



Chitosan Application as Edible Packaging Raw Material

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

Chitosan extracted from shrimp or crab can be made as raw material for biodegradable packaging. The purpose of this article is to review the advantages of chitosan as a raw material for packaging, the extraction method of chitosan, the properties and quality of chitosan, biodegradable packaging, manufacturing methods, and properties of packaging made from chitosan and the development of chitosan products for packaging materials. Chitosan is a raw material for packaging that can be parsed, commonly called biodegradable packaging, non-toxic and anti-bacterial. The method of making chitosan is removing minerals and removing protein from shrimp, crab, or ranjungan shells to obtain chitin, followed by the chitin acetylation process to obtain chitosan. The quality of chitosan is listed in SNI No.7949 (2013), where this standardization includes color, moisture content, ash content, nitrogen content, and degree of deacetylation. Chitosan has been used for edible coating material and edible film material, both of which have different manufacturing methods and characteristics. The development of chitosan as raw material for degradable packaging is that chitosan is made in nanoparticle size.

Keywords: *Nanoparticles; excellence; quality; method; extraction.*

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1. INTRODUCTION

Food packaging use materials that accordance with the food to be packaged. Food packaging serves to maintain and protect food until it reaches consumers safely. Edible packaging is biodegradable packaging and can preserve the quality of food ingredients during storage. Edible packaging for food packaging is divided into three types, namely edible coating, edible film, and encapsulation [1].

The edible coating is a package that is used as a coating for semi-wet food and fruits. Meanwhile, the edible film is a thin and continuous layer in polymer chain interactions, resulting in more extensive and more stable polymer aggregates. Encapsulation is a powder material that carries flavor. One of the potential edible packaging materials used is chitosan, a deacetylation product of chitin by heating it in an alkaline solution [2].

Chitosan is a polysaccharide derived from chitin which is the second-largest polysaccharide after cellulose. Chitosan is generally made from shrimp, crab, and small crab shells. Chitosan has properties that can form films, non-water, can be degraded in nature, is non-toxic, and can increase transparency, so that is a suitable packaging material (Kittur et al., 1998). Therefore, this article aims to review the advantages of chitosan as a raw material for packaging, chitosan extraction methods, properties and quality of chitosan, biodegradable packaging, manufacturing methods, and packaging properties made from chitosan as well as the development of chitosan products for packaging materials.

2. ADVANTAGES OF USING CHITOSAN AS RAW MATERIAL FOR PACKAGING

The advantages of chitosan as raw material for packaging include biodegradable, edible, and anti-microbial activity [2]. Chitosan is non-toxic and is a polymer that can be broken down by nature, and its structure is similar to cellulose, has a positive ion charge which can chemically bind to lipids, metal ions, and proteins [3].

The main characteristic of chitosan as a raw material for packaging is that it is anti-microbial. Factors that influence chitosan against microbes

include intrinsic and extrinsic properties of chitosan. The mechanism of chitosan as an anti-microbial is because chitosan has a porous membrane form that can absorb water in food to inhibit microbial growth in the food. The antibacterial of chitosan is an amine functional group and chitosan's absorption ability, which has a positive charge. Whereas in the cell, the microbial membrane is negatively charged. This negative-positive charge interacts electrostatically, which causes the membrane to experience permeable pressure causing the osmotic pressure in the cell to be unbalanced, thus preventing the growth of microbes [4].

3. CHITOSAN EXTRACTION METHOD

Chitosan is a natural compound that is a derivative of chitin. This chitosan comes from fishery waste such as shrimp shells or crab shells, containing a large amount of chitin, which is between 55 - 70% [5]. Raw materials for making chitosan can also use mushrooms, octopus, silkworms, and even insects and scorpions because they contain 5-45% chitin [5]. This chitosan is obtained from the distillation process of chitin. Distillation is a process to convert the acetyl group (-NHCOCH₃) in chitin to an amine group (-HHA₂) with a strong base (NaOH) [6]. Meanwhile, chitin can be obtained by deproteinating and demineralizing shrimp or crab shells [7].

The procedure for making chitosan is then continued with chitin acetylation to obtain chitosan as follows [9].

Fig 1. shows the chemical structure change in the structure of chitin (a) to chitosan (b). This changing structure is caused by a synthesis process that changes the acetamide group (-NHCOCH₃) into an amine group (NH-2). The first step of making chitosan is deproteination which aims to remove the protein contained in the shrimp shell powder using NaOH solution. The next step is to remove minerals using HCl at high temperatures or also called demineralization. Furthermore, or the final stage of making chitosan is deacetylation, this stage aims to remove acetyl groups using NaOH and HCl with high temperatures and concentrations, the result of this deacetylation will produce poly-2-amino-2-deoxy-β-D-Glucose or chitosan. Schematically, chitosan is made in the following flow diagram.

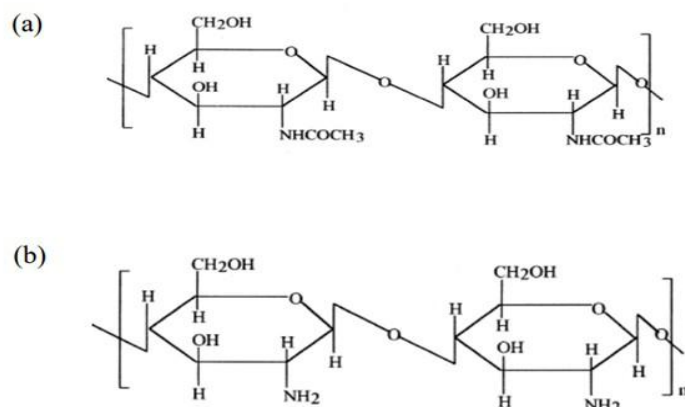


Fig. 1. Structure of Chitin (a) and Chitosan (b) [8]

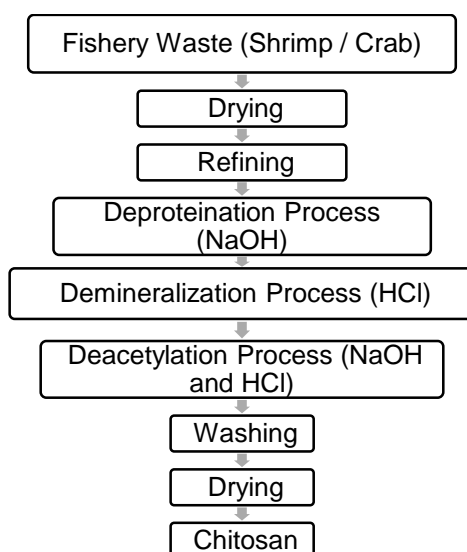


Fig. 2. Flowchart of Chitosan Making [9]

4. PROPERTIES AND QUALITY OF CHITOSAN

Chitosan has a water-retaining ability that can be applied in many fields. The ability to hold water in chitin and chitosan is different. This is due to differences in the number of forming groups, namely the salt and protein content of the material itself. The lower the protein content in the raw material for making chitosan, it will produce chitosan with good quality [10].

Chitosan is a fascinating natural polymer compound, and this is because chitosan is an electrolyte polymer used for fuel cell applications at low and medium temperatures. Dewi [11] stated that the application of the cell as a fuel had been developed since 1839 by William R.

Groove to overcome the critical period of oil. The results of this development, for example, are the hydrogen-polymer electrolyte fuel cell (PEFC) and direct methanol fuel cell (DMFC). Chitosan has a low conductivity value because the monomer structure contains only three H₂O atoms. These structures are strongly bonded, causing the bonds to be unable to generate electricity [12]. The quality standard of chitosan is listed in SNI No.7949 (2013). This standardization includes color, moisture content, ash content, nitrogen content, and degree of deacetylation.

4.1 Color

Based on the standardization of chitosan, it must be color light brown. The brown color of chitosan

is produced from the depigmentation process, which aims to remove the pigment (color) from astaxanthin which has a red-orange color.

4.2 Moisture Content

The water content in chitosan is influenced by making chitosan, starting from the drying time, the amount dried, and the surface area of the place to dry. During the hot drying process, the weather also dramatically affects the moisture content in the chitosan. To get chitosan with low water content, you can also use an oven apart from drying directly under the sun. This is because the oven has reasonably stable heat.

4.3 Ash Content

In chitosan, a high ash content indicates that the chitosan has high mineral content. The high ash content in chitosan will reduce the quality of chitosan. The high and low ash content in chitosan is influenced by the demineralization and washing processes. Good washing gives good results on the ash content in chitosan, and likewise, if the washing is done poorly, the minerals that should have been released can stick back together.

4.4 Total Nitrogen Level

The success of the deproteinization process is the decrease in nitrogen content produced in chitosan. NaOH concentration and the high temperature in the distillation process will cause nitrogen levels to get smaller [13].

4.5 Degree of Deacetylation

The degree of deacetylation is the parameter that most determines the quality of the resulting chitosan. The results of this degree of deacetylation can also be used to observe the effect of changing chitin to chitosan. After calculating the deacetylation degree of chitosan, if the chitosan produced meets it, it can be said to be an adsorbent requirement.

The following is a table of the quality standards of chitosan according to SNI No. 7949 (2013).

5. BIODEGRADABLE PACKAGING

Biodegradable packaging is a packaging film that can also be recycled. Biodegradable or bioplastic plastics are plastic products where part or even all of the component composition is made from recyclable raw materials. Biodegradable packaging can be used like conventional plastic, but there is a difference between biodegradable plastic and conventional plastic [14].

Biodegradable packaging based on raw materials is divided into two groups: packaging with petrochemical raw materials and packaging made from living things, both plants and animals. The use of petrochemical raw materials results in plastic being (non-renewable resources) or other words, recyclable. Meanwhile, using natural materials by utilizing organisms will produce renewable plastic, which means that this plastic can be recycled and can decompose quickly in the environment [14].

Table 1. Quality standards of chitosan

Parameters	SNI No. 7949 (2013)
Color	Light Brown to White
Water Content	≤ 12%
Ash Content	≤ 5%
Total Nitrogen Content	≤ 5%
Degree of Deacetylation	≥ 75%

Table 2. Types of plastics based on raw material classification and degradability

Type of Raw Material	Biodegradability
Renewable	Biodegradable Starch-based materials, Cellulose-based materials, PLA and PHA
Non-Renewable	PCL and PBS

Source : [14]

In the manufacture of biodegradable packaging films, biopolymers are used as the primary material. Biopolymers are divided into three groups [14] :

a. The Mixture of biopolymer with synthesis polymer

This film is made with a mixture of granule starch and synthetic polymer and additives such as oxidants and antioxidants. This material has low biodegradability value as well as limited fragmentation.

b. Microbiological polymer (Polyester)

This biopolymer is produced by fermentation using microbes of the genus *Alcaligenes*. Bacteria, fungi, and algae can degrade this material.

5.1 Agricultural Polymers

These biopolymers are natural materials that are not mixed with synthetic materials but use agricultural products. These polymers include Cellulose (part of the plant cell wall), Cellophane, Cellulose acetate chitin (in the skin of the Crustacea), Pullulan, or the fermentation of starch by *Pullularia pullulans*). Agricultural polymers have thermoplastic properties, so that they can be formed or printed into packaging films.

In addition to the differences in polymers, the following table includes some differences between conventional plastics, mixed plastics, and biodegradable plastic.

6. METHODS OF MAKING PACKAGING MADE FROM RAW CHITOSAN

Edible packaging, which is biodegradable packaging to maintain the quality of food ingredients when it's stored. One material that can use for edible packaging is chitosan, which is a deacetylation product of chitin by heating it in an alkaline solution.. There are two types of edible packaging, namely edible coating and edible film. The edible coating is a package used as a coating (coating) for semi-wet food and fruits. Meanwhile, the edible film is a thin and continuous layer in polymer chain interactions, resulting more extensive and more stable polymer aggregates.

6.1 Edible Coating

Chitosan is a raw material that can use for edible coating (Swastawati et al. 2008). The edible coating is a thin layer formed due to dipping, spraying the surface of food products to protect and increase the product's added value [15]. Meanwhile, edible coating packaging is used as a coating for perishable foods such as semi-wet food and fruits [2].

The process of making Edible coating packaging from chitosan is as follows [16].

Table 3. Comparison of conventional plastics, mixed plastics, and biodegradable plastics

Observations	Conventional Plastics	Mixed Plastics	Biodegradable Plastics
Composition	Synthetic Polymers	Synthetic Polymers and Natural Polymers	Nature Polymers
Nature and Raw Material	Non-renewable	Partly renewable	Renewable
Mechanical and Physical Properties	Non-renewable	Variety	Good and Variety but Limited Use
Biodegradability	Very good and Variety	Low	High
Compostability	None	Low	High
Combustion Product	None	Not Too Stable	Less Stable
Example	Stable PP PE	Pe + Starch Pe + Cellulose Synthetic Polymers and Natural Polymers	PLA PHA

Source: Vilpoux and Aveorous, 2006

- Production of chitosan uses ingredients from vannamei shrimp shells (*Litopenaeus vannamei*), NaOH, HCL, acetone, NaOCl, and distilled water deproteination, demineralization, bleaching, and deacetylation stages. Dissolve 1, 2, and 3 grams of chitosan in 30 mL acetic acid, then add 70 mL of distilled water.
- The solution is homogenized with a magnetic stirrer at 50°C for 60 minutes until the coating solution is completely dissolved. The selection of the chitosan solvent used to dissolve chitosan was acetic acid with a concentration of 1%.
- Then the dry film is removed and placed in a desiccator, for physicochemical analysis (thickness, color, and density); mechanical characteristics (tensile strength, and creep test /% elongation); and functional (WVTR, Water Vapor Transmission Rate).

6.2 Edible Film

The edible film is a thin film that can be consumed and used as a coating or barrier between food and the surrounding environment. Edible films are classified into three categories based on the nature of the components, namely hydrocolloids (proteins and polysaccharides), fats (fatty acids, acylglycerols, or waxes), and composites (a mixture of hydrocolloids and fats) [17].

The process of making chitosan edible film packaging is as follows [18]:

- Making chitosan using wet shrimp shell waste raw material cleaned of organic impurities with warm running water, then
- dried in the sun until it is quite dry in the sun for 2-3 days.
- Preparation of chemical solutions Solvents for extraction (3.5% and 505 NaOH solutions, HCl 1N NaOCl 0.32%) are prepared for extraction, deproteination, demineralization, decolorization, and deacetylation.
- Dissolving chitosan powder in 1% solution acetic acid. The mixture is stirred vigorously for 30 minutes
- Then immersed in boiling water for 10 minutes, cooled at room temperature, and
- Then filtered with a glass-wool filter to remove insoluble particles.
- The mixture is then divided into several 500 mL beaker glass containers. Each container is added glycerol as a plasticizer with a ratio (glycerol chitosan solution) 0.2 1 (w / w).
- The plastic film solution formed is then molded and allowed to dry at room temperature for 48 hours.

The formation of edible films can use several types of acids, such as acetic, lactic, formic, malic, and propionate acids. However, only acetic and formic acid produce flexible, transparent, and suitable films for packaging. This is because lactic and malic acids have more hydroxyl groups which increase the hydrophilic properties of chitosan. Thus, it is known that the type of acid greatly affects the characteristics of the film produced (Nadarajah et al., 2006).

The difference between making edible coating packaging and edible film is several additives (plasticizers). The process of making edible coating packaging does not use plasticizers. Meanwhile, making edible film packaging uses a plasticizer that functions to reduce polymer stiffness to obtain an elastic and flexible layer [2].

The most commonly used plasticizers are polyols (propylene glycol, glycerol, sorbitol, polyethylene glycol), oligosaccharides, and water. The additions are given in the range of 10–60% by weight of the hydrocolloid. Glycerol and sorbitol are effective plasticizers because they can reduce internal hydrogen bonds in intramolecular bonds [19]. But the results of films with lower tensile strength are due to the addition of a plasticizer that exceeds a certain amount [2].

7. CHARACTERISTICS, OF BIO-DEGRADABLE PACKAGING FROM CHITOSAN

7.1 Characteristics of Chitosan as an Edible Coating

Based on research conducted by [20], the characteristics of chitosan as an edible coating are water content and ash content of 8.5% and 1%. This value follow the quality standard of chitosan water content, namely <10%, and a maximum ash content of 2%. The degree of deacetylation of chitosan products has met the quality standards of chitosan that have been determined by the Proton Laboratory, namely ≥70%. It shows that the deacetylation process has been going well. The viscosity of chitosan produced in this study was in the high category, namely between 800-2000 cps, so that the

chitosan results of the survey qualify as edible coating food packaging.

7.2 Characteristics of Edible Film Packaging from Chitosan

The physicochemical characteristics of edible film that can be seen or observed visually are the colors on the film. edible film in clear color [18]. Film made from DMKA chitosan (deminalization, decolorization, and deacetylation) + sorbitol and DMKA chitosan + sorbitol + CMC (carboxyl methylcellulose additive) have a slightly brownish color [18]. While the thickness of the bioplastic film made from chitosan DPMA + glycerol and DMKA chitosan + glycerol with a thickness of 0.3 mm [18].

Based on JIS (Japanese Industrial Standard) 2 1707 + 1975 in Utami (1998), plastic films for food packaging that are categorized as films are those that have a maximum thickness of 0.25 mm. Meanwhile, the density is calculated by

dividing the mass and volume of the bioplastic (2 cm long and 1 cm wide). The measurement of density values is crucial because plastic density is closely related to the ability of plastics to protect products from several substances present in free air. Low-density plastics have a more open structure with greater porosity, so that the greater the density of the film, the better the quality (Nurmiah 2009). The highest bioplastic density was in the DPMA + sorbitol + CMC film with a value of 1.730 g / cm³, while the lowest density was in the DPMA + glycerol + CMC film with a value of 1.32 g / cm³.s water vapor, O₂, and CO₂.

Analysis of the mechanical characteristics of bioplastics includes tensile strength and% elongation (% elongation) tested using the Unit Testing Machine (UTM). The tensile strength and% elongation of bioplastics are influenced by the type of plasticizer used. A study conducted by [18] using glycerol and sorbitol plasticizers also carried out CMC.

Table 4. Characteristics of chitosan as an edible coating

Parameters	Commercial	Research Results
Particle Size	Flakes to Powder	Flakes to Powder
Moisture Content	≤ 10%	8.5%
Ash Content	≤ 2%	1%
Solution Color	Clear	Clear
Deacetylation Degree	≤70%	73.54%
Viscosity (cps)		
• Low	< 200 pcs	
• Medium	200 – 79cps	
• High	800 – 2000cps	1000 cps
• Extra	>2000 cps	

Source : Kristiana Yahya et al. 2015

Table 5. Results of analysis of physicochemical characteristics of various types of edible film

Edible Film Type	Color	Thickness (mm)	Density(gr/m)
DPMA +	Glycerol	0,30	1,298
	Glycerol + CMC	0,15	1,150
	Sorbitol	0,05	1,320
	Sorbitol + CMC	0,10	1,730
DMKA +	Glycerol	0,30	1,365
	Glycerol + CMC	0,25	1,256
	Sorbitol	0,20	1,295
	Sorbitol + CMC	0,20	1,302

Source: [18]

Table 6. Result of analysis of mechanical characteristics

Bioplastic		Tensile Strength (MPa)	Elongation (%)
DPMA +	Glycerol	1,15	38,62
	Glycerol	2,65	46,60
	Sorbitol	49,60	40,23
	Sorbitol + CMC	51,80	40,93
DMKA +	Glycerol	1,90	70,28
	Glycerol	2,25	45,60
	Sorbitol	75,25	29,70
	Sorbitol + CMC	27,10	35,73

Source: [18]

Tensile strength is the maximum tensile strength that can be achieved until the film remains before breaking [18]. The percentage of elongation (% elongation) is a measure of the film's ability to stretch when pulled [18]. The use of sorbitol in the manufacture of bioplastics shows a tensile strength value between 27.1-75.25 MPa, where this value is much greater than the use of glycerol plasticizer whose tensile strength is only between 1.15-2.65 MPa. While the percentage value of elongation obtained by using sorbitol plasticizer is relatively lower (29.7-40.93%) compared to the use of glycerol plasticizer (38.62-70.28%). So that the use of sorbitol plasticizer in the manufacture of bioplastics can provide a large tensile strength but not too elastic when compared to the use of glycerol plasticizers. The addition of an average CMC can increase the tensile strength of bioplastics, but the DPMA + sorbitol film with the addition of CMC has a lower tensile strength [18].

Analysis of functional characteristics was carried out by measuring the WVTR (water vapor transmission rate). WVTR measurements were carried out in a controlled temperature room. Measurements were made by covering a petri dish, a plate containing silica gel using a film, then the plates were stored in a desiccator containing 40% NaCl solution for 5 days with weighing every 24 hours. WVTR analysis was

carried out to determine the amount of water vapor that passes through the time union film divided by the area of the film area [18]. The WVTR value obtained from the research results [18], is relatively the same from all types of films, which is ranging from 3,2409-4,8858 g /day.m². This value is in line with JIS (Japanese Industrial Standard) 2 1707 † 1975, where bioplastics for food packaging that are categorized as films are those that have a maximum water vapor transmission rate value of 7 gr / m² per day.

8. CHITOSAN PRODUCT DEVELOPMENT FOR PACKAGING

The development of chitosan products as a packaging material, namely chitosan is made in the form of nanoparticles through simultaneous emulsification and self-assembly techniques. Innovation in packaging, which is often developed at this time, is innovative packaging, divided into active packaging and intelligent packaging. Nanotechnology is the method that has been studied the most for its application in the smart packaging material preparation process. The nanoparticles used as smart packaging materials function as active component carriers which act as biosensors or additives in food products (Silvestre, Duraccio, Cimmino, 2011).

Table 7. Results of functional characteristics analysis

Edible Film Type	WVTR (g/day.m ²)	
DPMA +	Glycerol	4,5792
	Glycerol + CMC	4,0573
	Sorbitol	3,2400
	Sorbitol + CMC	4,8858
DMKA +	Glycerol	4,0155
	Glycerol + CMC	3,4671
	Sorbitol	4,8392
	Sorbitol + CMC	4,2783

Source: [18]

Improving the properties of packaging materials can be done by combining polymer materials and nanoparticles into new materials known as nanocomposites. In active packaging, nanoparticles function as a carrier compound for active components that will release these active components if there is a change in the environment due to the initial process of degradation on the surface of food products [21].

Nanoparticles can adapt to the surrounding environment (biocompatibility) (Silvestre et al., 2011). The nanoparticles (properties) can also be adjusted according to the intended use by selecting the proper preparation process (Silvestre et al., 2011). The advantage of nanoparticles that can be utilized for applications in food technology as innovative packaging materials that can control the process of releasing active compounds into packaged food products. Migration of active compounds can be avoided by creating and designing nanoparticles as packaging materials that are responsive only to certain system conditions (e.g., pH, temperature, etc.).

The self-assembly technique is the most popular polymer processing technique for the synthesis of nanoparticles. Various morphologies can be produced through this preparation technique directly or using emulsification membrane equipment (Agustina, Tokuda, Minami, Boyer, Zetterlund, 2017). There are two types of self-assembly techniques used to form polymer nanoparticles, namely self-assembly techniques in bulk (non-solvent) and self-assembly techniques in liquid solvent media [22].

Polymer nanoparticles with various morphologies have been obtained through solvent manipulation techniques, including spherical micelles, worm-like micelles, rod-like, giant compound micelles, and vesicles [23]. In this technique, the polymer is dissolved in an organic solvent until it is thoroughly mixed. Then the second dissolution process was carried out using an undissolved co-solvent in one of the polymer blocks.

Developing the application of nanoparticles into innovative packaging materials is generally through the incorporation of bioactive compounds into the polymer matrix through a nano emulsification process. Wu and colleagues conducted research using the nanoemulsion method to synthesize polymer-based packaging materials as a biopolymer matrix and essential oil from citrus fruits as bioactive components [24].

Combining of the two emulsion techniques simultaneously is expected to be an alternative solution for preparing chitosan functional material (Shakeelet al., 2008). Simultaneously proceed with the encapsulation process of active compounds and self-assembly of nanoparticles through a second emulsion process, namely oil-in-water (O / W) emulsification (Li, Yujie, et al. 2019). Chitosan biopolymer was chosen as a nanoparticle material because it has good biodegradable and biocompatibility properties. It does not have side effects on the human body when used as a food packaging material.

9. CONCLUSION

Based on the results of reviews from various literature. The conclusion that the superiority of chitosan as a raw material for packaging is that it is degradable, non-toxic, and anti-bacterial. The method of making chitosan is removing minerals and removing protein from shrimp, crab, or ranjungan shells to obtain chitin, followed by the chitin acetylation process to obtain chitosan. The quality of chitosan is listed in SNI No.7949 (2013), where this standardization includes color, moisture content, ash content, nitrogen content, and degree of deacetylation. Chitosan has been used for edible coating and edible film, both of which have different manufacturing methods and characteristics. The development of chitosan as raw material for degradable packaging is that chitosan is made in nanoparticle size.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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Poly (n-butyl cyanoacrylate) nanoparticles via miniemulsion polymerization (1):

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