JOURNAL OF ENERGY, MATERIALS, AND INSTRUMENTATION TECHNOLOGY

Journal Webpagehttps://jemit.fmipa.unila.ac.id/

Exploration the Influence of Ag+ Ion Solution Concentration Variations Coated on Biofoam Based on Cassava Starch and Corn Fiber on Crystallinity, Density, and Porosity

Pariaman Ginting, Sutiarno, Galang Haedi Wijaya, Pulung Karo Karo, Ediman Ginting Suka, and Agus Riyanto*

Department of Physics, University of Lampung, Bandar Lampung, Indonesia, 35141

Article Information

Article history:
Received November 2, 2022
Received in revised form
May 24, 2024
Accepted May 27, 2024

Keywords: *bio-foam, biopolymer, crystallinity, density, porosity*

Abstract

The bio-foam in this study was made from biopolymers in the form of cassava starch, cellulose from corn stalks, and a binder polymer in the form of polyvinyl alcohol (PVA). The bio-foam was molded using the thermo pressing method at T = 150 °C and pressed for t = 3 minutes. Ag^+ ions were added as an initial review of the physics properties of the bio-foam to serve as a reference for future antibacterial bio-foam manufacturing. Ag^+ ions were produced using the electrolysis method from AgBr rods with an electric voltage of 15 volts. Then, the electrolyzed solution containing Ag^+ ions was varied in concentration (12 ppm, 17 ppm, 22 ppm, 27 ppm). The ions were then coated on the bio-foam using the dip coating method and dried at room temperature. Along with the increase in the concentration of the Ag^+ ion solution coated on the bio-foam, there was an increase in the bio-foam crystallinity with test values of 34.4%, 40.8%, 41.2%, and 42.6%, respectively. Based on these data, the crystallinity value of the bio-foam does not change significantly. It also aligns with the density test values, which tend to be constant with an average test value of 0.322 g/cm³. Furthermore, the concentration of the solution containing Ag+ ions did not influence the bio-foam's porosity properties, where the four samples tended to remain with an average value of 5.9%.

Informasi Artikel

Proses artikel:
Diterima 2 November 2022
Diterima dan direvisi dari 24 Mei 2024
Accepted 27 Mei 2024

Kata kunci: *biofoam, biopolymer, densitas, kristalinitas, porositas*

Abstrak

Biofoam pada penelitian ini terbuat dari biopolimer berupa pati singkong, selulosa dari batang jagung, dan polimer pengikat berupa Polivinil Alkohol (PVA). Pembuatan biofoam tersebut dicetak menggunakan metode thermopressing dengan T=150 °C dan ditekan selama t = 3 menit. Ion Ag^+ ditambahkan sebagai tinjauan awal sifat fisika dari biofoam untuk menjadi referensi pembuatan biofoam antibakteri di masa depan. Ion Ag^+ diproduksi menggunakan metode elektrolisis dari batang AgBr dengan tegangan listrik 15 Volt. Kemudian, larutan hasil elektrolisis yang mengandung ion Ag^+ divarisasikan konsentrasi (12 ppm; 17 ppm; 22 ppm; 27 ppm). Ion tersebut kemudian dilapiskan pada biofoam menggunakan metode dip coating, lalu dikeringkan pada suhu ruangan. Seiring dengan penambahan konsentrasi larutan ion Ag^+ yang dilapiskan pada biofoam menunjukkan adanya kenaikan kristalinitas biofoam dengan nilai pengujian berturut-turut 34,4%, 40,8%, 41,2% dan 42,6%. Berdasarkan data tersebut, nilai kristalinitas biofoam tidak terlalu berubah signifikan. Hal tersebut juga selaras dengan nilai uji densitas yang cendrung konstan dengan nilai rata-rata pengujian 0,322 g/cm³. Berikutnya, Konsentrasi larutan yang mengandung ions Ag^+ juga tidak menujukkan adanya pengaruh terhadap sifat porositas biofoam dimana Keempat sampel cendrung tetap dengan rata-rata senilai 5,9%.

* Corresponding author.
E-mail address: agus.riyanto@fmipa.unila.ac.id

1. Introduction

Most people continue to use and produce plastic packaging, including styrofoam, which contains various harmful chemicals and is not environmentally friendly due to its prolonged decomposition time. Even its production generates substantial waste, leading the Environmental Protection Agency (EPA) to categorize it as the world's fifth-largest hazardous waste producer. One alternative to petroleum-based and synthetic polymers is natural polymers such as starch and chitosan (Nurfitasari, 2018). Polystyrene plastic packaging, commonly known as styrofoam, is a polymer that degrades into polystyrene monomers (Cavallo et al., 2018). Exposure to polystyrene, the main ingredient in styrofoam, can cause neurological disorders and decrease hemoglobin levels (Dowly et al., 1976).

Additionally, styrofoam is unsuitable for food packaging due to the migration of chemicals (Lickly et al., 1995). The environmental harm from styrofoam waste, which is difficult to decompose and leads to accumulation, exacerbates natural conditions. Considering the negative impacts of styrofoam packaging, it is crucial to find alternative packaging materials.

Potential materials for biopolymers derived from agricultural waste, such as starch and cellulose, are renewable, abundantly available, inexpensive, environmentally friendly, and safe for human health. These materials can also facilitate the 3Rs: reuse, reduce, and recycle existing styrofoam packaging. Bio-foam, a replacement for styrofoam, can be made from starch and fiber. Starch can be extracted from carbohydrate-rich plants such as sago, cassava, corn, wheat, and sweet potatoes, as well as from fruit seeds like jackfruit, avocado, and durian seeds (Yuliasih et al., 2012). Agricultural waste such as bagasse, empty fruit bunches of oil palm, pineapple leaf fiber, cassava peel, and corn stalks is often discarded without further processing. Utilizing this waste for bio-foam offers an alternative food packaging material that is safe for health and environmentally friendly (Rusliana et al., 2014).

Several researchers have explored bio-foam manufacturing using various materials and methods. For instance, Coniwanti et al. (2018) examined the effect of NaOH concentration and the ratio of pineapple leaf fiber to bagasse on bio-foam production using the thermopressing method, with a pressing time of 30 minutes at 170°C. The NaOH concentrations used were 0%, 2.5%, 5%, 7.5%, and 10%. The fiber ratios of pineapple leaves to bagasse were 100:0, 75:25, 50:50, 25:75, and 0:100. The best bio-foam characteristics were achieved with a 5% NaOH concentration and a 75:25 ratio of pineapple leaf fiber to bagasse, resulting in a tensile strength of 16.35%, compressive strength of 3.70%, water absorption of 15.60%, water content of 6.90%, and 4.49% biodegradability. Sipahuitar (2020) researched bio-foam made from durian seed starch and tea dregs cellulose nanofibers using the baking process method. A mixture of durian seeds and PVA served as the matrix, mixed with nano cellulose fiber from tea dregs in ratios of 0%, 1%, 3%, and 5% with PVA compositions of 10%, 20%, and 30%, then baked at 80 °C for 60 minutes. The highest tensile strength was found in a 3% tea dregs nanocellulose fiber composition and 10% PVA, measuring 5.647 MPa. The lowest water absorption and content were observed in a composition of 1% tea dregs nanocellulose fiber and 30% PVA, with values of 21.505% and 1.515%, respectively. The lowest density, 1.022 g/cm^3, was observed at a ratio of 0% tea dregs nanocellulose fiber and 30% PVA. The highest percentage of mass loss, 60.256%, occurred at a ratio of 5% tea dregs nanocellulose fiber and 10% PVA, with a degradation time of 28 days.

Based on these descriptions, researchers have investigated bio-foam made from different materials, specifically cassava starch and corn stalk fiber reinforced with PVA, using a thermopressing technique (simultaneous pressing and heating). Cassava starch is a promising raw material for bio-foam due to its high starch content (97%). In Indonesia, cassava production has increased since 2015, while public consumption has decreased, making it a viable starch source without affecting consumption levels. Corn stalk waste, containing 90% cellulose, is another potential bio-foam source. Its high cellulose content forms strong white, insoluble fibers in water and organic solvents and is white (Hauw, 2017). PVA, with a density of 1150 kg/m^3 and a tensile strength of 65-79 MPa, enhances the composite strength as bio-foam (Ritonga, 2019). This study added Ag+ ions, known for their potent antimicrobial properties, to the bio-foam. Silver nanoparticles, including pathogenic bacteria, are effective antimicrobial agents without known bacterial resistance. The smaller the silver particle size, the stronger the antimicrobial properties.

Bio-foam characterization tests for cassava starch and corn stalk fiber waste included XRD crystallinity, density, and porosity measurements. The corn stalk fiber and PVA mixture is expected to produce bio-foam with physical, mechanical, and thermal properties similar to styrofoam. This bio-foam offers the advantages of styrofoam, commonly used in the market, without its carcinogenic, toxic, and non-biodegradable properties. It can serve as alternative food packaging and reduce environmental pollution caused by styrofoam waste disposal.

2. Methods

The materials used in this study were tapioca flour, Pt Budi Starch (Commercial), PVA, AgBr rods, aqua dest, ethanol, 99% NaOH, and 50% H_20_2. Then, the tools used were an oven, filter paper, petri dish, balance, blender, hotplate, magnetic stirrer, measuring cup, bio foam mold, beaker glass, round Ose, ruler, mesh 100, power supply, and TDS Meter. The research procedure carried out in this study is as follows.

2.1 Corn Stem Insulation

According to research by Sumardiono et al. (2021), corn stem cellulose was obtained by grinding corn stalks to a size of 100 mesh. Then, it was cooked with 15% NaOH at 100 °C, with the ratio between the solution and corn stalk powder being 1:12 (w/v). The cooking precipitate was washed with distilled water until it was free from the NaOH solution. The bleaching process is followed by adding 10% H202 and heating at a cooking temperature of 100 °C for 2 hours. The precipitate resulting from bleaching was washed until the pH was neutral and dried at room temperature.

2.2 Preparation of Ag Ion Solution from AgBr Stem by Electrolysis Method

The preparation of Ag+ ion solution using the electrolysis method refers to research by Palupi and Suparno (2020), which used two silver rods as electrodes with a distance of 1 cm in 400 mL of distilled water. Electrolysis is done by flowing an electric current of 15 volts. Every 10 minutes, the electrodes were cleaned and sanded, and the concentration was measured with a TDS meter. It is done for up to 100 minutes. After that, the finished solution is put into a dark glass bottle. Then, the resulting solution was made into four samples with concentrations of 12 ppm, 17 ppm, 22 ppm, and 27 ppm.

2.3 Samples Molding

Printing uses the thermo-pressing method, referring to research by Hendrawati et al. (2020). The samples were made in 4 variations with the composition based on **Table 1**. The molding of bio-foam was carried out in 3 stages. First, tapioca starch, corn stalk cellulose, and polyvinyl alcohol were mixed with distilled water in 1:1 (w/v). Then, stir until it forms a homogeneous dough. Then, the dough was molded at a temperature of 100 ℃ for 3 minutes using a thermo-pressing tool modified by a glass sealer. It was then coated Ag+ ions on antibacterial bio-foam using the dip coating method, dipping the bio-foam into a solution of electrolyzed Ag+ ions for 1 minute and drying at room temperature for 24 hours.

Table 1. Variation of Biofoam Sample Composition

No	Sample Name	Tapioca Starch (%)	Corn Stem Cellulose (%)	Polivinil Alcohol (%)	Ion Ag+ Solution
1	Sample A	48	16	36	12ppm
2	Sample B	48	16	36	17ppm
3	Sample C	48	16	36	22ppm
4	Sample D	48	16	36	27ppm

2.4 Characterization

The characterizations carried out in this study include XRD analysis, density analysis, and porosity analysis.

1. XRD Analysis. The crystallinity of bio-foam was tested using XRD Bruker D8. The sample is cut according to the shape of the holder with a diameter of 4 cm and then shot with X-rays with a wavelength of K Cu = 1.54060 so that an X-ray diffraction image is obtained in the graph of the relationship between intensity and 2. The Segal equation calculates the determination of the crystallinity index as per research conducted by Etikaningrum et al. (2016).

$$Crl\ (\%)\ =\ lc\ /\ (lc + l\alpha)\ \times\ 100\ \% \tag{1}$$

2. Density Analysis. The density of bio-foam is determined by cutting the sample with a size of 2 cm × 2 cm, after which the thickness of the sample is measured using a caliper, and then the volume is calculated. The pieces of bio-foam were weighed, and the density of the bio-foam was determined by dividing the mass of the test pieces (Mk) by their volume (V). After the test data was obtained, **Eq. 2** was used.

$$\rho = \frac{M_k}{V} \tag{2}$$

3. Porosity Analysis. The bio-foam's porosity was determined by cutting the sample to a size of 2 cm x 2 cm after measuring the thickness of the sample using a caliper and then calculating the volume. The pieces of bio-foam were weighed, and the dry weight was recorded. After that, the sample was immersed in water for 1 minute, then weighed, and the wet weight was recorded. Then, the porosity value was obtained using **Eq 3**.

$$\%P\ = \frac{(M_b - M_k)}{\rho_{air} V_t}\ \times\ 100\ \% \tag{3}$$

3. Results and Discussions

3.1 XRD Crystallinity Analysis Results

The crystallinity results in the bio-foam research were significant because they showed the regularity of bonds between molecular chains and materials. The diffractogram graph of the bio-foam sample is shown in **Figure 1**. The bio-foam crystallinity value in this study ranged from 34.6% to 42.6% (**Table 2**). The results of the bio-foam crystallinity test were in line with the research of Etikaningrum et al. (2016).

Table 2. Bio-foam crystallinity results

No	Sample Name	Ag	Crystallinity
1	A	12 ppm	34.4 %
2	B	17 ppm	40.8 %
3	C	22 ppm	41.2 %
4	D	27 ppm	42.6 %

Based on the crystallinity test results, it was concluded that adding Ag ions increased the crystallinity of bio-foam. According to Etikaningrum et al. (2016), the increase in crystallinity values was also due to the delignification

treatment and the addition of fiber, which enhanced the bio-foam's crystallinity because cellulose had a larger crystalline area than starch. The crystallinity value was closely related to the intermolecular forces between the materials mixed in bio-foam production. A high crystallinity value strengthened the bio-foam under various pressures when the material was used as food or beverage containers (Etikaningrum et al., 2016).

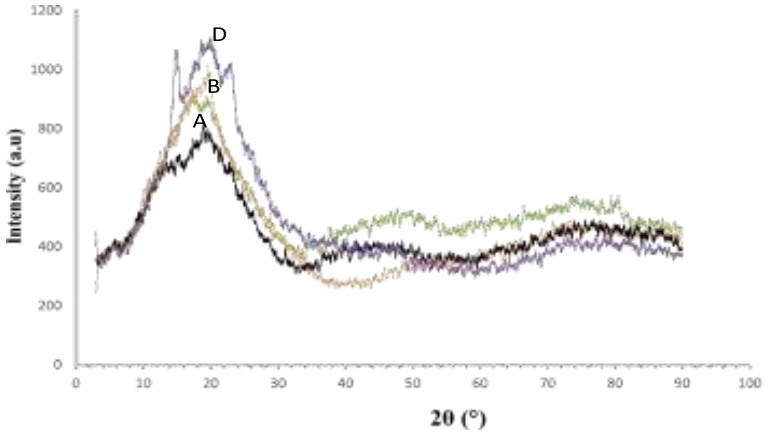

Figure 1. Difactogram sample of bio-foam

The higher the Ag+ concentration, the higher the crystallinity value of the bio-foam. These results align with the research conducted by Etikaningrum et al. (2016) and Parasmayanti (2014), which showed that zinc oxide nanoparticles in bio-foam made from natural rubber latex increased the crystalline portion. Parasmayanti (2014) also supported this finding by stating that silver, a metal with very high crystallinity and conductivity, increases the material's conductivity as its crystallinity increases. Additionally, the longer the sonication time, the more conductive the material becomes due to the PANI-Ag/Ni Thin Film becoming more crystalline.

3.2 Density Analysis Results

The bio-foam sample density test results in this study are shown in **Table 3** and calculated by **Eq. 1**, which produces a graph like **Figure 2**.

Table 3. Biofoam Density Test

No	Sample Name	Densitas Mass (g)	Calculations Volume
1	Sample A	0.341	0.322
2	Sample B	0.353	1.052
3	Sample C	0.338	1.044
4	Sample C	0.319	1.032

From **Figure 2**, it can be seen that there was not a significant change in the density test values. The results for sample A, with a concentration of Ag+ at 12 ppm, had a density of 0.322 g/cm³. For sample B, with a concentration of Ag^+ at 17 ppm, there was an increase in density to 0.335 g/cm³. However, samples C and D, with Ag^+ concentrations of 22 ppm and 27 ppm, showed a decrease in density to 0.323 g/cm³ and 0.309 g/cm³, respectively. From the data above, it can be concluded that the Ag^+ concentration did not affect the bio-foam density value. The density test results in this study ranged from 0.323 to 0.335 g/cm³.

Compared to commercially available styrofoam, which has a density of 0.03 to 0.12 g/cm³ (Marlina et al., 2021), these values are still relatively high. The density value of bio-foam indicates that a lower test value corresponds to lower starch expansion, making the bio-foam less water-absorbent, which can reduce the water absorption capacity of the bio-foam (Berutu et al., 2022). Thus, the lower the density value of the bio-foam, the better it is as a potential replacement for commercial styrofoam. When compared to previous research, such as Marlina et al. (2021), whose research on bio-foam made from paper fiber and orange peel waste had densities ranging from 0.18 to 0.48 g/cm³; Etikaningrum et al. (2016) with bio-foam made from oil palm empty fruit bunches fiber, ranging from 0.16 to 0.28 g/cm³; and Debiagi et al. (2011) with bio-foam made from starch, PVA, chitosan, and sugarcane stem fiber, ranging from 0.20 to 0.304 g/cm³; the density values in this study were still within the available range.

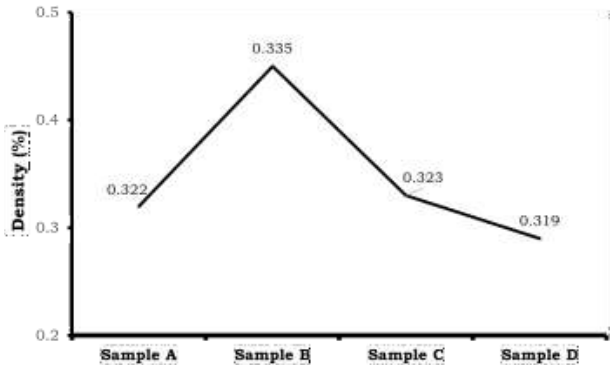

Figure 2. Bio-foam density test results

3.3 Porosity Analysis Results

Table 3 shows the results of the porosity testing of the bio-foam samples in this study. The porosity was calculated using **Eq. 2**, resulting in the porosity graph shown in **Figure 3**.

Table 4. Porosity calculation data

No	Sample Name	Porosity Calculation Initial Weight (g)	Final Weight (g)	Volume (cm^3)
1	A	0.341	O.397	1.056
2	B	0.353	0.411	1.052
3	C	0.338	0.412	1.044
4	D	0.379	0.319	1.032

Based on **Figure 3**, the change in the porosity value of bio-foam did not indicate a significant change. The four samples tended to stay at an average of 5.9%. It indicated that the bio-foam samples in this study met the established Indonesian National Standard (SNI). Adding Ag+ ions to the bio-foam did not affect the porosity value. The porosity of bio-foam had an inverse relationship with density. The higher the density value, the lower the number of cavities in the bio-foam. It made the bio-foam less prone to water infiltration, thus reducing the water absorption capacity of the resulting product (Soykeabkaew et al., 2004).

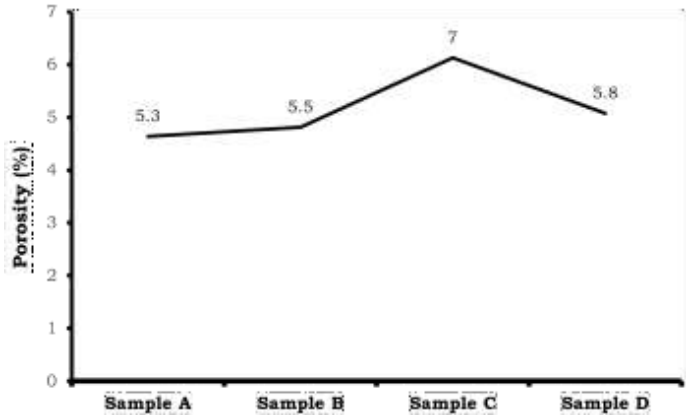

Figure 3. Graph of Porosity analysis data results.

According to Iriani and Soykeabkaew et al. (2019), the morphological structure affected bio-foam density and water absorption. Structures with large and many cavity sizes and high porosity produced bio-foam with low density because the cavities generally formed had thin pore walls, which broke quickly under pressure, and vice versa. Furthermore, according to Soykeabkaew et al. (2019), the higher the porosity of the bio-foam, the more water was absorbed to fill the cavities. Hence, the water absorption capacity of the bio-foam tended to increase. It followed the obtained density value, which tended to be lower, while the absorption and porosity tended to be high. Based on the information from the review conducted by the authors, bio-foam with good crystallinity should be able to increase the density value of the bio-foam. It significantly reduces the porosity value, making the bio-foam less fragile and less water-absorbent.

4. Conclusions

Based on the results, a higher concentration of Ag+ ion solution led to a higher crystallinity value of the bio-foam, but the effect was insignificant. It also aligns with the density test values, which tend to be constant with an average test value of 0.322 g/cm^3. Furthermore, the concentration of the solution containing Ag+ ions did not influence the bio-foam's porosity properties, where the four samples tended to remain with an average value of 5.9%.

Based on the data, the crystallinity value of the bio-foam did not change significantly. It was also consistent with the density test values, which tended to remain constant.

5. Bibliography

Abdullah, A.H.D., Chalimah, S., Primadona, I., & Hanantyo, M.H.G. (2018). 'Physical and Chemical Properties of Corn, Cassava, and Potato Starchs,' in IOP Conference Series: Earth and Environmental Science. *Institute of Physics Publishing*, pp. 1–7. doi:10.1088/1755-1315/160/1/012003.

Abraham, E., Deepa, B., Pothan, L.A., Jacob, M., Thomas, S., Cvelbar, U., & Anandjiwala, R. (2011). 'Extraction of nanocellulose fibrils from lignocellulosic fibers: A novel approach,' Carbohydrate Polymers, 86(4), pp. 1468–1475. doi:10.1016/j.carbpol.2011.06.034.

Aditama, A.G., & Ardhyananta, H. (2017) 'Isolasi Selulosa dari Serat Tandan Kosong Kelapa sawit untuk Nano Filler Komposit Absorpsi Suara: Analisis FTIR', *Jurnal Teknik ITS*, 6(2), pp. 1–4.

Akmala, A. & Supriyo, E. (2020) Optimasi Konsentrasi Selulosa pada Pembuatan Biodegradable Foam dari Selulosa dan Tepung Singkong. *Pentana: Jurnal Penelitian Terapan Kimia*, 1(1), 27-40

Boediono, M.P. (2012) *Pemisahan dan Pencirian Amilosa dan Amilopektin dari Pati Jagung dan Pati Kentang pada Berbagai Suhu*. Bogor Agricultural University (IPB). Available at: http://repository.ipb.ac.id/handle/123456789/58239.

Bourtoom, T. (2007). *Effect of Some Process Parameters on The Properties of Edible Film Prepared from Starch*. Available at: http://vishnu.sut.ac.th/iat/food_innovation/%0Aup/rice starch film.doc.

Cavallo, D., Tranfo, G., Ursini, C. L., Fresegna, A. M., Ciervo, A., Maiello, R., Paci, E., Pigini, D., Gherardi, M., Gatto, M. P., Buresti, G., & Iavicoli, S. (2018). Biomarkers of Early Genotoxicity and Oxidative Stress for Occupational Risk Assessment of Exposure to Styrene in the Fibreglass Reinforced Plastic Industry. *Toxicology Letters*, 298, 53–59. https://doi.org/10.1016/j.toxlet.2018.06.006

Coniwanti, P., Mu, R., Saputra, H. W., & Andre, M. R. A. (2018). Pengaruh konsentrasi NaOH Serta Rasio Serat Daun Nanas dan Ampas Tebu Pada Pembuatan Biofoam. *Teknik Kimia*, 24(1), 1–7.

Etikaningrum, Hermanianto, J., Iriani, E.S., Syarief, R. & Permana, A.W. (2016). Pengaruh Penambahan Berbagai Modifikasi Serat Tandan kosong Sawit Pada Sifat Fungsional Biodegradable Foam. *Jurnal Penelitian Pascapanen Pertanian*, 3(3), 146–155.

Nurfitasari, I. (2018). *Pengaruh Penbahan Kitson dan Gelatin Terhadap Kualitas Biodegradable Foam Berbahan Baku Pati Biji Nangka (Artocarpus Heterophyllus)*. Thesis. Universitas Islam Negeri Alauddin Makassar

Parasmayanti, F. (2014). *Pengaruh Lama Sonikasi Pada Pembuatan Film PANI-Ag/Ni Terhadap Kristalinitas dan Konduktvitasnya*. Thesis. Universitas Negeri Malang.

Ritonga, A. U. M. (2019). *Pembuatan dan Karakterisasi Biofoam Berbasis Komposit Serbuk Daun Keladi Yang Diperkuat Oleh Polivinil Asetat (PVAc)*. Thesis. Sumatera Utara University.

Rusliana, E., Saleh, M., & Assagaf, M. (2014). Penentuan Kodisi Proses Terbaik Pembuatan Biofoam Dari Limbah Pertanian Lokal Maluku Utara. *Seminar Nasional Sains Dan Teknologi 2014*, November, 10–13.

Singh, A. & Bishnoi, N.R. (2012). Enzymatic Hydrolysis Optimization of Microwave Alkali Pretreated Wheat Straw and Ethanol Production By Yeast. *Bioresource Technology*, 108, pp. 94–101. doi:10.1016/j.biortech.2011.12.084.

Singh, R.K. & Singh, A.K. (2013). Optimization of Reaction conditions for Preparing Carboxymethyl Cellulose from Corn cubic Agricultural Waste. *Waste and Biomass Valorization*, 4(1), pp. 129–137. doi:10.1007/s12649- 012-9123-9.

Sipahuitar, B. K. syahputra. (2020). *Pembuatan Biodegradable Foam (Durio Zibethinus) Dan Nanoserat Selulosa Ampas Teh (Camelia Sinentis) Dengan Proses Pemanggangan*. Universitas Sumatera Utara. http://repositori.usu.ac.id/handle/123456789/25568

Soykeabkaew, N., Supaphol, P., & Rujiravanit, R. (2004). Preparation and characterization of jute and flax reinforced starch-based composite foams. *Carbohydrate Polymers* 58: 53-63.

Wahyudi, T., Sugiyana, D. & Helmy, Q. (2011). Sintesis Nanopartikel Perak dan Uji Aktivitasnya Terhadap Bakteri Ecoli dan S.aureus. *Arena Tekstil*, 26(1), pp. 1–60.