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Research Paper

The Effects of Adding $CaCO_3$ and Glycerol as Filler in The Synthesis of Bioplastic Made from Corn Starch

Pengaruh Penambahan $CaCO_3$ dan Gliserol Sebagai Pengisi Pada Sintesis Bioplastik Berbahan Dasar Tepung Jagung

Ajeng Listiani Safira[a], Nurullia Suffah[a], Tri Yuni Hendrawati[b], Erna Astuti*[a]

[a]*Department of Chemical Engineering, Faculty of Industrial Technology, Universitas Ahmad Dahlan, Jl. Jend. Ahmad Yani, Tamanan, Banguntapan, Bantul, Yogyakarta, 55191, Indonesia*

[b]*Department of Chemical Engineering, Faculty of Engineering, Universitas Muhammadiyah Jakarta, Jl. Cempaka Putih Tengah 27, Jakarta, 10510, Indonesia*

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ABSTRACT: Plastic is one of the items widely used by the world's population, including in industry. Apart from being cheaper, plastic also has the advantage of being waterproof and having good strength to withstand the load carried. However, plastic has weaknesses in the decomposition process and can cause environmental pollution. Corn has a high percentage of starch content and is easy to cultivate in Indonesia, so corn has the potential to become one of the raw materials used in making bioplastics to reduce the plastic waste produced. This research aims to determine the effect of adding $CaCO_3$ and glycerol fillers in synthesizing bioplastics from corn starch. The method applied in this research is pouring the solution. Based on research, the reaction to make bioplastic from corn starch with $CaCO_3$ filler and glycerol is an esterification reaction. The higher the $CaCO_3$ content in bioplastics, the smaller the percent elongation value produced, the tensile strength properties will decrease, and this will result in the %w loss value decreasing. The low tensile strength is due to the presence of empty holes in the bioplastic product. Meanwhile, more glycerol will cause an increase in the percent elongation value, a decrease in tensile strength, and easy degradation. The bioplastic that has specification close to SNI standards was obtained using 5 ml of glycerol and 2 grams of $CaCO_3$ for 100 g of corn starch.

Keywords: Bioplastics; $CaCO_3$; Filler; Glycerol; Corn starch

ABSTRAK: *Plastik merupakan salah satu barang yang banyak digunakan oleh penduduk dunia, termasuk di industri. Selain lebih murah, plastik juga mempunyai keunggulan karena tahan air dan memiliki kekuatan yang baik dalam menahan beban yang dibawa. Namun plastik memiliki kelemahan dalam proses penguraiannya dan dapat menyebabkan pencemaran lingkungan. Jagung memiliki persentase kandungan pati yang tinggi dan mudah dibudidayakan di Indonesia, sehingga jagung berpotensi menjadi salah satu bahan baku pembuatan bioplastik untuk mengurangi sampah plastik yang dihasilkan. Penelitian ini bertujuan untuk mengetahui pengaruh penambahan bahan pengisi $CaCO_3$ dan gliserol dalam sintesis bioplastik dari pati jagung. Metode yang diterapkan dalam penelitian ini adalah penuangan larutan larutan. Berdasarkan penelitian, diketahui bahwa Reaksi pembuatan bioplastik dari pati jagung dengan bahan pengisi $CaCO_3$ dan gliserol merupakan reaksi esterifikasi. Semakin tinggi kandungan $CaCO_3$ pada bioplastik maka nilai persen elongasi yang dihasilkan akan semakin kecil, maka sifat kekuatan tarik akan semakin menurun, dan hal ini akan mengakibatkan nilai %w loss semakin menurun. Rendahnya kekuatan tarik bioplastik disebabkan adanya lubang-lubang kosong pada produk bioplastik. Sedangkan gliserol yang semakin banyak akan menyebabkan nilai persen elongasi meningkat, kekuatan tarik menurun, dan mudah terdegradasi.Bioplastik yang diproduksi dan mendekati standar SNI diperoleh dengan menggunakan 5 ml gliserol dan 2 gram $CaCO_3$ untuk 100 gram tepung maizena.*

Kata Kunci*: Bioplastik; $CaCO_3$; Filler; Gliserol; Tepung jagung*

1. INTRODUCTION

As the human population on earth increases, new concerns emerge, namely the problem of plastic waste. Indonesia has a large population, based on data from Biro Pusat Statistik [1], namely around 269.603 million people in 2020. This population increased by around 2.7 million people in 2019. Plastic is widely used in various industries. The increase in demand for plastic is due to plastic being cheaper, lighter than other

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Department of Chemical Engineering
Faculty of Industrial Technology
Universitas Muslim Indonesia, Makassar
Address
Jalan Urip Sumohardjo km. 05 (Kampus 2 UMI) Makassar- Sulawesi Selatan
e-mail : jcpe@umi.ac.id

Corresponding Author *
erna.astuti@che.uad.ac.id

materials such as metal and ceramic, water-resistant, and more robust. The weakness of plastic is that its slow decomposition is non-renewable and causes environmental pollution. According to National Waste Management Information System data, in 2021, plastic waste will contribute to 52.72% of the total waste composition in Indonesia. Ratnawati [2] states that plastic waste can decompose entirely within 100 years. Harunsyah [3] stated that plastic waste pollutes the environment due to its persistence in being decomposed by microorganisms. Processing plastic waste using burning and landfilling methods is not environmentally friendly [4].

The solution to the shortage of plastics is to use biodegradable polymers as the main ingredient in making plastics, thereby producing bioplastics. One biodegradable polymer that can be used is cellulose, a commonly used natural polymer. Biodegradable plastic is a type of plastic that can decompose in nature in a short time, so it is friendly to the environment [5]. *Biodegradable plastic* is generally defined as packaging that can undergo a recycling process and be degraded organically. The chemical structure of biodegradable plastics can vary [6]. Bioplastics are plastics made from organic components that decompose with the help of microorganisms. Bioplastics can degrade and decompose into CO_2, water, and biomass [7]. According to Kamsiati [8], environmentally friendly or biodegradable plastic has been developed in the same way as conventional plastic. Essential components such as sago [9][10], corn cobs [11], and cassava starch [12][13] is used to make biodegradable plastic. The production method for biodegradable plastic is primary but has many features, such as plastic packaging commonly used in the Indonesian industry, namely LPDE, HDPE, and PP. Therefore, plastic will be safer and less environmentally harmful [14].

Bioplastics can be produced from natural components found in animals, such as proteins and lipids, as well as from natural components, such as starch, cellulose, collagen, and casein [15]. In making bioplastics, sorbitol, starch, and chitosan are used as plasticizers, matrices, and preservatives [16]. Chitosan is generally applied as part of a starch combination in the production of biodegradable plastics to improve the mechanical quality of the final product. Chitosan, a biopolymer, is made from crustacean waste [17]. Bioplastics made from corn and rice starches show properties comparable to commercially available packaging materials [18]. According to Sen et al. [19], Corn starch and cotton fiber are hydrophilic and have good water absorption capacity and biodegradation characteristics.

Starch is more flexible and can be mixed with various polymer materials for various applications. Various physical or chemical modifications such as derivation, graft copolymerization, and blending have been investigated to improve the characteristics of starch bioplastics. The most effective method to increase bioplastic strength is fillers [20]. Starch has a semi-crystalline granular structure that is heterogeneous. *Starch is* a biodegradable polymer that can replace existing synthetic polymers. Several sources of starch, namely potatoes, corn, rice, wheat, beans, and soybeans, can be used in making bioplastics. There are two types of polymers in starch: glucose, amylose, and amylopectin. Starch can be obtained by extracting plants from plants using the wet milling method [21].

This research uses raw materials in the form of starch to make bioplastics. Starch can work as a material that can be used as an effective packaging agent. When it undergoes modification, a filler is formed, producing adequate mechanical properties with higher tensile strength, flexibility, and elongation. Bioplastic from starch is made by adding several mixtures, namely plasticizers with other materials, using genetic or chemical modification [22]. Glycerol [13] and sorbitol [23][10] are examples of plasticizers. According to Kumoro and Purbasari [24], excessive use of glycerol as a plasticizer allows the formation of starch structures. The addition of glycerol will increase the elasticity of the resulting polymer. [23] stated that increasing the plasticizer content in biodegradable films will increase flexibility and make them less brittle.

This research studied the effect of adding $CaCO_3$ and glycerol fillers on the synthesis of bioplastics from corn starch. $CaCO_3$ is the filler chosen and used in the research. $CaCO_3$ can increase the thermal stability and mechanical properties of biocomposites. Apart from that, $CaCO_3$ is also able to increase flexural strength and tensile strength as well as hot deformation [25]. The bioplastic characteristics analyzed are the functional groups contained in the bioplastic, tensile strength, elongation, and biodegradation.

2. RESEARCH METHODOLOGY

2.1 Materials

The ingredients used are corn starch with Maizenaku brand; chitosan, glycerol, and $CaCO_3$ from Alfa Kimia Shop; acetic acid, 95% alcohol, and distilled water.

2.2 procedures

There are two variations in this research, namely the addition of glycerol and the addition of $CaCO_3$.

Table 1. Bioplastic variables

No	Variable	Amount
1	$CaCO_3$	1 gr; 2gr; 3 gr 4 gr; 5 gr
2	Glycerol	4 ml; 5 ml; 6 ml; 7 ml; 8 ml

Making bioplastics begins by dissolving 10 grams of corn starch in 70 ml of 2% acetic acid and stirring using a magnetic stirrer at a temperature of 65°C-70°C. The gelatinization process will take around 20-25 minutes at a temperature of 30°C. The following is the process of dissolving 1 gram of chitosan with 80 ml of 2% acetic acid by stirring for 30 minutes at a temperature of 65°C. After that, the chitosan solution was added to the corn starch solution, and the stirring process was carried out for 15 minutes until it reached homogeneity without any heating process. Then, 6 ml of glycerol is added, and the stirring and heating process is carried out for 15 minutes at 65° Celsius. Then add $CaCO_3$ filler, which has been dissolved in acetic acid solution in a ratio of 1:1, and the solution will be left to sit for 5 minutes to avoid the formation of holes in the plastic.

Before casting, a cleaning process is carried out using 95% alcohol. The biodegradable plastic solution is poured into the prepared mold. Dry the mixture at room temperature for 24 hours. After drying, the bioplastic can be released from the mold, and its characteristics can be tested by cutting it into several parts to be used as a sample in the testing process. The performance of the resulting bioplastic was tested using tensile strength tests, elongation tests, biodegradation tests, and analysis using Fourier Transform Infrared Spectroscopy (FTIR). In the biodegradation test, the percentage of weight loss from bioplastics is calculated using the formula:

$$\text{Percent weight loss}(\%) = \frac{W1-W2}{W1} \text{ x } 100\% \text{ (1)} \ldots\ldots\ldots\ldots\ldots\ldots \text{(1)}$$

With:

W1 = Plastic weight before testing

W2 = Plastic weight before testing

FTIR analysis was conducted at the Integrated Research and Testing Laboratory at Gadjah Mada University.

3. RESULTS AND DISCUSSION

There are five types of modified starch: cationic starch, etherified starch, esterified starch, resistant starch, and pre-gelatinized starch [26]. Modification by etherification and esterification are the most studied fields in the synthesis of bioplastics. There are 3 starch modification schemes with esterification: Fischer's esterification; by a nucleophilic substitution reaction between the electrophilic carbonyl carbon of vinyl carbonate and the hydroxyl group of starch via an acid-base reaction between cesium carbonate and vinyl carbonate; by removes protons of the cationic ether [27]. Because this research uses calcium carbonate, the same type as cesium carbonate, this research is included in the second scheme.

The most popular starch ester is starch acetate [28]. The mechanism of esterification with acetic acid in this study is approached by esterification with vinyl acetate.

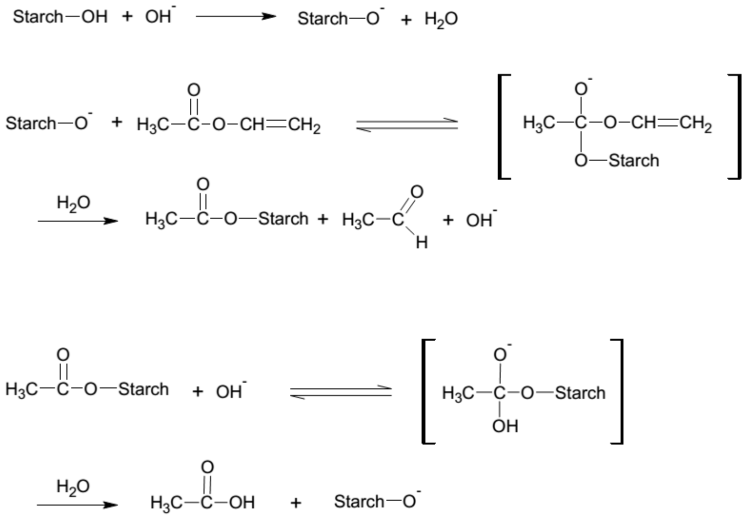

Figure 1. Mechanism of starch acetylation using vinyl acetate [28]

The following are bioplastic products produced from the process that has been carried out. Based on the picture below, bioplastics have different colors from one another. This is caused by the $CaCO_3$ content contained in each bioplastic. The higher the $CaCO_3$ content, the more lime deposits are formed, resulting in chalky white bioplastic. The bioplastic results are shown in Figure 1.

Figure 2. Bioplastics from Starch

3.1. Variation of $CaCO_3$

In the testing process using the tensile strength method, the plastic surface area of the samples tested and the respective loads for bioplastics with variations in $CaCO_3$ and glycerol. Based on the research that has been carried out, several data were obtained which are presented in table 2. All data written in this article is the average of data produced from experiments.

Table 2. Maximum load that bioplastics can withstand

Sample	$CaCO_3$ (gr)	Load (kg)
1	1	0.4148
2	2	0.8465
3	3	0.6503
4	4	0.6503
5	5	0.2674

This load is the maximum load that the bioplastic can withstand until the bioplastic tears and breaks, where the maximum load that the resulting bioplastic can withstand is 0.8465 kg with a $CaCO_3$ content contained in the bioplastic of 2 grams and the lowest load value obtained is 0. 2674 kg with a $CaCO_3$ content of 5 gr. So, based on the research results, the higher the $CaCO_3$ content contained in bioplastics will reduce the plastic's ability to withstand loads.

Meanwhile, the tensile strength test results obtained from the test results that have been carried out are as follows:

Table 3. Results of tensile strength tests on bioplastic samples with variations in $CaCO_3$

Sampe l	$CaCO_3$ (gr)	Tensile strength (Mpa)
1	1	0.0346
2	2	0.0705
3	3	0.0542
4	4	0.0542
5	5	0.0223

Based on the research results above, it can be concluded that the higher the level of $CaCO_3$, the more Ca is formed, resulting in the plastic having hard and stiff properties and tensile strength properties that decrease due to this stiffness. The reaction between $CaCO_3$ and CH_2COOH is as follows:

$$14\ CaCO_3 + CH_2COOH \rightarrow 7\ Ca + 6\ H_2O + 22\ CO_2$$

The elongation test aims to determine the percent elongation produced from bioplastics with variations in $CaCO_3$ and glycerol. Based on the test results, results were obtained for bioplastics with variations in $CaCO_3$ with 1 gram of chitosan and 6 ml of glycerol. Based on the results of the research, the following data were obtained:

Table 4. Elongation test results on bioplastic samples with variations in $CaCO_3$

Sample	$CaCO_3$ (gr)	Elongation value, %
1	1	28
2	2	40
3	3	30
4	4	22
5	5	20

The data in Table 4 states that increasing the $CaCO_3$ content contained in bioplastics will reduce the percent elongation value. Meanwhile, the highest percent elongation value is bioplastic with a $CaCO_3$ variation of 2 gr with a percent elongation value of 30%.

The ability of the biodegradation process in soil on bioplastics was tested using the planting method. Samples are weighed and measured first before being buried in the ground. Samples are buried in the ground at a certain depth. Checks will be carried out every four days after the landfill process until the plastic is completely biodegraded. Based on biodegradable testing that has been carried out with bioplastics with $CaCO_3$ variations, the results of % w loss are as follows:

Table 5. Biodegradation test results on bioplastic samples with variations in glycerol

Sample	$CaCO_3$ (gr)	W loss (%)
1	1	91,43
2	2	66,13
3	3	36,12
4	4	71,03
5	5	83,56

From the data in Table 5, it can be concluded that the higher the $CaCO_3$ content contained in bioplastics or the greater the amount of $CaCO_3$ contained in bioplastics, the %w loss value will decrease, with the enormous %w loss value being 91.43% with variations in $CaCO_3$.

In the research that has been carried out, the results of FTIR analysis on variations of bioplastics with $CaCO_3$ are as follows:

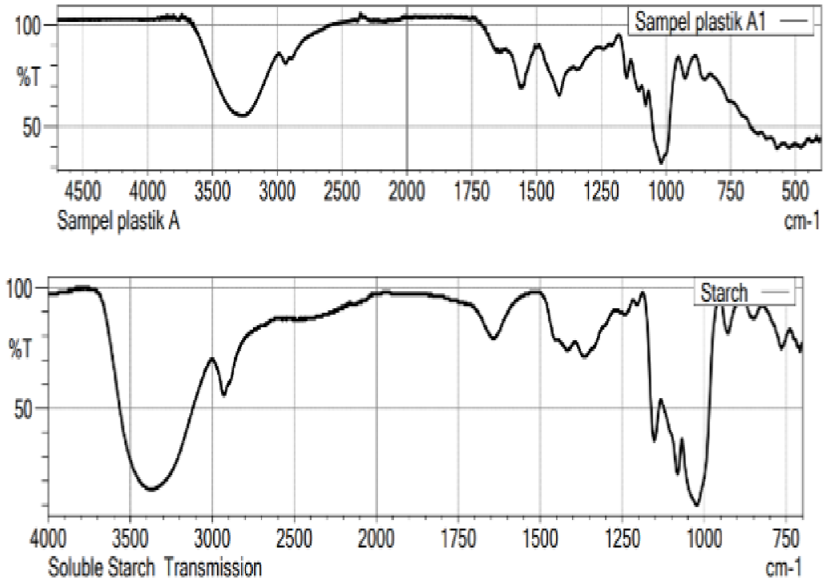

Figure 3. f FTIR Testing results on Bioplastic Samples with $CaCO_3$ Variations

The results of FTIR testing for bioplastic samples with variations in $CaCO_3$, C-H, C-O, and NO_2 bonds were obtained. In the bioplastic sample, it was found that the C-H bond in the alkyne compound was at the point 3300 cm^{-1}. The C-O bond itself is at the point 1150 cm^{-1}. Meanwhile, a small amount of NO_2 compounds are located at the 1550 cm^{-1} point.

The microstructure of bioplastics is obtained from analysis with Scanning Electron Microscope (SEM).

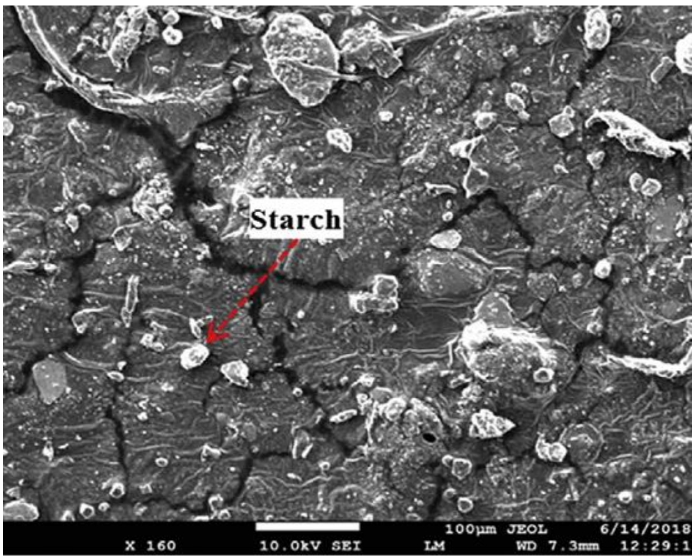

Figure 4. Scanning Electron Microscope of bioplastic from corn starch [29]

The microstructure of bioplastic made from corn starch can be seen in Figure 3. It can be seen that there are insoluble corn starch residues. There are also several empty holes which reduce tensile strength.

3.2. Glycerol Variations

When making bioplastics with variations of glycerol, the maximum weight that the bioplastic can hold until it breaks is obtained. Based on research and testing, the following data can be obtained:

Table 6. The maximum load that bioplastics can withstand with variations in glycerol

Sample	Glycerol (ml)	Load (kg)
1	4	1.3844
2	5	1.4494
3	6	0.8544
4	7	0.7688
5	8	0.5867

The resulting bioplastic was cut into samples for testing with a surface area of 12 cm2. The tensile strength test results were obtained as follows:

Table 7. Tensile strength test results on bioplastic samples with variations in glycerol

Sample	Glycerol (ml)	Tensile strength (Mpa)
1	4	0.1154
2	5	0.1208
3	6	0.0712
4	7	0.0641
5	8	0.0489

Based on the results of the glycerol variation test, it can be concluded that the higher the glycerol variation, the lower the tensile strength, but the drying process will take longer.

Meanwhile, for variations in glycerol with fixed variables in the form of 1 gram of chitosan and 2 grams of $CaCO_3$, the results of the elongation test were obtained as follows:

Table 8. Elongation test results on bioplastic samples with variations in glycerol

Sample	Glycerol (ml)	Elongation value, %
1	4	28
2	5	30
3	6	30
4	7	30
5	8	60

Based on research results, bioplastics have a percent elongation value that is initially stagnant and then increases drastically due to the glycerol contained therein. Furthermore, the %w loss values are shown at table 9.

Table 9. Biodegradation test results on bioplastic samples with variations in glycerol

Sample	Glycerol (ml)	W loss (%)
1	4	16,67
2	5	40.94
3	6	66,13
4	7	24,72
5	8	50,00

Based on the data above, it can be concluded that the greater the volume of glycerol, the easier it will be for bioplastics to degrade.

The FTIR results for variations in glycerol are as follows:

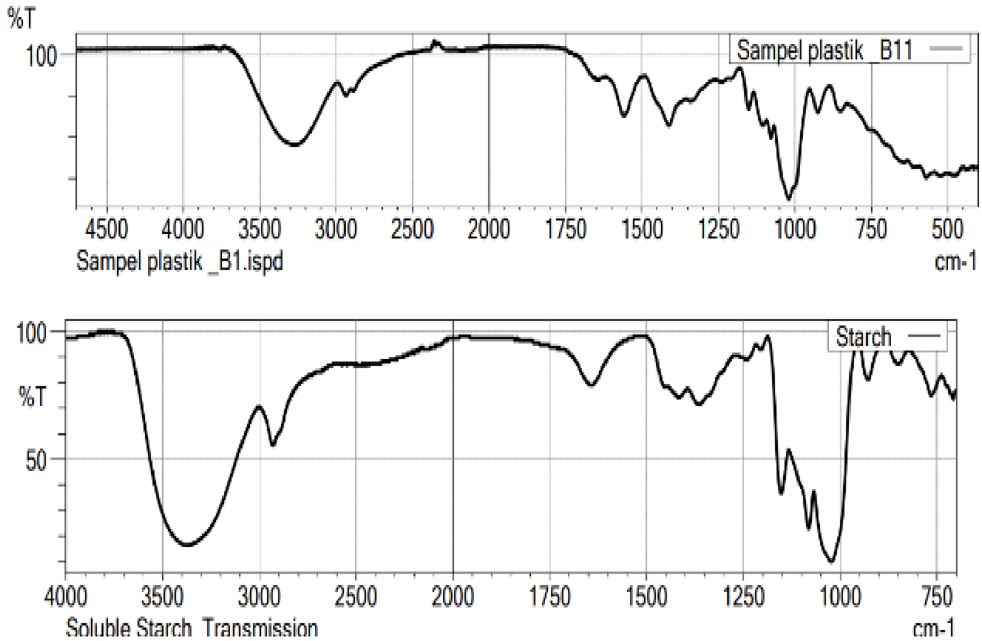

Figure 5. FTIR testing results on bioplastic samples with variations in glycerol

Meanwhile, for bioplastic samples with variations in the form of glycerol, bioplastic samples with variations in glycerol obtained C-H, C-O bonds, and NO_2 compounds. In the FTIR test, it was found that the C-H bond in the alkyne compound was at the point 3300 cm $^{-1}$. The C-O bond itself is at the point 1050 cm $^{-1}$. Meanwhile, the NO_2 compound is located at 1560 cm $^{-1}$. The presence of C-O functional groups in bioplastics has biodegradability. This is because C-O is a group that it is hydrophilic, which has the ability to bind water moleculeswhich comes from the environment and causes microorganisms to enter plastic matrices [30].

3.2. Comparison with other studies

Starch is a raw material that is often used in making bioplastics. The raw materials commonly used are potato, corn, cassava, and wheat [26]. The Indonesian plants that have been used as raw materials for bioplastics are kepok banana and sago.

Table 10. comparison of this research with other research

Raw Material	Filler	Analysis	Results
Cassava Starch [3]	ZnO	Tensile and Elongation	a. Tensile: Zno (0.6%) and plasticizer (25%) = 22.30 kgf/mm^2 b. Elongation: ZnO (0.6%) and plasticizer (30%) = 122.80 %

Corn Starch [6]	Chitosan	Tensile, Elongation, Degradation and %Swelling	a. Tensile: Chitosan (41.7%) and glycerol (0.7%) = 3.9 Mpa b. Elongation: Chitosan (0%) and glycerol (4%) = 37.8% c. Degradation: 20 days d. %Swelling: Chitosan (41.7%) and glycerol (0.7%) = 26.76%
Sago Starch [10]	Microfibrillated Cellulose Bamboo (MFC)	Tensile and FTIR	a. Tensile: MFC (5%)and KCl (1%) = 17.99 Mpa
Kepok Banana Peel Starch [14]	Chitosan and ZnO	Tensile, Elongation	a. Tensile: Chitosan (4%) = 1.212 Mpa ZnO (5%) = 0.601 Mpa b. Elongation: Chitosan (4%)= 0.2 Mpa ZnO (5%) = 0.18 Mpa
Corn Starch	$CaCO_3$ and Glycerol	Tensile,Elongation and FTIR	a. Tensile= 0.1208 Mpa b. Highest elongation =60%

Table 10 shows that the tensile strength of the bioplastic as a result of this research is relatively low, meaning that the bioplastic produced is more easily broken. The advantage of this research is the highest elongation value. This means that the resulting bioplastic has high elasticity. It is possible to use corn starch and $CaCO_3$ as materials for synthesis of bioplastics on a higher scale. In future research, it is necessary to carry out techno-economic research on synthesizing bioplastics with CaCO3 and glycerol fillers.

4. CONCLUSION

The reaction to make bioplastic from corn starch with CaCO3 filler and glycerol is an esterification reaction. The reaction mechanism and scheme for modifying corn starch into bioplastic are known. Based on the research results, it was concluded in this bioplastic synthesis research that the higher the $CaCO_3$ content contained in bioplastics, the resulting percent elongation value would decrease, thus causing an increase in the $CaCO_3$ content contained in bioplastics. Apart from that, the reason was that the lower the percent value elongation produced by the presence of calcium. The low tensile strength is due to the presence of empty holes in the bioplastic product. The higher the glycerol variation, the lower the tensile strength, the higher the elongation percentage will increase, and the easier it will undergo biodegradation. Bioplastics with maximum capacity can be found in bioplastic variations with a $CaCO_3$ content of 2 g and 5 ml glycerol. Further research is needed to increase the scale of bioplastic production using the methods presented in this research.

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