^2 = 1.17 \text{ mrd}^2$$

$$ODF_{max} = 3.5 \text{ mrd}$$

Agitation	Épaisseur (Å)
faible	406(0.6)
moyenne	12601(8)
forte	25102(24)

Agitation = moyenne

Taille des Cristallites:  
886.1(1) Å

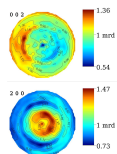
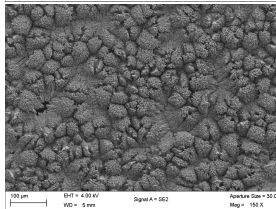
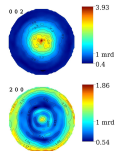
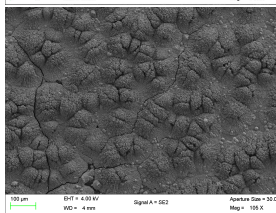
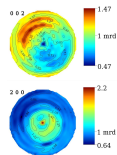
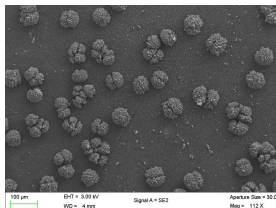
$Z_{C-O_1} = 0.0868 \text{ Å}$

$$F^2 = 2.0 \text{ mrd}^2$$

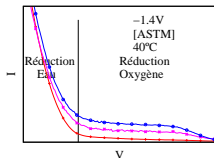
$$ODF_{max} = 33.4 \text{ mrd}$$

$$F^2 = 1.08 \text{ mrd}^2$$

$$ODF_{max} = 3.3 \text{ mrd}$$



# Influence de l'Agitation



$$F^2 = 1.17 \text{ mrd}^2$$

$$ODF_{max} = 3.5 \text{ mrd}$$

Agitation	Épaisseur (Å)
faible	406(0.6)
moyenne	12601(8)
forte	25102(24)

Agitation = moyenne

Taille des Cristallites:  
886.1(1) Å

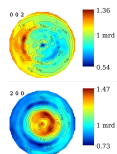
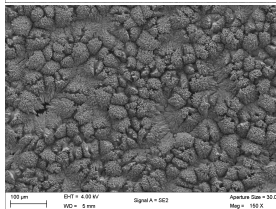
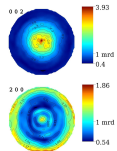
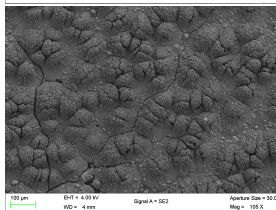
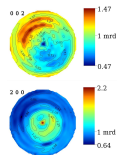
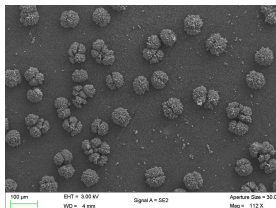
$$Z_{C-O_1} = 0.0868 \text{ Å}$$

$$F^2 = 2.0 \text{ mrd}^2$$

$$ODF_{max} = 33.4 \text{ mrd}$$

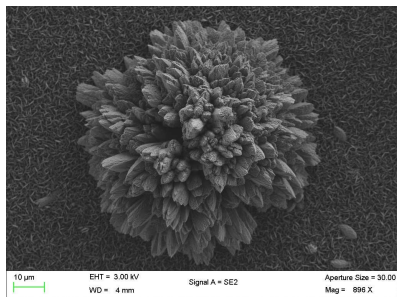
$$F^2 = 1.08 \text{ mrd}^2$$

$$ODF_{max} = 3.3 \text{ mrd}$$



# Bilan sur les Dépôts Minéraux

## Mode de Croissance:



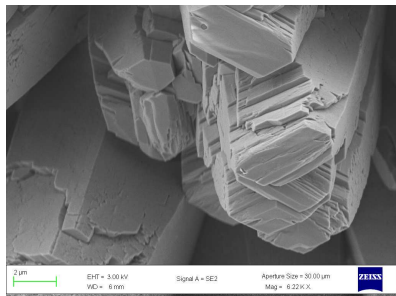
- ▶ Choux-fleurs composés de bâtonnets de section pseudo-hexagonale
- ▶ Texture de fibre
- ▶ Modèle de Voronoï proposé pour *Pinctada margaritifera*
- ▶ Croissance alternée en Couches liées par Maclage<sup>1</sup>

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<sup>1</sup>Mutvei H. (2009). Personal Communication

# Bilan sur les Dépôts Minéraux

## Mode de Croissance:



- ▶ Choux-fleurs composés de bâtonnets de section pseudo-hexagonale
- ▶ Texture de fibre
- ▶ Modèle de Voronoi proposé pour *Pinctada margaritifera*
- ▶ Croissance alternée en Couches liées par Maclage<sup>1</sup>

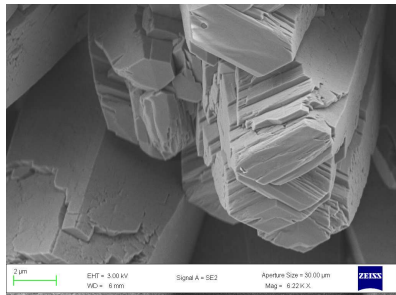
---

<sup>1</sup>Mutvei H. (2009). Personal Communication



# Bilan sur les Dépôts Minéraux

## Mode de Croissance:



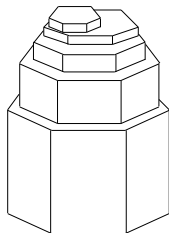
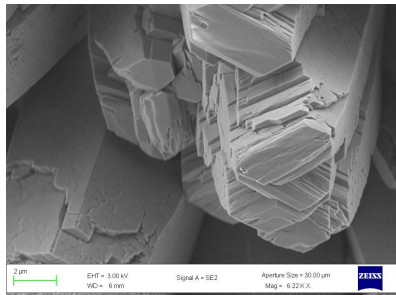
- ▶ Choux-fleurs composés de bâtonnets de section pseudo-hexagonale
- ▶ Texture de fibre
- ▶ Modèle de Voronoi proposé pour *Pinctada margaritifera*
- ▶ Croissance alternée en Couches liées par Maclage<sup>1</sup>

---

<sup>1</sup>Mutvei H. (2009). Personal Communication

# Bilan sur les Dépôts Minéraux

## Mode de Croissance:

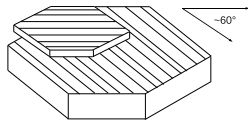
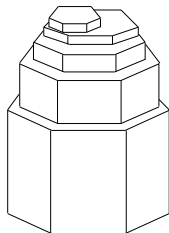
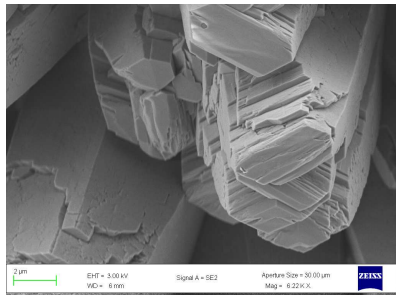


- ▶ Choux-fleurs composés de bâtonnets de section pseudo-hexagonale
- ▶ Texture de fibre
- ▶ Modèle de Voronoï proposé pour *Pinctada margaritifera*
- ▶ Croissance alternée en Couches liées par Maclage<sup>1</sup>

<sup>1</sup>Mutvei H. (2009). Personal Communication

# Bilan sur les Dépôts Minéraux

## Mode de Croissance:

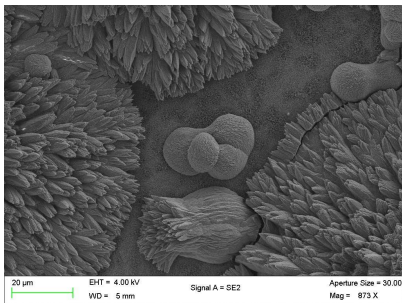


- ▶ Choux-fleurs composés de bâtonnets de section pseudo-hexagonale
- ▶ Texture de fibre
- ▶ Modèle de Voronoï proposé pour *Pinctada margaritifera*
- ▶ Croissance alternée en Couches liées par Maclage<sup>1</sup>

<sup>1</sup>Mutvei H. (2009). Personal Communication

# Bilan sur les Dépôts Minéraux

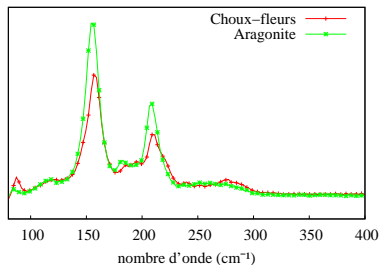
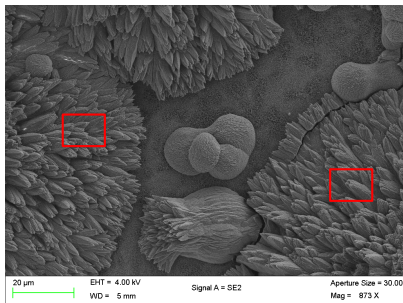
## Stade de Croissance:



- ▶ Choux-fleurs, à base de boule, composés d'Aragonite
- ▶ Boules (symétrie d'axe 3): calcite
- ▶ Transformation de la Calcite en Aragonite (plus stable dans les conditions expérimentales)

# Bilan sur les Dépôts Minéraux

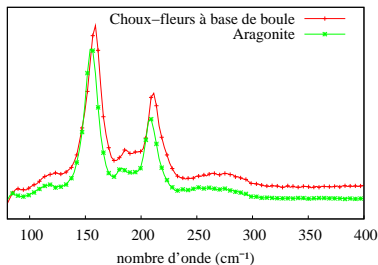
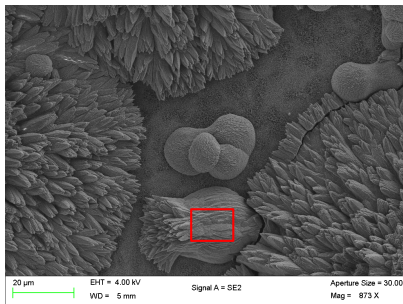
## Stade de Croissance:



- ▶ Choux-fleurs, à base de boules, composés d'Aragonite
- ▶ Boules (symétrie d'axe 3): calcite
- ▶ Transformation de la Calcite en Aragonite (plus stable dans les conditions expérimentales)

# Bilan sur les Dépôts Minéraux

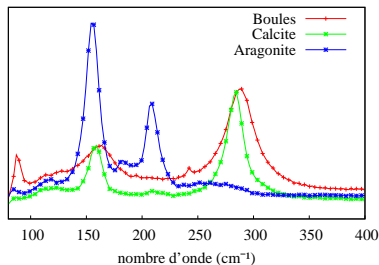
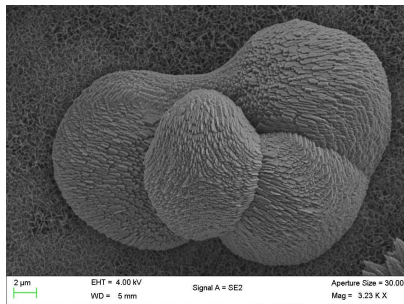
## Stade de Croissance:



- ▶ Choux-fleurs, à base de boule, composés d'Aragonite
- ▶ Boules (symétrie d'axe 3): calcite
- ▶ Transformation de la Calcite en Aragonite (plus stable dans les conditions expérimentales)

# Bilan sur les Dépôts Minéraux

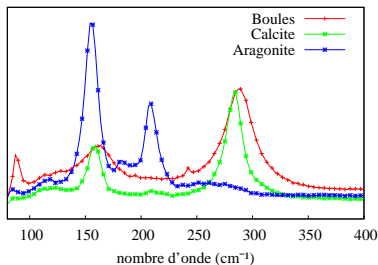
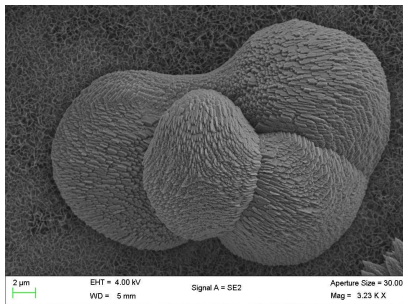
Stade de Croissance:



- ▶ Choux-fleurs, à base de boule, composés d'Aragonite
- ▶ Boules (symétrie d'axe 3): calcite
- ▶ Transformation de la Calcite en Aragonite (plus stable dans les conditions expérimentales)

# Bilan sur les Dépôts Minéraux

Stade de Croissance:



- ▶ Choux-fleurs, à base de boule, composés d'Aragonite
- ▶ Boules (symétrie d'axe 3): calcite
- ▶ Transformation de la Calcite en Aragonite (plus stable dans les conditions expérimentales)

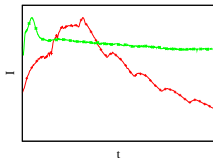


## Dépôts minéraux-organiques d'aragonite

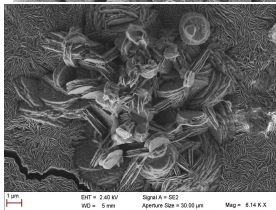
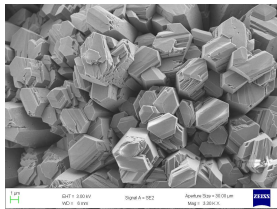
- ▶ Influence des Molécules Organiques
  - ▶ Chitosan
  - ▶ Phases Extraites de la nacre de *Pinctada maxima*
    - ▶ Phases Polaires
    - ▶ Phases Apolaires
- ▶ Modes de croissance
- ▶ Effets inter- et intracristallins

# Influence du Chitosan

Solution de Chitosan (1%) + Acide polyacrylique (1%)

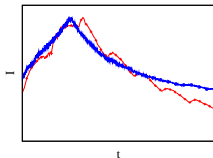


Dépôt minéral  
Solution de Chitosan:  
avant procédure



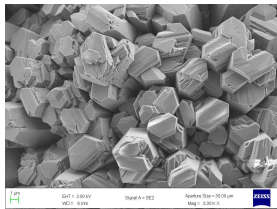
# Influence du Chitosan

Solution de Chitosan (1%) + Acide polyacrylique (1%)



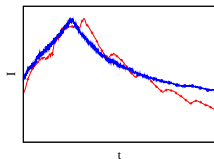
Dépôt minéral

Solution de Chitosan:  
gouttes à gouttes

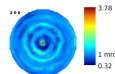
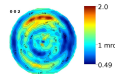
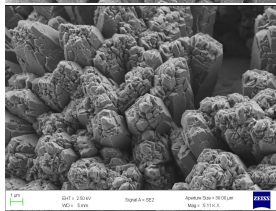
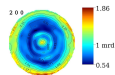
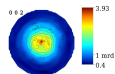
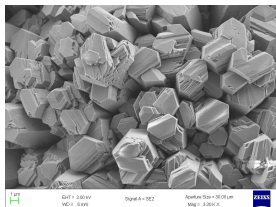


# Influence du Chitosan

Solution de Chitosan (1%) + Acide polyacrylique (1%)



$$F^2 = 2.0 \text{ mrd}^2$$
$$ODF_{\text{max}} = 33.4 \text{ mrd}$$

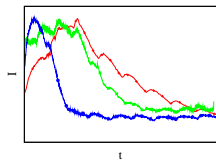


Dépôt minéral

Solution de Chitosan:  
gouttes à gouttes

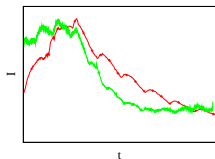
$$F^2 = 2.03 \text{ mrd}^2$$
$$ODF_{\text{max}} = 73.5 \text{ mrd}$$

# Influence des Phases Organiques Polaires

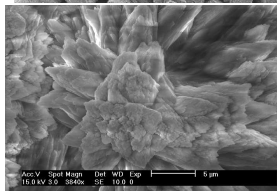
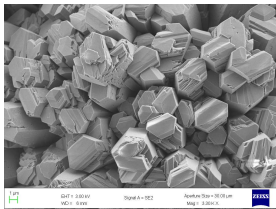


Ajout	Cristallite (Å)
0	886.1(1)
10mg	1284(0.7)
20mg	1150(1)

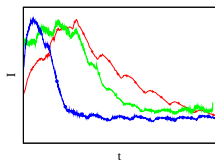
# Influence des Phases Organiques Polaires



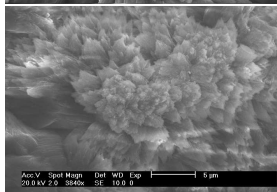
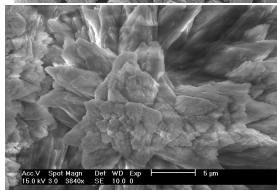
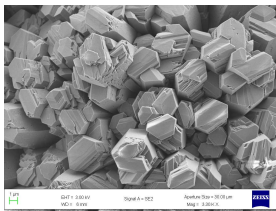
Ajout	Cristallite ( $\text{\AA}$ )
0	886.1(1)
10mg	1284(0.7)
20mg	1150(1)



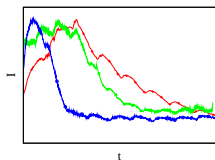
# Influence des Phases Organiques Polaires



Ajout	Cristallite (Å)
0	886.1(1)
10mg	1284(0.7)
20mg	1150(1)



# Influence des Phases Organiques Polaires



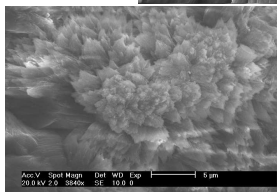
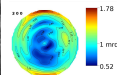
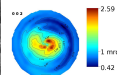
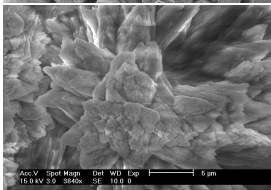
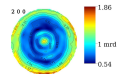
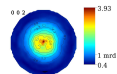
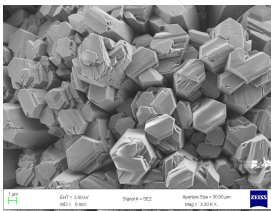
$$F^2 = 2.0 \text{ mrd}^2$$

$$ODF_{max} = 33.4 \text{ mrd}$$

Ajout	Cristallite (Å)
0	886.1(1)
10mg	1284(0.7)
20mg	1150(1)

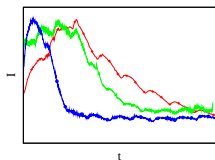
$$F^2 = 1.59 \text{ mrd}^2$$

$$ODF_{max} = 17.2 \text{ mrd}$$





# Influence des Phases Organiques Polaires



$$F^2 = 2.0 \text{ mrd}^2$$

$$ODF_{max} = 33.4 \text{ mrd}$$

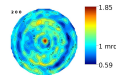
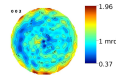
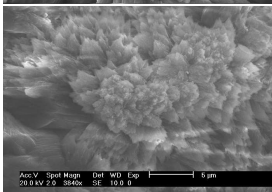
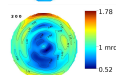
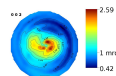
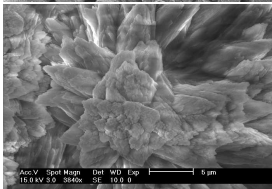
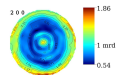
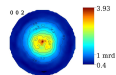
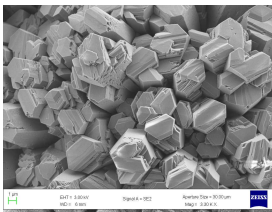
Ajout	Cristallite (Å)
0	886.1(1)
10mg	1284(0.7)
20mg	1150(1)

$$F^2 = 1.59 \text{ mrd}^2$$

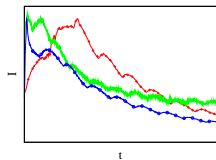
$$ODF_{max} = 17.2 \text{ mrd}$$

$$F^2 = 2.13 \text{ mrd}^2$$

$$ODF_{max} = 39.6 \text{ mrd}$$

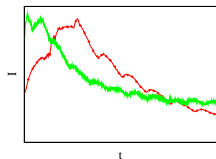


# Influence des Phases Organiques Apolaires

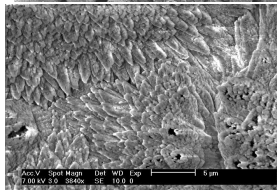
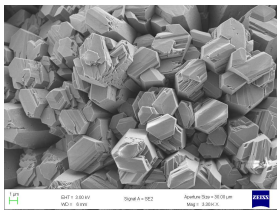


Ajout	Cristallite (Å)
0	886.1(1)
10mg	1211.13(8)
20mg	1126.5(5)

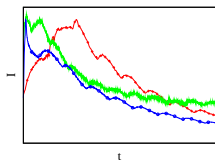
# Influence des Phases Organiques Apolaires



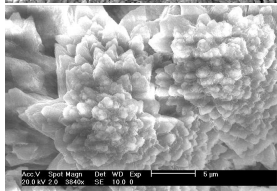
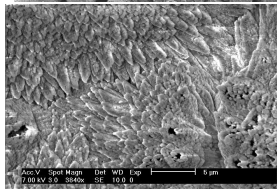
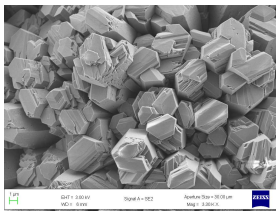
Ajout	Cristallite ( $\text{\AA}$ )
0	886.1(1)
10mg	1211.13(8)
20mg	1126.5(5)



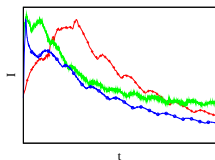
# Influence des Phases Organiques Apolaires



Ajout	Cristallite ( $\text{\AA}$ )
0	886.1(1)
10mg	1211.13(8)
20mg	1126.5(5)



# Influence des Phases Organiques Apolaires



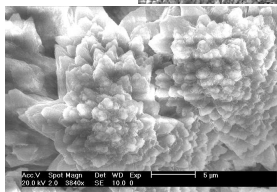
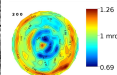
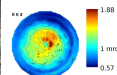
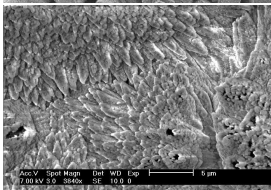
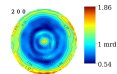
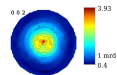
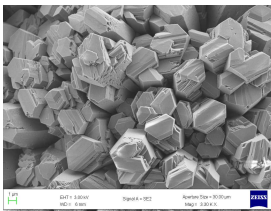
$$F^2 = 2.0 \text{ mrd}^2$$

$$ODF_{\text{max}} = 33.4 \text{ mrd}$$

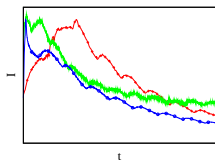
Ajout	Cristallite (Å)
0	886.1(1)
10mg	1211.13(8)
20mg	1126.5(5)

$$F^2 = 1.21 \text{ mrd}^2$$

$$ODF_{\text{max}} = 10.4 \text{ mrd}$$



# Influence des Phases Organiques Apolaires



$$F^2 = 2.0 \text{ mrd}^2$$

$$ODF_{max} = 33.4 \text{ mrd}$$

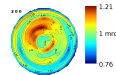
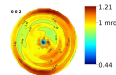
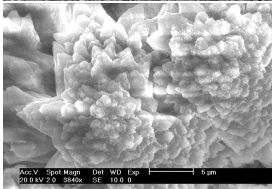
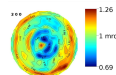
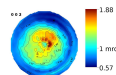
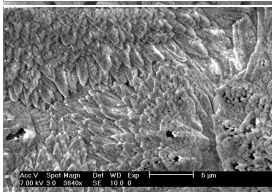
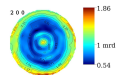
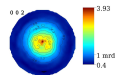
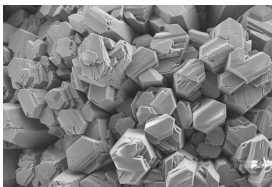
Ajout	Cristallite (Å)
0	886.1(1)
10mg	1211.13(8)
20mg	1126.5(5)

$$F^2 = 1.21 \text{ mrd}^2$$

$$ODF_{max} = 10.4 \text{ mrd}$$

$$F^2 = 1.06 \text{ mrd}^2$$

$$ODF_{max} = 5.2 \text{ mrd}$$



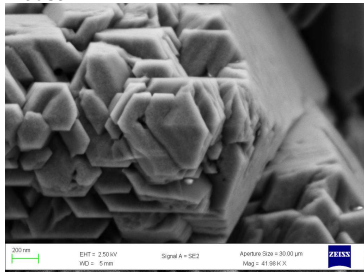
# Influence des Molécules Organiques sur le Mode de Croissance

Croissance en choux-fleurs

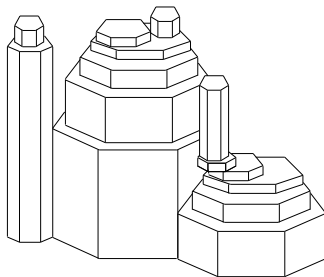
# Influence des Molécules Organiques sur le Mode de Croissance

## Croissance en choux-fleurs

### Chitosan



Bâtonnets de section  
pseudo-hexagonale

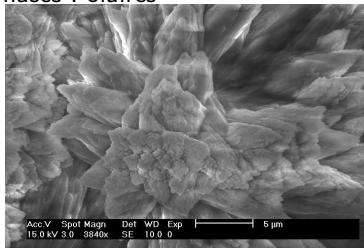




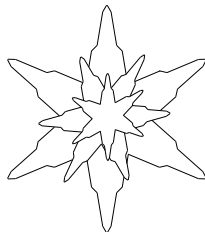
# Influence des Molécules Organiques sur le Mode de Croissance

## Croissance en choux-fleurs

### Phases Polaires



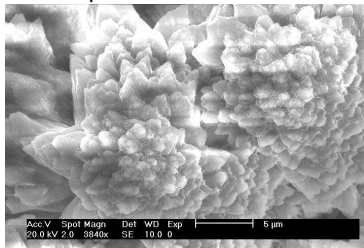
Aiguilles



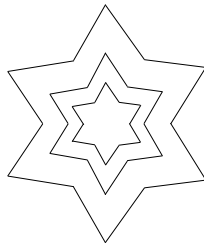
# Influence des Molécules Organiques sur le Mode de Croissance

Croissance en choux-fleurs

Phases Apolaires



Feuillets



# Influence des Molécules Organiques sur la Cristallinité de l'Aragonite

- ▶ ↑ taille cristallites: effet intracristallin
- ▶ ↑ texture: effet intercristallin

# Influence des Molécules Organiques sur la Cristallinité de l'Aragonite

- ▶ ↑ taille cristallites: effet intracristallin
- ▶ ↑ texture: effet intercristallin

Chitosan:

	Cristallite (Å)	texture ( $mrd^2$ )	$Z_{C-O_1}$ (Å)
chitosan	1272.1(1)	2.03	0.04
minéral	886.1(1)	2.0 ( $\vec{c}$ )	0.0868
<i>Pinctada maxima</i>	3696(0.8)	625.66 ( $\vec{c}$ )	0.0455

# Influence des Molécules Organiques sur la Cristallinité de l'Aragonite

- ▶ ↑ taille cristallites: effet intracristallin
- ▶ ↑ texture: effet intercrystallin

Chitosan:

	Cristallite (Å)	texture ( $mrd^2$ )	$Z_{C-O_1}$ (Å)
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Chitosan:

	Cristallite (Å)	texture ( $mrd^2$ )	$Z_{C-O_1}$ (Å)
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minéral	886.1(1)	2.0 ( $\vec{c}$ )	0.0868
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# Influence des Molécules Organiques sur la Cristallinité de l'Aragonite

- ▶ ↑ taille cristallites: effet intracristallin
- ▶ ↑ texture: effet intercristallin

Phases Organiques extraites (faible concentration 10mg):

	Cristallite (Å)	texture ( $mrd^2$ )	$Z_{C-O_1}$ (Å)
polaire	1284(0.7)	1.59 ( $\vec{c}$ )	0.1341
apolaire	1211.13(8)	1.21 ( $\vec{c}$ )	0.1730
minéral	886.1(1)	2.0 ( $\vec{c}$ )	0.0868

# Influence des Molécules Organiques sur la Cristallinité de l'Aragonite

- ▶ ↑ taille cristallites: effet intracristallin
- ▶ ↑ texture: **effet intercrystallin**

Phases Organiques extraites (faible concentration 10mg):

	Cristallite (Å)	texture ( $mrd^2$ )	$Z_{C-O_1}$ (Å)
polaire	1284(0.7)	1.59 ( $\vec{c}$ )	0.1341
apolaire	1211.13(8)	1.21 ( $\vec{c}$ )	0.1730
minéral	886.1(1)	2.0 ( $\vec{c}$ )	0.0868



# Influence des Molécules Organiques sur la Cristallinité de l'Aragonite

- ▶ ↑ taille cristallites: effet intracristallin
- ▶ ↑ texture: effet intercristallin

Phases Organiques extraites (faible concentration 10mg):

	Cristallite (Å)	texture ( $mrd^2$ )	$Z_{C-O_1}$ (Å)
polaire	1284(0.7)	1.59 ( $\vec{c}$ )	0.1341
apolaire	1211.13(8)	1.21 ( $\vec{c}$ )	0.1730
minéral	886.1(1)	2.0 ( $\vec{c}$ )	0.0868

# Influence des Molécules Organiques sur la Cristallinité de l'Aragonite

- ▶ ↑ taille cristallites: effet intracristallin
- ▶ ↑ texture: effet intercristallin

Phases Organiques extraites (faible concentration 10mg):

	Cristallite (Å)	texture ( $mrd^2$ )	$Z_{C-O_1}$ (Å)
polaire	1284(0.7)	1.59 ( $\vec{c}$ )	0.1341
apolaire	1211.13(8)	1.21 ( $\vec{c}$ )	0.1730
minéral	886.1(1)	2.0 ( $\vec{c}$ )	0.0868

Phases Organiques extraites (forte concentration 20mg):

	Cristallite (Å)	texture ( $mrd^2$ )	$Z_{C-O_1}$ (Å)
polaire	1150(1)	2.13 ( $\vec{c}/\parallel$ )	0.0810
apolaire	1126.5(5)	1.06	0.0862
minéral	886.1(1)	2.0 ( $\vec{c}$ )	0.0868
<i>Haliotis tuberculata</i>	2507(26)	211.05 ( $\vec{c}$ )	0.0895

# Influence des Molécules Organiques sur la Cristallinité de l'Aragonite

- ▶ ↑ taille cristallites: effet intracristallin
- ▶ ↑ texture: **effet intercrystallin**

Phases Organiques extraites (faible concentration 10mg):

	Cristallite (Å)	texture ( $mrd^2$ )	$Z_{C-O_1}$ (Å)
polaire	1284(0.7)	1.59 ( $\vec{c}$ )	0.1341
apolaire	1211.13(8)	1.21 ( $\vec{c}$ )	0.1730
minéral	886.1(1)	2.0 ( $\vec{c}$ )	0.0868

Phases Organiques extraites (forte concentration 20mg):

	Cristallite (Å)	texture ( $mrd^2$ )	$Z_{C-O_1}$ (Å)
polaire	1150(1)	2.13 ( $\vec{c}/\parallel$ )	0.0810
apolaire	1126.5(5)	1.06	0.0862
minéral	886.1(1)	2.0 ( $\vec{c}$ )	0.0868
<i>Haliotis tuberculata</i>	2507(26)	211.05 ( $\vec{c}$ )	0.0895

# Influence des Molécules Organiques sur la Cristallinité de l'Aragonite

- ▶ ↑ taille cristallites: effet intracristallin
- ▶ ↑ texture: effet intercristallin

Phases Organiques extraites (faible concentration 10mg):

	Cristallite (Å)	texture ( $mrd^2$ )	$Z_{C-O_1}$ (Å)
polaire	1284(0.7)	1.59 ( $\vec{c}$ )	0.1341
apolaire	1211.13(8)	1.21 ( $\vec{c}$ )	0.1730
minéral	886.1(1)	2.0 ( $\vec{c}$ )	0.0868

Phases Organiques extraites (forte concentration 20mg):

	Cristallite (Å)	texture ( $mrd^2$ )	$Z_{C-O_1}$ (Å)
polaire	1150(1)	2.13 ( $\vec{c}/l$ )	0.0810
apolaire	1126.5(5)	1.06	0.0862
minéral	886.1(1)	2.0 ( $\vec{c}$ )	0.0868
<i>Haliotis tuberculata</i>	2507(26)	211.05 ( $\vec{c}$ )	0.0895

## Conclusions et Perspectives

# Conclusions

- ▶ Dépôt minéral: croissance orientée d'aragonite sous forme de bâtonnets (portants l'axe  $\vec{c}$ ), croissance en plaquettes pseudo-hexagonales liées par maclage  $\Rightarrow$  effet thermodynamique
- ▶ Dépôt minéral-organique: modification des faciès, texture,
  - ▶ effet intracristallin ( $\uparrow$  taille des cristallites): faible concentration de phases polaires et apolaires
  - ▶ effet intercristallin ( $\uparrow$  texture): chitosan et forte concentration de phases polaires
- ▶ Comparaison avec des nacres naturelles grâce au groupement  $\text{CO}_3^{2-}$ :
  - ▶ texture *nacre colonnaire*: minéral
  - ▶ texture *nacre mur de brique*: organique

# Conclusions

- ▶ Dépôt minéral: croissance orientée d'aragonite sous forme de bâtonnets (portants l'axe  $\vec{c}$ ), croissance en plaquettes pseudo-hexagonales liées par maclage  $\Rightarrow$  effet thermodynamique
- ▶ Dépôt minéral-organique: modification des faciès, texture,
  - ▶ effet intracristallin ( $\uparrow$  taille des cristallites): faible concentration de phases polaires et apolaires
  - ▶ effet intercristallin ( $\uparrow$  texture): chitosan et forte concentration de phases polaires
- ▶ Comparaison avec des nacres naturelles grâce au groupement  $\text{CO}_3^{2-}$ :
  - ▶ texture *nacre colonnaire*: minéral
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