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The Degree of Deacetylation Chitosan Various Marine Shells

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Abstract

Chitosan is a natural product derived from biopolymers such as chitin and cellulose and is found in the exoskeleton of marine sources. Chitosan was produced through 3 subsequent processes i.e. deproteinization, demineralization and deacetylation. The aim of this study is to determine degree of deacetylation chitosan. The degree of deacetylation was calculated from IR spectrum using the Fourier Transform Infrared spectroscopy (FTIR). The degree of deacetylation of chitosan from *Penaeus monodon*, *Scylla* sp, and *Mytilus viridis* shells, were 89.05%, 87.64%, and 77.80%. Marine shells used as raw material of chitosan and enhancement degree of deacetylation potential to be used as natural coagulant.

Keywords: *Penaeus monodon*, *Scylla* sp, *Mytilus viridis*, Chitosan, Degree of deacetylation.

Introduction

Chitosan, a natural linear biopolyaminosaccharide, is obtained by deacetylation of chitin, which is the principal component of protective cuticles of crustaceans such as crabs, shrimps, lobsters, and shellfish and of some fungi such as aspergillums and mucor. Chitin can be extracted from marine sources. The chitin found most in crab as 50%-60%, shrimps 42%-57%, squid 40%, and shellfish 14%-35% [1]. Chitin is the most abundant natural

amino polysaccharide and is estimated to be produced annually almost as much as cellulose. It has become of great interest not only as an underutilized resource, but also as a new functional material of high potential in various fields, and recent progress in chitin chemistry is quite noteworthy.

Chitosan which high purity expressed by the degree of deacetylation. Preparation of chitosan through several subsequent each stage affects the degree of deacetylation of the chitosan. Moreover raw materials also affect the quality of chitosan. Therefore, to compare the characteristics of chitosan based materials, then the research are done by making chitosan from shrimp shell (*Penaeus monodon*), crab shell (*Scylla sp*), and green mussels shell (*Mytilus viridis linneaus*).

Methodology

Materials

Shell materials were obtained from the wastes of *Penaeus monodon*, *Scylla sp*, and *Mytilus viridis linneaus* shells, were collected from a Indonesian waters, 80 mesh crushed shells each one as much as 100 gr.

Preparation of chitosan

Chitosan extracted by adopt Knorr method [2] with some modifications. Deproteinization step was carried out with 3,3% NaOH 1:10 (w/v) at 65°C for 2 h, washed with deionized water to neutral pH and dried at 60°C for 24 h. Demineralization with 1 N HCl 1:15 (w/v) at 50°C for 30 s, washed with deionized water to neutral pH and dried at 60°C for 24 h. Deacetylation process with chitosan was prepared by alkali treatment of chitin using 60% NaOH 1:20 (w/v) in distilled water at 120°C for 1 h. The reactants were filtered, washed with deionized water to neutral pH and dried at 80°C for 24 h.

Degree or Deacetylation

The degree of deacetylation (DD) of chitosan was determined by a form IR spectrum using the Fourier transform infrared spectroscopy. Chitosan samples prepared in the forms of potassium bromide (KBr) disk was prepared according to the method of Sabnis and Block [3]. Approximately 1 mg of chitosan powder and KBr 1% w/w was mixture then pressed to form pellets. KBr pellets were obtained footage and put into place the infrared absorption spectra recorded the absorbance at 4000-650 cm⁻¹. The DD of the chitosan samples was calculated using standard method.

The hydroxyl group was the absorbance at 3450 cm⁻¹ and the amide group was the absorbance 1655 cm⁻¹. The DD of the chitosan samples was calculated using the following equation:

$$\%DD = \left[1 - \left[\frac{A_{1655}}{A_{3450}} \times \frac{1}{1333} \right] \right] \times 100\%$$

Where A₁₆₅₅ were the absorbance at 1655 cm⁻¹ of the amide and A₃₄₅₀ were the absorbance at 3450 cm⁻¹ of the hydroxyl.

Results

Preparation of chitosan through 3 subsequent processes i.e. deproteinization, demineralization and deacetylation. The highest protein composition found on

chitosan from *Scylla sp* shell. Protein composition of the shells is shown in Figure1.

Figure1. Protein composition of the shells

The highest mineral composition found in chitosan from *Mytilusviridislinneaus* shells. Mineral composition of the shells is shown in Fig.2.

Figure2. Mineral composition of the shells

The Fourier Transform Infrared (FTIR) Spectra analyzed to characterization of chitosan isolation from shells have been done, shown at figure 3.

Figure 3. The degree of deacetylation by FTIR Method.

The degree of deacetylation of chitosan from *Penaeus monodon*, *Scylla sp.*, and *Mytilus viridis* shells, were 89.05%, 87.64%, and 77.80% mean that the chitosan samples containing minerals were less effective than products from which minerals had been removed.

Discussion

Proteins in the shells cannot be eliminated entirely when deproteinization process, this is due to the protein contained in the shells attached by covalent bonds to form a stable complex. In demineralization process, main mineral content in the shells are CaCO_3 . HCl solution reacts with the mineral shells resulting salts are soluble in the solvent and CO_2 gas.

Deacetylation of chitin was done by removing the acetyl group binds to the amine group using high concentrated of NaOH that C-N bonds of chitin can be interrupted, thus forming the amine group ($-\text{NH}_2$) on chitosan.

DD indicates high purity and potential applied use of chitosan as a coagulant. Most an acetyl group on chitin was lost in the process that shows the extent of (%) deacetylation of the chitosan.

The profiles of FTIR spectroscopy of the chitosan extracted from shells were almost similar, revealed the main adsorption $3200\text{--}3600\text{ cm}^{-1}$ and 2521.97 cm^{-1} (O-H stretching and stretching vibration), 2927.03 cm^{-1} , 1466.89 cm^{-1} and 862.20 cm^{-1} (C-H stretching of CH_4 and CH_2 symmetry), 1083.05 cm^{-1} (C-O stretching), 3300 cm^{-1} and 3500 cm^{-1} (N-H bending of amide bonds). A new revealed adsorption with sharp intensity at 3445.89 cm^{-1} of chitosan spectra designed N-H bending of amine bonds (primary amines). These spectra were characteristic and profile the main chitosan groups (NH_2 primary), see fig. 3. Chitosan have amine groups ($-\text{NH}_2$), positively charged in acidic conditions, and it acquires interesting cationic character which is responsible for its flocculating activity. Amine content of the chitosan used as a coagulant allows water purifier [4]. Prosperity to form a flock of chitosan in the coagulation-flocculation process will influenced by the degree of deacetylation [5, 6].

Conclusion

Chitosan is potential to be applied as coagulant in a coagulation /flocculation to remove turbidity and colloidal material in water treatment and substitute for aluminum salts and synthetic polyelectrolytes in water treatment.

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