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Par :

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Sur le thème

Polymeric Biomaterial extracted from Local Marine Sources: Properties and application in coagulation/flocculation process

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Dedication

I dedicate this work:

To the most precious people in the world; the source of my pleasure and delight and who have always sacrificed to see me succeed: My parents.

To my siblings: Mohamed Ridha, Kheireddine and Fadia for their encouragement and support.

To my companion and sister Nihel with whom I did this work and spent all of my university years.

And to my best friend Aya and everyone who means a lot to me.

M.Achwak WIssal

Dedication

I have the great pleasure to dedicate this modest work:

To my dearest mother, who always gives me hope to live and who has never stopped praying for me.

To my dearest father, for his encouragement, his support and especially for his sacrifice.

To my sisters, Khaoula, Kawter, Lamis, Hasna, Sarah and Bouchra.

To my brothers, Aymen, Moussab, Hammam, Abdsalam and Souhil.

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To my best friends: Aya, Asma.

To my dear aunts: Nadira and Saliha.

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K.NIHEL

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CHAPTER I: LITERATURE REVUE

CHAPTER III: RESULTS AND DISCUSSION

LIST OF ABREVIATIONS

***DD1**: Degree of deacetylation for one deacetylation process.*

***DD2**: Degree of deacetylation for two deacetylation processes.*

***ER**: Elimination rate.*

***Chitosan 1**: Chitosan extracted from crab shells.*

***Chitosan 2**: Chitosan extracted from shrimp shells.*

***XRD**: X-ray diffraction.*

***IR**: Infrared.*

***FTIR**: Fourier-transform infrared spectroscopy.*

***HNMR**: nuclear magnetic resonance of proton.*

***TU_0**: Initial turbidity.*

***TU_f**: Turbidity of solution after a settling time.*

INTRODUCTION

Nowadays, the increase in the number of industrial areas and their waste discharged into different parts of the environment increases the pollution rate of the soil, the air and especially the water of the seas and rivers**[1]**. Nature is a collection of different unusable substances the reason why the thought of using these substances in pollution control is increasing.

Sea waste such as crab shells, shrimp and crayfish shells are often dumped on the shores of seas and ports, there is growing interest in the extraction of biopolymers from these natural products for biomedical and industrial applications. Some of these important molecules are chitin and chitosan, which has attracted great interest due to its wide range of applications, especially in biomedicine, agriculture, food and technology, and various industries**[2]**.

Worldwide, 1011 tons of chitin is produced as a by-product per year, with an estimated 10,000 tons of industrial use per year**[3]**.

Chitin is a polysaccharide derived from the ocean; it is one of the most abundant and natural polymers in the world and this biopolymer are mainly extracted from the shells of crustaceans, mainly shrimps and crabs**[4]**. Its chemical structure is composed of N-acetyl-D-glucosamine units linked by B-type bonds. The main derivative of chitin is chitosan which is obtained by partial deacetylation in alkaline medium.

Chitosan is an acid soluble amino-polysaccharide with high potential due to its poly-cationic properties which distinguish it from other natural polysaccharides and polymers (usually anionic). The macromolecular chains of chitin and chitosan are characterized by their molecular weight and their degree of acetylation (DD)**[5].**

The aim of this study is as follow, chitin and chitosan are extracted, so it is necessary to find a high DD ratio for the use of chitosan as a coagulant for the treatment of water by the technique of coagulation flocculation by jar-test and verify that turbidity decreases**[6]**.

The presented work is divided into three chapters:

- The first chapter is composed of four sections:

-The first contains a generalization on chitin, its chemical structure, physicochemical properties, its source, also the extraction of chitin from the nature and the degree of deacetylation.

-The second one discusses a generalization about chitosan, its chemical structure, physicochemical properties, source and degree of deacetylation and application of chitosan in several fields.

-The third part dedicated to a brief overview of bentonite, discussing the properties, areas of use and studies of natural settling.

-The fourth is devoted to the method of coagulation flocculation, talking about its definition, principle of technique, type of coagulants flocculants, factors that affect the quality of process, effective agents of chitin and chitosan for water treatment.

- The second chapter represents the products used in our experiment, the operating mode of the extraction of chitin and chitosan as well as the various methods of characterization.
- The third chapter gives a presentation of the different results and discussions of:

- Synthesis results (yield and structure, solubility test).

-Characterization of chitin and chitosan by infrared (IR) and X-ray diffraction (XRD).

- Calculation of DD by potentiometry and conductimetry titration.

- Study of coagulation flocculation on jar-test by chitosan for bentonite removal.

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LITERATURE REVUE

PARTI: CHITIN

I.1. Generalities

Chitin is a polymer frequently found in nature, a polysaccharide similar in structure to cellulose with the difference on the presence of an acetamide group in the first **[1]**.Its chemical structure is a chain of N-acetyl-β-D-glycos-amine units linked by glycosidic bonds (1→4)**[2].**

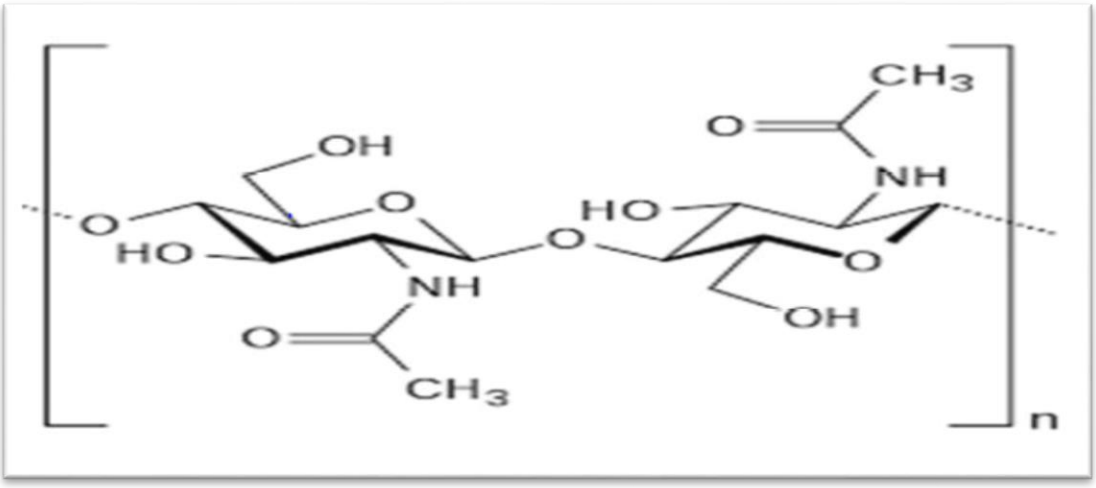

Figure I.1 *Structure of chitin**[3]**.*

The first discovery of chitin was in 1811 by a Frenchman: Hennery Bracannot, who extracted it from mushrooms. Several researchers were interested in processing this new substance using different chemicals and some obtained bravery on the extraction or use of chitin**[2]**.

I.2. Structure

"Animal cellulose"**,** is the second appellation given to chitin because of the similarity between these two most used polymers. It is a polymer: formed with one type of monomer units (homopolymer) which is β-1, 4 N- acetyl-glycos-amine**[4]**.

Chitin is a crystalline material; this character is given to it due to the presence of high inter-molecular forces for the existence of hydrogen bonds between hydroxyl and acetamide groups. It presents in three crystalline forms:

α-chitin

The alignment of the macromolecular chains is anti-parallel. It is the most abundance and the stable form between the three forms because of the existence of hydrogen bonds; it has the higher crystallinity rate**[5]**.

β-chitin

This structure is less stable because of the parallel alignment of the chains; it is characterized by the high affinity for water**[2]**.

γ-chitin

It is a combination of the two structures alpha and beta with intermediate properties**[2]**.

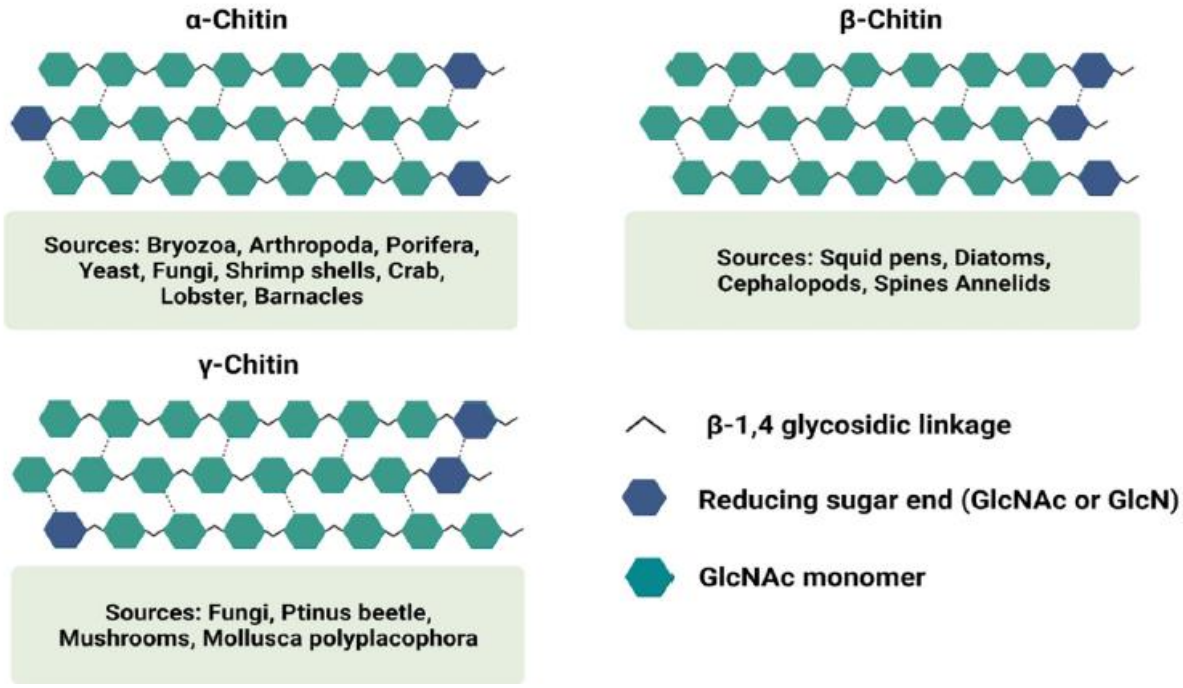

Figure I.2 *Chemical arrangement of chitin chains in the different forms**[6]**.*

I.3. Physicochemical properties of chitin

Chitin is non-soluble in all the common solvents**.** The high attraction between the chains of the polymer makes it difficult to separate and gives to the polymer a mechanical stability**[4]**. This character of insolubility makes its manipulation more difficult to achieve, for that several derivatives are prepared from chitin using modification reactions to have polymers easily implemented**[2]**.

I.3.1. Degree of deacetylation

An alkali treatment can make changes in the structure of chitin; with the presence of a base, the acetamide groups will be transformed to amino groups with acetyl elimination which decreases the rate of acetylated monomers in the polymer.The quantification of the changed monomers is named the degree of deacetylation (DD)**[2],** whereas the degree of acetylation (DA) refers to the acetylated monomers in the chains.

DD is an important parameter in chitin which affects all its properties such as solubility and crystallinity. In chitin, DD value is between 5 and 15%**[4]**and the DA is up to 50%**[2].**

Chitin has other characteristics such as the white color, rigidity, non-toxicity, the capacity to be naturally degradable**[4]**and its decomposition temperature around 250°C**[2].**

I.3.2. Macromolecular weight

The macromolecular weight of chitin can reach 8×10^5 to 10^6 g/moles for DD values in the range of 70%**[5].**

This important character can be determined using viscosity measurement of chitin solution using the relation of MARK HOWINK**(A),[7]**:

$$[\eta] = K \times (M_v)^a \text{……. (A)}$$

With

$[\eta]$: Intrinsic viscosity.

K and a: Constants depending of the nature of solutions and temperature.

M_v: Viscosimetric molar weight.

I.4. Extraction methods of chitin

The extraction of chitin from the natural sources can be achieved using several methods; the most commonly used is the chemical extraction which is based on the use of chemical reagents (products).

A purification of the crustacean (or the chosen source) should be done to remove intercalated impurities in the shells, and then the purified product is crushed into powder in order to start the important steps on the production of chitin.

I.4.1. Chemical extraction

The chemical extraction is composed by two important steps:

Deproteinization: In this stage shell powder is treated with alkaline solution, NaOH is generally used in this step to eliminate proteins**[8]**.

Demineralization: Carbonate calcium is separated from the product obtained by the deproteinization using acidic treatment with nitric acid, sulfuric acid … but chlorhidric acid is recommended to be used in this stage**[8]**.

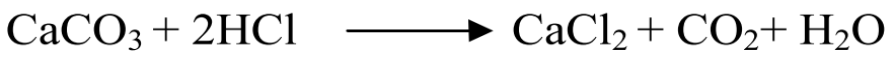

Figure I.3 *Demineralization reaction for chitin production**[3]**.*

A decolorization process can be carried out to obtain a colorless product. This step can produce damages for the structure of the polymer because of the need to use an oxidant reagent and the polymer will be risked to be oxidized or dissociated**[2]**.

Another interesting method for the extraction of chitin is the biological extraction which is based on the use of enzymes and bacteria.

I.4.2. Comparison between biological and chemical extractions efficiencies

K.K Gadgeyand al. [8]had reported chemical and biological extraction of chitin from crab shells done by several researchers. For the biological method they used lactic acid as reagent for the demineralization and specific bacteria for the deproteinization using a protease and fermentation processes while the demineralization of the chemical method was carried out using four inorganic acids to see the effect of each one and deproteinization using alkali treatment.

The results show that chitin obtained from chemical extraction had several qualities according to the acids used, but chitin extracted with bacterial process had a high quality. A

positive point of the use of lactic acid is that the residual proteins and minerals can be reusable in animal nutrients.

I.5. Sources of chitin

As mentioned above, chitin is a biopolymer; its sources are generally found in nature. The table below shows some sources and the chitin content in each source:

Table I.1 *The rate of chitin in some sources**[9]**.*

The source	% of chitin
Brown shrimp shells	21.53
Pink shrimp shells	23.72
Squid pens	49
Crab shells	16.73

PART II: CHITOSAN

II.1. Generalities

Chitosan is a polysaccharide with special characteristics. The first manufacture of this species took place in 1859 by a chemical researcher C. Roget who involved a modification of chitin by adding a basic solution at a high temperature. The name chitosan was first introduced by H. Seyler to distinguish between the two polymers in 1894**[2]**.

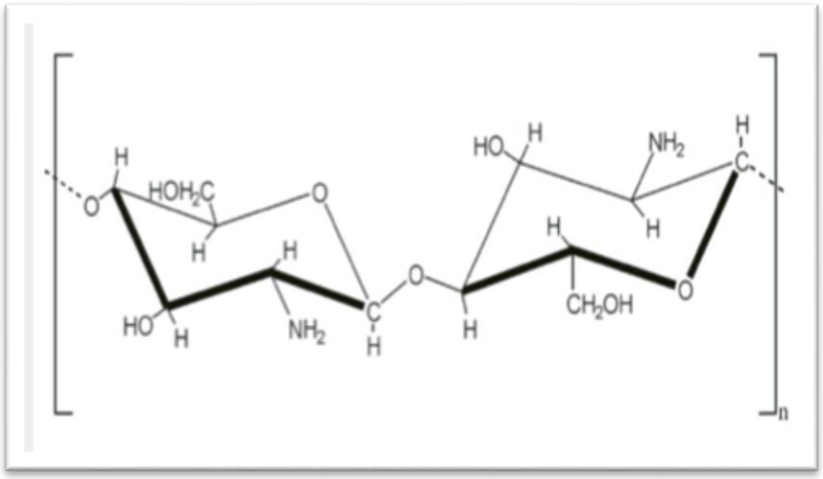

Figure I.4 *Structure of chitosan**[10]**.*

Chitosan has a structure related to that of chitin; the principal structural differentiation between these polymers consists in the acetamide groups of chitin which are replaced by amine groups in chitosan**[11].**

II.2. Chemical structure of chitosan

Chitosan is a linear copolymer with a random arrangement of monomer units and one of the derivatives of chitin**[2]**.

The structural changes of the starting polymer influence its crystallinity character. The substitution of acetamide groups by amine groups decreases the cohesive forces between the polymer chains with a decrease in the number of hydrogen bonds which give the polymer an ordered structure. For that chitosan has less ordered structure comparing to chitin but it still has a semi-crystalline form because of the incomplete deacetylation of all the monomers.

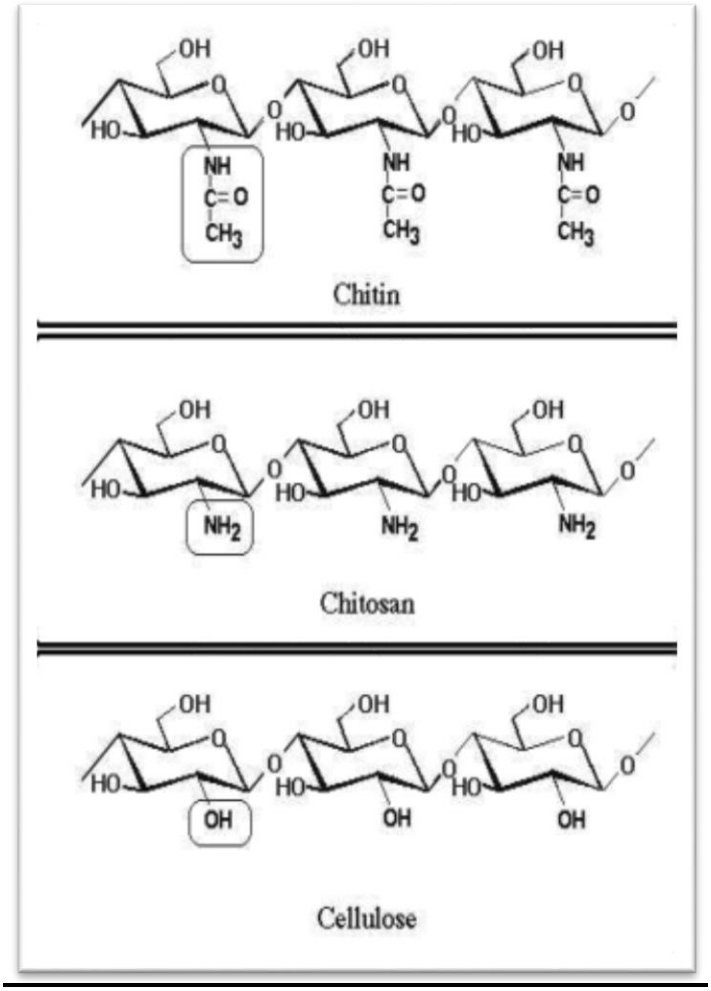

Figure I.5 *Comparison between cellulose, chitin and chitosan structures**[4]**.*

II.3. Physicochemical properties

The anti-microbial, anti-bacterial, anti-oxidant properties and also its nature as a poly-electrolyte which can be positively charged are the most important characteristics of chitosan**[11].**

Chitosan is a biodegradable and hydrophobic polymer for to the presence of acetamide groups in its structure**,** whereas it is soluble in acidic diluted solutions for the protonation of amine groups in pH under 6 **[11].**

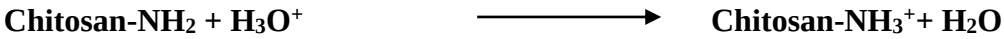

Figure I.6 *The protonation of amino groups in acidic solution**[3]**.*

The degree of deacetylation in chitosan is from 70% to 95% [4] which means that in chitosan the number of amino groups is up to the number of acetamide groups and the

macromolecular weight is in increase because of the difference of weight between the two groups**[11].**

II.3.1. Determination of the degree of deacetylation of chitosan

ES.ABDOU et al.[9]extracted chitin and chitosan from different sources(crab, shrimp…), characterized the products and determined the DD using different analytical methods:

Potentiometric titration

Prepared solutions of alpha, beta chitin and chitosan composed of water and chlorhydric acid was titrated using basic solution (the titrant NaOH), the set of pH values obtained after each addition of HCl gives a curve with two inflections. The determination of the volumes added in these two points serves to calculate the DD values.

NMR spectroscopy

^{1}HNMR aims to the detection of protons and the determination of the proportion of each type of hydrogen in the structure to analyze.

Chitosan extracted from pink shrimp shells, with 40%NaOH, was characterized by ^{1}HNMR , the results are represented in the figure **I.7**.

The most important peaks on this study are those of acetamide and amino groups (situated on 2.06 and 4.9 ppm respectively) which refer to the amount of acetylated and deacetylated monomers in chitosan and the DD is calculated using the number of H in each peak:

$$DD\% = \left(\frac{numberofHon\ 4.9}{numberofHon\ 4.9 + (numberofHon\ 2.06)/3}\right) \ldots\ldots\ldots (B)$$

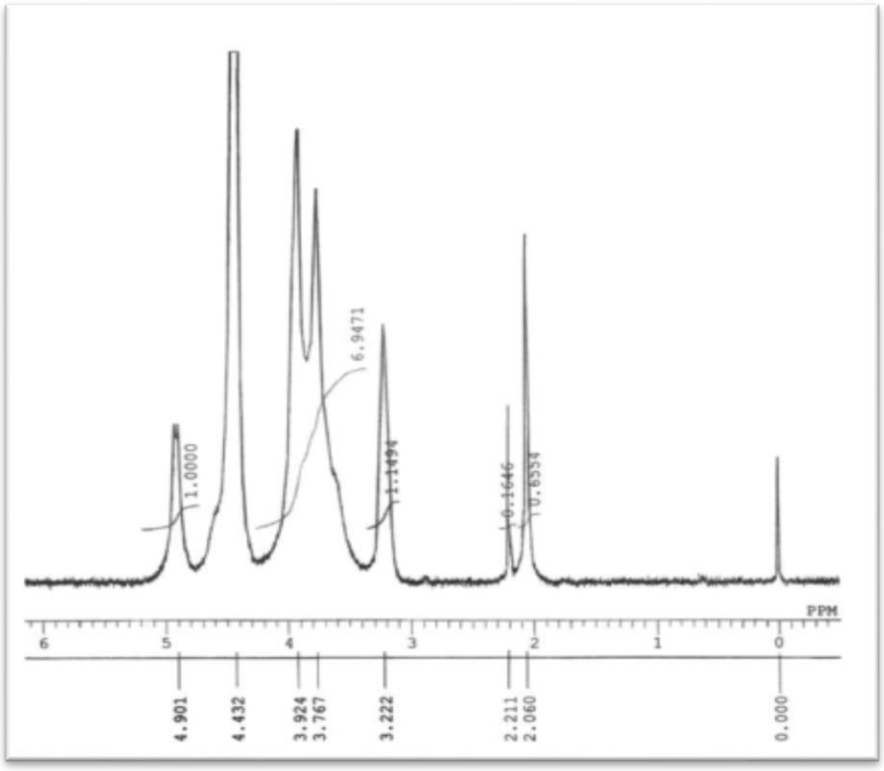

Figure I.7 *^{1}HNMR results of chitosan extracted from pink shrimp with 40%NaOH and 1 day of deacetylation in autoclave**[9]**.*

II.4. Sources and chemical extraction

Generally, chitosan is extracted from chitin by a reaction of deacetylation which partially transforms acetamide groups into amino groups. This procedure needed an alkali treatment at a high temperature**[2]**.

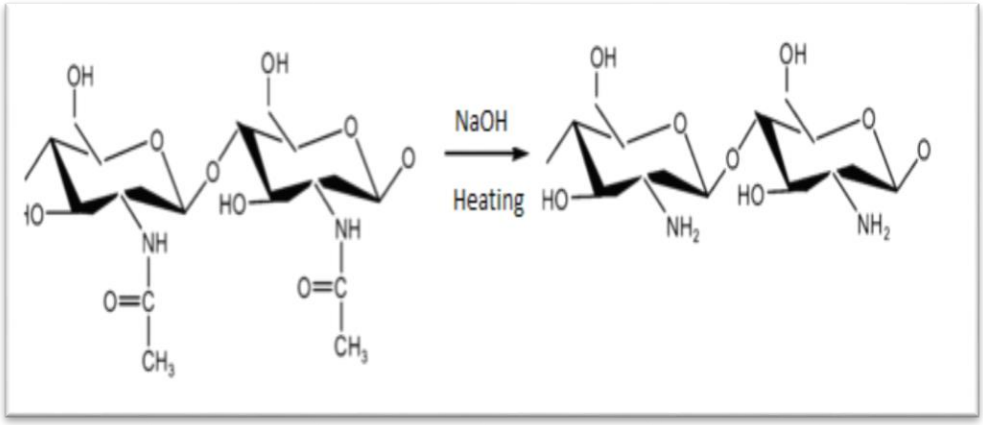

Figure I.8 *Deacetylation reaction**[12]**.*

II.5. Application Fields of chitosan

Chitosan has several chemical and biological properties the reason why its domains of application are varying. It can be used in pharmaceutical and medicinal uses, paper manufacture, textile sewage treatment, agriculture, cosmetics, and food processing**[12].**

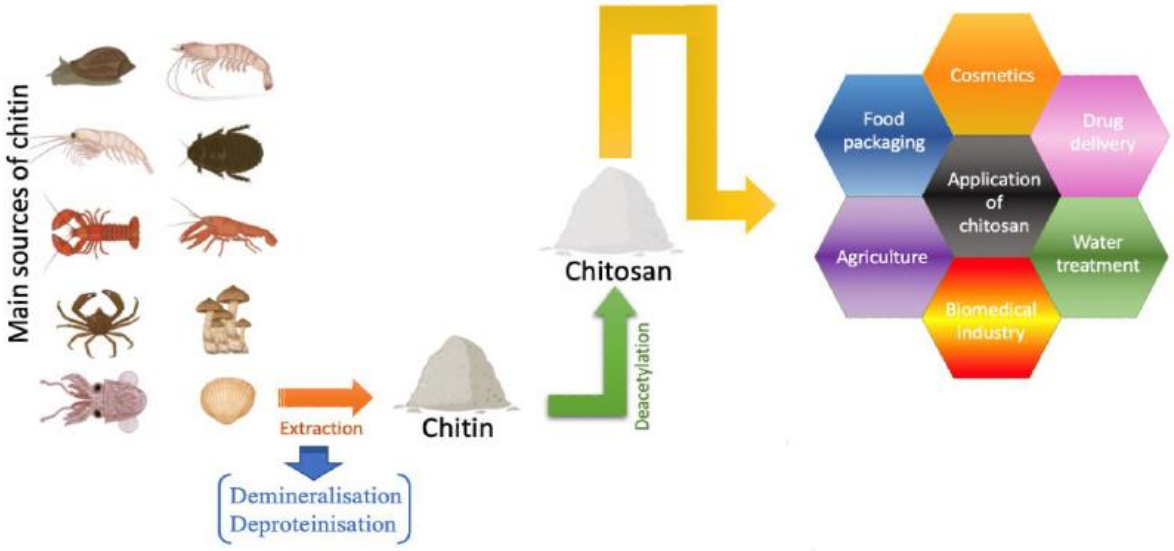

Figure I.9 *Resume of chitin, chitosan extraction and some applications**[11]**.*

PART III: BENTONITE

III.1. Generalities

Clay minerals are formed from the physical and chemical weathering of rocks they are often major constituents of soils. Bentonite is a type of these clays which main mineralogical component is montmorillonite. It is characterized by a high capacity for adsorption, ion exchange and swelling**[13]**.

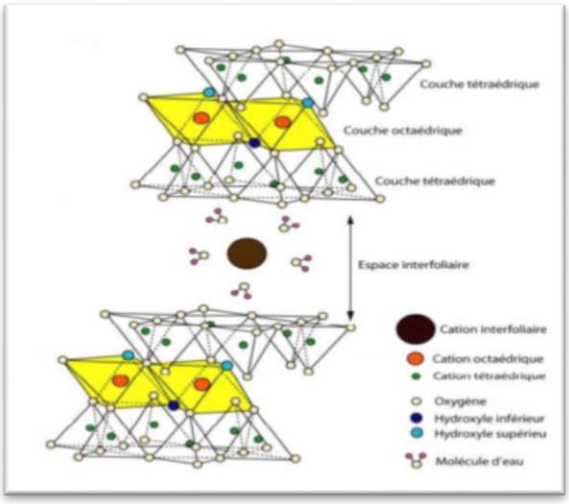

Figure I.10 *Montmorillonite structure**[13].***

III.2. Properties and domains of use

The interest of scientists and researchers in introducing bentonite into various fields of study is due to its interesting properties. It has a high adsorption capacity because of the important specific surface which include the exterior area and the surface between the sheets of montmorillonite. This character also implies the capacity of swelling especially in case of intercalation of water molecules between the layers and involved their separation**[13]**.

Bentonite can be employed in several fields such as cosmetics, pharmaceutical applications and especially in the retention of heavy metals for water treatment**[13].**

III.3. Study of the natural sedimentation of bentonite

T.Hocine [13]had studying the effect of pH (3, 7 and 9) and the turbidity level (30ppm, 100ppm and 500ppm)on the natural decantation of bentonite. The solution of high content of turbidity presented a high and fast rate of sedimentation. The solution of 30ppm was the most difficult to be decanted.

The rate of sedimentation was in increase with the decrease of pH values because of the positive surface charges of bentonite in acidic medium for the protonation of hydroxyl groups and the possibility of interaction between these charges and the permanent negative charges for the other particles.

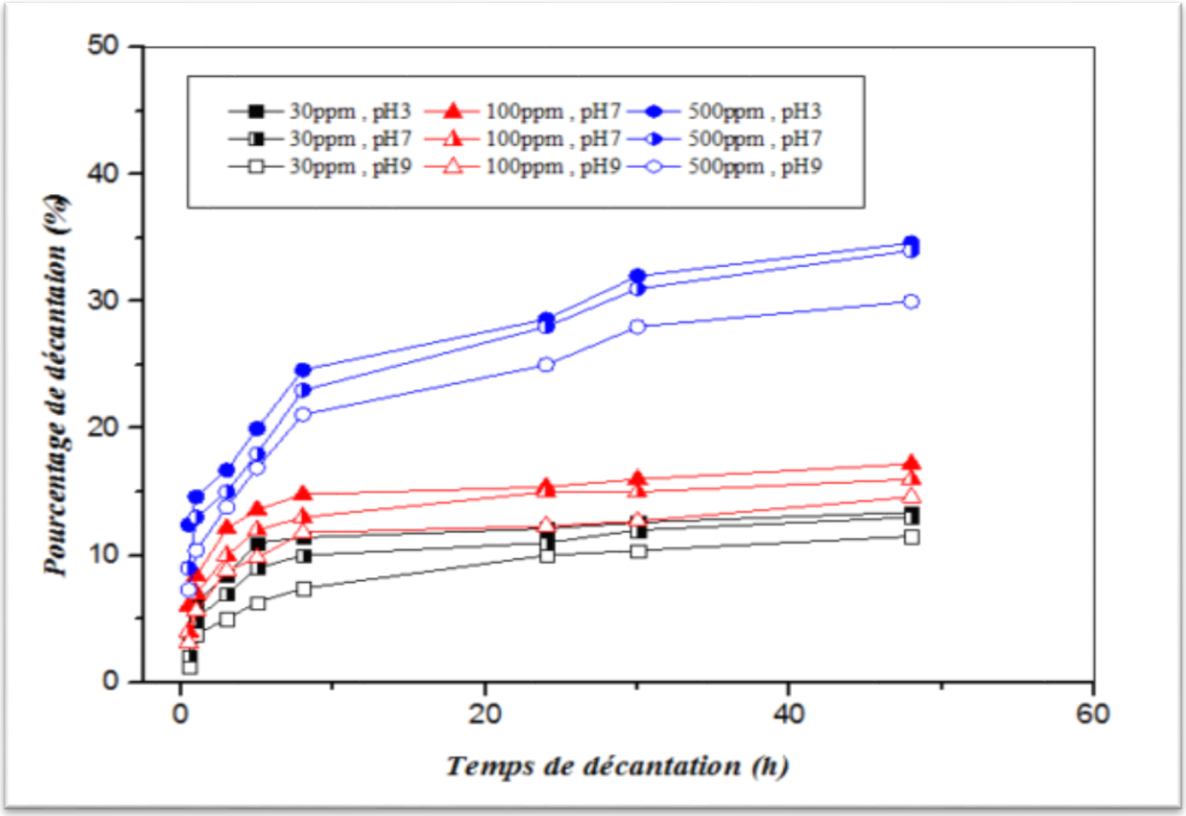

Figure I.11 *Results of natural sedimentation of bentonite as a function of concentration and suspension pH* ***[13]****.*

PART IV: COAGULATION/FLOCCULATION PROCESS

IV.1. Definition and principle

Coagulation-flocculation is a process often used to treat polluted factories water before being discharged into rivers or marines' water, or even purified for industrial reutilization. It serves to eliminate suspensions and colloids**[14]**. This procedure is composed of two parts:

The coagulation: In this stage, the colloidal suspensions are destabilized by the neutralization of charges using a coagulant such as polyelectrolytes and metal salts**[7].**

The flocculation: In this step the destabilized molecules are combined and constitute flocks which can be eliminated by a simple decantation or floating and then filtration**[7].**

IV.2. Types of coagulants/flocculants:

The use of coagulants is based on the nature of pollutants to eliminate, the table below shows some types of these species currently used in water treatment:

Table I.2 *Coagulants/flocculants types and application for water treatment**[15]**.*

Type of coagulant/flocculent type	Examples	Domain of use
Metal salts	-calcium oxide CaO. -Ferric chloride $FeCl_3$. -Ferrous sulphate FeSO4.	-Removal of heavy metals (metallurgical industry). -Removal of oils and greases -Removal of phosphate from wash water
Polymerized metal salts	Aluminum-based and iron-based	Water treatment for turbidity elimination
Biological polymers	-Alginates -Starch. -Chitosan.	Several uses with aluminum salts

IV.3. Factors influences the quality of coagulation flocculation process

There are several parameters which influence the coagulation/flocculation such as:

a. **Temperature:** A low temperature can affect the contact speed of the particles which makes the process slow.
b. **Concentration of coagulant**: Augmentations of the concentration of coagulant produce a saturation of the colloidal substances and a limitation of the flocculation**[7]**.

c. **pH:** The pH of the solution to treat should be manipulated because of the presence of a value of pH specific for each product to analyze in which the elimination of turbidity or colors is optimal.

IV.4. Chitin and chitosan effective agents for water treatment

Chitin and chitosan were employed in various studies for the elimination of dyes and metals from wastewater.

D.S.Shirsath et al.[16]used chitin as adsorbent for dye removal from aqueous solutions and studies the influence of several parameters for the quality of elimination of Ponceau-S dyes. The process started by a chemical extraction of chitin from crab shells using chemical method then solutions of different concentrations on dye are prepared (from 10 to 100mg/L) and stirred to be homogenized. The effects of the initial concentration of colorant, contact time, pH of the solutions and the concentration of adsorbent in the rate of adsorption.

The results of the study indicate that the low concentrations on adsorbent and the low values of pH promoted the elimination of the pollutant until an optimal point then the adsorption was decreasing. The adsorption of dye decreased with its concentration decrease for the saturation of the adsorbance sites of chitosan. The last effect was the time of contact between chitosan and Ponceau-S dye which increased the rate of elimination with its increase.

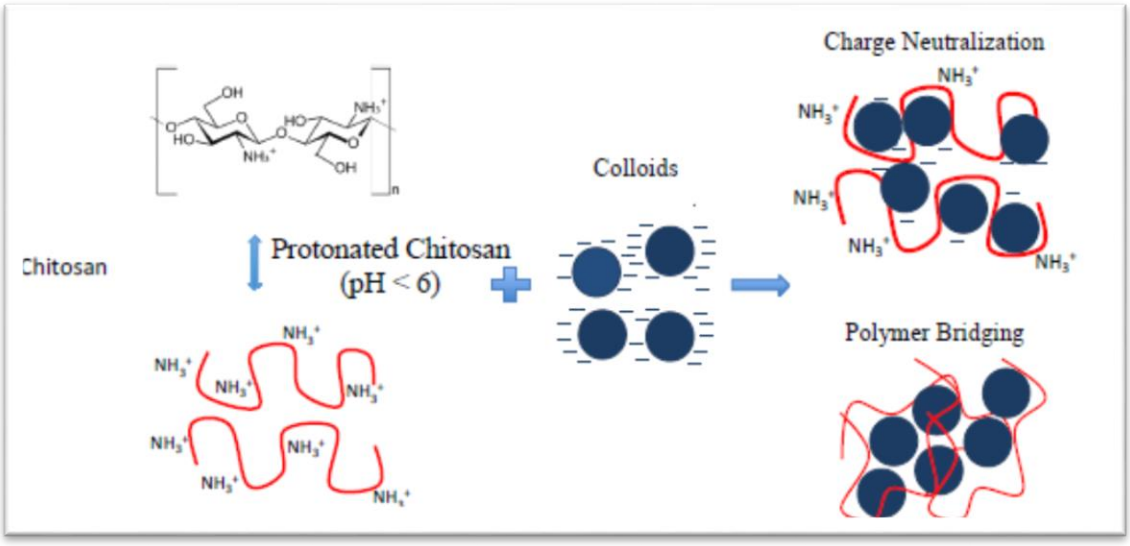

Figure I.12 *Chitosan elimination of colloids mechanism**[17].***

Another study concerns the elimination of heavy metals using chitosan done by **A.Elias [7]**who extracted chitosan from shrimp shells and used it for the retention of Cobalt, Nickel and Copper.

For testing the effect of the adsorbent concentration, several concentrations were prepared and added to a solution of 200 ppm of the metals at pH = 5.5. The elimination was increasing with the increase of the concentrations of chitosan.

Solutions of 100 ppm of chitosan were prepared with different pH values. The elimination was increasing with the decrease of pH because of the competition between protons resulting from HCl and metal ions.

As a last test, a solution of 100 ppm of chitosan was added to different concentrations of metals. The elimination was in increasing with the increase of concentration until the stabilization of the adsorption. This test informs about the capacity of adsorption of chitosan.

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MATERIALS AND METHODS

In this chapter, the materials and products used to achieve the aim of the study are presented in addition to the characterization analyses: definitions and principle of work.

Part I:The equipment

I.1. Chemical products

In our experience, we have used the following products:

-Shrimp and crab shells

-Distilled water

-Sodium hydroxide (NaOH)

-Hydrogen chloride (HCl)

-Bentonite

-Chloroform

-Acetic acid

-Ethanol

-Acetone

I.2. Glass wares

-Beakers of different capacities (10, 50, 250, 500 mL).

-Vials (25, 50, 100, 250 mL).

-Pipettes (50, 25, 5, 1 mL).

-Burettes.

-Erlenmeyer flask.

.-Heating plates.

-Magnetic stirrer.

-Spatulas.

-Petri dishes.

-Digital balance.

-Wash bottle.

Part II: Experimental Methods

II.1. Chemical Extraction of chitin and chitosan

The raw materials used in this experiment are natural 100%, which are shrimp and crab shells were collected from GHAZAOUET seaside in the west of Algeria, which were processed and extracted from chitin and chitosan in the laboratory of Abou- Baker Belkaid University, Faculty of Chemistry, using the following procedure.

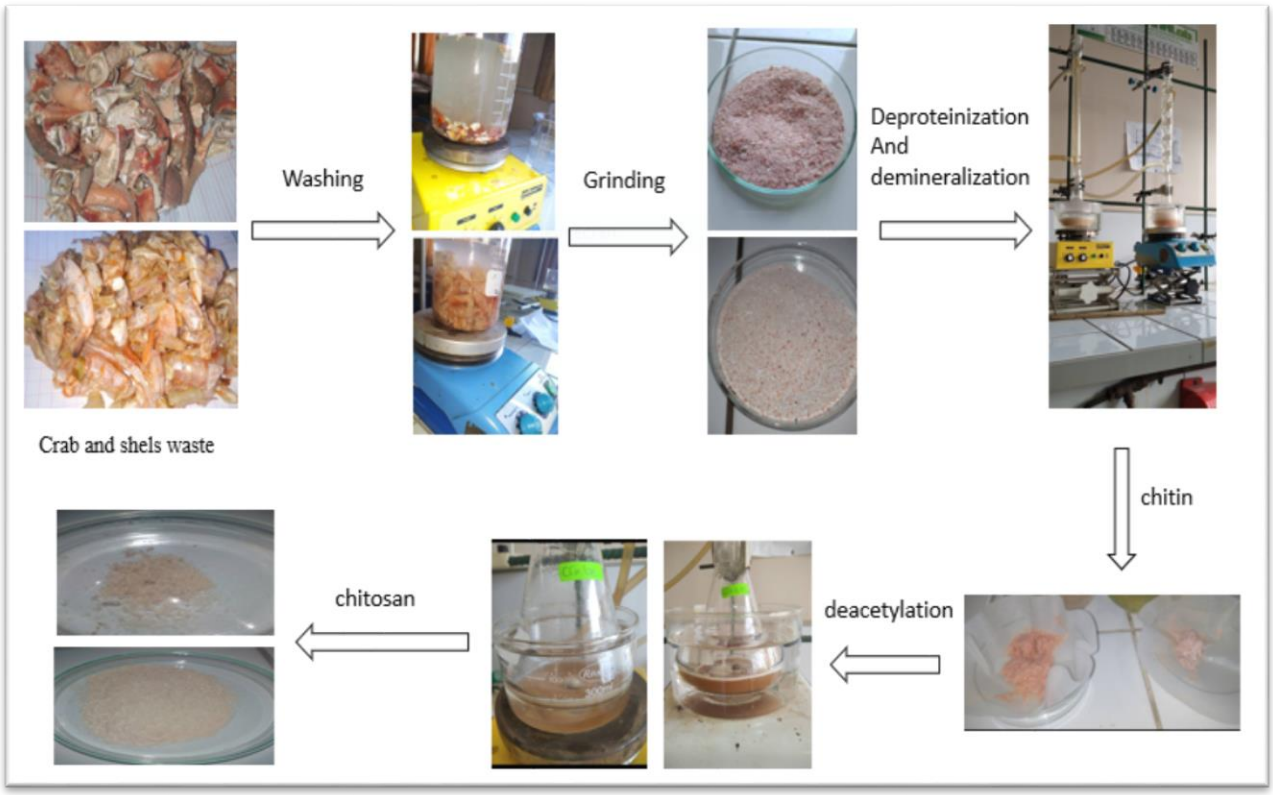

Figure II.1 *Diagram of the preparation of chitin and chitosan*

II.1.1. Purification of shells

In a 1000 mL beaker, take 40 g of shrimp and lobster, add 800 mL of distilled water and shake for one hour, then filter and check the pH (acid). After drying at 60 °C for 24 hours, in the last step, grind well into a slurry to facilitate further work.

II.1.2. Deproteinization

To remove proteins, dissolve 6 g of NaOH in a 100 ml flask of distilled water in duplicate, then in a ground flask, take 10g of crab and 10g of shrimp and add 100 ml of NaOH to each flask (ratio 1/10 w/v), reflux the mixture for 3h30min, filter (neutralize), and dry at 60°C (24h) in the oven.

II.1.3. Demineralization

This step consists in removing the calcium carbonate (CaCO3)

$$2\,HCl_{(aq)} + CaCO_{3(s)} \rightarrow CaCl_{2\,(aq)} + H_2O_{(l)} + CO_{2\,(g)} \ldots\ldots\ldots (1)$$

Prepare two solutions of 105 ml HCl (2N) (because the ratio is 1/15w/v), add 7g of crab and 7g of shrimp in a ground flask of 250 ml, bring to reflux for 2h 60°c, filter (adjust the pH until neutralization) and dry in the oven (24h).

II.1.4. Deacetylation

It resides in the removal of part of the acetyl group of chitin with sodium hydroxide NaOH.

$$R - NH - CO - CH_{3(s)} + NaOH_{(aq)} \rightarrow R - NH_{2\,(s)} + CH_3COONa_{\,(aq)} \ldots.(2)$$

Preparea solution of 60% NaOH (30g NaOH 50ml H_2O). We have 1.15g of chitin extracted from crab and 3.10g of shrimp, so in an Erlenmeyer take 1.15g of chitin extracted from crab with 11.5 ml NaOH and 3.1g of shrimp with 30.1 ml of NaOH, bring to the reflux for 3h (120°C), filter, wash (neutralization) and repeat this handling twice and before drying the products.

II.1.5. Deacetylation 2

Due to the first deacetylation, we recovered 0.70 g of crab and 2.06 g of shrimp, so prepare 50 mL of NaOH (30 g ratio 1/10), take 0.7 g and 7 mL of NaOH, 2.06 g and 20.6 mL of NaOH in an Erlenmeyer flask, Reflux for 2 hours (95°C), filter (neutralize) and dry the chitosan.

II.2. Preparation of the solution for conductimetry/potentiometry (determination)

Deacetylation 2 gave 0.45 g of crab chitosan and 1.79 g of shrimp chitosan.

Take 0.2 g of chitosan and 20 mL of HCl (0.1 M) in a 50 ml flask and adjust with distilled water, shake to dissolve our product, titrate with NaOH (0.1 M).

II.3. Preparation of the coagulant solution

In a 25 ml flask introduced 0.025g of chitosan plus 1% of acetic acid (C2H4O2) and adjust with distilled water.

II.3.1. Procedure of coagulation-flocculation on jar –test

-Fill the 500 mL beakers with 100 ppm bentonite suspension (put 0.025g of bentonite with 250 mL of distilled water for each beaker).

-Measure the initial turbidity (TU_0).

- Add different concentrations of copolymer per beaker (0.5, 1, 2 and 4).

- Set the stirring speed for coagulation at 150 rpm for 5 min, then reduce the speed to 50 rpm for 7 min for the flocculation phase.

-After the end of the flocculation, wait for the mixture to settle for 5 minutes and measure the turbidity, then 15 minutes and 30 minutes**[1]**. To adjust the pH to the desired level in each beaker with a fixed concentration, use HCl and NaOH (1M).

-Measure the initial pH and repeat the same speed mode, then measure the turbidity for the same time as the first step.

This method used for chitosan from crab, shrimp and also for commercial chitosan.

Part III: Characterization methods

III.1. Infrared spectroscopy (IR)

III.1.1. Principle

It is an analysis technique used for liquids, solids and gases.

Infrared spectroscopy allows to identify the presence of certain covalent bonds within a molecule and thus of characteristic groups. The absorption of these radiations corresponds to an internal vibration of the molecule (modifying the inter-atomic distances or the normal angles of bonds).

III.1.2. Equipment

IR equipment(FTIR CARY 600 SERIES) used for characterization and calculation of DD. The prepared chitosan samples were characterized in KBr pellets by means of a model IR spectrophotometer (4100 yasco, Japan) in the diagram from 400 to 4000 cm $^{-1}$, and they determined the DD by the following formula**[2]**:

$$DD\% = \left[1 - \left(\frac{A1665}{A3450} \times \frac{1}{1.33}\right)\right] \times 100 \quad \text{(III.1)}$$

Figure II.2 *Infrared Spectroscopy (FTIR CARY 600 SERIES).*

III.2. X-ray diffraction(XRD)

III.2.1. Principle

This method is mainly applicable to crystalline materials (solid or powder, mono-crystalline or polycrystalline) and aims to specify the structure of the material, measure the mesh parameters, the size of crystallites and the orientation statistics of crystallites. The diffraction is done according to Bragg's lawwith:

$\eta\,\lambda = 2d \sin\theta$

θ: The angle between the incident beam and the diffracting planes.

λ: The wavelength of the incident beam.

d: the reticular distance between the diffracting planes.

III.2.2. Equipment

We used a DRX device of type "RIGAKU ULTIMA-IV" with copper CBO radiation Kα (λ=1.54 Å) at 20°C under 40 kV and 30 mA.

S.Govindan and his group work with these parameters:

The structure of the prepared samples was analyzed using a Rich Siefert 3000 XRD apparatus with Cu Kα1 radiation (λ = 1.5406 Å), and the surface morphology of CS-**Ag** nanocomposites was observed using a HITACHI-SU 6600 FESEM. Group confirmation was evaluated by Shimadzu FTIR infrared affinity spectrometer**[3]**.

The diffraction patterns were recorded between 2° and 80° (2θ). Scanning speed 5°/min to characterize the crystalline material. We used this device for characterization and comparison with commercial chitosan.

Figure II.3 *X-ray diffraction apparatus.*

III.3. The pH (potentiometer)

III.3.1. Definition

A pH meter is a scientific instrument used to measure the activity of hydrogen ions in aqueous solutions, expressing their acidity or alkalinity in terms of pH. A pH meter measures the potential difference between a pH electrode and a reference electrode.

III.3.2. Equipment

In our work, we used the pH scale for the measurements as well as to know the value of DD**[4]**.

Figure II.4 *Potentiometer device OHAUS STARTER 2C (LAEPO)*

III.4. Conductimetry

III.4.1. Definition

Conductimetry is the quantitative study of the conductivity of electrolytes, i.e. solutions that conduct current (ionic solutions).

III.4.2. Principle

Two flats parallel stainless-steel electrodes of area S are inserted face to face at a distance L from each other in the electrolyte.

A sinusoidal AC voltage of rms value U (in V) is applied between these electrodes, and we note that L is the rms current (in A) of the current flowing through the solution.

III.4.3. Equipment

In our work, we used the conductivity meter for the measurements and the calculation of DD**[4].**

Figure II.5 *Conductivity meter device CDM210 (LAEPO)*

III.5. JAR- TEST experiments

III.5.1. Definition

In our coagulation-flocculation experiment, we use the jar-test apparatus, which allows the study of the efficiency of our method.

III.5.2. Principle

In the following experiments, coagulation-flocculation tests were performed on Jar-tests of the following types**[1]**:

-Prepare the solution to be treated.

-pH adjustment of the solution.

-Add the polymer solution followed by rapid agitation for a short period.

-Low speed agitation.

-Stop agitating and pay attention to the decantation kinetics.

III.5.3. Equipment

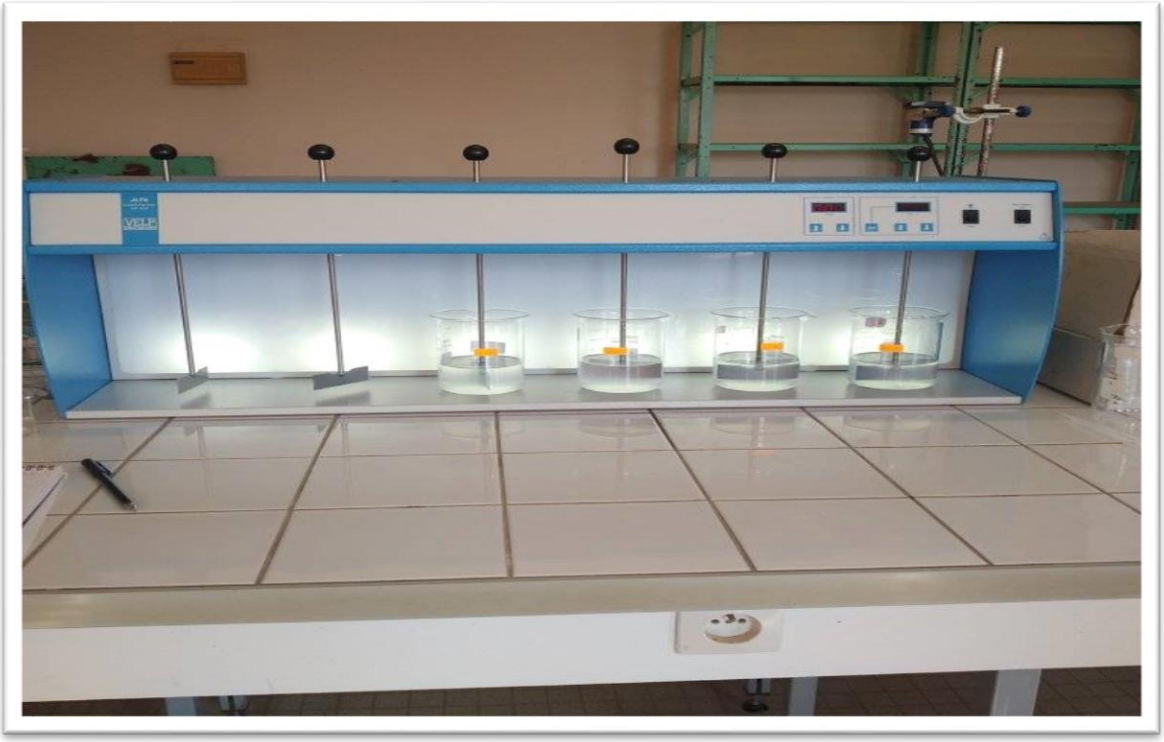

Figure II.6 *Jar-test device (LAEPO).*

III.6. Turbidimetry

III.6.1. Definition

Suspended matter in the water represents turbidity, which includes clay, silt, organic particles, plankton and other tiny organisms. The presence of suspended particles in the water is indicated by turbidity. A turbidimeter is used to determine turbidity. At an angle of 90° to the incident light beam, this device detects the light scattered by the suspended particles.

We used a HANNA HI-93703C turbidimeter to study the flocculation capacity of the different polymers used to remove turbidity from bentonite solutions.

III.6.2. Equipment

Figure II.7 *Turbidimeter Hanna (LAEPO).*

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RESULTS AND DISCUSSION

This chapter focused on the characterization of chitin and chitosan produced in this study and the use of chitosan for turbidity elimination for its efficiency in the removal of a variety of pollutants from water**[1]**.

PART I: Results of chitin and chitosan extraction

Crab and shrimp shells collected from sea waste were cleaned for 2 hours with distilled water, dried and then treated with NaOH and HCl for the extraction of chitin. The product obtained was deacetylated with concentrated solution of NaOH 60%; the process was repeated twice for having a high level of DD.

I.1. Overview of extraction

The masses of the products, used and obtained, are listed in the following table:

Table III.1 *The results of the extraction of chitin and chitosan from crab and shrimp shells*.

	Shrimp shells		**Crab shells**	
Process	The mass used (g)	The mass obtained (g)	the mass used (g)	The mass obtained (g)
Purification	40	16.72	40	38
Deproteinization	10	7	10	7.7
Demineralization	7	4.5	7	2
Deacetylation 1	3	2.06	1	0.7
Deacetylation 2	2.06	1.79	0.7	0.45

The results of obtaining chitin method from marine waste show that each step done is followed by a loss of mass which means the content of the product analyzed in material to be eliminated.

The important loss of mass was on the step of demineralization and especially for crab shells which is due to the high content of calcium carbonate on this first and the results were similar to those of the precious works. The table below presents the percentages of loss of mass in crab and shrimp for the two important steps on the production of chitin and a simple comparison with the literature**[2]**:

Table III.2 *The percentage of proteins and minerals in crab and shrimp shells used.*

	Crab		**Shrimp**	
	Experimental	Literature	Experimental	Literature
% proteins	23	16.68	30	34.02
% minerals	71.42	66.58	35.71	42.26

I.2. Test of solubility

Chitosan is a polymer with a high solubility in acid solutions in the range of pH=4; this is the main difference between this first and its acetylated form**[3]**. This solubility is limited by the degree of deacetylation: as the product has a high DD value, the more its solubility increases in acidic medium, so that the NH_2 groups will be present in a large number in a deacetylated product (with a high DD) and in the presence of an acidic medium will be easily protonated, transforming them into NH^{3+}, which are easily soluble.

The following table shows the results of solubility test carried out using different solvents for chitosan:

Table III.3 *Results of chitosan solubility in different solvents.*

	Hydrochloric acid	Water	Chloroform	Acetone	Ethanol	Acetic acid
DD1	–	–	–	–	–	+
DD2	–	–	–	–	–	+

With: – means insoluble and + means soluble.

The results show that synthesized chitosan is not soluble in all the solvents used, except with acetic acid, where a partial solubility of the product was encountered which can gives an idea about the DD(less than 50%).

PART II: Study of chitin and chitosan quality

II.1. Characterization of chitin and chitosan

The efficiency of the extraction procedure can be evaluated using several analytical methods for testing the crystallinity, the rate of deacetylation and the functional groups present in the obtained product.

II.1.1. Characterization of the obtained products with FTIR spectroscopy

The study of our products by FTIR analysis aims to determine their characteristic bands and to compare them with those of the literature. The results of the study are presented in the figures **III.1** and **III.2**:

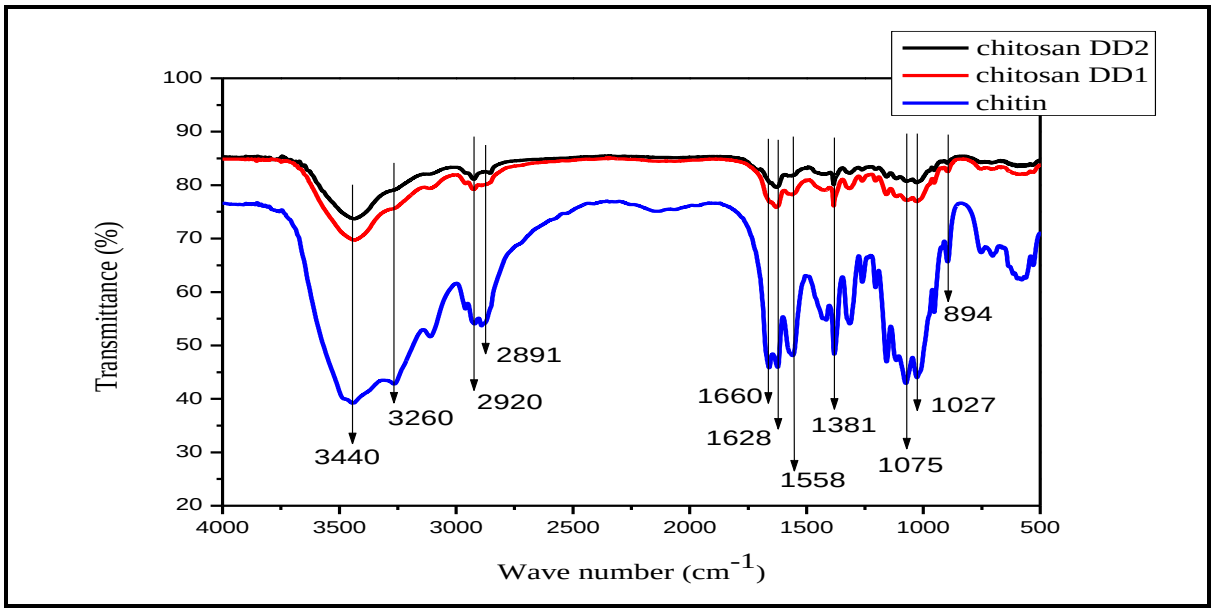

Figure III.1 *FTIR spectrum of chitin, chitosan DD1 and Chitosan DD2 extracted from shrimp shells.*

The principal functional groups of chitin and chitosan were detected with FTIR spectroscopy.

Adsorption bands on 3440 and 3260 cm^{-1} is due to stretching vibration of O-H and the N-H (amine) groups. Peaks on 2920 and 2891 cm^{-1} can be attributed to the asymmetric stretching of C-H in CH_3 and CH_2 groups.

The stretching of C=O bonds of the secondary amid results a peak at 1660 $cm^{-1}$and another at 1558 cm^{-1}.

The stretching of CH_2-CH_3 bonds in the products explains the existence of the peak on 1381 cm^{-1}.

The adsorption band on 1075 cm^{-1} is assigned to C-O bonds stretching for (C-O-C), moreover, the vibration of the glucosamine ring (-COC-) gives an adsorption peak at 1027cm^{1}.And finally the ring stretching of the $\beta(1,4)$ glycosidic bond is located on the fingerprint of the spectrum at 894 cm^{-1}.

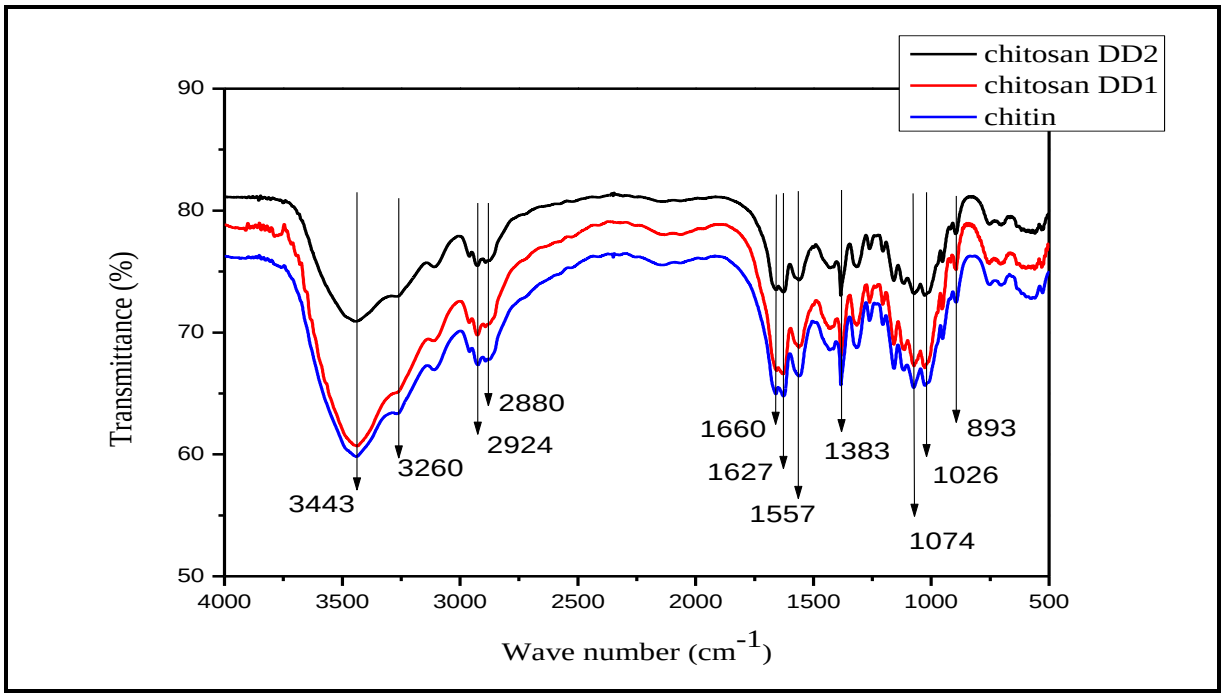

Figure III.2 *FTIR spectrum of chitin, chitosan DD1 and chitosan DD2 extracted from crab shells.*

The characteristic bands of chitosan extracted from crab are almost totally similar to those obtained for shrimp.

The difference between chitin and chitosan can be seen in the absorption bands of NH and amide groups: As the NH groups are present in chitosan more than in chitin, the intensity of the bands at 3260 cm^{-1} will be greater in chitosan DD1 than in chitin and in chitosan DD2 than in chitosan DD1. The same thing for acetyl groups (amid) which are present in chitin more than chitosan, for that the peaks on 1627 and 1557 cm^{-1} will be more visible in chitin.

The table below resumes the most interesting characteristic bands of chitosan extracted from the two sources compared with the literature**[4]**:

Table III.4 *Comparison between IR spectroscopy bonds of chitosan obtained and results of the literature.*

Functional groups	Wave number (cm^{-1})		
	Chitosan crab shells	**Chitosan shrimp shells**	**Chitosan (Literature)**
Stretching O-H Stretching N-H	3437-3260	3440-3260	3440;3442
Asymmetric Stretching C-H	2924-2880	2920-2891	2920;2921
Stretching C=O	1627	1628	1626;1628
Amid Ⅱ	1660	1660	1658;1657
Stretching C-O (C-O-C)	1074	1075	1070;1074
Stretching $\beta(1,4)$ glycosidic	893	894	897

II.1.2. X-ray diffraction (XRD) characterization

The analysis of the samples by XRD analysis gives an idea about the crystallinity of the products studied. The results of the analysis of chitin and chitosan extracted from crab and shrimp were translated into the following diffractograms:

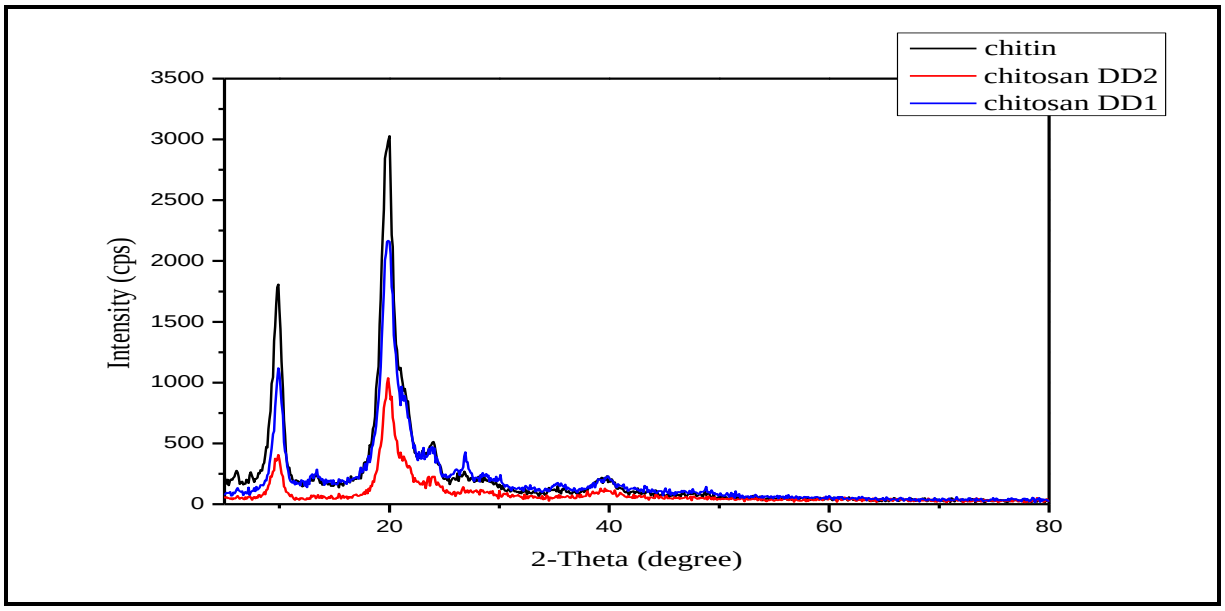

Figure III.3 *XRD patterns of chitin, chitosan DD1 and chitosan DD2 obtained from shrimp shells.*

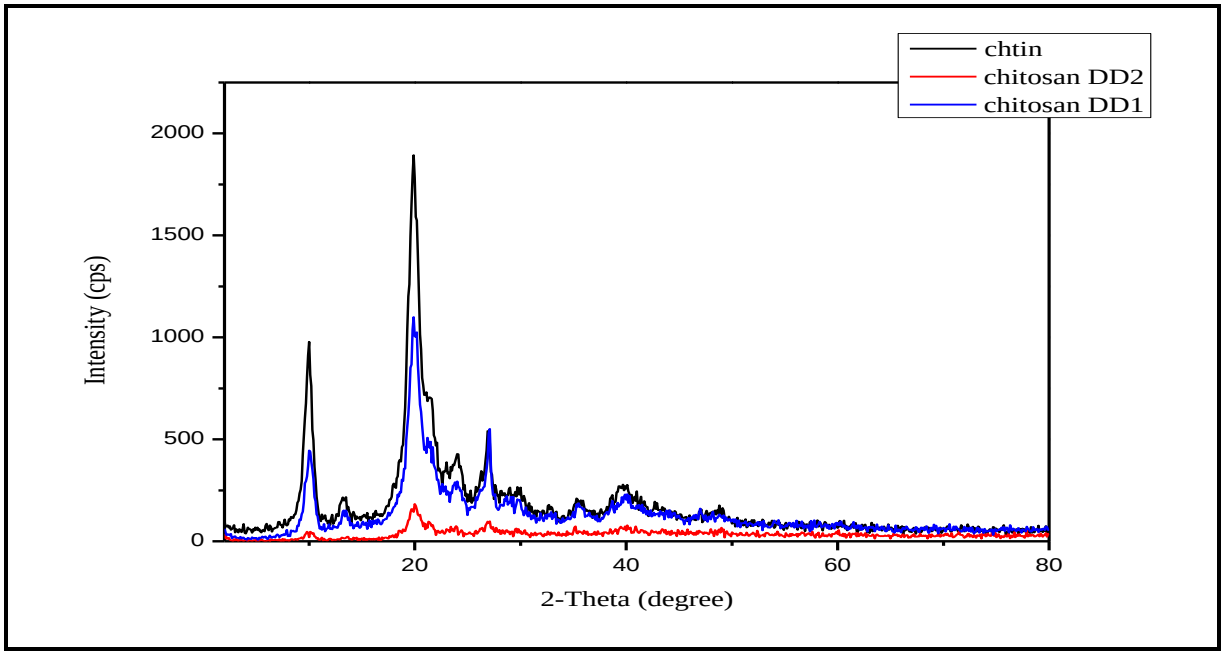

Figure III.4 *XRD patterns of chitin, chitosan DD1 and chitosan DD2 obtained from crab shells.*

The XRD patterns present five maximum peaks in the order of 10, 20, 24, 27 and 29°. A simple comparison between the three patterns gives an idea about the changes of the morphology in the structures: moving from chitin, DD1 to DD2 the intensity of peaks was decreasing which means that the disorder has become more frequent from one structure to another in other words, chitin is more crystalline than chitosan and the crystallinity percent decreases with the increase of DD, all that can be illustrated as following:

Chitin has a semi-crystalline form; its deacetylation by changing the acetyl groups with hydrogen atoms decreases the inter-chain forces in the polymer and the chains become very weakly connected to each other which causes a non-ordered structure, the formation of amorphous zones and thus a decrease of the crystallinity rate. The same results have been obtained by previous works**[4, 5]**.

II.1.3. Conductimetric titration

The conductimetric titration of the product obtained is intended to determine the conversion of chitin to chitosan (**for twice deacetylated chitosan**). The changes in the conductivity of the chitosan solutions (chitosan+ HCl) with basic dosage by NaOH make it possible to draw the following curves:

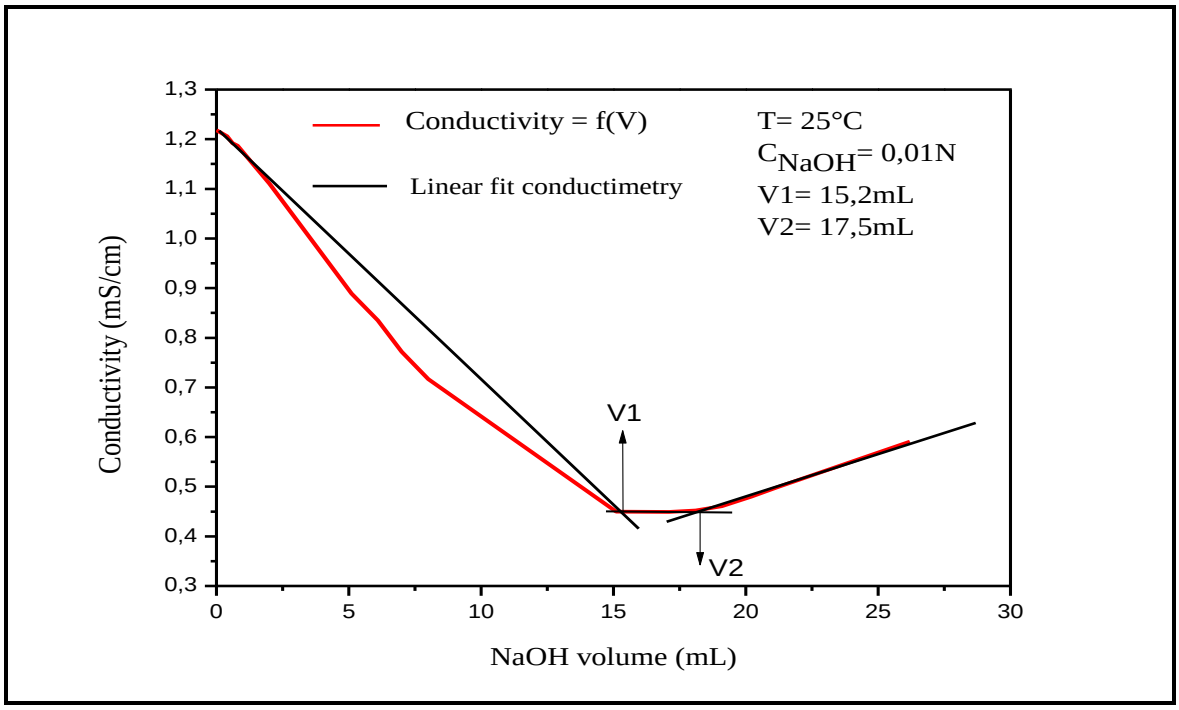

Figure III.5 *Results of conductivity changes for chitosan DD2 extracted from shrimp shells.*

The plots of conductivity versus volumes of NaOH added are divided into three parts:

The first part indicates a decrease in conductivity due to the decrease in the number of H^+ ions in the reaction medium by neutralization of the latter by OH^- ions.

The second part indicates a stabilization of the conductivity as a function of the volume of base added, which is due to the transformation of $NH3^+$ ions on NH_2.

The last part indicates a sudden increase in conductivity which is due to the total neutralization of ammonium ions and the presence of an excess of OH- ions.

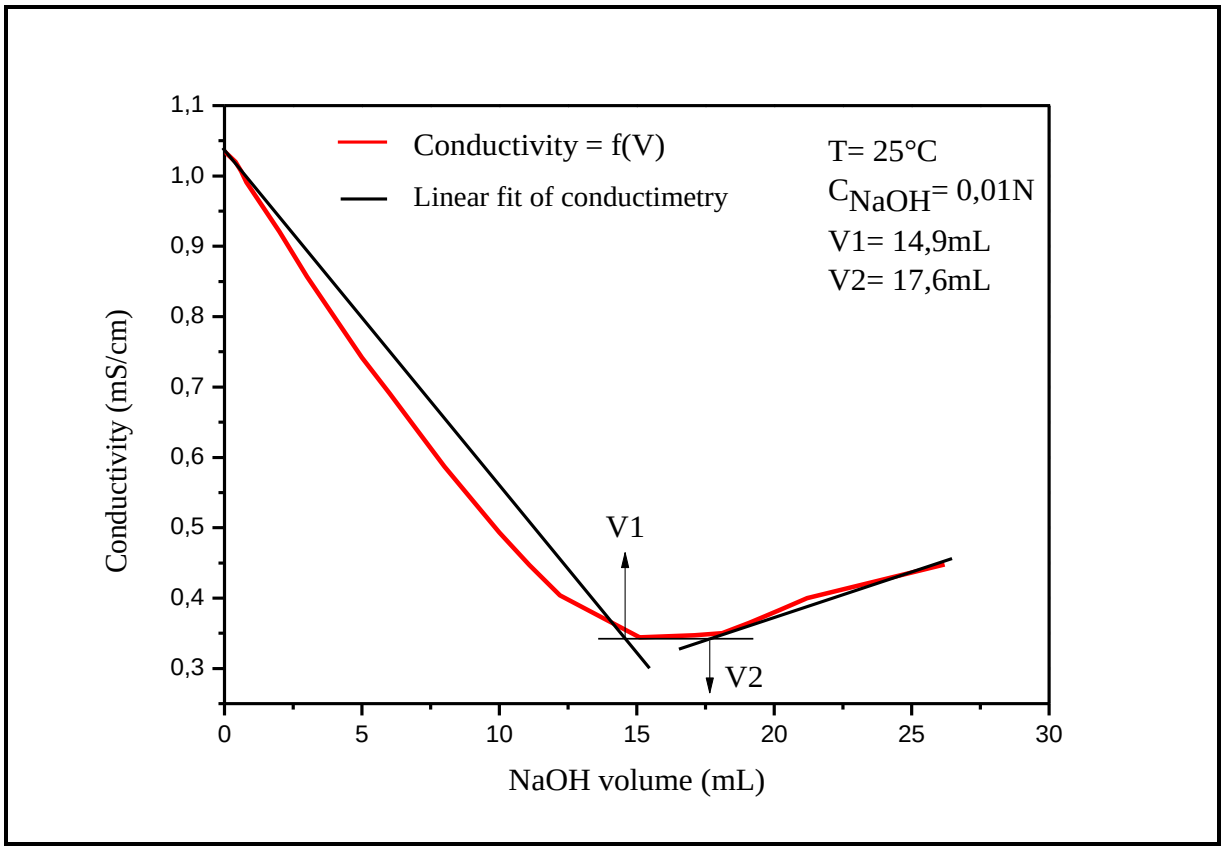

Figure III.6 *Results of conductivity changes for chitosan DD2 extracted from crab shells.*

The curves present two inflection points; the first named V1 and the second point V2:

V1: Refers to the volume of sodium hydroxide necessary to neutralize the hydronium ions coming from the hydrochloric acid which did not participate in the protonation of the NH_2 groups of chitosan.

V2: Indicates the volume of NaOH needed to protonate the NH_3^+ groups of chitosan.

The difference between the two volumes V1 and V2 represents the volume of NaOH that was needed to convert NH_3^+ ions to NH_2.

TableIII.5 *Results of the NaOH volumes obtained by conductimetric analyses.*

	Crab shells	**Shrimp shells**
V1(mL)	14.9	15.2
V2(mL)	17.6	17.5

II.1.4. Potentiometric titration

A pH-metric dosage of chitosan solutions serves to determine the equivalent volumes, of the neutralization of amino groups and H^+ ions, for the determination of the DD values. It has the same principle as conductimetric titration. The following curves present the results obtained in this study:

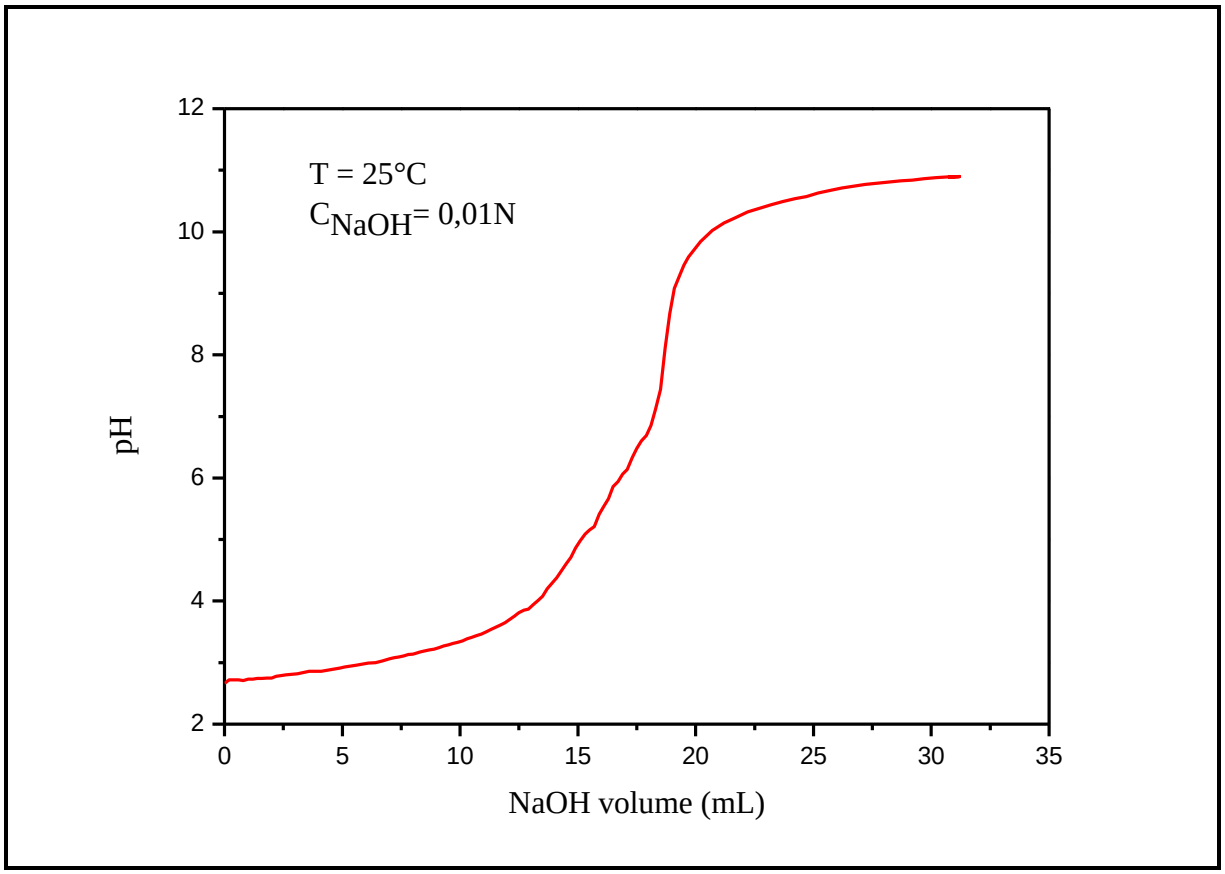

Figure III.7 *Potentiometric results for chitosan 2 titration.*

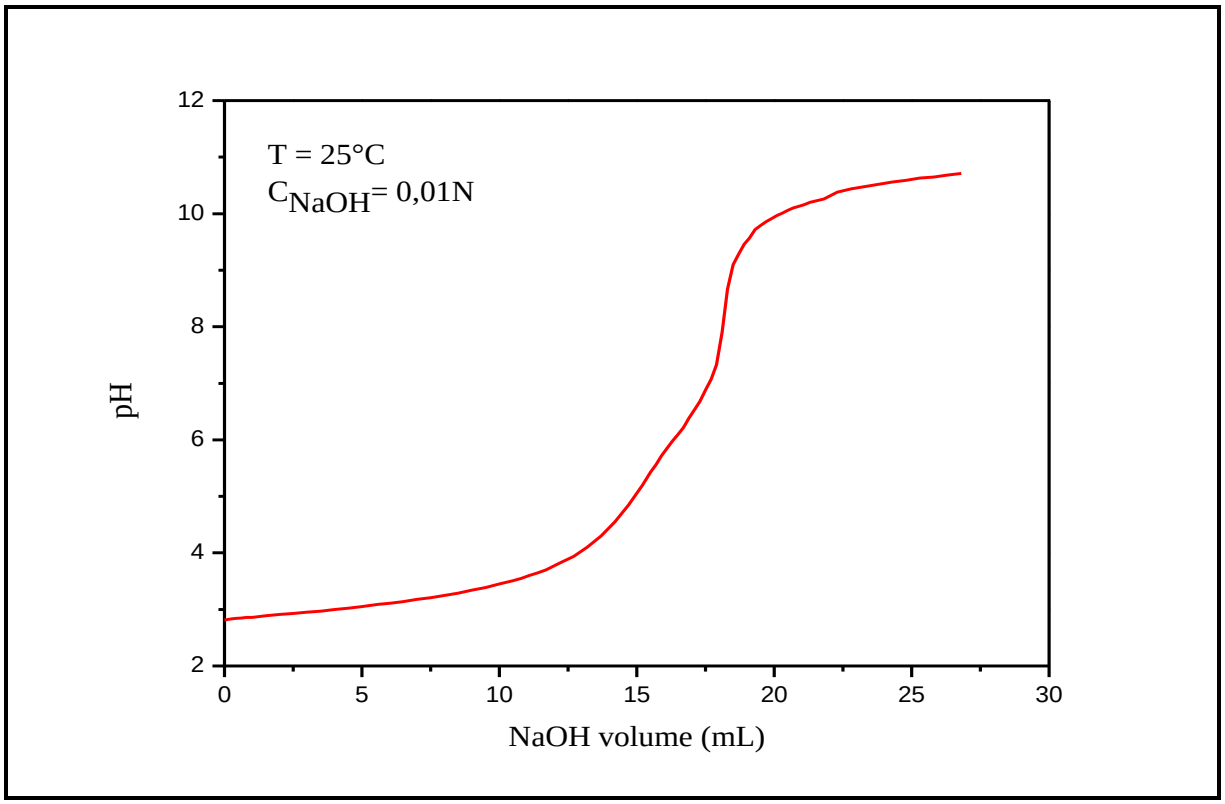

Figure III.8 *Potentiometric results for chitosan 1 titration.*

II.2. Determination of DD values

The methods of characterization used in this study serve to determine the degree of deacetylation of chitosan:

II.2.1. DD with FTIR spectroscopy:

The determination of the deacetylation degree values by infra-red analysis can be done using the following expression**[6]**:

$$\%DD = \left[1 - \left(\frac{A1665}{A3450} \times \frac{1}{1.33}\right)\right] \times 100$$

With:

A1660: The absorbance at 1665cm^{-1}, corresponds to amid Ⅱ absorbance.

A3440: The absorbance at 3440cm^{-1}, corresponds to the absorbance of NH_2 groups.

1.33: The ratio of A1660/ A3440 for chitosan that has been totally acetylated (theoretical constant).

A remark to be noted:

The results obtained previously give the values of the transmittance as a function of the wave number, for that it is necessary to use the relation between the absorbance and the transmittance to be able to use the foregoing relation:

$$A = -\log(T)$$

Using the previous formula we calculate the DD1 of chitosan extracted from shrimp (an example of calculation):

$$\%DD1 = \left[1 - \left(\frac{\log(76.39)}{\log(69.56)} \times \frac{1}{1.33}\right)\right] \times 100$$

➔$\%DD1_{shrimp}$ = 23.15%

II.2.2. Conductimetric analysis:

The conductimetric assay allowed us to find the volume necessary to protonate the deacetylated monomers, this volume will be used to calculate the DD value of each synthesized sample based on the following relation:

$$\%DD = \left[203 \times \frac{(V2-V1) \times N}{m + 42 \times (V2-V1) \times N}\right] \times 100 .$$

With:

V1: The volume of NaOH for the excess HCl neutralization.

V2: The volume of NaOH for $-NH^{3+}$ neutralization.

m: The mass of chitosan used (on mg).

N: The normality of titrant solution (mol/L).

203: The macromolecular mass of the acetylated sample.

42: The difference between the macromolecular masses of deacetylated chitosan and acetylated chitosan.

An example of calculation (DD1 of chitosan extracted from crab):

$$\%DD2 = \left[203 \times \frac{(17.6-14.9)\times 0.01}{20 + 42 \times (17.6-14.9)\times 0.01}\right] \times 100$$

➔%DD2 = 25.93%

The following table summarizes the calculation results obtained by the two methods used:

TableIII.6 *Results of DD values determined by IR spectroscopy and conductimetric analyses.*

		Shrimp shells	Crab shells
FTIR Spectroscopy.	DD1	23.15	23.03
	DD2	23.30	24.14
Conductimetric Analyses.	DD2	23.19	25.93

The results of DD values calculated with the data obtained by IR analyses of the synthesized polymers are in good agreement with those obtained by conductimetry.

PART III: Application of chitosan for the elimination of turbidity

The natural sedimentation of bentonite is a lengthy process; the rate of sedimentation during one week can only reach less than 20% for a solution of 100 ppm of bentonite**[7]**the

reason why the use of a coagulant and flocculent is necessary to activate the process. For several reasons, the use of a bio-coagulant (and flocculent) is recommended**[8]**.

In this part the results of the elimination rate of bentonite using three types of chitosan: the two products obtained in our study and a commercial one. The quantity of pollutant eliminated is calculated as following**[9]**:

$$ER\ (\%) = (TU_0 - TU_f) / TU_0$$

With:

TU_0: Turbidity of the polluted solution.

TU_f: Turbidity of solution composed from (coagulant + turbidity) after a settling time.

III.1. Study of the effect of polymers concentration on the decantation of bentonite suspensions

For studying this effect, several concentrations of polymers used (chitosan 1, chitosan 2 and commercial chitosan) are added to a 100 ppm solution of bentonite and stirred with 150 rpm for 5 minutes then 50 rpm for 7 minutes.

The initial turbidity and the final turbidity (after 5, 15 and 30 minutes of decantation times) are measured using a turbidity meter.

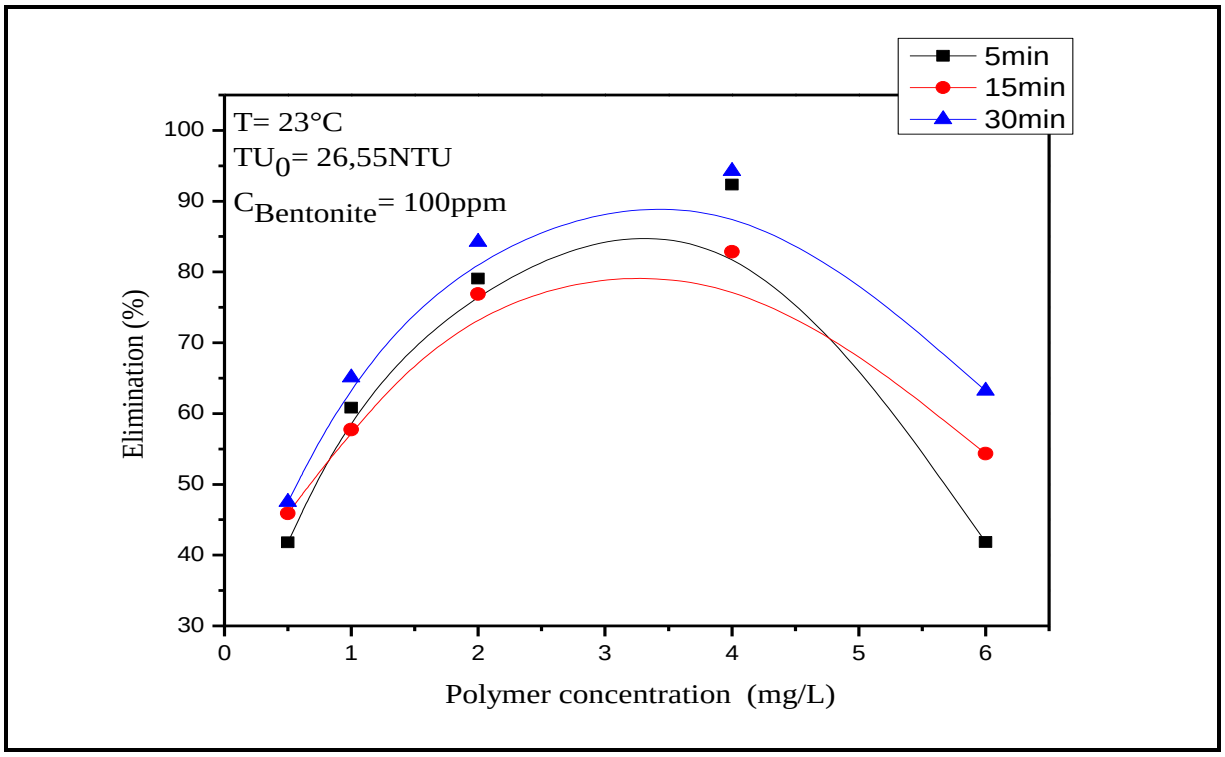

Figure III.9 *Effect of the polymer concentration (chitosan 2) ;(T= 23°C; TU_0=26.55NTU 150 rpm/5min; 50 rpm/7min).*

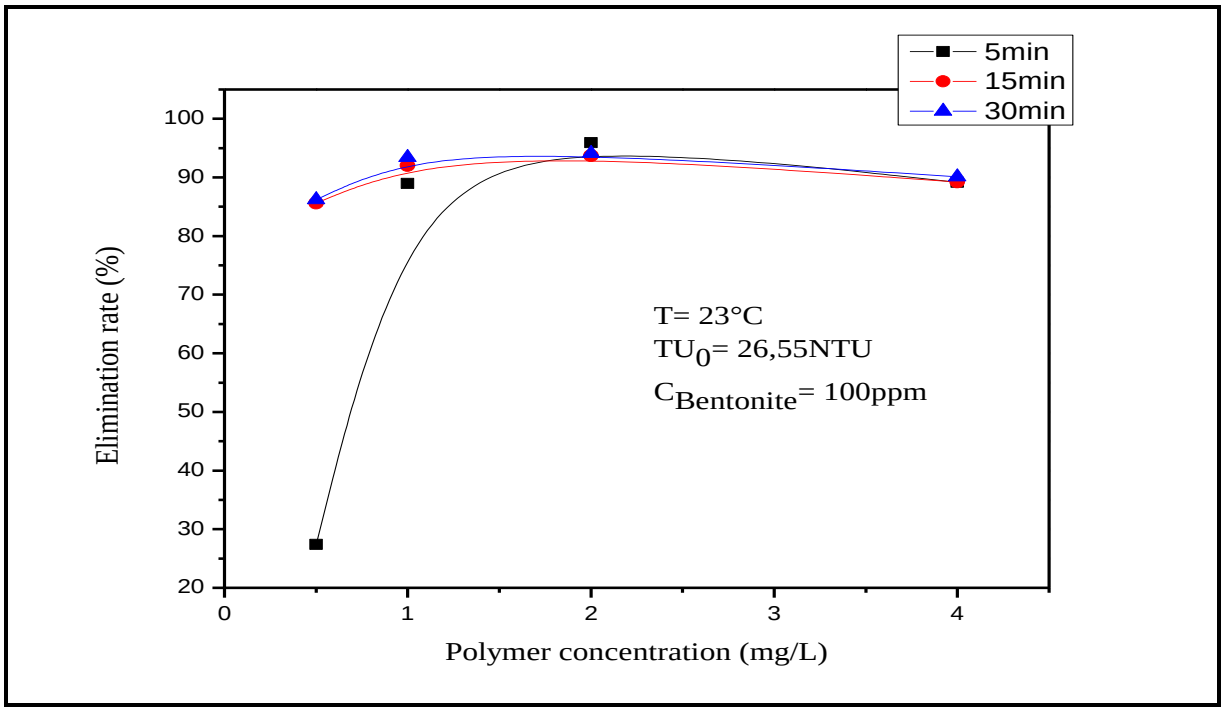

Figure III.10 *Effect of the polymer concentration (chitosan 1) ;(*T= 23°C; TU_0=26.55NTU; 150rpm/5min; 50rpm/7min).

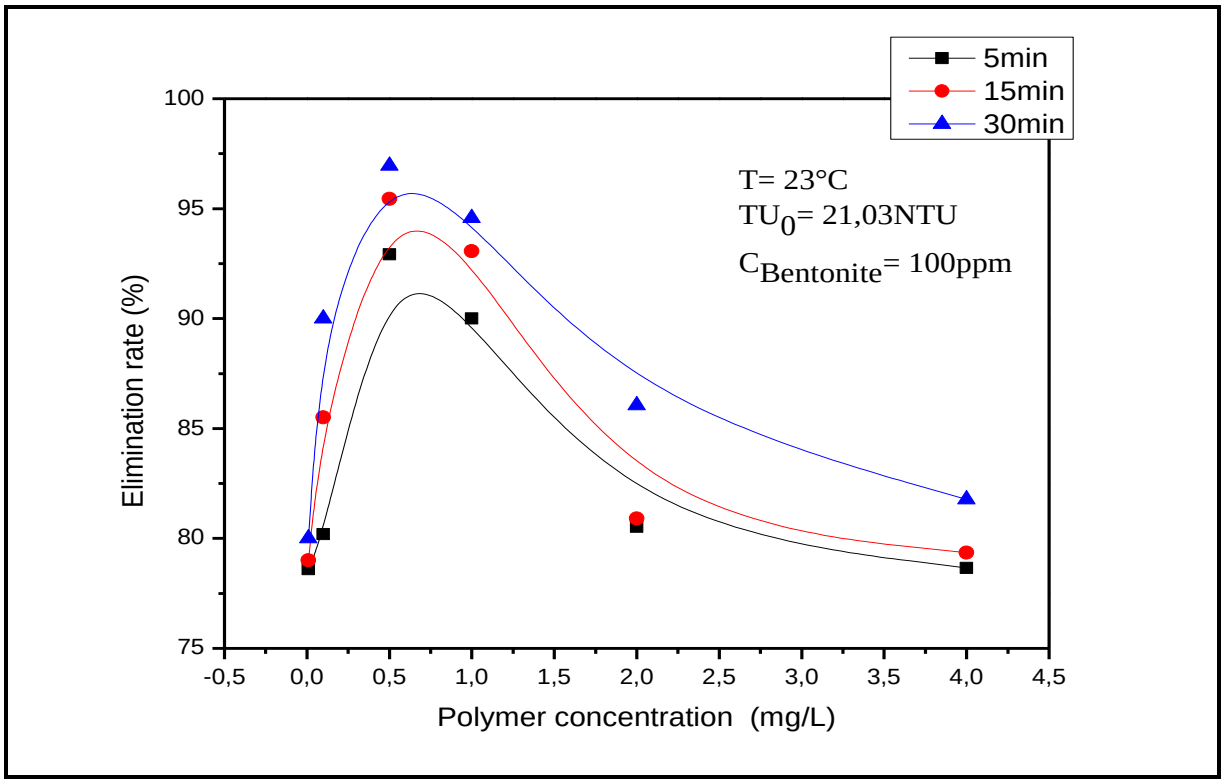

Figure III.11 *Effect of the polymer concentration (commercial chitosan) ;(*T= 23°C; TU_0=21.03NTU; 150 rpm/5min; 50 rpm/7min).

The plotted figures show the elimination rate of the turbidity as a function of the concentration of coagulant for different settling times. The first curves have the same shape (for chitosan extracted from shrimp and crab shells) while the last curve corresponding to the elimination efficiency of commercial chitosan has a special shape but all of them present a maximum point which correspond to the optimal elimination. Chitosan 1 has an elimination of 94.24% for the optimal concentration 4mg/L; chitosan 2 has 94.12% for 2mg/L while for commercial chitosan 96.9% of elimination for 0.5mg/L.

Chitosan 1 and chitosan 2 have almost equal rates of elimination but have different optimal concentrations because the two polymers have a difference in the DD values (chitosan 1 have DD up to chitosan 2), whereas for the commercial one for a DD value > 70%, the elimination was near to the two first polymers but the difference was in the optimal concentration.

III.2. Effect of the pH of the solution on the elimination of turbidity

The polymers have the capacity to be positively charged in the presence of H^+ ions, for that the effect of the pH was studied with varying the pH of the bentonite solution (pH= 1, 2,

4, 6 and 9) using the optimal concentration found in the precious part of the study. The augmentation or diminution of the pH of the medium was effected using NaOH and HCl solutions.

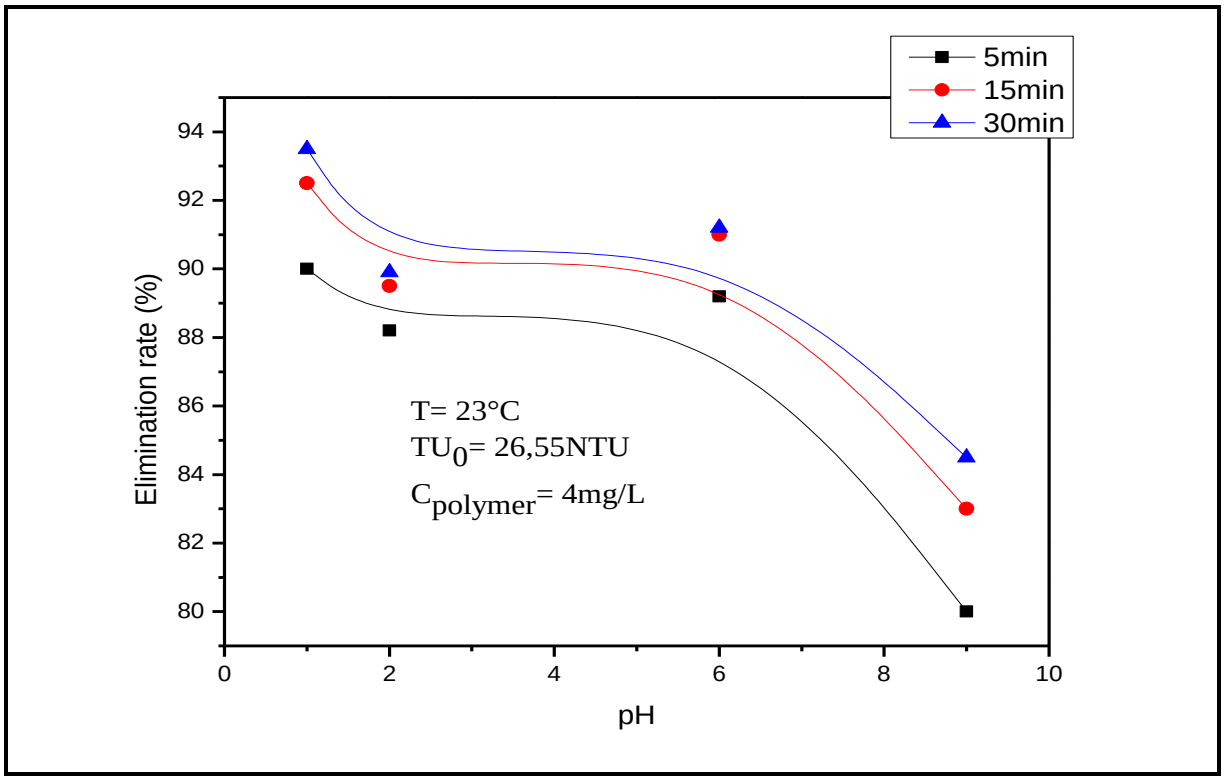

Figure III.12 *Effect of the solution pH (chitosan 2); (T= 23°C; TU_0=26.55NTU; $C_{polymer}$=4mg/L; 150 rpm/5min; 50 rpm/7min).*

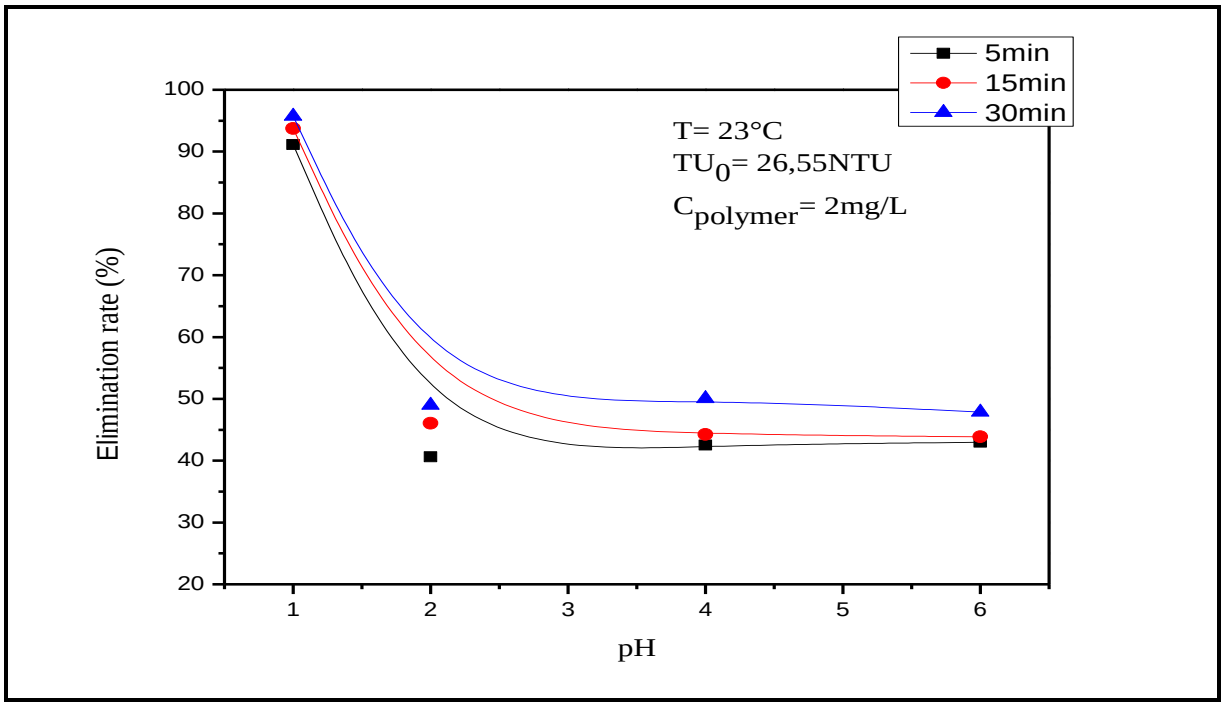

Figure III.13 *Effect of the solution pH (chitosan 1); (*T= 23°C; TU_0=26.55NTU; 150rpm/5min; $C_{polymer}$=2mg/L 50rpm/7min).

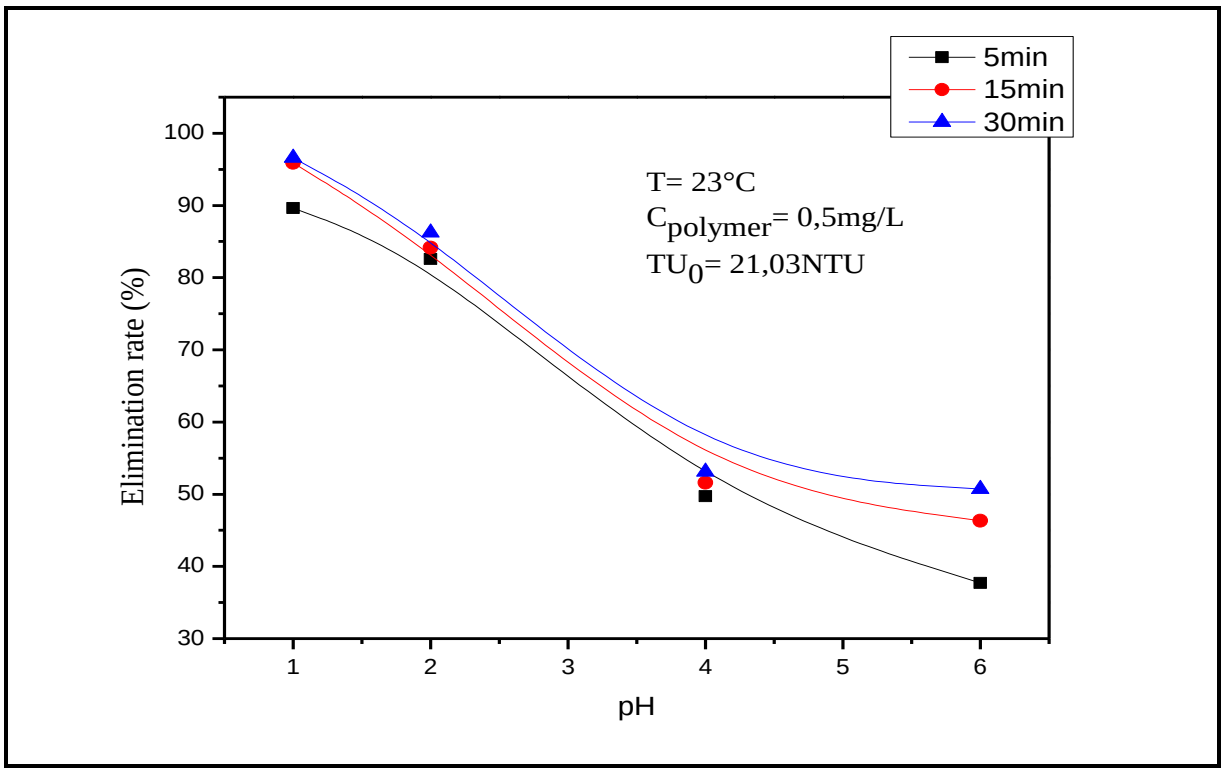

Figure III.14 *Effect of the solution pH (commercial chitosan); (*T= 23°C; TU_0=21.03NTU; 150rpm/5min; 50rpm/7min).

As presented in the figures, the elimination of bentonite suspensions decreases with the increase of the pH or with the increase of H^+ ions in the medium.

A maximal elimination was seen in the lowest pHs for the three polymers used: chitosan 2 eliminated 93% of suspensions at pH= 1 while an elimination of 85% was in pH= 6 which include an enhancing of the elimination. The same results for the other polymers, 95% of decantation with from 45 to 50% of amelioration on the efficiency of elimination.

The protonation of amino groups of chitosan decreases its superficial positive charges and facilitates the fixation and the destabilization of pollutant suspensions the reason why the diminution of turbidity of the medium was in increase in low pH values.

The results obtained in the study of these two factors in this part are reliable to the efficiency of the extraction of chitosan and the quality of the products. The high degrees of deacetylation involve the high capacity to fixe bentonite particles in acidic medium and facilitate the elimination of turbidity.

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CONCLUSION

The main objective of this work was to extract chitin and chitosan in order to use this polymer as coagulant and flocculent in a Jar-Test coagulation-flocculation technique to remove bentonite from water, and this work was divided into three parts.

-The first step: was devoted to the synthesis of chitin and chitosan, after the preparation we find a loss of mass which means that the content of the analyzed product in matter to be eliminated (the important loss located at the level of the stage of demineralization especially for the carapaces of crab 71.42%.also chitosan has a high solubility in acidic solutions in the pH=4 range, and the more the product has a DD value the more the solubility increases (the results show that our product soluble with acetic acid)

-The second step: Characterization of chitin and chitosan by FTIR and XDR, so the results show that the main functional groups of chitin and chitosan detect by IR and we find DD1 (for shrimp= 23.15, crab=23.03) and a DD2 (for shrimp=23.30, crab=24.14) and XDR gives an idea about the crystallinity of our products and that is super imposable that the commercial products.

Also, we use conductimetry titration and potentiometry to calculate the DD and we find that the DD results calculated by IR analysis are in good agreement with those of conductimetry.

-The third step: To test the effectiveness of chitosan in the treatment of water by the technique of coagulation - flocculation for the removal of bentonite. Based on the experimental results we have come to the following conclusions:

- Effect of the concentration:

Chitosan 1 makes an elimination of 94.24% for an optimal concentration of 4 mg/l, also chitosan 2 has an elimination of 96.9% for a concentration of 2 mg/l, on the other hand commercial chitosan eliminate 96.9% for a weak concentration which equals 0.5 mg/l it is because of their DD which is greater than 70%

- Effect of pH:

The elimination of the bentonite suspension decreases with the increase of pH, also the chitosan 2 eliminated 93% of suspension at pH =1, (same results for the other polymers).

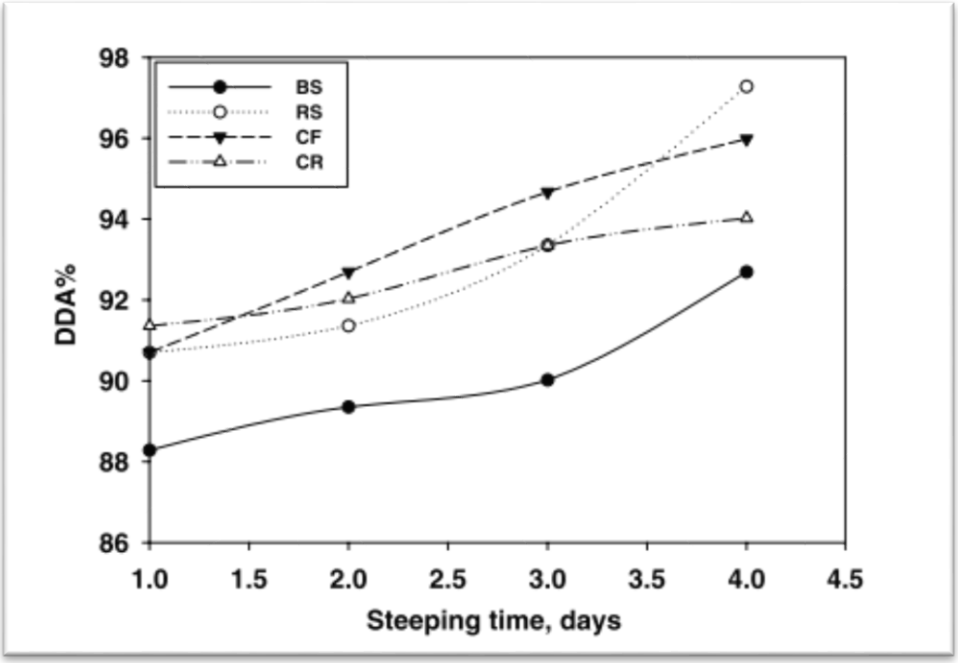
All the results obtained in this study reliable for the efficiency of the extraction of chitosan and the quality of product synthesized in our laboratory.

Perspectives

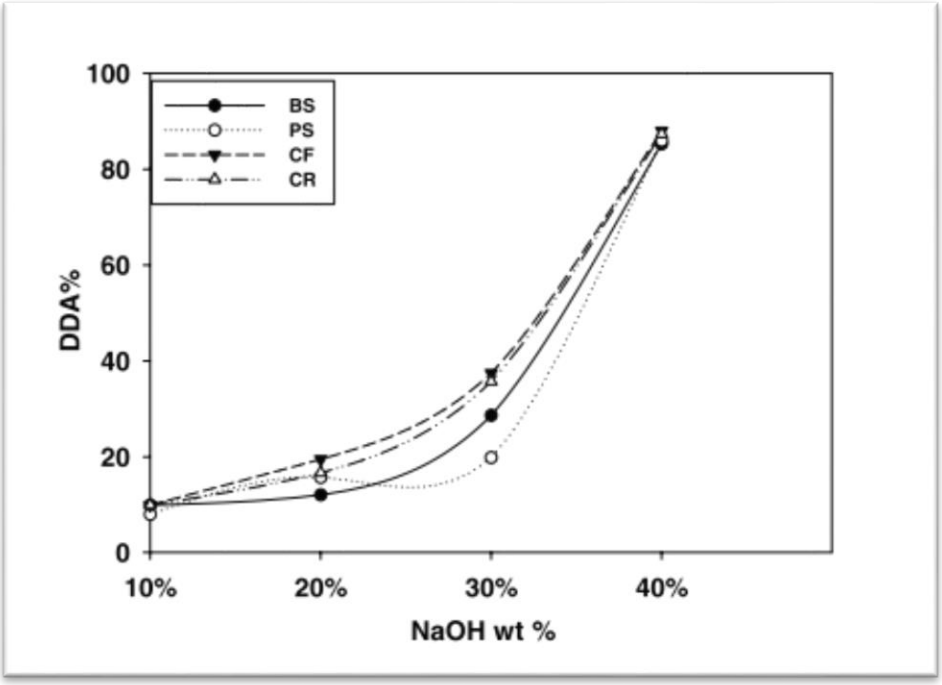
Other studies could be carried out in our study such as viscosymetric analyses for the determination of the macromolecular weight of the obtained product, RNM characterization for the determination of the degree of deacetylation and the study of the influence of: temperature, concentration of NaOH and HCl, time of reaction and the pH on the DD values.

ANNEXES

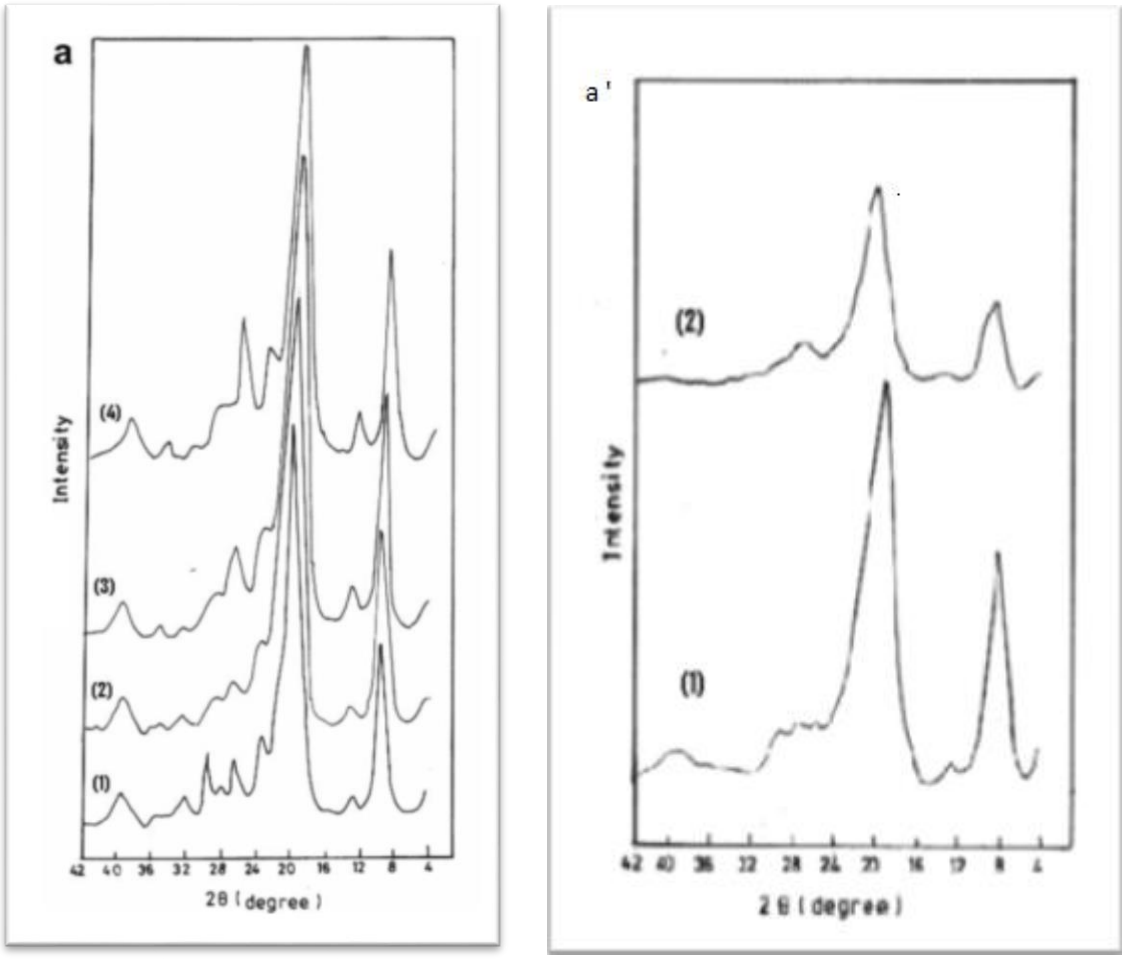
Annexes



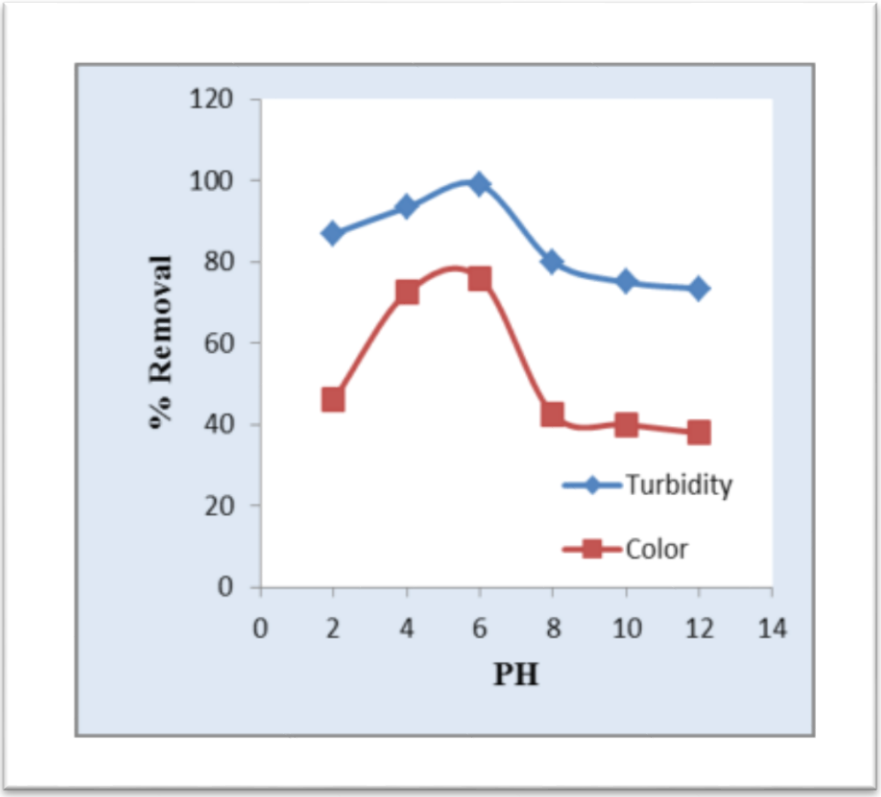
Annex 1: Effect of steeping time in 40% NaOH upon the DD% after heating in an autoclave for 1h, (BS: Brown shrimp, RS: Pink shrimp, CF: Crayfish shells, CR: Crabs shells).



Annex 2: Effect of NaOH concentration of the steeping bath on the DDA% after heating for 1 h in the autoclave, (BS: Brown shrimp, RS: Pink shrimp, CF: Crayfish shells, CR: Crabs shells).



Annex 3: (a): X-ray diffraction patterns of (a) α-chitin from (1) BS (2) PS (3) CR (4) Cray Fish, (a'): X-ray diffraction patterns of (a) b-chitin from (1) CT (2) SQ (Squid pens).



Annex 4: pH effect on turbidity and color removals. Dye 5 mg/l (Acid blue elimination with chitosan)

ملخص

مع زيادة الكثافة السكانية و كثرة النفايات جذبت مشكلة التلوث البيئي بجميع أنواعه انتباه العديد من الباحثين لإيجاد حلول لهذه المشكلة على وجه الخصوص باستخدام مواد و وسائل أقل ضررا بالبيئة و لهذا السبب، أصبح استخدام و إعادة تدوير النفايات ضرورة وخاصة ذات الأصل البحري والطبيعي.

تم عرض العمل المنجز على استخراج الكيتين و الكيتوزان من قشور الجمبري و سرطان البحر و استخدامهما في معالجة المياه ثم إجراء معايرة المواد التي تم الحصول عليها باستخدام التحليل الطيفي للأشعة تحت الحمراء (FTIR) وحيد الأشعة السينية (XDR) وأظهرت النتائج التي تم الحصول عليها أن نسبة DD أقل من 50%لكنها أثبتت فعاليتها في استخلاص البنتونيت من المياه الملوثة حيث بلغ معدل الإزالةأكثر من90% في الوسط العادي و في الوسط الحمضي.

الكلمات المفتاحية: كيتين, كيتوزان, بنتونيت, معالجة المياه, العكارة.

Abstract

With the increase in population density and the large number of wastes, the problem of environmental pollution of all kinds has attracted the attention of many researchers to find solutions for this problem in particular by using less harmful materials to the environment. For this reason, the use and recycling of waste, especially of marine and natural origin, has become a necessity.

In this research paper, the work done on the extraction of chitin and chitosan from shrimp and crab shells and their use in water treatment are presented. The characterization of the materials obtained was carried out using infrared spectroscopy (FTIR), X-ray diffraction (XRD). The results obtained showed that the chitosan obtained had a DD rate of less than 50%, but it proved its effectiveness in the extraction of turbidity (bentonite) from polluted water in which the removal rate reached more than 90% in normal and in acidic medium.

Key words: Chitin, chitosan, bentonite, water treatment, turbidity.

Résumé

Avec l'augmentation de la densité de population et le grand nombre de déchets, le problème de la pollution environnementale de toutes sortes a attiré l'attention de nombreux chercheurs pour y trouver des solutions pour ce problème notamment en utilisant des matériaux et des moyens moins nocifs pour l'environnement. Pour cette raison, l'utilisation et le recyclage des déchets, en particulier d'origine marine et naturelle, sont devenus une nécessité.

Dans ce document de recherche, les travaux effectués sur l'extraction de la chitine et du chitosane des carapaces de crevettes et de crabes et leur utilisation dans le traitement de l'eau sont présentés. La caractérisation des matériaux obtenus a été effectuée à l'aide de la spectroscopie infrarouge (FTIR), diffraction des rayons X (DRX). Les résultats obtenus ont montré que le chitosane obtenu avait un taux de DD inférieur à 50%, mais il a prouvé son efficacité dans l'extraction de la turbidité (bentonite) de l'eau polluée dans laquelle le taux d'élimination atteigne plus de 90% en milieu neutre et en milieu acide.

Mots clés: Chitine, chitosane, bentonite, traitement des eaux, turbidité.