Characteristic of Chitosan Adhesive from Shell Shrimp Litopenaeus vannamei and Their Application For Producing Particleboard

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Characteristic of Chitosan Adhesive from Shell Shrimp Litopenaeus vannamei and Their Application For Producing Particleboard

D W Ningsi[1*], Suhasman[1], S Saad[1]

[1]Faculty of Forestry, Hasanuddin University, Makassar, Indonesia

*Email: dwi.wahyuningsih33@gmail.com

Abstract.The shrimp shell that rich compounds of chitin can be converted to produce polysaccharides called chitosan. The chitosan can be utilized as bioadhesive for substitute chemical based adhesive for producing particleboard. The purpose of this study was to evaluate the effect of different concentration acetic acid (CH_3COOH) as the solvent to made chitosan adhesive and physical and mechanical properties of the produced particleboard by using sawdust waste. Chitosan adhesive formulation will be used as an adhesive by dissolving chitosan powder with CH_3COOH solvent. This research focused on optimization the solvent content wich is CH_3COOH in different concentration such as 0.5%, 2% and 4%. Particleboard with size of 25 cm x 25 cm x 0.7 cm with 8%, target density 0.75 g/cm^3 were produced using sawdust waste of Paraserianthes falcataria. The processing condition were 180°C in temperature during 12 minutes with pressure of 25 kg/cm^2. The research showed, there is no particular trend in particle board characteristics related to solvent concentration. Physical and mechanical Properties fulfill requirement of JIS A 5908-2003 for Moisture Content, MOE and IB. The produced particleboard that using chitosan as bioadhesive and sawdust waste as particle is potentially to be developed technically based on the quality of the particleboards produced.

1. Introduction

South Sulawesi is one of shrimp production resource which able exported in large number. The volume of shrimp were exported from 11 shrimp industry in South Sulawesi at 2016 reached out 6,884,5 tonnes. The exported shrimp are the frozen formed shrimps without skin and the potential waste of shrimp skin left was quite large. Therefore, the use of shrimp skin residues is very potential to be used as a raw material for chitosan adhesives.

Research on chitosan has been done by some researchers such as investigate raw material, temperature and reaction time on characteristics of chitosan produced [1]. Sinaga (2009) study the effect of chitosan by modifying the stages of demineralization, deproteination, and deacetylation. Adhesive application using chitosan with maple wood particles [2] and utilizing chitosan as an adhesive for producing particleboard from Sengon wood [3]. A number of those researchers using acetic acid (CH_3COOH) to dissolve the chitosan to used as an adhesive. Based on research for the use of chitosan, the concentration of solvents applicated at chitosan has never been studied before. Therefore this study focuses to answer the problems related to it.

Other main materials needed for the production of particle board are wood particles or other lignocellulosic materials. The advantage, the particle board does not require raw materials of high

quality particles. For example, coconut fiber [4], rattan wastes [5] and the collection waste of acacia wood [10] as a material for the production particle board.

In other side, plenty wood waste from sawmill industry remains less use. Waste from sawmill industries often left to pile up, decomposed and burned. The average volume of sawn timber in 2014-2015 in South Sulawesi is 87785 m3 [6]. The amount of waste produced by the sawmill industry was on average 40.48% per year [14]. The high volume of waste produced can cause environmental pollution if not controlled. So, this study focuses on the effect of different concentration of CH_3COOH solvents to chitosan in the manufacture of particle boards by utilizing sawn wood waste.

2. Materials and Method

The wood particles used were waste particles from the sawmill and then were milled to particle by using knife ring flaker. The proportion of sawdust particles size was identified by 6 screens levels: passed 6 mesh/hold 9 mesh, passed 9 mesh/hold 12 mesh, passed 12 mesh/hold 22 mesh, passed 22 mesh/hold 40 mesh, passed 40 mesh/hold 60 mesh, and passed 60 mesh/hold 80 mesh, respectively. The proportion of the particles size distribution data shown in Figure 1. Furthermore, the particles were dried until the moisture content was about 4%.

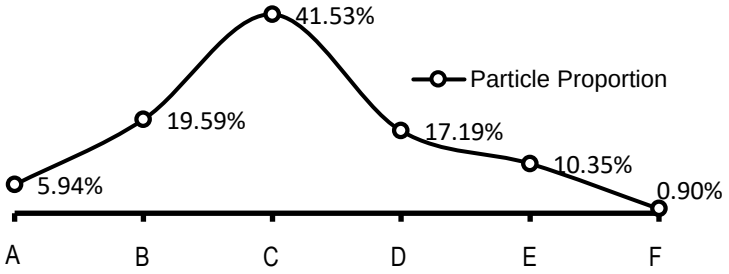

Figure 1. Particles size: (A) Passed 6/hold 9 mesh, (B) Passed 6/hold 12 mesh, (C) Passed 12/hold 22 mesh, (D) Passed 22/hold 40 mesh, (E) Passed 40/hold 60 mesh, (F) Passed 60/hold 80 mesh

The shell shrimp (*Litopenaeus vannamei*) were obtained from PT. Multi Monodon Indonesia. The shell shrimp then washed from residual meat were still attached and air dried and crushed with *hammermil*. There are 3 (three) steps for producing chitosan, namely Demineralization, Deproteinization, and Deacetylation [8].

2.1 Chitosan Production

Demineralization of shrimp shell has been carried out with concentration HCl 2 M with a solid solvent ratio 1:15 (w/v) into a baker. The solution was heated at 70°C for 4 hours while stirring every 10 minutes. The residual was washed with water flows until neutral pH then the shell shrimp were air dried. Deproteinization of shrimp shell was done with 3.5% NaOH with a solid solvent ratio 1:10 (w/v) at 70°C for 4 hours while stirring every 10 minutes. Protein Solution was removed and washed with water flows until netral pH. Then purified chitin was air dried.

The chitosan was prepared by removal of acetyl groups from chitin. It was experimented using concentration of NaOH 60% with solid solvent ratio 1:20 (w/v) at 110°C for 4 hours while stirring every 10 minutes. The residual was washed until neutral pH with water flows. The resulting of chitosan then dried and prepared for characterization.

2.2 Characterization of chitosan produced

Quality of chitosan produced was checked following by Protan Laboratory Standard consist of rendemen, moisture content, viscosity, solubility, solution colour, and deacetylation degree.

2.2.1 Rendemen

Rendemen of chitosan was determined according to the following equation:

$$\text{Rendemen (\%)} = \frac{\text{Output}}{\text{Input}}\ x\ 100 \quad (1)$$

where output is the chitosan weight and input is the chitin weight before heated for deacetylation

2.2.2 Moisture content

Moisture content of chitosan was determined by *moisture analyzer.* About 2 g chitosan was put inside *mositure analyzer*. Then the measured values showed was calculated according to the following equation:

$$\text{Moisture content (\%)} = \frac{w0-w1}{w1}\ x\ 100 \quad (2)$$

where w0 is wet weight of the sample and w1 is dry weightof the sample.

2.2.3 Ash content

Ash content measured with about 2 g chitosan put into pourcelain then it was dried at 100°C in the oven to constant weight. The sample was placed in a desicator and measured the weight. Furthermore, the sample was put on a muffle furnace and heated at 750°C for 6 hours. Then placed in a desicator for minimum of one hour. The sample was allowed to cool and the weight of ash residue was weighed. The ash content was calculated according to the following equation:

$$\text{Moisture content (\%)} = \frac{w1}{w0}\ x\ 100 \quad (3)$$

where w0 is dry weight sample after ovened and w1 is weight of ash residue.

2.2.4 Solubility

Solubility of chitosan was checked with dissolved 2 g chitosan in 2% Acetic Acid with solid solvent ratio 1:100 (w/v). Then the sample was filtered using vacuum filtration and chitosan residue was heated at 100°C for 1-2 hours until get a constant weight. The sample placed in a desicator and the weight was weighed. Solubility of chitosan was calculated according to the following equation:

$$\text{Solubility (\%)} = \frac{w0-w1}{w0}\ x\ 100 \quad (4)$$

where w0 is initial weight of chitosan before dissolved and w1 is final weight of chitosan insoluble after heated and get a constant weight.

2.2.5 Viscosity

Viscosity of chitosan and chitosan adhesive was determinated using *Brookfield Viscometer.* Chitosan solution was prepared in 1% acetic acid at 1% concentration on dry basis. The value are reported in centipoise (cP). For chitosan adhesive, 30 g chitosan dissolved in different concentration of acetic acid (0.5%, 2%, and 4%) with solid solvent ratio 1:5 (w/v). Then the value are reported in centipoise (cP).

2.2.6 Degree of deacetylation

Degree of deacetylation (DD) determined with base line method using FTIR (*Fourier Transform Infrared Spectroscopy*) instrument with frequency range of 4000–400 cm^{-1}. The DD of chitosan sample was calculated based on the following equation:

$$\text{DD} = \left(1 - \left(\frac{A1655}{A3450} X\ \frac{1}{1.33}\right)\right) \text{X } 100\% \quad (5)$$

where A was log (P0/P) for absorbance, A1655 and A3450 were the absorbance at 1655 cm−1 of the amide-I band as a measure of the N-acetyl group content and 3450 cm−1 of the hydroxyl band as an internal standard to correct for thickness.

2.2.7 Particleboards production

Chitosan adhesive was made using dissolved chitosan powder with different concentration of acetic acid (0.5%, 2%, and 4%) with solid solvent ratio 1:5 (w/v). The solid content was used at 8% based on the weight of the oven-dried particles. Furthermore, chitosan gel was distributed into the particles until evenly mixed. The mixture was hand-formed into a mat by using a forming box of 25 x 25 cm with a target density of 0.75 g/cm2. The metal plate was put in the side of the mat to control the board thickness to 0.7 cm. Then the mat was given preliminary pressure and the forming box was removed. After that, the mat that has been formed was pressed using a hot press, which was sealed with thin stainless steel frame. The pressure, temperature and time of press were 25 kg/cm2, 180°C, and 12 min, respectively. Three replications of each production were performed in this study. Then particleboard were conditioned at room temperature for about 14 days.

2.2.8 Evaluation of board properties

The conditioned particleboard tested were physical (moisture content (MC), density (D), thickness swelling (TS), and water absorption (WA)) and mechanical properties (modulus of elasticity (MOE), modulus of rupture (MOR), and internal bond (IB)) according to Japanise Industrial Standard for particleboard (JIS A 5908-2003). The MC and D test were performed on a 10 x 10 cm specimen. The TS and WA test were performed on a 5 x 5 cm specimen from each board after they underwent water immersion for 24 h at room temperature. The weight and thickness of the specimens were recorded for each specimen before and after immersion. The IB was test in spacimens of the same size of those used. The bending properties of the boards were evaluated by a bending test on a 15 x 5 cm specimen for each board in dry conditions.

3. Result and Discussion

The properties of chitosan can be observed qualitatively and quantitatively. According to the Standard Protan Laboratory there were several criteria to evaluate the quality of chitosan [4, 19]. The properties of chitosan according to the Standard Protan Laboratory and the results of the study showed in Table 1.

Tabel 1. Properties of chitosan according to the Standard Protan Laboratory and research results.

Parameters	Standard Protan Laboratory	Chitosan produced	Remark.
The yield (%)*			
a. Shell shrimp powder-demineralization	-	29.61	-
b. Demineralization-deproteination (chitin)		84	
c. Deproteination-deacetylation (chitosan)		82	
d. Shell shrimp powder-chitosan		20.6	
Moisture content (%)	≤ 10	4.8	Fulfilled
Ash content (%)	≤ 2	0.55	Fulfilled
Viscosity (cP)			
a. Low	<200	44.32	Low
b. Medium	200-799		
c. High	800-2000		
d. Very High	>2000		
Solubility in 2% acetic acid	Soluble	Soluble	Fulfilled
Deacetylation (%)	≥ 70	64.02	Unfulfilled
Viscosity of chitosan adhesive (cP)*			
a. Solvent consentration 0.5%	-	4.752	-
b. Solvent consentration 2%		(E)**	
c. Solvent consentration 4%		(E)**	

*) Not specified in the Standard Protan Laboratory

3.1 The yield

The chitosan production process started from demineralization process, which is removing the minerals content at the shell shrimp using HCl solution. In this stage, the minerals contained will reacted with HCl. The effervescence were appear during of heating the shell shrimp was indicated as a reaction of HCl with minerals salt. In this stage, the yield obtained was 29.61%. Furthemore, result of demineralization process was reacted with NaOH solution on waterbath therefore protein contents were dissolved in NaOH solution [9]. A little of effervescence were formed on the surface of the solution, condenced and reddish colour. The condenced was occured because the protein contents were released and linked with Na+ ions and resulted natrium proteinat. The ions Na+ were binding the end of the negatively charged protein chain until settled [9]. After this stage the remaining content of the shrimp

skin powder was chitin. The yield of chitin in this work was 84%. Isolation of chitosan compounds was obtained by performing a deacetylation reaction on chitin. The chitosan was extracted by converting the acetyl groups into amine group in chitin using strong alkalis (NaOH). Therefore, the yield of chitosan in in this study was 82%. The chitosan yield from shell shrimp was 67.08% which is higher than their study [8].
Therefore, the yield of chitosan obtained from this study begined on the shell shrimp powder until chitosan was 20.60%. The calculated of chitosan was not required on the Standard Protan Laboratory but useful for calculating the amount of chitosan that will be applied.

3.2 Moisture and ash content

The moisture content in the present study according to the past works [3, 8]. The commercial chitosan products required by less than 10% moisture content. It was well within the range of chitosan produced. Generally, the moisture content contained in chitosan expressed as H_2O which is bound to polymer functional groups especially amine groups, N-acetyl and hydroxyl through hydrogen bonds [9]. The moisture content of chitosan can be influenced by the process during drying, the drying time, the amount of dried chitosan and the surface in which the chitosan was dried [8].

The ash content of chitosan was indicates as inorganic compounds contained in the raw material of chitosan. The ash content of chitosan in this study and commercial chitosan by Standard Protan Laboratory were 0.55% and less or same than 2%, respectively.

3.3 Viscosity and solubility

The value viscosity of chitosan in this study was 44.32 cP. The value was in low category according to Standard Protan Laboratory. Information about viscosity of chitosan was related to its application. In the pharmaceutical field was required chitosan with low viscosity, whereas for the purposes of thickening or hardening necessary groceries chitosan with high viscosity [11].

The solubility of chitosan on acetic acid is also one of the parameters in determining the quality of chitosan. The higher solubility of chitosan, the better quality of the chitosan produced [8]. In this study, solubility of chitosan was 94.37% which was fulfilled in Standard Protan Laboratory required. Several factors affected the solubility of chitosan were temperature, concentration of alkaline (NaOH), ratio of chitin and alkaline (NaOH) and the size of chitosan powder [12].

3.4 Deacetylation degree

The deacetylation process was carried out to transform chitin into chitosan. Deacetylation was the process to removed acetyl group in chitin to produced chitosan. The amount of acetyl group missing from chitin was called the degree of deacetylation (DD). DD of chitosan can be indicated from function group formed used FTIR analysis. The results of functional group analysis and FTIR reference are shown in Tabel 2.

Funcional Group	**Wave number (cm^{-1})**	
	FTIR reference * (cm^{-1})	**Chitosan produced (cm^{-1})**
OH	3448	3444.87
C-H	2891.1	2881.65
NH_2 scissoring, N-H bending	1655.0	1653
NH_2 wagging and twisting	850.0-750.0	896

*)[12; 1]

Chitosan FTIR spectra showed the emergence of OH strech which indicated absorption in the wave number area 3444.87 cm-1 and amide (C = O) at the 1660.71 cm-1 wave. disappearance of C=O groups in waves 1680-1660 cm-1 indicates the loss or reduction of the acetyl group on chitosan [13]. The reaction of chitosan formation from chitin is a hydrolysis reaction of an amide from a base. Chitin acts as an amide and NaOH as a base. In this process, the -OH group enters into the NHCOCH3 group and then eliminates the $CH\,COO^-$ group and resulting in an amine, namely chitosan [14].

The chitosan DD obtained in this study was 64.02%, The result did not satisfy the Standard Protan Laboratory. According to Pujiastuti (2001), the DD chitin compared to chitosan usually ranges from

70-100% depending on its use. The low chitosan of DD from the results of this study may be caused by the non-optimal isolation process, especially in the stirring process. The stirring factor and heating temperature used were influence the chitosan DD [8].

3.5 Viscosity of adhesive

In this study chitosan was used as an adhesive for production particleboard. The viscosity value of the chitosan formulated in tadhesives is presented below:

Tabel 3. Viscosity of chitosan adhesive

Solvent concentration of CH_3COOH (%)	Viscosity (cP)
0.5	4.752
2	E*
4	E*

*)*Over range*

Based on the results test of adhesives with a solvent concentration of 0.5%, the viscosity value was 4752 cP. While for solvent concentrations 2 and 4%, the viscosity value were illegibled because the maximum viscosity that can be measured is 12,000 c,so the value were over ranged. It is happened because dissolved chitosan in concentrations 2 and 4% resulted too condenced adhesive. The viscosity of chitosan adhesive was higher with increased the solvent concentration [15].

3.6 Characteristic of particleboard

3.6.1 Physical properties

Determination physical properties of particleboard in this study were moisture content, density, thickness swelling and water absorption.

3.6.1.1 Moisture content

The average value of moisture content of the particle board in this was range about 7.9–8.5%. The relationship between chitosan solvent concentration and moisture content of the particleboard showed in Figure 3.

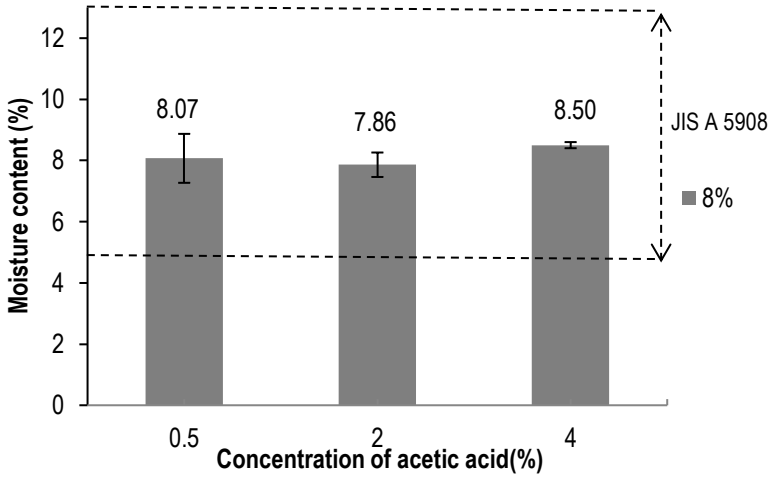

Figure 2. Moisture content of particleboard

Figure 2 showed that the moisture content value of particleboard in this study were the treatment with a solvent concentration 2% and 4% for the lowest and the highest value, respectively. The moisture content of the particleboard in this study was satisfied with JIS A 5908-2003 that required the value in ranged 5–13%. The moisture content of the particle board depends on the surrounding air conditions. In addition, wood particles are hygroscopic the adhesive used (chitosan) also has hydrophilic properties [9].

3.6.1.2 Density

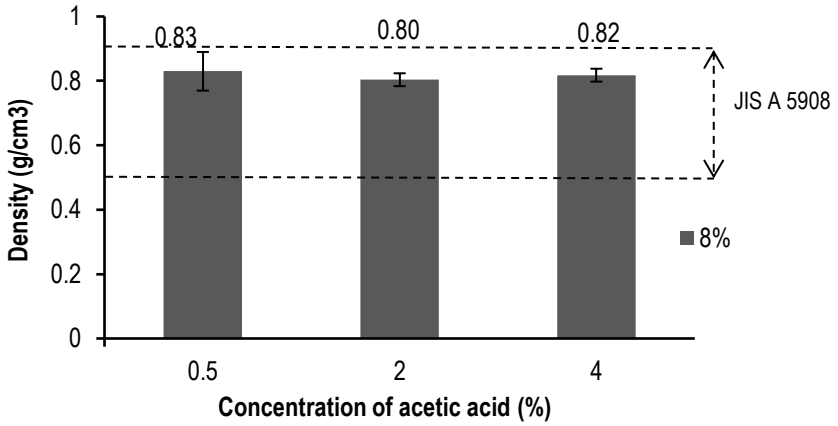

Figure 3. Density of particleboard

Based on figure 3 it can be seen that the average density value produced in this study ranged from 0.80 to 0.83 g/cm^3. The lowest density was obtained on particleboard using a solvent concentration of 2% and the highest density was found in a treatment with a solvent concentration of 0.5%. JIS A 5908-2003 requires the densities of a good particleboard produced between 0.5-0.9 g /cm^3. Overall, the density of the particleboard made in this study met the standard, but not in fulfilled with the targeted density.

3.6.1.3 Thickness swelling

The results of the particle panel test showed that the average of thickness swelling after being immersed for 24 hours at around 17.6-25.8%. The highest and the lowest value thickness swelling was found on the board using 2% and 0.5% solvent concentrations, respectively. The value of the thickness swelling obtained can be seen in Figure 4.

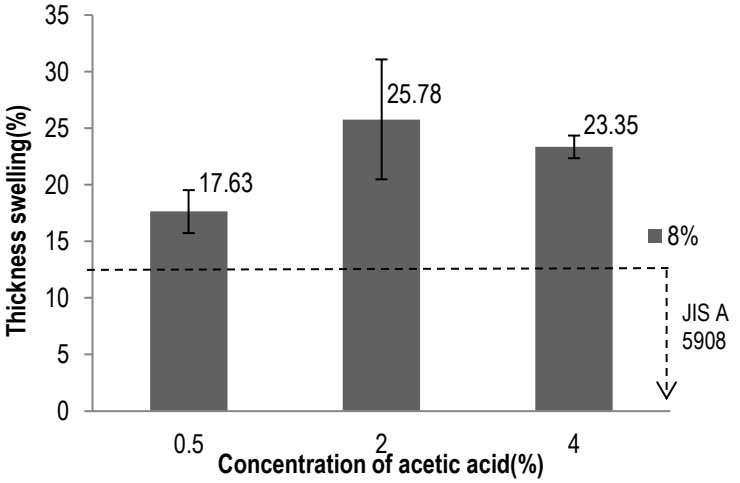

Figure 4. Thickness swelling of particleboard

Based on JIS A 5908-2003, the resulting particleboard does not fully met the standards that require 12%. The absence of the certain trends in thickness swelling that associated with of solvents concentration may be caused the adhesive was not dispersed and the moisture content of the particles and the excess of adhesive. Visual observations showed there was different color in the center of the particleboard indicated the particles and adhesive water was trapped during the pressing process.

3.6.1.4 Water absorption

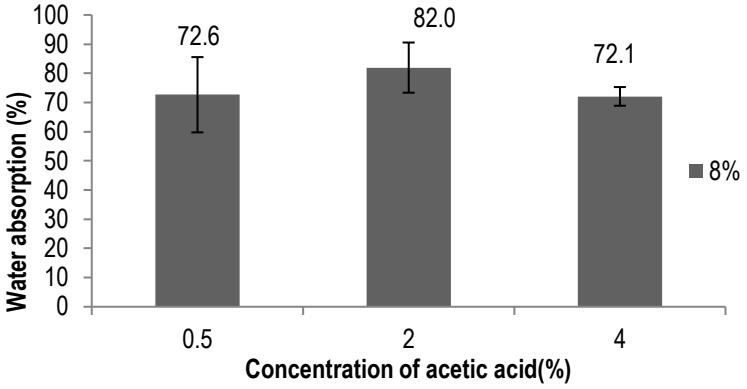

Figure 5. Water absorption of particleboard

Water absorption is the physical properties table that shows the board ability to absorb water. The average water absorption value obtained in this study ranged from 72.1 to 82%. The highest water absorption was produced by the particleboard using 2% solvent concentration and the lowest water absorption on the particleboard with a solvent concentration of 4%. The value of water absorption is not required in JIS A 5908-2003. Decreased water absorption indicated, the adhesive enters into empty spaces inter- particles. Therefore, the space that can be inserted by the water was decreases [16].

3.6.2 Mechanical properties

3.6.2.1 Internal bond (IB)

IB shows the bond value between the particles so the internal bond can be used as a good reference to determine the quality of the particleboard produced [17]. The average IB value of the particleboard ranges from 3.4 to 5.5 kg/cm^2. The highest value was found on the board using a solvent concentration of 0.5%. The IB value can be seen in Figure 7. The highest value was produced by the board using a solvent concentration of 0.5% while while the board using solvent concentrations of 2% and 4% almost resulted the same value.

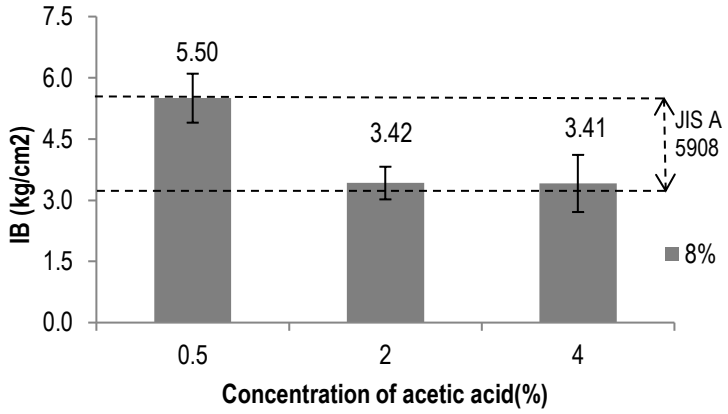

Figure 6. Internal bond of particleboard

Data in Figure 6 showed the IB value of the particleboard tends to decrease with increase in solvent concentration. This probably caused of the high viscosity of the adhesive with solvent concentrations of 2% and 4% (Table 3). Viscosity greatly affects the quality of an adhesive. The higher the adhesive viscosity, the ability of the adhesive to flow, move, retain penetration and wetting were lower, its caused the quality of the bonded product was also low [18]. JIS A 5908-2003 required the internal bond of the particleboard is at least 1.5 kg/cm$^{2.}$ Therefore, the IB value of the resulting particleboard has met the standard.

3.6.2.2 Modulus of elasticity (MOE)

The young's strength test is performed to show the strength of the particleboard to withstand the compressive forces. However, this parameter important because the utilities of particleboard in furniture always requires in flat usage [9]. The MOE value of the particleboard is shown in Figure 7. The average MOE values resulted ranged from 22983 to 24472 kg/cm^2. The highest MOE value was produced by the board using a solvent concentration of 0.5% and the lowest value on the board using a solvent concentration of 4%.

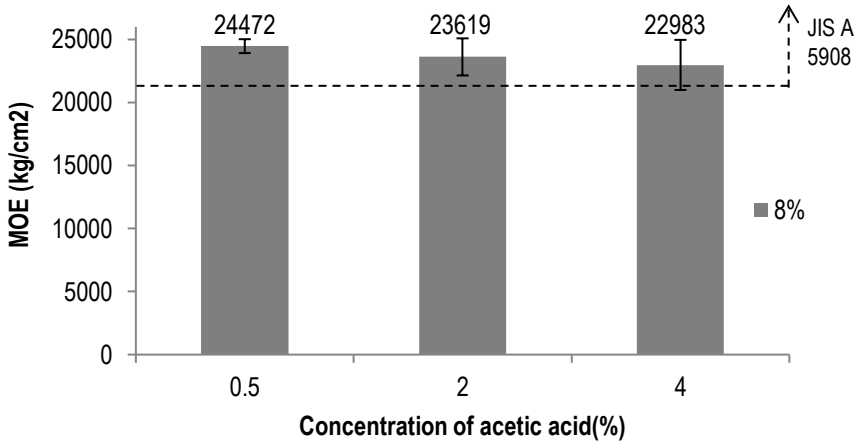

Figure 7. Modulus of rupture of particleboard

Data in Figure 8 showed that the highest concentration of solvent used, then resulted MOE value lower. Based on JIS A 5908-2003 which required a minimum bending value at least 20400 kg / cm^2, so all the particleboard produced met the standard.

3.6.2.3 Keteguhan Patah (MOR)

The Rupture streght is a measure of the maximum load that can be received by the wood. The average values of the MOR ranged from 95.82 to 166.72 kg/cm^2. The MOR value is shown in Figure 8.

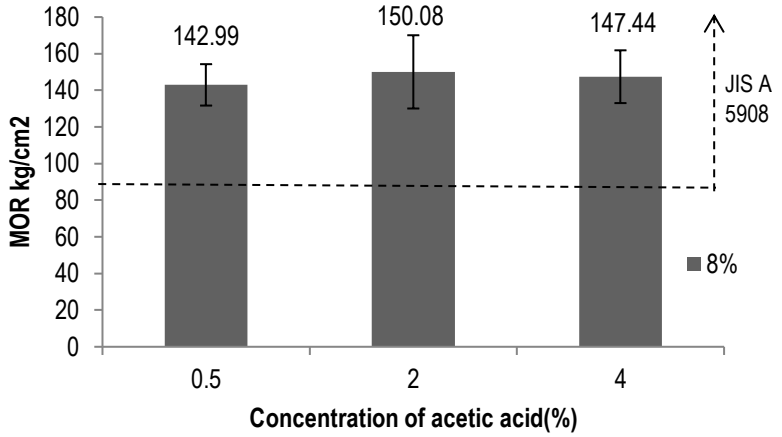

Figure 8. Modulus of rupture of particleboard

The highest MOR value was resulted on particleboard using 2% solvent concentration while the lowest MOR value was on the particle oard using a solvent concentration of 0.5%. Compared to the JIS A 5908-2003, as a whole the chitosan-adhesive particleboard has met the standard which requires a minimum MOR value of 82 kg/cm^2.

3.7. General discussion

The results of this study did not show a particular tendency on the particle board characteristics related to solvent concentrations. The solvent used in this study was CH_3COOH solution. The use of this solution is caused insoluble of chitosan in water. Therefore it is necessary to add CH_3COOH in water. Theoretically, the concentration of the solvent will affect the solubility of adhesive, thus ultimately increasing the adhesive dispersion by increasing the concentration solution.

However, this phenomenon was not found in this study. Variations in solvent concentrations associated with dimension change, adhesive strength and tensile strength, the best values were found on particleboard using a solvent concentration of 2%. While the best value for the elasticity is on the panel with a solvent concentration of 0.5%. It is caused the DD value produced in this study is lower than the required standard, at least 70% so the expected quality objective can be achieved with a lower solvent concentration. The value of DD must be higher than 60% to be soluble in acid even for about 50% of the solvent used is neutral ph [18].

Overall, even the resulting value is not the best, but considering the technical, standard, and economic aspects, the concentration of the solvent at 0.5% is the optimal treatment.

4. Conclusions

The characteristics of the chitosan from the shrimp skin tested in this study met almost all the standards specified in the Protan Standard Laboratory except for DD%. The use of chitosan adhesive in particleboard production shows that the optimum concentration of the solvent is adhesive using a concentration of 0.5% CH^3COOH. Except for the thickness swelling parameters, all the physical and mechanical properties of the particle board parameters fulfill the standards set by JIS A 2908 - 2003.

Based on the data of the tested parameters, it is indicated that the production of adhesives from shell shrimp is very feasible and has the potential to be technically developed based on the quality of the particleboard produced.

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