Synthesis and Characterization of Biodegradable Plastic with Basic Materials of "Singkong Onggok" - Pectin Peel of "Jeruk Bali" (Citrus Maxima) – Plasticizer

Laelatun Maghfiroh¹, Kartini Fauziah Hanum², Endaruji Sedyadi¹, Irwan Nugraha¹, Fatchul Anam Nurlaili¹

¹Chemistry Department, ²Chemistry Education Department, Faculty of Science and Technology, UIN Sunan Kalijaga JI. Marsda Adisucipto No. 1 Yogyakarta 55281, Indonesia. Tel. + 62-274-540971, Fax. + 62-274-519739 ²Email: kartinihanum@gmail.com

Abstract. Biodegradable plastic made from pectin pell of "jeruk bali" has been carried out. Pectin is obtained by extraction methods and for the manufacture of biodegradable plastics with hot-blending methods. The characteristics of the mechanical biodegradable plastics are determined by tensile strength and elongation, and biodegradation test of biodegradable plastics. The results of manufacture biodegradable plastic with the addition of pectin 5g was the best result with a tensile strength value of 195.35 MPa, and an elongation value of around 15.73 - 33.40%. Plastics with the variation of sorbitol starch composition as much as 1.5 g with 1 ml sorbitol each having a tensile strength of 61.29 MPa and elongation value of 14.30%. Plastic with a variation of 1.5 ml sorbitol has a tensile strength value of 198.93 MPa and an elongation value of 16.73%. Plastic with a variation of 2 ml sorbitol has a tensile strength value of 79.67 MPa and an elongation value of 17.63%.

Keywords: Biodegradable, Canna Tuber, Pectin, Pell of 'jeruk bali"

INTRODUCTIONS

Plastic is a material used by Indonesia people, because it is flexible and easy to use. But plastic has a bad impact on the environment because it is not easily broken down and takes about 300-500 years to break down completely. This is because plastic made from synthetic polymers. One effort to overcome this problem is to make *biodegradable* plastic. The advantages of *biodegradable* plastic are easily biodegradable and the materials used to make it easily found in nature. Biodegradable plastics can be made from materials derived from nature or living things.

Materials be used that can to make *biodegradable* plastics include protein lipids, carbohydrates, pectin and chitosan compounds, cassava starch, potatoes and corn. Pectin is a polysaccharide biopolymer compound that has the ability to form gels. The content of pectin with a fairly high level is found in "jeruk bali" peel (Citrus maxima), but research making biodegradable plastics with basic ingredients of "jeruk bali" culture has not been widely done, Sutopo (2011). In the manufacture of *biodegradable* plastics made from pectin from peel of "jerk bali", it is necessary to add chitosan and starch to strengthen the layers. In this study, cassava starch was added the manufacture in of biodegradable plastics, while plasticizer was needed to increase the elasticity of the layer.

Plasticizer was added so that *biodegradable* plastic was soft, resistant to water and increased elasticity (Yadaf and Satoskar, 2007). In this study sorbitol *plasticizers were* used. Sorbitol is used

because it is environmentally friendly, is abundant in nature, and is non-toxic and can inhibit evaporation of water in products (Purwanti, 2010). Pectin used in the manufacture of *biodegradable* plastics in this study was obtained by extraction method and for the manufacture of *biodegradable* plastics the hot-blending method was used. The characteristics of the mechanical properties of *biodegradable* plastics were determined by tensile strength and elongation, as well as *biodegredation* tests of *biodegradable* plastics.

MATERIALS AND METHODS

The main ingredients used in the manufacture of *biodegradable* plastics are among others the skin of "jeruk bali" and "singkong ongkok", 96% ethanol solution, 40% citric acid, sorbitol, and aquades.

Procedures

Making Peel of "Jeruk Bali" Powder

Making pell of "jeruk bali" powder is made by drying the peel of "jerk bali" that has been cut and washed first. Drying is carried out for one to two hours until it has a constant weight. After drying, then finely ground like flour and sifted.

Pectin Extraction

Extraction is done by mixing 25 grams of "jeruk bali" peel with 500 ml of distilled water in a 1000 ml cup of glass and stirring until blended to help in the extraction process. Next, 40% citric acid was added to pH 3 and heated at a constant temperature of 80 °C for 60 minutes. Then filtered so that the filtrate in the form of liquid and pulp is obtained. The filtrate is then added with ethanol 96% with a ratio of 1: 2. After the addition of ethanol, a gel and liquid will be obtained which is then filtered to separate the two parts. "jeruk bali" pectin gel obtained is then dried at 50 °C for 5 hours. The results obtained form dried sheets of "jeruk bali" pectin extract. Then pound until smooth and sifted. After pectin powder was obtained, FTIR spectra were analyzed.

Manufacture Starch of "Singkong Onggok"

Making cassava starch starts with crushing cassava cassava and adding water to it produce pulp. Then filtered in stages so that the pulp and filtrate can be separated. The results obtained are then allowed to stand until the starch settles. The results of the sediment are then dried use the oven at 60 $^{\circ}$ C.

Data Analysis

Viscosity Test

The viscosity of cassava starch extract and pell of "jeruk bali" extract (*Citrus maxima*) were carried out by dissolving the sample to a volume of 100 ml. The sample concentration is varied to 0.005; 0.010; 0.015; 0.020; 0.025 M. Then allowed to reach room temperature, then the solution is measured by *brofield viscometer*.

FTIR Function Cluster Analysis

Analysis of FTIR pectin functional groups is done by placing pectin powder that has been made pellet into the *sample holder*, then the sample is *scanned* at 4000-400 cm⁻¹ wave number. The sample in the form of film is placed into the *set holder* on the infrared spectrophotometer so that it can the functional groups were identified and compared with the cassava starch spectra.

Testing of Mechanical Properties

The mechanical properties of *biodegradable* plastics are determined by *tensil strength* and *elongation* based on ASTM. D. 882-02. The sample was tested using a *tensile strength* and *elongation tester*. *Biodegradable* plastic is conditioned at room temperature 30 °C, RH 68% for 24 hours before measurement.

WVTR Test

Testing the water vapor transmission rate using the gravimetric method (ASTM 1983). Pengujia is carried out by means of plastic placed on the mouth of the cup in a circle with a diameter of 7 cm, an outer diameter of 8 cm and a depth of 2 cm which contains silica gel 10 g. The edge of the cup and plastic are covered with wax or insulation. The cup is then put into a jar containing 40% (b / v) NaCl solution. Water vapor diffused through plastic will be absorbed by silica gel and will increase the weight of the silica gel.

Biodegradability Test

Test biodegradability of plastic *biodegradable* done by weighing the *biodegradable* plastic that has been generated is measured to determine the severity of the initial weight. Then the plastic is piled up in the soil and weighed once every 2 days for 10 days.

RESULTS AND DISCUSSION

The part of "jeruk bali" peel that will be extracted is albedo because it has a greater pectin content than the other parts, which is 40-60%. The yield of the extract process of "jeruk bali" pectin extracted was 27.3%. The pectin powder that was produced was then tested using an FTIR spectrophotometer to determine the character of the resulting functional group. The resulting spectrum is presented in figure 1.

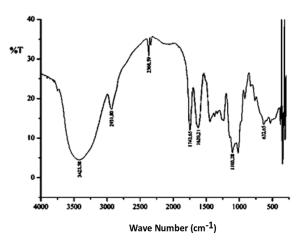


Figure 1. The resulting spectrum extract pell of "jeruk bali".

The results obtained from the graph are widening uptake at wave number 3425.58 cm⁻¹ which indicates the presence of a hydroxyl (-OH) group. At wave number 2931.80 cm⁻¹ there is absorption which indicates the presence of a CH group. The absorption peak at wave number 1743.65 cm⁻¹ shows the presence of C = O carbonyl group and at wave number 1103.28 cm⁻¹ there is an absorption which indicates the presence of a secondary alcohol group. Identification of uptake at several wave number points shows that the groups are functional groups found in pure pectin compounds.

The next *step* after extracting the "jeruk bali" peel is the viscosity test carried out using the *brofieldfield viscometer* instrument. Based on the viscosity test obtained the molecular weight of "jeruk bali" extract extract sample results of 88,206,535 g / mol. The value of pure molecular weight of pectin is around 50,000 g / mol (Mutimmah et al. 2012) so that it can be concluded that the extracted compound is pectin.

The starch used as the base material for making plastic in this study was obtained from cassava onggok (pulp), and obtained through extraction methods. The results obtained in this stage were then tested by FTIR to determine the group of compounds contained therein. The spectrum of the "singkong ongkok" extract test results is shown in figure 2.

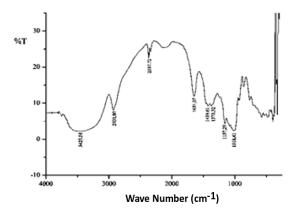


Figure 2. The spectrum results of the "singkong ongkok" extract.

From the spectrum there are visible absorptionabsorption at several wave number points. There is a wide absorption at wave number 3425.58 cm⁻¹ which indicates the presence of *stretching* hydroxyl (-OH) groups . Coupled with the overlap of the wave number 2931.80 cm⁻¹ which indicates the presence of CH groups and is amplified by the presence of absorption around 1373.32 cm⁻¹ which indicates the presence of CH ₂ groups . Uptake around the wave number 1018.41 cm⁻¹ shows the presence of an ether (CO) group, and uptake at 1651.07 cm⁻¹ indicates the presence of – OH *bending* group .

The resulting "singkong ongkok" extract compound then carried out a viscosity test to determine the molecular weight of cassava starch compounds produced. Based on the results of the viscosity test obtained the molecular weight of cassava extract samples of 15,426.67 g / mol with a pure starch molecular weight ranging from 10,000-50,000 g / mol (Mutimmah et al. 2012). From the two tests above, it can be said that the compounds produced are starch.

Biodegradable plastic with starch-sorbitol composition with the best mechanical properties used in subsequent studies. The plastic is added with pectin with the aim of increasing the quality of the plastic produced. The method for making plastic with the addition of pectin is the same as before, using the *hotblending* method (Sedyadi et al., 2016).

From the previous data obtained *biodegradable* plastic which has maximum tensile strength is plastic with a composition of starch 1.5 grams and 1.5 ml sorbitol. The data is used to be varied with pectin to find out how the effect of pectin the value tensile strength on of possessed by *biodegradable* plastics. The results of the tensile strength test on *biodegradable* plastic with variations in pectin can be presented in the graph in figure 3.

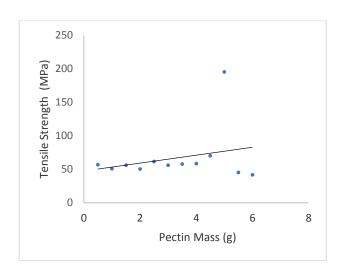


Figure 3. The results of the tensile strength test on *biodegradable* plastic.

While the results of the *biodegradable* plastic elongation test with variations in pectin are presented in graphical form in figure 4.

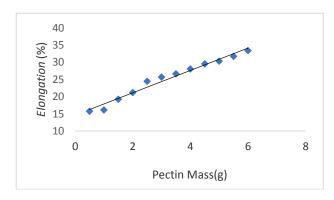


Figure 4. The results of the biodegradable plastic elongation test.

The thickness of biodegradable plastic with the variation of pectin produced can be presented in the graph in Picture 5. The graph shows the thickness of biodegradable plastic increases with increasing mass of pectin added.

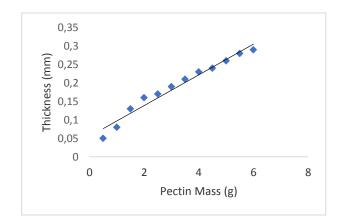


Figure 5. Grafik of thickness Plastik *Biodegradable* Variasi Pektin.

The resulting plastic was then identified as a compound group using an FTIR spectrophotometer. The resulting spectrum is presented in figure 6

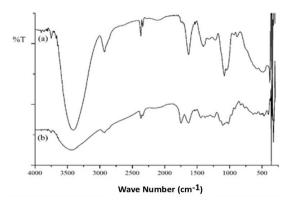


Figure 6. Spektrum FTIR Plastik Biodegradable (a) with pectin (b) with out pectin.

The spectrum in picture 6. shows the absorption pattern of biodegradable plastic with the addition of pectin having an absorption pattern that widens around the wave number 3000 cm⁻¹. The uptake that shows the presence of the -OH group is slightly different from the pattern of absorption patterns in biodegradable plastics without pectin. Significant differences in patterns are possible because of differences in the composition of the materials used in making plastics, thus affecting the number of hydrogen bonds contained in the compounds formed so that the formed absorption patterns are different.

WVTR *biodegradable* plastic test results with variations of pectin are presented in the graph in picture 7. WVTR value on *biodegradable* plastic with а composition of 1.5 grams of starch, 1.5 ml sorbitol and the addition of 5 grams of pectin raises the amount of WVTR value possessed by *biodegradable* plastics with a composition 1.5 gram starch and 1.5 ml sorbitol because pectin has the same hydrophilic properties as starch and sorbitol. Whereas when compared with *biodegradable* plastic with sorbitol 2 ml composition the *WVTR* value of plastic with the addition of lower pectin.

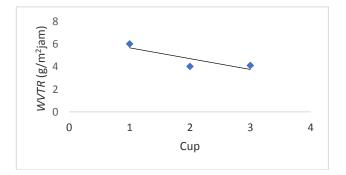


Figure 7. WVTR.

The biodegradation test carried out on plastic with a variety of pectin was carried out by the same method. *Biodegradable* plastic with variation of pectin with the best tensile strength which is then tested for *biodegradation*. The *biodegradation* test results are presented in the table.

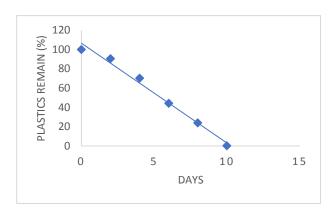


Figure 8. Rate of degradation after added pectin.

From the data listed on the table. showed that the degradation rate experienced by plastic with the addition of pectin increased slightly. Plastics with the addition of pectin decompose faster than plastic without pectin.

CONCLUSIONS

The addition of pectin increases the biodegradation rate of *biodegradable* plastic on the ground. Though not so significant, but there is a rise in the rate of biodegradation of the *biodegradable* plastics with the addition of pectin produced compared with *biodegradable* plastics without the addition of pectin.

REFERENCES

- Agra, I. B. Warnijati, S., dan Pujianto. 1973. Hidrolisa Pati Ketela Rambat Pada Suhu Lebih Dari 100°C. Forum Teknik. Vol 3. 115-129.
- Bruice, P. Y. 2001. *Organic Chemistry*. Prentice Hall International, Inc., New Jersey.
- Cervera, M.F., Heinamaki, J., Krogars, K., and Jorgensen, A.C., 2005. Solid-State and Mechanical Properties of Aqueous Chitosan-Amylose Starch Films Plasticized With Polyols. AAPS PharmSciTech. Vol 5. 15-20.
- Dann, S. E. 2000. Reaction an characterization of solids, royal society of chemistry UK.
- Kertesz, Z. I., 1951. *The Pectin Substances*. New York. Interscience Publisher Inc.
- Kim, J, choi. H-J Sohn, T. Kang. 1999. J. Electrochem. Soc., 146, 4401.
- Niyomdharn, C. 1992. *Citrus moximo (BurrL) Merr*. Di dalam: Verheij EWM. Coronel RE (ed). Edibie Fruits and Nuts. Bogor: Prosea. 128-132.
- Othmer, Kirk. 1967. Encyclopedia of Chemical Technolog, Edisi 2, Vol 14. John Willey and Sons, Inc. New York.

Pantastico. 1997. Fisiologi dan Teknologi Pasca Panen. UGM Press. Yogyakarta.

- Pearson, David. 1977. The Chemical Analysis of Foods, 6th ed. Chemical Publishing Company Inc. New York.
- Pigman, W. W. 1946. Advance in carbohydrat Chemistry, Vol 2. London Academic Press.
- Platt, David K. 1984. *Biodegradable Polymers*. United Kingdom, New York. Smithers Raphra Limited.
- Purwanti, Ani. 2010. Analisis Kuat Tarik dan Elongasi Plastik Kitosan Terplantasi Sorbitol. Jurnal Teknologi. Vol 2(3). 99-106.
- Rusmadiyanto. 2010. Pembuatan bioplastik dari tepung iles-iles (Amorphophallus onchophyllus) dengan plasticizer gliserol. Skirpsi. Yogyakarta. Universitas Gajah Mada 7-14
- Sastrohamidjojo, Hardjono. 2007. Spektroskopi. Liberty. Yogyakarta
- Sedyadi, E., Aini, S. K., Anggraini, D., Ekawati, D. P. 2016. Starch-Glycerol Based Edible Film and Effect of Rosella

(Hibiscus Sabdariffa Linn) Extract and Surimi Dumbo Catfish (Clarias gariepinus) Addition on Its Mechanical Properties. *Biology, Medicine, & Natural Product Chemistry.* **5(2)**: 33-40

- Suppakul, P., 2006. Plasticizer and Reaktive Humidity Effects in Mechanical Properties of Cassava Flour Films. Skripsi. Bangkok, Thailand. Department of Packaging Technology, Faculty of Agro-Industry, Kasetsart University.
- Sutopo, 2011. Penangan Panen dan Pasca Panen Buah Jeruk. Batu. Kpri Citrus.
- Wirawan, Sang Kompiang., Agus Prasetya, dan Ernie. 2012. Pengaruh Plasticizer pada Karakteristik Edible Film dari Pektin. *Reaktor*. Vol 14(1). 61-67.
- Yadaf, G. D dan D. V. Satoskar. 1997. Kinetics of epoxidation of Alkyl Esters of Undecyclenic Acid Comparison of Traditional Rout vs Ishii-Venturello Chemistry. JAOCS. Vol 74(4). 397-407.

THIS PAGE INTENTIONALLY LEFT BLANK