Degree of Acetylation Chitosan Gonggong Snail Shells

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Abstract. Chitosan is a polysaccharide obtained from the deacetylation of chitin, which is generally derived from crustacean animal waste and animal skins other sea. One marine animals that have compounds that can be processed chitin chitosan is derived from the snail Gonggong marine waters of Riau Islands province. The purpose of this study was to determine the degree of chitosan from the shells of snails asetilisasi Gonggong. This research is an experimental research laboratory. The results of this study indicate that the degree of chitosan shell snail deasetilisasi Gonggong is 70.27%.

1. Introduction
Indonesian nation consists of 17,502 islands and a coastline of 81,000 km with a total area of fishing in the sea about 5.8 million km², which consists of the archipelagic waters and territorial area of 3.1 million km² as well as the waters of the Indonesian Exclusive Economic Zone (ZEEI) area of 2.7 million km². These facts indicate that the prospects for development of fisheries and marine Indonesia is considered very bright and become one of the strategic economic activities. Live fish resources in the territorial waters of Indonesia has a level of biodiversity (bio-diversity) the highest.

Fishery potential in Riau Islands Province, including in the area of the South China Sea fishery management and Natuna through the last boundary line Indonesian Exclusive Economic Zone (ZEEI). In 2006, the fishery production amounted to 220,570.61 tons. Some 217,094.91 tons (99.5%) came from capture fisheries at sea. Followed by marine aquaculture production amounted to 3279.05 tons (0.4%), production of 174.66 tons of freshwater aquaculture and brackish water aquaculture production (farms) amounting to 21.99 tons (0.1%). Marine waters in Riau Islands have the potential of marine life is abundant and diverse (Ministry of Maritime Affairs and Fisheries, 2011). One of the potential of the Riau archipelago are found around the coast of Bintan and Batam is Snail Gonggong. Snail Gonggong in Tanjungpinang production in 2013 reached 147.6 tons, where the average searcher gonggong got less than 6 kg of snails barking day [1].

Gonggong snail is a typical food from Tanjungpinang. Gonggong snails are found in seafood restaurants in Tanjungpinang. Waste generated from Snail shell Gonggong reached 75% of the amount Gonggong snails are eaten. From 1 kg of waste obtained Snail shell Snail Gonggong ± 750 grams. Waste shells of snails Gonggong only used for crafts such as key chains and ornaments on souvenirs, but it is not uncommon shell snail Gonggong dumped and left to rot which eventually cause negative
impacts on the environment such as causing odor that spoil the aesthetics of the environment and be
garbage piling of the coast or under the house eating seafood. One alternative effort snail shell
Gonggong waste utilization in order to have the value and usability of Gonggong Snail shell waste into
products of high economic value is the process into chitin and chitosan [2]. Chitosan is poly (2-amino-2-deoxy-β- (1-4) -D-glucopyranose) with molecular formula (C6H11NO4)n which can be obtained from the deacetylation of chitin (Figure 2.2). Chitosan is also found naturally in
some organisms [3].

Figure 1. The structure of the polymer chitosan

Chitosan deacetylation process can be done by chemical or enzymatic. Chemical process using a
base, for example NaOH, and can produce chitosan with a high degree of deacetylation, which reached
85 to 93% [3]. So that the physical and chemical properties of chitosan are not uniform. In addition,
the chemical process can also cause environmental pollution, difficult to control and involves many
side reactions that can decrease yield. Enzymatic processes can cover the shortage of the chemical
process. Basically, the enzymatic deacetylation is selective and does not damage the structure of the
chitosan chains, resulting in chitosan with more uniform characteristics in order to extend the field of
application [3].

Chitosan has unique properties that can be used in a variety of ways and has a variety of uses, such
as adhesives, additives for paper and textiles, drinking water purification, as well as to accelerate
wound healing, and improve the binding properties of color. Chitosan is a powerful adhesive to
transition metal ions [4].

Chitosan can be isolated from snail shell waste Gonggong through three stages namely
deproteinization, demineralization and deasetilisasi. Deacetylation process aims to break the covalent
bond between chitin acetamide group so it turns amine (-NH2). The magnitude of the removal of acetyl
group in chitin acetamide group known as the degree deacetylation (DD). According to Khan [5]
suggest that chitin with a DD of 75% or more commonly known as chitosan. Deacetylation degree is
one of the most important chemical characteristics as DD chitosan affects performance in many
applications [5].

2. Methods
This study was an experimental study carried out in laboratories Environmental Health Department of
the Ministry of Health Polytechnic Tanjungpinang from March to June 2017. The chitosan FTIR
radiation measurements carried out in the Laboratory of Chemistry, State University of Padang.
The tools used are Oven, filter paper, pH meter, universal indicators, flask, glass beaker, stir bar, glass
funnel, measuring cup, pipette volume, analytical balance, hot plate. Materials used are NaOH, HCl,
distilled water, Whatman filter paper, Paper Label, Gonggong snail shell. Stages of this research is the
sample preparation Gonggong snail shells, chitin isolation o
f snail shells Gonggong deasetilisasi and
FTIR measurements.
1. Sample preparation
Waste Gonggong Snail shells cleaned of residual dirt and food debris attached. Snail shells Gonggong
repeatedly washed until completely clean. Snail shells Gonggong dried at a temperature of 50°C oven
for 6 hours and blended to a powder.
2. Isolation Chitin
Chitin isolation process is done by modifying the method Hang in Fahmi [6] and Susilowati [7]. Snail shell chitin isolation from Gonggong done through the stages of deproteinization and demineralization.

3. Deacetylation
Ten grams of chitin added 200 mL of 50% NaOH. The reaction is conducted at a temperature of 120°C for 4 hours. The solution is separated from the residue and the residue was neutralized by washing using aquades do the repeatedly. The residue was dried in an oven for 4 hours at a temperature of 60°C. After drying blended until smooth with a size of 150 mesh. The residue is further characterized using FTIR.

4. FTIR measurements
Chitosan is stored in a desiccator for one day to remove the water content. Furthermore, the manufacture of pellets from powders containing samples of ± 1 mg and 10-100 mg KBr. The mixture is subjected to grinding until a homogeneous powder and pressed with a hydraulic pump. The results obtained are in the form of pellets, the pellets will be analyzed by FTIR spectrophotometer base-line method.

3. Results and Discussion
Waste bark snail shells cleaned and dried at 50 °C using the oven for 6 hours and blended to a powder. A total of 500 g of powdered shells of snails Gonggong put in a glass beaker and added with 1.5 M HCl in the ratio 1: 5 (w / v) for 4 hours at a temperature of 65°C, stirring constantly. The continue cooled briefly and then filtered. Then washed with distilled water until neutral and tested with AgNO3 to detect residual Cl ions. Furthermore, the solids dried in an oven with a temperature of 80°C for 24 hours to obtain powdered shells of snails Gonggong without minerals.

Snail Shells Gonggong powder demineralized put in a glass beaker and added with NaOH 3.5% with a ratio of 1:10 (w / v) for 4 hours at a temperature of 65-70°C. Then cooled briefly and then filtered. Then washed with distilled water until neutral. Furthermore, the solids dried in an oven with a temperature of 80°C for 24 hours in order to obtain chitin.

Chitin obtained from entered deproteinization process into a glass beaker and added with NaOH 50% with a ratio of 1:20 (w / v) for 4 hours at a temperature of 120°C, stirring constantly. Subsequently cooled briefly and then filtered. Then washed with distilled water until neutral and tested with AgNO3 to detect residual Cl ions. Furthermore, the solids dried in an oven with a temperature of 60°C for 4 hours to obtain chitosan.

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![Figure 2. Chitosan shells of snails Gonggong dried.](image)

Chitosan Deacetylation degree Snail Shells Gonggong degree of deacetylation of chitin is determined by the method of the base-line. This method is based on comparison of the value of spectrum absorbance of infrared absorption bands at wave number 1655 cm⁻¹ and 3450 cm⁻¹. Absorbance (A) is expressed as the equation (1), while the value of DD is expressed as the equation (2).
\[ A = \log \frac{P_0}{P} \]  

where:

\[ P_0 = \text{% transmittance at base-line (maximum absorption)} \]
\[ P = \text{% transmittance at the maximum} \]

\[ \text{absorption\% DD} = 100 - \left(\frac{A_{1665}}{A_{3450}} \times 115\right) \]  

Value of A1665 and A3450 is a value corresponding to an absorption band 1665 cm\(^{-1}\) and 3450 cm\(^{-1}\). Absorption band 1665 cm\(^{-1}\) is the absorption band of N-acetyl carbonyl group while the absorption band 3450 cm\(^{-1}\) is an absorption band group NH2 [5,8].

The results of calculations using the equations 2 FTIR spectrum acquired 70.27% DD. The process of removal of acetyl groups (-CH\(_3\)COO-) of the acetamide group (-NHCOCH\(_3\)) forming the amine group (NH\(_2\)) is also called deacetylation. If the acetyl group separated from the chitin is called deacetylation of chitin. The mechanism of deacetylation of chitin occurs in an alkaline solution. In alkaline solution an ester carbonyl carbon can be attacked by a nucleophile without prior protonation [9]. Process acetyl group at acetamide deacetylation of chitin can be explained as follows: carbon carbonyl group is attacked by a nucleophile OH\(_{-}\), resulting in an addition reaction to form intermediates. These intermediates elimination subsequent reactions so that an acetyl group on the loose form of chitin acetamide acetate.

The process of release of acetyl groups from chitin acetamide groups associated with OH\(_{-}\) ion concentration in the solution. OH\(_{-}\) concentration will be greater in strong alkaline solution. The stronger a greater alkaline OH\(_{-}\) concentration in the solution.

DD chitin isolation results obtained are 70.27% these results suggest that isolation already a chitosan because, according Terbojevich [10] if DD 40-100% called chitosan.

**Conclusion**

Acetylation study was to determine the degree of chitosan from the shells of snails Gonggong can be concluded that chitosan DD Gonggong Snail Shells 70.27%.

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