

Mekanika: Majalah Ilmiah Mekanika

Effect of Alkaline Treatment and Fumigation (Fumigation) on the Mechanical Properties of Fiber Unsaturated Polyester-Cantala Composite with Compression Molding Method

Muhamad Saifuddin Salim¹, M. Rafidya Bintang Ramadhan², Elvira Wahyu Arum Fanani², Dody Ariawan^{2*}, Eko Surojo²

1 School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia, Nibong Tebal 14300, Malaysia

2 Department of Mechanical Engineering, Sebelas Maret University, Surakarta 57126, Indonesia

*Corresponding Author's email address: dodyariawan@staff.uns.ac.id

Keywords:

Cantala fiber
Microcrystalline cellulose
UPRs

Abstract

This research examines the strength of the Unsaturated Polyester Resins (UPRs)-Cantala composite with the addition of filler Microcrystalline Cellulose (MCC). Composites were created with a Volume Fraction (VF) of 30% and a 45° angle. This angle variation received the same treatment as the others, including untreated, alkaline, and fumigation. The treatment time for alkali treatment was six hours, while the treatment time for fumigation was 10 hours. The strength of each angle variation was determined, as well as its treatment of tensile strength, modulus of elasticity, and Poisson ratio UPRs-Cantala composites. According to the results, the alkali treatment produced the highest tensile strength and modulus of elasticity values. The highest Poisson ratio value was discovered without treatment at a 45°. The alkaline treatment yielded the highest tensile strength and modulus of elasticity test results. The pullout fiber fracture dominated the untreated composite fracture, whereas the fiber breakage fracture dominated the alkaline treatment.

1 Introduction

There are many developments in alternative materials in manufacturing and support for the development of Science and Technology (*Ilmu Pengetahuan dan Teknologi-IPTEK*), one of which is in composite material innovation. A Polymer Reinforced Fiber (PRF) is a composite material of a polymer matrix and high-strength fibers [1]. A composite is a material of two parts: reinforcement and matrix. The combination of these components results in unique mechanical and thermal properties that a single material cannot achieve [2,3]. Environmental concerns lead to environmentally friendly and renewable materials like biocomposites. Biocomposites are materials made from environmentally friendly matrices and reinforced with natural fibers. Natural fibers are derived from plants or animals with advantages over synthetic fiber composites, such as good thermal insulation, high specific strength, environmental friendliness, sustainability, and stiffness properties due to low density [4,5]. Natural fibers are a potential replacement for glass fibers in composite materials due to these advantages. The thermoset is a polymer matrix that is currently in use.

<https://dx.doi.org/10.20961/mekanika.v21i2.51493>

Revised 30 July 2022; received in revised version 10 August 2022; Accepted 21 August 2022
Available Online 30 September 2022

2579-3144

© 2022 Mekanika: Majalah Ilmiah Mekanika. All right reserved

Salim et al.

Unsaturated polyester, epoxy, and phenol are the most commonly used thermoset polymers [6]. Cantala fibers are natural fibers that can be used in bio-composites. Agave cantala is a plant in the Asparagaceae family. The plant has been cultivated in the Philippines since 1783 and is currently growing in Indonesia [7]. They are widely available, inexpensive, have good mechanical properties, low density, are non-abrasive during processing, and contain cellulose [8]. Cantala fiber has a high potential as a composite reinforcing material due to its high cellulose content of approximately 64.23% [9]. The unsaturated polyester matrix is made up of two polymers: a linear polymer with an ester bond and a polymer with an unsaturated double bond [10]. Unsaturated polyester has the advantages of being easy to process, having good mechanical properties, and being inexpensive [11]. Cellulose is a compound that influences natural fiber polymer composites. Microcrystalline cellulose is a promising natural material derived from native cellulose that could be used to create nanocomposite or polymer composite materials [12]. Microcrystalline Cellulose (MCC) is pure cellulose isolated from alpha-cellulose using mineral acids that has the advantages of being renewable, having high mechanical properties, being free of toxins, and being biocompatible [13]. Microcrystalline cellulose can improve composite material properties and characteristics [14]. It is well known that alkali treatment and the addition of microcrystalline improved flexural strength and modulus of elasticity, similar to the study conducted by Sakuri et al. [15].

The fiber and matrix adhesion bond influences the mechanical properties of polymer composite fiber reinforcement. Fumigation is a technique used to reduce the moisture content of fibers, causing the surface to roughen and the mechanical properties of composites to improve [16]. Palungan et al. [17] investigated the effects of fumigation on king pineapple leaf fiber hemicelluloses and lignin content, causing the fiber surface to become rough and grooved as the water content decreased and fiber density increased. Alkali treatment is another method for influencing the adhesion bond in polymer composites. Alkali treatment can increase the tensile strength of a composite by increasing the bond between the fiber and matrix by removing lignin and hemicellulose in natural fibers [18]. Figure 1 shows natural fiber's simplified lignin and hemicellulose [19].

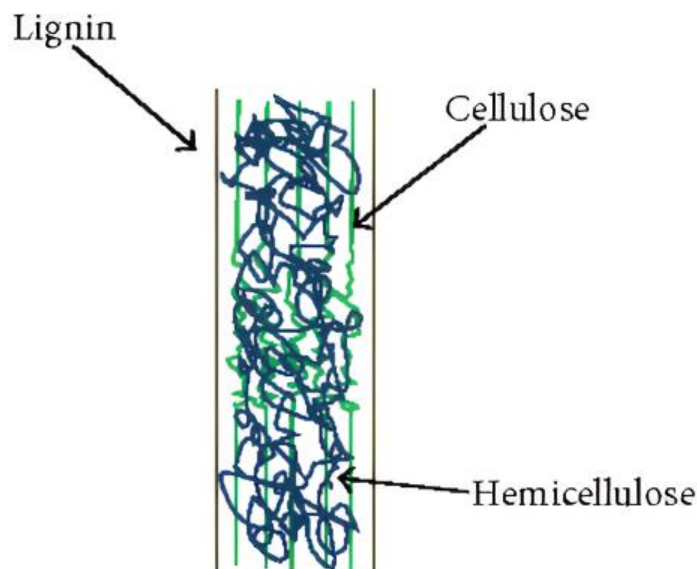


Figure 1. Lignin and hemicellulose in natural fiber [19]

The ability of a composite is highly dependent on its mechanical properties and the relative content of the reinforcing material and the matrix material. The composite also depends on many parameters, including fiber orientation [20]. Based on this, a composite study of the effect of alkaline treatment and fumigation with the addition of MCC on the tensile strength and Poisson ratio of unsaturated polyester composites reinforced with Cantala fiber is required description. The treatment included six hours of 6% concentration (NaOH) and 10 hours of fumigation. By varying the angle by 45°.

Salim et al.

2 Experimental Methods

2.1 Materials

1. Cantala Fiber

One of Indonesia's many natural fibers is agave Cantala. Indonesia first encountered agave Cantala in the nineteenth century. Agave Cantala is characterized by its bluish-gray color, densely pointed spiny leaves, excellent drought resistance, and relatively low fiber output [21]. The extraction method is used to obtain fiber from the agave Cantala plant. During the extraction process, the leaves to be extracted are clamped with a unique tool and then pulled at one end to separate the fibers [22]. Cantala fiber from Pengasih Village, Kulon Progo District, Yogyakarta Special Region Province is shown in Figure 2.



Figure 2. Cantala fiber

2. Unsaturated Polyester

Unsaturated Polyester of the Ortho-Phthalic type, YUKALAC 157-BQTN EX, is used as a binder between fibers and is available from PT Justus Sakti Raya.

3. Microcrystalline Cellulose (MCC)

Microcrystalline cellulose is a promising natural material derived from natural cellulose that could be used to create nanocomposites or polymer composites [11]. Sigma-Aldrich Pte Ltd Singapore supplied the microcrystalline cellulose. Initially, MCC was widely used in the food, cosmetic, and medical industries, among others. MCC was then widely used as a reinforcing agent in polymer composites because of its low cost, low density, and low abrasion to equipment [13].

Coconut husks similar to those in Figure 3 were purchased from Pasar Palur, located at Jl. Raya Terminal Palur in Banaran, Ngringo, Kec. Central Java, Karanganyar Regency, Central Java. The smoking procedure will make use of this coconut shell.



Figure 3. Coconut husk

Salim et al.

2.2 Fabrication

For six hours, the Cantala fibers were immersed in an alkaline solution composed of distilled water and NaOH crystals at a concentration of 6%. The Cantala fibers were then immersed in a 1% acetic acid solution and rinsed with distilled water until clean, and the pH reached 7. The Cantala fibers were then dried in an oven for 10 hours at 60 °C. After that, cut it into 10 cm pieces. A fumigation process for Cantala fibers is carried out for 10 hours while maintaining the fumigation temperature of 40-60 °C. The fiber is then cut to a length of 10 cm. The input of all compositions, including Cantala fibers, unsaturated polyester matrices, Methyl-Ethyl-Ketone-Peroxide (MEKPO) catalyst (1% of total volume fraction of unsaturated polyester), and MCC (5% of total volume fraction of unsaturated polyester), into the mold, is the first step in the creation of composites. The fibers are then arranged at a 45°. After that, print the composite using the compression molding technique at room temperature for 12 hours with a pressure of 10 MPa. The composite was then placed in the oven for a two-hour post-cure at a temperature of 60 °C. The composite was then cut following American Society for Testing and Material (ASTM) D638. One chemical treatment that affects surface roughness in a composite is Cantala fiber fumigation. The fumigation process lasts 10 hours and is carried out with smoke generated by burning coconut fibers. Figure 4 displays the equipment used in the fumigation procedure. The temperature used in the fumigation process ranges from 40-60 °C to generate a large amount of smoke while not heating the Cantala fibers. Cantala fiber was alkali-treated by immersing it in a NaOH solution for six hours. Cantala fiber was washed in a 1% CH₃COOH solution to remove any treatment traces.



Figure 4. Equipment for the fumigation process

Cantala fiber, unsaturated polyester resin, catalyst, and Microcrystalline Cellulose (MCC) were weighed with a volume fraction of 30%, matrix volume fraction of UPRs 70%, and MCC added at 5%. The Cantala fiber must be inserted into the mold. Before inserting the mold, the top and bottom must be waxed to prevent sticking. Then, until the mixture is evenly distributed, pour the matrix composition of unsaturated polyester resin, catalyst with a composition of 1%, and microcrystalline cellulose with a composition of 5% of the total volume fraction of unsaturated polyester into the mold. Following that, an emphasis of 10 kg/cm² was applied for 12 hours. The specimens are placed in a 50 °C oven for 12 hours when the process is finished.

2.3 Testing method

Tensile strength is the mechanical property investigated in this study. Tensile testing is performed following ASTM D638. Figure 5 shows the JTMUTS510 machine used in the Engineering Materials Laboratory, Mechanical Engineering Study Program, Faculty of Engineering, Sebelas Maret University.



Figure 5. Universal testing machine

3 Results and Discussion

The tensile strength and modulus of elasticity of the UPRs - Cantala composite can be seen in Figures 6 and 7. The values for both the tensile strength and the modulus of elasticity have comparable values. The alkali treatment has the most significant tensile strength value and modulus of elasticity of 23.30 MPa and 3.51 GPa. Then fumigation treatment has a tensile strength value and modulus of elasticity of 13.05 MPa and 3.106 GPa. Without treatment, the tensile strength and modulus of elasticity were the smallest compared to the alkali and fumigation treatment, with values of 11.38 MPa and 3.047 GPa.

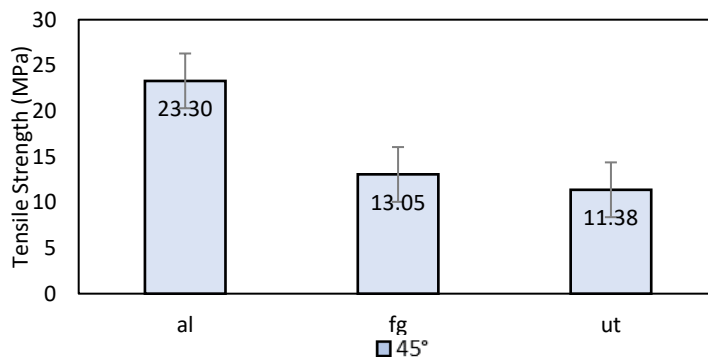


Figure 6. Tensile strength on 45° orientations of UPRs-Cantala composite fibers

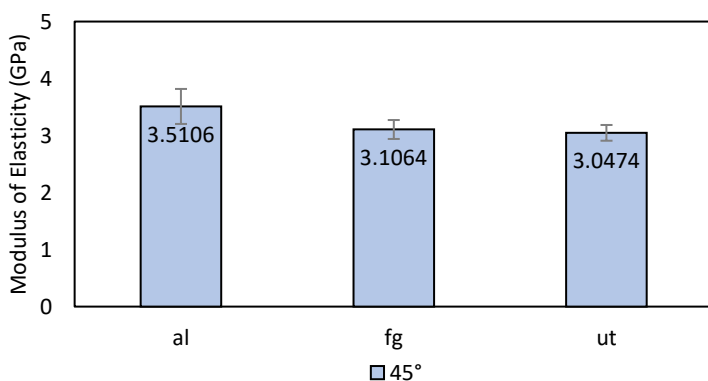


Figure 7. Modulus of elasticity on 45° orientations of UPRs-Cantala composite fibers

Figures 6 and 7 show that the alkali treatment has the greatest tensile strength and modulus of elasticity. This is because natural fibers have high hemicellulose, pectin, and lignin that can cause mechanical properties to decrease correctly, and one way to improve is by alkaline treatment [23]. The alkaline treatment also aims to reduce the hydrophobic properties that cause the optimal hydrophobic interfacial with thermoset or thermoplastic matrices to be more optimal [24].

Table 1. Poisson ration value on UPRs - Cantala composite

Treatment	ϵ_x	ϵ_y	V_{xy}
UT 45°	0.0046	0.0118	0.389
AL 45°	0.0048	0.0131	0.366
FG 45°	0.0042	0.0111	0.378

Poisson ratio (V_{xy}) value was obtained after conducting a strain gauge test where the results obtained were a comparison of the value of strain in the direction of the lateral axis (ϵ_x) to the axial axis (ϵ_y) [25]. Table 1 shows that the results of alkaline treatment have the lowest Poisson ratio value compared to no treatment or fumigation. This is because the alkaline treatment for six hours will cause the amorphous area in the UPRs-Cantala composite to disappear, causing the value of the hybrid strain to decrease and will result in the value of the Poisson ratio also decreasing [26]. Meanwhile, it can also be seen from the data above that the UPRs-Cantala composite without treatment had the highest Poisson ratio value compared to alkaline and fumigation treatment. This is because the fibers in the untreated composites still have a lot of amorphous properties and can withstand more loads, so the strain value increases, and the value of the compound Poisson ratio increases [27].

Figures 8 and 9 show the results of a Scanning Electron Microscopy (SEM) photo of the fracture of an unsaturated polyester polymer composite with an angular orientation of 45° without treatment and alkali treatment. In the alkaline treated fiber with an angle orientation of 45°, it can be seen that the bond between the fiber and matrix is better than in the untreated composite. In Figure 4, it can also be seen that the composite has a fiber breakage fracture, and there is a tight bond because the lignin and hemicellulose content in the fiber has disappeared. The alkaline treatment also improves the bond between the fiber and the matrix to be more optimal [28]. Sakuri et al. [15] have also carried out these results regarding observing the microscopic Cantala fiber. In the observation research, untreated Cantala fibers still have a smooth surface, and the content of lignin, hemicellulose, and other impurities is still visible. Meanwhile, the Cantala fiber with alkaline treatment shows that lignin, hemicellulose, and other impurities have started to thin out and effectively make the surface of the Cantala fibers broader and coarser, which forms a better bond with the matrix.

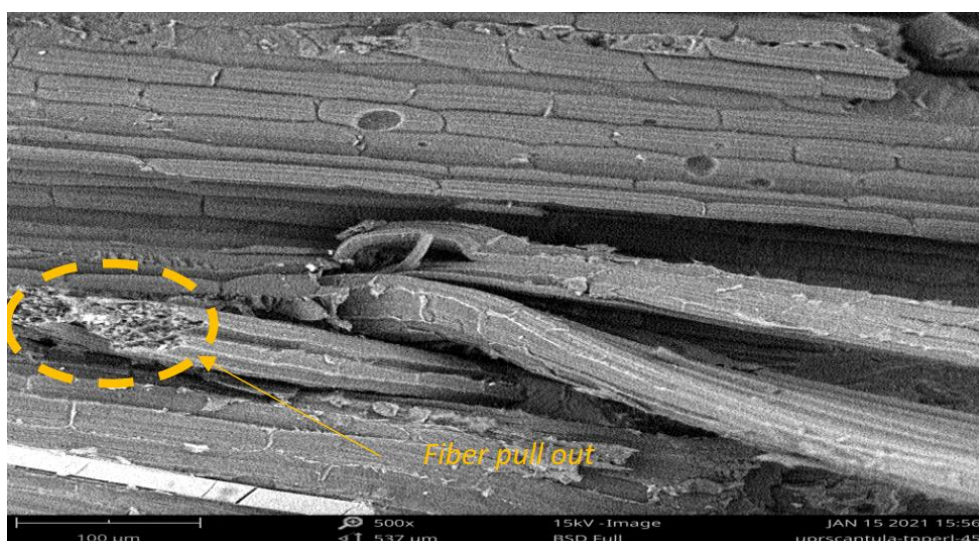


Figure 8. Composite fracture observation untreatment

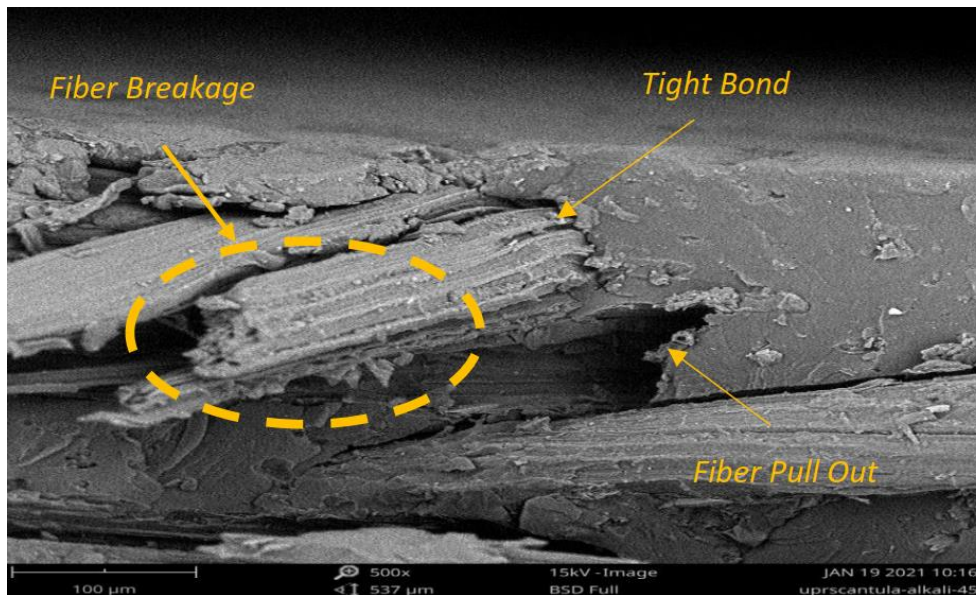


Figure 9. Composite fracture observation alkali treatment

4 Conclusion

According to the findings of the research and discussion of unsaturated polyester composites with Cantala reinforcement, the alkali treatment had the highest tensile strength and modulus of elasticity at an orientation angle of 45° . In contrast, the untreated had the lowest value of 1.5 GPa. Compared to fumigation and no treatment, alkali treatment can increase tensile strength and modulus of elasticity. The UPRs-Cantala composite's Poisson ratio reached its maximum value with no treatment and 0.389. The fiber pulls out fracture type predominated in the outcomes of the untreated UPRs-Cantala composite fracture. At the same time, numerous types of fiber breaking in the alkali-treated composite are breaking.

References

1. H. Ku, H. Wang, N. Pattarachaiyakoop, and M. Trada, "A review on the tensile properties of natural fiber reinforced polymer composites," *Compos. Part B Eng.*, vol. 42, no. 4, pp. 856-873, 2011.
2. R. Rahman and S. Z. F. S. Putra, *Tensile properties of natural and synthetic fiber-reinforced polymer composites*. Amsterdam: Elsevier Ltd, 2018.
3. E. W. A. Fanani, E. Surojo, A. R. Prabowo, and H. I. Akbar, "Recent progress in hybrid aluminum composite: Manufacturing and application," *Metals (Basel)*, vol. 11, no. 12, article no. 1919, 2021.
4. P. S. Sari, S. Thomas, P. Spatenka, Z. Ghanam, and Z. Jenikova, "Effect of plasma modification of polyethylene on natural fibre composites prepared via rotational moulding," *Compos. Part B Eng.*, vol. 177, article no. 107344, 2019.
5. M. R. Sanjay, S. Siengchin, J. Parameswaranpillai, M. Jawaid, C. I. Pruncu, and A. Khan, "A comprehensive review of techniques for natural fibers as reinforcement in composites: Preparation, processing and characterization," *Carbohydr. Polym.*, vol. 207, pp. 108-121, 2019.
6. L. Y. Mwaikambo and M. P. Ansell, "Chemical modification of hemp, sisal, jute, and kapok fibers by alkalization," *J. Appl. Polym. Sci.*, vol. 84, no. 12, pp. 2222-2234, 2002.
7. Britannica, *cantala*, Chicago: Encyclopedia Britannica. Available in <https://www.britannica.com/plant/cantala> (Accessed in August 25, 2022).
8. B. Zuccarello, G. Marannano, and A. Mancino, "Optimal manufacturing and mechanical characterization of high performance biocomposites reinforced by sisal fibers," *Compos. Struct.*, vol. 194, pp. 575-583, 2018.
9. D. Purwanto, E., Wisnu, W., and Ariawan, "Kekuatan Tekan dan Konduktivitas Panas Komposit Semen Serbuk Aren-Cantala.," *Prosiding Seminar Nasional Sains dan Teknologi*, vol. 1, pp. 21-25, 2011. (in Indonesian).
10. Y. Gao, P. romero, H. Zhang, M. Huang, and F. Lai, "Unsaturated polyester resin concrete: A review," *Constr. Build. Mater.*, vol. 228, article no. 116709, 2019.
11. A. Gharbi, R. B. Hassen, and S. Boufi, "Composite materials from unsaturated polyester resin and olive nuts

Salim et al.

- residue: The effect of silane treatment,” *Ind. Crops Prod.*, vol. 62, pp. 491-498, 2014.
12. B. Y. Alashwal, M. Saad Bala, A. Gupta, S. Sharma, and P. Mishra, “Improved properties of keratin-based bioplastic film blended with microcrystalline cellulose: A comparative analysis,” *J. King Saud Univ. - Sci.*, vol. 32, no. 1, pp. 853-857, 2020.
 13. L. K. Kian, M. Jawaid, H. Ariffin, and O. Y. Alothman, “Isolation and characterization of microcrystalline cellulose from roselle fibers,” *Int. J. Biol. Macromol.*, vol. 103, pp. 931-940, 2017.
 14. A. Kiziltas, D. J. Gardner, Y. Han, and H. S. Yang, “Mechanical Properties of Microcrystalline Cellulose (MCC) Filled Engineering Thermoplastic Composites,” *J. Polym. Environ.*, vol. 22, no. 3, pp. 365-372, 2014.
 15. S. Sakuri, E. Surojo, and D. Ariawan, “Thermogravimetry and interfacial characterization of alkaline treated cantala fiber/microcrystalline cellulose-composite,” *Procedia Struct. Integr.*, vol. 27, pp. 85-92, 2020.
 16. M. B. Palungan, R. Soenoko, Y. S. Irawan, and A. Purnowidodo, “The effect of fumigation toward the engagement ability of king pineapple leaf fibre (Agave Cantala Roxb) with epoxy matrix,” *ARNP J. Eng. Appl. Sci.*, vol. 11, no. 13, pp. 8532-8537, 2016.
 17. M. B. Palungan, B. Tangaran, S. Salu, and K. Tikupadang, “The Effect of Fumigation Treatment of King Pineapple Leaf Fiber (Agave Cantala Roxb) on Length of Fiber Critical Using Epoxy Matrix,” *J. Phys. Conf. Ser.*, vol. 1464, article no. 012057, 2020.
 18. M. Asim, M. Jawaid, K. Abdan, and M. R. Ishak, “Effect of Alkali and Silane Treatments on Mechanical and Fibre-matrix Bond Strength of Kenaf and Pineapple Leaf Fibres,” *J. Bionic Eng.*, vol. 13, no. 3, pp. 426-435, 2016.
 19. Z. Liu and B. Fei, *Characteristics of Moso Bamboo with Chemical Pretreatment-Sustainable Degradation of Lignocellulosic Biomass - Techniques, Applications and Commercialization*, Rijeka: InTech, 2013.
 20. A. Lotfi, H. Li, D. V. Dao, and G. Prusty, “Natural fiber-reinforced composites: A review on material, manufacturing, and machinability,” *J. Thermoplast. Compos. Mater.*, vol. 34, no. 2, pp. 238-284, 2021.
 21. B. Santoso, “Peluang pengembangan agave sebagai sumber serat alam,” *Perspektif*, vol. 8, no. 2, pp. 84-95, 2009. (in Indonesian).
 22. D. Ariawan, W. W. Raharjo, and Windiarjo, “Pengaruh Model Anyaman 3D Serat Cantala terhadap Karakteristik Serapan Bunyi Komposit Unsaturated Polyester Resin (UPR S) - Cantala 3D,” *Mekanika Jurnal Ilmiah Mekanika*, vol. 7, no. 2, pp. 50-57, 2009. (in Indonesian).
 23. G. Goud and R. N. Rao, “Effect of fibre content and alkali treatment on mechanical properties of Roystonea regia-reinforced epoxy partially biodegradable composites,” *Bull. Mater. Sci.*, vol. 34, no. 7, pp. 1575-1581, 2011.
 24. X. Li, L. G. Tabil, and S. Panigrahi, “Chemical treatments of natural fiber for use in natural fiber-reinforced composites: A review,” *J. Polym. Environ.*, vol. 15, no. 1, pp. 25-33, 2007.
 25. C. C. Ihueze, C. E. Okafor, and C. I. Okoye, “Natural fiber composite design and characterization for limit stress prediction in multiaxial stress state,” *J. King Saud Univ. - Eng. Sci.*, vol. 27, no. 2, pp. 193-206, 2015.
 26. K. Mahato, S. Goswami, and A. Ambarkar, “Morphology and mechanical properties of sisal fibre/vinyl ester composites,” *Fibers Polym.*, vol. 15, no. 6, pp. 1310-1320, 2014.
 27. M. H. Zin, K. Abdan, N. Mazlan, E. S. Zainudin, and K. E. Liew, “The effects of alkali treatment on the mechanical and chemical properties of pineapple leaf fibres (PALF) and adhesion to epoxy resin,” *IOP Conf. Ser. Mater. Sci. Eng.*, vol. 368, article no. 012035, 2018.
 28. S. H. Aziz and M. P. Ansell, “The effect of alkalization and fibre alignment on the mechanical and thermal properties of kenaf and hemp bast fibre composites: Part 1 - polyester resin matrix,” *Compos. Sci. Technol.*, vol. 64, no. 9, pp. 1219-1230, 2004.