

Synthesis And Characterization Of Biodegradable Foam (Biofoam) From Taro Starch (*Colocasia Aesculenta*)-Chitosan

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Abstract

Biodegradable foam can be used as an alternative packaging which is safe for health and environment. The raw material used to produce biofoam is starch. Taro consist of 67.42% starch. Contrarily, biofoam produced from starch has fragile mechanical properties. The addition of chitosan aims to improve the mechanical properties of biodegradable foam. Beside that, Chitosan is expected to reduce water absorption and increase tensile strength of biodegradable foam without reducing degradability. Biodegradable foam was synthesized by three steps, first step was isolation of starch from taro, second step was synthesis of biodegradable foam from taro starch-chitosan which used various concentration of chitosan to study the effect of chitosan addition for mechanical properties of biodegradable foam. Various concentration of chitosan was 0%, 1%, 2%, 3%, and 4% (w/w). Third step was characterization of biodegradable foam. The Analysis of biodegradable foam was carried out included FTIR functional group, mechanical properties, biodegradability and water absorption. The result showed that the best tensile strength test was 1% chitosan addition with a value of 3.75 MPa; based on water absorption test, the optimum sample was 4% chitosan addition with a value of 11.66%. The results of the functional group analysis of FTIR biodegradable foam contained of C=O (amide), CN, NH and OH groups which indicated the existence of chitosan in the sample.

Keywords: *Biodegradable foam, Taro starch, Chitosan.*

Introduction

Nowadays, styrofoam has commonly used as food packaging, this is due to the practicality of styrofoam. In addition, Styrofoam also has the characteristics of being lightweight, easy to shape, water resistant, heat resistant, and has an economical price. Behind the advantages of styrofoam, it turns out that styrofoam has a bad impact on health and the environment.

The World Health Organization (WHO) and other health institutions have categorized

styrofoam as a carcinogenic material because the raw material used is styrene. Styrene is a chemical that is difficult to dissolve by the digestive system and difficult to remove through urine and feces so that these substances accumulate more and can trigger cancer [1]. While the impact of styrofoam utilization is environmental damage due to the difficulty of styrofoam to decompose and it will produce dioxins when burned [2].

Biodegradable foam can be used as an alternative to styrofoam for food packaging

which is safe for health and environment, because the raw material to produce biodegradable foam is starch. Biodegradable foam or biofoam can be made from tuber starch and agricultural waste, such as EFB (Empty Palm Oil Bunches), bagasse, etc. which is easily decomposed by microorganism in the soil [3].

The main precursor in the manufacture of biodegradable foam is starch. Starch can replace up to 70% polystyrene utilization in the manufacture of biodegradable foam. Starch is widely used in the manufacture of biodegradable foam because it is inexpensive, has low toxicity and density, and is easily decomposed [4]. One of the plants that has a high starch content is taro. Taro was used in this study to improve the economic value. Taro consist of 67.42% starch (2.25% amylose and 65.17% amylopectin).

High levels of amylopectin will produce biodegradable foam with smaller pores and lower density compared to biodegradable foam with high amylose content [5]. Starch derived from Taro, a tuber growing in tropical regions, can be used owing to its granule size, which is in the range of 1–5 μm [6]. The amylopectin in Taro is short and possesses a long average chain length which aids in the formation of a firm gel with high elasticity when heated [7].

However, biodegradable foam from starch still has a low water resistance. Therefore, to improve the physical properties, such as absorption capacity of the biodegradable foam and the mechanical properties, it is necessary to innovate in the manufacture of biodegradable foam by adding chitosan as a reinforcement. This is because chitosan is hydrophobic. Chitosan in biodegradable foam is expected to reduce water absorption and increase tensile strength without reducing degradability. Chitosan is a biopolymer which is abundant and has many excellences, they are easily to decompose, easily to be absorbed by biological tissues, and non-toxic [6].

This research was conducted to study the effect of chitosan addition to the biodegradable, physical and mechanical properties of taro

starch foam. The product of biodegradable foam was then analyzed for its functional group, tensile strength, biodegradation, and water absorption capacity. The research about biodegradable foam based on starch was done before, but the used of taro as starch resources for biodegradable foam application was never been done before, therefore the novelty of this research was the resources of the starch and the combination of taro starch with chitosan for biodegradable foam application, which was potentially increased mechanical property of biodegradable foam.

Experimental

Equipment

The tools used in this study were 1000mL, 250mL and 100mL beakers, 100mL measuring cups, Erlenmeyer, test tubes, petri dishes, dropping pipettes, stirring rods, glass plates, desiccators, FTIR (Fourier Transform Infra Red), tensile strength machines (mechanical universal testing machine), magnetic stirrer, electric oven, analytical balance, blender, basin, and knife.

Material

The materials used was taro tubers, and some materials which produced by Merck with p.a quality, They are distilled water (H_2O), chitosan powder ($\text{C}_6\text{H}_{11}\text{NO}_4$), 1% acetic acid solution (CH_3COOH), magnesium stearate powder ($\text{Mg}(\text{C}_{18}\text{H}_{35}\text{O}_2)_2$) 5%, polyvinyl alcohol (PVA) 10%, iodine reagent (I_2), compost, filter paper.

Methods

Taro Starch Isolation

The taro was washed thoroughly in running water, then weighed as much as 500gr and pulverized with a blender with the addition of distilled water for the refining process with a ratio of 2:1 (w/v). The fine taro was filtered through gauze and put into a 1000mL beaker. The filtrate is allowed to stand until a precipitate forms. The precipitate obtained was washed with aquadest, then filtered and dried using an oven. The taro starch obtained was then tested for functional groups using FTIR.

Analysis of Taro Starch

Preparation of the test solution following the method used [7]. 1.0 gram of starch sample then dissolved with 20 mL of distilled water, take the upper phase as a test solution. Pipette 1 mL of the test solution and put it into a test tube, then add 1 drop of iodine reagent, then homogenize. The end result was that a blue-black or purple precipitate will be formed in the solution if the sample contains starch.

Synthesis of Biodegradable Foam

Biodegradable foam starch (BS) and biodegradable foam starch-chitosan (BSC) were prepared by adding 11mL of distilled water to 8 grams of starch (80% w/v), 5% magnesium stearate (w/w starch), 10% PVA (w/w starch), and chitosan with variations of 0%, 1%, 2%, 3%, and 4% which had previously been dissolved in 10 mL of 1% acetic acid. They were Mixed using a magnetic stirrer at high speed until homogeneous. The next step was to pour the mixture into the mold and then put it in the oven at 250°C for 15 minutes. Then the Foam cooled at room temperature then the functional group of the product was characterized by FTIR.

Mechanical Strength Test

The tensile strength test is carried out using a Tensile Strength tool, the workings of the tensile strength test are by pulling the specimen from two directions so that the length increases and the diameter decreases. The test calculation uses the following formula [8].

$$TS = F/A$$

Information:

F = maximum force

A = magnitude outside the initial surface

Biodegradation Test (Decomposition)

A total of 0.05 grams of sample was planted in 100 grams of compost soil to a uniform depth in a transparent plastic cup for a certain time interval, then observed how long it took for the biodegradable foam to completely disappear.

Water Absorption Test

The biodegradable foam sample with a size of 2.5 x 5 cm was cut, then the sample was weighed as the initial weight, then the sample was immersed in water for 1 minute and the sample was dried with a tissue so that no residual water sticks to the sample. The sample was weighed again and it was calculated so that the % water absorption is obtained [9]. The calculation of the added weight was done by the following equation:

$$\% \text{ Water absorption} = (W_1 - W_0) / W_0 \times 100\%$$

Information:

W_1 = Sample weight after immersion

W_0 = Initial sample weight
Peralatan/Instruments

Results and Discussion

The results of the iodine test conducted on taro starch showed positive results for the presence of starch, the results obtained were in accordance with the opinion of Fessenden et al (1986) [10], due to the presence of glucose units in the starch solution which formed helical chains which resulted in starch forming complex with an iodine molecule that can enter the spiral to form a blue-black color in the complex.

Functional group analysis of taro starch was carried out at wave numbers 4000-400 cm^{-1} . The FTIR results of taro starch synthesis were found at wave numbers 3269.31 cm^{-1} ; 2928.58 cm^{-1} ; 1640.14 cm^{-1} ; 1336.32 cm^{-1} ; 1077.75 cm^{-1} and 860.44 cm^{-1} . As seen in Figure 1 and Table 1.

The wave number of 3269.31 cm^{-1} indicates the presence of the O-H functional group which is a hydrogen bond in the starch molecule. The wave number of 2928.58 cm^{-1} indicates the absorption value of the C-H alkane group. The C=O functional group was indicated by the wave number 1640.14 cm^{-1} . The wave number detected was then 1336.32 cm^{-1} which indicated the presence of the C-N functional group, this functional group identified the presence of protein contained in the resulting taro starch.

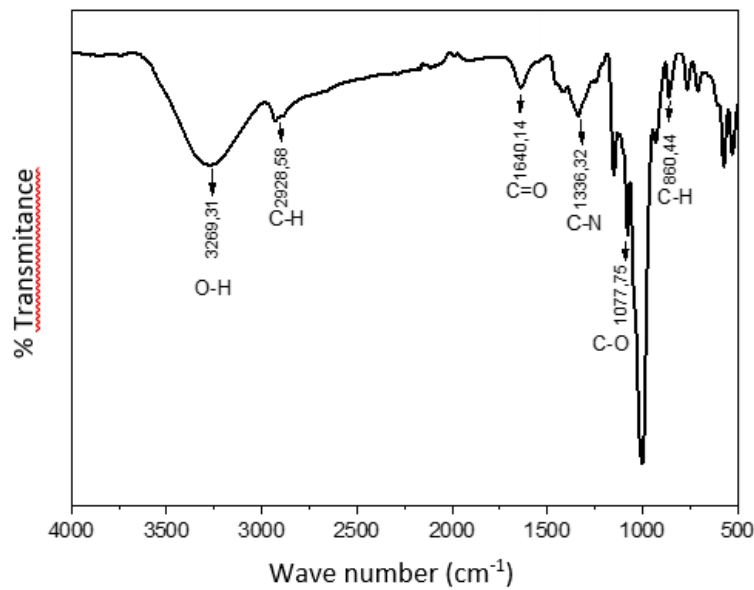


Figure 1. FTIR analysis of Taro starch

Table 1. The result of FTIR analysis of Taro starch

No.	Functional Groups	Wave Number (Cm ⁻¹)	Wave Number Range (Cm ⁻¹)
1.	O-H	3269,31	3200-3600
2.	C-H	2928,58	2850-2970
3.	Alkana C=O	1640,14	1600-1760
4.	Amida C-N	1336,32	1180-1300
5.	C-O Ester	1150,14	1000-1300
6.	C-H Aromatic	860,44	690-900

The wave number of 1077.75 cm⁻¹ indicates the presence of a C-O ester group. While the wave number of 860.44 cm⁻¹ indicates the presence of an aromatic C-H group. The results of the FTIR analysis already represent the starch content of taro which consists of amylose, amylopectin and reducing glucose (C₆H₁₀O₅)_n [11].

FTIR characterization of taro tuber starch resulted that there were functional groups of O-H, C-H, C=O, C-O and C-H aromatic with a wave number of 3433.29 respectively cm⁻¹; 2931.80 cm⁻¹; 1643.35cm⁻¹; 1149.47 cm⁻¹ and

850.39 cm⁻¹. This showed that the results of the FTIR test carried out have similar results with previous studies from [12].

Functional group characterization was carried out to identify functional groups indicated in the biodegradable foam. The results of characterization of biodegradable foam using FTIR are shown in Figure 2 and Table 2.

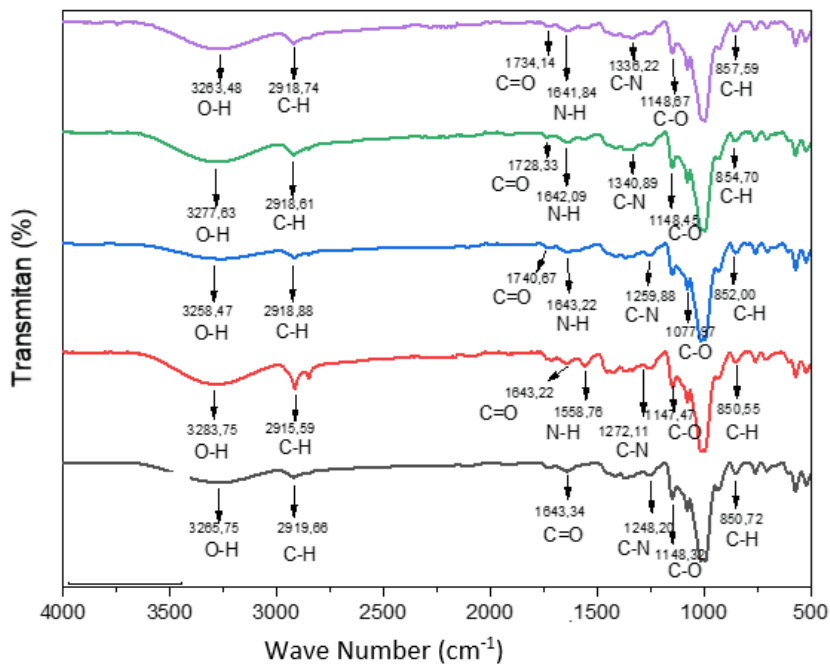


Figure 2. FTIR analysis of Biodegradable foam

Table 2. The result of FTIR analysis of Biodegradable foam

No	Functional Groups	Wave Numbers (cm^{-1})				
		BS	BSC 1%	BSC 2%	BSC 3%	BSC 4%
1.	O-H	3265.75	3283.75	3258.47	3277.63	3263.48
2.	C-H	2919.66	2915.59	2918.88	2918.61	2918.74
3.	Alkana C=O	1643.34	1643.22	1740.67	1728.33	1734.14
4.	Amide N-H	-	1643.22	1643.22	1642.09	1641.84
5.	Amine C-N	1248.32	1272.11	1259.88	1340.89	1336.22
6.	Ester C-O	1148.32	1147.47	1148.09	1148.45	1148.67
7.	Aromatic C-H	850.72	850.55	852.00	854.70	857.59

The results of the FTIR characterization of BS and BSC in the $3200\text{-}3600\text{cm}^{-1}$ area were typical areas of the O-H functional group, in the $2850\text{-}2970\text{cm}^{-1}$ absorption area are typical areas of the C-H alkane functional group, the absorption area of $1600\text{-}1760\text{ cm}^{-1}$ is the C=O functional group amide, the absorption area of $1180\text{-}1360\text{ cm}^{-1}$ is a typical area of the C-N amine functional group, the absorption area of $1050\text{-}1300\text{cm}^{-1}$ is a typical area of the C-O ester functional group, the absorption area of $690\text{-}900\text{ cm}^{-1}$ was a typical area of the aromatic C-H

functional group and the absorption area of $1550\text{-}1650\text{ cm}^{-1}$ which was a typical area of the N-H functional group which was a characteristic chitosan absorption group. The shift that occurs in the absorption area of the N-H group at wave number $3300\text{-}3500\text{ cm}^{-1}$ was due to the presence of bonds in the molecule which results in the elongation of the N-H bond so that the wave number shifts to the right [13].

The results of chitosan uptake can be seen in Figure 3 and Table 3.

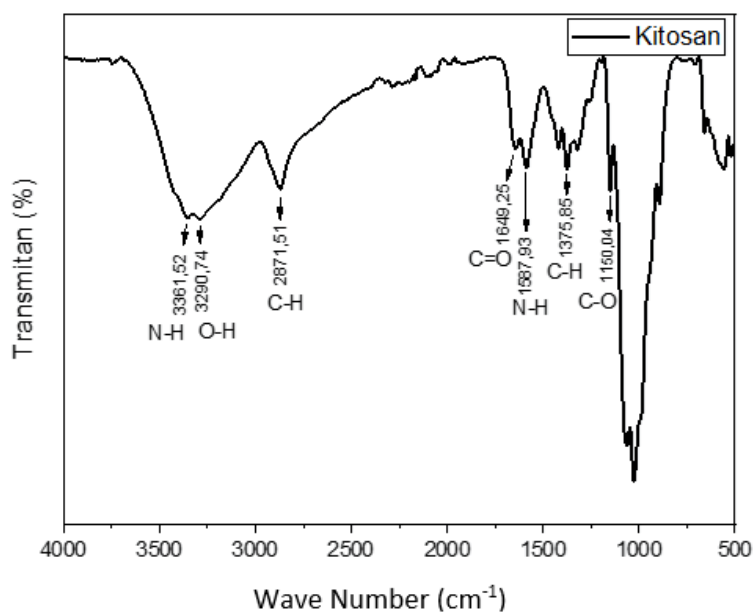


Figure 3. FTIR analysis of Chitosan

Table 3. The result of FTIR analysis of Chitosan

Functional Groups	Wave Number (cm ⁻¹)	Absorption Area (cm ⁻¹)
N-H Amide	3361.52	3300-3500
O-H	3290.74	3200-3600
C-H Alkane	2871.51	2850-2970
C=O Amide	1649.25	1600-1760
N-H Amide II	1587.93	1550-1650
C-O Ester	1150.04	1050-1300

FTIR analysis of chitosan showed that there was an absorption peak at 3361.52 cm⁻¹ which was a typical area of the N-H functional group, this group was a characteristic absorption of chitosan. The appearance of wave number 3290.74 cm⁻¹ indicated the presence of the O-H functional group. The C-H functional group of the alkane was indicated by the presence of a wave number of 2871.51 cm⁻¹. The wave number 1649.25 cm⁻¹ was a typical region of the

C=O functional group of amides. While the wave number of 1150.04 cm⁻¹ indicated the presence of the C-O ester functional group. Indications of the formation of chitosan due to weak stretching absorption at wave number 1649.25 cm⁻¹ which was a typical area of the C=O group in the bond (-NHCOCH₃)^[14].

Mechanical Properties Test

Tensile strength and percent elongation tests were carried out to determine the tensile strength and elongation values of the synthesized biodegradable foam. The resulting tensile strength values were shown in Table 4.

In this study, the tensile strength value of biodegradable foam with chitosan variations was less stable because it experienced an increase and decrease in each variation. At BS, the tensile strength value was 3.18 Mpa and then it increased at 1% BSC with a value of 3.75 MPa. However, it tended to decrease at 2%, 3% and 4% BSC with a tensile strength value of 1.88 MPa; 2.03 MPa and 0.91 MPa. This happened due to the high temperatures in the drying process of biodegradable foam. The higher the drying temperature, the lower the tensile strength value ^[15].

The high temperature in the drying process would affect the evaporation of chitosan and PVA in the biodegradable foam, so that the resulting biodegradable foam contained little chitosan and PVA. Made Heni Epriyanti et al. (2016) ^[16] in their research also revealed that high temperatures and long drying times will showed low tensile strength values, due to the tendency to weaken the plasticizer used. The results obtained in this study were inversely proportional to previous research. In Nurfitasari's research (2018) ^[17] stated that the addition of chitosan could increase the tensile strength value of biodegradable foam due to the presence of amine, primary and secondary hydroxyl functional groups which resulted in

the formation of hydrophobic hydrogen bonds due to the high reactivity of chitosan. Chitosan in the manufacture of biodegradable foam would experience interactions with amylopectin and amylose contained in starch. There was an interaction between the O or N atom molecules present in chitosan interacting with H atoms from amylose, amylopectin or from chitosan itself, this interaction was a hydrogen bond ^[18].

The elongation test was included in the tensile strength test section, the elongation test was intended to determine the stretch length of the biodegradable foam after the tensile force was applied before and after it was broken ^[19] The percent elongation values could be seen in Table 5.

The elongation test that has been carried out obtained results that were inversely proportional to the theory. Elongation was the opposite of tensile strength. The elongation value of biodegradable foam would decrease with the addition of chitosan. Ginting et al. (2016) ^[20] argued that the decrease in intermolecular bond distances was due to the amount of chitosan added. The decrease in intermolecular bond spacing was due to the increase in the number of hydrogen bonds formed in the chitosan-amylose-amylopectin molecule. This caused the percent elongation value to be low and the resulting biodegradable foam became stiff and less elastic ^[21]. In this study the highest percent elongation results were 4% BSC and the lowest percent elongation were BS and 2% BSC.

Table 4. Mechanical properties of Biofoam

No.	Biodegradable foam	Tensile Strength (Mpa)
1.	BS	3.18
2.	BSC 1%	3.75
3.	BSC 2%	1.88
4.	BSC 3%	2.03
5.	BSC 4%	0.91

Table 5. Biodegradation test of biofoam

Various concentration of chitosan addition in biofoam	Weight of sample (gram)	Time (days)
0% (BS)	0.05	8
1% (BSC)	0.05	10
2% (BSC)	0.05	10
3% (BSC)	0.05	12
4% (BSC)	0.05	12

Biodegradation Test

The biodegradation test aimed to determine the time required for the synthesized biodegradable foam to be completely degraded. The results of the research conducted were obtained in Table 5.

The results obtained from the biodegradation test with the highest degradation rate at BS and the lowest degradation rate at 3% and 4% BSC. The higher the concentration of chitosan, the more difficult the biodegradable foam would be decomposed by the soil. This was because the addition of chitosan to biodegradable foam made from starch would form strong hydrogen bonds between the NH_3^+ groups of chitosan and the OH groups of starch. In chitosan the NH_2 group undergoes protonation to become the NH_3^+ group in acetic acid solution. As the amount of chitosan used increased, the value of the NH_3^+ group increased too, so that the resulting biodegradable foam became stronger and more difficult to degrade by microbes in the soil [22].

According to international standards (ASTM 5336) the time needed for styrofoam to be degraded (biodegraded) was 60 days to completely decompose (100%). Meanwhile, according to the European Union Standard (EN 13432), the maximum time for biodegradable foam to decompose is 6 months to 9 months [23]. In this study, the degradation time of biodegradable foam was 12 days for the lowest degradation rate, with a degradation rate of

100%. This could happen because the amount of the sample affected how fast the sample could degrade in the soil, the sample used in this research was small with a weight of 0.05 grams so the time needed was relatively short but it still could achieve the international standard of ASTM and EN.

Water Absorption Test

Absorption test was carried out to determine the resistance of the synthesized biodegradable foam to water contact. Biodegradable foam samples were weighed before and after being immersed in water. Then the amount of water absorbed by the sample is calculated and written as the percentage of water absorbed. The results of the water absorption test that has been carried out were obtained in accordance with Table 6.

The amount of chitosan added affects the percentage of water absorbed, the more chitosan added, the lower the water absorption. It means that the lower water absorption, the higher water resistance. Setiani et al. (2013) [18] in his research explained that due to the presence of an amine group (NH_2) at the C2 position, primary and secondary hydroxyl at the C3 and C6 positions in chitosan. The presence of amino acids with a positive charge caused the molecule to be able to bond with a negative surface charge through ionic or hydrogen bonds.

Table 6. Water absorption test of Biofoam

No.	Biodegradable foam	Water absorption (%)
1.	BS 0%	24.54
2.	BSC 1%	22.64
3.	BSC 2%	15.58
4.	BSC 3%	13.43
5.	BSC 4%	11.66

This bond caused chitosan to be hydrophobic or difficult to bind with water and it caused increasing of water resistance and decreasing of water absorption percentage.

BS has the lowest water resistance which mean that it had the highest water absorption percentage, this was because the biodegradable foam was fully made from starch which has hydrophilic properties that tend to bind with water. This argument was related to Ansori, A. et al (2023) [23] which reported that The resistance of Cassava starch films is influenced by the number of hydroxyl groups in the polymer molecule, where the hydroxyl groups bind to water molecules by forming hydrogen bonds. The increase in the number of hydroxyl groups in the cassava starch film can occur from the intramolecular interaction of the cassava starch with PVA polymer mixture, which can significantly increase the binding of water molecules. This property made biodegradable foam impervious to water because the water absorption that occurred in BS was higher. The results of the BSC absorption test of 1%, 2%, 3%, and 4% showed an increase in water resistance, which showed by the decrease of water absorption percentage. The value of water absorption percentage of BSC 0%, 1%, 2%, 3%, and 4% was respectively 24.54%, 22.64%; 15.58%; 13.43%; and 11.66%. The product with 4% chitosan addition had the best water resistance value, this is in accordance with the hydrophobic nature of chitosan so that the addition of chitosan to biodegradable foam could increase the water resistance value of the resulting samples.

Conclusions

Based on the results of the study it can be concluded that the addition of chitosan in the process of making BSC increased its biodegradation value from 8 days to 12 days; reduce the value of water absorption from 24.54% to 11.66%; and the results of the FTIR test stated that the BS and BSC variations of 1, 2, 3 and 4% contained the functional groups of OH, CH alkanes, CN amines, CO esters and CH aromatics. The difference between BS and BSC was the presence of the NH functional group in BSC, which was a characteristic of chitosan functional group. The effect of the addition of chitosan on the mechanical properties of BSC in this study showed the best value of 4% addition of chitosan with the value of tensile strength was 3.75 Mpa.

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