Characterization of Sago Starch Based Degradable Plastic with Calcium Carbonate ($CaCO_3$) as Filler

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***Received**: 15 November 2023; **Accepted:** 29 November 2023; **Published:** 5 December 2023*

Abstract: Research on finding substitute to plastic commercial has received massive attentions due to the environmental effect of plastic waste. Degradable plastic can be used as an alternate to synthetic plastic even though the properties especially mechanical characteristic. Sources of degradable plastic can be starch, cellulose, poly lactic acid, etc. Starch available in large quantities, cheap and renewable. The purpose of this study was to determine the effect of Calcium Carbonate ($CaCO_3$) filler on characteristics of sago based degradable plastic. The degradable plastic properties analyzed were mechanical, chemical, thermal, water absorption and degradation rate. The preparation of degradable plastics was done in several stages, starting with the preparation of sago starch, synthesis of degradable plastic and characterization. Variations of $CaCO_3$ composition and sorbitol plasticizer were used to observe their effect towards plastic properties. $CaCO_3$ filler variations used were 2, 4, 6, 8% and sorbitol plasticizer variations were 25, 30, 35%. The highest tensile strength, Young's Modulus and elongation at break obtained were 6.24 MPa, 89.92 MPa and 154.80% respectively, at 0.8% calcium carbonate and 35% sorbitol. Fourier Transform Infra Red (FTIR) test results showed in thermoplastic starch from sago there were more free -OH hydroxyl groups due to the reduction of atoms that are hydrogen bonded. The absorption peaks in the range of wave numbers 2931.80 cm-1 indicated the presence of saturated aliphatic hydrocarbon chains (C-H), wave numbers of 1411.89 cm-1, 1334.74 cm-1, 1207.44 cm-1, 1149.57 cm-1, and 1078.81 cm^{-1}. It showed typical areas of C-O groups. Most of the compounds were hydrophilic which binds water, hence can be degraded by microbial activity in the soil. Thermal characterization using Differential Scanning Calorimetry (DSC) thermogram test indicated degraded plastic has a thermogram peak at 137.25°C. This peak indicates physical changes due to the loss of water groups content in plastic. The highest swelling value was 103.96 % obtained at 2% calcium carbonate and 35% sorbitol. The addition of $CaCO_3$ filler improved the water resistance properties of degradable plastics. The degradation of sago starch-based plastics with $CaCO_3$ filler was 16-24 days depending on the filler composition and has complied with ASTM D-20.96 (degradable plastics should decompose before 180 days).

Keywords: degradable plastic; sago starch; calcium carbonate; filler; characteristic

Introduction

Plastic pollution has emerged as a major threat to human and natural ecosystems. According to a United Nations report, the world is producing around 430 million tonnes of plastics every year of which two-thirds are for short-term use. The production and consumption of plastic products have grown exponentially since the 1950s. A 2022 report by the Organisation for Economic Co-operation and Development (OECD), titled ‘Global Plastics Outlook: Policy Scenarios to 2060’, states that if the present trends persist global plastic production will triple and exceed 1 billion tonnes by the year 2060.

Type of commercial plastic used today produce waste and are harmful to health and environment because they cannot be decomposed naturally for thousands of years. The issue of recycled plastics is still controversial due to its safety and health effects and high processing costs. Currently, the world needs a solution to the growing problem of plastic waste. Conventional plastics such as PET take about 23 to 48 years to break down in the environment (Robert Adhitama et al., 2023). Methods of dealing with plastic waste are recycling, incineration, and burial. However, burning plastic waste can produce toxic substances and it is harmful to human and environment. Meanwhile, bury plastic waste is ineffective because it is difficult to degrade and takes thousand years (Denny Akbar Tanjung et al., 2022).

One possible solution is to develop plastic products using materials that easily decomposed by bacteria such as degradable plastics. Some of basic materials to be used such as starch, cellulose, poly lactid acid, etc. Starch has the properties of biodegradability, low cost, renewable, and abundance, so starch is considered a "green line" as a raw material to produce porous substances such as aerogels, biofoams, and bioplastics (K. J. Falua et al., 2022). In its granular form, starch is mostly composed of linear amylose and highly branched amylopectin. Thus, starch can be considered as a crystalline material (Nafchi et al, 2013). Thermoplastic starch (TPS) is a gelatinized starch-based material. Gelatinization aimed to destroy the crystalline structure in starch granules and involves granular swelling, native crystalline melting and molecular solubilization. These materials can be reheated or melted and form new shapes without significant properties changes, as the result of the absence of chemical crosslink in these polymers (Jiang et all, 2020). Plastics synthesized from starch normally containing small amounts of water, hence its often brittle.

Sago contains starch, fiber, protein, fat, and ash. Sago starch contains cellulose as a macromolecular material (E. R. M. Saleh et al., 2022). Sago starch quality standards in Indonesia are listed in the Indonesian National Standard (SNI 3729: 2008), with composition of moisture content, ash content, crude fiber, amylose content, amylopectin content and starch content (Maryam et al., 2022). One of the disadvantages of starch-based degradable plastics is that their mechanical properties still not comparable to conventional plastics, therefore technology is needed to improve their performance (R. Alebooyeh et al., 2012). Calcium Carbonate ($CaCO_3$) fillers have ideal properties and have been used in various polymer applications. $CaCO_3$ is widely used in thermoplastics, thermosets and elastomers. Its main use is in elastomers and PVC (Rothon R and Paynter, C., 2016).

J´essica D.C. Santos et al., 2023 conducted experiments on the consolidation of calcium carbonate as a filler for LDPE-based plastics resulting in stiff polymer properties. In their study, the addition of $CaCO_3$ at concentrations of 30%, 45% and 50% significantly increased Young's Modulus from 221 MPa and 569 MPa to 542 MPa; elongation at break from 29.0% and 14.1% to 7.2% and 5.8%; and 19.6 MPa and 20.5 MPa to 15.3 MPa and 14.2 MPa, respectively.

In this study, Calcium Carbonate ($CaCO_3$) is used as filler in sago starch-based degradable plastic to improve degradable comparison of the effect of $CaCO_3$ filler addition on the characteristics of degradable plastics derived from sago starch. The $CaCO_3$ concentrations used were 0.2, 0.4, 0.6, 0.8% and sorbitol plasticizer variations were 25, 30, 35% of starch weight.

Different concentrations and plasticizer variations was used to determine the best characteristics of the degradable plastic produced in terms of mechanical properties, thermal properties, functional groups, water resistance and degradation rate.

Methodology

The research method consists of several stages, extracting starch from sago, synthesizing degradable plastics and characterization. The equipment used in this research include blender, volumetric pipette, stirrer, oven, thermometer, beaker glass, hot plate, Erlenmeyer, digital scale, plastomile flask, magnetic stirrer, sieve, and mold. The materials used in this research are sago starch, calcium carbonate ($CaCO_3$), distilled water, and sorbitol. All material purchased from Merck and used without any further purification.

Degradable Plastic Synthesis

Extraction of sago starch was done by chopping the sago stems and then washing them thoroughly, mashed using a blender and added water. After becoming sago porridge, filtered using a cloth and the filtrate is deposited for 24 hours. The precipitate was separated from the water part and then dried using an oven until it became starch. Next, the process of synthesizing degradable plastic with the addition of $CaCO_3$ filler. Sago starch was weighed 10 grams and put into a beaker glass, added 200 ml of distilled water. Then, heated and stirred at 100 rpm until cooked to becomes a gel at a gelatinization temperature of 70 °C, about 30 minutes. Sorbitol (25, 30, 35% of starch weight) and $CaCO_3$ with various doses (0.2, 0.4, 0.6, 0.8% of starch weight) were added. Molding used was glass casting and dried in the oven for 24 hours at 70°C. Plastic was taken out and cooled at room temperature.

Study on Effect CaCO3 as filler to Degradable Plastic

Sago starch contains cellulose as a macromolecular material (E. R. M Saleh *et al.*, 2022). Sago starch-based degradable plastic is an environmentally friendly product. Since sago starch is derived from natural materials, it is completely safe. Amylose is found in sago starch, and quickly binds with other substances. When heated to 70 - 120 degrees Celsius, sago starch will become gelatinized (Pratik Dilip Patil, 2022). This starch has the potential to be used as a raw material for synthesizing bioplastics. The mechanical properties of starch-based degradable plastics are currently still low. Poor mechanical properties and higher hydrophilicity are some of the characteristics of Thermoplastic Starch/TPS that make it unsuitable for various applications, such as food packaging. For this reason, several modifications have been explored to modify its properties. Incorporation of reinforcing agents, chemical modification and blending with other polymers are the main ways to improve the optimal characteristics or minimize the drawbacks of starch-based materials.

One of the strategies to increase the mechanical strength of degradable plastics is the addition of $CaCO_3$ filler. The addition of $CaCO_3$ also helps accelerate the biodegradability process of degradable plastics. This study used $CaCO_3$ filler to see the comparison of the effect of mechanical properties, thermal properties, water resistance and optimal biodegradability on sago starch degradable plastics. Fillers are required when synthesizing bioplastics to improve stiffness, strength and hygroscopicity. Calcium carbonate is an important mineral and has many functions for plastics, and there is no doubt that it will continue to be one of the main filling materials used by the polymer industry. Edi Syafri et al., 2017 studied the effect of calcium carbonate on the physical, mechanical properties of cassava starch-based bioplastics showing an increase in tensile strength from 1.65 MPa to 3.38 MPa with the addition of 4% $CaCO_3$ (w/w). The modulus of elasticity increased from 174.61 MPa to 645.15 MPa with the addition of 4% $CaCO_3$. The addition of 4% w/w $CaCO_3$ significantly affected the strain properties of the bioplastic composite, the strain decreased from 53.14% to 39.91%.

Degradable Plastic Characterization

Mechanical characteristics of sago starch degradable plastic with $CaCO_3$ filler were carried out by tensile test, elongation and Young's Modulus test. The tensile strength test used the ASTM D-638 (American Standard Testing and Material) standard. Analysis of tensile strength and elongation was carried out using a Mechanical Universal Testing Machine. Functional group analysis was carried out with Fourier Transformation Infra-Red (FTIR) to determine the functional groups contained in the degradable plastic produced. Testing the thermal properties of degradable plastics was done by Differential Scanning Calorimetry (DSC) analysis to determine the energy absorbed. Plastic resistance to water was done by swelling test (water absorption) and microbial decomposition rate analysis was done by burial in the soil.

Result and Discussion

Mechanical Properties Analysis

Measurement of mechanical properties, consist of tensile strength, elongation and young's modulus, was carried out using a texture analyzer. The tensile strength value is inversely proportional to the elongation value and the young modulus is proportional to the elongation value. The tensile strength test results of sago starch-based degradable plastics with variations of $CaCO_3$ filler of 0.2, 0.4, 0.6, 0.8% and sorbitol plasticizer 35% can be seen in Table 1.

Table 1. Mechanical properties of degradable plastics

Sorbitol (%)	$CaCO_3$ (%)	Tensile Strength (MPa)	Elongation at Break (%)	Modulus Young (MPa)
35	0.2	4.72	60.35	76.68
35	0.4	4.56	154.80	29.02
35	0.6	5.44	84.65	62.76
35	0.8	6.24	68.55	89.92

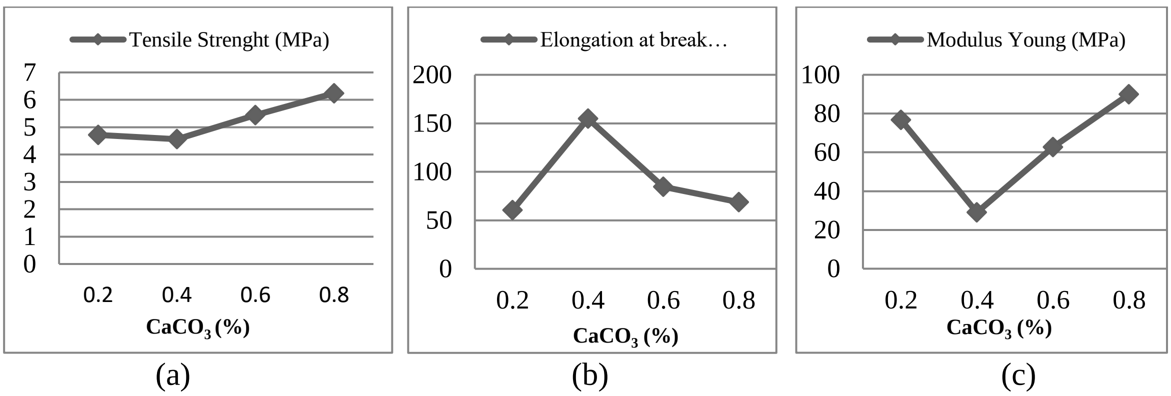

Figure 1. Effect of $CaCO_3$ filler percentage on mechanical properties of degradable plastics: (a) Tensile strength (MPa), (b) Elongation at break (%), and (c) Modulus Young (MPa)

Figure 1 (a) showed the tensile strength value of sago starch-based degradable plastic with $CaCO_3$ variation obtained in this study ranged from 4.56-6.24 MPa. The addition of $CaCO_3$ filler used affects the mechanical properties of degradable plastics. The tensile strength value criteria for the Moderate Properties group is 10-100 MPa. Meanwhile, according to SNI standards (Indonesia Standard for bioplastic), the tensile strength for plastics is 24.7-302 MPa. The tensile strength values in this study are not comparable to the tensile strength obtained by LDPE (10-12 MPa), PET (55-79 MPa), and PP (100-600 MPa) synthetic plastics (Yuliasih I et al., 2019).

From Figure 1 (b), it can be seen that the highest elongation value was obtained at 0.4% of $CaCO_3$ filler concentration of 154.80%. The SNI value for bioplastic elongation is 21-220%. The elongation value obtained in this study is in accordance with SNI standards. The elongation value obtained was also comparable to the elongation value of PET (15-165%) (Nor Izaida Ibrahim et al., 2021). In Figure 1 (c), the elastic modulus value of sago starch-based degradable plastic using $CaCO_3$ filler is 29.02-89.92 MPa. The greater the modulus of elasticity, the more rigid the material will be and not easily deformed. The type of filler used can affect the elastic modulus of bioplastics. Lailatin Nuriyah et al., 2019 studied the effect of $CaCO_3$ addition on the mechanical properties of cassava starch-based bioplastics with glycerol as a plasticizer. The characteristics of the mechanical properties studied were tensile and elongation tests. The tensile test results showed that the tensile strength was obtained at 0.4% $CaCO_3$ as of 22.88 ± 1.46 MPa. While the addition of 0.5 to 1.0% $CaCO_3$ decreased the tensile strength. Elongation value obtained with the addition of 0.8% $CaCO_3$ was 27.57 ± 0.14%.

Chemical Characterization Analysis

To identify chemical bonds in organic materials, polymers, metals and various materials, FTIR can be used. FTIR on degradable plastic samples with 0.6% $CaCO_3$ filler and 35% sorbitol can be seen in Figure 2.

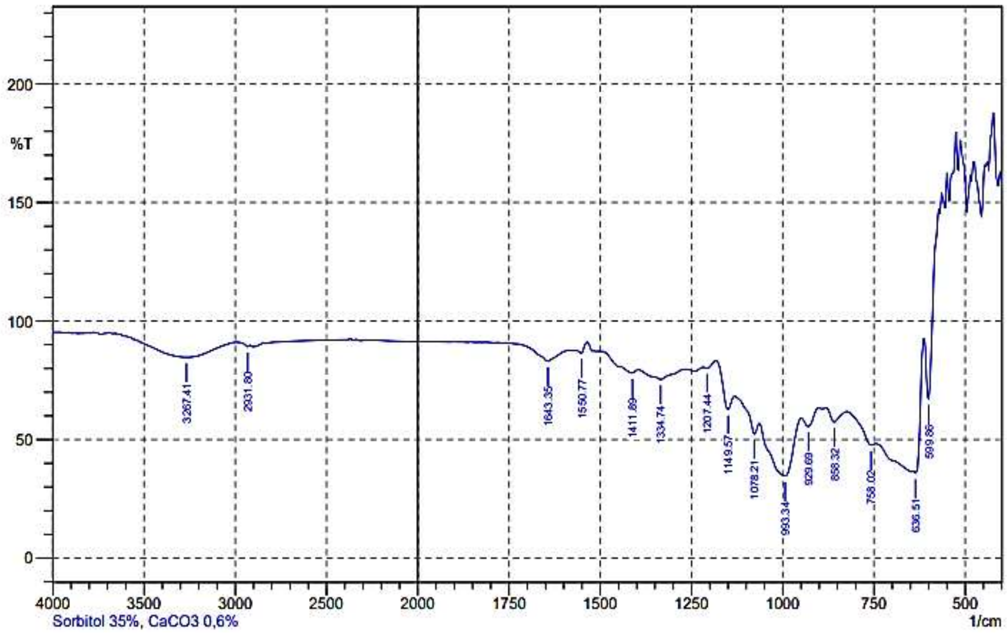

Figure 2. FTIR results of degradable plastic

The degradable plastic contains hydroxyl group (O-H) which is at wave number 3267.41 cm^{-1}. This shows that in thermoplastic starch from sago there are more free -OH hydroxyl groups due to the reduction of atoms that are hydrogen bonded. The absorption peaks in the range of wave numbers 2931.80 cm^{-1} indicated the presence of saturated aliphatic hydrocarbon chains (C-H), wave numbers of 1411.89 cm^{-1}, 1334.74 cm^{-1}, 1207.44 cm^{-1}, 1149.57 cm^{-1}, and 1078.81 cm^{-1} showed typical areas of C-O groups with wave number frequencies of 1000 cm^{-1} - 1450 cm^{-1}. Based on the interpretation of the FTIR spectra of the resulting degradable plastics, there were no changes in new functional groups, shifts in some wave numbers and changes in the intensity of the absorption bands, which indicated the interactions that occur between starch polymers, sorbitol, and $CaCO_3$.

Rajesh Jesudoss Hynes Navasingh et al., 2023 reported that the wave numbers 250 and 3550 cm^{-1} represent the O-H stretching of the alcohol group in starch-based bioplastics with the addition of additives such as glycerol, sorbitol and $CaCO_3$ filler. These peaks indicate that the sample contains glycerol, which has hydroxyl groups. C-O-H groups can be seen at the level where between 1624 cm^{-1} and 1759 cm^{-1} move from one to another. The wave number peaks between 2883 cm^{-1} and 3000 cm^{-1} indicated the presence of C-H groups. The inclusion of starch has led to the presence of these groups. This is also because the sample has been plasticized with glycerol. Tan et al., 2022 stated that the hydrogen bond between the OH group of starch and the COOH group of Cellulose Nano Crystals (CNC) has a high tensile strength value and has a low elongation value.

Thermal characterization Analysis

One of the indicators of plastic quality is its properties or resistance to heat, which can be analyzed by DSC. The basic principle underlying the DSC analysis technique is that when a sample undergoes a physical transformation such as a phase transition, a change in heat will be required to flow from the reference and sample to maintain both at the same temperature. Whether less or more heat is required to flow to the sample depends on whether the process is exothermic (heat release) or endothermic (heat absorption). In this study, DSC was conducted to determine how much energy was absorbed by the degradable plastic. The test results on degradable plastic using 0.6% $CaCO_3$ filler and 35% sorbitol can be seen in Figure 3.

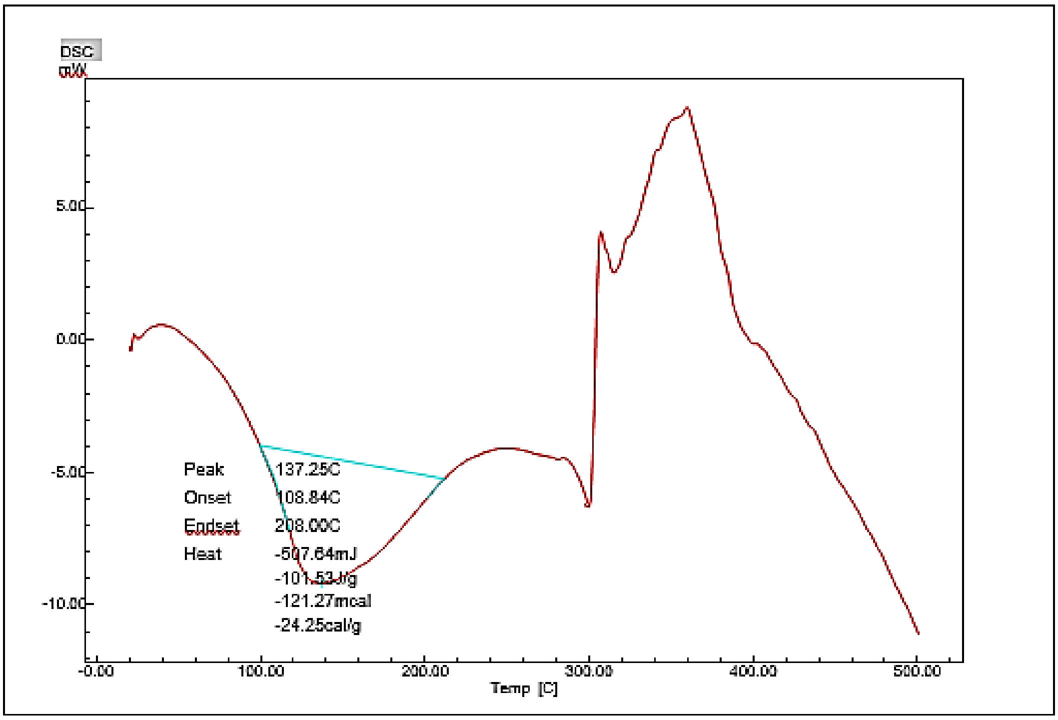

Figure 3. DSC results of degradable plastic

The thermogram of the degradable plastic showed some artifacts at 137.25°C exhibit a sharp thermogram peak. This peak shows physical changes, because the loss of water groups that are still present in the plastic. It was known that water begins to evaporate at 100°C. This dehydration is related to the loss of H_2O molecules from the sample that are physically bound to its surface. The addition of filler to the matrix has increased the melting point of the degradable plastic which proves that cross-linking has occurred. Higher the melting point lead to more cross-links occurred. The melting point was influenced by the hydrogen bonds contained in the plastic. DSC analysis pointed degradable plastic have good thermal characteristics and stability. The more hydrogen bonds in a plastic, the higher its melting point will be. This is because the energy required to break the bonds will also be greater (Oneesha, H, P et al., 2021).

Water Absorption Analysis

The water resistance of degradable plastics was determined by the swelling test, which is the percentage of degradable plastic development due to the presence of water. Analysis of the swelling percent was done to determine the amount of liquid absorbed until degradable plastic expands. Table 2 showed the results of the water resistance test of degradable plastics with variations of $CaCO_3$ filler of 0.2, 0.4, 0.6, 0.8%) and variations of sorbitol of 25, 30, 35%).

Table 2. Results of water resistance test on sago starch degradable plastic by swelling test

Comparison		Swelling Analysis		
Sorbitol (%)	$CaCO_3$ (%)	Start Weight (gr)	Final Weight (gr)	Swelling (%)
25	0.2	0.0599	0.1159	93.48915
	0.4	0.0603	0.1130	87.39635
	0.6	0.0582	0.1077	85.05155
	0.8	0.0564	0.1033	83.15603
30	0.2	0.0554	0.1083	95.48736
	0.4	0.0549	0.1039	89.25319
	0.6	0.0802	0.1464	82.54364
	0.8	0.0761	0.1399	83.83706
35	0.2	0.0757	0.1544	103.96301
	0.4	0.0608	0.1213	99.50658
	0.6	0.0601	0.1146	90.68219
	0.8	0.0568	0.1063	87.14789

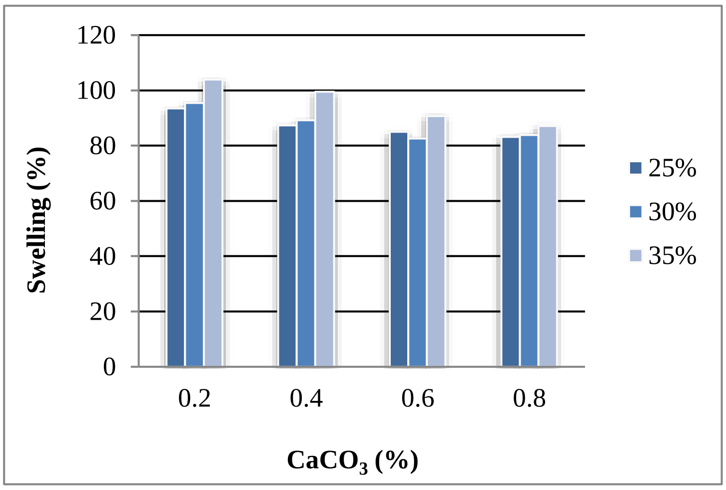

Figure 4. Water resistance analysis of degradable plastic

The most important properties for biodegradable materials are water absorption capacity and degradability. Microorganisms tend to grow and consume materials as an energy source when water is absorbed in the material (H. Ismail and N.F. Zaaba., 2014). Figure 4 pointed that the swelling value obtained of degradable plastics with $CaCO_3$ filler and 25% sorbitol plasticizer were 83.15603 - 93.48915%; while 30% sorbitol were 82.54364 - 95.48736%, and 35% sorbitol were 87.14789 - 103.96301%. The swelling value obtained from the different plasticizers tends to be high. The use of $CaCO_3$ filler concentration greatly influenced the water resistance properties of degradable plastics. The highest swelling percentage was obtained at 0.2% filler from all ranges of sorbitol plasticizer variations used. According to Nur Nadia Nasir and Siti Amira Othman, 2021, the highly hydrophilic nature of starch and its high sensitivity to moisture causes water to easily move into the amorphous region of starch during immersion. The use of plasticizers also has an effect on the water resistance of degradable plastics. Nur Diyana et al., 2021 used $CaCO_3$ filler and glycerol in cassava-based bioplastics. Through water absorption test, the highest water absorption rate was found at 40% glycerol addition of 28.64 ± 0.16% while the lowest absorption rate at 20% was 24.49 ± 0.21%. The water absorption rate increased when the concentration of glycerol in the degradable plastic increased. This was due to the hydrophilic properties of glycerol and starch. These properties increase the affinity between glycerol and water, thus increasing water absorption. According to H. Ismail and N.F. Zaaba., 2014 in their research that the lower water absorption capacity of modified sago starch plastic films contributes to the increase in film hydrophobicity where the hydroxyl groups in starch molecules are replaced by phosphate groups so as to increase water resistance.

Biodegradability Rate Analysis with Soil Burial Method

The biodegradability test aimed to determine the degradation rate of degradable plastics. The biodegradability rate is done by burial test in the soil and attacked by microbes. The results of the biodegradability test of degradable plastic with variations of $CaCO_3$ filler of 0.2, 0.4, 0.6, 0.8% and sorbitol variations of 25, 30, 35%) can be seen in Table 3 and Figure 5 below.

Table 3. Biodegradability test results on sago starch degradable plastics by burial in soil

Sorbitol (%)	$CaCO_3$ (%)	% Biodegradability Day					
		4	8	12	16	20	24
25	0.2	0	16.224	57.959	83.503	100	100
	0.4	0	15.503	64.361	82.084	100	100
	0.6	0	10.208	23.624	60.882	84.362	100
	0.8	0	4.874	27.301	64.428	84.236	100
30	0.2	13.589	55.206	77.939	100	100	100
	0.4	8.589	32.902	60.316	88.214	100	100
	0.6	0	19.024	39.182	79.473	92.065	100
	0.8	0	10.180	28.908	70.276	85.066	100
35	0.2	18.605	39.808	68.831	85.036	100	100
	0.4	11.925	38.139	61.413	86.221	100	100
	0.6	0	22.181	42.897	69.283	90.623	100
	0.8	0	16.256	40.762	68.769	88.429	100

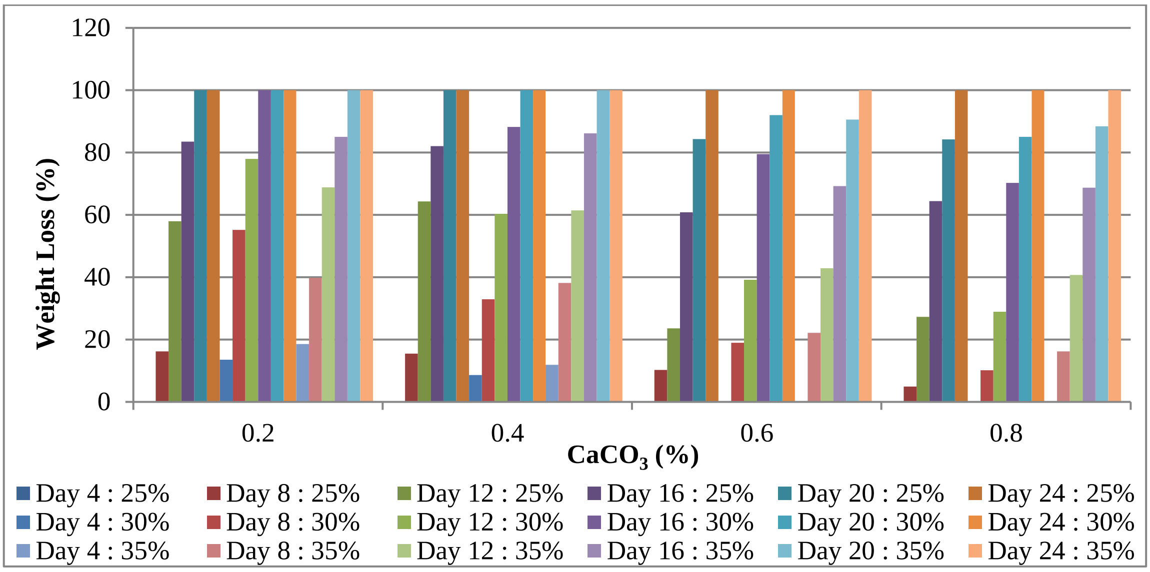

Figure 5. Biodegradability rate analysis of degradable plastic

Microorganisms such as bacteria and fungi in the soil cause degradation of degradable plastics after burial. Samples should be buried in soil of known weight for different time intervals, depending on the material and application of the plastic product used. This study uses sago starch as a base material with the addition of $CaCO_3$ filler (0.2, 0.4, 0.6, 0.8%) using sorbitol variations at (25, 30, 35%) to see the effect of the resulting degradation rate. From Table 3 and Figure 5, it can be seen that the results of the biodegradation of sago starch-based degradable plastics using $CaCO_3$ and sorbitol with certain variations decomposed completely within 16-24 days in the soil. Sago starch-based plastics with the addition of 0.2% $CaCO_3$ filler and 30% sorbitol tend to decompose faster on day 16. The use of $CaCO_3$ filler affects the level of biodegradation, the more $CaCO_3$ filler used, the longer the plastic degradation time. The use of plasticizer variations used in this study tends to affect the speed of plastic degradation.

Biodegradable plastic standards (ASTM D-6002: Guide for Assessing the Compostability of Environmentally Degradable Plastics and ASTM D-20.96 on Environmentally Degradable Plastic), which states for products consisting of a single polymer (homopolymer or random copolymer), 60% of the organic carbon must be converted to carbon dioxide by the end of the test period which is a maximum of 180 days. Therefore, the decomposition duration of this degradable plastic has met the standard. Research conducted by Mrithula Shanmathy et al., 2021 on the use of bentonite fillers into taro starch-based bioplastics affects the degradation rate. The results of the degradation rate obtained were more bentonite concentration added has caused slower degradation rate of bioplastics. Wouter Post et al., 2021 using mineral fillers called talc and calcium carbonate in biodegradable polymer composites and the result showed type of filler affects overall biodegradability characteristics of the composite.

Conclusions

The mechanical properties of sago starch-based degradable plastics with the addition of CaCO3 as filler have poor tensile strength, elongation and young modulus, with tensile strength test of 4.56-6.24 MPa and not yet in accordance with SNI for bioplastics. Compound cluster contained in the FTIR analysis of sago starch degradable plastic with $CaCO_3$ filler has organic groups, thus it is hydrophilic and has O-H, C-H and C=O groups. Thermal characteristics by DSC thermogram test showed degradable plastic had a thermogram peak at 137.25°C. This peak shows physical changes, caused by the loss of water groups were still present in the plastic. The swelling value obtained in sago starch degradable plastic with variation of $CaCO_3$ filler (0.2, 0.4, 0.6, 0.8%) and sorbitol plasticizer 25% were 83.15603 - 93.48915%; sorbitol 30% were 82.54364 - 95.48736%, while sorbitol 35% were 87.14789 - 103.96301%. The degradation rate of plastic degradable using variations of $CaCO_3$ filler and sorbitol plasticizer took place to decompose completely at 16-24 days and was in accordance with ASTM D-20.96 for degradable plastics. The higher the percent of $CaCO_3$ filler added to the degradable plastic, the longer the degradation time in the soil will be.

Acknowledgements

This research was done at the Chemical Engineering Laboratory, Faculty of Engineering, Malikussaleh University, Aceh. This research was funded by PNBP Malikussaleh University Year 2023 and DRTPM Ministry of Education, Culture, Research and Technology in 2022.

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