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Pertanika J. Sci. & Technol. 27 (3): 1441 - 1450 (2019) Improved Mechanical and Thermal Properties of Modified Thermoplastic Starch (TPS) from Sago by using Chitosan Rozanna Dewi1*, Nasrun1, Zulnazi1, Medyan Riza2 and Harry Agusnar3 Department of Chemical Engineering, Universitas Malikussaleh, Lhokseumawe, Aceh, Indonesia Department of Chemical Engineering, Universitas Syiah Kuala, Banda Aceh, Aceh, Indonesia Department of Chemistry, Science Faculty, Universitas Sumatera Utara, Medan, Indonesia ABSTRACT The use of starch as bioplastic has been generally examined; however, the quality should be improved before it can substitute the commercial plastic.

Sago is one of starch sources that can be used for this reason and is accessible in impressive sums in Indonesia. Past exploration to combine an adjusted sago starch thermoplastics (TPS) in-situ by reacting plasticized starch with diphenylmethane diisocyanate (MDI) and castor oil; at the same time to create polyurethane prepolymer (PUP) in more homogeneous stage and littler size has been successfully done. At this stage, chitosan of 0.5, 1, 1.5, 2 and 2.5 gram was included into the compound and went about as filler to improve the mechanical and warm properties of TPS.

The chitosan was first diluted into acetic acid derivation corrosive and blended thoroughly with the starch and PUP. Sorbitol was included as plasticizer. The modified TPS-chitosan was then characterized mechanically, thermally, and biodegradability.

ARTICLE INFO __ Article history: Received: 30 June 2018 Accepted: 23 January 2019 Published: 24 July 2019 __ E-mail addresses: rozanna.dewi@gmail.com (Rozanna Dewi) nasrun_ibrahim@yahoo.com (Nasrun) zoelnazri@yahoo.co.id (Zulnazri) medyan.riza@gmail.com (Medyan Riza) harryagusnar@yahoo.com (Harry Agusnar) * Corresponding author _The results of this study showed that the ideal mechanical properties of adjusted TPS were acquired with an expansion of 1 gram chitosan; tensile strength and elongation were 200.04 MPa and 24.96 % respectively.Thermal Gravimetric Analysis indicated that adjusted TPS-chitosan had a decent thermal steadiness and must be corrupted at high temperature of 534?.

DSC result indicated that changed TPS-chitosan had a high softening purpose of 385.41? and ?H152.61 J/g. This value is higher than melting point of TPS alone which is 104.69?. Modified TPS-chitosan

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Rozanna Dewi, Nasrun, Zulnazi, Medyan Riza and Harry Agusnar was degraded earlier than TPS which was 19 days in nature compared to 2-4 months.

Overall, mechanical and thermal properties of modified TPS are improved with the addition of chitosan. Keywords: Biodegradability, chitosan, mechanical properties, Modified Thermoplastic Starch (TPS), sago, thermal characteristics INTRODUCTION Essential material used to produce plastic is polypropylene and polyethylene. The waste produced is very high and therefore increases the cost to recycle the material.

Reused plastic despite everything has a lot of contention related with the degree of safety and health impact to the user. The guideline by Head of Drug and Indonesian Food Control Body Regulation on Food Packaging Materials No. HK 00.05.55.6497 as for food packaging material dated on August 20, 2007 has entered into force in August 2008 which disallow the utilization of reused plastics (Pudjiastuti and Listyarini, 2012).

Biodegradable plastics industry will shape into a significant industry later on with the goal that it is basic to convey plastic materials that have prevalent attributes and can be debased commonly. Starch has been commonly utilized as crude material for biodegradable plastic in view of its biodegradability, renewability and availability in huge quantity.

However, plastic orchestrated from starch can't match the conventional plastic in term of its mechanical and warm properties. Hence, the bioplastic ought to be changed to improve its mechanical and warm properties. Wu et al. (2008), integrated adjusted thermoplastic starch (TPS) utilizing corn starch with polyurethane prepolymer (PUP) from diphenylmethane diisocyanates (MDI) and polyols got from castor oil. This change produces littler scope particles fillers (PUP).

Assurance of nature can be acknowledged when the polyol supplanted with inexhaustible materials, for instance, vegetable oil (Lu et al., 2005). Among numerous kinds of plant oils, castor oil has three hydroxyl social affairs, subsequently is a better than average chance to coordinate poliuretan due to its ability to improve the mechanical properties or toughness against water (Ferrer et al., 2008) Rozanna et al.

(2014) had synthesized modified thermoplastic sago starch (TPS) through in-situ mechanism by reacting sago starch with MDI and castor oil simultaneously, resulting the formation of more homogenous and better size polyurethane prepolymer (PUP), just as the adjustment response that occured not just on the outside of the PUP particles yet in addition in the mass stage.

The uprightness of this investigation separated from in-situ process which gave more ideal situation than the standard method was sago starch. as a crude material of TPS which is still not being maximally utilized in Indonesia notwithstanding of its high creation. The capacity of sago starch has changed of late from principle food in the eastern of Indonesia into a cultivation grain.

The mechanical and warm properties of altered TPS is better than bioplastic; be that as it may, it is as yet not serious contrasted with traditional plastic. 1442 Pertanika J. Sci. & Technol. 27 (3): 1441 - 1450 (2019)

Improved Properties of Modified Thermoplastic Starch (TPS) Through this research, the mechanical and warm properties of adjusted TPS are being improved by including chitosan into the blend.

Chitosan has been known as a material that can improve the mechanical properties of plastic and can lessen its capacity to assimilate water. The adjusted TPS-chitosan is relied upon to have a serious mechanical property like the customary one just as zero waste to the earth. MATERIALS AND METHODS Materials Sago starch, Castor oil, 4, 4'-methylenedi-p-phenyl diisocyanate (MDI), chitosan, acetic acid (2%), and sorbitol plasticizer were used without any further pretreatment.

Synthesis of Modified TPS-Chitosan Chitosan with five unique loads as referenced in the Table 1 were weakened with acidic corrosive (2%) and included into a 15.5g of sago in 500ml Erlenmeyer jar. The blend was warmed and mixed at a temperature of 70°C for around 30 minutes to frame a gel. Subsequently, castor oil and MDI were poured directly into the sago gelatinization to frame the polyurethane prepolymer (PUP).

The blend was mixed overwhelmingly for a couple of moments and sorbitol was included as plasticizer. The blend was then imprinted on a sheet of glass and dried at encompassing temperature for 24 hours. Sago starch plastic (PS) and PUP-chitosan were orchestrated for examination. Table 1 Composition in five samples of modified TPS - chitosan No.

_Sample _Sago starch (g) _Chitosan (g) _MDI (ml) _Castor oil (g) _Sorbitol (g) _ _1 _TPS - Chitosan 1 _15.5 _0.5 _1 _2 _7 _ _2 _TPS - Chitosan 2 _15.5 _1 _2 _3 _7 _ _3 _TPS - Chitosan 3 _15.5 _1.5 _3 _4 _7 _ _4 _TPS - Chitosan 4 _15.5 _2 _4 _5 _7 _ _5 _TPS - Chitosan 5 _15.5 _2.5 _5 _6 _7 _ _ Analysis Conducted A tensile strength test was performed to test the mechanical properties of the modified TPS-chitosan.

Whereas the thermal characteristic, stability and biodegradability rate of the modified TPS-chitosan were measured by using a Thermal Gravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC). Pertanika J. Sci. & Technol. 27 (3): 1441 - 1450 (2019) 1443

Rozanna Dewi, Nasrun, Zulnazi, Medyan Riza and Harry Agusnar RESULTS AND DISCUSSIONS Mechanical Properties Tensile strength is the estimation of the force required to pull something to where it breaks.

The tensile strength for the altered TPS-chitosan was performed by utilizing Universal Testing Machines Electronic System reliant on the standard ASTM D638, 1991. The rigidity for each example is recorded in Table 2. Table 2 Tensile strength for each sample No _Sample _Tensile strength _ _Elongation (%) _ _ _ _(Kgf/mm2) _(MPa) _ _ _1 _TPS - Chitosan 1 _13.30 _130.41 _15.08 _ _2 _TPS - Chitosan 2 _20.40 _200.04 _24.96 _ _3 _TPS - Chitosan 3 _8.50 _83.35 _21.32 _ _4 _TPS - Chitosan 4 _9.40 _92.17 _19.76 _ _5 _TPS - Chitosan 5 _8.50 _83.35 _11.84 _ _6 _Bioplastic sago + Sorbitol _0.26 _0.03 _57.00 _ _7 _PUP + Chitosan _15.20 _149.05 _13.40 _ _ Figure 1.

Tensile strength for various TPS - Chitosan sample As showed in Figure 1, the expansion of 0.5 g and 1 g of chitosan has expanded the elasticity to 13.30 kgf/mm2 (130.41Mpa) and 20.40 kgf/mm2 (200.04 Mpa) individually. Conversely, the expansion of 1.5g, 2.0g and 2.5g of chitosan has diminished the elasticity to 8.50 kgf/mm2 (83.35 Mpa), 9.40 kgf/mm2 (92.17 Mpa), and 8.50 kgf/mm2 (83.35 Mpa) individually.

The tensile strength of changed TPS - chitosan got for every one of the five examples have figured out how to surpass the Moderate Class Plastic mechanical properties of 1-10 MPa. Past exploration led by Rozanna et al. (2014) additionally uncovered that the rigidity for TPS without chitosan was just 0.40-0.47 kgf/mm2, while the tensile strength of unadulterated PUP was 1.72 kgf/mm2.

This is because the higher the concentration of chitosan, the more hydrogen bonds formed in the TPS-chitosan. 1444 Pertanika J. Sci. & Technol. 27 (3): 1441 - 1450 (2019)

Improved Properties of Modified Thermoplastic Starch (TPS) Thusly, chemical bond becomes more grounded and hard to break as it expects vitality to break the bond. Be that as it may, this simply occurs inside a scope of centralization of chitosan. For this situation, the expansion of 0.5

g and 1g of chitosan has expanded the tensile strengt, while expansion of 1.5 g, 2.0 g and 2.5 g of chitosan has decreased the tensile strengt. Along these lines, the best conditions for TPS-chitosan printing happen on the expansion of chitosan 0.5 g and 1 g. The outcome got was like to research done by Hartatik. Hartatik et al.

(2014) considered that the expansion of chitosan of 1% and 2% into cassava starch expanded the estimation of elasticity of bioplastics, while the expansion of chitosan of 3% and 5% diminished it (Hartatik et al., 2014). The more chitosan included, the tensile strength of bioplastics will diminishes further as the bioplactics become delicate (Hartatik et al., 2014).

Tensile strength of TPS-chitosan was likewise affected by PUP responded in-situ from MDI and castor oil. It had likewise prompted an expanded adaptability of adjusted TPS-chitosan because of the nearness of cross connecting between the PUP and the starch grid which hence had expanded the sub-atomic load of the starch and prolongation of altered TPS.

This is on the grounds that the castor oil included as materials for PUP goes about as an effect modifier that expanded the stretching and brought down the elasticity properties. Therefore, the tensile strength properties of altered TPS sheet was impacted by the reasonable increment in sub-atomic weight and starch substance of castor oil (Wu et al., 2008). Information in Table 2 show that the rigidity of bioplastic comprised of sago just is lower (0.26 kgf/mm2) than bioplastic made up from the mix of TPS-chitosan (15.20 kgf/mm2). Additonally, the plasticizer added had additionally added to the expanded flexibility.

Figure 2. Elongation for various TPS-chitosan samples Elongation demonstrates a flexibility/elasticity of a film by measuring the maximum length it can reach before breaks. The elongation of TPS-chitosan was influenced by the amount of chitosan added as shown in Figure 2. Higher concentration of chitosan will Pertanika J. Sci. & Technol.

Rozanna Dewi, Nasrun, Zulnazi, Medyan Riza and Harry Agusnar reduce the elongation of TPS-chitosan due to the lower distance of its intermolecular bond. Elongation of all TPS-chitosan sample has fulfilled the moderate properties group which was 10-20%. The moderate properties group in plastic refer to the plastic with medium elongation capability.

Elongation of a plastic is determined by its application, thus it has to be adjusted accordingly. Previous research done for TPS (15.5 g) without chitosan has shown that the elongation was 15-19.5 % while for PS (Plastic starch made from sago and plasticizer only) the elongation was 10 %. Polyol derived from castor oil used to form PUP has improved the flexibility of TPS (Rozanna et al., 2014). Research done by Wu et al.

(2008) on corn TPS had shown that PUP would improved the tensile and elongation of TPS at certain level; however, after optimum figure, the tensile strength would decrease (Wu et al., 2008). Thermal Characteristics Melting Point. Melting point is the temperature at which a substance change from solid to liquid state. The thermal stability analysis help to determine if a specific plastic is good for packaging and can survive a certain temperature depends on its durability.

Differential Scanning Calorimetry (DSC) test was performed for TPS – Chitosan 1 and TPS – Chitosan 2 to test its thermal stability. Figure 3. DSC for modified TPS-chitosan 1 

Improved Properties of Modified Thermoplastic Starch (TPS) Figure 4. DSC for modified TPS-chitosan 2 Figure 3 and 4 above show the DSC test result for modified TPS - Chitosan 1 and TPS – Chitosan 2. The melting point was 363.93oC and 385.41oC; and the latent heat of fusion (?H) was 87.64 J/g and 152. 61 J/g, respectively.

The result shows an increase in chitosan concentration has increased the melting point. Melting point of modified TPS - Chitosan mixture was higher than in TPS only was due to cross link formed between TPS and chitosan. Addition of chitosan into the mixture had improved the melting point. The higher melting point showed that more cross links occurred and the higher the tensile strength.

Rozanna et al. (2014) found that melting point of PUP was 105.55oC and ?H was 224.38 J/g, while for TPS only, its melting point was 104.69oC and ?H was 234.27 J/g. Another means to determine thermal characteristic of TPS-chitosan was through Thermal Gravimetric Analysis (TGA). Figure 5 below shows the result of TGA performed on TPS – Chitosan 1.

TPS-chitosan 1 started to lose weight at 6th minute and 165oC, and was fully decomposed at 24th minute and 534oC. Drastic weight lost took place at 11th minute and 271oC until 16th minute and 375oC. The result for TPS-chitosan 1 and 2 was almost similar. Previous study done by Rozanna et al. (2014) showed that the PUP began to decompose at temperature of 300 - 500oC.

Meanwhile, TPS started to decompose at 150 – 200oC in small quantities, continued with weight loss steeply and completely discharged Pertanika J. Sci. & Technol. 27 (3): 1441 - 1450 (2019) 1447

Rozanna Dewi, Nasrun, Zulnazi, Medyan Riza and Harry Agusnar at temperature of 500oC. Modified TPS-chitosan lost weight faster than PUP at the first transition because starch had a lower thermal stability compared to PUP.

Meanwhile, TPS- chitosan showed higher temperature to decompose compared to TPS only. Figure 5. TGA for modified TPS-chitosan 1 Biodegradability As shown in Figure 6, biodegradability rate of TPS-chitosan at various composition was affected by composition of MDI and chitosan. High amount of MDI and chitosan in the mixture has led to lower rate of biodegradability.

Starch is a natural polymer that can be easily degraded in nature, while chitosan has slower rate of degradation. Rate of biodegradability of polymer depends on its structure. Since crystallinity of chitosan was higher than starch, hence higher concentration of chitosan in the mixture reduced TPS- chitosan rate of biodegradability.

A hundred percent weight loss occurred in 19 days for all TPS-chitosan, except TPS-chitosan 4 and 5. The biodegradability rate was higher compared to the previous research, where TPS was degraded at 10-16 weeks depending on PUP concentration (Rozanna et al., 2014). This was due to weather condition where the samples was buried in a rainy season; therefore, the soil moisture has increased. Hartatik et al.

(2014) also revealed a similar finding that after 15 days buried almost 75% of bioplastic had degraded from cassava-chitosan. This shows that higher amount of chitosan in the mixture reduces the 1448 Pertanika J. Sci. & Technol. 27 (3): 1441 - 1450 (2019)

Improved Properties of Modified Thermoplastic Starch (TPS) biodegradability rate.

Biodegradability of cassava-chitosan was almost fully degraded in 45 days (Lazuardi & Cahyaningrum, 2013). Figure 6. Biodegradability rate of various TPS-chitosan CONCLUSION Addition of chitosan has improved the mechanical and thermal properties of modified sago starch thermoplastics (TPS). The optimum concentration of addition of 1 g chitosan has resulted in increased tensile strength (200.04 MPa) and improved elongation (24.96%).

The melting point of modified TPS - Chitosan (385.41oC and ?H is 152.61 J/g) was higher than TPS (104.69oC and ?H is 234.27 J/g). A modified TPS-chitosan also has a good thermal stability up to 534oC and will be degraded in nature in 19 days. ACKNOWLEDGEMENT Authors would like to thank their departments for all the support provided in the smooth conduct of their research. REFERENCES Ferrer, M. C. C.,

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