



## Study of the adsorption of Nickel ions on Sodium alginate polymeric membrane in hydrometallurgical effluents

[Étude de l'adsorption des ions de Nickel sur une membrane polymérique d'alginate de sodium dans les effluents hydrométallurgiques]

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### Abstract

Mining extraction activities in the Democratic Republic of Congo result in the production of hydrometallurgical effluents containing varying concentrations of metallic ions. These effluents are often discharged into the environment without proper treatment, leading to ecological imbalances and severe illnesses in animals and humans. It is crucial to treat these effluents before releasing them into the natural environment. This study presents a technical approach for developing polymeric membranes capable of adsorbing metallic ions from aqueous solutions of hydrometallurgical effluents. A sodium alginate polymeric membrane was synthesized and characterized using various techniques including X-Ray Diffraction, Fourier Transform Infrared Spectroscopy, X-Ray Fluorescence, Transmission Electronic Microscopy, Differential Scanning Calorimetry, and Mechanical Traction. Adsorption experiments were conducted using aqueous solutions of nickel sulphate prepared in the laboratory and hydrometallurgical aqueous solutions from a factory. The results showed that the adsorption of nickel ( $\text{Ni}^{2+}$ ) ions on the polymeric membrane is faster in the hydrometallurgical solution compared to the nickel sulphate solution. This phenomenon appears to be mainly governed by short-range forces such as Van der Waals forces. The Hill-Langmuir model was used to describe the adsorption experiments, and the analysis of the model parameters indicated that the adsorption of  $\text{Ni}^{2+}$  ions on the sodium alginate polymeric membrane is more efficient in solutions containing only one type of ion compared to complex aqueous solutions. This is due to the competition between different metallic ions present in complex solutions, which are not the case in the nickel sulphate solution where only  $\text{Ni}^{2+}$  ions are present. Furthermore, the analysis showed that the coordination number (n) for  $\text{Ni}^{2+}$  ions in a "receiving" site of the polymeric membrane is smaller in the nickel sulphate solution (2.22) compared to the hydrometallurgical aqueous solution (2.85).

Keywords: Hydrometallurgical effluents, adsorption, polymeric membrane, sodium alginate, Hill-Langmuir model.

### Résumé

Les activités d'extraction minière en République démocratique du Congo génèrent des effluents hydrométallurgiques chargés en ions métalliques à des concentrations variables. Ces effluents sont parfois rejetés dans la nature sans aucun traitement approprié. Les ions métalliques présents dans ces effluents sont directement impliqués dans l'apparition de déséquilibres au niveau des écosystèmes ou à l'origine de maladies graves pouvant conduire à la mort, aussi bien chez les animaux que chez l'homme. Il est impératif de traiter certains effluents hydrométallurgiques avant leur rejet dans l'environnement naturel. Ce travail présente une approche technique

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pour développer des membranes polymériques capables d'adsorber les ions métalliques contenus dans les solutions aqueuses d'effluents hydrométallurgiques. Une membrane polymérique d'alginate de sodium a été élaborée et caractérisée à l'aide de différentes techniques, notamment la diffraction des rayons X, la spectroscopie infrarouge par transformée de Fourier, la fluorescence des rayons X, la microscopie électronique à transmission, la calorimétrie différentielle à balayage et la traction mécanique. Des expériences d'adsorption ont été réalisées en utilisant des solutions aqueuses de sulfate de nickel préparées en laboratoire et des solutions aqueuses hydrométallurgiques provenant d'une usine. Les résultats des expériences d'adsorption ont montré que l'adsorption des ions nickel ( $\text{Ni}^{2+}$ ) sur la membrane polymérique est plus rapide dans la solution hydrométallurgique que dans la solution de sulfate de nickel. Ce phénomène semble être principalement régi par des forces à courte portée telles que les forces de Van der Waals. Le modèle de Hill-Langmuir a été utilisé pour décrire les expériences d'adsorption, et l'analyse des paramètres du modèle a indiqué que l'adsorption des ions  $\text{Ni}^{2+}$  sur la membrane polymérique d'alginate de sodium est plus efficace dans les solutions contenant un seul type d'ion que dans les solutions aqueuses complexes. Cela est dû à la compétition entre différents ions métalliques présents dans les solutions complexes, ce qui n'est pas le cas dans la solution de sulfate de nickel où seuls les ions  $\text{Ni}^{2+}$  sont présents. De plus, l'analyse a montré que le nombre de coordinations ( $n$ ) pour les ions  $\text{Ni}^{2+}$  dans un site "récepteur" de la membrane polymérique est plus petit dans la solution de sulfate de nickel (2,22) par rapport à la solution aqueuse hydrométallurgique (2,85).

Mots-clés : Effluents hydrométallurgiques, adsorption, membrane polymérique, alginate de sodium, modèle de Hill-Langmuir.

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## 1. Introduction

Hydrometallurgical effluents are treated by processes that aim to reduce the polluting load to a level judged acceptable for the environment according to the World Health organization. They use some methods as the adsorption, the precipitation, the electrolysis, etc. The adsorption is the method that implies some adsorbants materials in the process of treatment.

The adsorbants materials present themselves as membrane that one immerses in an aqueous solution of hydrometallurgical effluent. After some time of immersion, the membrane charged of the pollutants coupled to the surface is removed from the aqueous solution. Some adsorbants materials present themselves as a powder that can be pulverized in an aqueous solution of hydrometallurgical effluents. This aqueous solution is filtered then to rid it of the pollutants kept in the fine particles of the adsorbants materials.

The study on the natural adsorbants materials as the frost of silica, the chitine, the chitosane and the clayey materials, worried good number of the researchers. The treatment of hydrometallurgical effluents using some polymeric materials for example, constituted an important topic of research. The mechanisms by which the metallic ions set on an adsorbant in an aqueous solution make the more intervene often of the electrostatic interactions, the strengths of Van Der Waals, of the links covalentes etc. The groupings of the loaded adsorbants negatively as the groupings carboxylic, hydroxyles and phosphoric, are known for adsorb metallic ions in an aqueous solution.

The researchers studied the adsorption of the metallic ions on balls of sodium alginate in an aqueous solution. We mention among these ions, the copper (Veglio et al., 2002), the cadmium and lead (Papageorgiou et al., 2006), the manganese (Gotoh et al., 2004) and the chromium (Araujo & Texeira, 1997). The works of these authors showed that the balls of sodium alginate can serve for the treatment of hydrometallurgical effluents.

The objective of this work is to study the adsorption of ions of nickel of hydrometallurgical effluents by the polymeric membrane of sodium alginate that we elaborated. Other metallic ions as the Mercury, the Copper, the Nickel, the Cobalt, etc., appear among the metallic ions to strong content in

the hydrometallurgical effluents of mining extraction in Democratic Republic of Congo.

## 2. Materials and methods

In this study, the methodology is at a time experimental, objective and systemic. The method consists in observing the phenomenon of adsorption of the ions of nickel on polymeric membrane in an aqueous solution of hydrometallurgical effluents. It also consists in describing in an objective way and to modelise the phenomenon of adsorption observed. For that to make, the polymeric membrane of sodium alginate have been characterized to determine their various physico-chemical properties.

### 2.1. Materials

The alginate of sodium is a natural polymer of chemical formula  $NaC_6H_7O_6$ . This polymer has been gotten in trade. It is synthesized from the alginic acid, extracted from brown algae and made up of carbohydrate units forming a chain (figure 1). It presents itself as a white, odorless powder and without flavor. It is water soluble and insoluble in the alcohol. It is a long chain including a lot of negative loads because of the groupings carboxylic ( $-COO$ ) whose load negatives is counterbalanced by the load positive of the  $Na^+$  ions (figure 1) (Fernandez et al., 2000 ; Lazaridis & Charalambou, 2005 ; Kim et al., 2008 ; Emilie, 2010).

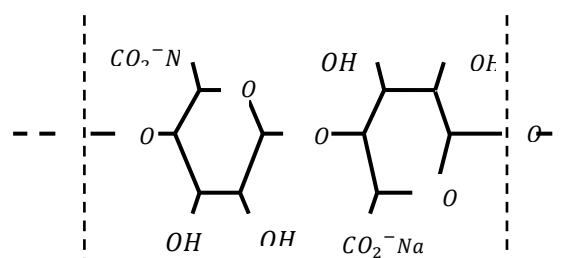


Figure 1. Sodium alginate structure

When the powder of sodium alginate is pulverized in an aqueous solution containing of pressed them divalents as the calcium, the  $Na^+$  ions of sodium alginate are going to exchange themselves with the  $Ca^{2+}$  ions of the aqueous solution. The  $Ca^{2+}$  ions being divalents, every  $Ca^{2+}$  ion is going to replace two  $Na^+$  ions and to interact with two groupings carboxyliques ( $-COO$ ), making a bridge between two parallel chains of sodium alginate (figure 2). This phenomenon is called « polymerization ». Polymerization succeeds to the formation of a frost.

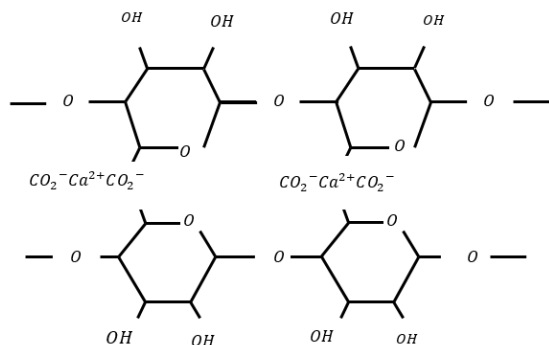


Figure 2. Polymerization in presence of the calcium ion

The used polymer is studied not as powder or frost but rather as a polymeric membrane. The membrane form has been chosen for the applications considered of treatment of the hydrometallurgical solutions. As the powder of sodium alginate used contains some  $Ca^{2+}$  ions, it was sufficient us to pulverize a quantity of this powder in water to get a frost. It is from this frost that the polymeric membrane has been prepared.

To the ambient temperature, 4 g the powder of sodium alginate is pulverized in 4 g of water while the mixture is agitated with the help of a magnetic bar. A transparent and steady frost is gotten after 24 h agitation of the mixture. The stability of the frost depends on the length and the speed of agitation and also of the temperature. A slow gelling to low temperature during a long length of agitation to weak speed drives to a neater structure, and therefore to a frost of bigger stability. A bigger concentration of the powder of sodium alginate in water produces a frost of bigger rigidity. A rigid frost is not adapted for the preparation of the polymeric membrane (Benchabane & Bekkour, 2004). The gotten frost is spread on a plane surface in glass to get a fine layer. The fine layer has been dried to the ambient temperature during 72 h. We then obtain an polymeric membrane of sodium alginate of  $\pm 0,7$  mm thickness.

The polymeric membrane of sodium alginate have been characterized by the X-Ray Diffraction, the Fourier Transform Infrared Spectroscopy, the X-Ray Fluorescence, Transmission Electronic Microscopy, Mechanical traction and by Differential scanning calorimetry.

## 2.2. X-Ray Diffraction

The figure 3 present the X-Ray Diffraction spectrum of the polymer. The peak around 15° watch the amorphous state of the polymer. This result shows that the sodium alginate is an amorphous thermoplastic polymer

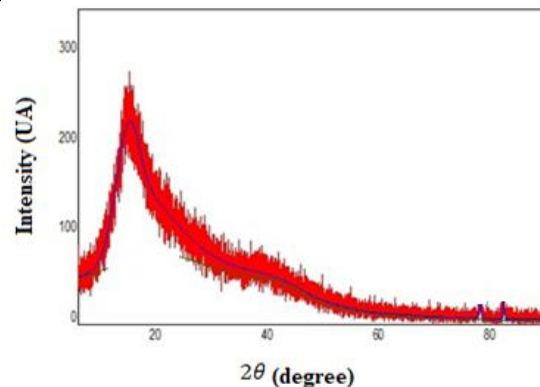


Figure 3. X-Ray Diffraction spectrum of the sodium alginate

## 2.3. X-Ray Fluorescence

The table 1 watch the elements contained in the sodium alginate. In agreement with the works of Kim et al. (2008) and of Singh et al. (2009), the  $Na^+$  ions constitute the main exchangeable ions and assure the balance of the loads in the polymeric chain.

Table 1. Elements contained in the sodium alginate

Elements	Content (%)
Na	11,67
Ca	0,81
Zn	1,45

## 2.4. Fourier Transform Infrared Spectroscopy

The infrared spectrum of the sodium alginate (figure 4) is characterized by strips of vibrations around  $1000\text{ cm}^{-1}$ ,  $1400\text{ cm}^{-1}$  and  $1600\text{ cm}^{-1}$  and a large strip between  $3700\text{ cm}^{-1}$  and  $3000\text{ cm}^{-1}$ .

The two strips to  $1600\text{ cm}^{-1}$  and to  $1400\text{ cm}^{-1}$  are specific of the vibrations of the carbonyl  $C=O$ . The strips of the vibrations observed around  $1000\text{ cm}^{-1}$  corresponds to the vibrations  $C-O$ . The strip of the vibrations observed to  $2934\text{ cm}^{-1}$  corresponds to the vibration of valence  $C-OH$ .

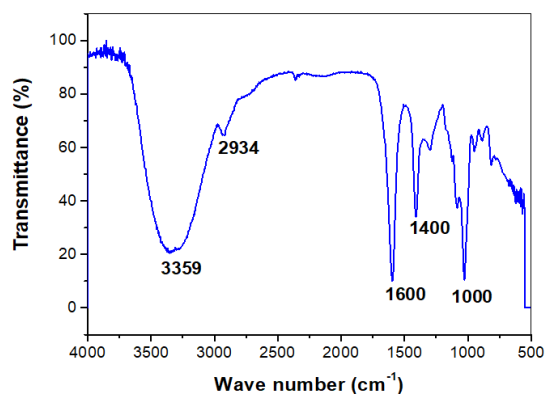


Figure 4. Infrared spectrum of the sodium alginate

The strip of vibrations between  $3700\text{ cm}^{-1}$  and  $3000\text{ cm}^{-1}$  is due to the vibration of valence  $O - H$  features of the natural polysaccharides in agreement with the studies of Abdellahi (2010), Parikh et al. (2006) and Tripathi et al. (2012). According to the same studies, the large observed strip between  $3700\text{ cm}^{-1}$  and  $3000\text{ cm}^{-1}$  account for the sites of retention and absorption of water of the sodium alginate.

### 2.5. Transmission Electronic Microscopy

The energy dispersive spectroscopy Spectrum (figure 5) and the transmission Electronic Microscopy Picture (figure 6) show that the polymer presents a homogeneous and smooth surface with a predominance of the  $Na^+$  ions.

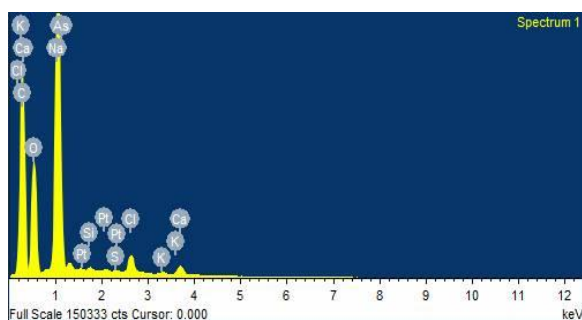


Figure 5. Energy dispersive spectroscopy Spectrum duo to the transmission Electronic Microscopy of the polymer surface

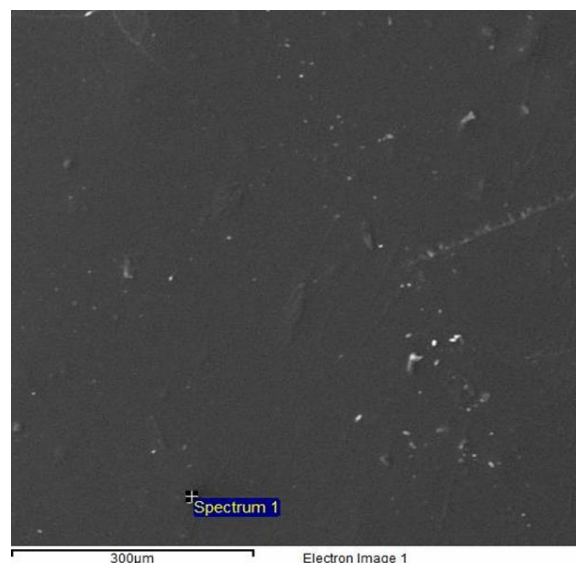


Figure 6. Transmission Electronic Microscopy Picture of the polymer surface

### 2.6. Differential scanning calorimetry

These experiments have been achieved in continuous in the active temperature domain of  $25\text{ °C}$  to  $350\text{ °C}$  with a speed of heating of  $1,7\text{ °C s}^{-1}$ . The steam of nitrogen has been considered like sample of reference. The well stocked excess electric power to the sample compared to the one well stocked to the reference according to the temperature is presented on the figure 7.

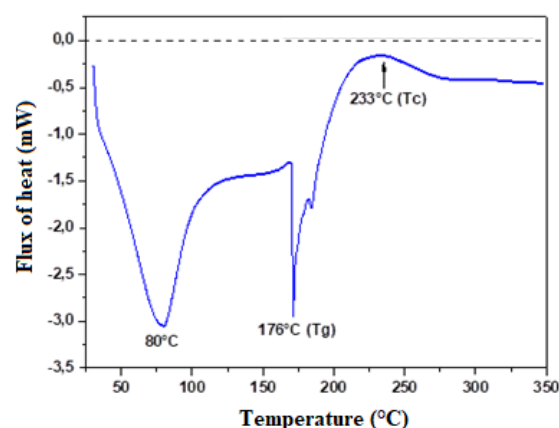


Figure 7. Differential scanning calorimetry curve of the sodium alginate

This figure shows that the studied sample absorbs the energy of  $25\text{ °C}$  until around  $225\text{ °C}$ .

In the interval between 25 °C and 150 °C, the absorption of power is necessary for the dehydration of the sample. While in the interval between 150 °C and 225 °C, the absorption corresponds to the vitreous transition  $T_g$  of the materials. This transition takes place to the temperature of 176 °C. The behavior of the material beyond 225 °C is joined to crystallization. These results illustrate the domain of stability of the polymeric membrane of sodium alginate in relation to the temperature. This stability is compromised beyond the vitreous transition temperature because the crystallization.

### 2.7. Mechanical traction

These experiments of traction have been done on samples of the sodium alginate membrane in the shape of the ribbon of 50 mm length, 10 mm width and 0,7 mm thickness. These ribbon have been stretched with a certain strength to the speed of 10 mm min<sup>-1</sup> until the rupture. The strength of traction  $F$  and the elongation  $\Delta L$  of the ribbon have been recorded.

The table 2 present the strength  $F$  of traction, the elongation  $\Delta L$ , the relative elongation to the rupture  $A_R$ , the resistance to traction  $R_T$  and the springiness module  $E$ .

Table 2. Elastic properties of the polymeric membrane of the sodium alginate

Material	$F(N)$	$\Delta L(mm)$	$R_T(Nmm^{-2})$	$A_R(\%)$	$E(Nmm^{-2})$
Polymeric membrane	42,77	10,5	6,11	21	29,09

### 2.8. Adsorption experiments and results

The adsorption experiments of  $Ni^{2+}$  ions on the polymeric membrane of sodium alginate have been achieved of the following manner : samples of the polymer of 2x2 cm<sup>2</sup> surface and of 0,08 g mass have been prepared. Every sample of the membrane has been immersed during a certain length is in 10 mL an aqueous solution of nickel sulphate, either in an hydrometallurgical solution initially to 10 mgL<sup>-1</sup> in  $Ni^{2+}$  ions. The vestigial solution gotten, after the polymer has been withdrawn, has been analyzed by the atomic absorption spectroscopy to measure the concentration of the  $Ni^{2+}$  ions in the vestigial solution. The concentration of the  $Ni^{2+}$  ions adsorbed on the polymeric membrane has been gotten while making the difference between the concentration of the  $Ni^{2+}$  ions in the vestigial

solution, measured by the atomic absorption spectroscopy, and the initial concentration of the  $Ni^{2+}$  ions, of 10mgL<sup>-1</sup>. The values of this concentration are presented in the figure 3 according to the time of immersion of the polymeric membrane in the considered solution.

Table 3. Concentration  $C(mgL^{-1})$  of the  $Ni^{2+}$  ions adsorbed according to the immersion time of the polymeric membrane in the aqueous solution of nickel sulfate and in the hydrometallurgical solution initially to 10 mgL<sup>-1</sup> in  $Ni^{2+}$  ions

Time (min)	Nickel sulfate aqueous solution	Hydrometallurgical solution
	$C(mgL^{-1})$	$C(mgL^{-1})$
0	0,00	0,00
30	0,22	0,16
60	0,48	0,33
90	0,64	0,42
120	0,70	0,42
150	0,75	0,42
180	0,75	0,42
210	0,75	0,42
240	0,75	0,42

## 3. Results and discussion

In the tables 3, the values of the concentration of the  $Ni^{2+}$  ions adsorbed increase and stretch toward a certain saturation when the immersion time of the polymeric membrane in the aqueous solution increases.

Some researchers showed that the equation of Morgan-Mercer-Flodin could be used to explain the phenomena of the adsorption and the desorption of a species ( $L$ ) named « Ligand » by another species ( $R$ ) named « Receptor » (Muntumosi et al, 2017 ; Gesztely et al., 2012). The equation of Morgan-Mercer-Flodin come from the combination of the equation of Hill-Langmuir and the one of the logistical model of Pierre François Verhulst. The equation of Morgan-Mercer-Flodin is given by the expression :

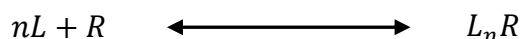
$$[L_n R] = [R_0] + \frac{\beta - [R_0]}{1 + \left(\frac{t}{t_0}\right)^n} \quad (1)$$

Where  $[L_n R]$  represent the concentration of the complex « Ligand-Receptor » ;  $[R_0]$  the total concentration of sites « Receptor » ;  $[L]$  the concentration of the « Ligand »,  $\beta$  a limits value toward which stretches the complex formed in receiving sites,  $t$  is the time of reaction,  $t_0$  represent



the necessary time so that the concentration of the ligands adsorbed by receiving sites is close to the half of the initial concentration of the ligands and  $n$  the named factor coefficient of Hill, represent the number of coordinations for the «Ligand» species in a «Receptor» site. It provides a means to quantify the degree of interaction.

The «Receptor» sites are in the polymer, while the «Ligand» species are  $Ni^{2+}$  ions.



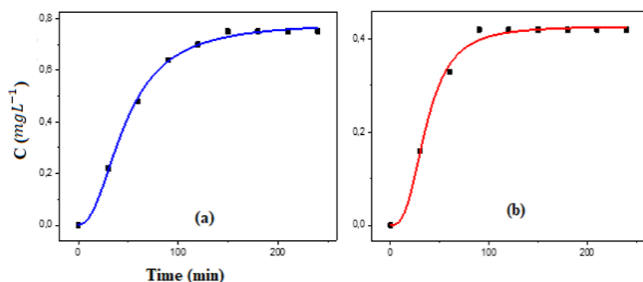
We used a variant of the logistical equation of growth proposed by the software OriginPro8 that is like the equation of Morgan-Mercer-Flodin to analyze the experimental data. The form of this equation is :

$$y(t) = a_2 + \frac{a_1 - a_2}{1 + \left(\frac{t}{t_0}\right)^n} \quad (2)$$

A set of the figures has been gotten. On these figures, the values  $y$  represent the concentration of the  $Ni^{2+}$  ions adsorbed on the polymeric membrane while the values of  $x$  represent the immersion time in the aqueous solution. On these figure, the points indicate the measured values, while the strong curve that ensues of the model shows the behavior of the phenomenon of adsorption of  $Ni^{2+}$  ions in the aqueous solution.

Thus, the figure 8 show the behavior of the concentration of the  $Ni^{2+}$  ions adsorbs according to the immersion time of the polymeric membrane in the aqueous solution of nickel sulphate and in the hydrométallurgical solution.

We observes on these figures that the concentration of the  $Ni^{2+}$  ions adsorbs increases a certain manner when the immersion time of the polymeric membrane in the aqueous solution increases.



**Figure 8.** Behavior of the concentration of the  $Ni^{2+}$  ions adsorbes on the polymeric membrane according to the immersion time of the polymeric membrane in the aqueous solution of nickel sulphate and in the hydrométallurgical solution initially to  $10 \text{ mgL}^{-1}$  in  $Ni^{2+}$  ions

The parameters intervening in the equation of Morgan-Mercer-Flodin adjusted to the experimental data as proposed by the software OriginPro8 are  $a_1 = \beta$ ,  $a_2 = [R_0]$ ,  $t_0$  and  $n$ . These values are presented in the figure 4.

**Table 4.** Parameters intervening in the equation of Morgan-Mercer-Flodin adjusted to the experimental data of the polymeric membrane in the aqueous solution of nickel sulfate and in the hydrometallurgical solution initially to  $10 \text{ mgL}^{-1}$  in  $Ni^{2+}$  ions.

Parameters	Aqueous solution of nickel	Hydrometallurgical solution
$\beta(\text{mgL}^{-1})$	$0,78 \pm 0,01$	$0,48 \pm 0,01$
$[R_0](\text{mgL}^{-1})$	$0,25 \pm 0,01$	$0,08 \pm 0,01$
$t_0(\text{min})$	$47,21 \pm 1,72$	$36,48 \pm 1,75$
$n$	$2,22 \pm 0,16$	$2,85 \pm 0,34$
$R^2$	0,99	0,99
$(\beta - [R_0])(\text{mgL}^{-1})$	$0,53 \pm 0,02$	$0,40 \pm 0,02$

The analysis compared of the parameter toward which stretches the complex formed in receptor sites, the concentrations of the receptor sites  $[R_0]$  and the concentrations of the reactional sites  $(\beta - [R_0])$  for the aqueous solutions of sulphate of Nickel and the hydrometallurgical solution in the figure 4 shows that the value limits ( $\beta$ ) toward which stretches the complex formed in receptor sites in the aqueous solution of Nickel sulphate ( $0,78 \text{ mgL}^{-1}$ ) is from afar bigger than the one toward which stretches the complex formed in receptor sites in the hydrometallurgical solution ( $0,48 \text{ mgL}^{-1}$ ).

The concentration of the receptor sites  $[R_0]$  for the aqueous solution of Nickel sulphate ( $0,25 \text{ mgL}^{-1}$ ) is from afar bigger than the concentration of the receptor sites for the hydrometallurgical solution ( $0,08 \text{ mg/L}$ ). What explains the time of adsorption of  $Ni^{2+}$  ions,  $t_0$  faster in the hydrometallurgical solution that in the aqueous solution of Nickel sulphate.

The concentration of the reactional sites  $(\beta - [R_0])$  of the polymeric membrane in the aqueous solution of Nickel sulphate and in the hydrometallurgical solution aqueous is respectively  $0,53 \text{ mgL}^{-1}$  and  $0,40 \text{ mgL}^{-1}$ .

He gets away of this comparison that the adsorption of the ions by polymeric membrane of

sodium alginate is a lot better in solution including only one type of ion that in complex aqueous solution that is the solutions including several ions.

This situation explains itself indeed by the existence of the competition between the ions in the complex solution, besides  $Ni^{2+}$  ions, the hydrometallurgical solution contains other metallic ions. During the process of adsorption, there is competition therefore between these two types of ion. It is that not the case in the aqueous solution of Nickel sulphate where one meets of it that of the  $Ni^{2+}$  ions.

As for the number of coordinations  $n$  for  $Ni^{2+}$  ions in a Receptor site of the polymeric membrane, we observe that this number is smaller in the aqueous solution of Nickel sulphate ( $n = 2,22$ ) than in the hydrometallurgical Solution ( $n = 2,85$ ).

#### 4. Conclusion

Of the characterization of the polymeric membrane of sodium alginate sample, we keep what follows: an predominance of the  $Na^+$  ions to the surface of the polymer. These ions constitute the main exchangeable ions ; the presence of the sites of absorption and retention of water in the polymer in agreement with the studies of authors ; the polymeric membrane of sodium alginate can not be used beyond  $176\text{ }^\circ\text{C}$  and present a big module of springiness, either  $29,09\text{ Nmm}^{-2}$ .

The study of the phenomenon of adsorption of the  $Ni^{2+}$  ions on the polymer in the aqueous solution of nickel sulphate and hydrometallurgical solution showed that : that when the polymer is immersed in an aqueous solution, it adsorbs the ions of the aqueous solution and this adsorption is very weak ; that the  $Ni^{2+}$  ions that are eliminated in the aqueous solution go in the available sites for the adsorption in the polymer. During this process, the number of the adsorption sites decreases in the polymer until all available sites are saturated by the presence of the  $Ni^{2+}$  ions. The number of the sites of adsorption of the  $Ni^{2+}$  ions on the polymer, limit the phenomenon of adsorption; the adsorption of the  $Ni^{2+}$  on the polymer is faster in the hydrometallurgical solution than in the aqueous solution of Nickel sulphate. The observed phenomenon seems to be an adsorption of the nickel ions to the surface of the polymer governed mainly by strengths of short range, of Van der Waals for example. The efficiency of this

phenomenon is a matter for the specific surface of the polymer.

The analysis compared of the parameters gotten thanks to the model of Hill-Langmuir,  $\beta$  toward which stretches the complex formed in receptor sites, of the concentrations of the receptor sites  $[R_0]$  and the concentrations of the reactional sites  $(\beta - [R_0])$  for the aqueous solutions of sulphate of Nickel and the hydrometallurgical solution shows that the adsorption of the  $Ni^{2+}$  ions by the polymeric membrane to basis of sodium alginate is a lot better in solutions including only one type of ion that in complex aqueous solutions.

The model also showed that the number of coordinations  $n$  for  $Ni^{2+}$  ions in a Receptor site of the polymeric membrane is smaller in the aqueous solution of Nickel sulphate (2,22) than in the Solution hydrométallurgique (2,85).

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