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Effect of Hydrochloric Acid Concentration as Chitosan Solvent on Mechanical Properties of Bioplastics from Durian Seed Starch (*Durio Zibethinus*) with Filler Chitosan and Plasticizer Glyserol

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Abstract

The production of starch based bioplastic from durian seed as polymer matrix with addition of chitosan as filler and glycerol as plasticizer were investigated. The aim of this research to obtain the effect of hydrochloric acid concentration as chitosan solvent on the mechanical properties of bioplastics included density, tensile strength, elongation at break, functional group using FT-IR, and surface morphology using SEM. Starch is the raw material for bioplastics which extracted by the durian seeds, then characterized to determine its chemical composition. The production of bioplastic method used in this research was casting method. Variation of hydrochloric acid concentration used 0.9%v, 1.0%v, 1.1%v, 1.2%v and 1.3%v. Bioplastic were analyzed physical and chemical properties. From the analysis, best condition of bioplastics obtained at hydrochloric acid concentration 0.9 %v with temperature 72.5 oC for tensile strength 10,137 MPa, elongation at break 8.416 %, and density 1.618 gr/cm3. From the results of FT-IR analysis indicated O-H group and N-H group on bioplastics due to the addition of chitosan and glycerol. The results of mechanical properties were supported by Scanning Electron Microscopy (SEM) showed the bioplastic with chitosan as filler and plasticizer glycerol have the fracture surfaces were a bit rough and jagged.

Keywords— Durian Seed, Bioplastic, Chitosan, Hydrochloric Acid Concentration, Glycerol

Introduction

Plastic are widely used for packaging and other applications because of their several advantages compared to other materials. However, these artificial macromolecular subtances are usually originating from petroleum and most of the conventional ones are regarded as non-biodegradable (Janssen & Moscicki, 2009; Nair & Laurencin, 2007). Based of that case we need solutions to manage this environmental problem, one of the solution is evolving the material or substance for producing biodegradable plastics (Darni, Ismiyati, & Marbun, 2010). Bio-plastic or organic plastic is a polymer material obtained from renewable biomass sources such as vegetable oil, corn starch, potato starch, and pea starch unlike fossil-fuel plastics derived from petroleum (Kipngetich & Hillary, 2013). This type of plastic are naturally degradable by microorganism to become environmentally substances. Materials that use to make biodegradabel plastic are compounds such as cellulose, starch, and lignin that are can be obtain from the plants, and casein, protein and lipid from the animals (Averous, 2004).

Among all biodegradable polymers, starch has been considered as one of the most promising one due to its easy availability, biodegrad- ability, lower cost and renewability (Xu, Kim, Hanna, & Nag, 2005). Starch consists primarily of branched and linear chains of glucose molecules, namely amylopectin and amylose, respectively. Amylose is essentially a linear molecule with a few branches, whereas amylopectin is a highly branched molecule (Tharanathan, 2003). Durian, or its scientific name, Durio Zibethinus Murr, is a seasonal fruit grown in South East Asia. Durian is normally eaten fresh (Osman & Zakaria, 2012). In a previous study by Amiza et al., fresh durian seeds contain some small amounts of protein (about 1.6%), but consist largely of carbohydrates (about 46%). Oil or fat content is reported to be roughly 0.5%.

However, wide application of starch film is limited by its mechanical properties and efficient barrier against low polarity compounds (Azeredo, Faria, & Azeredo, 2000; Kester & Fennema, 1986). In order to overcome its shortcoming, Bourtoom and Chinnan (2008) blend rice starch with different chitosan to increase the tensile strength. Chitosan is insoluble in water but soluble in acidic solvents below pH 6. Solubility of chitosan in inorganic acid solvent is quite limited. Chitosan is soluble in 1 % hydrochloric acid but insoluble in sulfuric and phosphoric acids

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(Nadarajah, 2005). A plasticizer is a major component, required to overcome film brittleness and helping to avoid chipping and cracking of films during subsequent handling and storage. The plasticizer reduces intermolecular forces and increase polymeric chains mobility. Moreover, the plasticizer reduces the glass transition temperature of the thermoplastic wheat gluten proteins (Mangata, Baudin, Boutevin, & Gontard, 2001; Matveev, Grinberg, & Tolstoguzov, 2000). As plasticizers polyol plasticizers such as glycerol and sorbitol (Teixeira et al., 2009) have been used. With regard to polyols, glycerol and sorbitol, glycerol appears to give the best results in decreasing the friction between starch molecules (Janssen & Moscicki, 2006).

Literature Review

Bioplastic

Biobased plastics or bioplastics are sustainable, largely biodegradable and biocompatible. They reduce our dependency on depleting fossil fuels and are CO_2 neutral (Pilla, 2011). According to a review study by Flieger et al. (2003), there are three groups of bio- degradable polymers that can be utilized in the produc- tion of bioplastics: biopolymers by chemical synthesis, biopolymers through fermentation process by microor- ganisms, and biopolymers from chemically modified natural products.

Starch

Starch is organized in discrete particles, granules whose size, shape, morphology, composition, and supramolecular structure depend on the botanical source. The diameters of the granules generally range from less than 1 μm to more than 100 μm, and shapes can be regular (e.g., spherical, ovoid, or angular) or quite irregular. Starch granules are par- tially crystalline particles composed mainly of two homopolymers of glucopyranose with different structures: amylose, which is composed of units of D-glucose linked through α–D–(1–4) linkages and amylopectin, the branching polymer of starch, composed of α–D–(1–4)-linked glu- cose segments containing glucose units in α–D (1–6) branches (Tang, Mitsunaga, & Kawamura, 2006).

Starch, which is a biodegradable natural polymer and produced in abundance at low cost is reported to be one of the most promising candidates for fabrication of bioplastics (Huangu, Yu, & Ma, 2004). Numerous studies have been conducted to optimize the properties of starch based bioplastics (Janssen & Moscicki, 2009).

Chitosan

Chitosan is a carbohydrate polymer that can be derived from crustacean seafood wastes such as shells of crabs, shrimps and crawfish. Chitosan has a wide range of applications in diverse fields ranging from medical sutures and seed coatings to dietary supplements and coagulants for waste treatment. Physicochemical properties of chitosans and their functionalities are affected by their sources (Rhazi et al., 2004). Chitosan is soluble in weekly acidic solutions resulting in the formation of a cationic polymer with a high charge density and can therefore form polyelectrolyte complexes with wide range of anionic polymers (Khor & Lim, 2003).

Chitosan relatively low cost, widespread availability from a stable renewable source such as shellfish waste of the sea food industry, along with chitosan's ability to form a good film, are primary reasons to seek new applications of this polymer. Numerous investigations have been reported on the studies of films made from chitosan (Park, Marsh, & Rhim, 2002) and chitosan blends with starch. Zhai et al. (2004) investigated the effect of chitosan on corn starch film properties. The tensile strength and the flexibility of starch film were improved largely after incorporation of 20% chitosan into starch film.

Glycerol

Plasticizers are the most important materials that can increase the flexibility and processibility of thermoplastic starch. Besides, use of plasticizer would lead to change in properties of starch, which is depend on the amount of plasticizer used. For example, glass transition temperature (Tg) of cassava starch is reported as decreased with the increasing amount of glycerol (Bergo, Sobral, & Prison, 2009). Glycerol (1,2,3-propanetriol) is a plain alcohol which used in various industries. Glycerol is a by-product generated in large amounts in the biofuel industry, and is becoming nowadays a waste product that must be disposed of with additional costs (Wang, Jian, Huiying, & Bernard, 2001; Yazdani & Gonzalez, 2007).

Methodology

Starch Extraction of Durian Seeds

Starch that was used in this research of bioplatic was starch that was extracted from durian seeds. Durian seeds was obtained from merchants durian located at Wahid Hasyim and A.H Nasution, Medan, Indonesia. Durian seeds that had been collected were then cleaned and peeled.

Futhermore, durian seeds were cut, cleaned, and dried under the sun. Seeds that had been dried were blended with water at ratio 1 : 5 (w/v), then those were filtered. The filtrate was precipitated for 2 × 24 hours and the sediment

was wet starch.Wet starch was dried by direct sunto obtain the dry starch. Dry starch was refined and sieved with 100 mesh sieve.

Starch durian seed was analyzed the water content, starch content, amylose content, amylopectin content, ash content, fat content, and protein content based on standard SNI-01-2891-1992, SNI-01-3194-1992, Fourier Transform Infra-Red (FTIR), and Scanning Electron Microscope (SEM).

Film Preparation

Durian seed starch were weighed 0,2 w/v from 100 ml aquadest and chitosan were weighed 0,04 m/v from strach solution. Starch solution was made by dissolving starch with distilled water (H_2O). Chitosan solution was also prepared by dissolving chitosan which had been weighed before with a solution of hidrolic acid with concentration variation 1,0% ; 1,1% ; 1,2% ; 1,3% ; and 1,4% (v). Gliserol were weight with concentration 25% (w) from chitosan strach mass. Glass beaker containing starch solution was placed on a magnetic stirrer hot plate while heated. Chitosan solution was then poured into a glass beaker containing starch solution. After fewminutesglycerol added to the starch-chitosan solution, afterwards stirred until heating temperature reached. The solution was heated on constant heating temperature for 15 minutes, the magnetic stirrer was turned off. Glass beaker containing a solution of bioplastic was cooled briefly before printing. The solution was poured into a mold bioplastic acrylic. Bioplastic solution was dried on room temperature for 2 × 24 hours. After the bioplatic was dry, bioplastic was removed from the mold and then stored in a desiccator. Bioplastic was ready to be analyzed.

Characterization of Bioplastic

Mechanical Properties

Test of mechanical properties involves tensile strength and elongation at break. Product of bioplastic chosen and cut forming specimen for tensile strength test based on standard of ASTM D 638.

Fourier Transform Infra Red (FTIR)

Analyzing of FTIR was observed at Research Laboratory, Faculty of Pharmacy, University of Sumatera Utara.

Scanning Electron Microscope (SEM)

Analyzing of SEM was observed at Minerale and Advance Material Laboratory, Faculty of Mathematics and Natural Sciences, University of Negeri Malang.

Density

Analyzing bioplastic density was observed based on standard of ASTM D 792-91 1991.

Results and Discussion

Characterization of Durian Seeds Starch

The yield of starch extracted from the durian seeds is 20.58%, in which the starch in the form of powder and white with the particle size of 100 mesh. The chemical composition of durian seeds starch are presented in Table 1. Durian seeds starch is quite potentially be used as bioplastics due to starch contained in the starch extracted from durian seeds is quite high, that was 86.82%, with the ratio of amylase : amylopectin 36.32 : 50.50. The high water content was decreased starch content relatively (Yulianti & Ginting, 2012). Higher starch water content can effect a shorter starch saving time because it will faster contaminated by microorganism (Setiani, Sudiarti, & Rahmidar, 2013). The presence of protein in durian seed starch caused browning reaction, so that bioplastic from durian seed starch were not cleared (Cornelia, Syarief, Effendi, & Nurtama, 2013). Beside that the presence of fat in durian seed starch has been investigated too. The presence of endogenous lipids in starch may have an ad- verse effect on the swelling of individual granules by repelling the water (Us, Gönenç, & Aytunga Arik Kibar, 2010).

Table 1:
Chemical Composition of Durian Seeds Starch

Component	Amount (%)
Starch	86.82
- Amylose	36.32
- Amylopectin	50.50
Moisture	15.7
Ash	0.13
Fat	0.07
Protein	0.81

Mechanical Properties of Bioplastic

Tensile Strength

The effect of variation Hydrochloric acid concentration as chitosan solvent and heating temperature of bioplastic on the bioplastic tensile strength are shown in fig. 1.

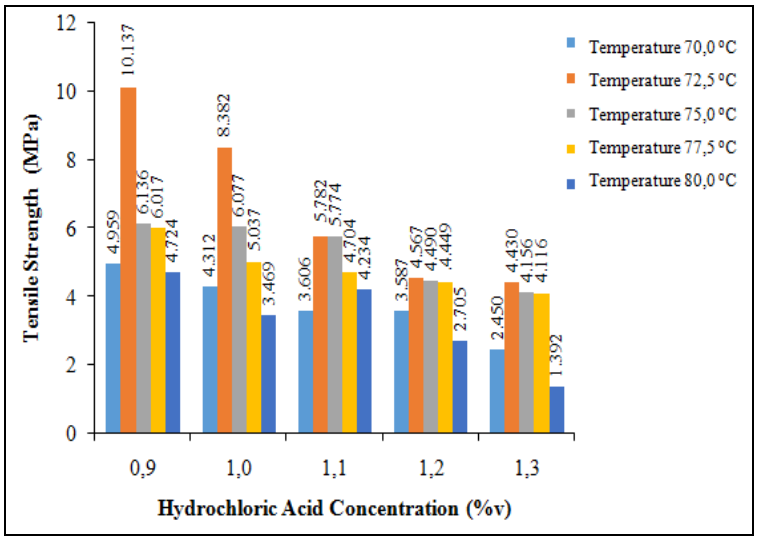

Figure 1: The Effect of Variation Hydrochloric acid Concentration as Chitosan Solvent on Tensile Strength of Bioplastic

From fig. 1 could be seen that variation of Hydrochloric acid concentration as chitosan solvent has effected to the tensile strength of bioplastic. These research using hydrochloric acid as chitosan solvent where the highest tensile strength value of bioplastics at concentration 0.9 %v with heating temperature 72,5 ^{o}C earned the magnitude 10.237 MPa. Chitosan solubility in Hydrochloric acid was decreased as increasing of Hydrochloric acid concentration, it could be seen with decreasing tensile strength value of bioplastic. Chitosan is insoluble in water but soluble in acidic solvents below pH 6. Organic acids such as acetic, formic and lactic acids are used for dissolving chitosan, and the most commonly used solvent is 1% acetic acid solution. Solubility of chitosan in inorganic acid solvent is quite limited. Chitosan is soluble in 1 % hydrochloric acid but insoluble in sulfuric and phosphoric acids (Nadarajah, 2005). The less homogeneous mixing process caused molecule distribution of plastic component were unequal, so that tensile strength of the material were decreased (Buzarovska et al., 2008).

Addition of filler is needed to overcome film deficiency such as film strength. It could increased plastic stiffness and strength, then decreased solubility and tendency to crooked (Widyaningsih, Senny, Dwi Kartika, 2012). Concentration of filler effect to mechanical properties (tensile strength) from composit. Tensile strength depend on strong interface bonding bitween matrix and fillers (Sudi, Irhamni, & Rahmi, 2013).

Elongation at Break

The effect of variation Hydrochloric acid concentration as chitosan solvent and heating temperature of bioplastic elongation at break are shown in fig. 2.

From fig. 2 could be seen that variation of Hydrochloric acid concentration as chitosan solvent has effected to the elongation at break of bioplastic. The elongation at break value of bioplastic was increased along with increasing Hydrochloric acid concentration as chitosan solvent. The highest elongation at break value at Hydrochloric acid concentration 1.3 %v with heating temperature 70 ^{o}C was 22.608 %. Xu et al. (2005) prepared and assessed the starch and chitosan blend films. The film's elongation at break was affected by the chitosan content. According to the results, the mechanical properties of the composite films increased with the addition of chitosan. The changes in mechanical properties of characterized by the plasticizers weakening the intermolecular forces between the chains of adjacent macromolecules, increasing the free volume and causing a reduction of mechanical resistant (Sobral, Menegalli, Hubinger, & Roques, 2001). Plasticizer used in this research was glycerol, it caused flexibility for plastic.

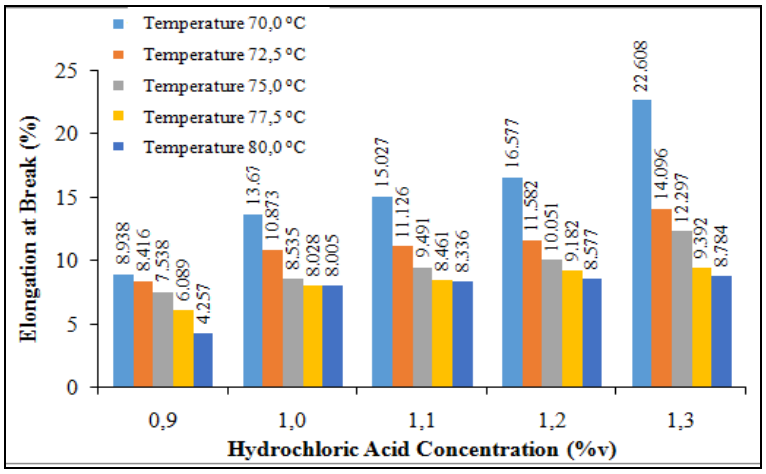

Figure 2: The Effect of Variation Hydrochloric acid Concentration as Chitosan Solvent on Elongation at Break of Bioplastic.

Increasing of chitosan solubility in Hydrochloric acid solution caused decreasing of bioplastic elongation at break value. This thing could be caused by the higher the compactness of intermolecular bonds in the bioplastic because the enhancement of hydrogen bond when adding chitosan, so bioplastic formed becomes stronger and more rigid. The addition of chitosan to bioplastic solution caused the decreasing of bioplastic elongation percent. The more chitosan added caused the decreasing of bioplastic elongation percent (Hartatik & Nuriyah, 2014).

Density

The effect of variation Hydrochloric acid concentration as chitosan solvent and heating temperature of bioplastic density are shown in fig. 3.

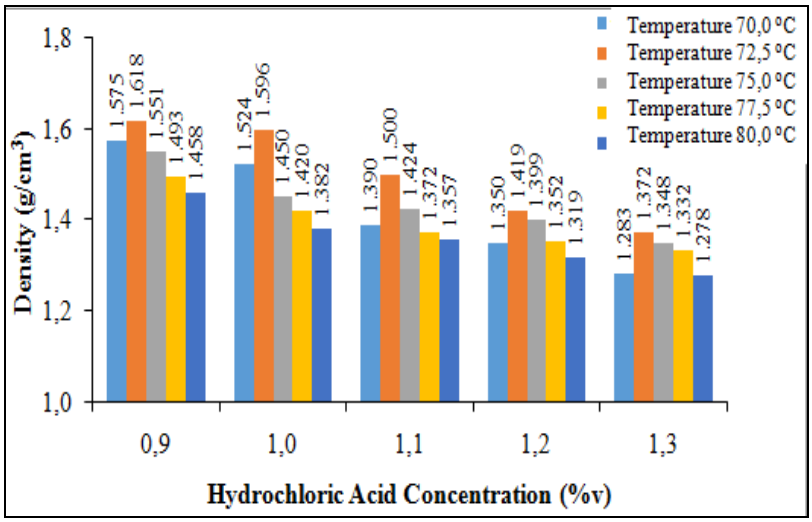

Figure 3: The Effect of Variation Hydrochloric acid Concentration as Chitosan Solvent on Bioplastic Densities

From fig. 3 could be seen that variation of Hydrochloric acid concentration as chitosan solvent has effected to the bioplastic densities. The increasing of Hydrochloric acid concentration as chitosan solvent caused the decreasing of bioplastic densities value. This is caused by decreased of chitosan solubility along with increasing Hydrochloric acid concentration from 0.9 %v to 1.3 %v. The highest density at Hydrochloric acid concentration 0.9 %v and heating temperature 72.5 ^{o}C was 1.618 gr/cm^{3}. At Hydrochloric acid concentration 0.9 %v bioplastic has the highest density value because chitosan has the best solubility at that concentration, so that with addition of chitosan can fill and increase the structure density of bioplastic. The increase in mechanical properties is due to the increase of cross-linking density. The cross-linking agents react with the -OH groups present in starch and make ether linkages with the available hydroxyl groups. This helps to increase the mechanical properties (Das et al., 2010).

The addition of filler and plasticizer produce the higher densities value. Density is proportional with mass (Widyaningsih, Senny, Dwi Kartika, 2012). The higher density make bioplastic mass increase (weigh more) (Melorose, Perroy, & Careas, 2013). These research used durian seeds starch, it has relatively small particle size. The

smaller particle size will form mass with higher density because of decreasing the cavity between particle (Jufri, Dewi, Ridwan, & Firli, 2006).

Fourier Transform Infra Red (FTIR)

The purpose of FTIR analysis is to identify the presence of hydrogen bonds that were formed in bioplastics. Fig. 4 below is the FTIR analysis result of durian seeds starch, chitosan, bioplastic from durian seed starch without chitosan, and bioplastic from durian seed starch with chitosan and glycerol.

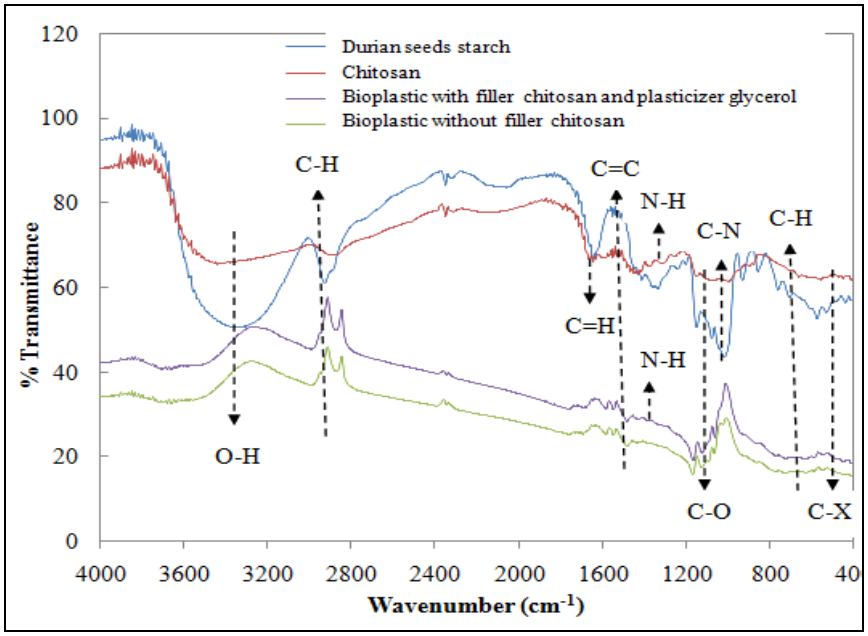

Figure 4: The FTIR Analysis Results of Durian Seeds Starch, Chitosan, Bioplastic From Durian Seeds Starch without Chitosan, And Bioplastic From Durian Seeds Starch with Chitosan And Glycerol

Analyzing of FTIR was observed to identify major functional group. From fig. 3 we could seen the presence of O-H hydrogen bonds, C-H aromatic, C-H alkanes, C-O ethers, and C-N amines group in the durian seeds starch. Main structure of starch is amylose and amylopectin and starch has C-O, O-H, and C-H group (Pascoal et al., 2013; Ratnayake, Hoover, & Warkentin, 2002). Besides that starch also has protein and fat content (Madruga et al., 2014). The existing of C-N group shows the presence of protein in durian seeds starch.

From the analyzing result of FTIR could be seen that chitosan owns simetrical N-H groups, C-H alkenes, C=O, N-H bonds, C-O esters. Chitosan is glucoseamine compound which shows the presence of O-H, NH_2, and C=O amide, and CH_3 group (Suendo, Ahmad, & Valiyaveetiil, 2010). Chitosan is produced by deacetylation of chitin. In the process of deacetylation, acetyl groups from the molecular chain of chitin are removed to form amino groups (Nadarajah, 2005).

From figure 4 could be seen the characteristics of bioplastic without adding filler chitosan and bioplastic with adding chitosan and plasticizer glycerol have the same functional groups yet the behaviour of spectrum on bioplastic without adding filler chitosan and plasticizer glycerol lies under spectrum of bioplastic with adding chitosan and plasticizer glycerol. It have the same functional groups with durian seeds starch as its constituent component. From the result of analyzing FTIR bioplastic with chitosan could be seen the peak exists on the wavelength 1546,91 cm^{-1}. This wavelength shows the existance NH groups (Zhong & Xia, 2008). This thing shows that filler chitosan has been spread in bioplastic film.

Scanning Electron Microscope (SEM)

The purpose of the SEM analysis was to observe the morphology of fracture surface of the bioplastics, whether all components of bioplastics has been mixed homogeneously.

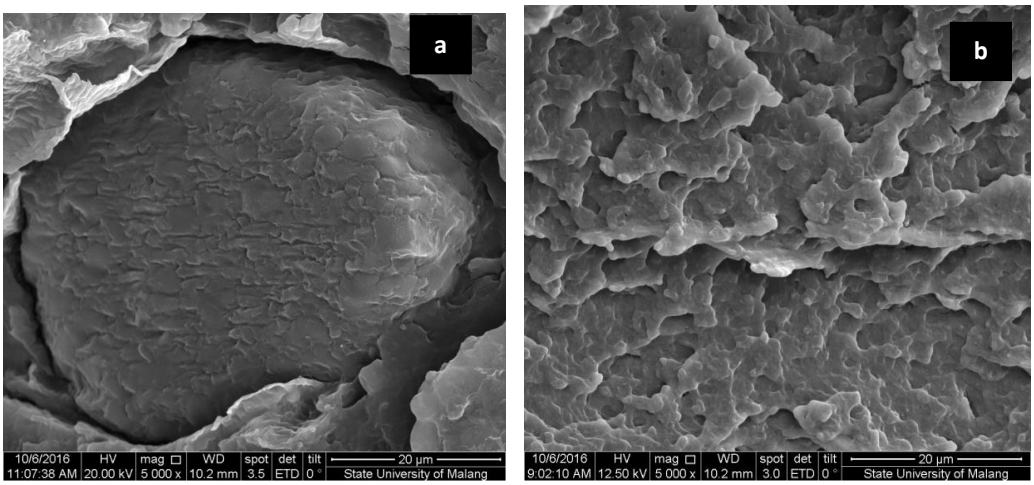

Figure 5: SEM micrographs of fractures: (a) Bioplastics without fillers chitosan; (b) Bioplastics with chitosan filler and plasticizer glycerol

Scanning electron microscopy of fracture of bioplastic samples using a magnification of 5000x are prensented in Fig. 5. When we compared differences in the structure of fracture of bioplastic in Fig. 5 (a) and Figure 5 (b), it can be seen that bioplastics in Fig. 5 (b), bioplastics with filler chitosan and plasticizer glycerol has more dense and compact structure because the chitosan as a filler has been distributed homogeneously and fills empty spaces in the bioplastics film, thus it is increasing the density of bioplastics. From fig. 5 (a) could be seen bioplastic without filler chitosan has cavity in its fracture surface. However both of those figures shows that each fracture surface seem a bit rough and jagged. The jagged surface of the plastic also illustrated the amount of force that was required to break the plastic as the break would have been sudden and drastic (Carvalho et al., 2003). Homogenity structure of bioplastics is one indicator that can indicate improvements in the value of the mechanical strength of the bioplastics (Amanda, Carmen, Fabio, & Laurindo, 2010). This supports the results of this research that an increase in tensile strength of bioplastics is caused by the increasing concentrations of chitosan which has been distributed homogeneously.

Conclusions

Concentration of hydrochloric acid as chitosan solvent affect the tensile strength value of bioplastics. Tensile strength and density value increase along with decreasing of hydrochloric acid from 1,3 %v to 0,9 %v. But it made bioplastics to be compact and rigid, so that the the elongation at break value of bioplastics decreased. From analyzing of FTIR could be seen that functional group of bioplastics has similarity with its constituent components. From analyzing SEM shows that the fracture surface of bioplastic seem a bit rough and jagged.

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