

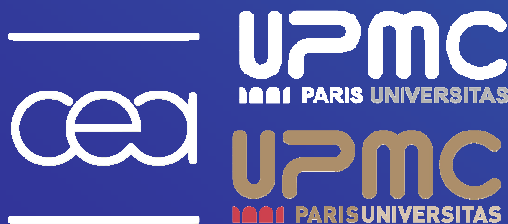


# Greffage de Films Organiques par Polymérisation Radicalaire Électro-amorcée en Milieu Aqueux Dispersé

Lorraine TESSIER

Guy DENIAU

Bernadette CHARLEUX



Vendredi 16 octobre 2009

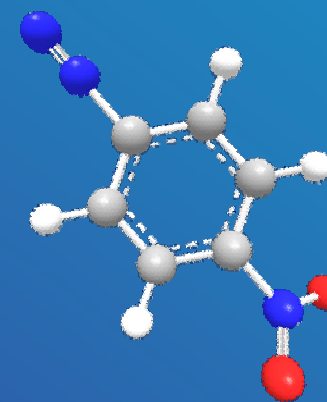
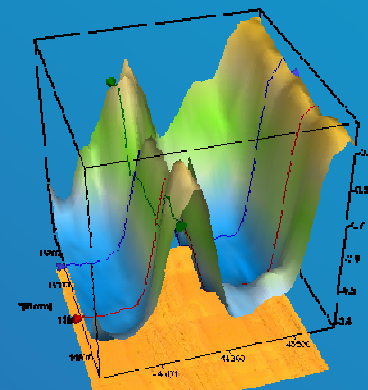
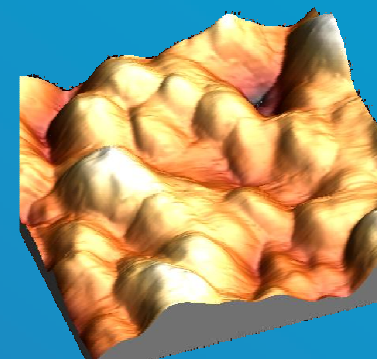
Laboratoire de Chimie des Surfaces et Interfaces

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Greffage de Films Organiques par  
Polymérisation Radicalaire  
Électro-amorcée en Milieu Aqueux  
Dispersé



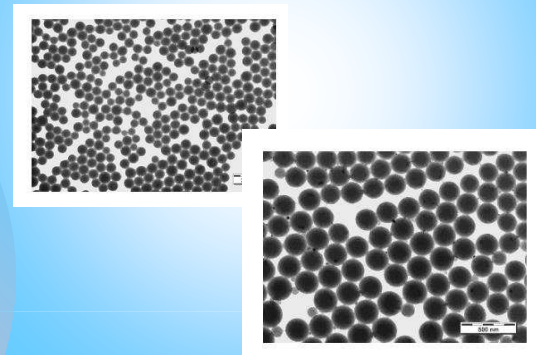
## Objectif:

Etudier le mécanisme réactionnel impliqué dans le procédé SEEP

Laboratoire de Chimie des Polymères

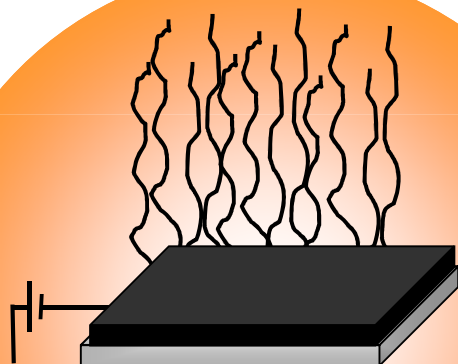
Polymérisation radicalaire (contrôlée)

Polymérisation en émulsion



## SEEP

*(Surface Electroinitiated Emulsion Polymerization)*



Fonctionnalisation de Surface  
(Electrogreffage)

Laboratoire de Chimie des Surfaces et Interfaces

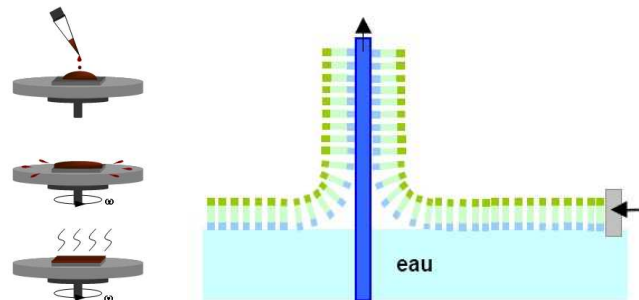
J. Nicolas, B. Charleux, O. Guerret, S. Magnet; *Angew. Chem, Int. Ed*; **2004**; 43; 6186

S. Palacin, C. Bureau, J. Charlier, G. Deniau, B. Mouanda, P. Viel; *ChemPhysChem*; **2004**; 18; 1468-1481

G. Deniau, L. Azoulay, L. Bougerolles, S. Palacin; *Chem. Mater*; **2006**; 18; 5421-5428

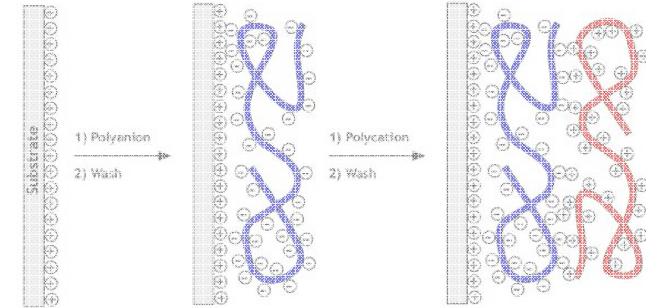
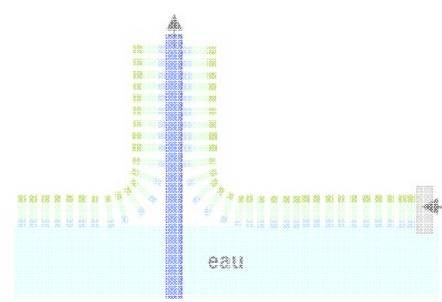
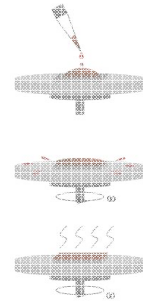
## Techniques de physisorption

- Spin coating
- Langmuir-Blodgett



## Techniques de physisorption

- Spin coating
- Langmuir-Blodgett
- Layer by layer

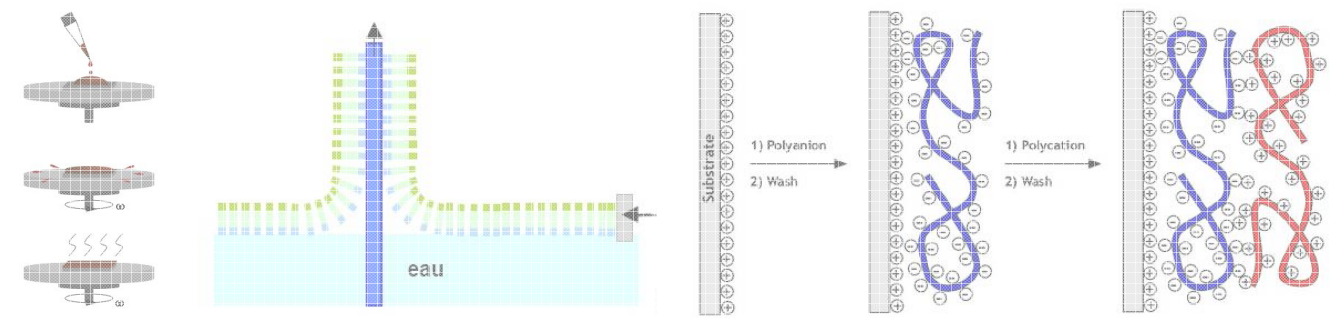


➔ ☹ Interactions faibles → fragilité du revêtement

## Techniques de chimisorption

## Techniques de physisorption

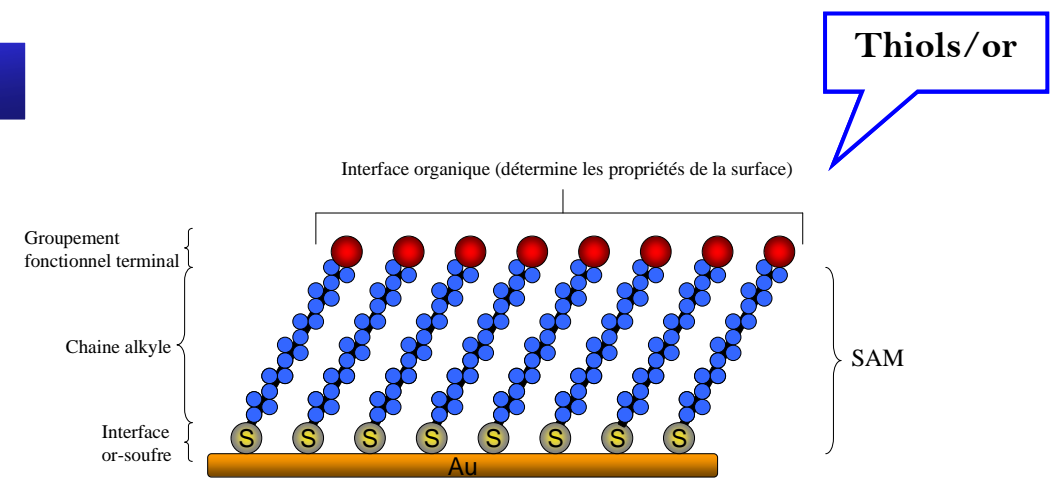
- Spin coating
- Langmuir-Blodgett
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## Techniques de chimisorption

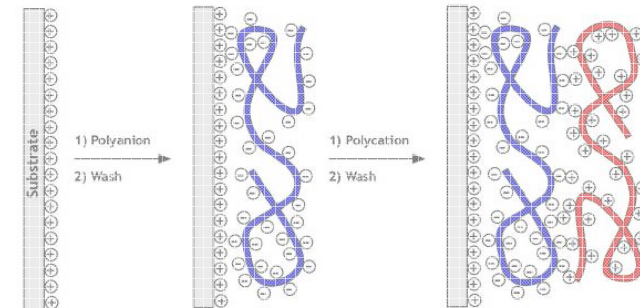
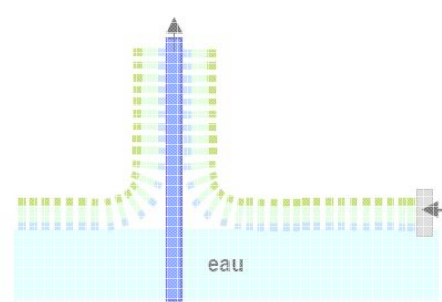
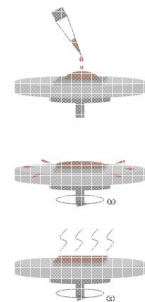
- Monocouche auto-assemblée



- ☹ Films fins
- ☹ Interface fragile

## Techniques de physisorption

- Spin coating
- Langmuir-Blodgett
- Layer by layer

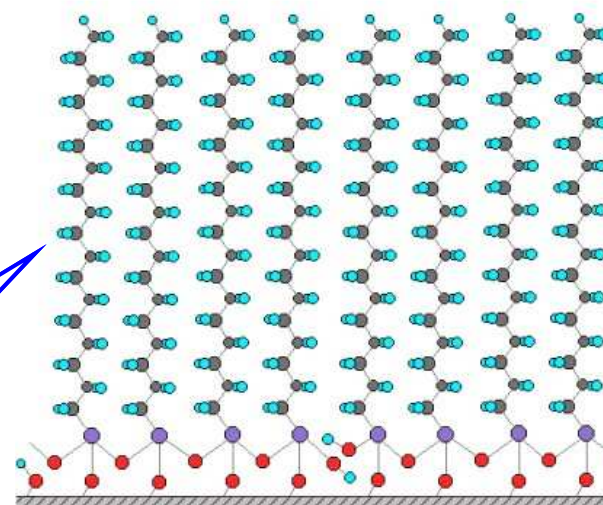


➡ ☹ Interactions faibles → fragilité du revêtement

## Techniques de chimisorption

- Monocouche auto-assemblée

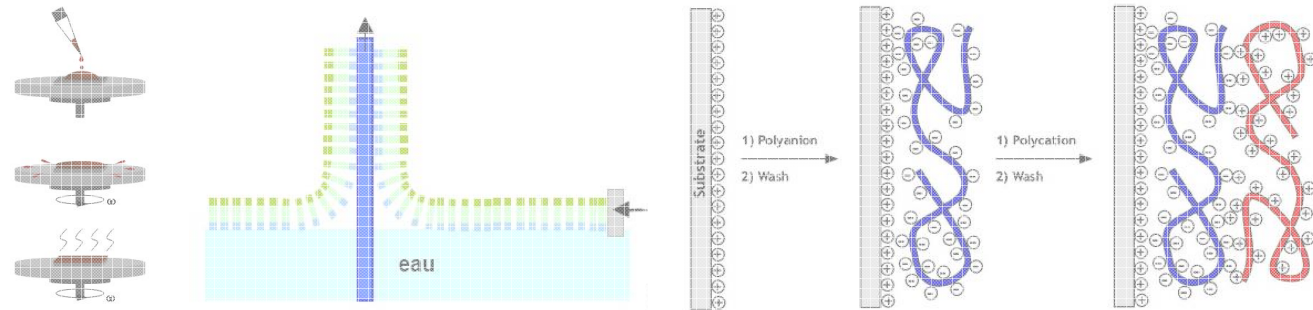
Chlorosilanes/silice



- ☹ Films fins
- ☹ Principalement sur surfaces oxydées

## Techniques de physisorption

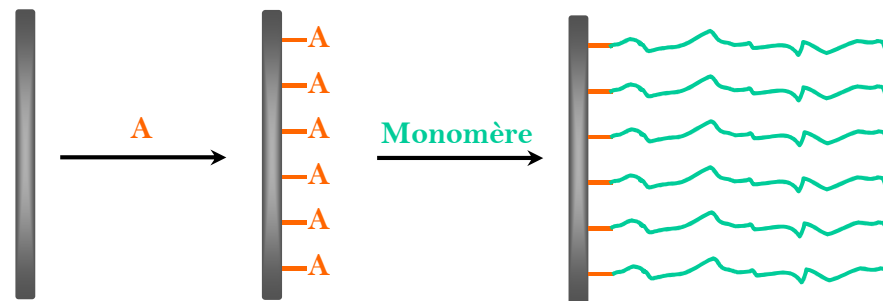
- Spin coating
- Langmuir-Blodgett
- Layer by layer



➡ ☹ Interactions faibles → fragilité du revêtement

## Techniques de chimisorption

- Monocouche auto-assemblée
- Polymérisation amorcée sur/à partir de la surface

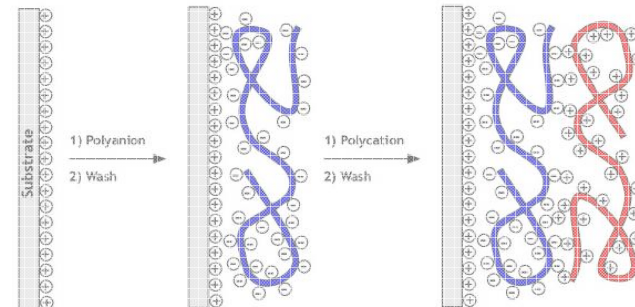
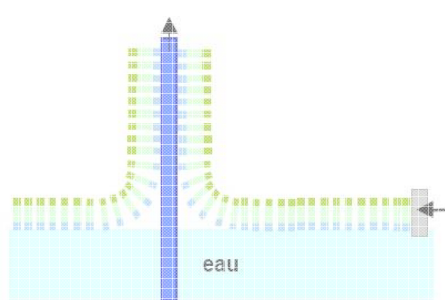
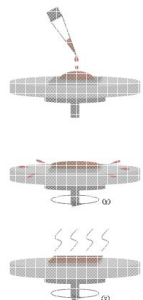


☹ Plusieurs étapes  
☹ Temps et température élevés



## Techniques de physisorption

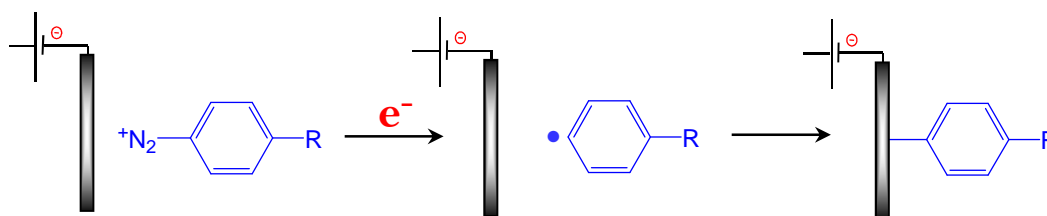
- Spin coating
- Langmuir-Blodgett
- Layer by layer



➔ ☹ Interactions faibles → fragilité du revêtement

## Techniques de chimisorption

- Monocouche auto-assemblée
- Polymérisation amorcée sur/à partir de la surface



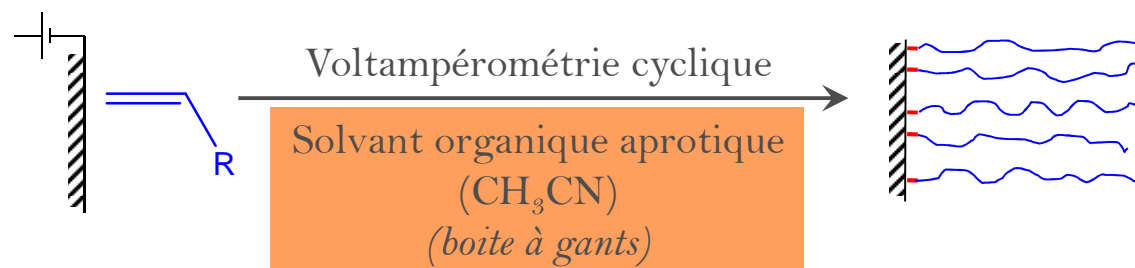
- Électrogreffage de sels d'aryldiazonium
- Électrogreffage cathodique de monomères vinyliques

☹ Revêtement type polyaryle

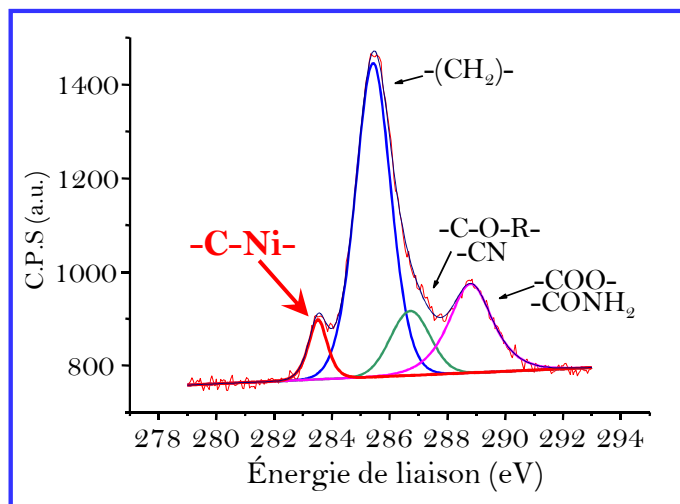
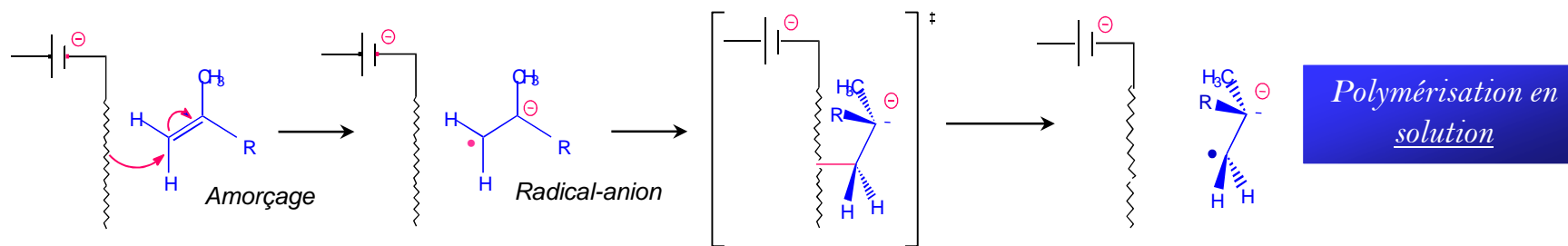
# I. Introduction

# Électrogreffage Cathodique

Principe



Mécanisme



➡ Mécanisme anionique

## **I. Introduction**

## **II. SEEP: Principe & Protocole expérimental**

## **III. Résultats**

Caractérisation des films

Mécanisme

## **IV. Discussion : Mécanisme de SEEP**

## **V. Application**

## **VI. Conclusion**

**I. Introduction**

**II. SEEP: Principe & Protocole expérimental**

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III. Résultats

Caractérisation des films

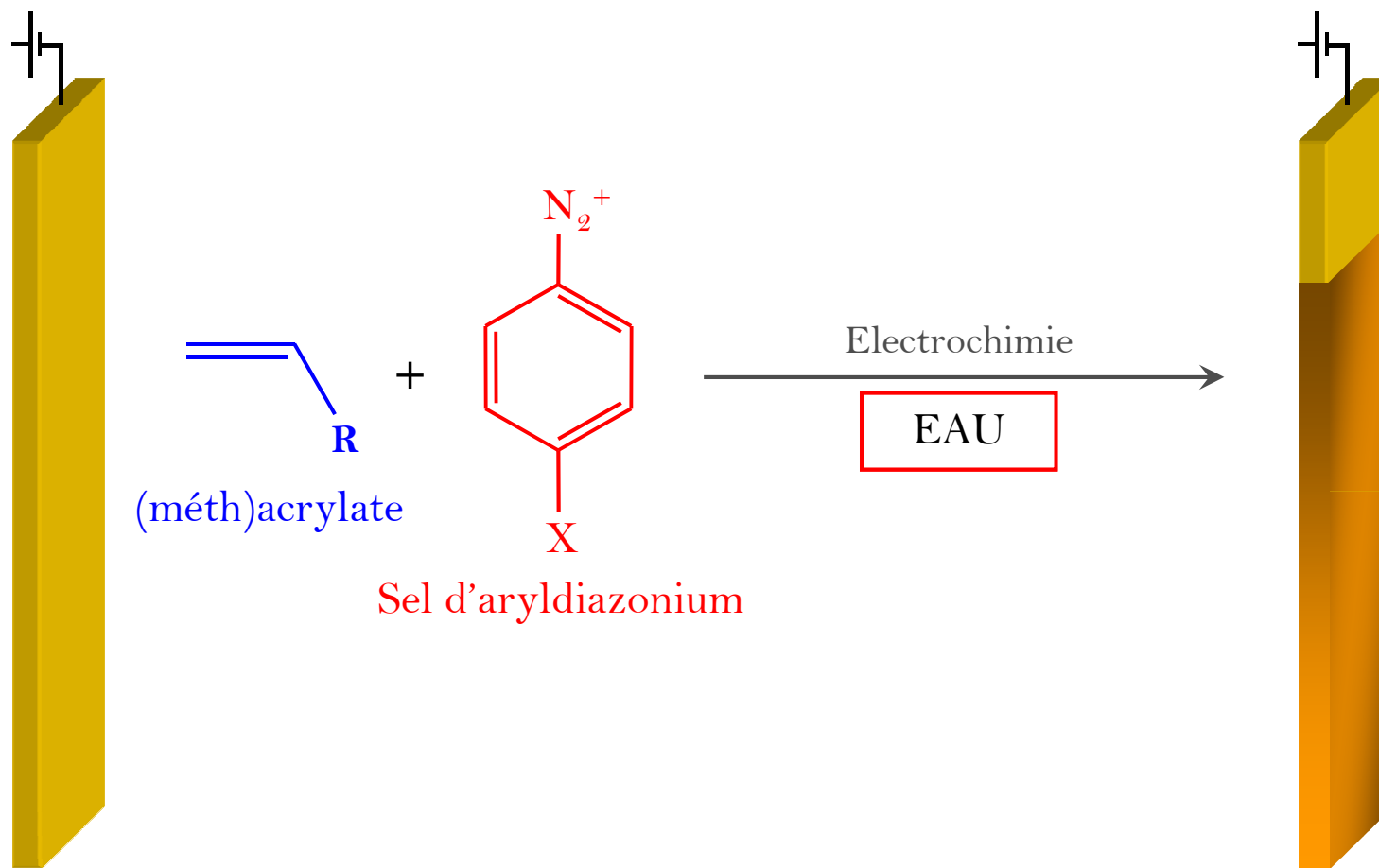
Mécanisme

IV. Discussion : Mécanisme de SEEP

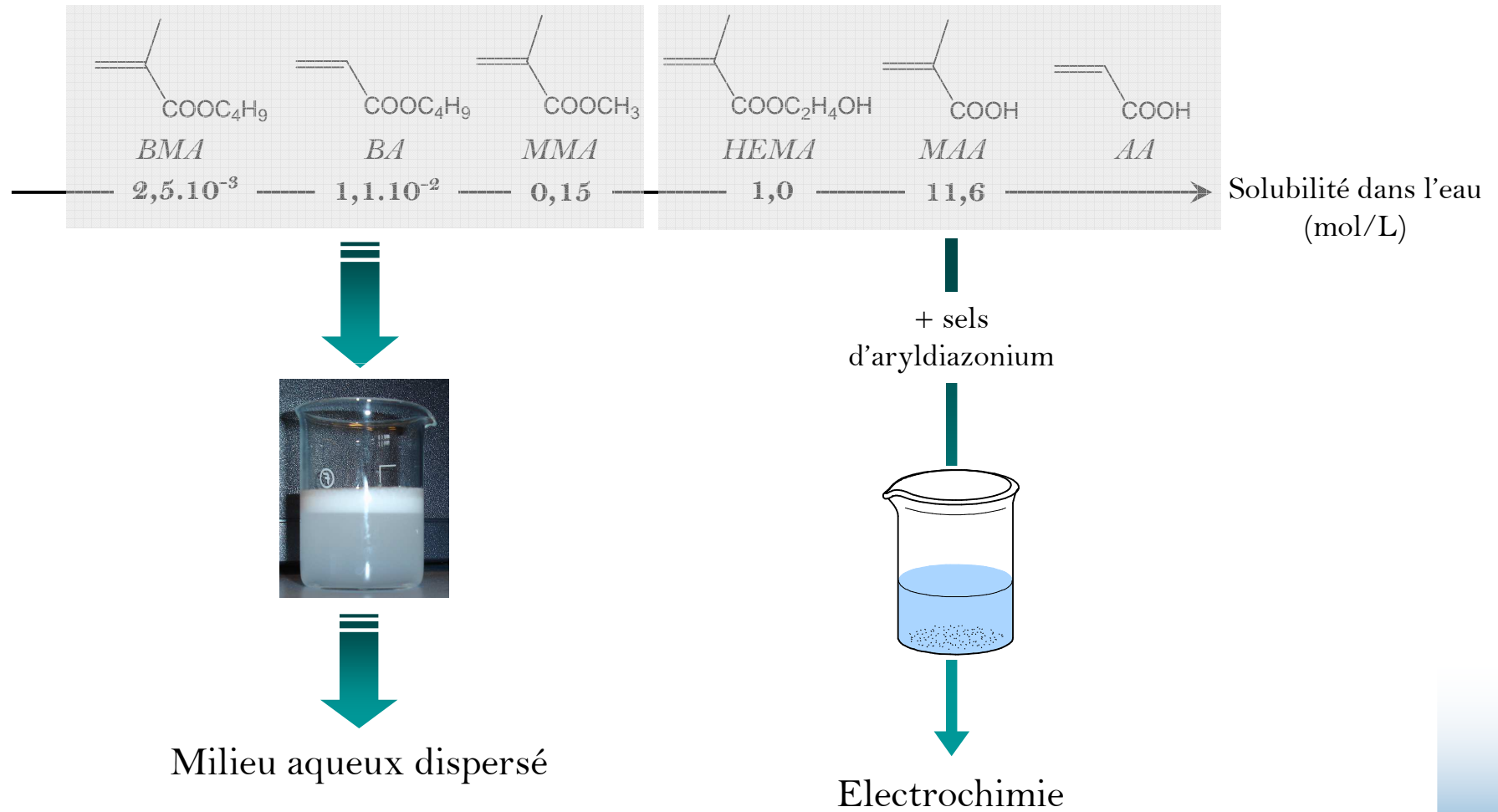
V. Application

VI. Conclusion

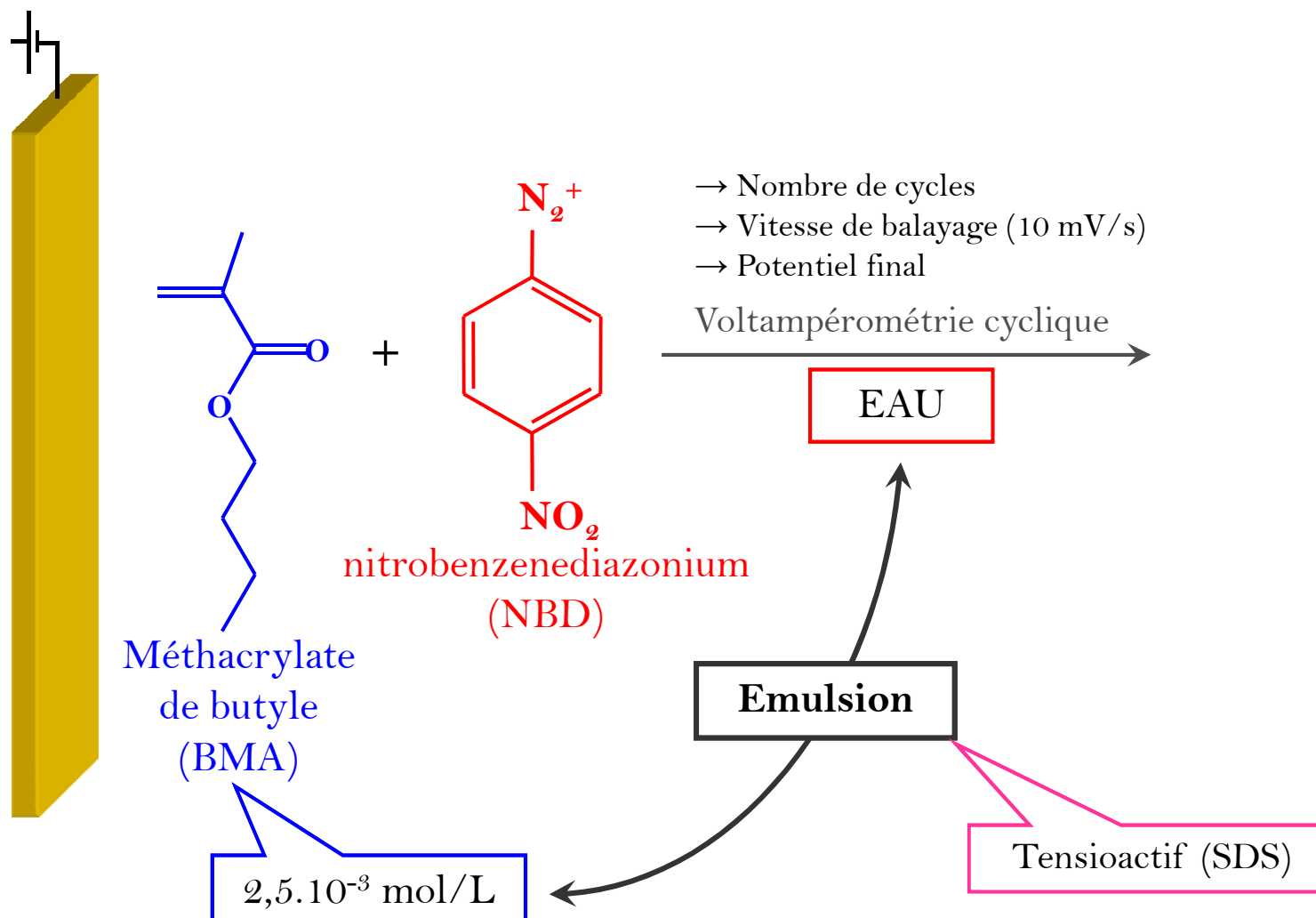
### *Surface Electronitiated Emulsion Polymerization*



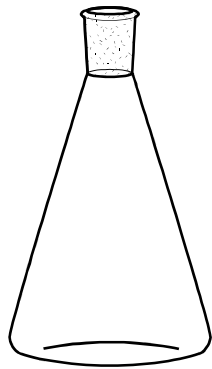
## Surface Electronitiated Emulsion Polymerization



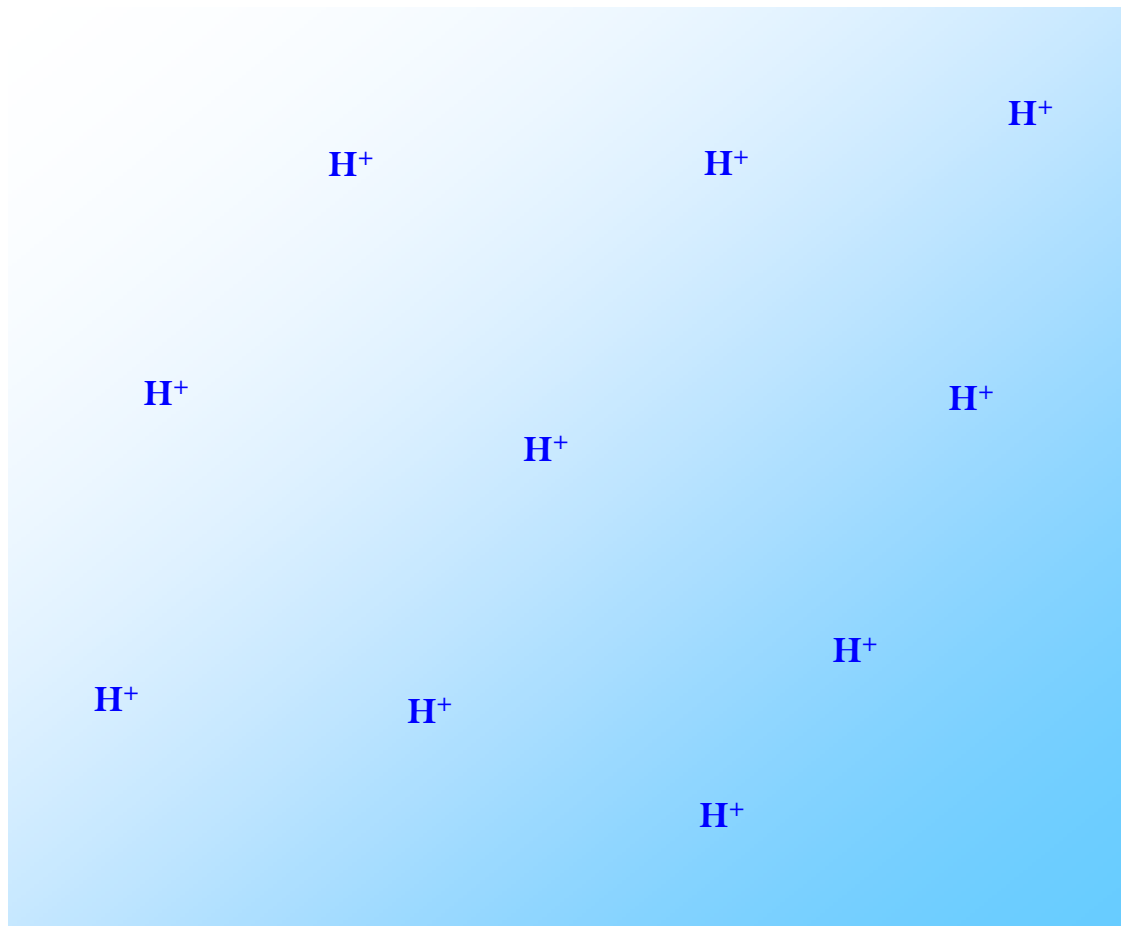
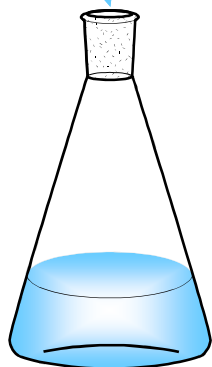
### *Surface Electronitiated Emulsion Polymerization*







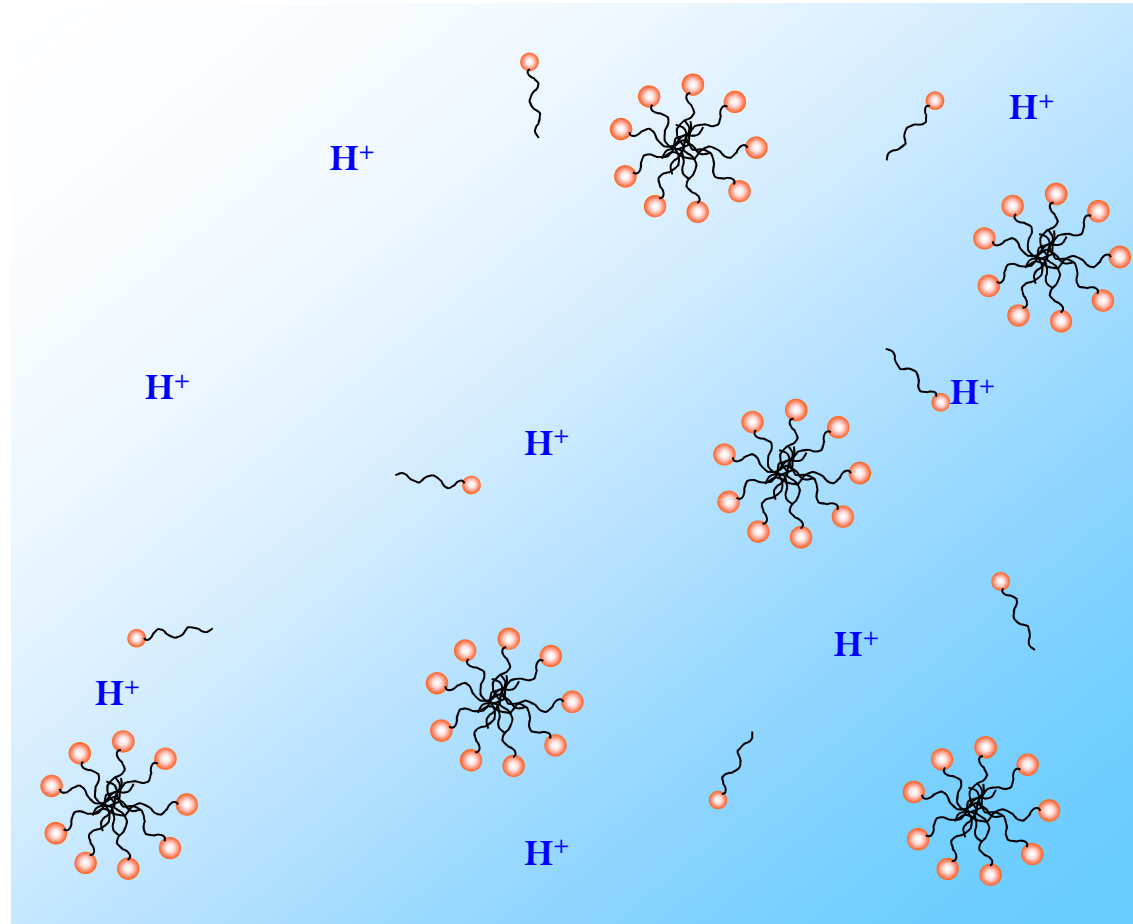
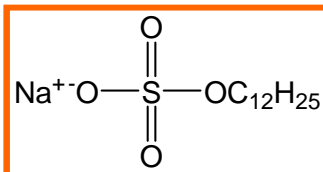
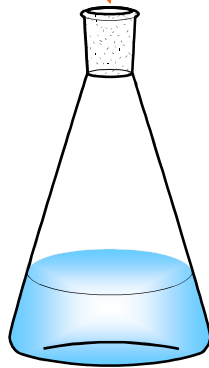
H<sub>2</sub>O DI  
H<sub>2</sub>SO<sub>4</sub> 0,01 M  
pH < 2



## II. Expérimental

## Préparation du mélange réactionnel

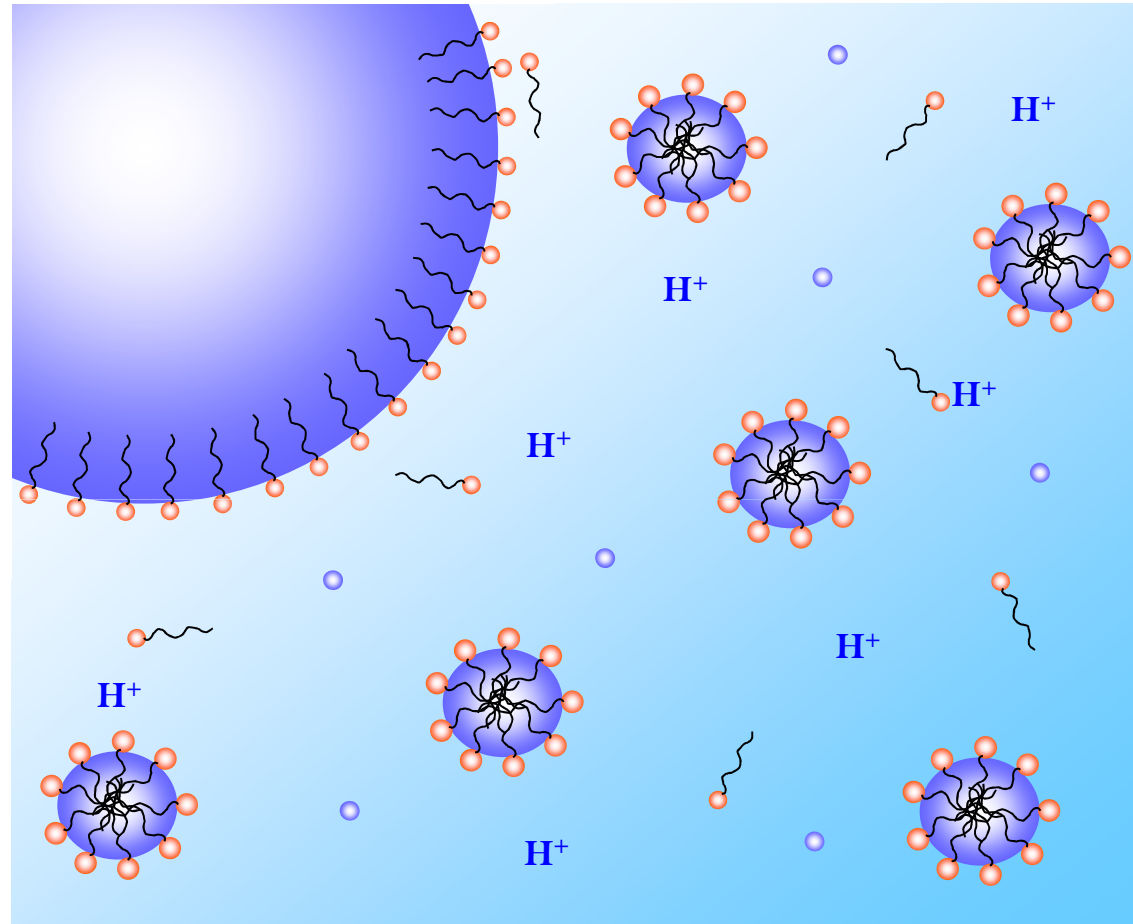
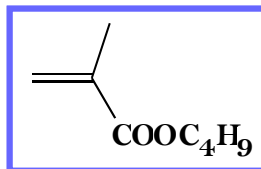
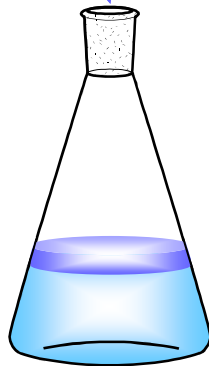
Dodécyle sulfate de sodium (SDS)  
 $9 \cdot 10^{-3}$  mol/L



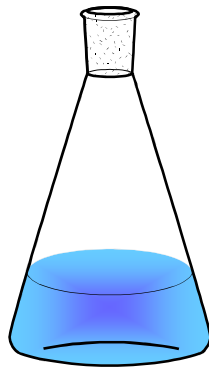
## II. Expérimental

## Préparation du mélange réactionnel

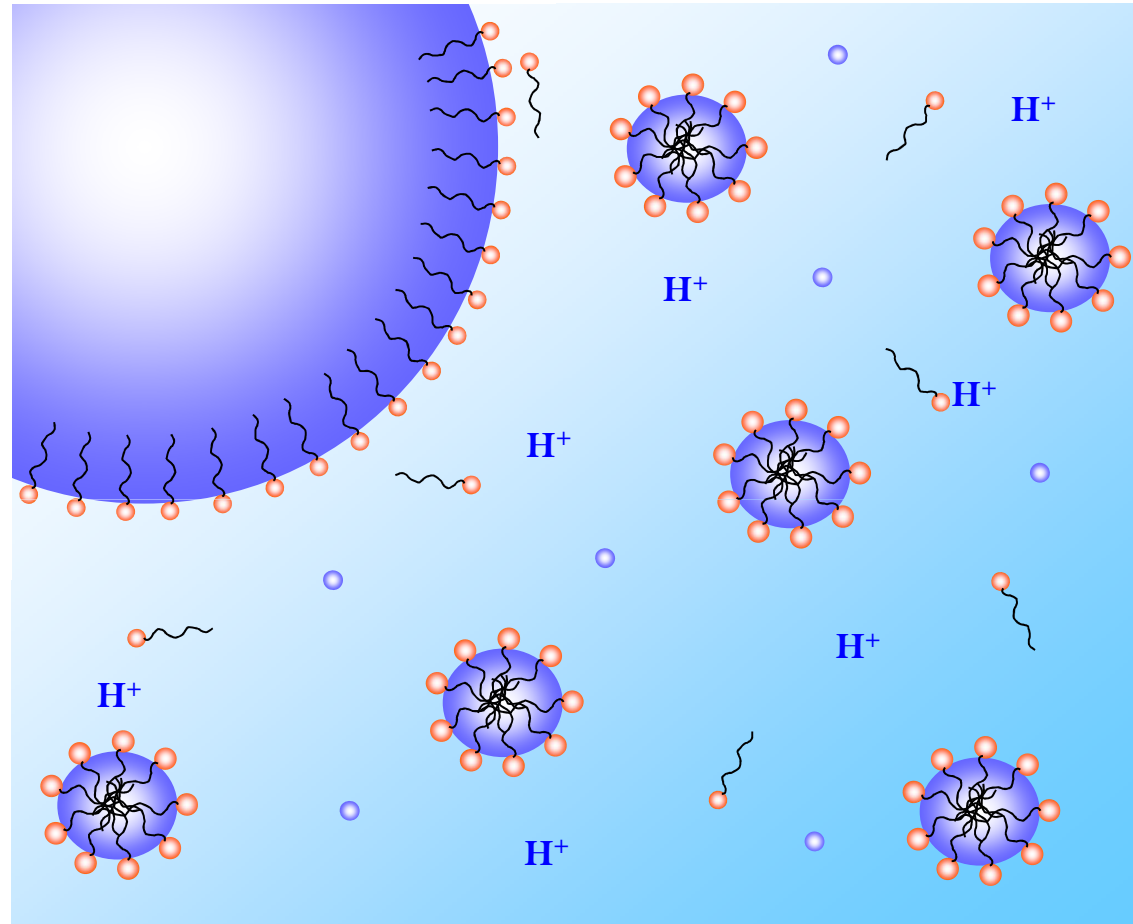
Méthacrylate de Butyle  
(BMA)  
0,7 mol/L



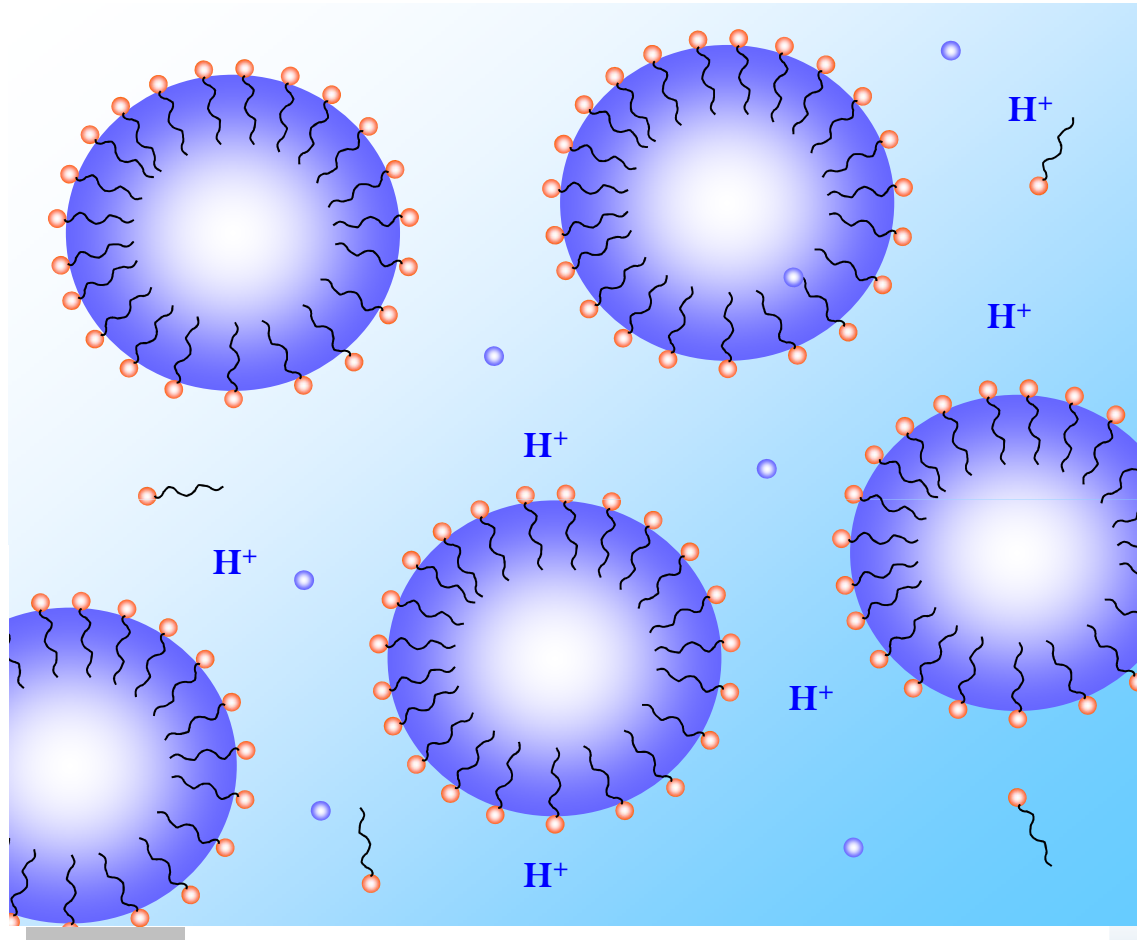
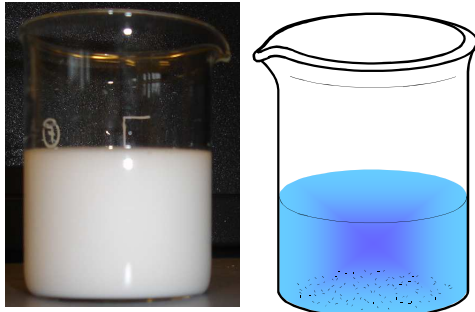
ÉMULSION



Sonde à ultrasons (10 minutes / 60 %)



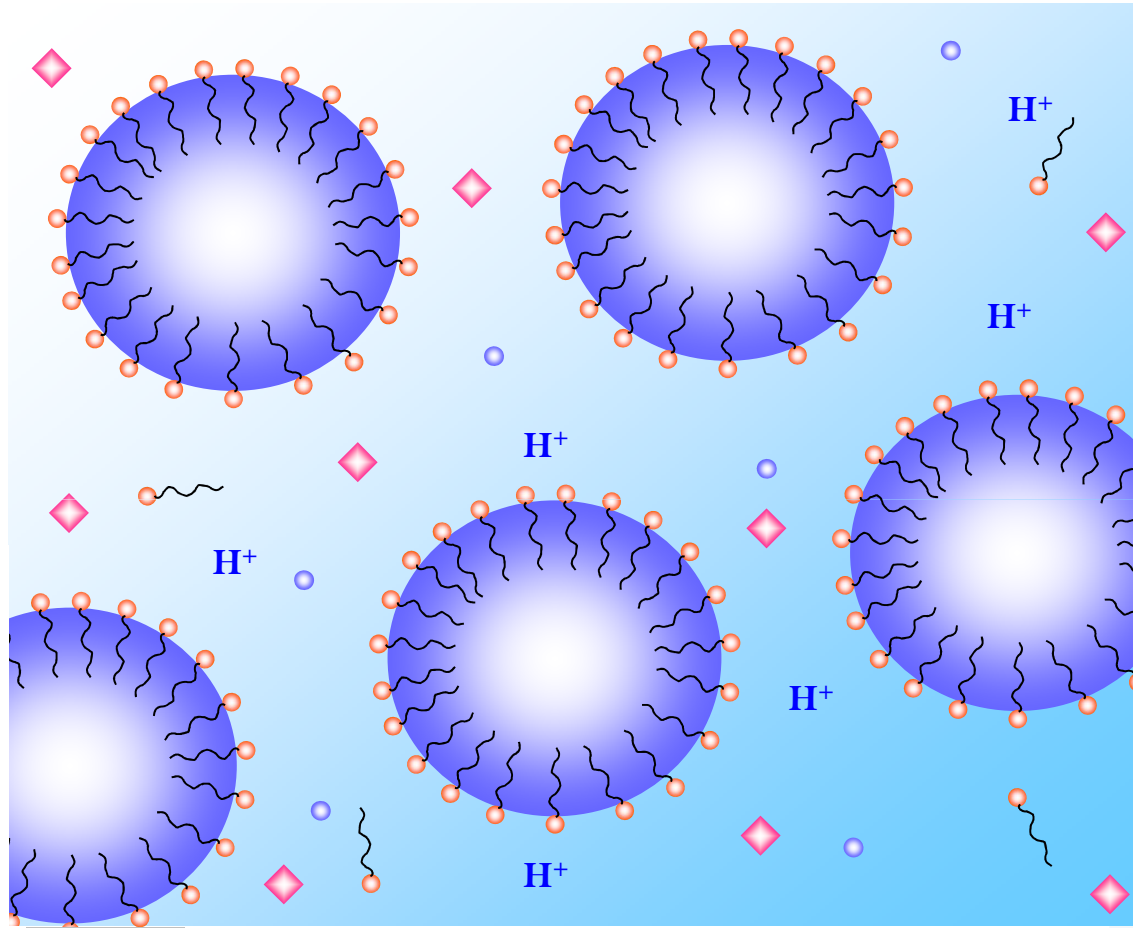
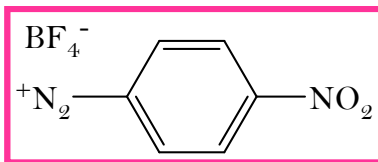
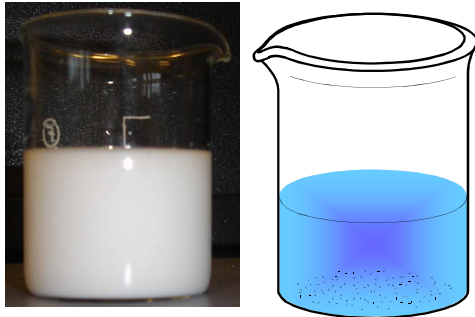
### MINIÉMULSION



## II. Expérimental

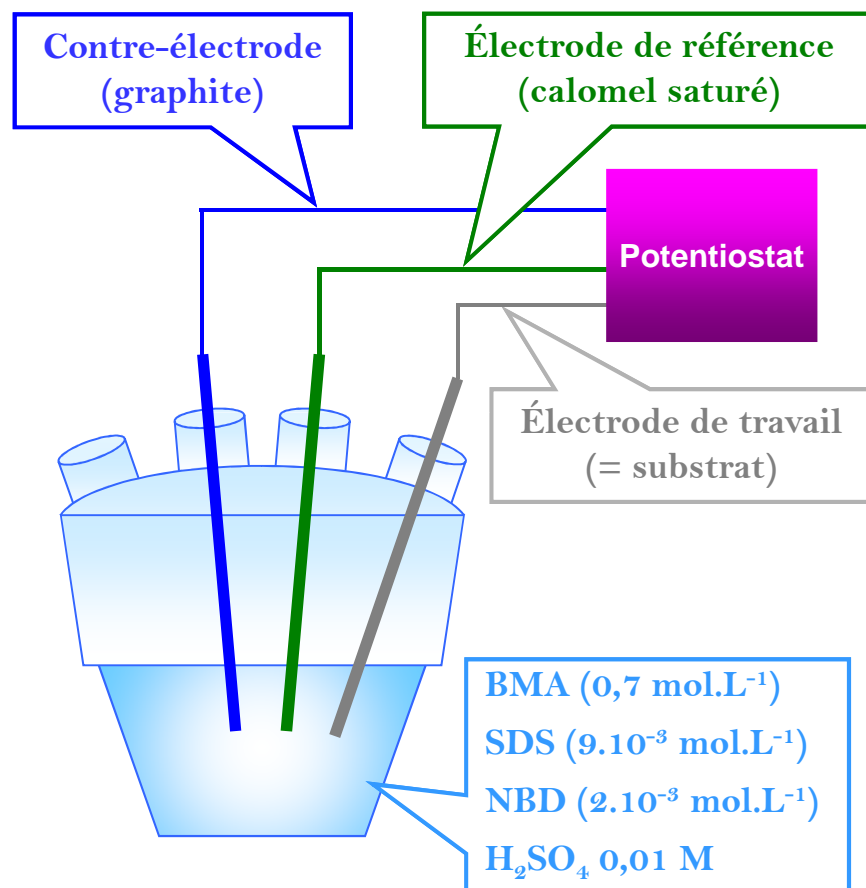
## Préparation du mélange réactionnel

Nitrobenzene diazonium  
tetrafluoroborate (NBD)  
 $2 \cdot 10^{-3}$  mol/L



☺ Commercial

☺ Sonde interne pour la caractérisation des films



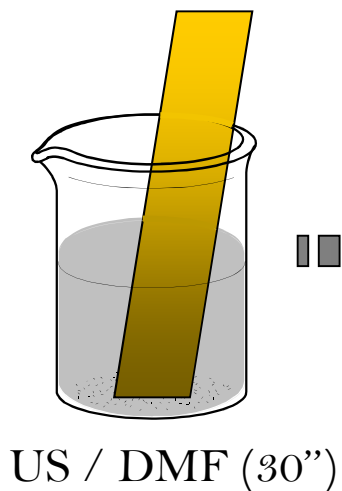
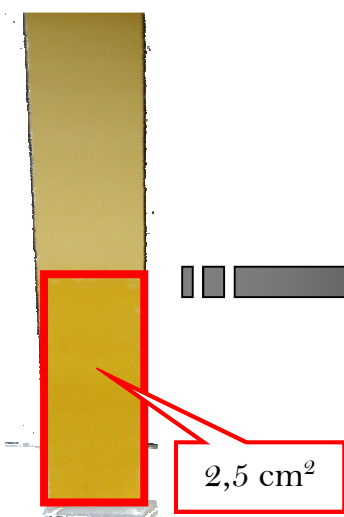
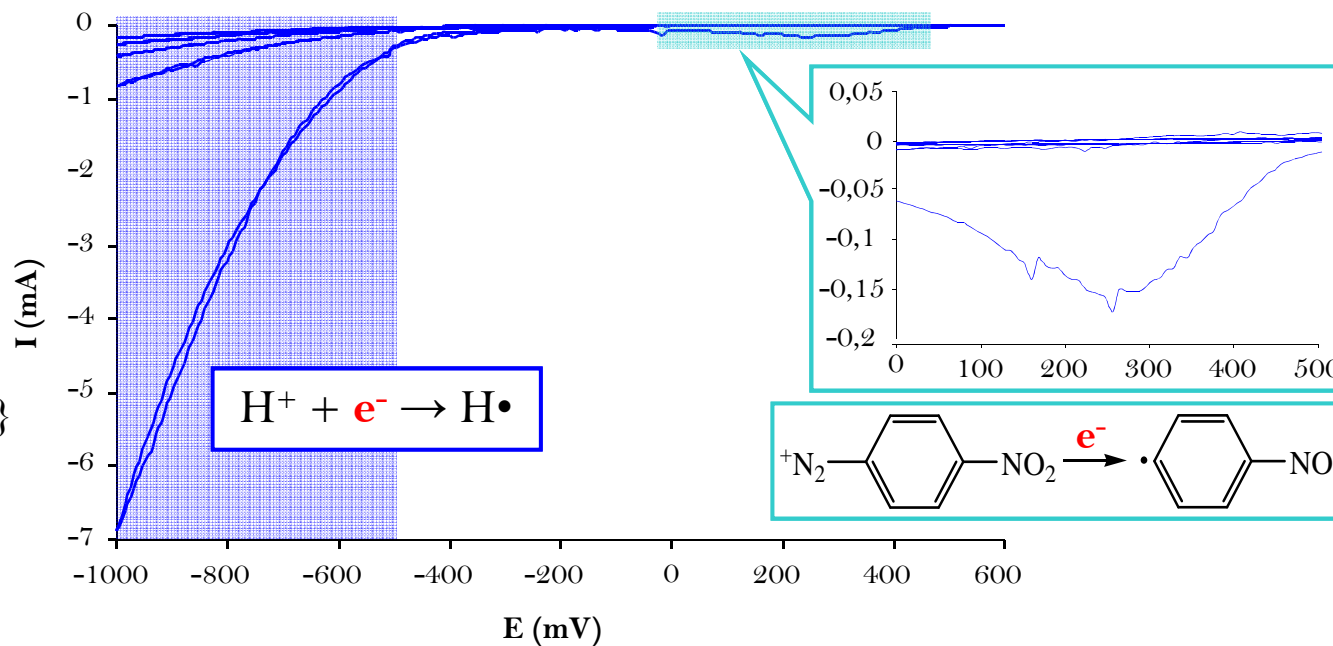


Voltampérométrie  
Cyclique

10 mV/s

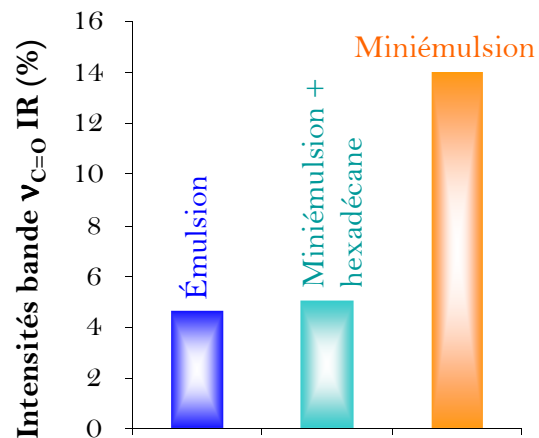
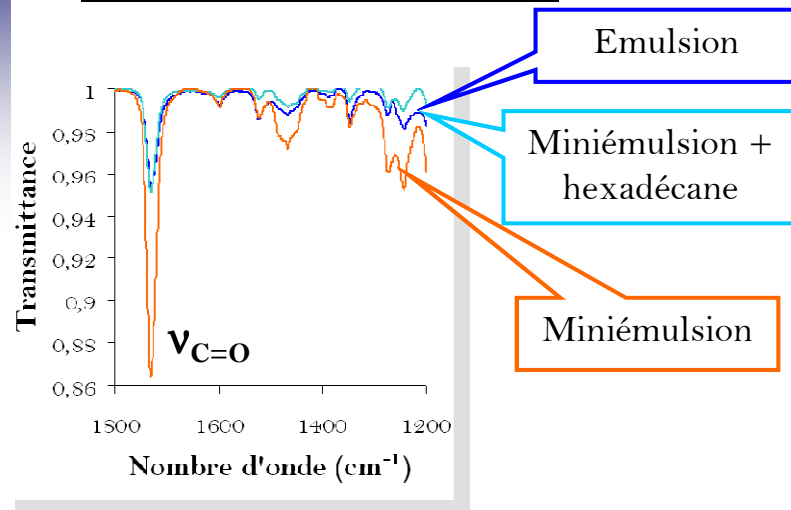
5 cycles (1 à 10)

$E_{\text{éq}}(0,5 \text{ V}) \rightarrow \{-0,4 \text{ V}; -1,0 \text{ V}\}$



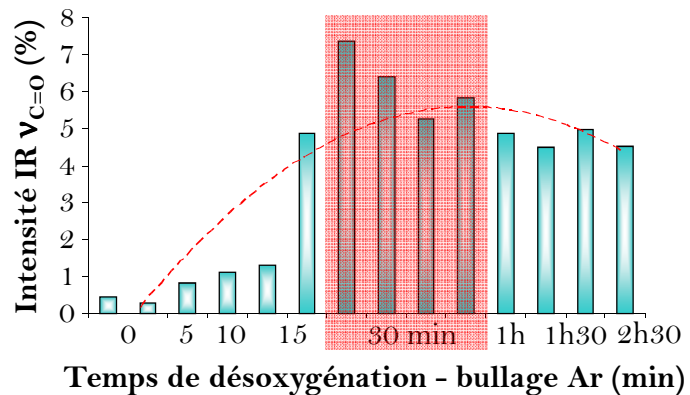
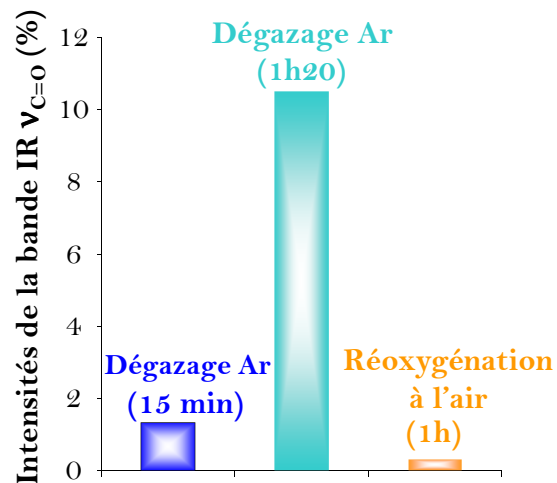
**Caractérisations  
des films**  
(IR, XPS, angle  
de contact, AFM)

### ① Émulsion / Miniémulsion



➡ **Miniémulsion**

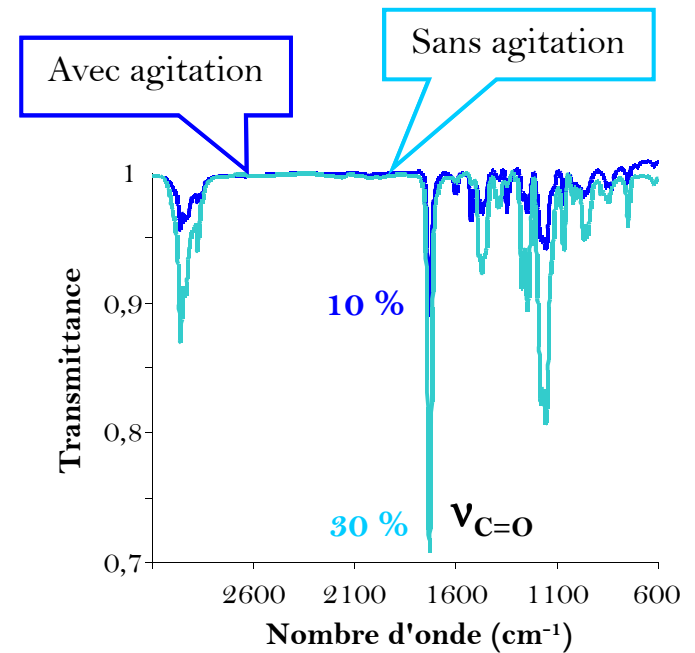
### ② Désoxygénation



➡ Miniémulsion

➡ Dégazage d'Argon (30 min)

### ③ Agitation du mélange réactionnel



⇒ Miniémulsion

⇒ Dégazage d'Argon (30 min)

⇒ Sans agitation

## I. Introduction

## II. SEEP: Principe & Protocole expérimental

## III. Résultats

### Caractérisation des films

- Densité / Compacité
- Composition chimique
- Structure moléculaire

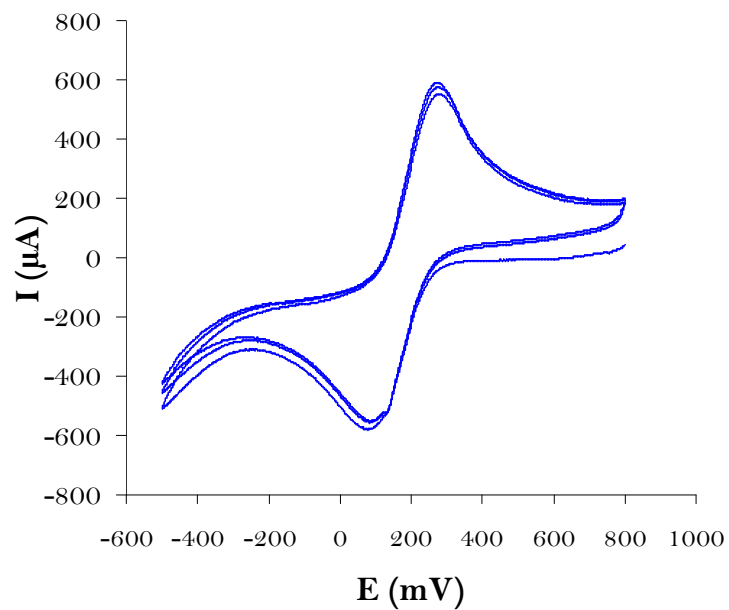
### Mécanisme

## IV. Discussion : Mécanisme de SEEP

## V. Application

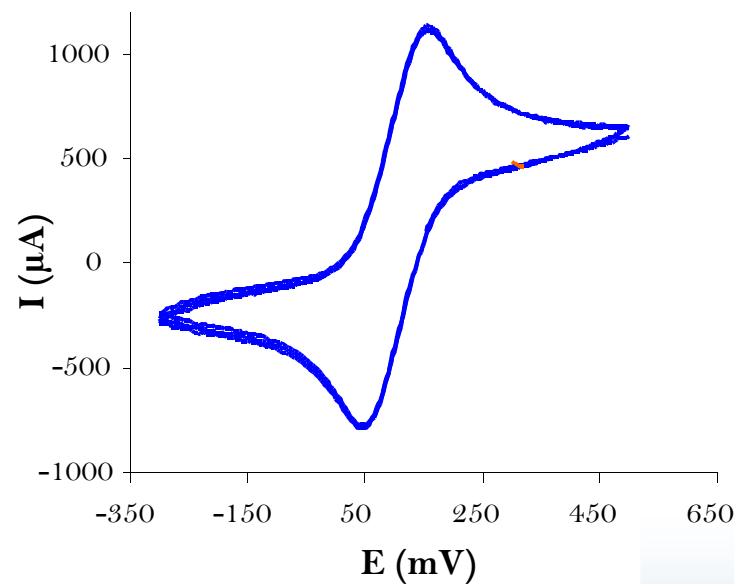
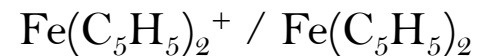
## VI. Conclusion

Milieu aqueux ( $H_2O$ )



Au vierge

Milieu organique ( $CH_3CN$ )

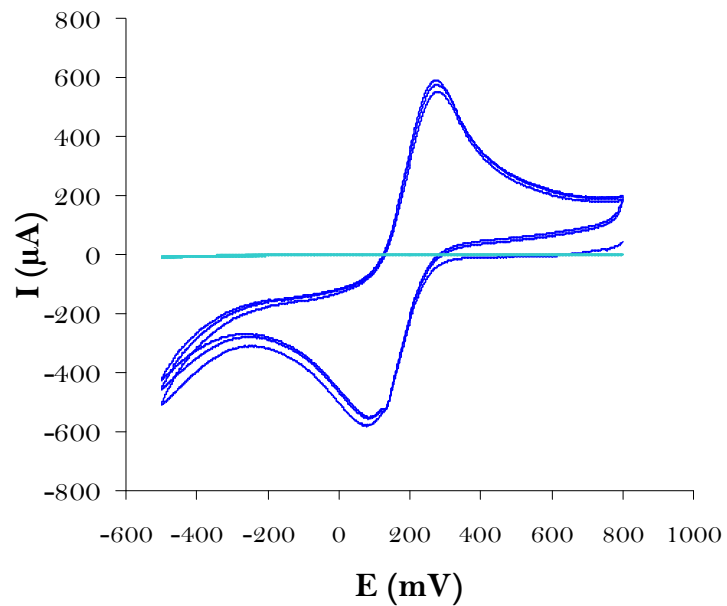


Au vierge

### III. Caractéristiques des films

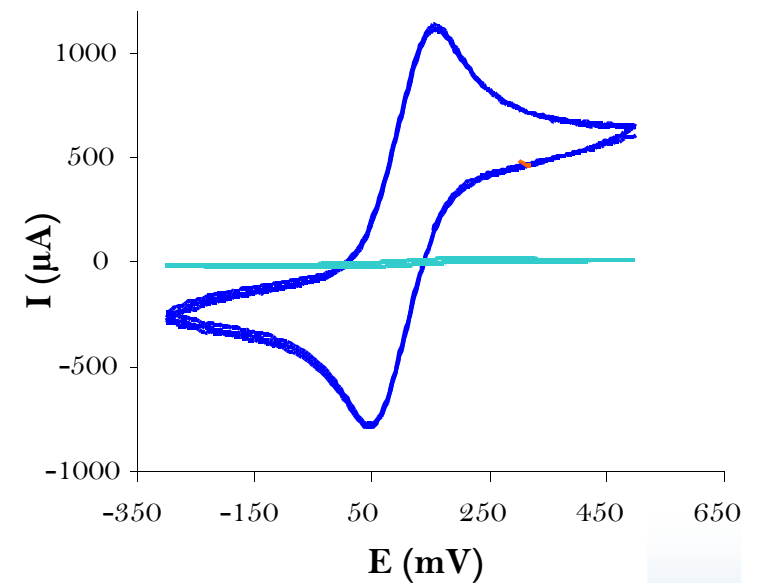
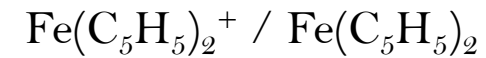
### Densité / Compacité

Milieu aqueux ( $\text{H}_2\text{O}$ )



Au vierge  
PBMA (5 cycles)

Milieu organique ( $\text{CH}_3\text{CN}$ )



Au vierge  
PBMA (5 cycles)

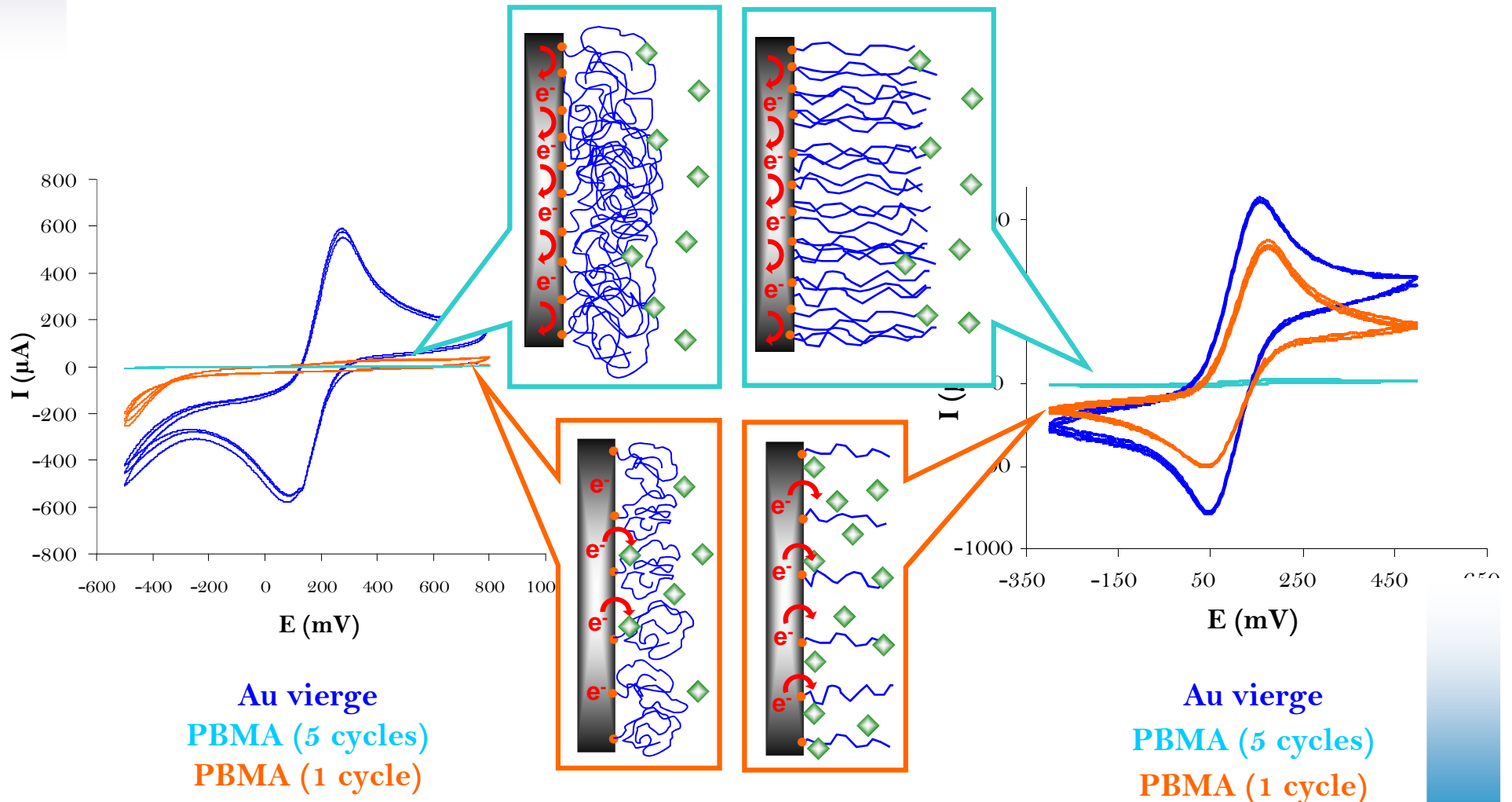
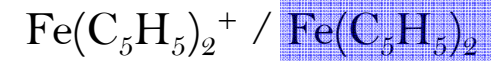
### III. Caractéristiques des films

### Densité / Compacité

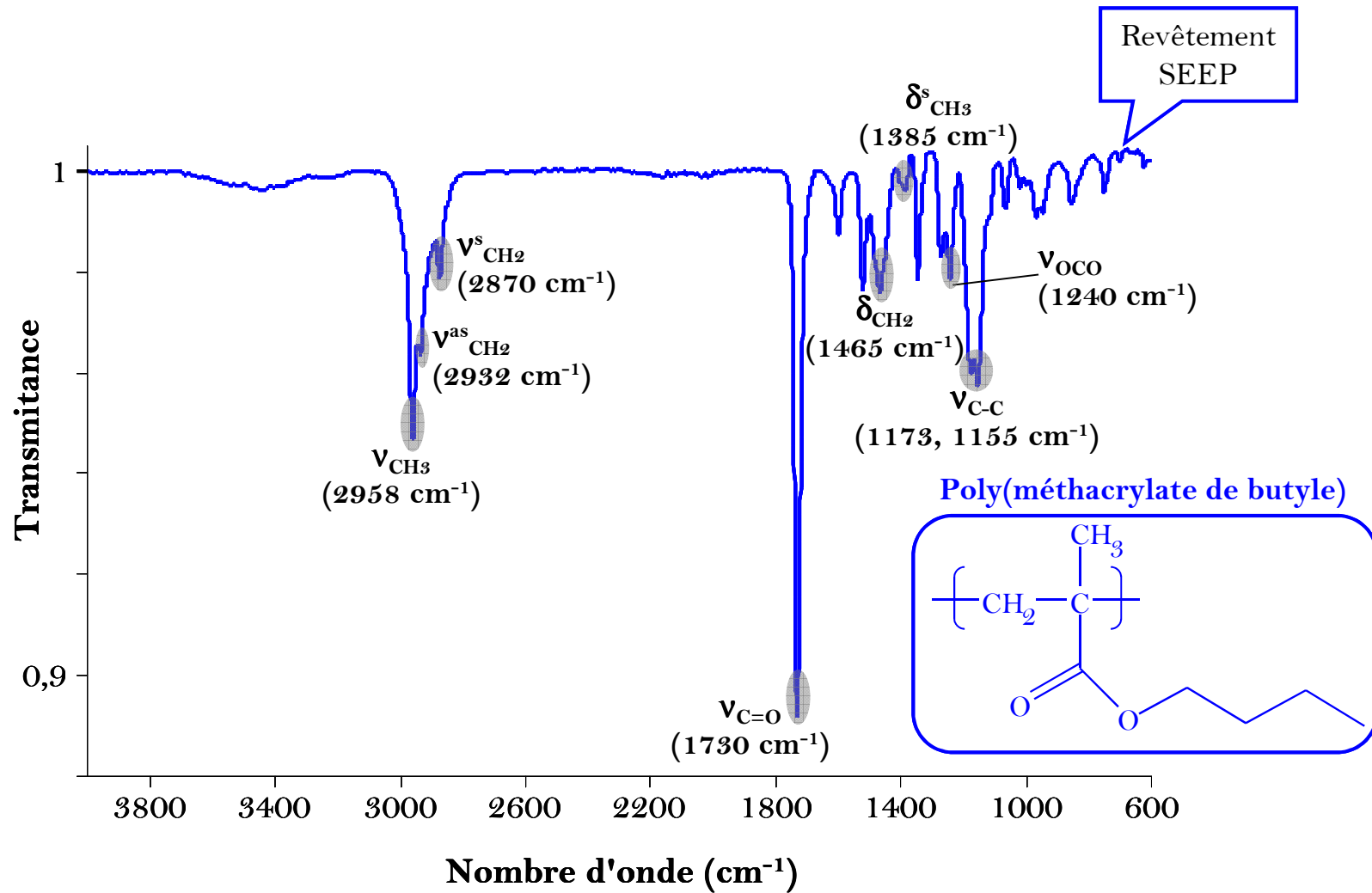
Milieu aqueux ( $H_2O$ )

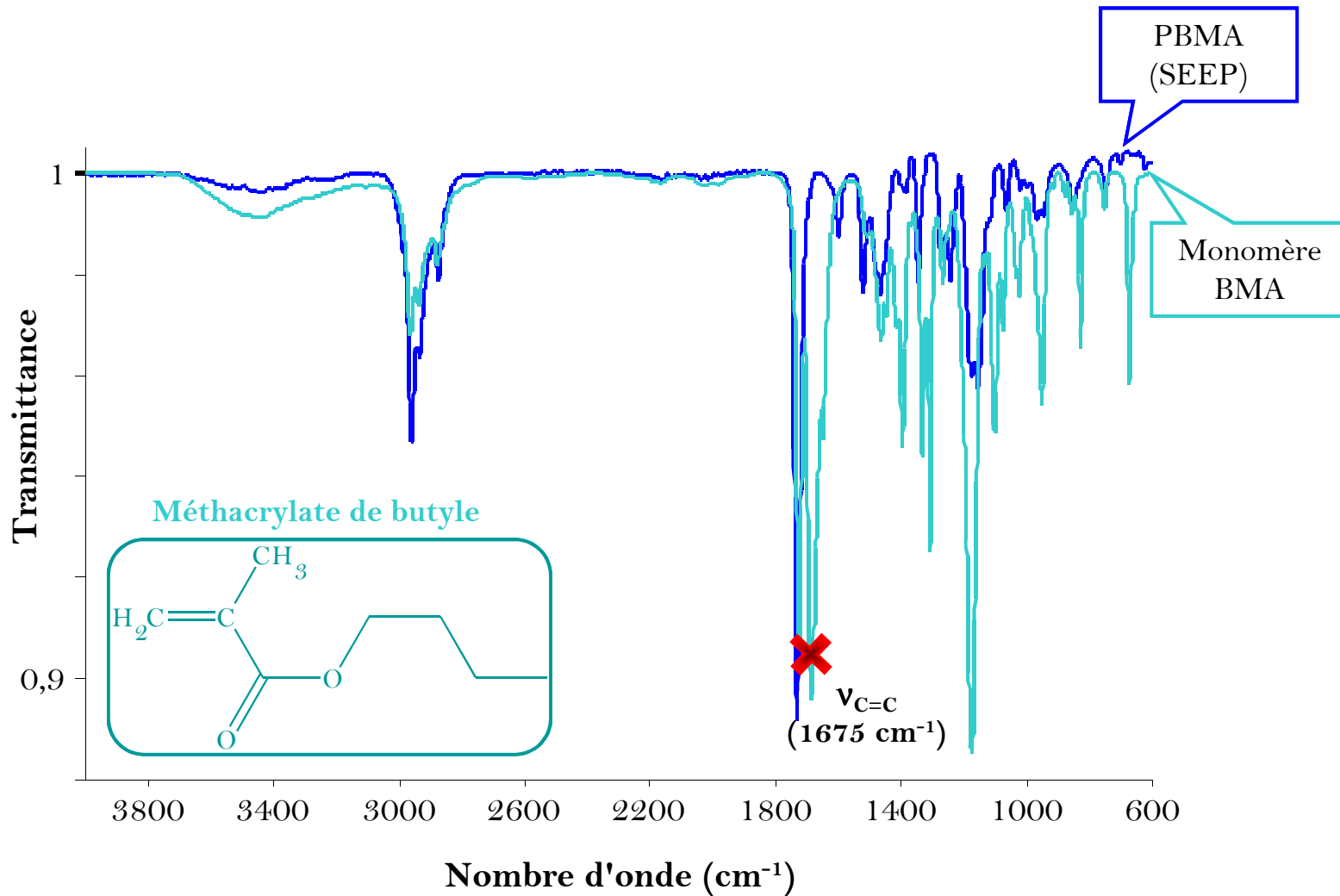


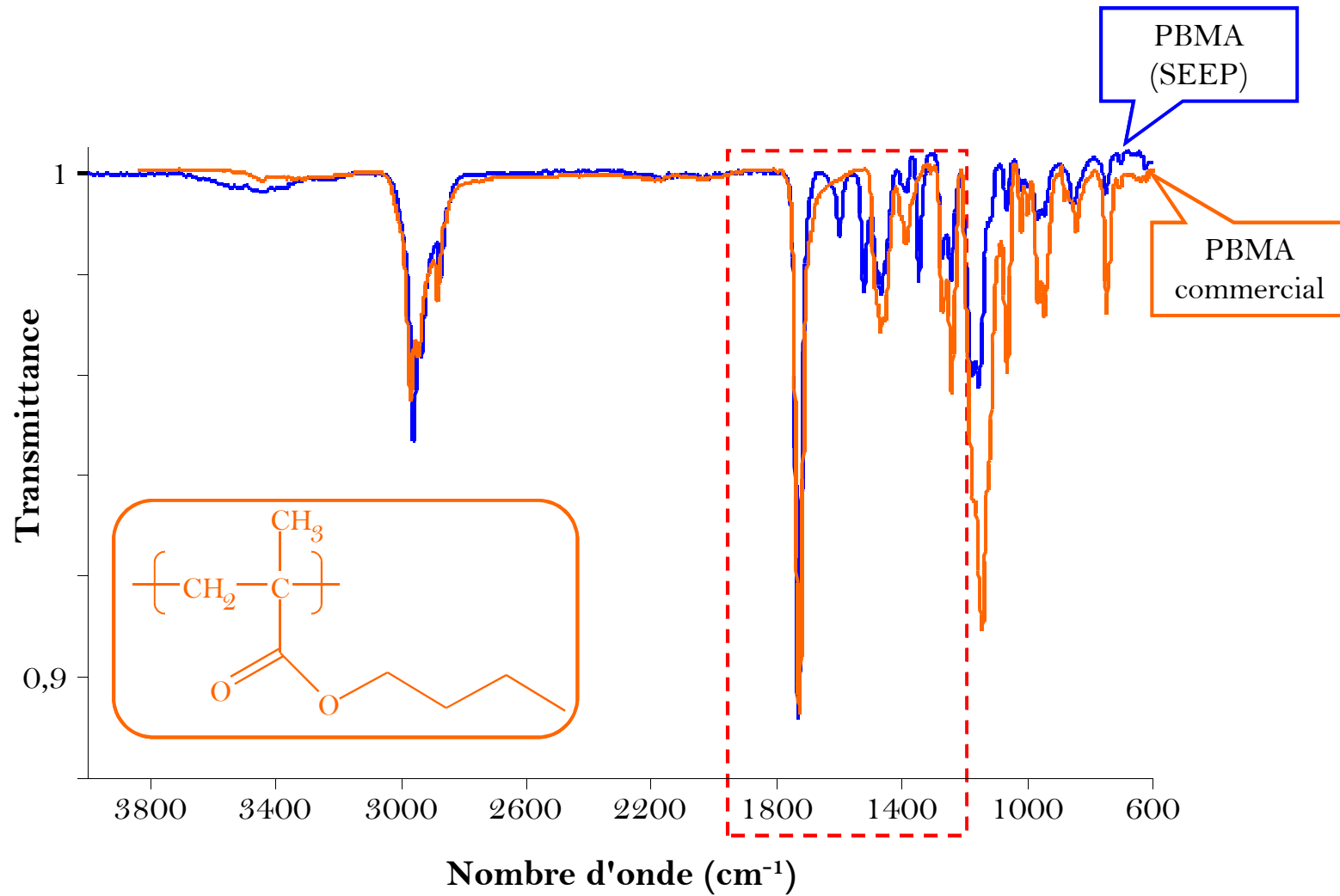
Milieu organique ( $CH_3CN$ )

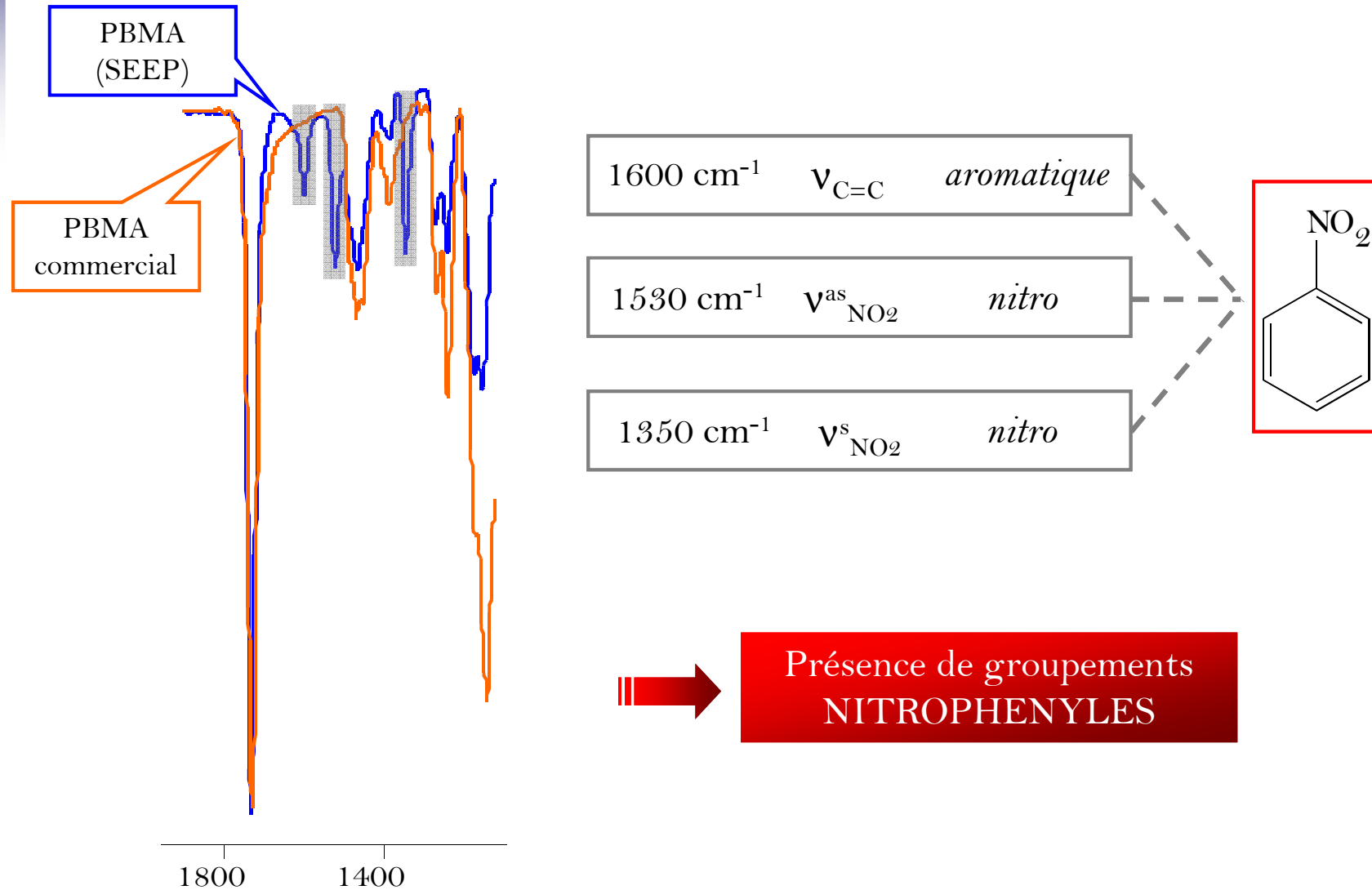












m/z Fragments  $\oplus$

29,0303  $C_2H_5$

27,0285  $C_2H_3$

41,0432  $C_3H_5$

43,0450  $C_3H_7$

55,0579  $C_4H_7$

57,0597  $C_4H_9$

69,0414  $C_4H_5O$

98,0405  $C_5H_6O_2$

141,0855  $C_8H_{13}O_2$

m/z Fragments  $\ominus$

25,0267  $C_2H$

41,0120  $C_2HO$

55,0267  $C_3H_3O$

73,0450  $C_4H_9O$

85,0268  $C_4H_5O_2$

107,0819  $C_7H_7O$

183,1297  $C_{11}H_{19}O_2$

269,1574  $C_{15}H_{25}O_4$

m/z Fragments  $\oplus$

29,0303  $C_2H_5$

27,0285  $C_2H_3$

41,0432  $C_3H_5$

43,0450  $C_3H_7$

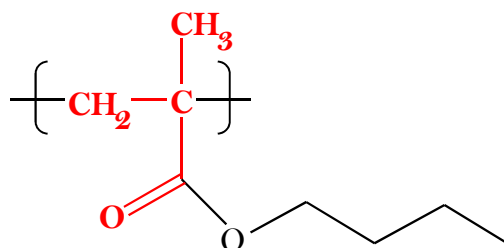
55,0579  $C_4H_7$

57,0597  $C_4H_9$

**69,0414  $C_4H_5O$**

98,0405  $C_5H_6O_2$

141,0855  $C_8H_{13}O_2$



m/z Fragments  $\ominus$

25,0267  $C_2H$

41,0120  $C_2HO$

55,0267  $C_3H_3O$

73,0450  $C_4H_9O$

85,0268  $C_4H_5O_2$

107,0819  $C_7H_7O$

183,1297  $C_{11}H_{19}O_2$

269,1574  $C_{15}H_{25}O_4$

### III. Composition chimique

### ToF-SIMS (spectroscopie de surface)

m/z Fragments  $\oplus$

29,0303  $C_2H_5$

27,0285  $C_2H_3$

41,0432  $C_3H_5$

43,0450  $C_3H_7$

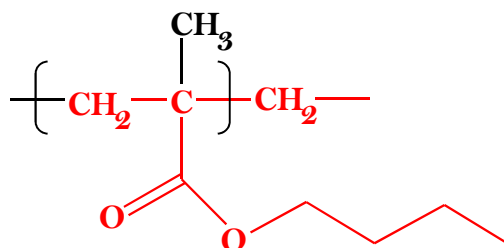
55,0579  $C_4H_7$

57,0597  $C_4H_9$

69,0414  $C_4H_5O$

98,0405  $C_5H_6O_2$

**141,0855  $C_8H_{13}O_2$**



m/z Fragments  $\ominus$

25,0267  $C_2H$

41,0120  $C_2HO$

55,0267  $C_3H_3O$

73,0450  $C_4H_9O$

85,0268  $C_4H_5O_2$

107,0819  $C_7H_7O$

183,1297  $C_{11}H_{19}O_2$

269,1574  $C_{15}H_{25}O_4$

### III. Composition chimique

### ToF-SIMS (spectroscopie de surface)

m/z Fragments  $\oplus$

29,0303  $C_2H_5$

27,0285  $C_2H_3$

41,0432  $C_3H_5$

43,0450  $C_3H_7$

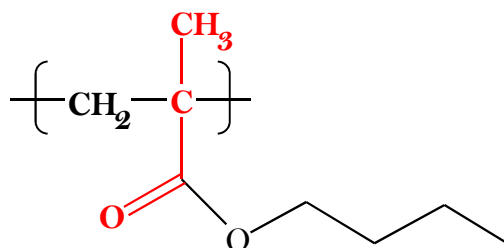
55,0579  $C_4H_7$

57,0597  $C_4H_9$

69,0414  $C_4H_5O$

98,0405  $C_5H_6O_2$

141,0855  $C_8H_{13}O_2$



m/z Fragments  $\ominus$

25,0267  $C_2H$

41,0120  $C_2HO$

**55,0267  $C_3H_3O$**

73,0450  $C_4H_9O$

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183,1297  $C_{11}H_{19}O_2$

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### III. Composition chimique

### ToF-SIMS (spectroscopie de surface)

m/z Fragments  $\oplus$

29,0303  $C_2H_5$

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41,0432  $C_3H_5$

43,0450  $C_3H_7$

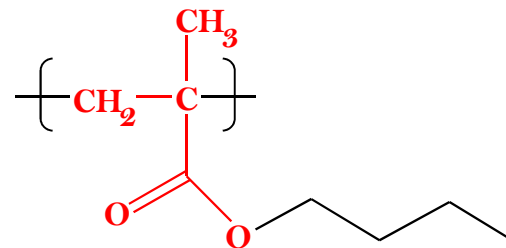
55,0579  $C_4H_7$

57,0597  $C_4H_9$

69,0414  $C_4H_5O$

98,0405  $C_5H_6O_2$

141,0855  $C_8H_{13}O_2$



Structure du  
PBMA

m/z Fragments  $\ominus$

25,0267  $C_2H$

41,0120  $C_2HO$

55,0267  $C_3H_3O$

73,0450  $C_4H_9O$

**85,0268  $C_4H_5O_2$**

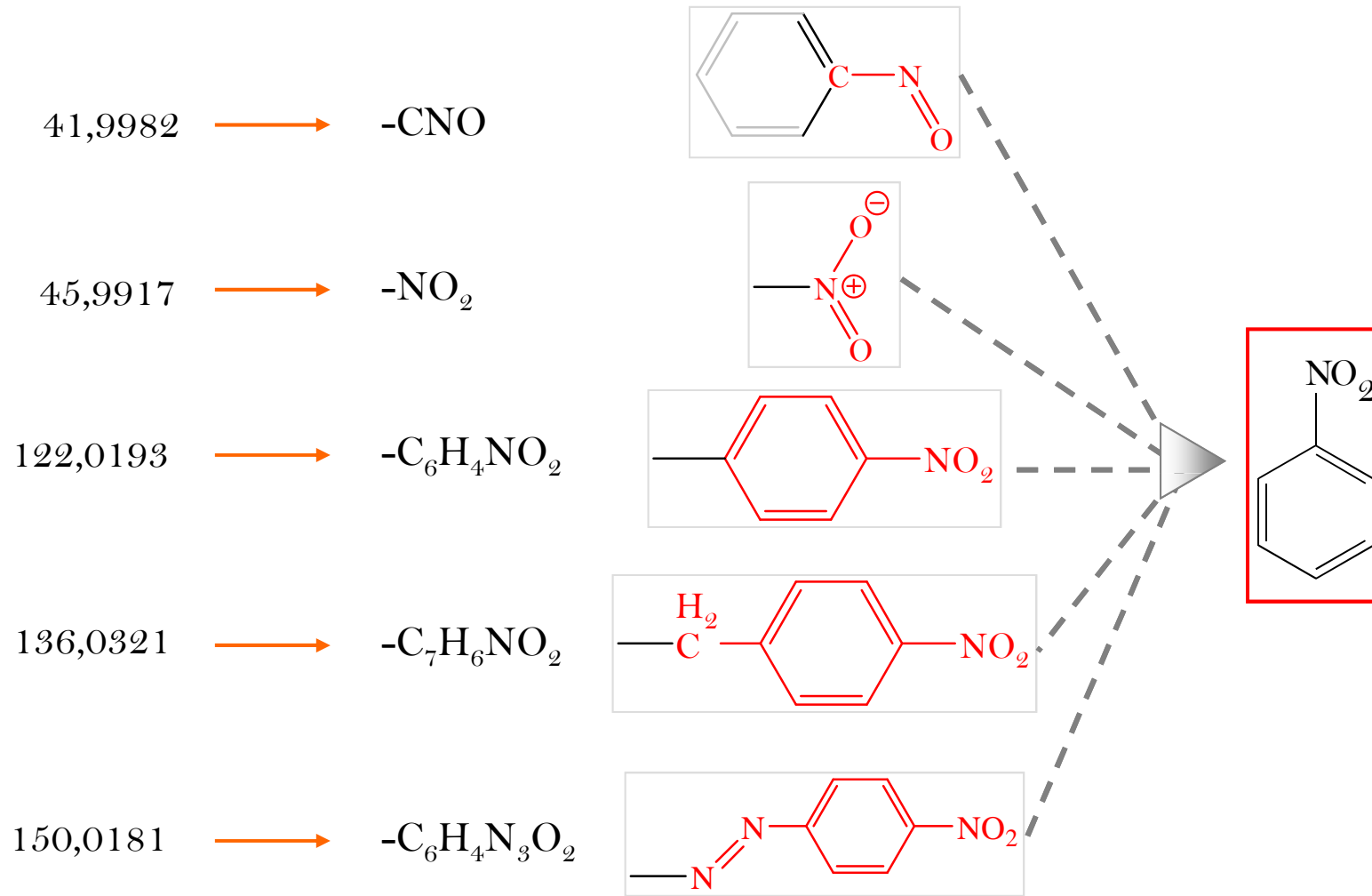
107,0819  $C_7H_7O$

183,1297  $C_{11}H_{19}O_2$

269,1574  $C_{15}H_{25}O_4$

### III. Composition chimique

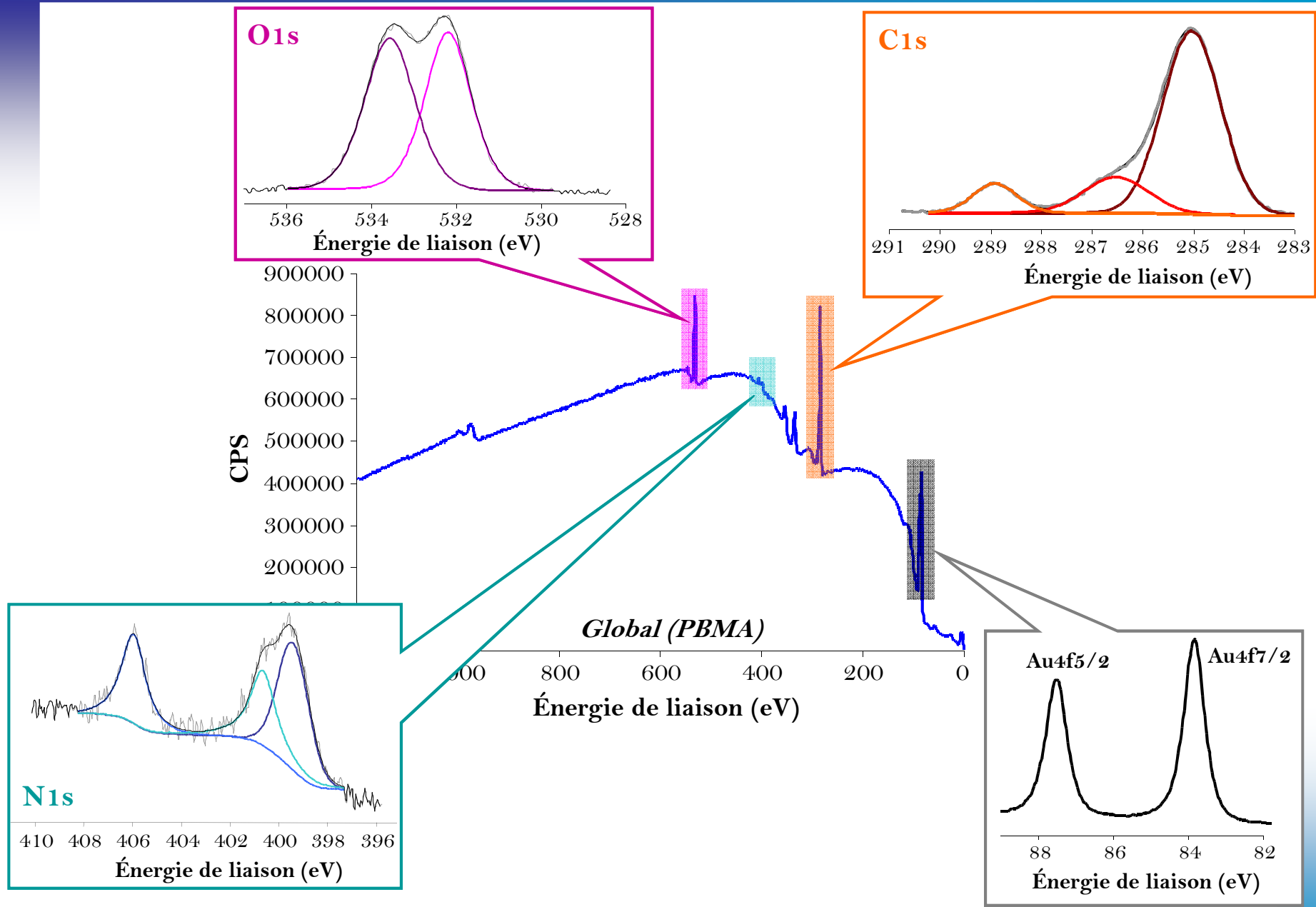
### ToF-SIMS (spectroscopie de surface)

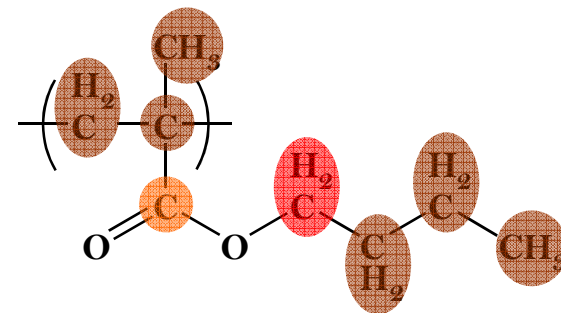
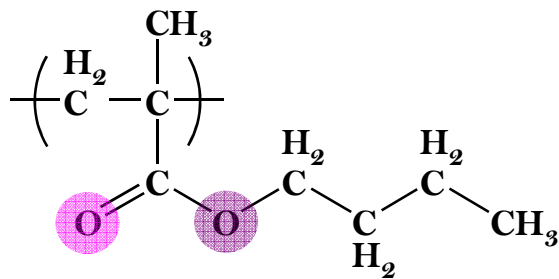
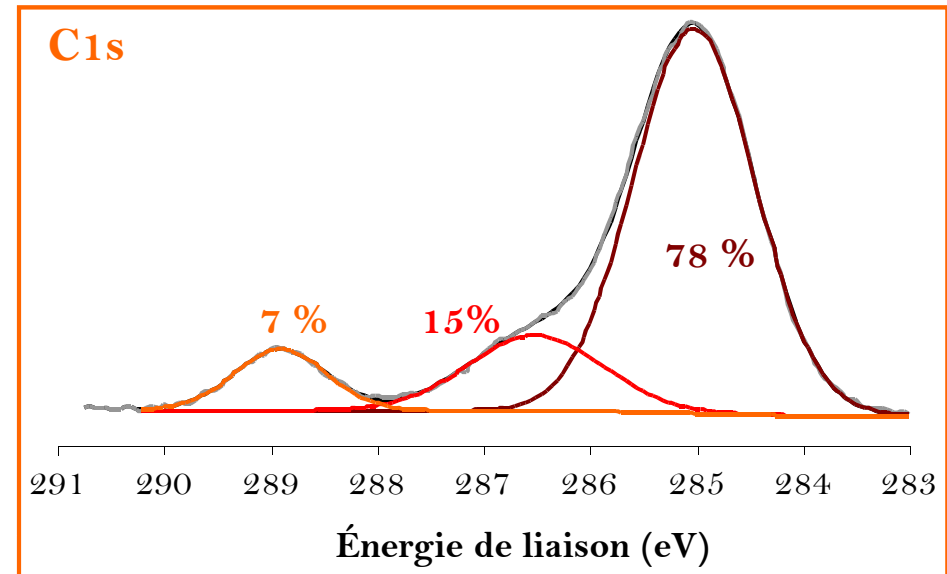
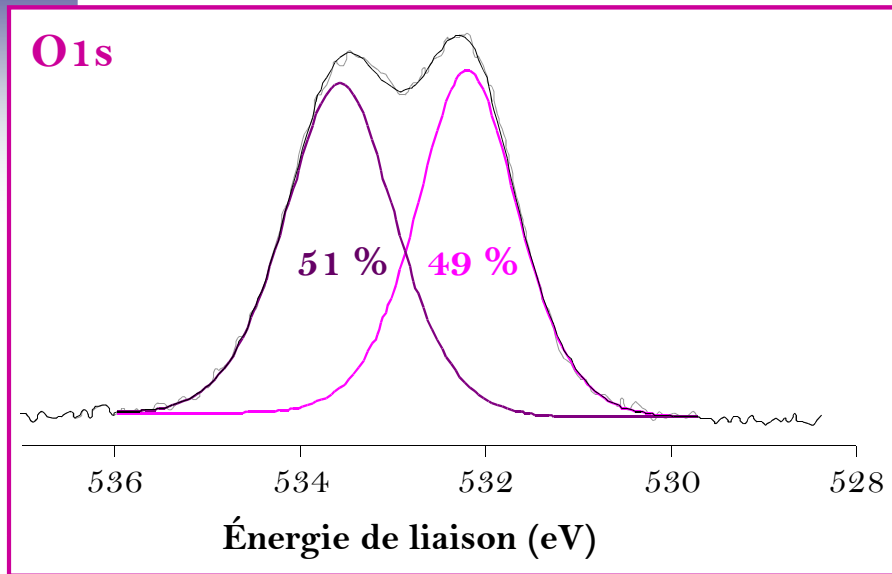


Présence de groupements NITROPHENYLES

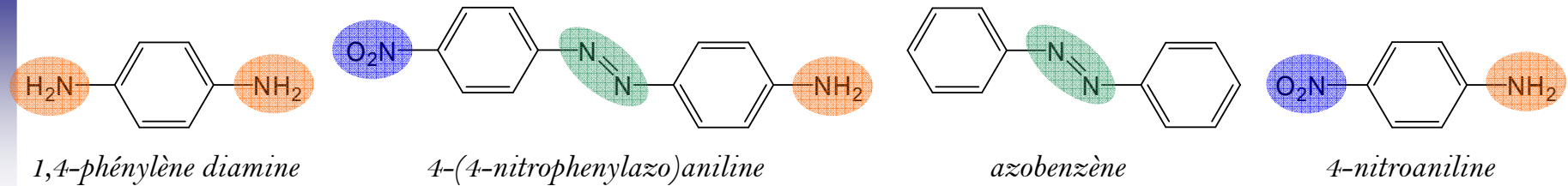
### III. Composition chimique

### Spectroscopie de photoélec. X (XPS)





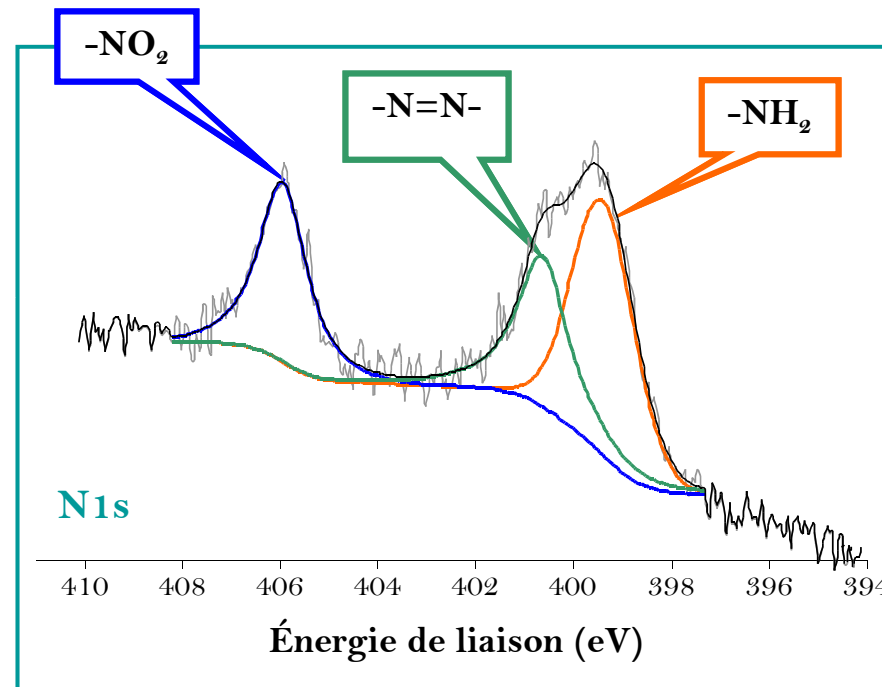
Composition du PBMA



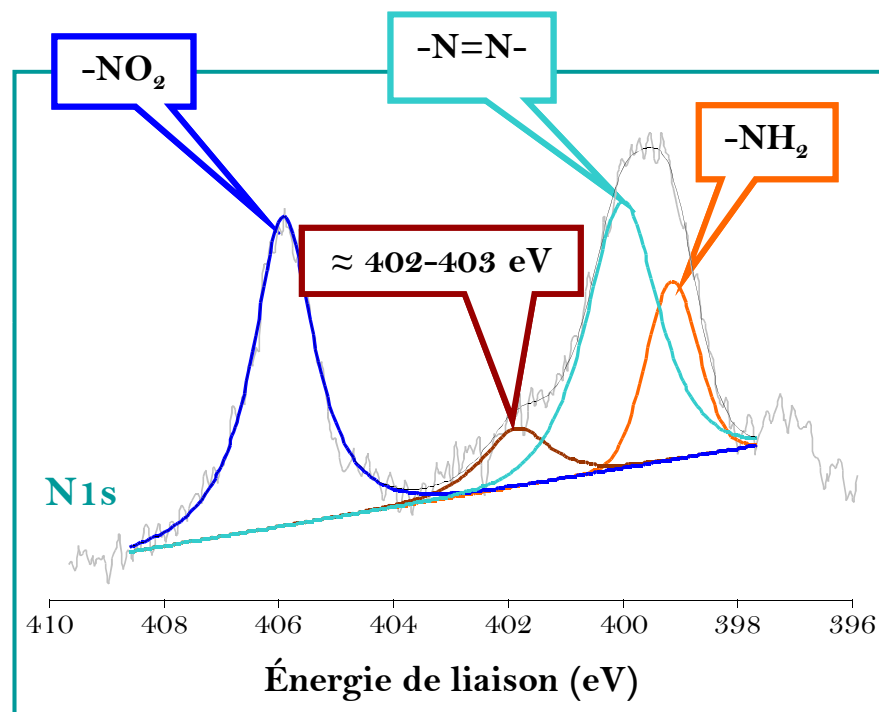
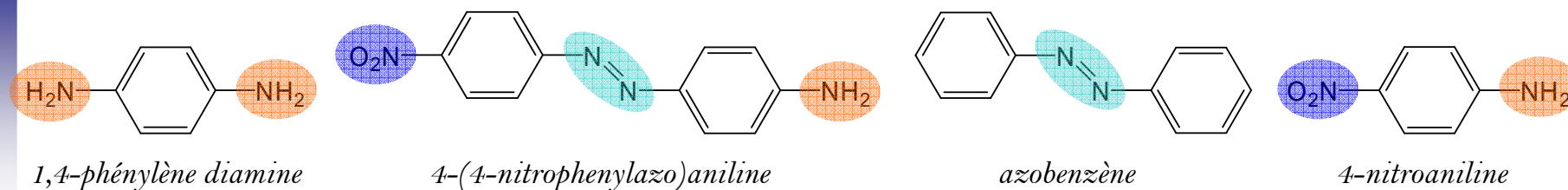
405,95 eV

400,64 eV

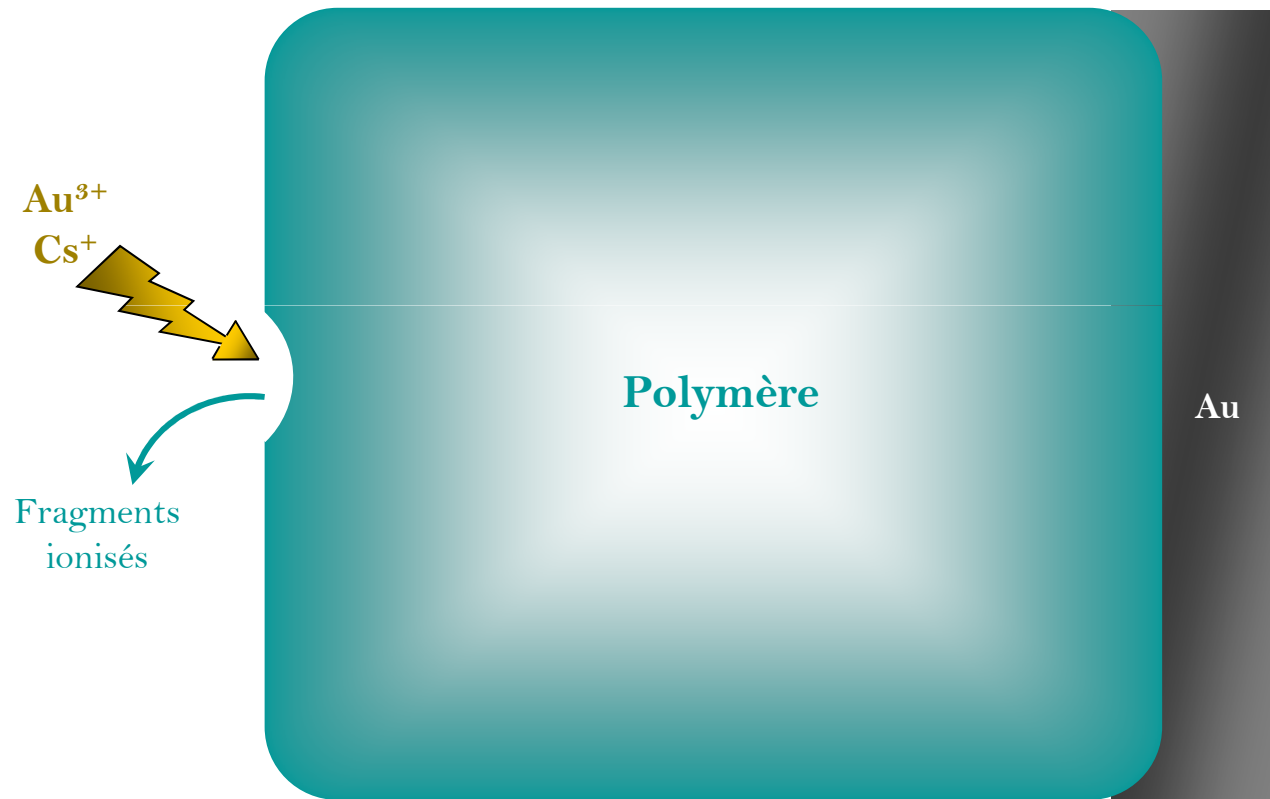
399,41 eV

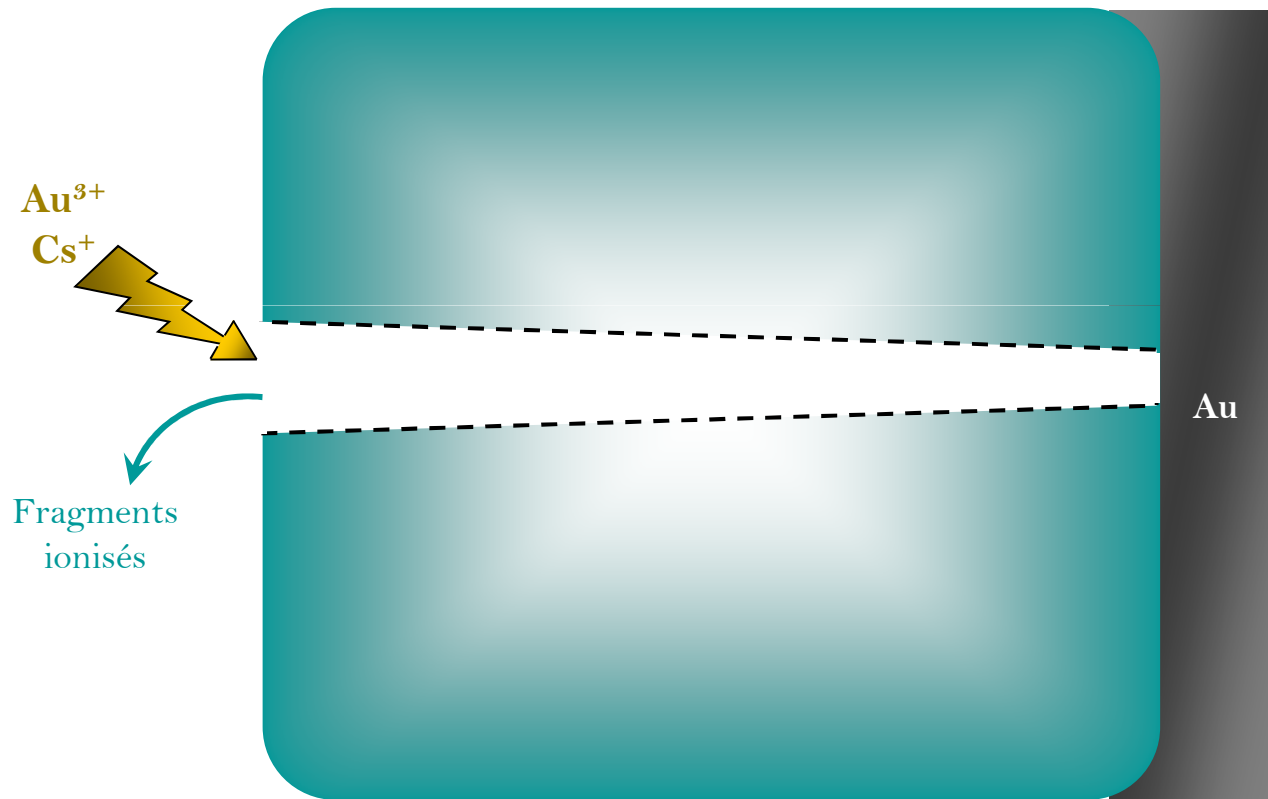


- NO<sub>2</sub> → nitrobenzene diazonium
- NH<sub>2</sub> → réduction des NO<sub>2</sub> en NH<sub>2</sub> (électrochimie ou XPS)
- N=N → mécanisme controversé (3 propositions)

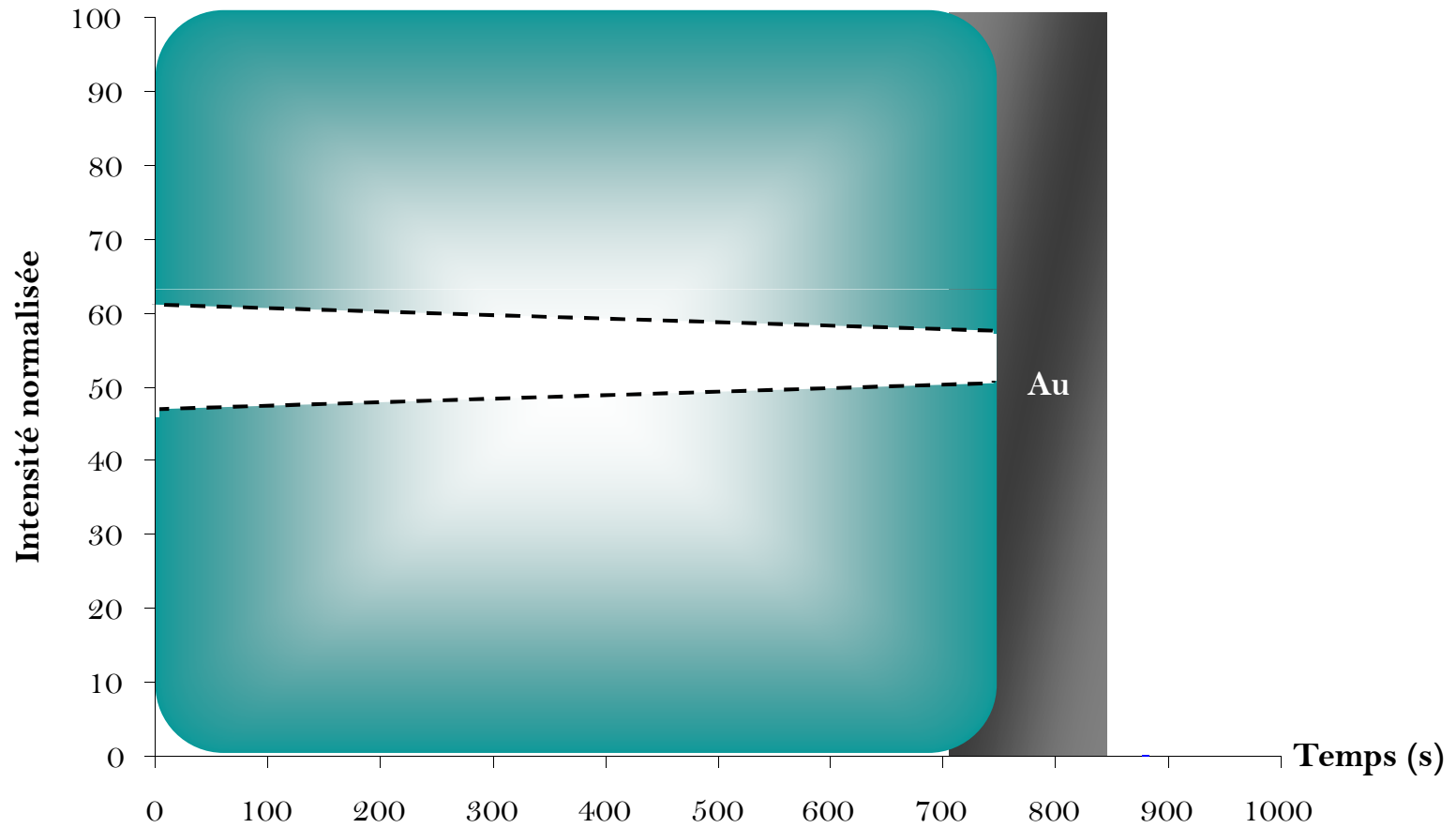


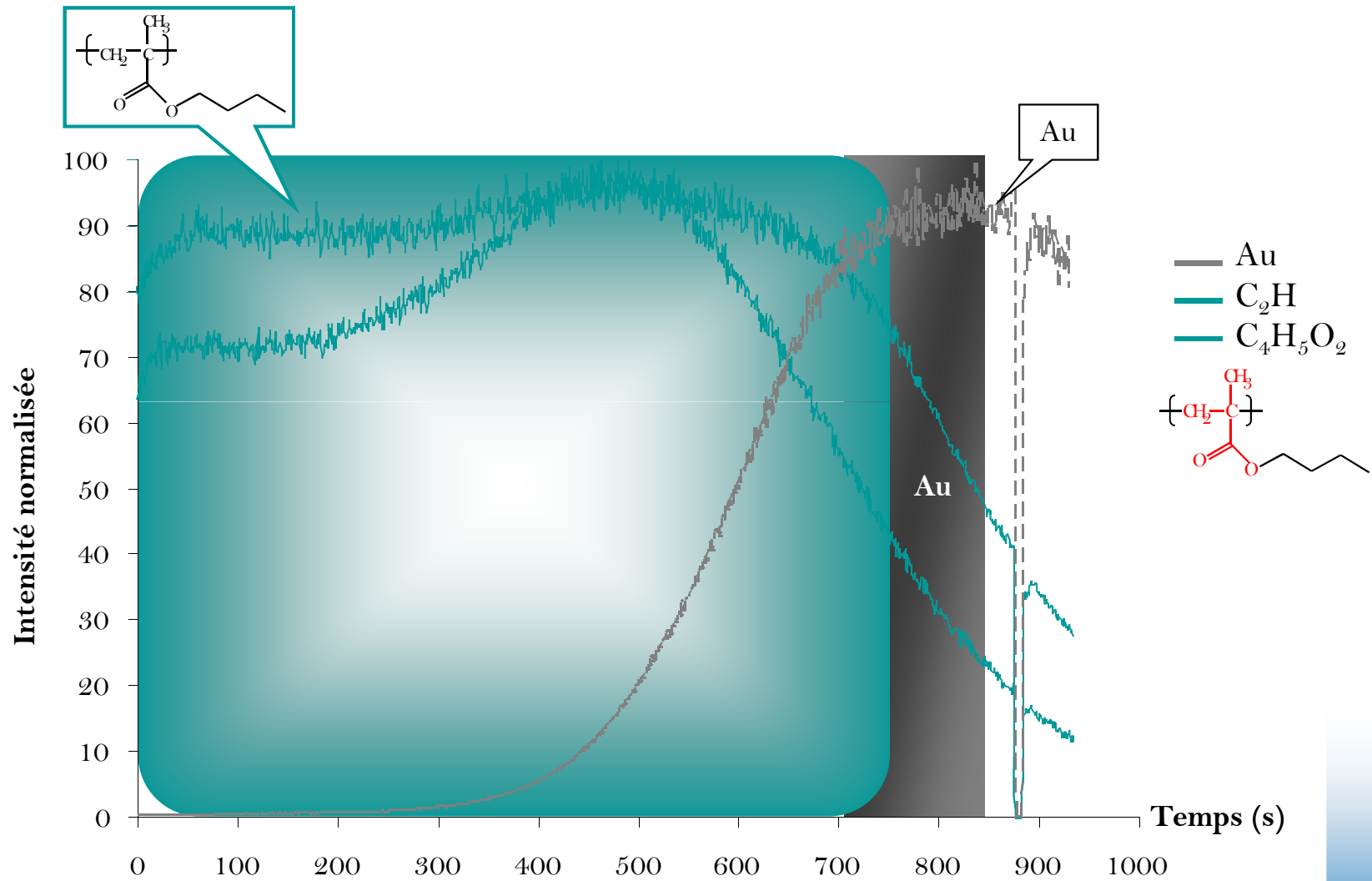
- $\text{NH}_3^+$  → Protonation des amines
- $\text{NHOH} / \text{N=O}$  → Produits intermédiaires de la réduction des  $\text{NO}_2$  en  $\text{NH}_2$

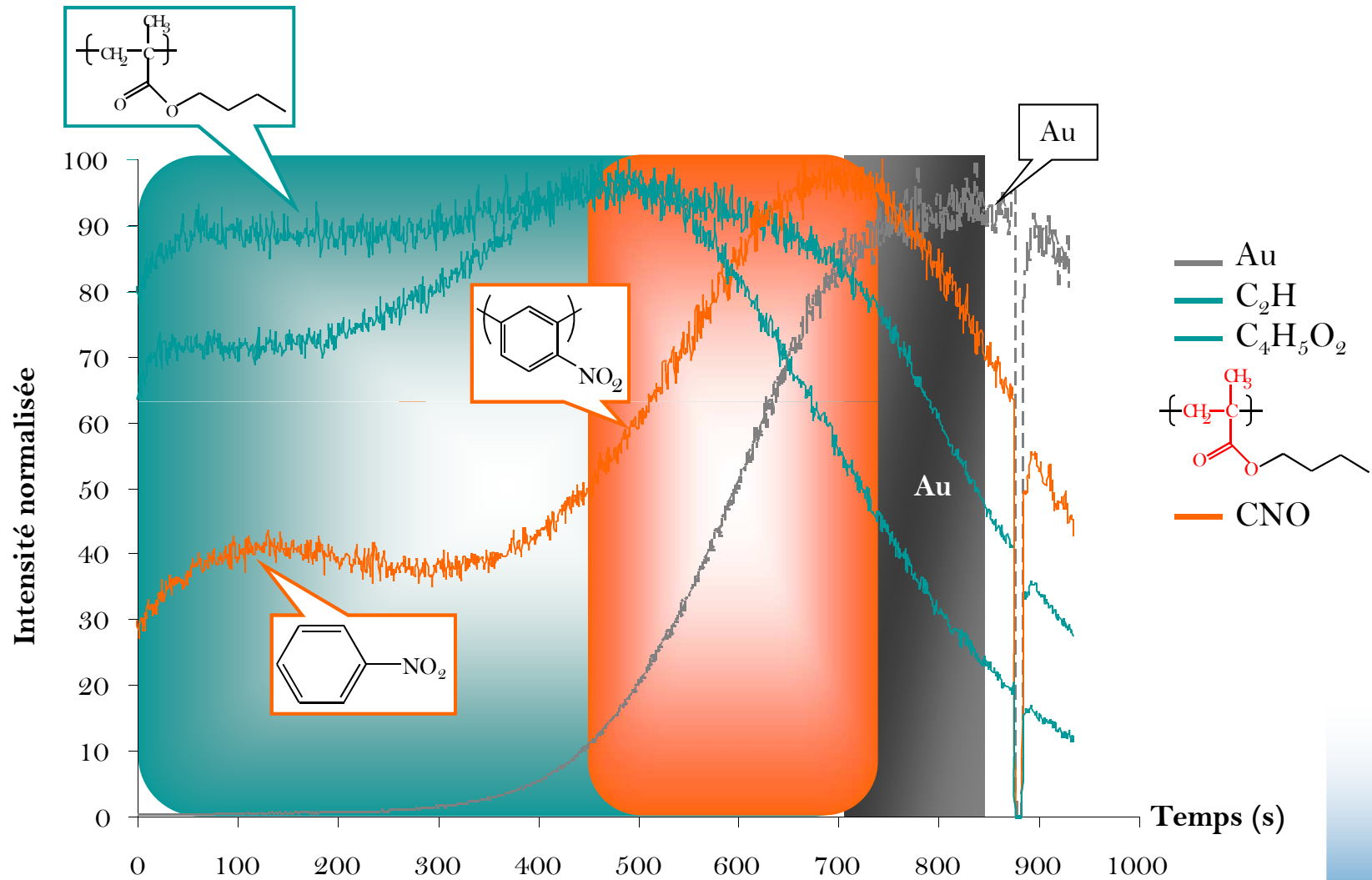


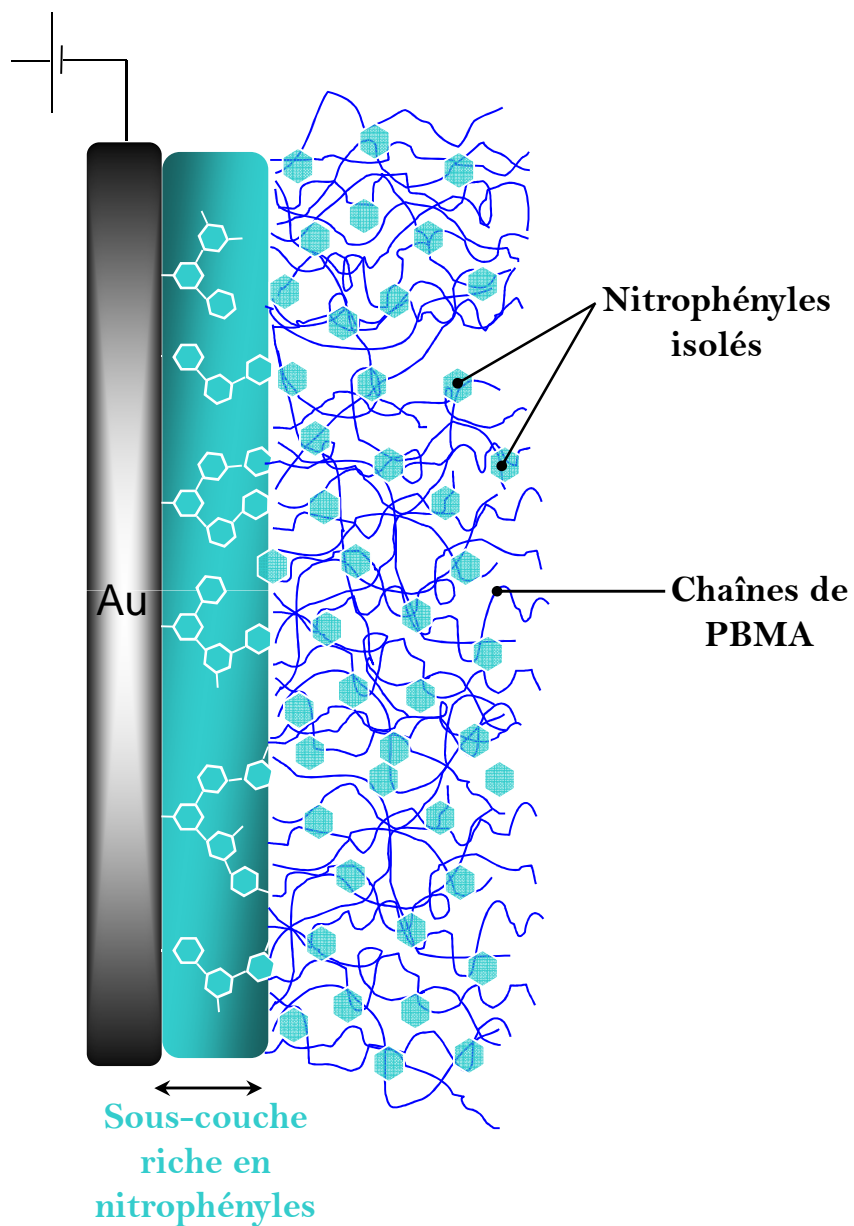












## I. Introduction

## II. SEEP: Principe & Protocole expérimental

## III. Résultats

Caractérisation des films

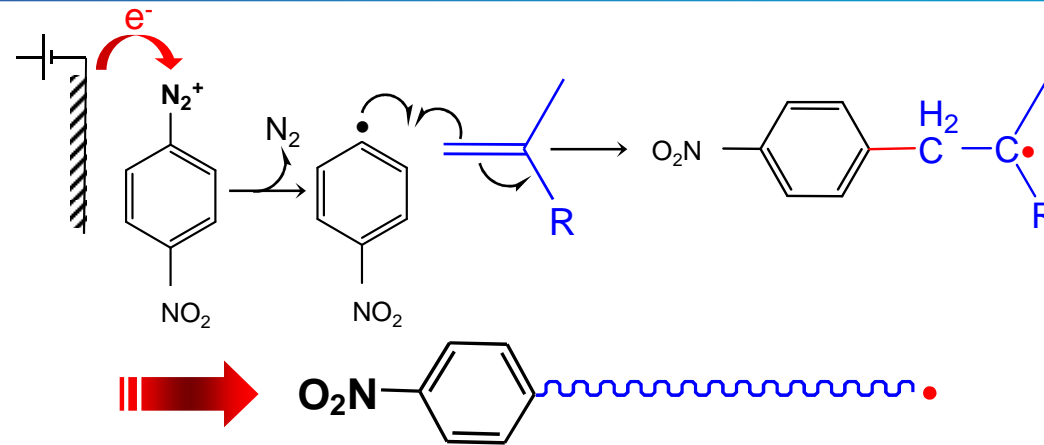
Mécanisme

## IV. Discussion : Mécanisme de SEEP

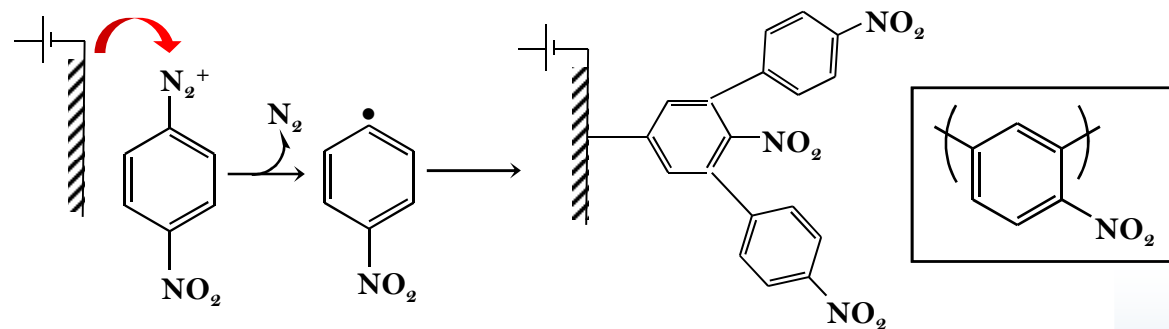
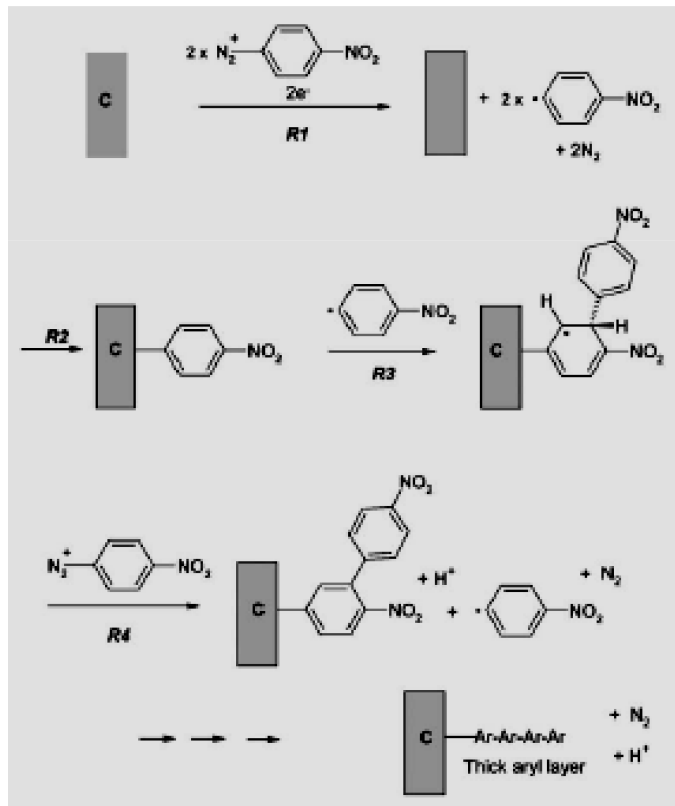
## V. Application

## VI. Conclusion

① Amorceurs de polymérisation radicalaire

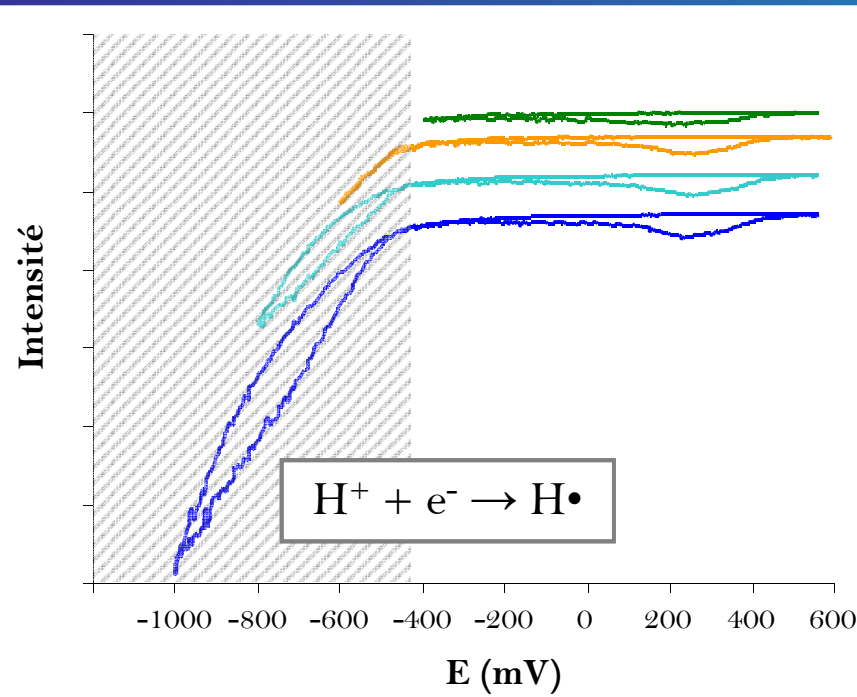


② Electrogreffage des sels d'aryldiazonium  
→ films de polyaryles



- Milieu organique ( $\text{CH}_3\text{CN}$ )
- Milieu acide (= conditions SEEP)

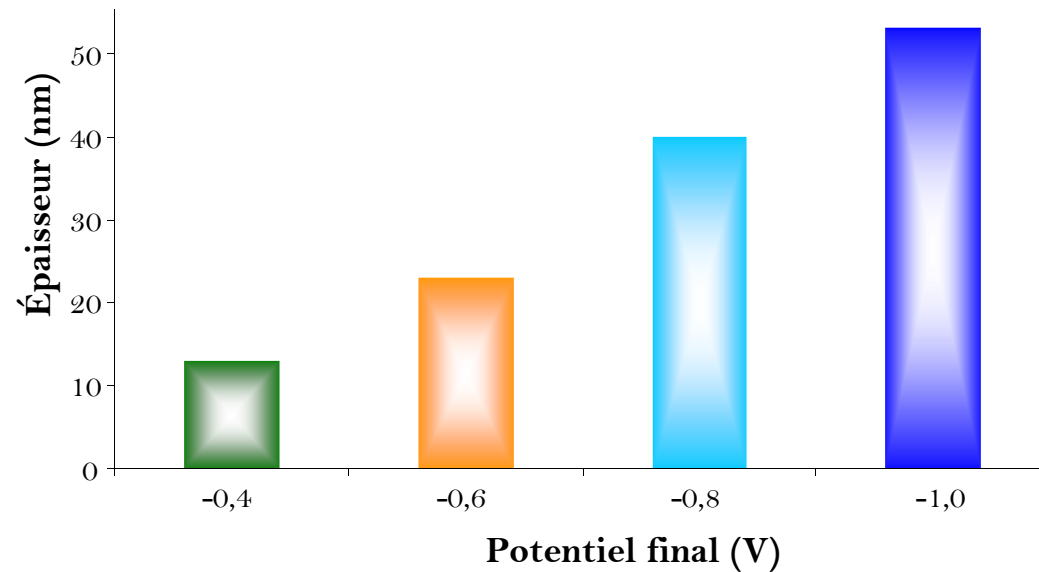
Polynitrophenylene  
(PNP)



Influence du  
potentiel final

5 cycles  
10 mV/s

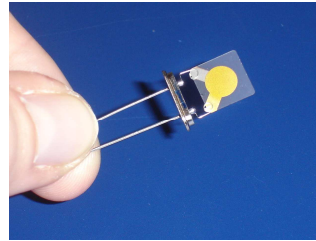
⇒ Importance de la  
réduction des  
protons



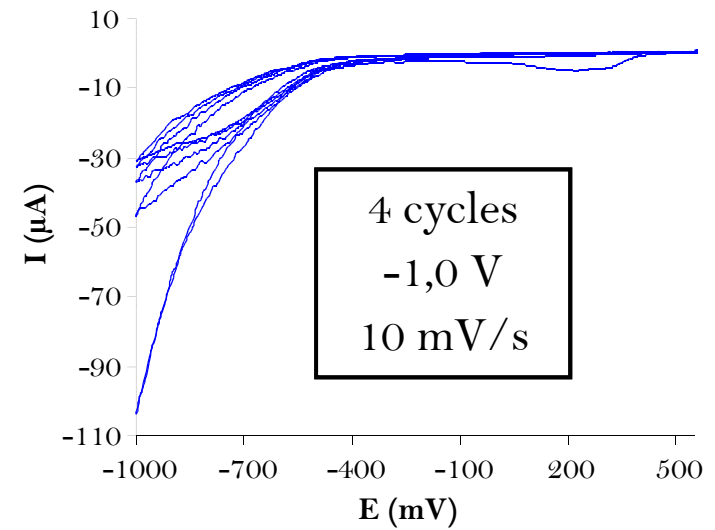
## IV. Résultats

Microbalance à  
Cristal de Quartz  
(MCQ)

$$\Delta f = \frac{-2\Delta m f_0^2}{A\sqrt{\rho_q \mu_q}}$$

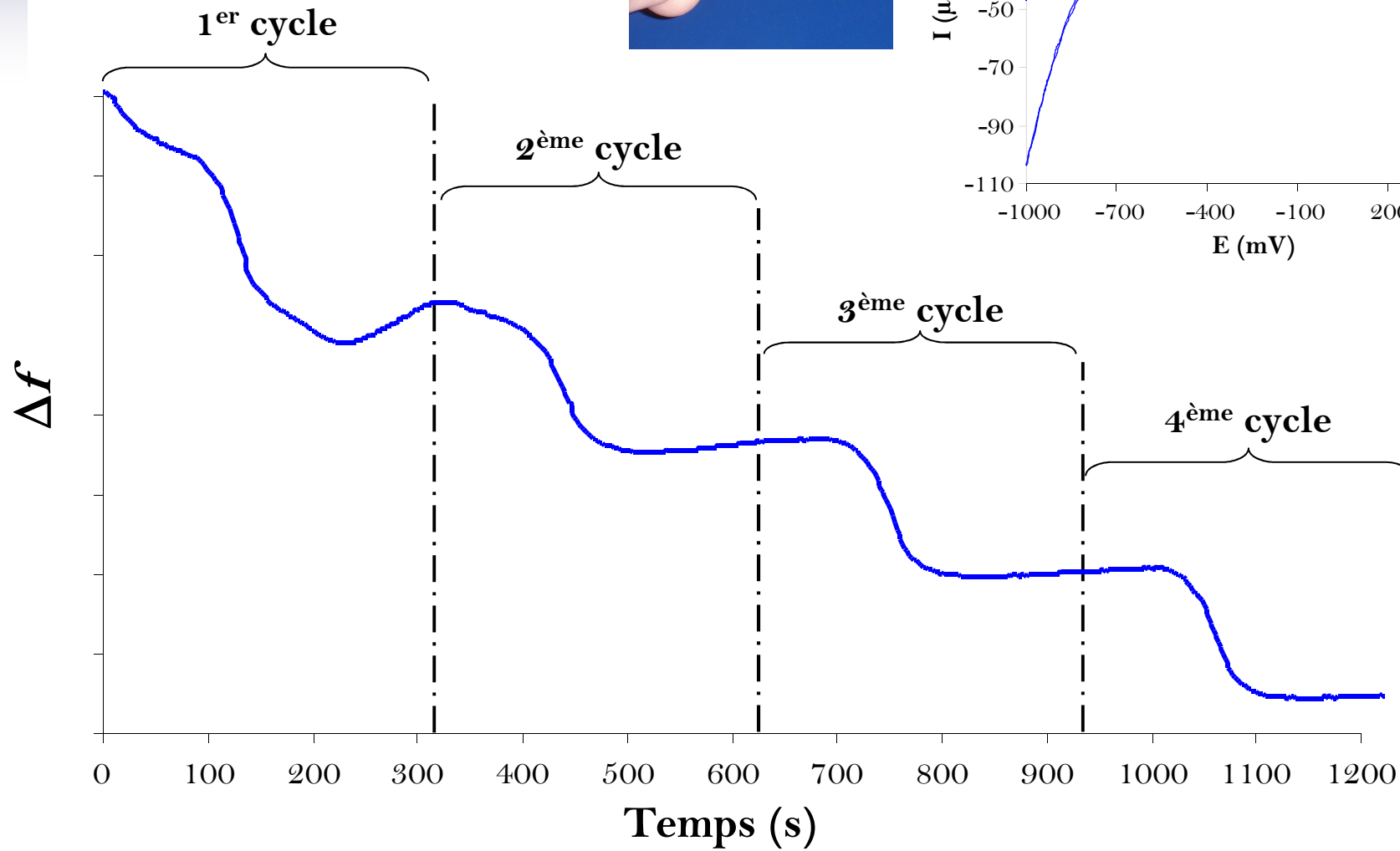
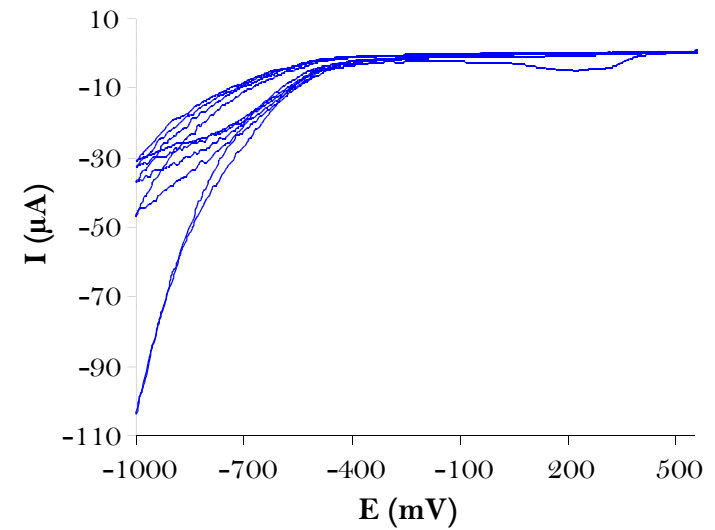
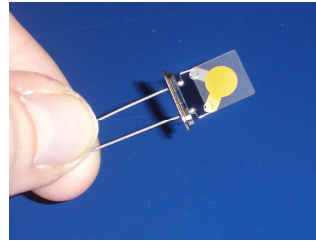


## Rôle des protons



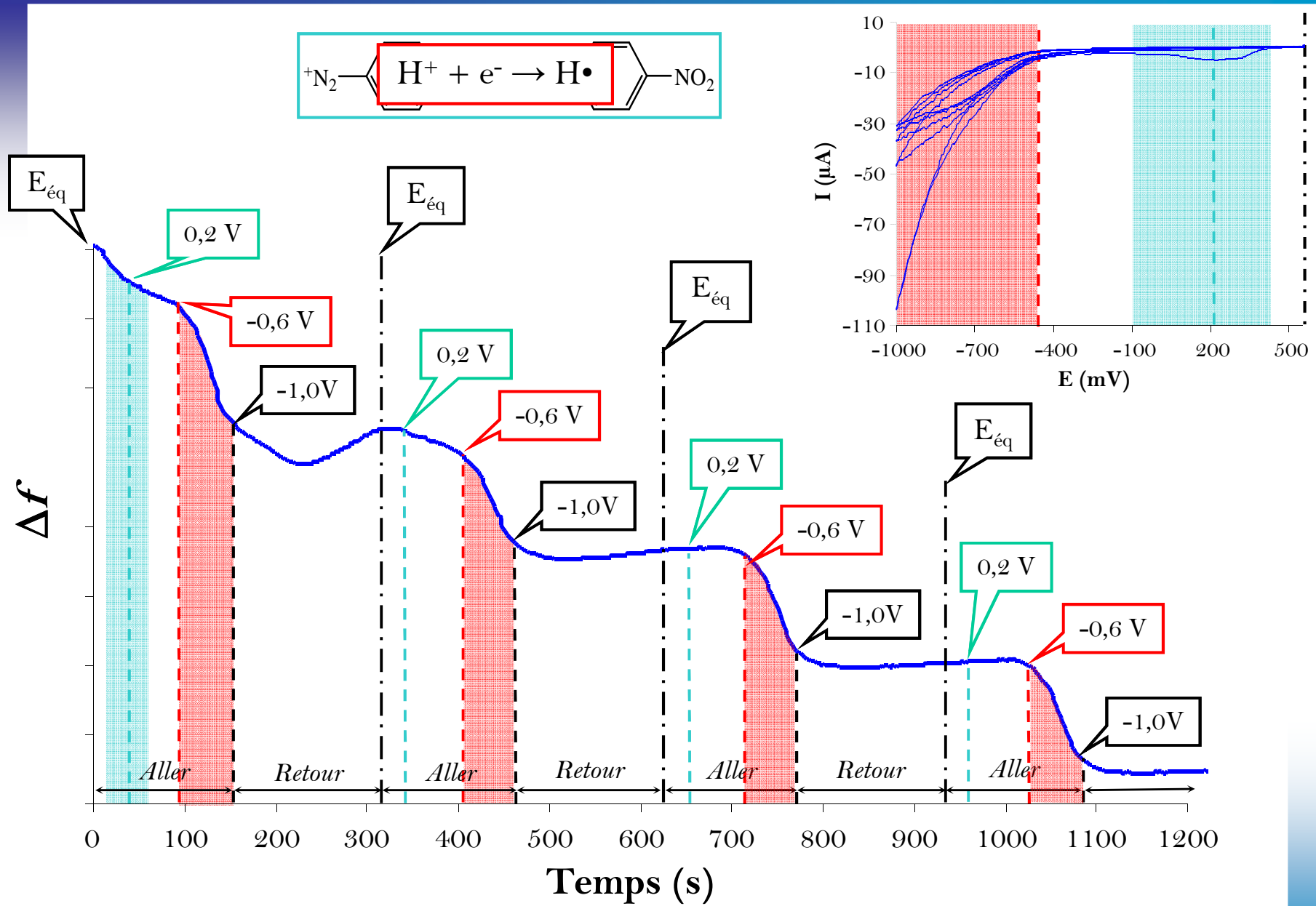


$$\Delta f = \frac{-2\Delta m f_0^2}{A\sqrt{\rho_q \mu_q}}$$

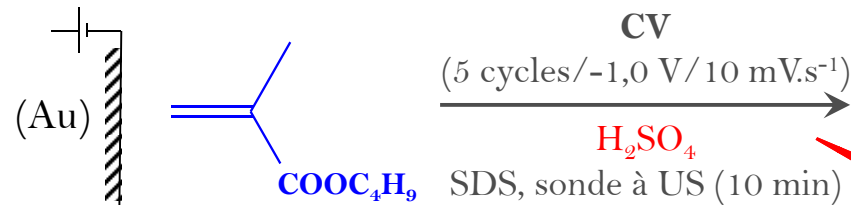


# IV. Résultats

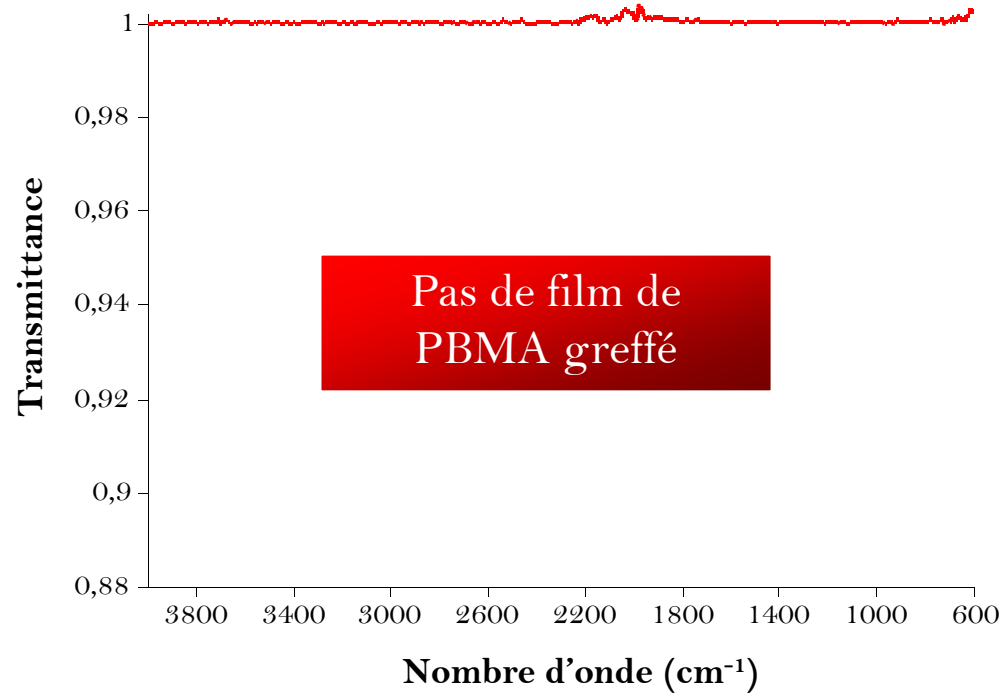
# Rôle des protons



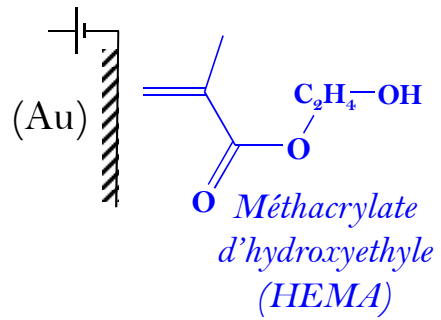
## Miniémulsion de BMA sans sel de diazonium



**SANS sel de diazonium**



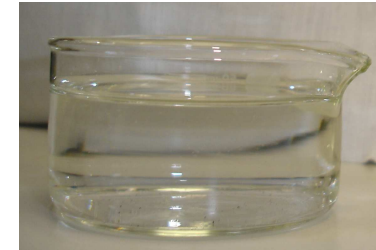
## Solution de HEMA sans sel de diazonium



Chronoamperométrie  
 (-1,0 V/15h)



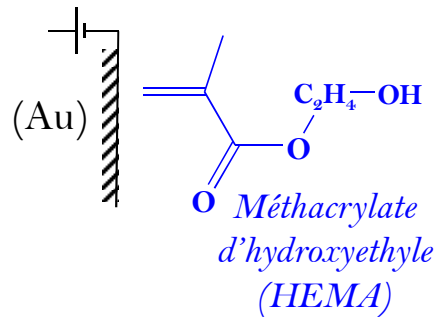
SANS sel de  
 diazonium



Evaporation sous vide (20°C)  
 ≈ 25h à 1-3.10<sup>-1</sup> mbar



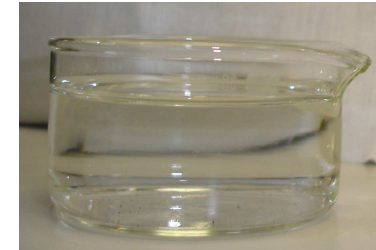
## Solution de HEMA sans sel de diazonium



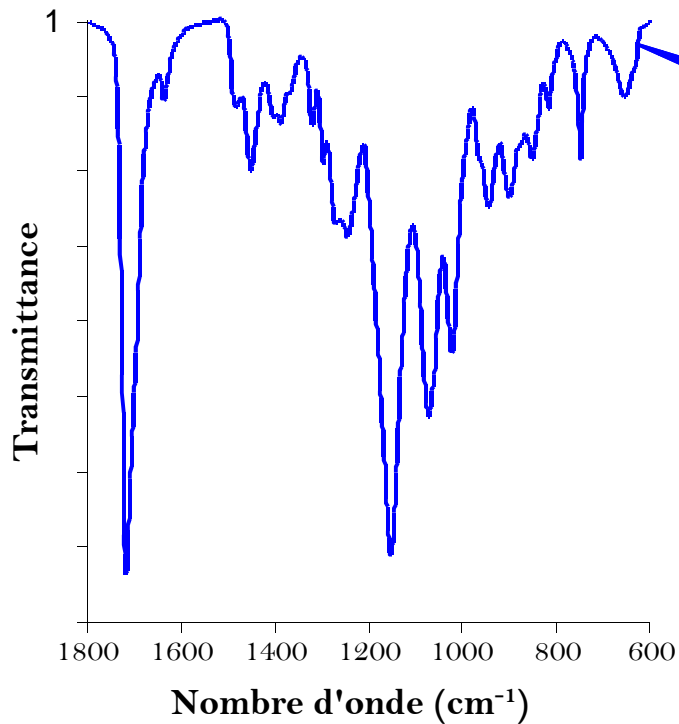
Chronoamperométrie  
 (-1,0 V/15h)



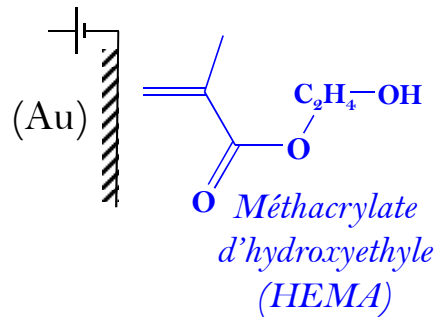
SANS sel de  
 diazonium



Evaporation sous vide (20°C)  
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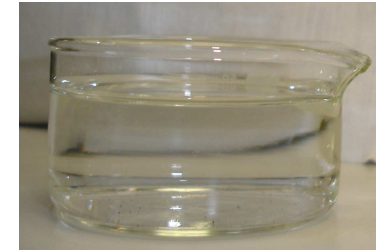
Solution de HEMA sans sel de diazonium



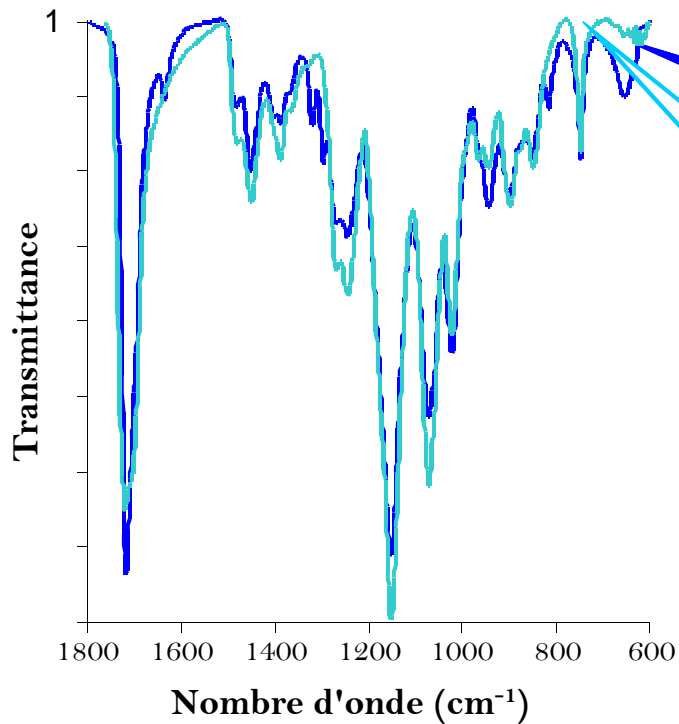
Chronoamperométrie  
(-1,0 V/15h)



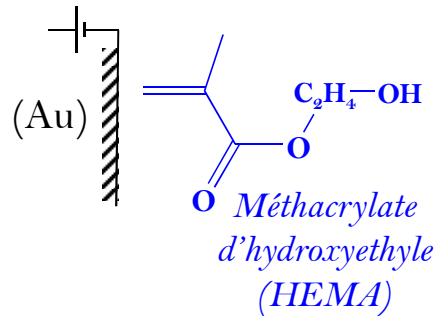
SANS sel de diazonium



Evaporation sous vide (20°C)  
≈ 25h à  $1-3 \cdot 10^{-1}$  mbar



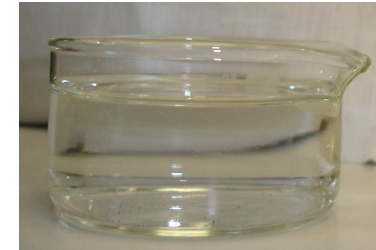
Solution de HEMA sans sel de diazonium



Chronoamperométrie  
(-1,0 V/15h)



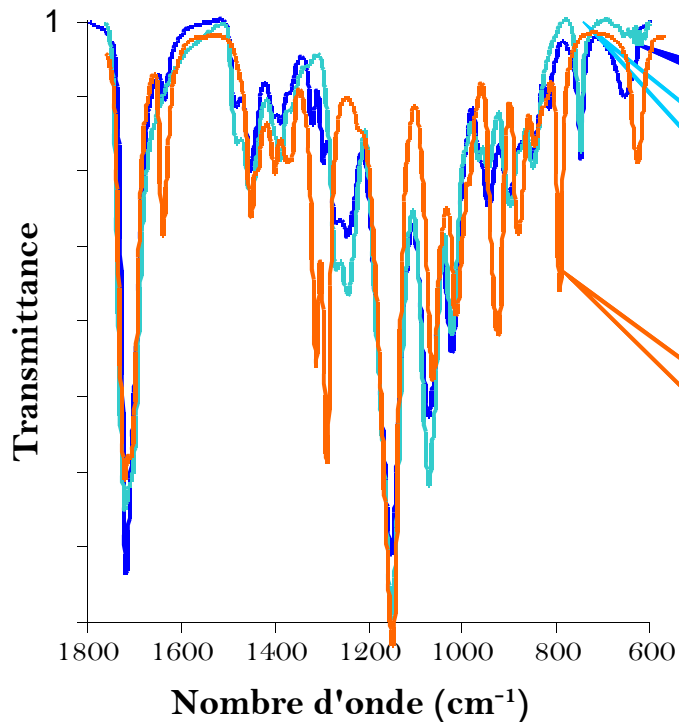
SANS sel de diazonium



Evaporation sous vide (20°C)  
≈ 25h à 1-3.10<sup>-1</sup> mbar



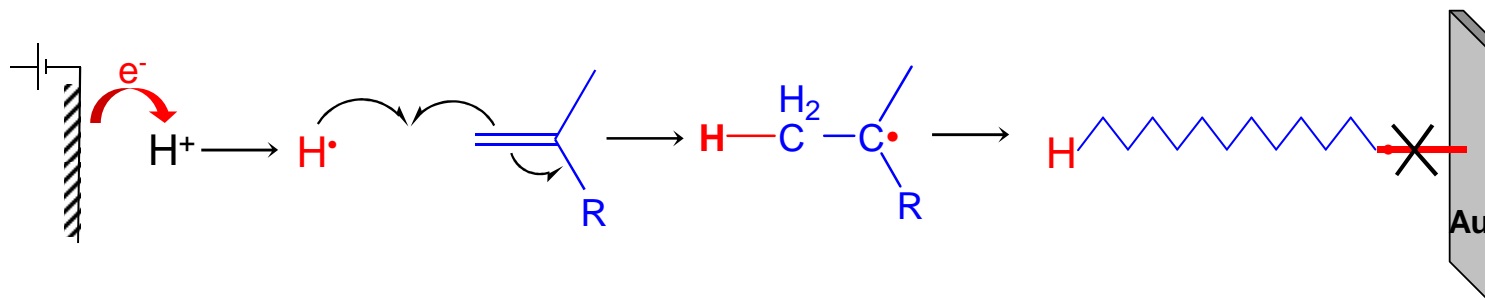
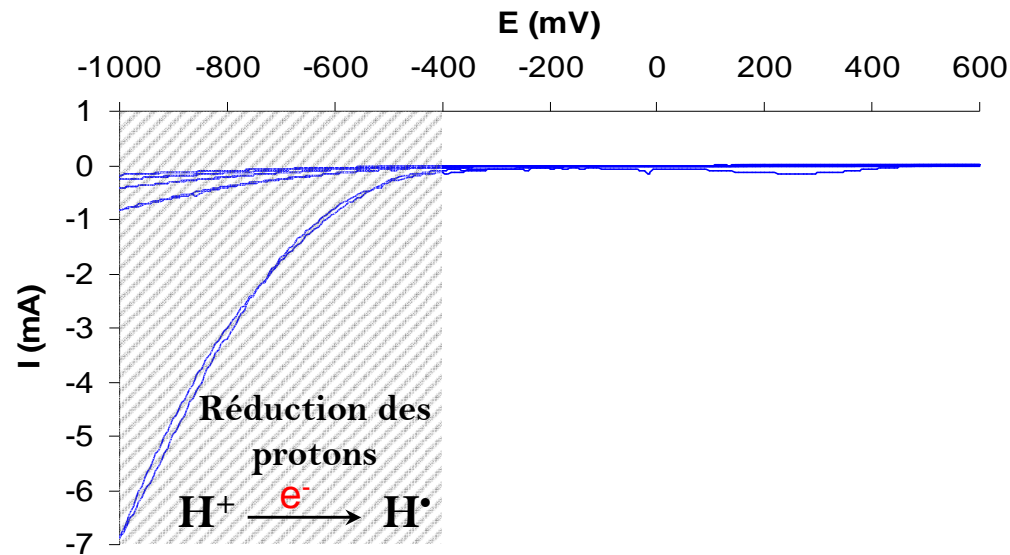
Amorçage de la polymérisation par les radicaux hydrogènes  $H^+ \rightarrow H\bullet$



PHEMA commercial

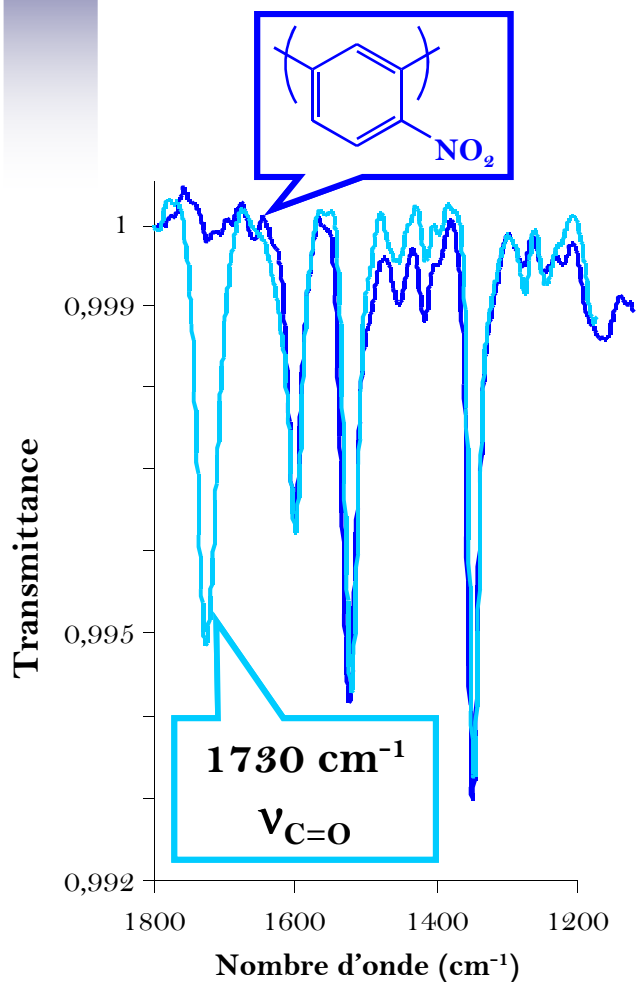
HEMA monomère

Amorçage de la polymérisation par les radicaux hydrogènes  $H^+ \rightarrow H^\bullet$

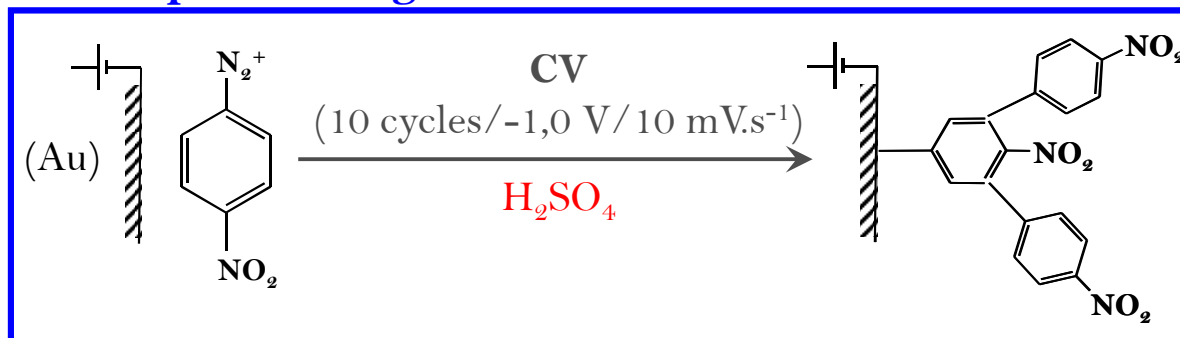




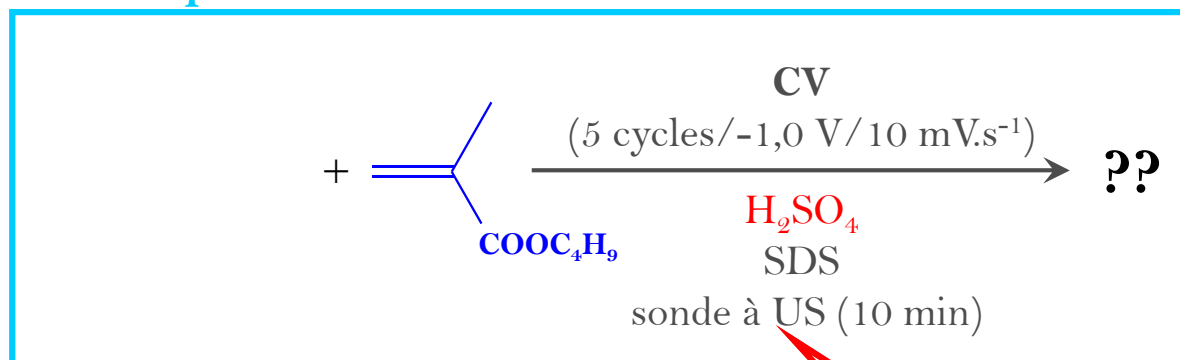
## Greffage en 2 étapes



## 1<sup>ère</sup> étape : Greffage d'un film de PNP



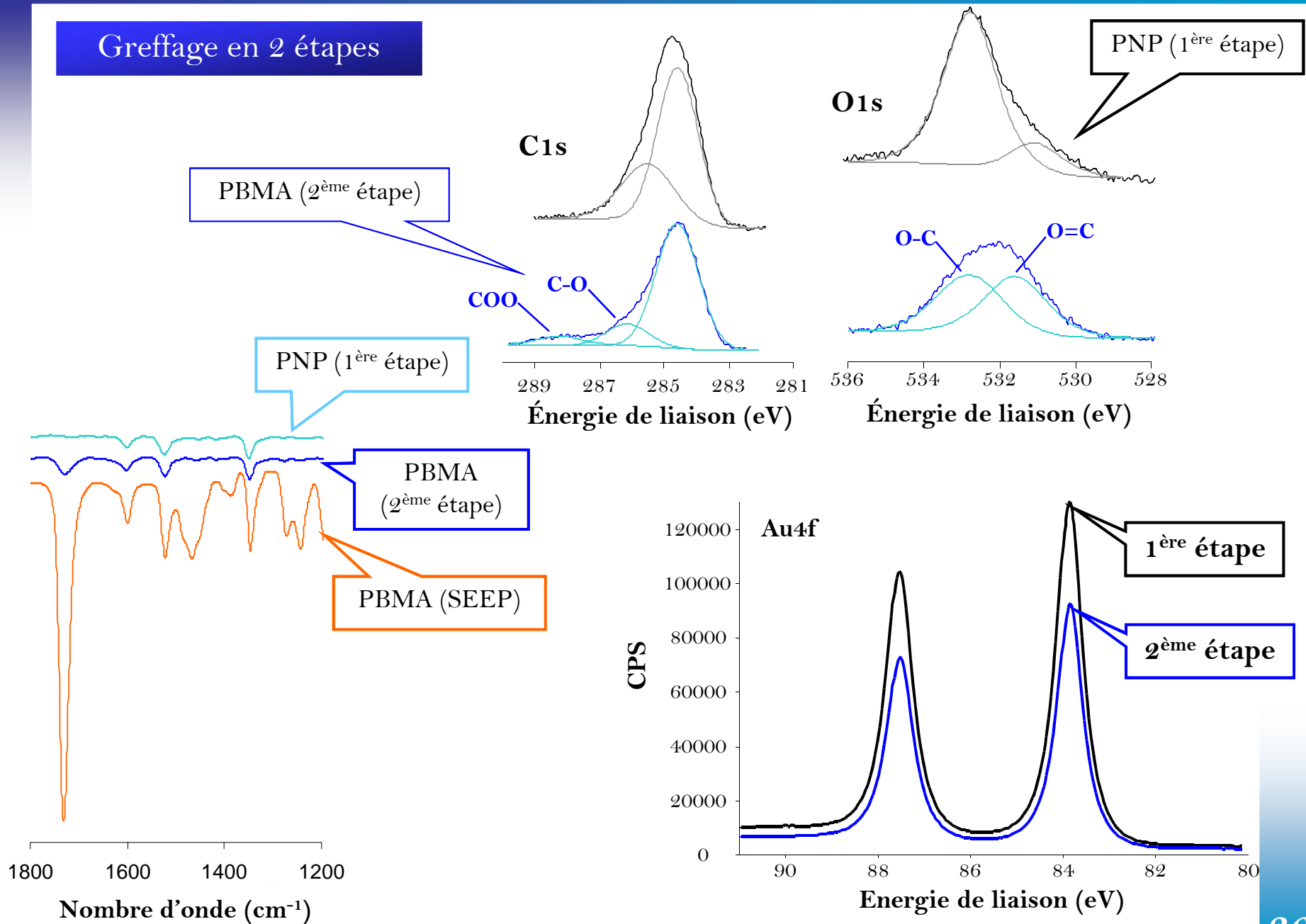
## 2<sup>ème</sup> étape : Miniémulsion de BMA



Formation de PBMA greffé dans la 2<sup>ème</sup> étape (sans sel de diazonium)

SANS sel de diazonium

## Greffage en 2 étapes



I. Introduction

II. SEEP: Principe & Protocole expérimental

III. Résultats

Caractérisation des films

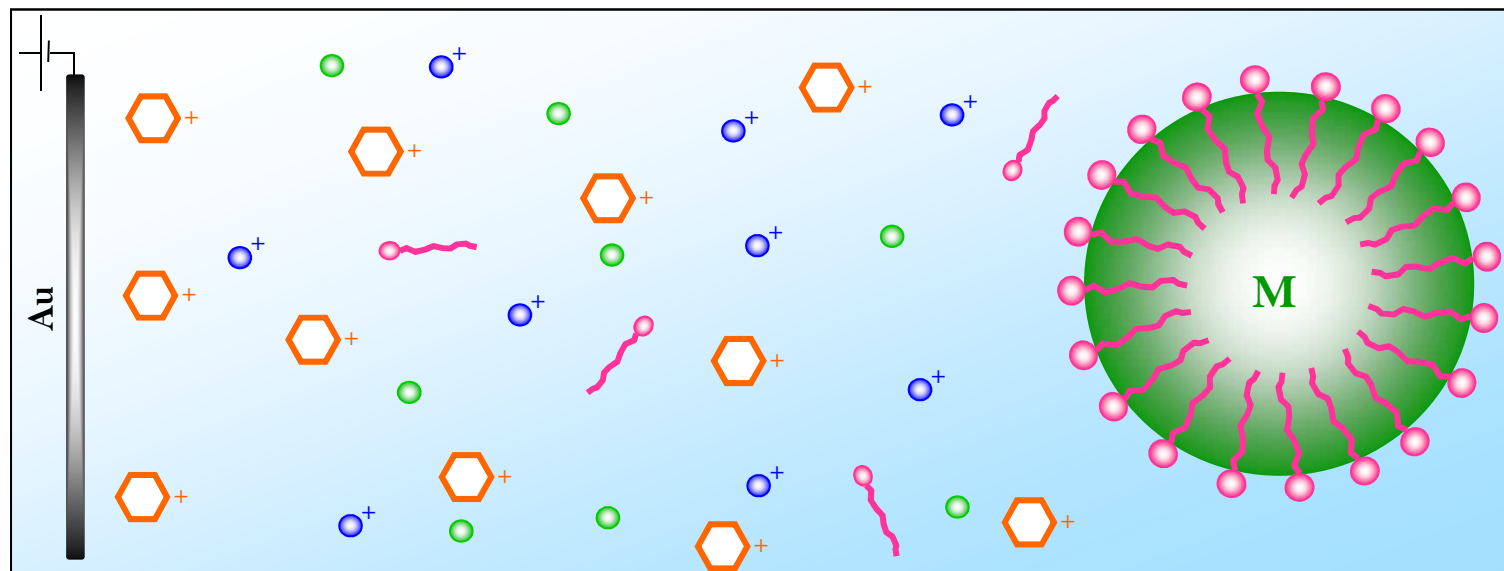
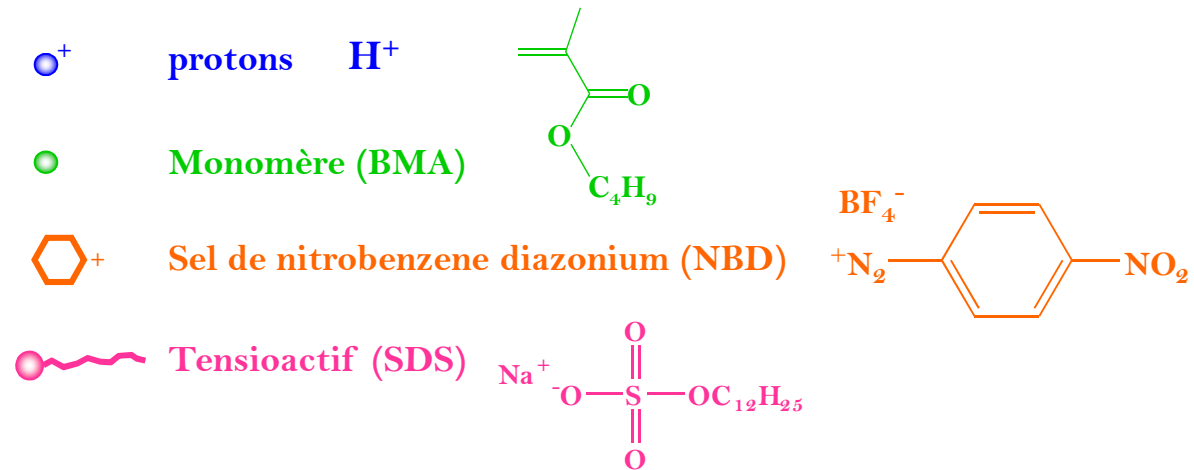
Mécanisme

**IV. Discussion : Mécanisme de SEEP**

V. Application

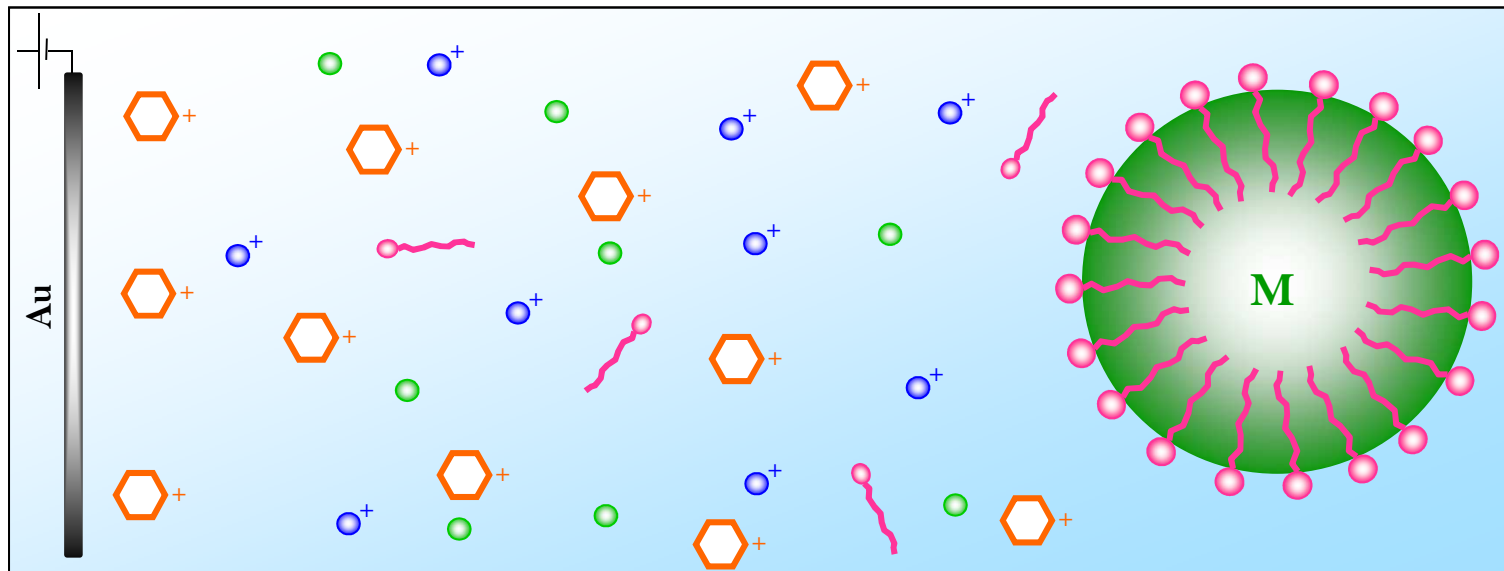
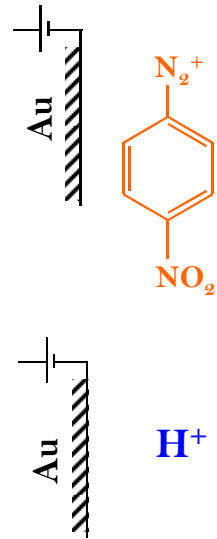
VI. Conclusion

## ① Amorçage



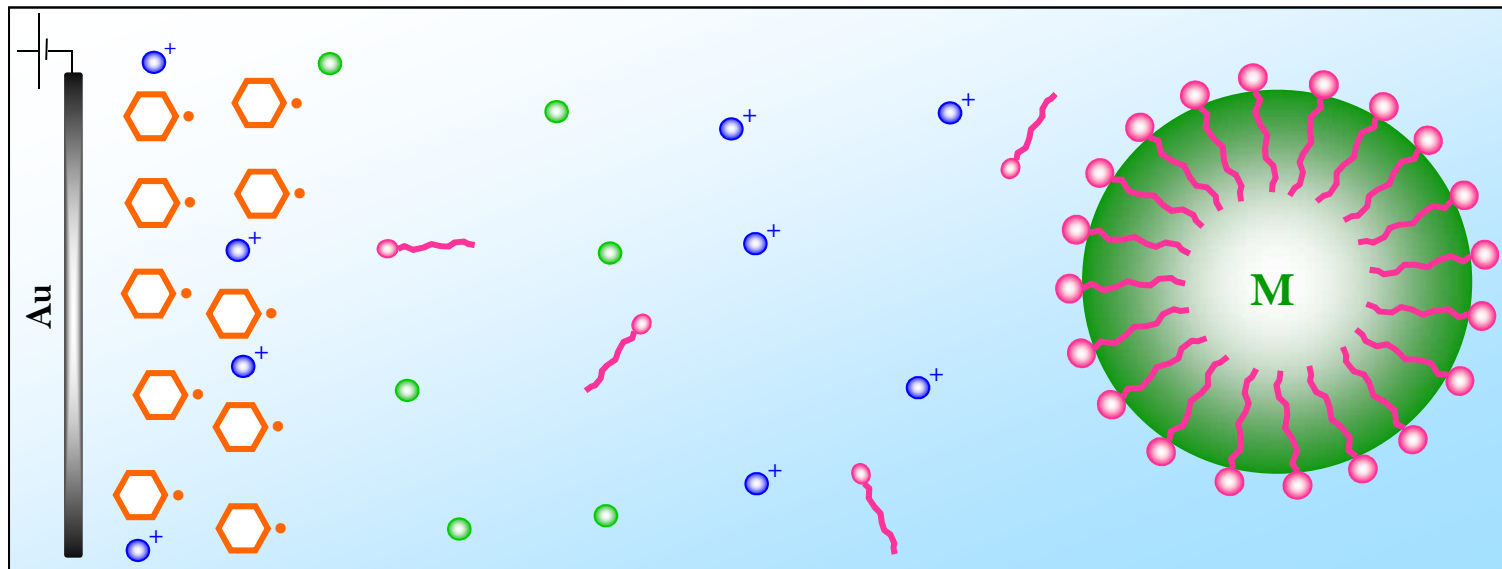
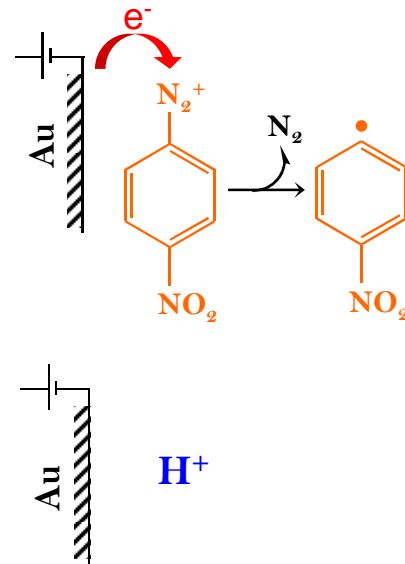
## ① Amorçage

$E_{\text{éq}}$



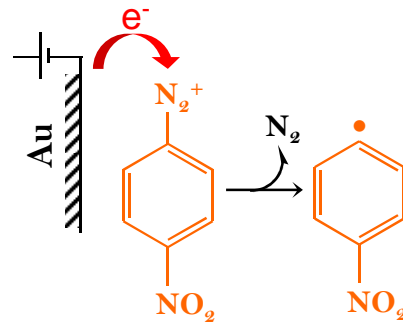
## ① Amorçage

$E \approx 0,2 \text{ V}$

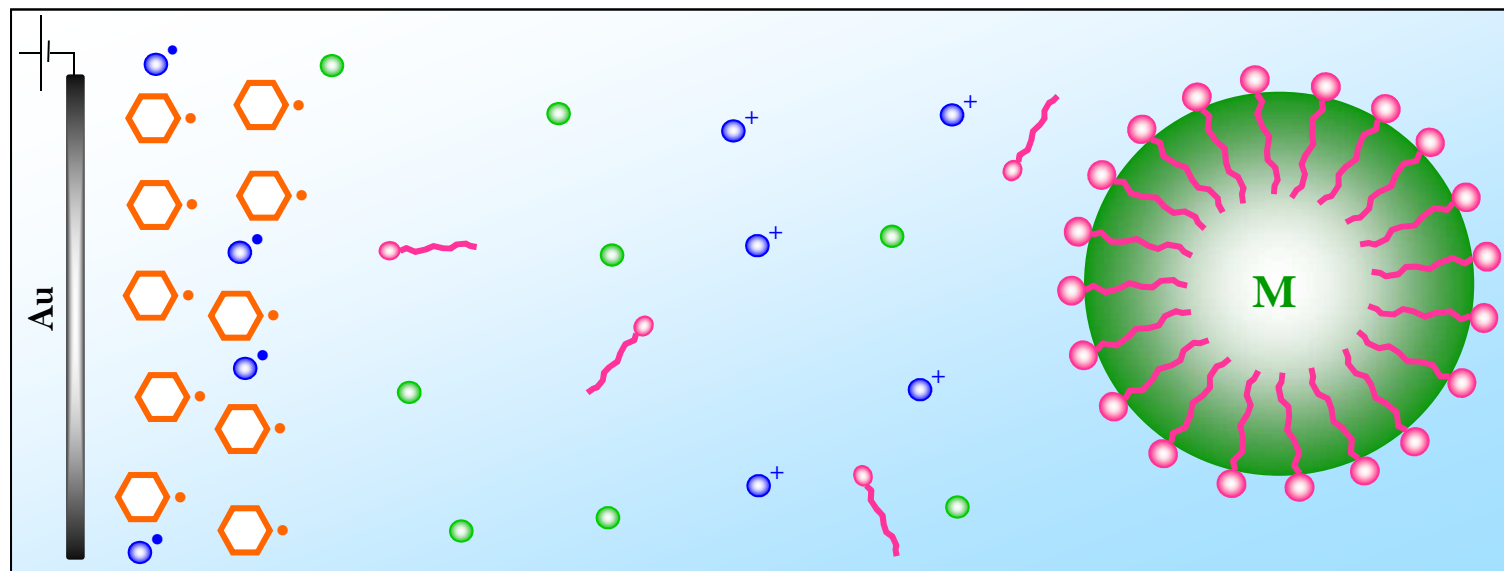
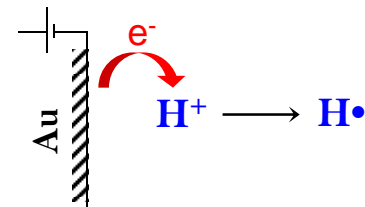


## ① Amorçage

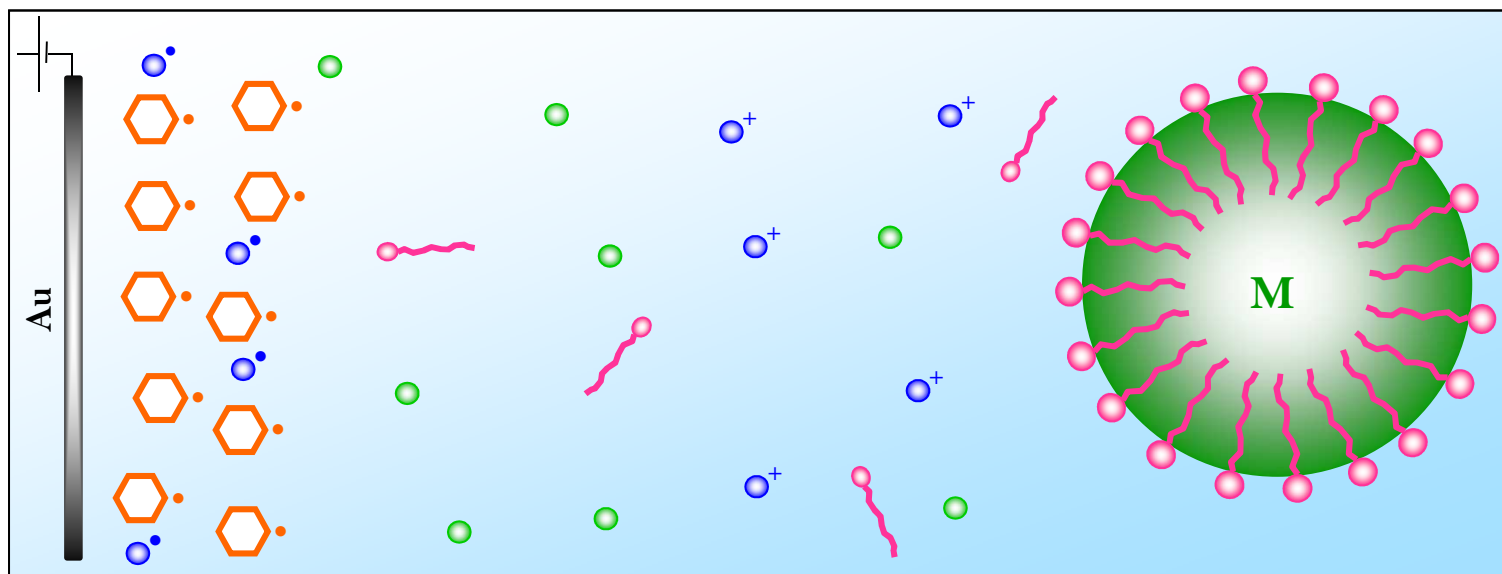
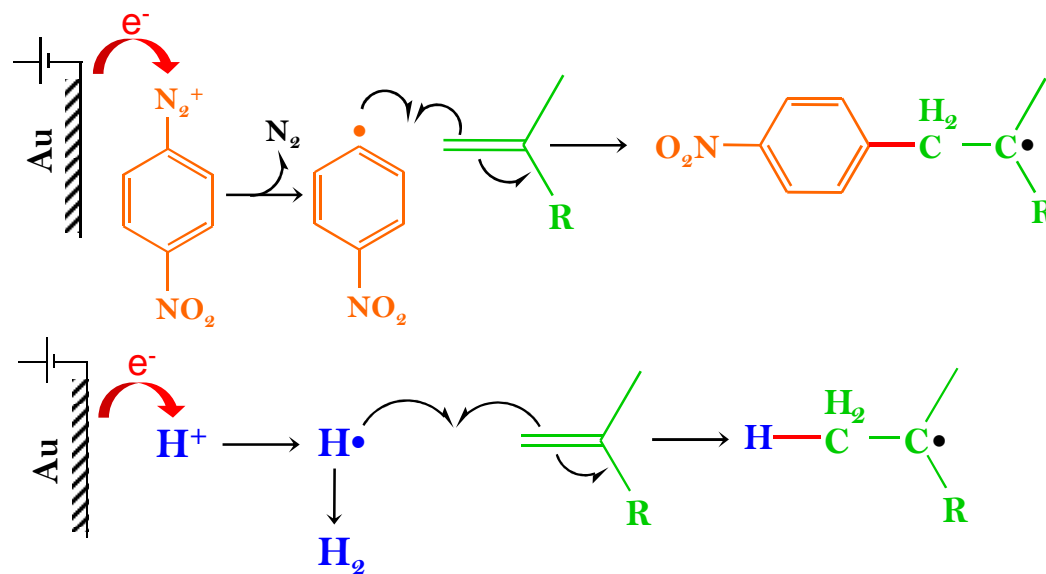
$E \approx 0,2 \text{ V}$



$E \approx -0,6 \text{ V}$

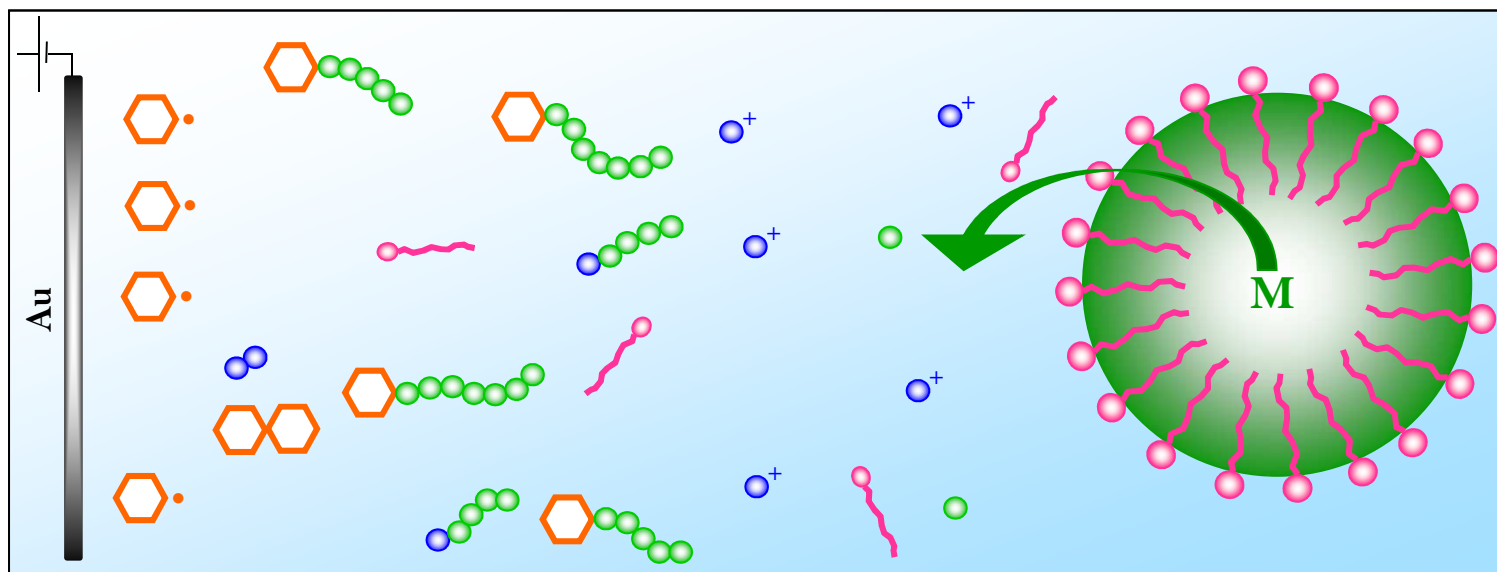
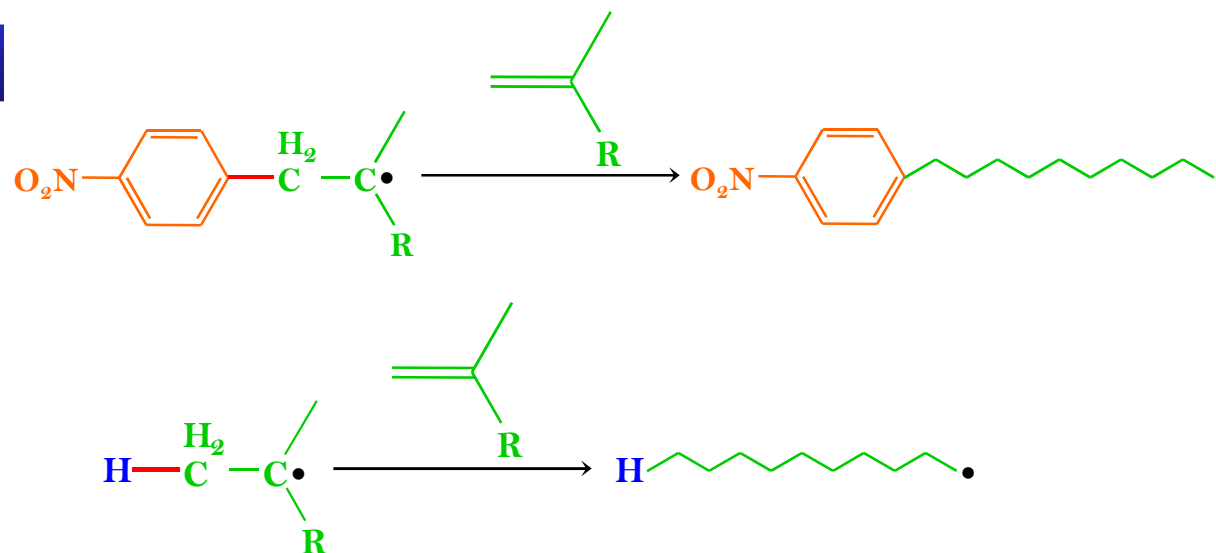


## ① Amorçage

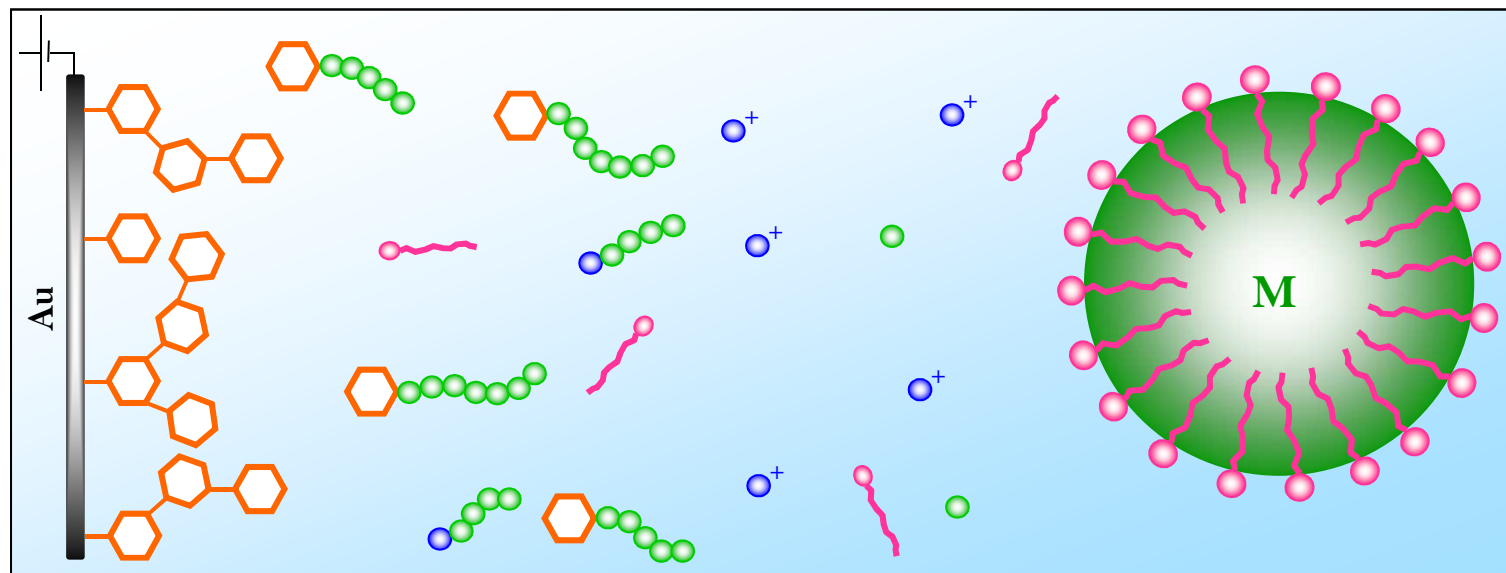
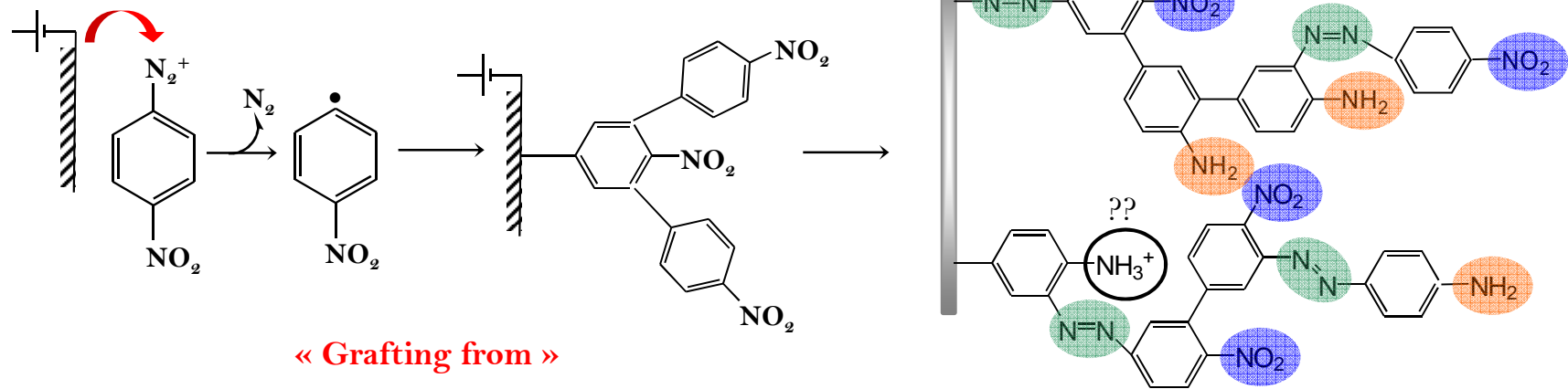




## ② Propagation

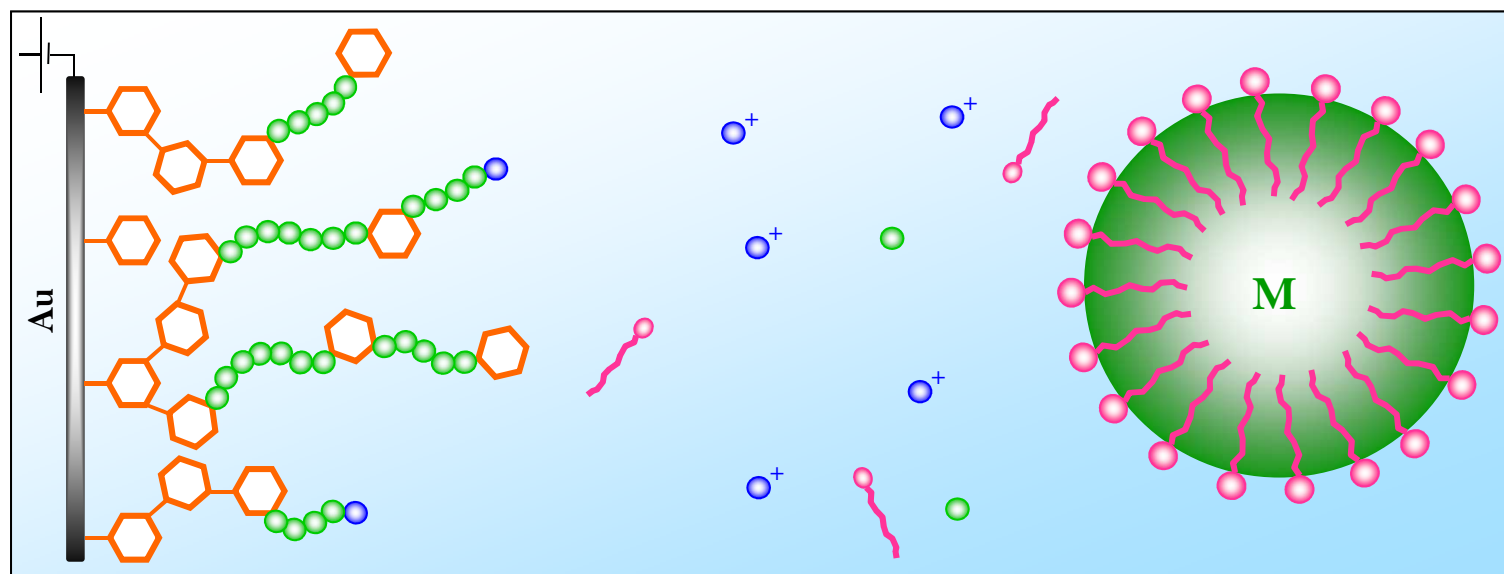
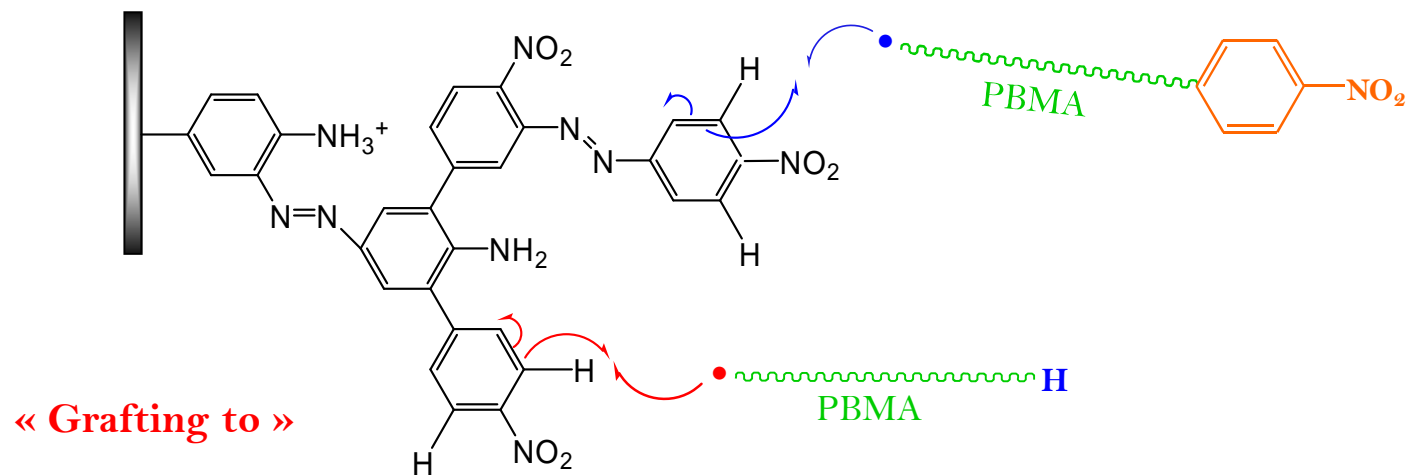


## ③ Formation de la sous-couche de PNP



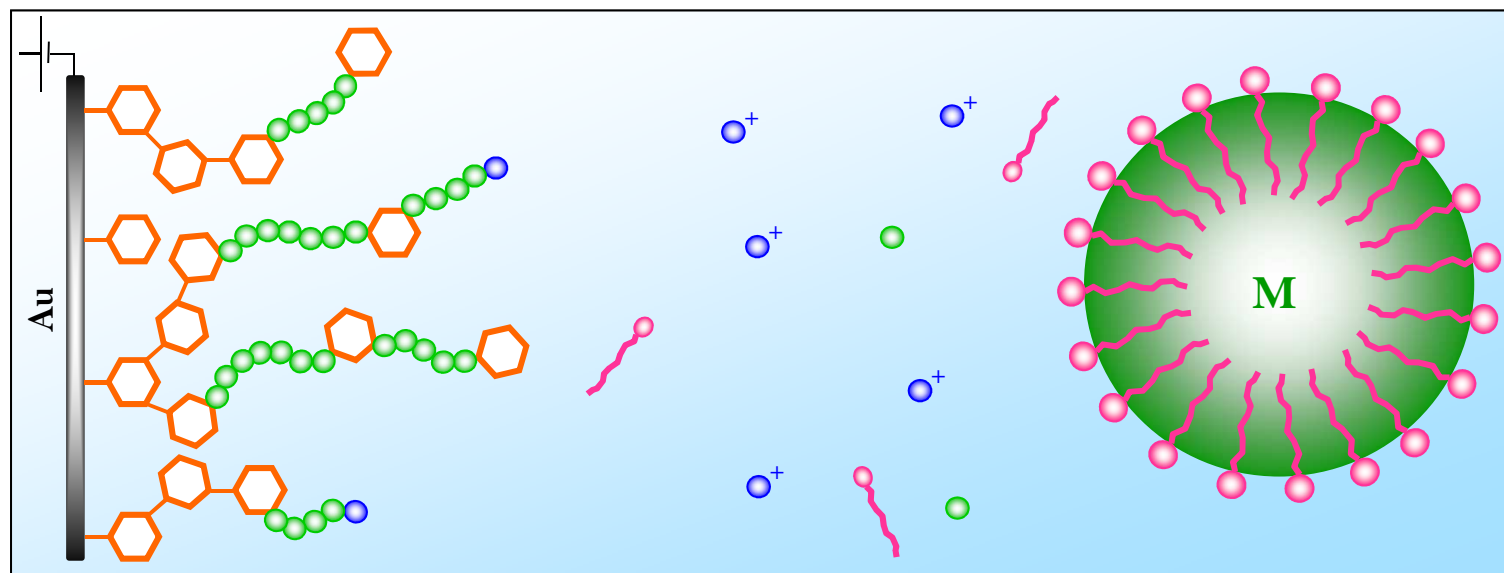
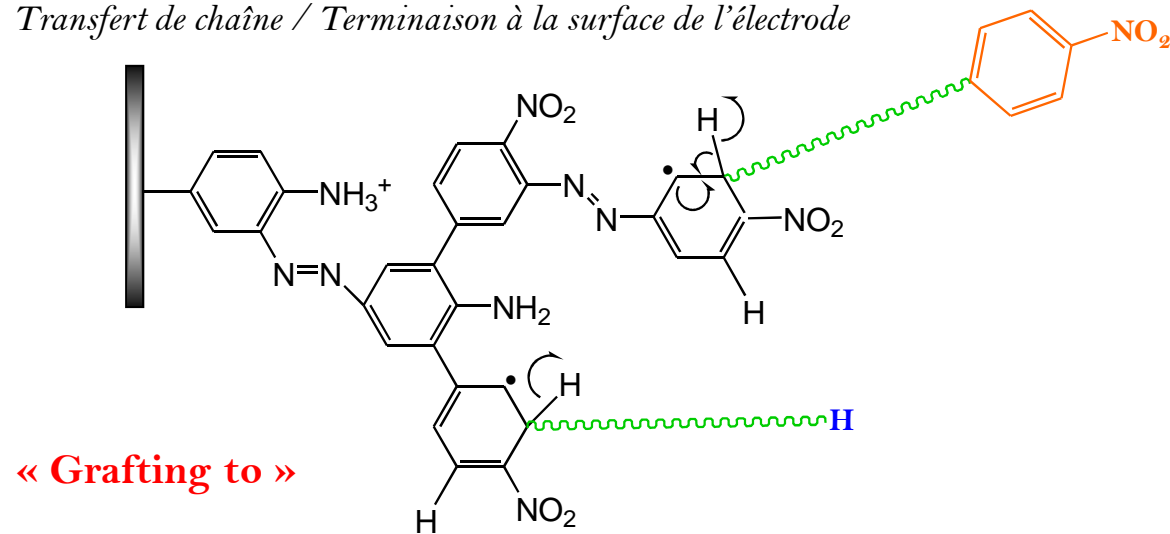
## ⑤ Accroche des chaînes sur la sous-couche de PNP (réaction de transfert)

*Transfert de chaîne / Terminaison à la surface de l'électrode*



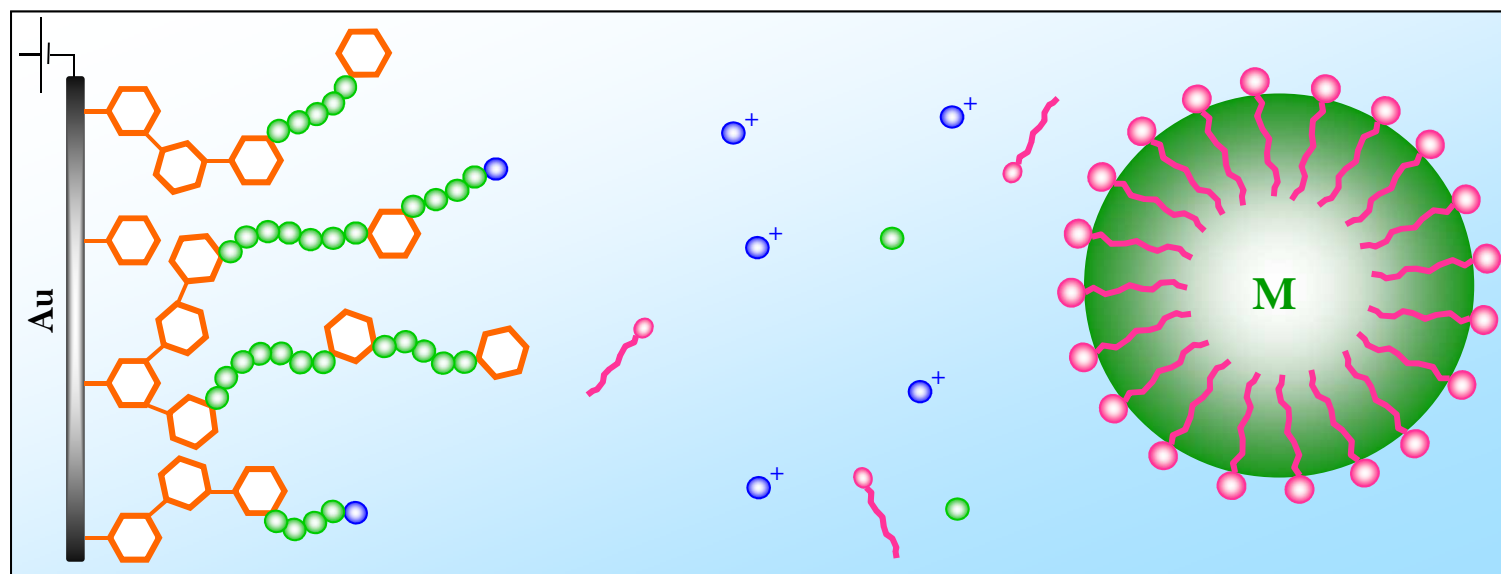
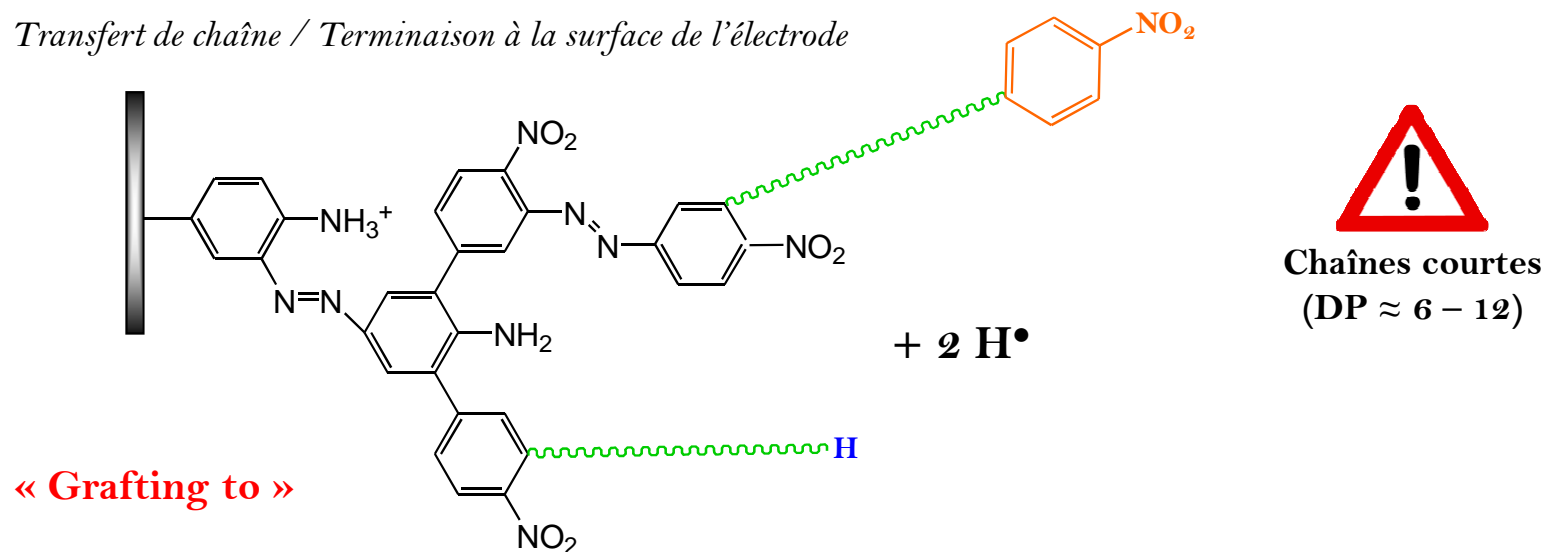
## ⑤ Accroche des chaînes sur la sous-couche de PNP (réaction de transfert)

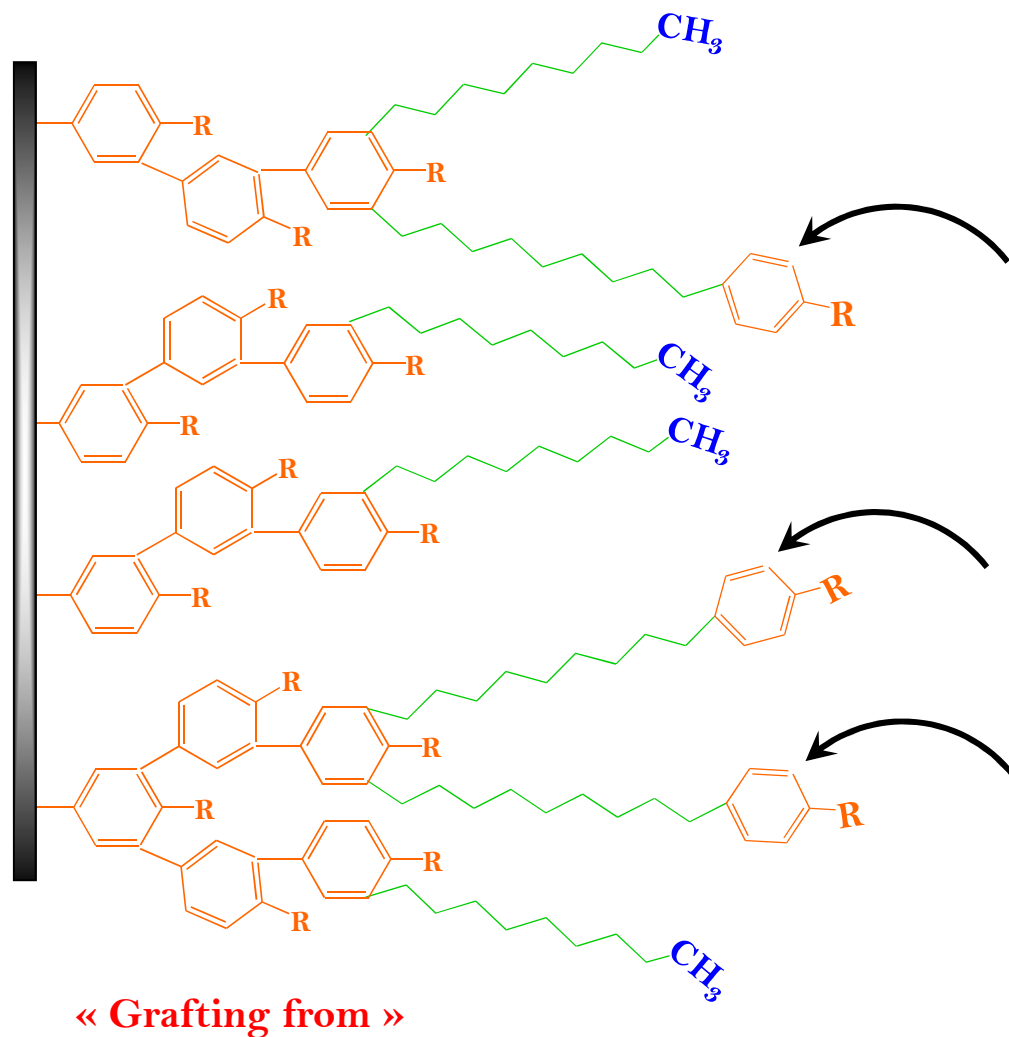
*Transfert de chaîne / Terminaison à la surface de l'électrode*



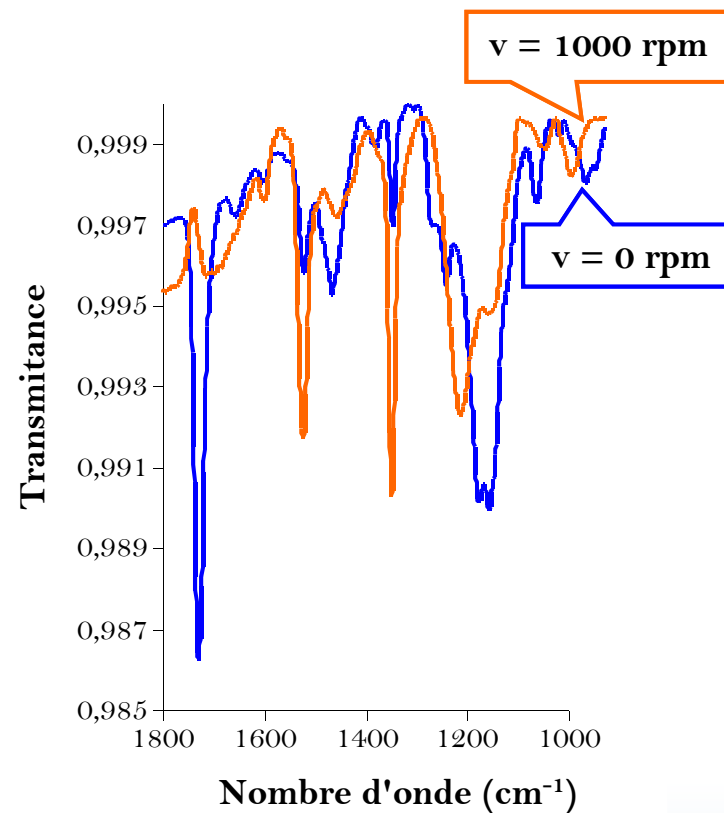
## ⑤ Accroche des chaînes sur la sous-couche de PNP (réaction de transfert)

*Transfert de chaîne / Terminaison à la surface de l'électrode*

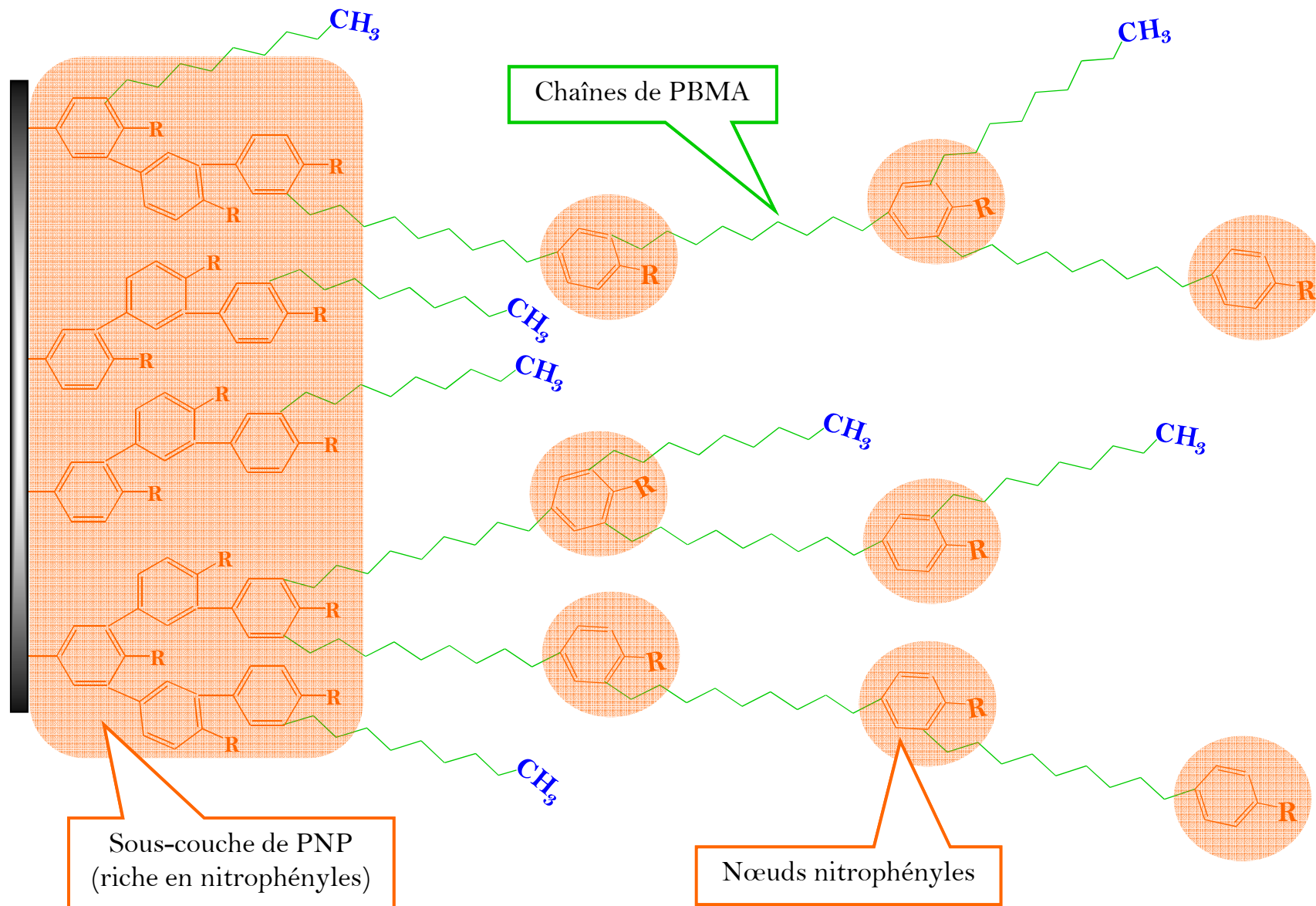


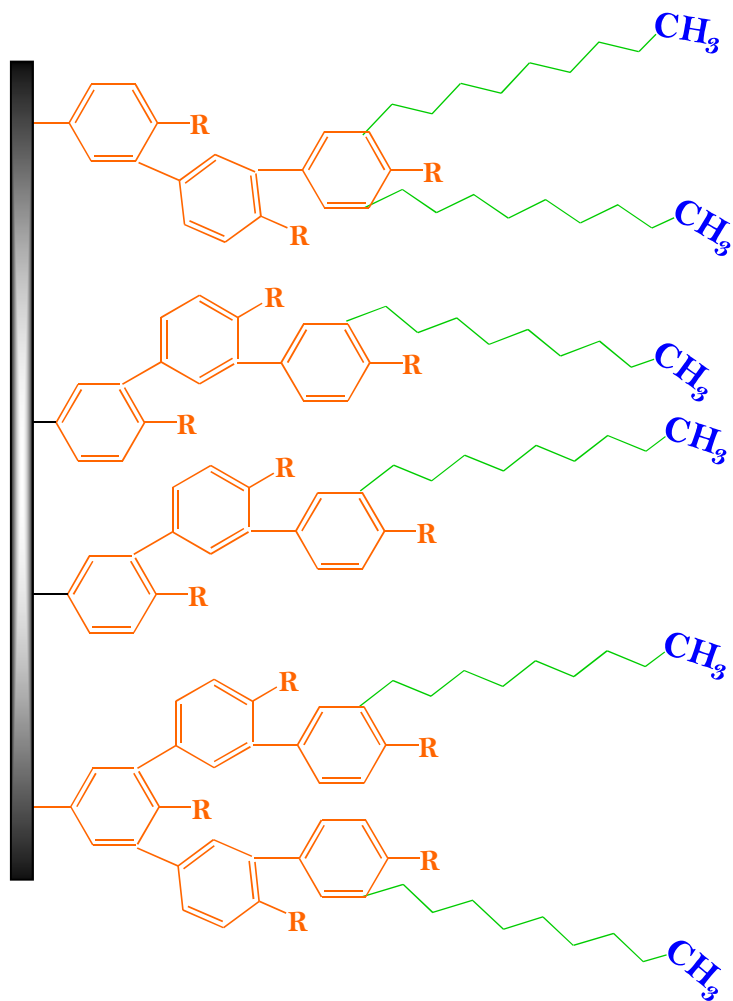


Électrode tournante



« Grafting to »







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## III. Résultats

Caractérisation des films (morphologie)

Composition chimique & Structure moléculaire

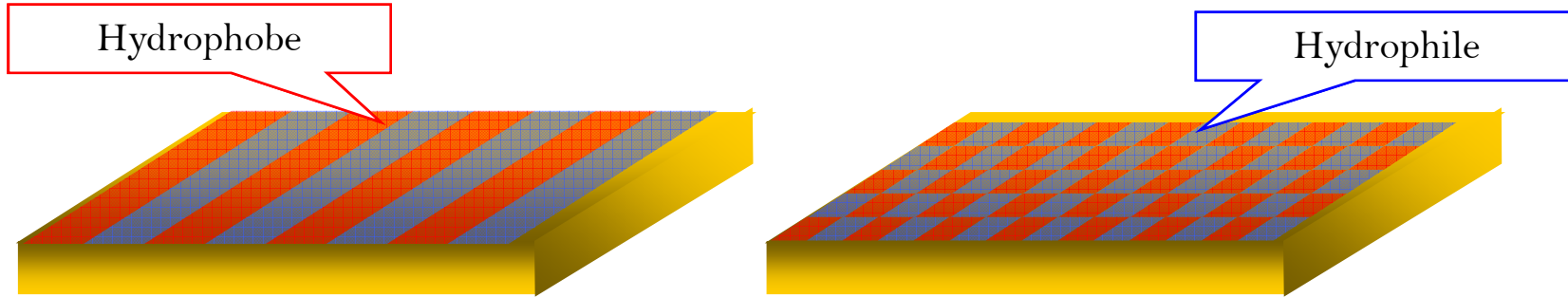
Mécanisme

## IV. Discussion : Mécanisme de SEEP

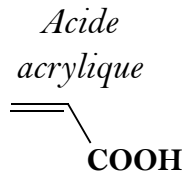
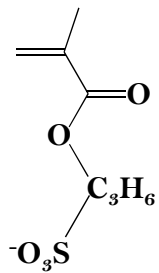
## V. Application

## VI. Conclusion

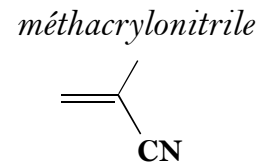
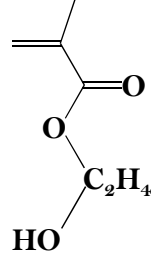
Réalisation de surfaces à motifs alternés hydrophile / hydrophobe



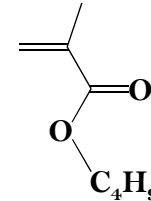
Méthacrylate de 3-sulfopropyle



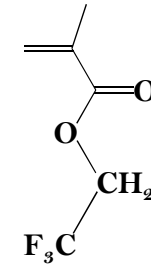
Méthacrylate de hydroxyéthyle



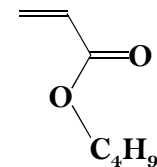
Méthacrylate de butyle



Méthacrylate de trifluoroéthyle

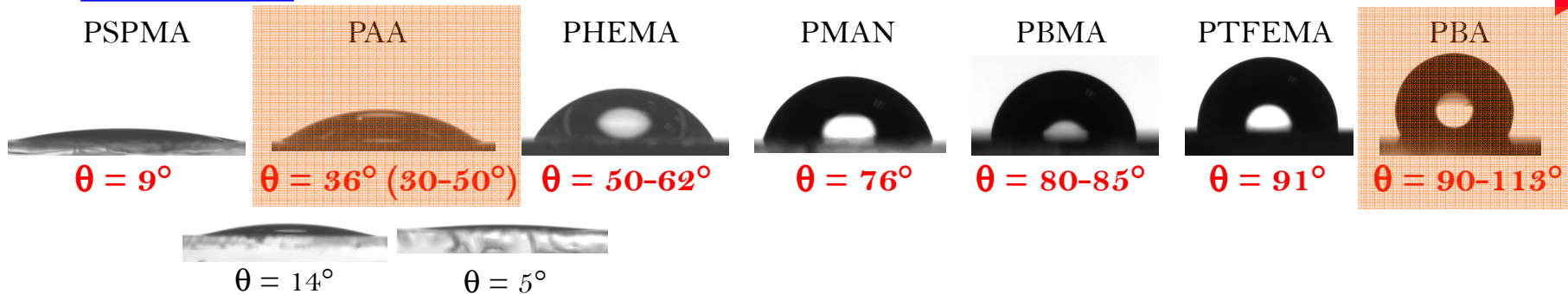


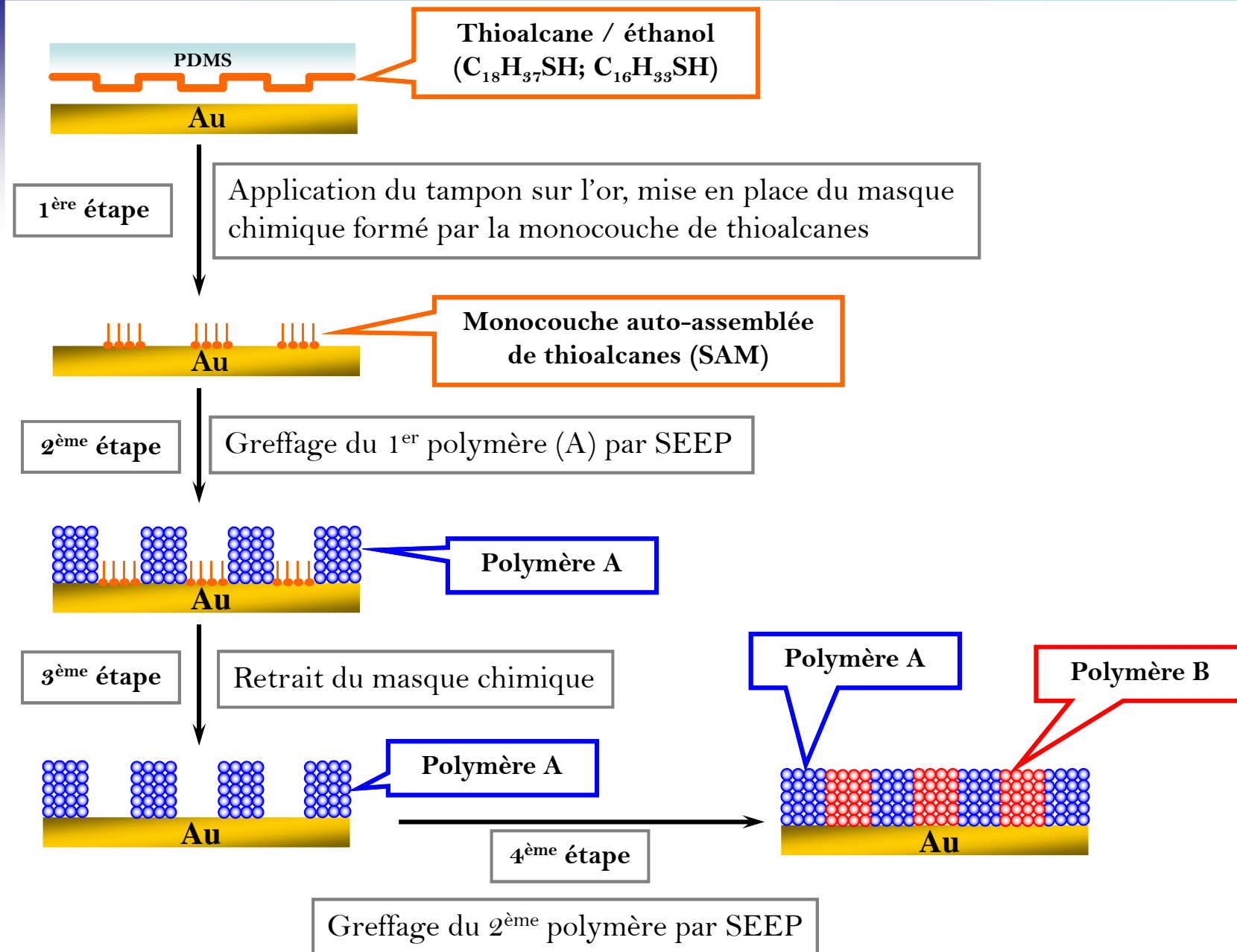
Acrylate de butyle



Hydrophile

Hydrophobe





# V. Application

## Surfaces à motifs alternés hydrophile/hydrophobe

Echelle macroscopique



Echelle microscopique



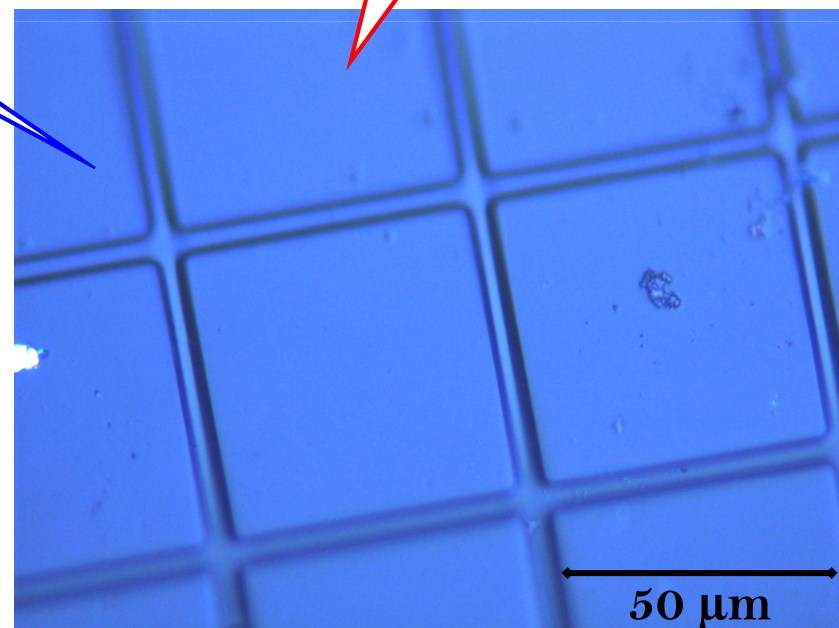
Or vierge

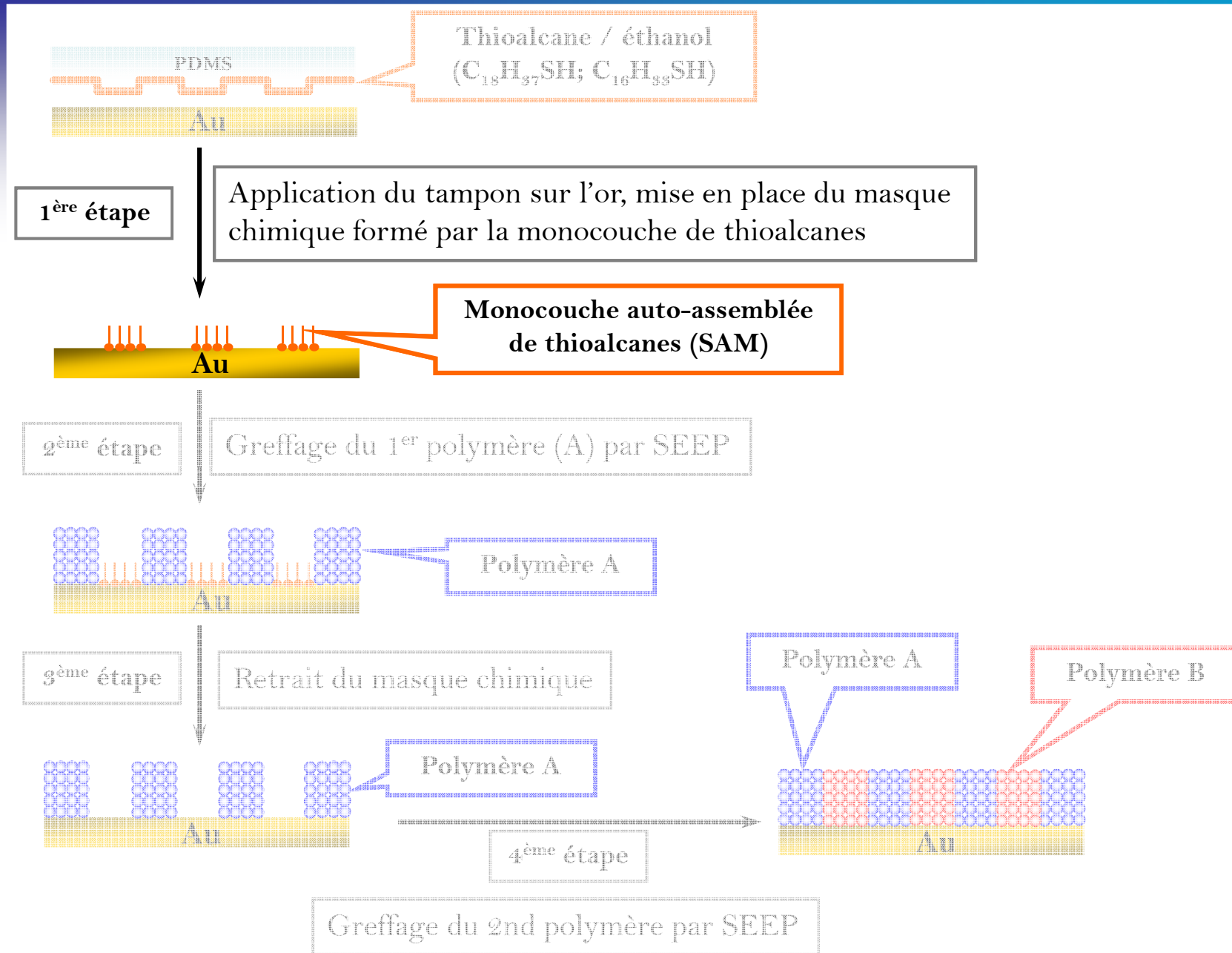


Monocouche de thioalcanes

Creux

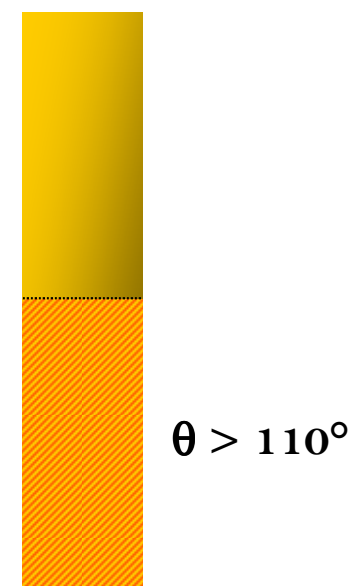
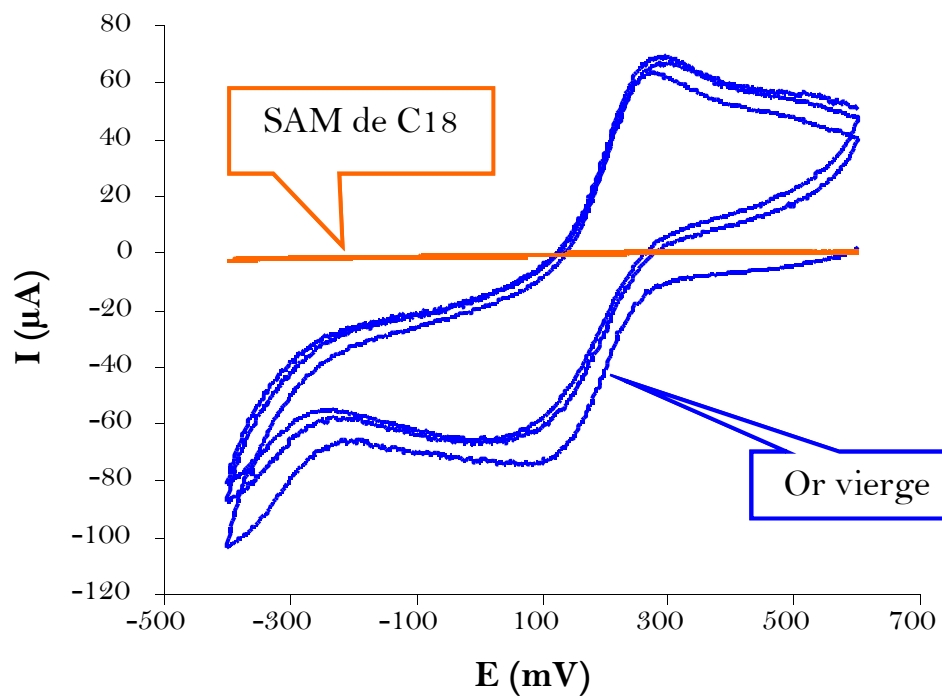
Relief

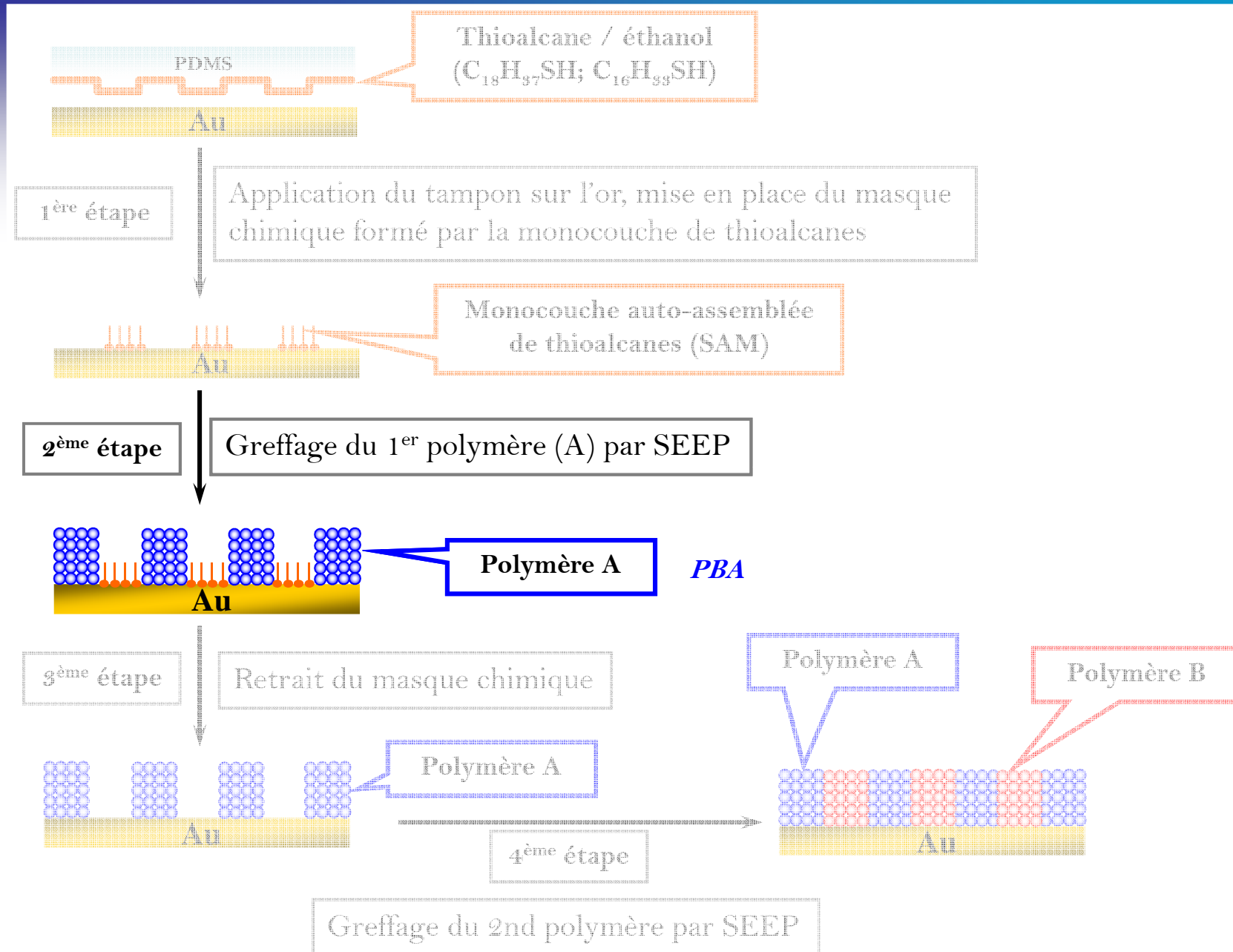




1<sup>ère</sup> étape

SAM réalisée par tampon





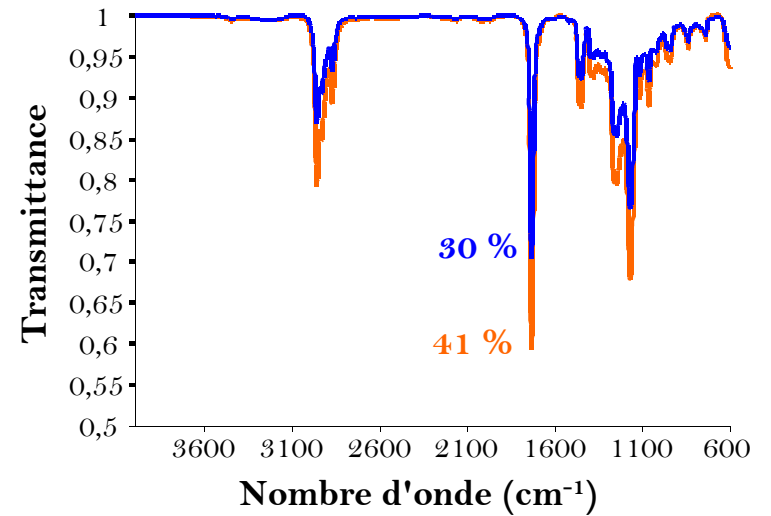
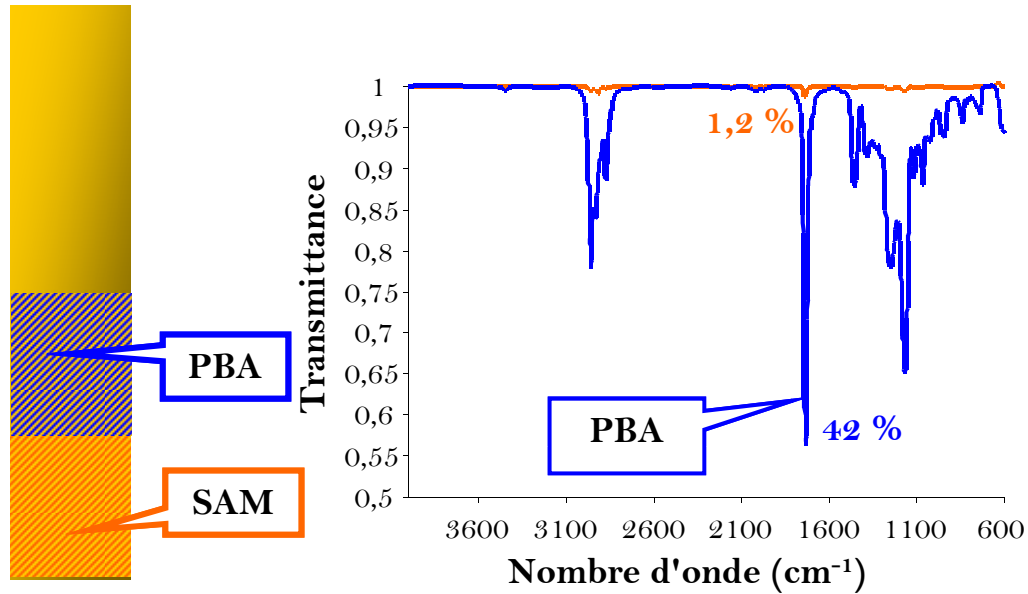
# V. Application

## Surfaces à motifs alternés hydrophile/hydrophobe

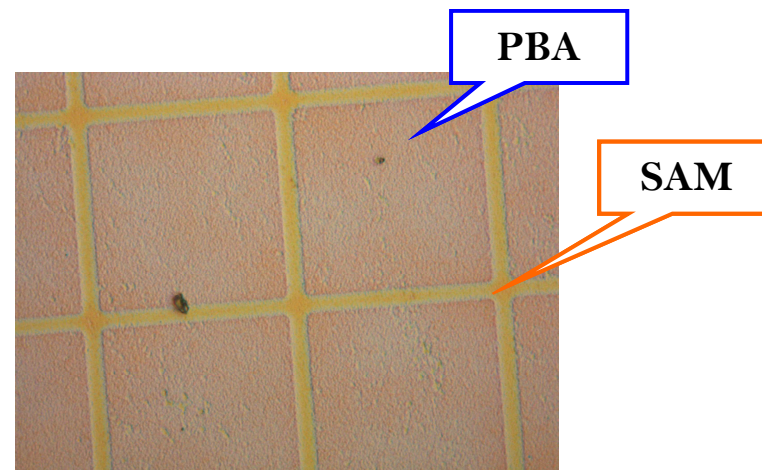
2<sup>ème</sup> étape

Greffage du PBA par SEEP:  
5 cycles / -1,0 V

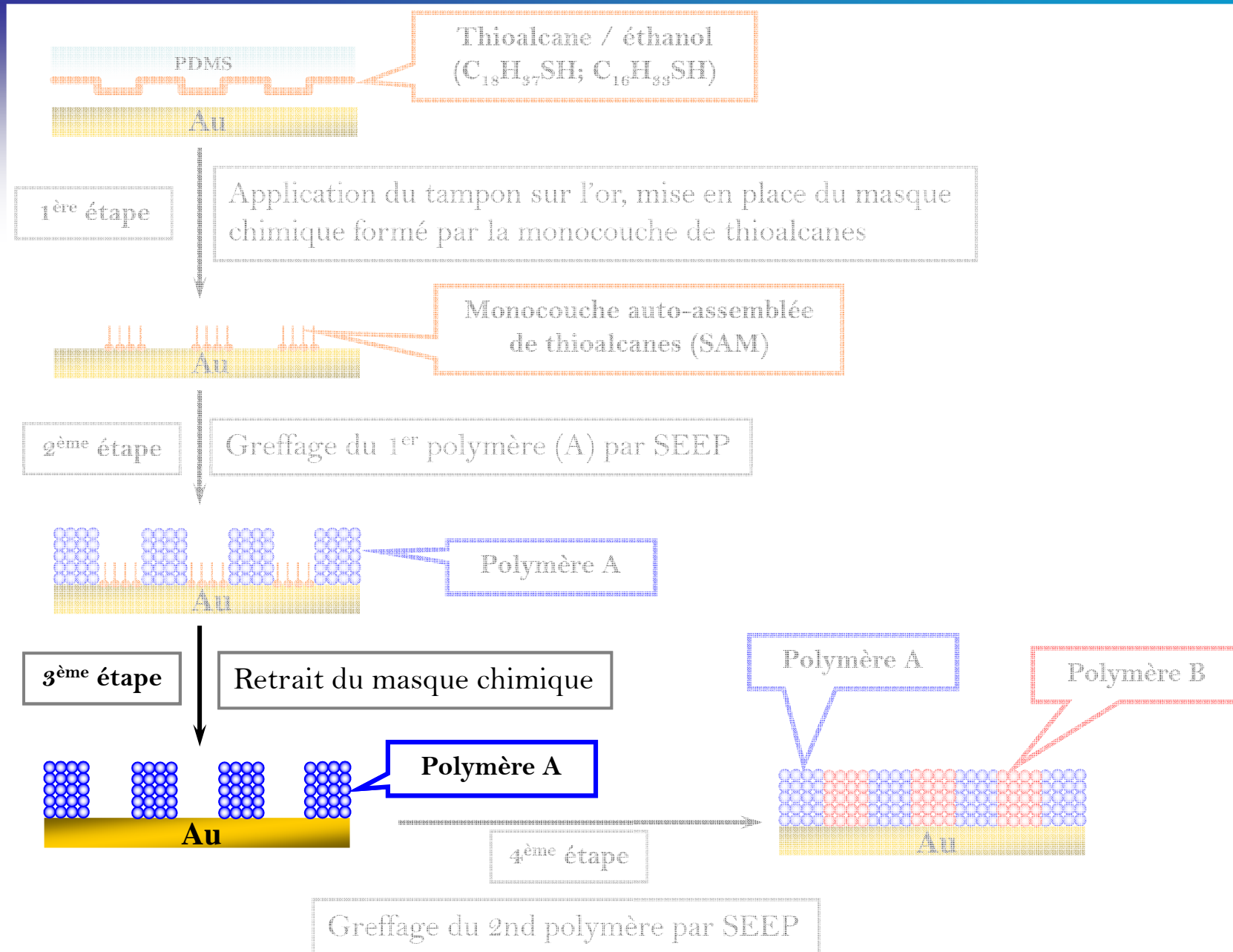
Echelle macroscopique



Echelle  
microscopique



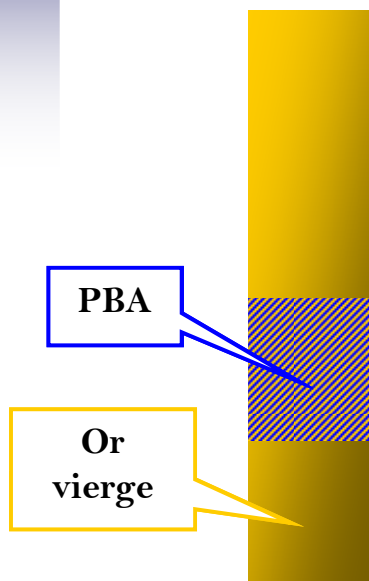




# V. Application

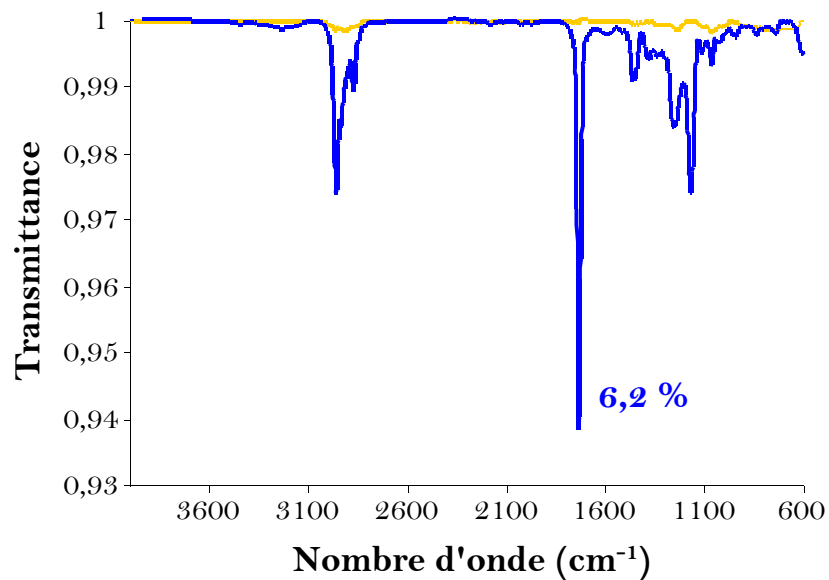
## Surfaces à motifs alternés hydrophile/hydrophobe

Echelle macroscopique

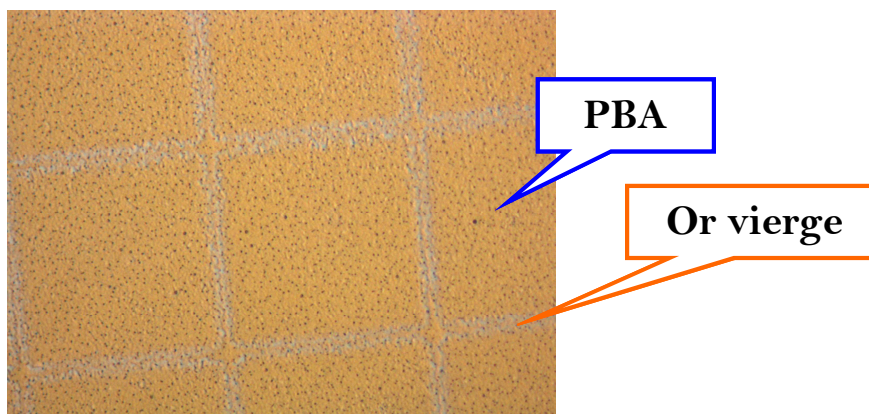


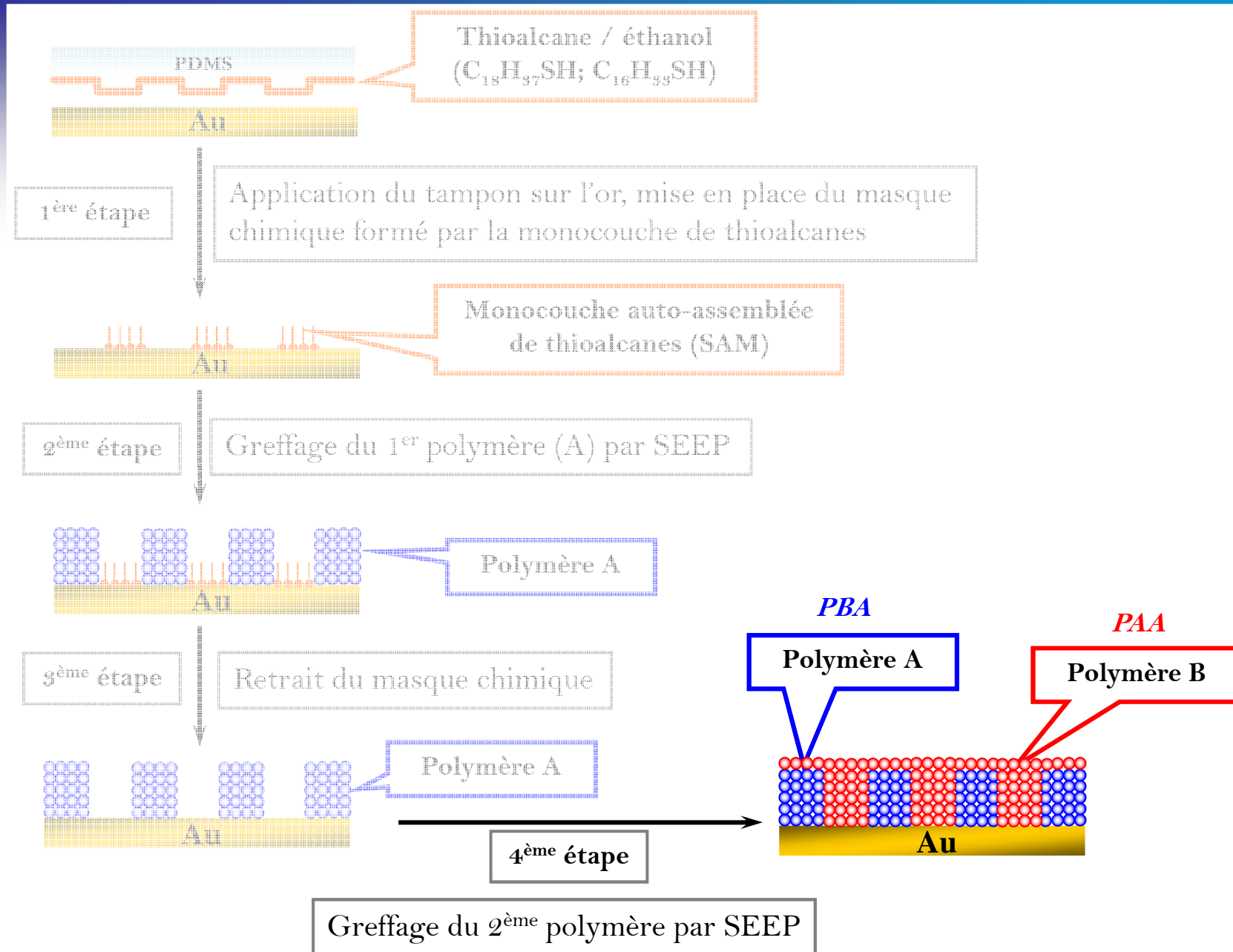
3<sup>ème</sup> étape

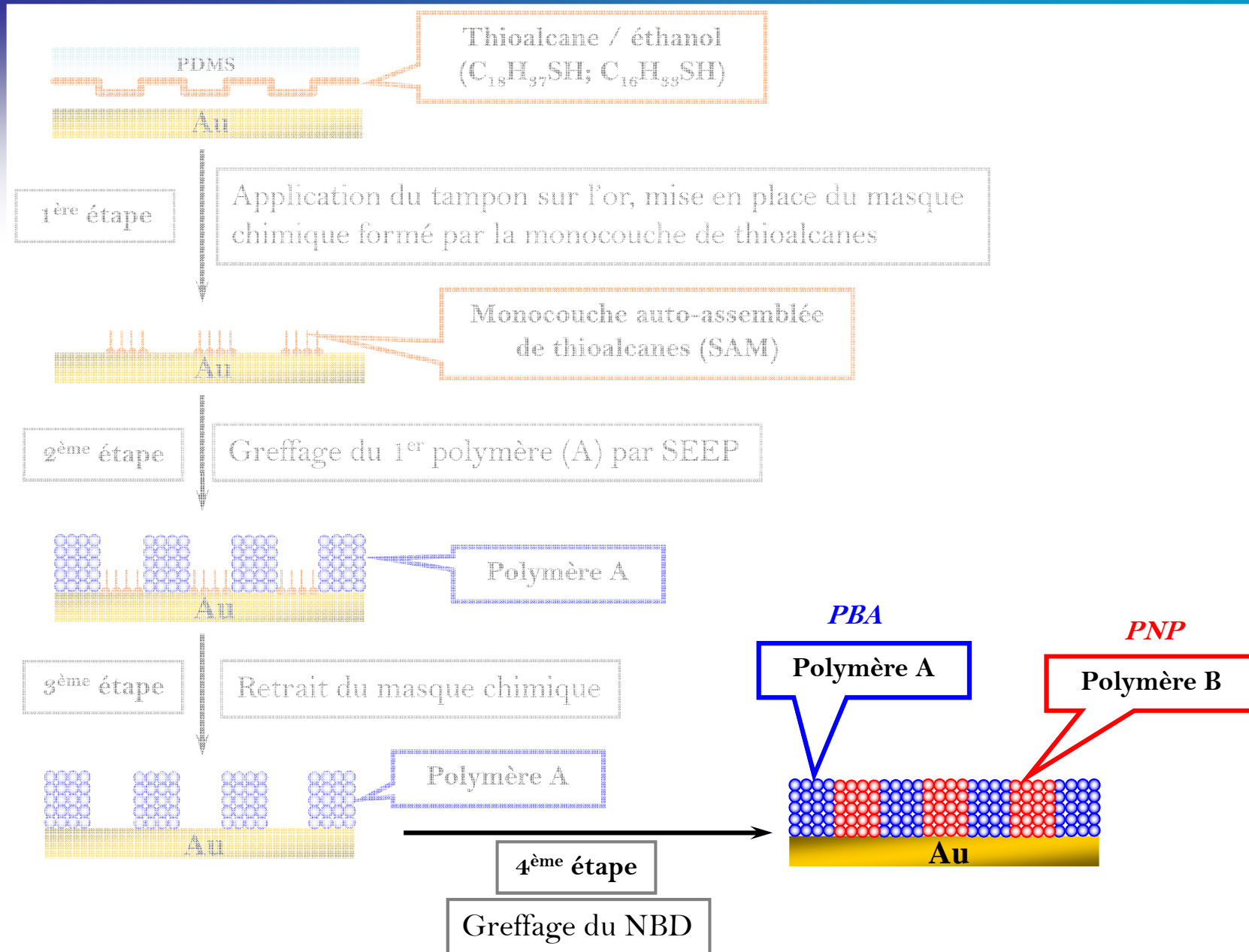
Désorption de la monocouche:  
Réduction -2,5 V / CH<sub>3</sub>CN



Echelle microscopique







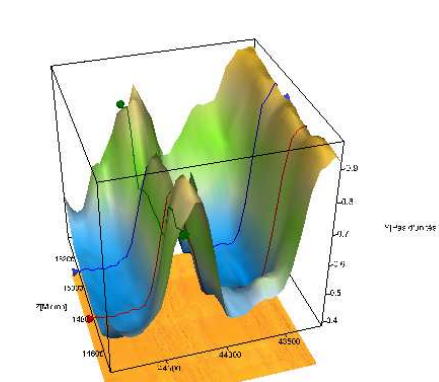
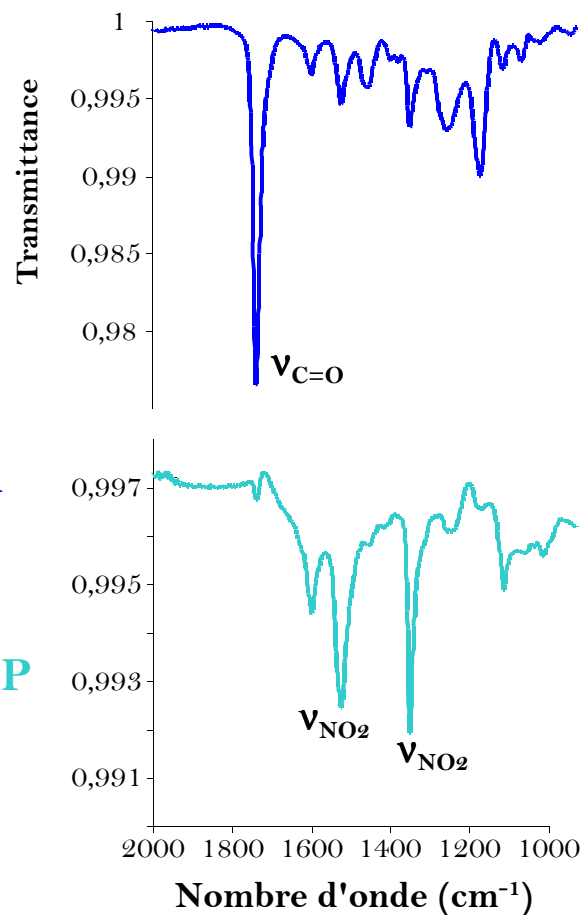
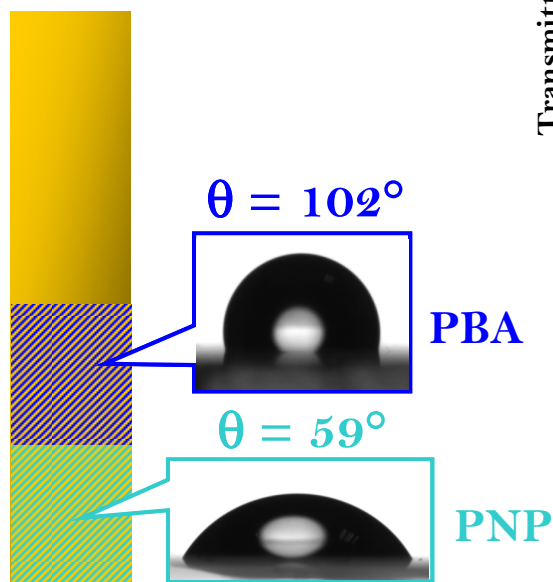
# V. Application

## Surfaces à motifs alternés hydrophile/hydrophobe

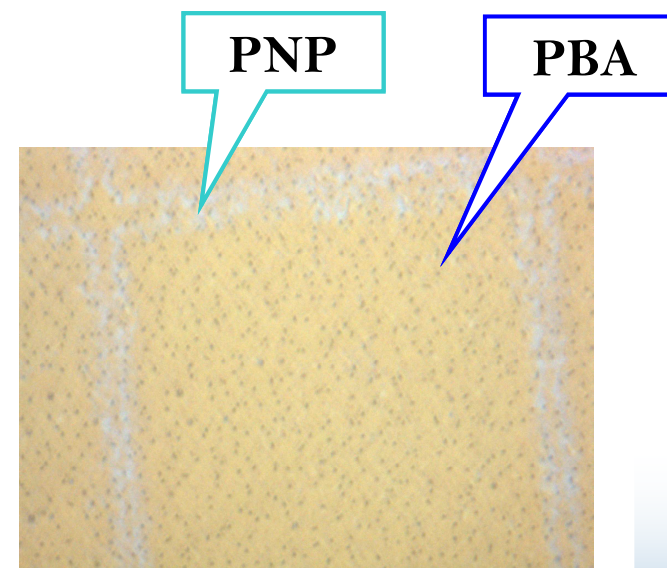
4<sup>ème</sup> étape

**Greffage du NBD / H<sub>2</sub>SO<sub>4</sub>  
3 cycles, 10 mV/s, E<sub>éq</sub> → -1,0 V**

Echelle macroscopique



Echelle microscopique



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## III. Discussion : Mécanisme de SEEP

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## Procédé SEEP

- Revêtements de PBMA à partir de solutions aqueuses
- Nombreux revêtements

## Mécanisme $\Rightarrow$ objectifs atteints



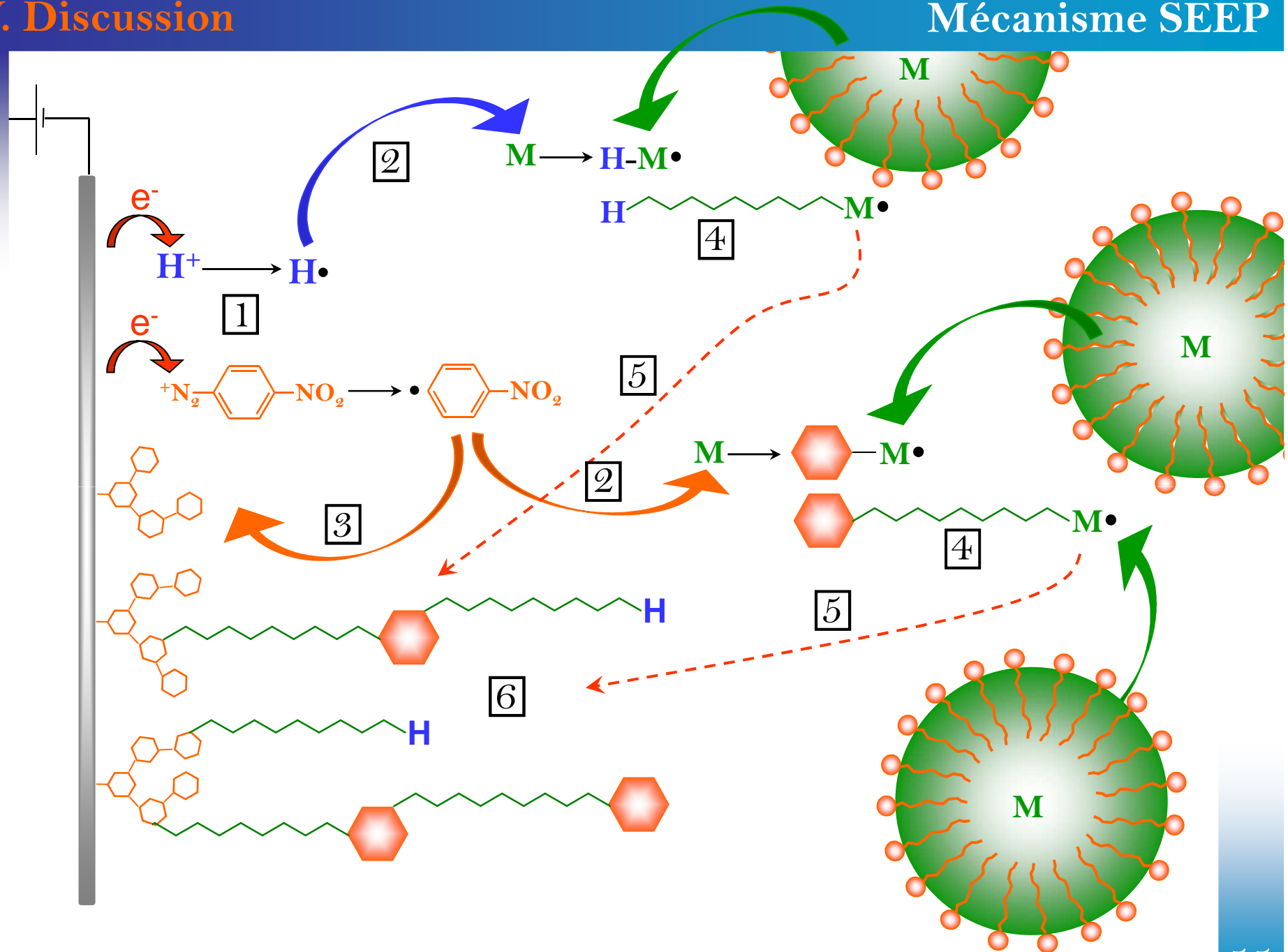
- Amorceurs =  $\text{NO}_2\text{-}\Phi\cdot$  ( $\leftarrow \text{NO}_2\text{-}\Phi\text{-N}_2^+$ ) et  $\text{H}\cdot$  ( $\leftarrow \text{H}^+$ )
- PNP =  $(\Phi\text{-NO}_2)_n$  sous-couche
- Procédé par “Grafting to” (PBMA) / “Grafting from” (PNP)
- Chaînes courtes ( $\text{DP} \approx 10\text{-}12$  estimé par XPS)
- Construction “multicouches”
- Structure des films :  $\Phi\text{-NO}_2 \equiv$  cross-linkers

• Influence du tensioactif

• Influence de l'agitation

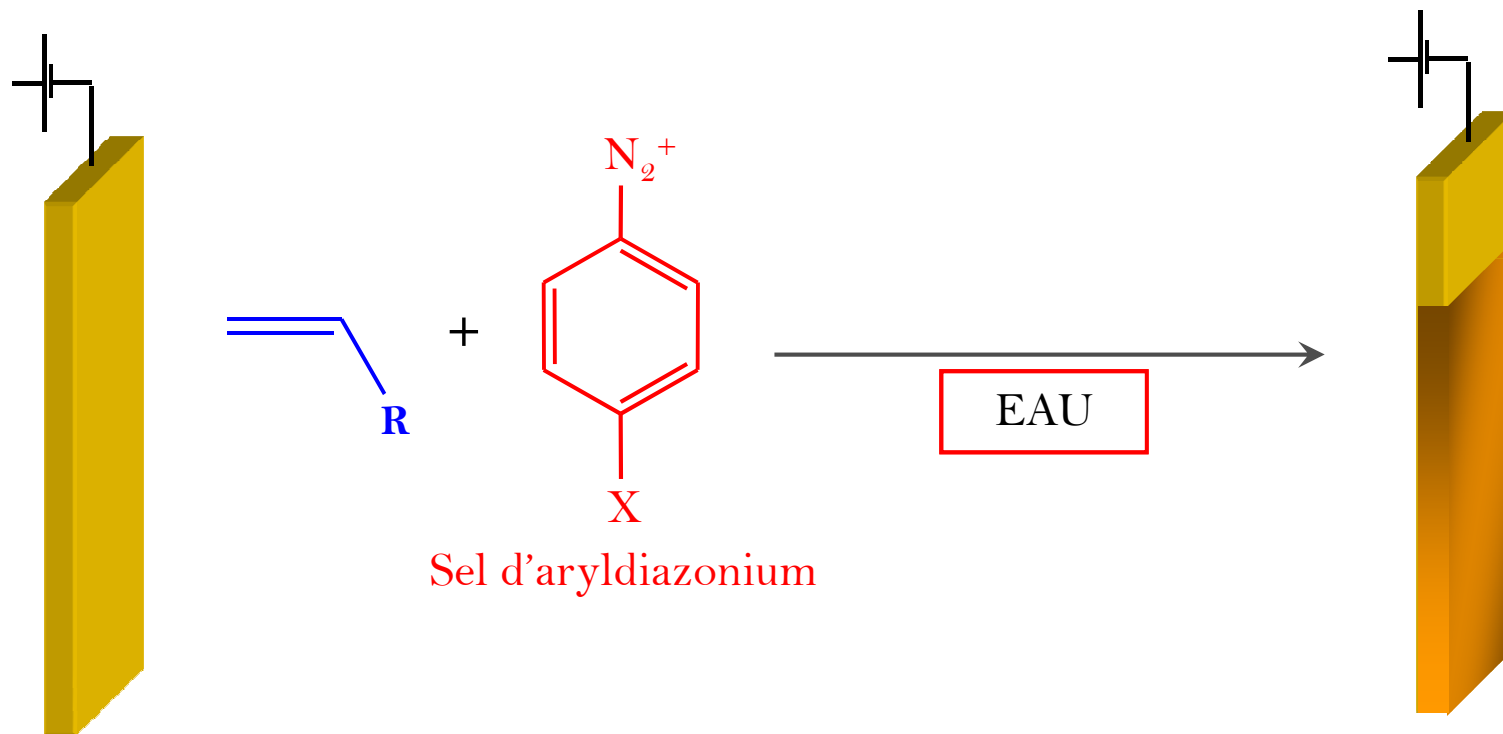


• Interaction spécifique NBD / SDS

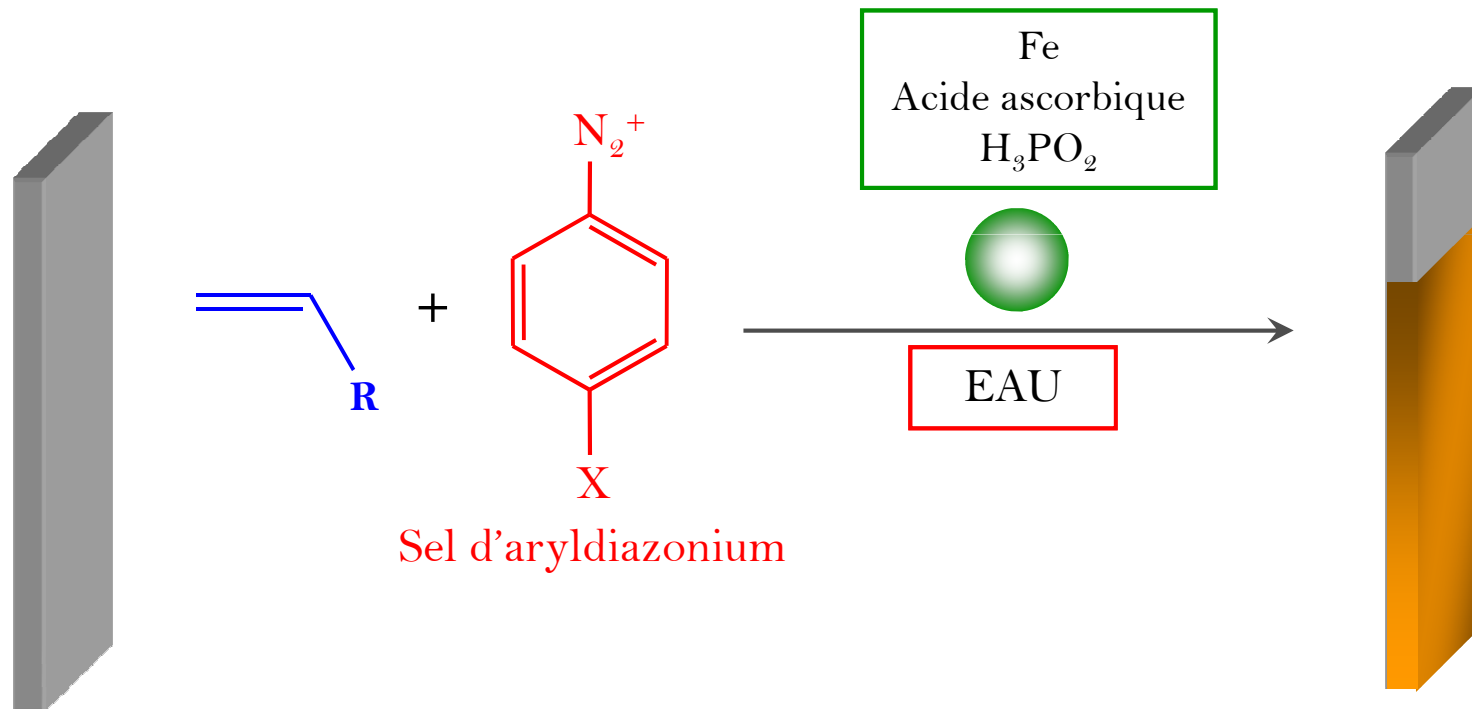




- Liaison d'interface
- Amorçage chimique → Réduction par voie chimique



- Liaison d'interface
- Amorçage chimique → Réduction par voie chimique



☺ Tous types de surfaces

# Remerciements

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Pierre-Antoine CURET

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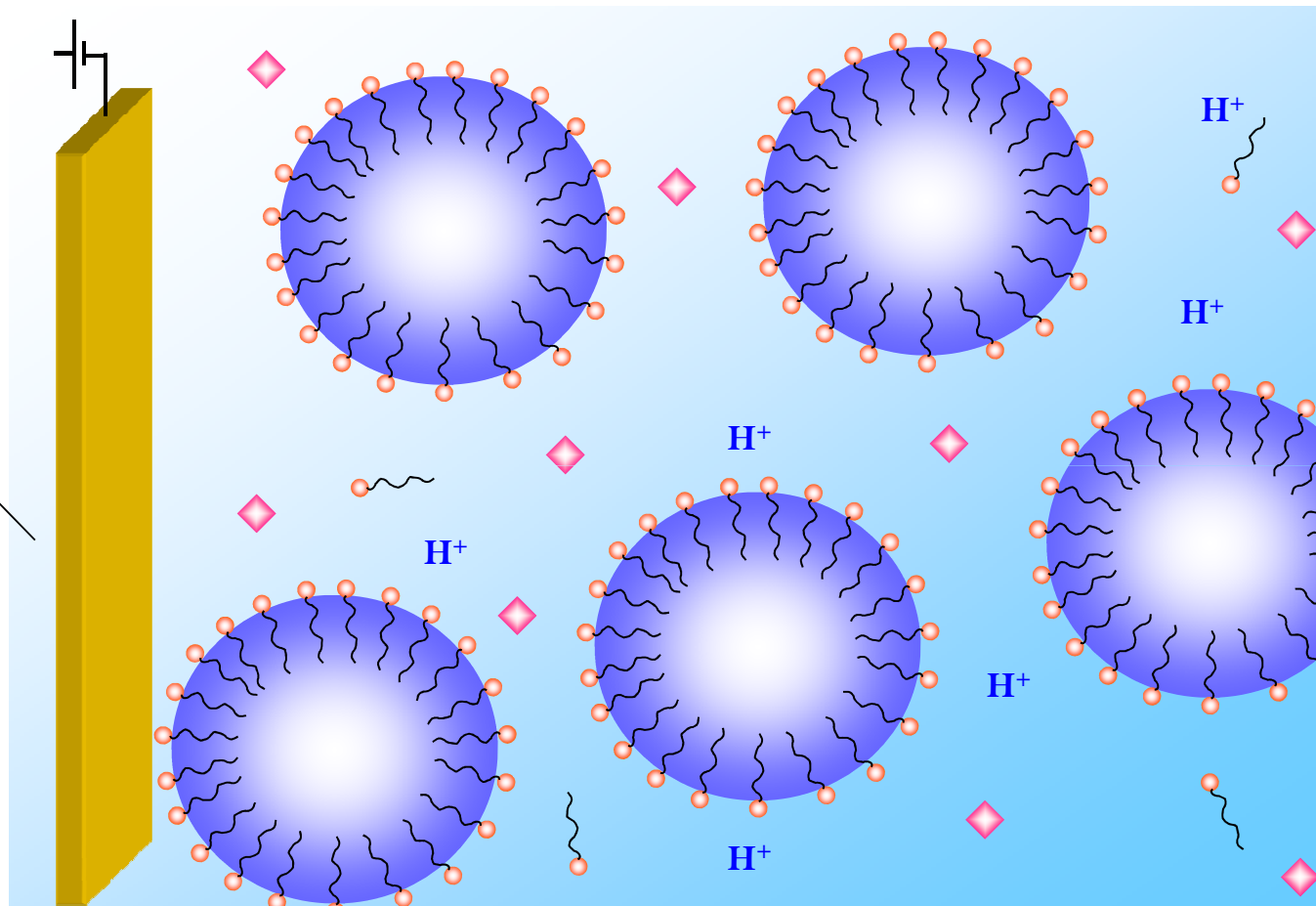
**MERCI pour votre ATTENTION**

• Introduction

- Résultats
- Discussion
- Conclusion
- Perspectives

# Protocole Expérimental

Électrode de travail  
(lame d'or)

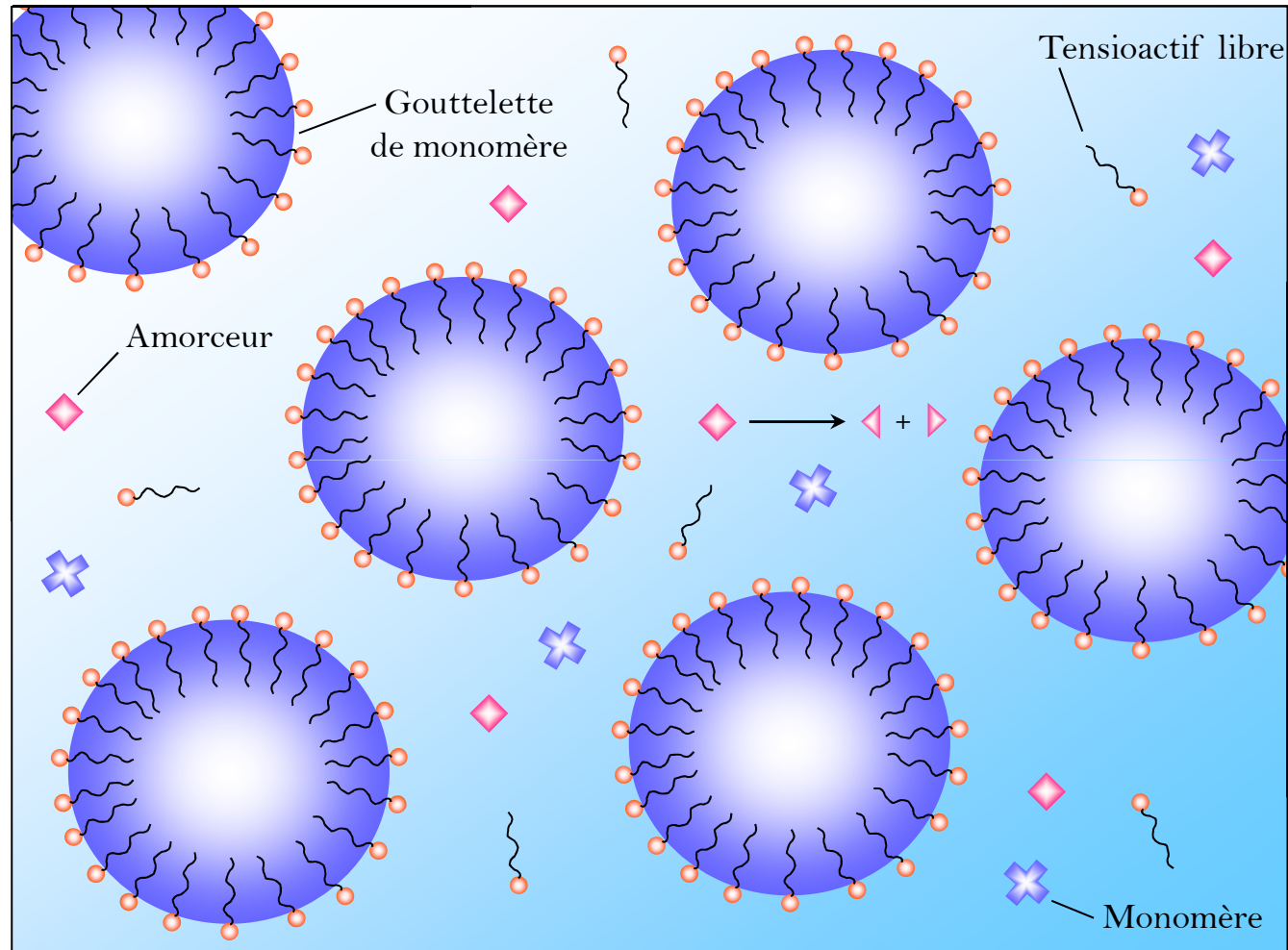


# I. Introduction

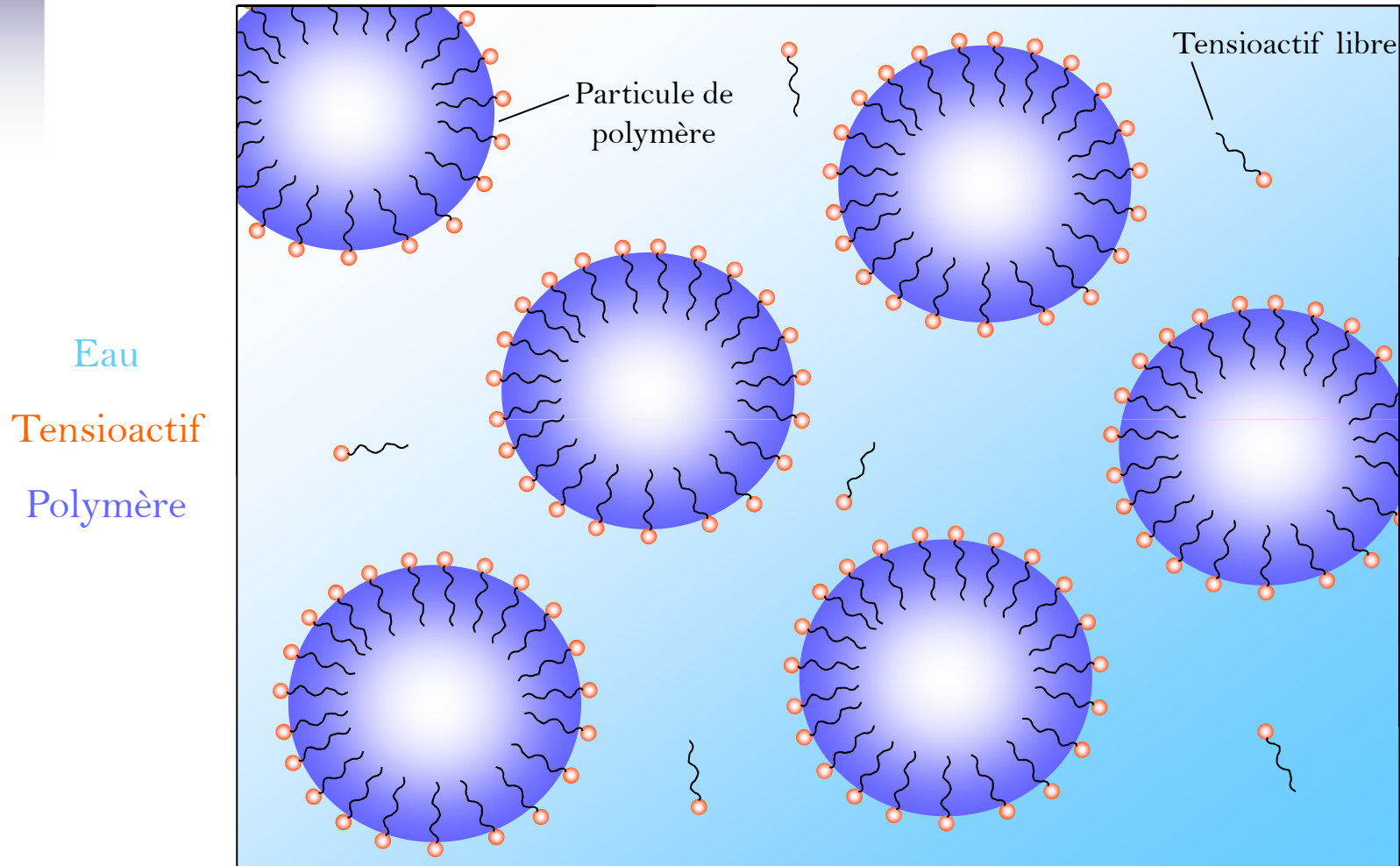
# Polymérisation radicalaire en Miniémulsion

État INITIAL

Eau  
Tensioactif  
Monomère hydrophobe  
Amorceur hydrosoluble



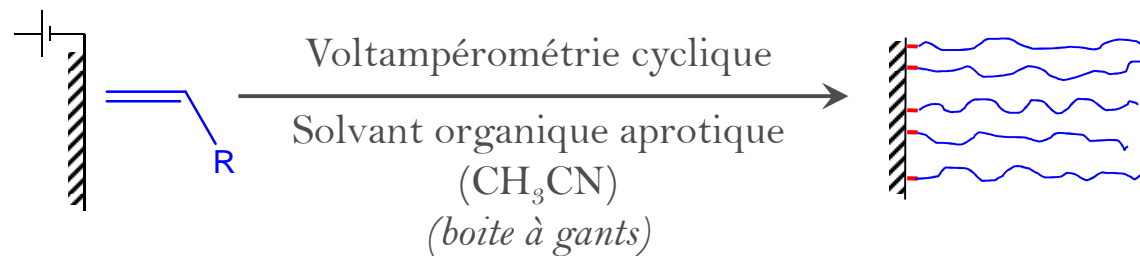
État FINAL



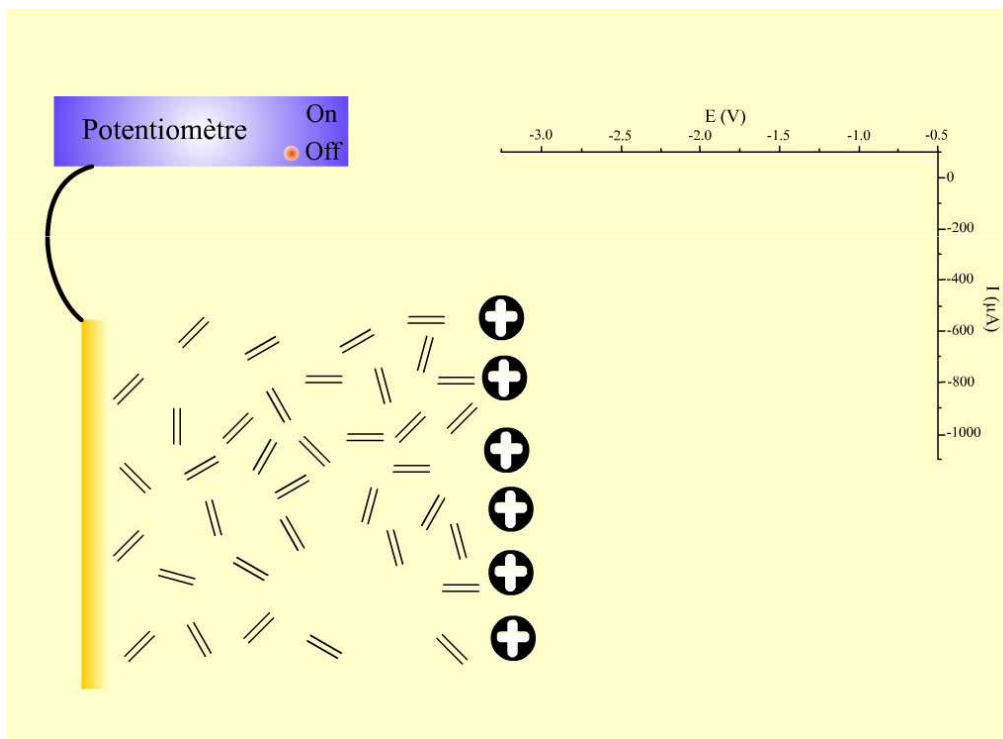
# I. Introduction

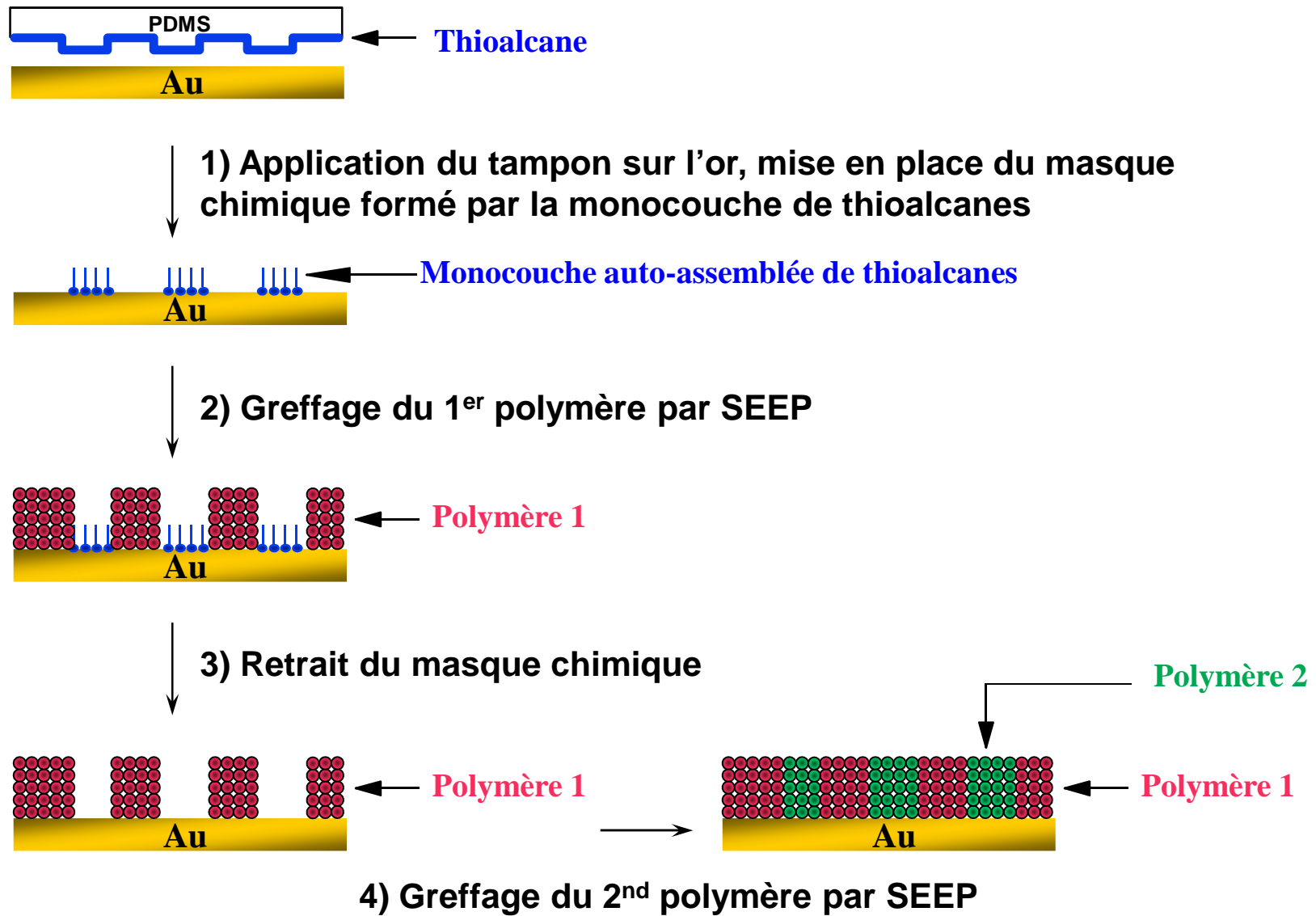
# Électrogreffage Cathodique

Principe

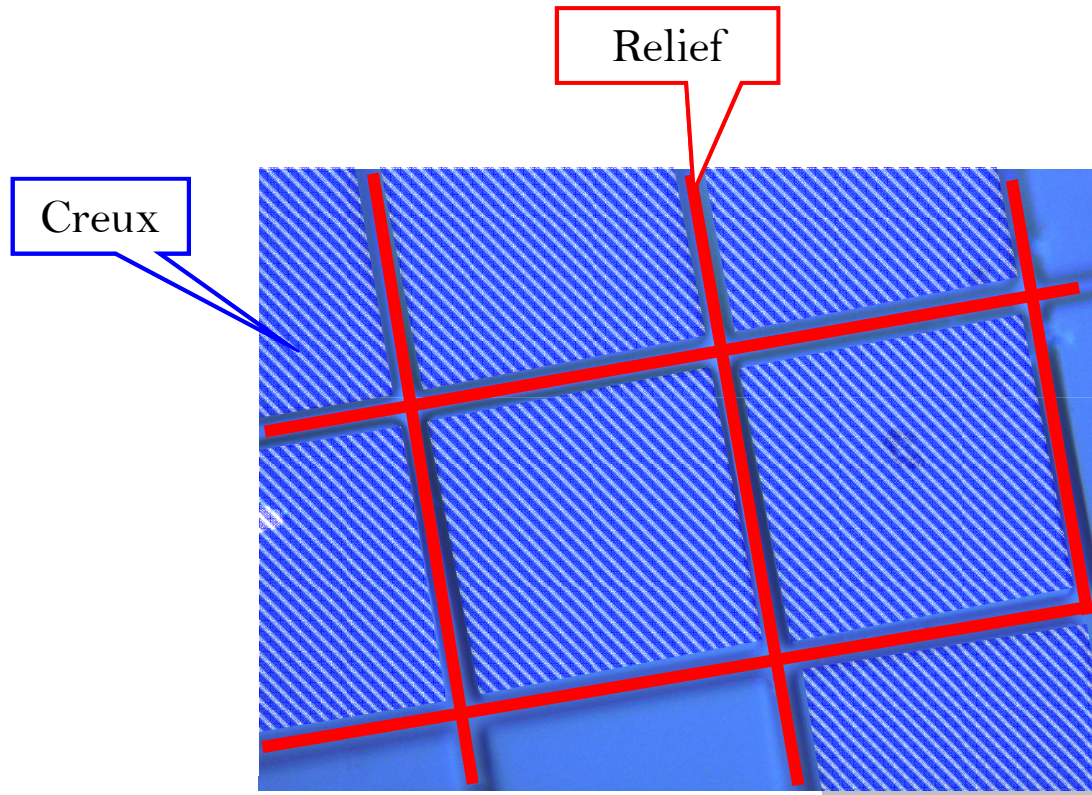


Mécanisme

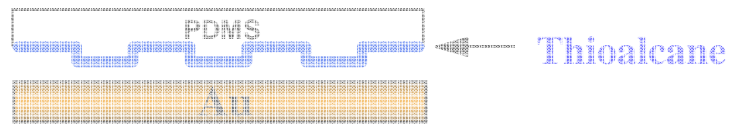








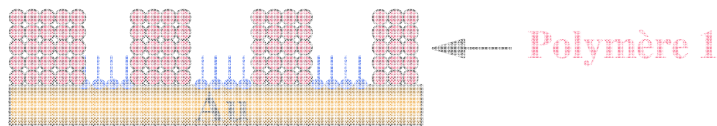
50  $\mu\text{m}$



1) Application du tampon sur l'or, mise en place du masque chimique formé par la monocouche de thioalcanes



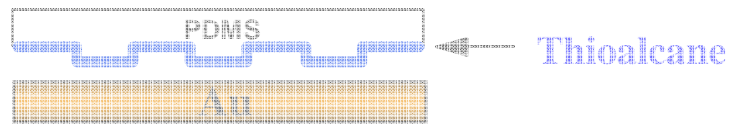
2) Greffage du 1<sup>er</sup> polymère par SEEP



3) Retrait du masque chimique



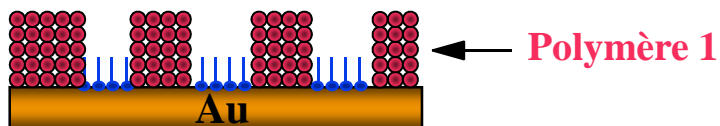
4) Greffage du 2<sup>nd</sup> polymère par SEEP



1) Application du tampon sur l'or, mise en place du masque chimique formé par la monocouche de thioalcanes



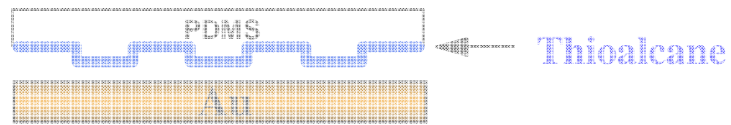
2) Greffage du 1<sup>er</sup> polymère par SEEP



3) Retrait du masque chimique



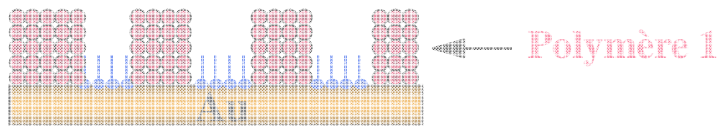
4) Greffage du 2<sup>nd</sup> polymère par SEEP



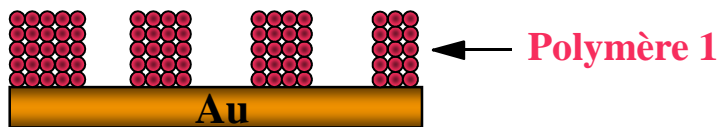
1) Application du tampon sur l'or, mise en place du masque chimique formé par la monocouche de thioalcanes



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3) Retrait du masque chimique



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