

Study and Characterization of Biofoam From Bagasse (*Saccharum officinarum* L.) with Chitosan Addition

Rismawati^{1*}, Paulina Taba², Fahrudin³

¹ Environmental Management Study Program of Graduate School, Hasanuddin University, Makassar, Indonesia

² Department of Chemistry, Faculty of Mathematics and Natural Sciences, Hasanuddin University, Makassar, Indonesia

³ Department of Biology, Faculty of Mathematics and Natural Sciences, Hasanuddin University, Makassar, Indonesia

* Corresponding author's e-mail: rismaimma288@gmail.com

ABSTRACT

The increase in styrofoam waste can cause environmental pollution, therefore efforts are required to reduce the use of styrofoam. One of them is by replacing it with biofoam made from agricultural industry waste such as bagasse because if this garbage is not used, it may cause environmental issues. Bagasse has a potential to be utilized as biofoam. This research aims to extract cellulose from bagasse through delignification and bleaching processes, as well as synthesize and characterize biofoam using cellulose from bagasse with the addition of chitosan variations of 2, 3.5, 5, and 6.5 g. The stages of this research are the extraction of cellulose from bagasse waste with FTIR and XRD analysis, making biofoam and biofoam characterization tests. The results of cellulose extraction from bagasse are O-H, C-H and C-O functional groups that indicate the presence of cellulose. In XRD analysis, the cellulose sample has a crystallinity index of 70.74%. Biofoam based on sugarcane bagasse cellulose with the addition of 2 g chitosan has the best characterization, which has a density of 1.23 g/mL, with a water absorption value of 46.03% at 24 hours immersion and biodegradability of 20.68% for 28 days.

Keywords: sugarcane bagasse waste cellulose, chitosan, biofoam and degradation

INTRODUCTION

Styrofoam, a synthetic polymer variant of polystyrene, is utilized as a filler in food packaging and as a support for throwaway electronic equipment. As a result, discarded packaging contributes to environmental issues (Setyowati, 2014; Yanto et al., 2019). Styrofoam waste is non-decomposable and difficult to decompose in nature, it can only build up and harm the ecosystem (Kim et al., 2021; Wulan et al., 2020). Data from the Ministry of Environment and Forestry reported that Indonesia produces 175.000 tons of waste per day or 0.7 kg per person, and reaches 67.1 million tons per year of which 16% is plastic waste (Ismawati et al., 2022). Burning the waste produces harmful gases such as styrene, polycyclic aromatic hydrocarbons (HAP), hydrochlorofluoro carbon (HCFC) and carbon monoxide

(CO) will be generated. Therefore, the use of Styrofoam should be reduced (Verma et al., 2016; Zhou et al., 2016; Fikri and Veronica, 2018). An effort to reduce the use of styrofoam is to develop materials that do not significantly burden the environment. Biodegradable foams (biofoams) can replace styrofoam. The materials are good alternative materials because they decompose naturally by microbial activity and can increase soil fertility (Yao et al., 2022; Darni et al., 2023). Numerous investigations on the synthesis of biofoam have been carried out. One such study used the cellulose and starch from banana stems to construct biofoam, which decomposes 90% in 30 days and absorbs 11% of the water in just one minute of soaking (Fatrozi et al., 2020). Sumardiono et al. (2021) made biofoam by utilizing cassava flour and corn fiber which produces biofoam with 25.45% water absorption in 1 minute of soaking

time and decomposes in the soil 20.25% within 14 days. Based on these studies, biofoams are a foam made from natural products such as agricultural waste (Sumardiono et al., 2021; Alexander et al., 2023). One of these is bagasse, where the Camming sugar mill produces in excess of 24.614 tons annually and generates trash that can account for up to 90% of processed sugar cane. The processing of bagasse waste is still less than optimal (Perwitasari et al., 2021; Rizal et al., 2020; Wani et al., 2023). Only little volumes of bagasse waste are used as animal feed and fuel. As a result, this kind of trash that is not needed is typically built up outside the mill, which might contaminate the surrounding area. Sugarcane bagasse waste can be used as a basic material for making biofoam because bagasse has a cellulose content of 47% (Mahmud and Anannya, 2021). The high cellulose content can be used as an alternative choice in making biofoam (Indarti et al., 2021). Sugarcane bagasse waste must be extracted first to separate cellulose and lignin by delignification and bleaching processes (Hisbiyah et al., 2021; Watkins et al., 2015). Delignification aims to dissolve the lignin content of bagasse waste so as to facilitate the separation of lignin and cellulose (Karp et al., 2013; Abdel, 2014). However, the manufacture of biofoam made from cellulose still has shortcomings, namely poor water solubility, one of the innovations to improve the characteristics of biofoam is the addition of chitosan, which is expected to reduce water absorption without reducing the degradability of biofoam. Chitosans are a biopolymer that functions as a filler with hydrophobic functional groups so it is very reactive because it can form hydrogen bonds (Akopova et al., 2021). Thus, the purpose of this research is to extract cellulose from sugarcane bagasse through delignification and bleaching process, as well as synthesize and characterize cellulose-based biofoam from sugarcane bagasse with chitosan mass variation to obtain biofoam with low water solubility without reducing its degradability.

EXPERIMENTAL

Material

The materials used in the study were bagasse waste from the camming sugar factory, starch ($C_6H_{10}O_5)_n$, chitosan ($C_6H_{11}NO_4)_n$, distilled water (H_2O), sodium hydroxide (NaOH) Merck, sodium

hypochlorite ($NaOCl$), nitric acid (HNO_3) Merck, acetic acid (CH_3COOH) Merck, polyvinyl alcohol (PVA) ($C_4H_6O_2)_n$, glycerol ($C_3H_8O_3$), magnesium stearate ($Mg(C_{18}H_{35}O_2)_2$), and soil.

METHODS

Sugarcane bagasse preparation

The bagasse was cleaned of impurities by washing it thoroughly under running water. Then the bagasse was dried in the sun for 3 days. After drying, the bagasse was crushed using a blender. The bagasse powder was then stored in an airtight plastic container so that the raw material was not contaminated. Furthermore, chemical composition testing of bagasse waste is carried out to determine the ash content, lignin content, hemicellulose content and cellulose content contained in bagasse to be extracted.

Sugarcane bagasse cellulose extraction

The extraction process of bagasse cellulose was divided into two processes, namely the delignification process and the bleaching process. The delignification process refers to (Mzimela et al., 2020) by mixing 50 grams of bagasse powder with 2% NaOH as much as 500 mL into a glass beaker and stirring using a magnetic stirrer for 2 hours at 80 °C. The mixture was filtered and dried in an oven at 50 °C. The bleaching process refers to the procedure (Mandal and Chakrabarty, 2011; Hasan, 2014) by adding the delignified product with 250 mL of 5% NaOCl while stirring using a magnetic stirrer and heating at 70 °C for 1 hour. The mixture was then filtered and washed until the filtrate was neutral. The resulting residue was then added 0.05 N HNO_3 stirred at 70 °C for 1 hour then filtered and washed until the filtrate was neutral. The residue (cellulose extract) was dried in an oven at 50 °C. The extracted cellulose was sieved using an 80 mesh sieve to be used in making biofoam (biodegradable foam).

Characterization of bagasse cellulose

Infrared spectrum analysis

FTIR spectra of sugarcane bagasse waste cellulose were recorded using a Shimadzu Spectrophotometer. The sample was added

with KBr in the ratio of 1:100. The spectrum was recorded in the range of 4000–500 cm^{-1} with a resolution of 2 cm^{-1} and a scan count of 200. The spectrum data was then analyzed using Origin software.

X-ray diffraction analysis

The sample to be analyzed was placed on the sample holder and then irradiated using Cu K α radiation ($\lambda = 0.1539 \text{ nm}$) with a 2 theta angle ranging from 15°–70° with a step size of 0.01. The diffractogram obtained was then refined using Rietica software. The results refined with the model diffraction pattern show the lattice parameters of the sample. The crystallinity index (C_I) was calculated using Origin and Excel software, with Equation 1, measuring the peak heights of crystalline regions (I_{002}) and amorphous regions (I_{am}) (Johar et al., 2012).

$$C_I(\%) = \frac{I_{002} - I_{am}}{I_{002}} \times 100\% \quad (1)$$

Biofoam manufacturing process

The manufacture of biofoam refers to the previous procedure (Sari, 2012). The manufacture of bagasse-based biofoam was divided into three stages, namely making biofoam batter, baking process and testing the characteristics of the resulting biofoam. The manufacture of biofoam was carried out at room temperature starting with dissolving chitosan variations in 1% acetic acid up to 100 mL, stirred using a magnetic stirrer to form a gel. Each variation was added gram, PVA 10 g, glycerol 4.5 mL and magnesium stearate 1.8 put into one container, then the mixture was stirred with a mixer until it expanded. Starch as much as 15 g was added to the stirred mixture, added with 15 g cellulose that had been weighed, the mixture was stirred until homogeneous using a mixer. Next, the mixture was printed on aluminum foil and dried in an oven at 150 °C for 1 hour. The resulting

biofoam was then tested for its characteristics. Variations of chitosan and bagasse cellulose addition can be seen in Table 1.

Biofoam characterization

Characterization of biofoam from bagasse was carried out including:

- Biofoam density

Biofoam density measurements were carried out using measurement techniques previously carried out by (Yunita et al., 2023), namely by measuring the weight and volume of the sample. Density was measured using Equation 2.

$$\text{Biofoam density } (\rho) = \frac{M_b}{V_b} \quad (2)$$

where: ρ – biofoam density (g/cm^3), M_b – biofoam mass (g), V_b – biofoam volume (cm^3).

The sample was cut to a size of 10 × 10 mm and weighed using an analytical balance until a constant weight (biofoam mass). The volume of biofoam is obtained by multiplying the length, width and thickness of the sample, where the length, width and thickness of the sample are measured using a screw micrometer.

- Water absorption

The water absorption test used the TAPPI T 441 om-09 standard method. Biofoam samples were cut to a size of 25 × 25 mm. The sample was weighed to a constant weight (initial weight W_0). Then soaked in 200 mL of water for 24 hours. Next, the water on the surface of the sample was dried using a tissue and the sample was weighed after soaking (final weight W_1) (Indarti et al., 2023). Calculation of water absorption (%) was done using Equation 3.

$$\text{Biofoam density } (\rho) = \frac{M_b}{V_b} \quad (3)$$

where: W_0 – initial sample weight (g), W_1 – final sample weight (g).

Table 1. Variations of biofoam manufacturing

Sample code	Chitosan (%)	Magnesium stearat (g)	Glycerol (mL)	PVA (g)	Starch (g)	Sugarcane bagasse cellulose (g)
B _{1(kontrol)}	0	1.8	4.5	10	15	15
B ₂	2.5	1.8	4.5	10	15	15
B ₃	3.5	1.8	4.5	10	15	15
B ₄	5	1.8	4.5	10	15	15
B ₅	6.5	1.8	4.5	10	15	15

- Biodegradability

Testing the level of biodegradability using the soil burial test method refers to (Liao et al., 2022). Biofoam samples were directly contacted with the soil. The sample was calculated with a size of 30 × 30 mm, then weighed (W_0). The samples were buried in the soil and left for 7 days. Then, the weight after biodegradation was measured (W_1). Mass loss was measured using Equation 4.

$$\% \text{ Mass loss} = \frac{W_1 - W_0}{W_0} \times 100\% \quad (4)$$

where: W_0 – mass of sample before biodegradation (g), W_1 – mass of sample after biodegradation (g).

RESULTS AND DISCUSSION

Bagasse used in this study had composition cellulose of 44.06%, lignin of 11.24%, hemicellulose of 27.02%, and insoluble ash of 0.064%, indicating that the largest content is cellulose. Several studies that tested the composition of bagasse also showed the same results, where the largest content of bagasse is cellulose (Mahmud and Anannya, 2021). Although the amount varies because the bagasse tested is different. The difference in the amount of composition in each bagasse is caused by several things such

as variety, harvest time and growing conditions (Osuna et al., 2020; Hajiha and Sain, 2015).

Sugarcane bagasse cellulose extraction

The first stage of extraction was delignification using NaOH to break the bonds of lignin and can dissolve hemicellulose, mineral salts and ash characterized by a dense brown filtrate, resulting in brownish yellow cellulose in accordance with research (Sheltami et al., 2012; Deepa et al., 2011). It can be seen in Figure 1 that there is a bond break in the basic structure of lignin caused by hydroxide ions (OH^-) from NaOH. Sodium ions (Na^+) bind to lignin. The hydroxyl group of lignin was ionized and polar so that it is easily soluble in water. In this case, it is assisted with a temperature of 80 °C to accelerate the lignin degradation process which produces hot steam to help break the lignin structure.

The second stage of extraction is bleaching using NaOCl to break the ether bonds in the lignin structure. NaOCl is an oxidizing agent because it can degrade and remove lignin. So that in this process produces white cellulose, the resulting filtrate is brownish yellow which indicates that lignin compounds still remain in the delignification process and are soluble in water. In this bleaching process, sodium hypochlorite is hydrolyzed by water to produce hypochlorous acid that liberates oxygen, which acts as a bleaching agent (Mahmud and Anannya, 2021; Abdel, 2014).

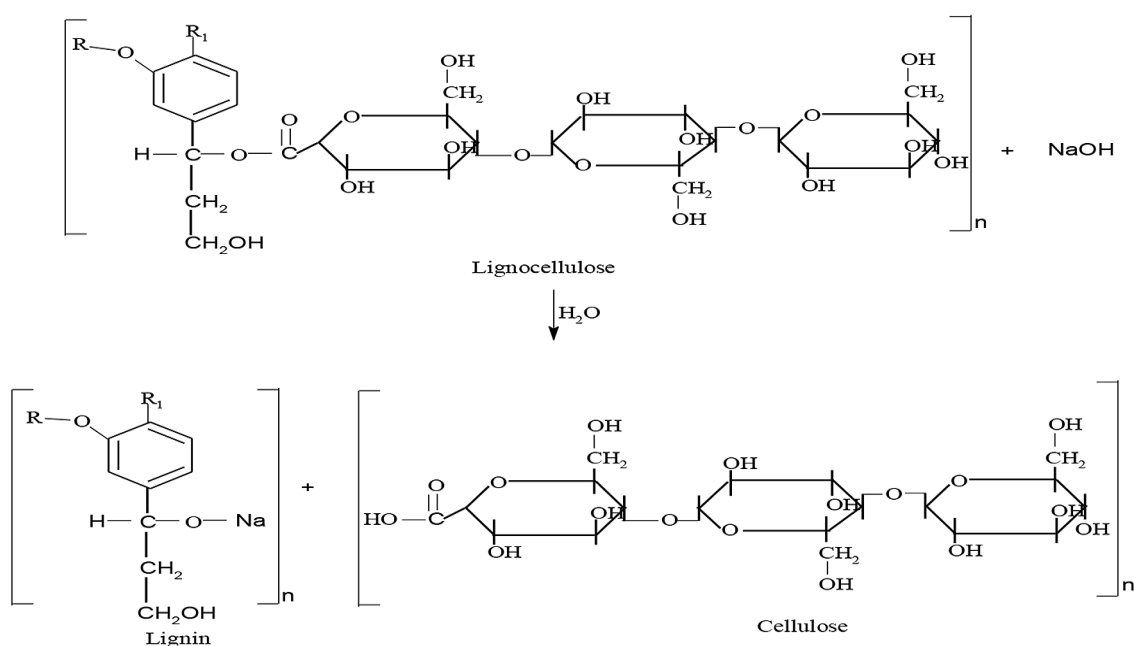


Figure 1. Bond breaking mechanism between lignin and cellulose (Soundarrajan et al., 2011).

To obtain high cellulose purity, a bleaching process is required. After that, acid hydrolysis using HNO_3 is carried out for bleaching to remove the amorphous part and the remaining lignin that still remains in the cellulose chain (Deepa et al., 2011). The discoloration process from bagasse to the resulting cellulose can be seen in Figure 2.

Characterization of bagasse cellulose

Infrared spectrum analysis

The FTIR spectra of cellulose are shown in Figure 3. The broad and strong absorption peaks located at wave numbers 3414 cm^{-1} and 3415 cm^{-1} are caused by the presence of O-H groups which are the main functional groups in cellulose. This peak is supported by the peaks at

wave numbers 1645 cm^{-1} and 1647 cm^{-1} caused by O-H bending vibrations. The broadening of the absorption peak occurs, possibly due to the presence of hydrogen bonds formed due to the presence of absorbed water (Abdel, 2014; Johar et al., 2012; Szymanska et al., 2019; Abraham et al., 2013). The absorption peaks at 2916 cm^{-1} and 2900 cm^{-1} are C-H stretching vibrations (Evans et al., 2019). The absorption peaks at wave numbers 1737 cm^{-1} and 1737 cm^{-1} indicate the presence of C=O vibrations of carboxylic acids. The absorption peaks at wave numbers 1460 cm^{-1} and 1433 cm^{-1} represent H-C-H bending vibrations, which are reinforced in the absorption peaks at wave numbers 1047 cm^{-1} and 1033 cm^{-1} are C-O stretching vibrations which are long chain linkers of β -glucose (Sheltami et al., 2012; Evans et al.,

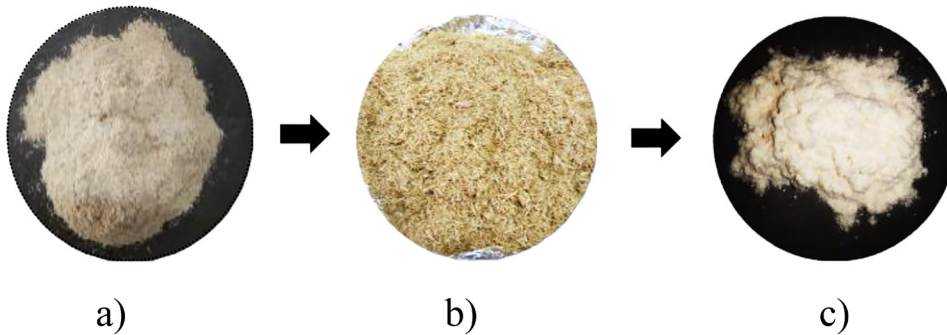


Figure 2. Color of (a) bagasse, (b) bagasse after delignification, (c) bagasse cellulose

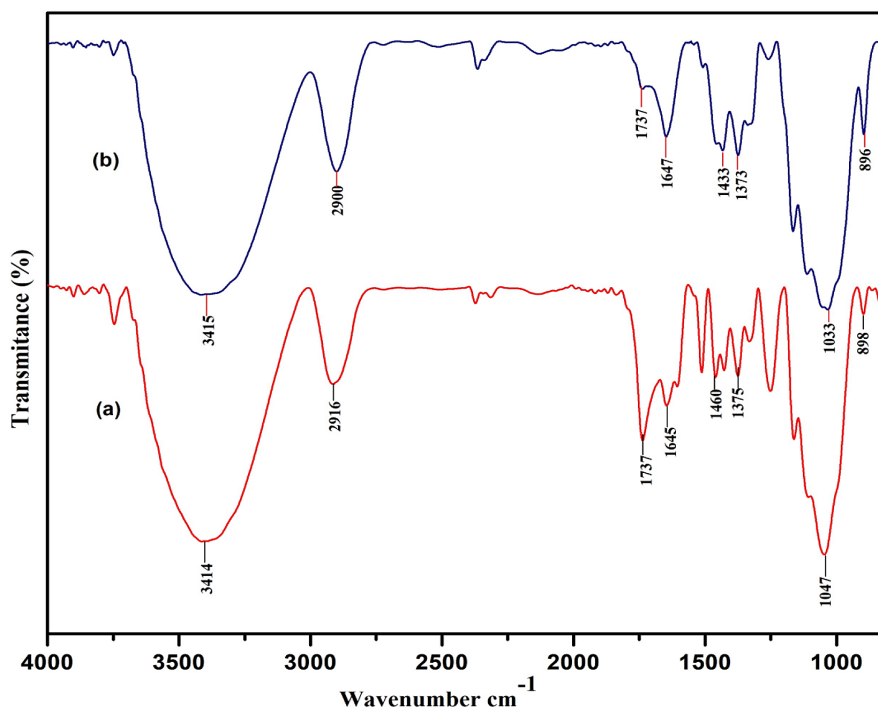


Figure 3. FTIR spectra of a) bagasse and b) bagasse cellulose

2019; Liu et al., 2005). The absorption peaks at wave numbers 898 cm^{-1} and 896 cm^{-1} indicate the formation of cellulose glycosidic bonds (Abraham et al., 2013). The peaks contained in the FTIR spectrum are the peaks generated from the cellulose spectrum. XRD diffractogram is used to prove that the result obtained from the extraction of bagasse is cellulose.

X-ray diffraction analysis

X-ray diffraction (XRD) analysis is used to determine the lattice parameters, the approximate size of crystals and the ratio of crystalline areas to amorphous areas (degree of crystallinity) (Sainorudin et al., 2018) in the sample can be seen in Figure 4, which shows that the sugarcane bagasse cellulose sample has crystalline areas and amorphous areas from the results of XRD analysis of sugarcane bagasse cellulose has three diffraction peaks, namely at $2\theta = 22.31^\circ$, 16.48° and 34.90° the peak is characteristic of cellulose and has a monoclinic structure (Klemm et al., 2005; Prieto et al., 2019; Thomas et al., 2013). The peak at maximum intensity found around $2\theta = 22.31^\circ$ corresponds to the (002) plane of the crystalline phase of cellulose. The peak at $2\theta = 16.48^\circ$ corresponding to the (110) plane and the peak at $2\theta = 34.90^\circ$ corresponding to the (004) plane are characteristic of cellulose as stated in References (Evans et al., 2019; Tibolla et al., 2014;

Rincon et al., 2022). The crystallinity index of cellulose obtained is 70.74%, this result is as described by Reference (Mzimela et al., 2020; Fauziyah et al., 2022; Sirvio et al., 2011).

Biofoam from bagasse cellulose with chitosan addition

The texture of biofoam produced from bagasse cellulose, chitosan and other additives can be seen in Figure 5. Biofoam without chitosan has a smooth surface, with a white color and solid. However, the higher the chitosan added, the rougher the surface of the biofoam produced, with colors varying from yellowish white to brownish white. The more chitosan added, the denser the biofoam structure produced, on the other hand, if the chitosan used is too little, the biofoam produced is more hollow. This is due to the expansion that occurs in the biofoam manufacturing process so that a hollow structure is obtained (Mathias et al., 2011; Obradovic et al., 2017).

Biofoam characterization

Density test

The effect of the addition of bagasse cellulose without chitosan and with the use of chitosan on the density of biofoam is shown in Figure 6. Biofoam without chitosan has a density of 1.21 g/mL while those using chitosan have a

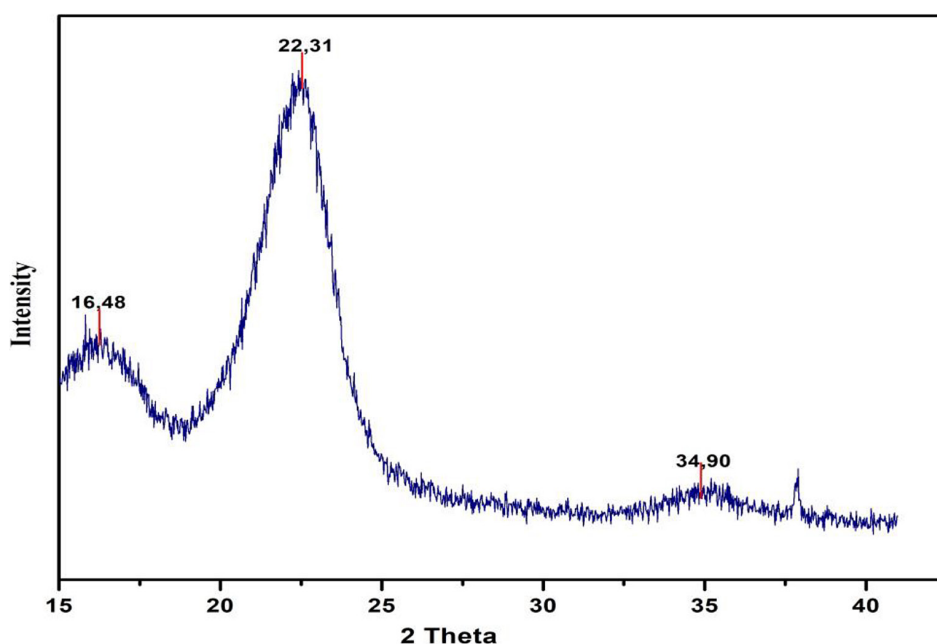


Figure 4. XRD diffractogram of bagasse cellulose

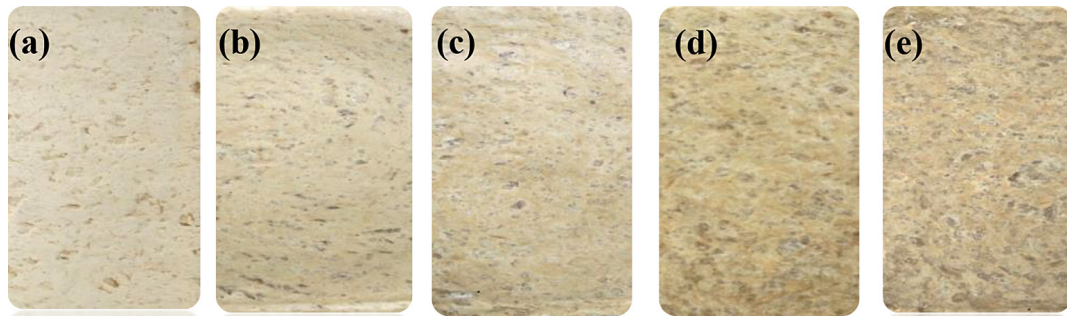


Figure 5. Texture of biofoam obtained from (a) cellulose (b) cellulose with 2 g chitosan added (c) cellulose with 3.5 g chitosan added (d) cellulose with 5 g chitosan added (e) cellulose with 6.5 g chitosan added.

density of 0.87–1.23 g/mL. When viewed from previous studies, the density of cellulose-based biofoam from rice straw, cotton and corn straw ranges from 0.22–0.96 g/mL (Sari, 2022; Mathias et al., 2011; Pan et al., 2023; Sanhawong et al., 2017). The standard density of Styrofoam is in the range of 0.90–1.10 g/mL (Indarti et al., 2021), so the density of biofoam sample without chitosan in this study is slightly higher than the range of biofoam density in general and is close to the standard density of Styrofoam. This is due to the inhibition of the expansion process resulting in a denser biofoam with a high density (Pan et al., 2023). However, the density of biofoam using chitosan falls within the range of biofoam in general and the standard density of styrofoam, namely the addition of 5 g chitosan with a density value of 0.91 g/mL. However, these variations are not only seen from the physical and mechanical properties but also from an economic point of view. Where biofoam with the addition of 2 g chitosan with a density value of 1.23 g/mL is considered more capable of reducing costs than biofoam with the addition of 5 g chitosan. The addition of chitosan affects the density of the resulting biofoam, where

the more chitosan is added, the lower the density value will be because when adding chitosan there will be times when an optimum point is reached which makes the biofoam density value decrease. This is because the more chitosan used the more uneven the interaction between the filler material and the matrix so that there are fillers that do not bind to the matrix (Sari, 2022). The higher density value means that it has a high density (Yunita et al., 2023), so that it can support biofoam from pressure and has a large pressure strength (Darni et al., 2023). Low density values can affect the decrease in mechanical strength values characterized by the brittle nature of biofoam, and many cavities.

Water absorption

Water absorption testing aims to determine the resistance of biofoam to direct contact with water (Majib et al., 2023). In Figure 7, shows the water absorption of biofoam without chitosan has a water absorption value of 96%, while biofoam with the addition of chitosan water absorption value decreased to 46% variation of 2 g chitosan. Biofoam without chitosan has higher water absorption because it has a mixture of starch which is hydrophilic and very

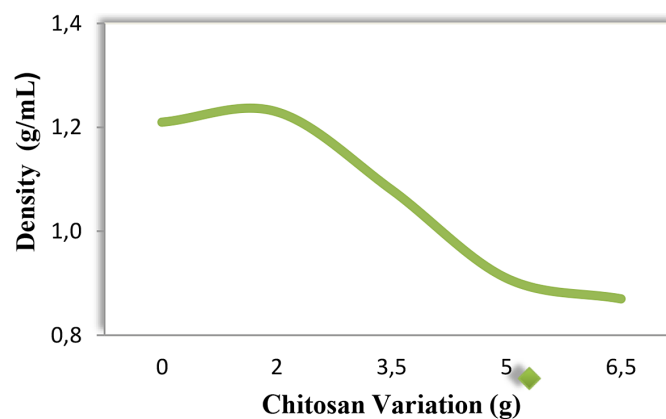


Figure 6. Relationship between chitosan addition (g) and biofoam density

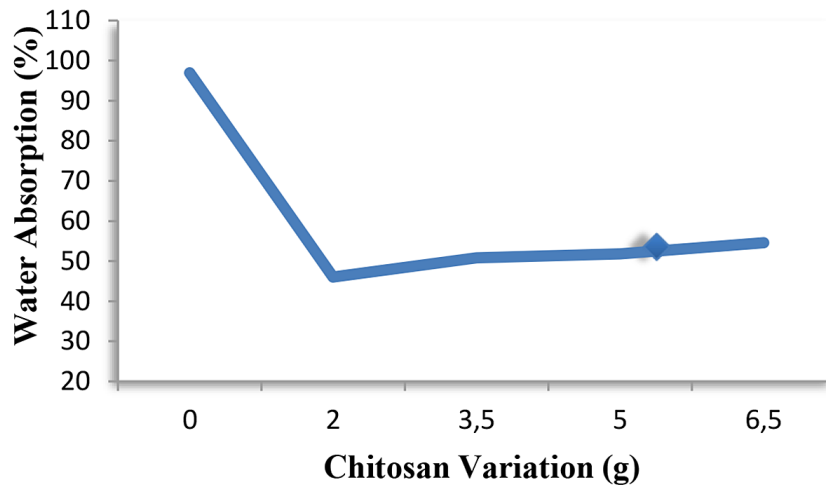


Figure 7. Relationship between chitosan variation (g) and water absorption (%) of biofoam.

easy to absorb water compared to other biopolymers (Soykeabkaew et al., 2015). Biofoam with the addition of chitosan is lower in water absorption because chitosan is a hydrophobic compound so that less water is absorbed to fill the cavity (Cahyani et al., 2023). In addition, due to the presence of NH_2^- groups in chitosan which will be protonated to NH_3^+ in acetic acid solution. Furthermore, NH_3^+ causes the molecule to bind OH^- through hydrogen bonds so that biofoam using chitosan is difficult to absorb water.

Biodegradability

The biodegradation test was conducted to determine the size of the sample decomposed by microorganisms in the soil (Darni et al., 2023; Indarti et al., 2021). This test was carried out by

soil burial method which is an effective technique to test the degradation of biofoam without chitosan and with chitosan addition. Degradation as a function of time through burial of the sample in the soil. It can be seen in Figure 8, which shows that the biofoam sample without chitosan lost 27.54% of its weight after being buried in the soil for 28 days, while the biofoam degradation decreased after the addition of 2 g chitosan lost 20.68% of its weight. Biofoam degradation began to increase upon the addition of 3.5 g, 5 g and 6.5 g chitosan, losing 25.22%, 26.95% and 34.38% weight respectively. The more chitosan used, the higher the degraded biofoam. This is likely due to the factor of imperfect homogeneity when mixing sugarcane bagasse waste cellulose with chitosan so that the biofoam is easily degraded by the soil. The

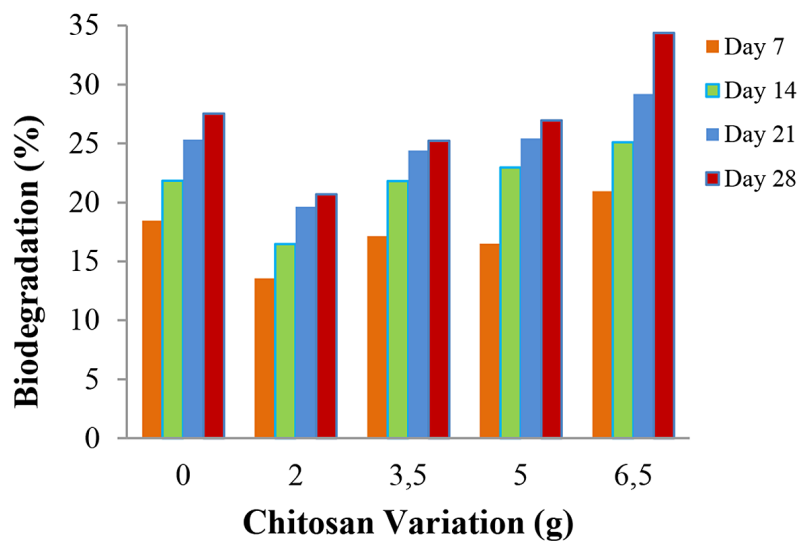


Figure 8. Relationship between time in the soil and the biodegradability of biofoam

inhomogeneous mixing process can cause holes and gaps that allow environmental variables such as water absorption in biofoam to occur more easily and accelerate the degradation process (Sari, 2022; Pan et al., 2023).

CONCLUSIONS

In this study, bagasse (*Saccharum Officinarum*) was used as a source of cellulose because it is a waste material, and the reuse of this material will be very helpful. Cellulose characterization data using FTIR instrument contained O-H, C-H and C-O functional groups, at wave numbers 3415 cm^{-1} , 2900 cm^{-1} , 1033 cm^{-1} , which indicated the presence of cellulose. In XRD analysis, the cellulose sample has a crystallinity index of 70.74%. Furthermore, cellulose was utilized as a base material for biodegradable foam with the addition of optimum chitosan mass of 2 grams, with a density value of 1.23 g/mL , water absorption value of 46.03% and biodegradation of 20.68%. The biofoam produced in this study is as expected by the researchers because it shows good characterization data. Researchers recommend the use of bagasse cellulose and chitosan in the industrial production of biofoam because both materials can improve the water absorption value of biofoam. As for the approach to the development of biofoam waste, it can be accepted by the environment because it is made of organic material so that it can decompose in the soil quickly.

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