

Comparative analysis of extracted chitosan versus commercial one: physicochemical effects of extraction

Aicha DEHANE^{*1,2}, Lisa KLAAL¹, Dalila HAMMICHE¹, Balbir Singh KAITH²

¹Université de Bejaia, Faculté de Technologie, Laboratoire des Matériaux Polymères Avancés Route De Targa- Ouzemmour, Bejaia 0600, Algérie

²Department of Chemistry, Dr. B.R. Ambedkar National Institute of Technology, Jalandhar 144 027, Punjab- India

Corresponding author*: aicha.dehane@univ-bejaia.dz

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Abstract

This study successfully extracted chitosan from waste generated by local shrimp shells. Functional and physicochemical properties of Chitosan produced have been evaluated by several methods such as FTIR, XRD and DSC. The obtained chitosan showed similar physicochemical properties compared to commercial one prepared. Structure was confirmed by Fourier transform infrared spectroscopy; X-ray diffraction and differential scanning calorimeter have same differences to refer to the effect of methods of extractions and type of shrimp shells waste.

Keywords: Chitosan, Extraction, shrimp shells, Commercial chitosan.

I. Introduction

Chitosan, a linear polysaccharide generated from the partial deacetylation of chitin, has sparked widespread attention due to its many uses in biomedicine, pharmacology, and agriculture. Its distinct features, including as biocompatibility, biodegradability, and antibacterial activity, have made it an important material in a variety of applications. Chitin, the precursor to chitosan, is one of the most prevalent biopolymers on Earth, essentially extracted from crustacean exoskeletons, insect cuticles, and fungal cell walls [1]. Shrimp shells represent a significant source of chitin, largely because of their widespread use as a by-product of the seafood industry [2].

Chitosan utilized in industrial applications is predominantly sourced from crustaceans, particularly the shells of crabs, prawns, and shrimp, which are readily available as byproducts of the food processing industry. However, it is increasingly available as a side-stream.

A product derived from the breeding of cocoons within the silk industry, serving as a by-product of protein extraction from insects for food and animal feed industries, as well as fungal fermentation. Fish scales, composed of chitin, are frequently discarded due to their low yield, representing only 1 wt% of the total weight [3].

Chitosan is commonly extracted from shrimp shells using three steps: demineralization, deproteination, and deacetylation. In one research, chitosan was extracted from *Penaeus monodon* shells using an optimized method that included 1 N HCl for demineralization at 50 °C, NaOH for deproteination, and an alkaline process for deacetylation. The ultimate chitosan production was 82%, with outstanding physicochemical qualities [4]. The extraction of chitosan is conducted through a stepwise procedure that incorporates both acid and alkaline treatments, producing high purity and

yield [5]. The selection of reagents, temperatures, and reaction durations during extraction markedly affects the characteristics of the resulting chitosan. The application of concentrated NaOH at elevated temperatures increases the extent of deacetylation; however, it may also lead to a reduction in the molecular weight of chitosan [1]. Furthermore, the application of H₂O₂ for bleaching has demonstrated an enhancement in the whiteness and purity of chitosan, thereby rendering it more suitable for uses that necessitate elevated optical clarity, particularly within the pharmaceutical domain [6]. This research investigation outlines an enhanced protocol for the extraction of chitosan from shrimp shell waste, detailing each step and comparing the results with commercial methods. The objective is to demonstrate the effectiveness and reproducibility of this protocol in producing high-quality chitosan, while also suggesting its potential as a sustainable material for industrial applications.

II. Material and methods

II.1 Used biomass origin

The used shrimp shell waste for the extraction of chitosan was collected from Mers El phare port on the Mediterranean Sea, (Algeria). Commercial chitosan from shrimp shells 75% deacetylation Min 75% Ash 1.0% moisture 10.0% viscosity (1% solution) Min 200cpc from Loba Chemie Ltd 107.

II.2 Extraction of chitosan

The extraction procedure that is being used is a laboratory modification of the industrial procedure [7,8].

a) Demineralization

Shrimp shells were gathered, cleaned, and dehydrated. The dried shells were subsequently pulverized into a fine powder. Fifty grams of the resultant powder were hydrated in a 1 N

HCl aqueous solution, with continuous stirring for six hours at 50 °C. The liquid phase was subsequently removed, and the solid material was washed with distilled water until a neutral pH was achieved. The demineralized material was dried overnight at 50 °C and subsequently utilized in the next step without additional purification.

b) Deproteination

The dried, demineralized material was immersed in an 8% NaOH solution and continuously stirred at 80-100 °C for a duration of 6 hours. The solution underwent filtration, followed by multiple washes of the solid phase with distilled water until a neutral pH was attained. The deproteinized material was then dried overnight in an oven at 45 °C.

c) Bleaching

The dried, deproteinized material underwent bleaching by immersion in a 0.3% H₂O₂ solution with continuous stirring for a duration of 2 hours. The bleached material underwent filtration, followed by washing with distilled water, and was subsequently dried in an oven overnight.

d) Additional HCl Treatment

The bleached powder was subsequently incorporated into a 12.5 M HCl solution and stirred for 2 hours at a temperature range of 80-100 °C. Following treatment, the material underwent several washes with distilled water until a neutral pH was attained. The final product was dried overnight in an oven at 45 °C.

II.3 Fourier transform infrared spectroscopy (FTIR)

Infrared spectra of powders were obtained over frequency range of 400–4000 cm⁻¹ using a FTIR Spectra (Bruker alpha-P) (with KBr).

II.4 Thermal analysis ATG/DTG

Thermal analysis was carried out by a differential scanning calorimeter (Netzsch DSC-214 polyma) where 10 mg of the samples was put in the crucible with a heating.

III. Results and discussion

III.1 Fourier transforms infrared spectroscopy (FTIR)

Spectra resulting from FTIR analysis of chitosan samples are shown in Figure 1 in comparison with the commercial one; all the characteristic peaks of chitosan were detected in all samples at their specific wavelengths: 3490, 2971, 2910, 1643, 1552, 1421, 1022, 893 and 752 cm⁻¹. Several bands have been proposed as internal reference bands of chitosan: A peak at 3480 cm⁻¹ corresponds to the -OH stretching vibrations of water and hydroxyls, as well as the NH₂ stretching vibrations of free amino groups; Symmetric CH₃ stretching and asymmetric CH₂ stretching bands within 2971-2910 cm⁻¹; The amide I and amide II bands absorption bands in the chitosan spectrum were around 1643 and 1552 cm⁻¹, respectively. The ethylene group's scissoring band was also seen at 1421 cm⁻¹. The absorption bands around 1022 and 893 cm⁻¹ correspond to vibration C-O-C bonds, confirming monomer bonding via -glycosidic links. This result is in agreement with those found by different authors [9-14]. Certain variations in peak wavelengths are most likely caused by variations in natural sources and the extraction technique. The presence of a band stretching pattern in the extracted

chitosan that corresponds to the band stretching of commercial standard chitosan indicates that the extracted material is indeed chitosan.

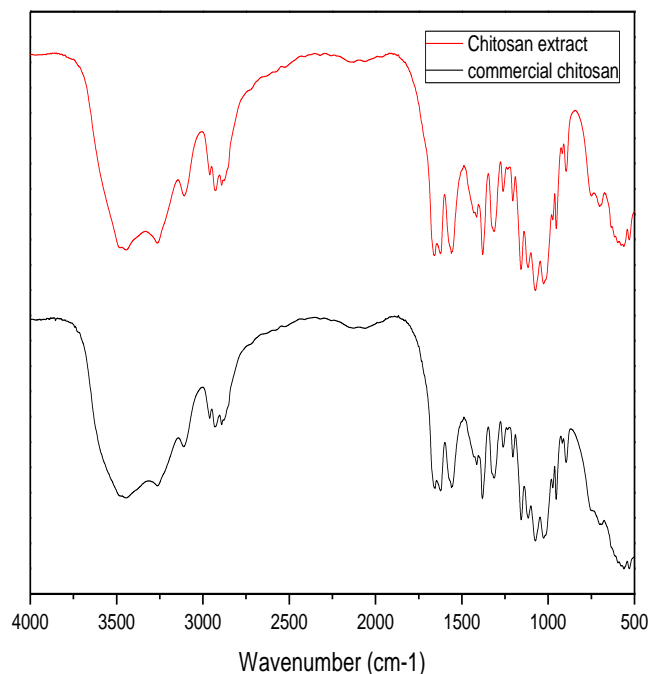


Figure 1: FTIR spectrum of the extracted chitosan and commercial chitosan

III.2 Thermal analysis ATG/DTG

The TGA curves of the Chitosan samples were plotted at two main stages of decomposition. The first was at 100 °C with a mass loss of 6.2% for Chitosan extracted and 12.5% for commercial Chitosan. Such loss is due to the evaporation of water molecules [15]. The second was variable, at 320 °C with a loss of 48.2% for Chitosan extracted, a loss of 49.2% for commercial Chitosan. This loss was caused by the depolymerization of the Chitosan chains [16]. Additionally, the details of the TGA are shown in the table below (Table 1)

Table 1: TGA Analysis results of extracted Chitosan and commercial Chitosan

TGA analysis	Extracted chitosan			Commercial chitosan		
	Temperature (°C)	100	320	650	100	320
Weight loss (%)	6.2	48.2	93.4	12.5	49.2	85.8

IV. Conclusions

In this study, chitosan was extracted from shrimp shell waste. The extracted chitosan was characterized. The results showed that the extracted chitosan had similar trends in the FTIR spectrum and TGA/DTG behavior compared to commercial chitosan indicating that extracted chitosan has comparable properties to commercial chitosan.

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