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Impact of Chemical Treatment on Diffusion Mechanism and Mechanical Strength in Acidic Medium (pH 3) of Banana Fiber Composites

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Abstract

Synthetic fibers are widely used in polymer-based composites for their high stiffness and strength. However, these fibers have notable drawbacks, including poor biodegradability, high processing costs, low recyclability, high energy consumption, machine abrasion, and health hazards. This study explores the use of unidirectional banana fiber mats as natural reinforcements. These mats were carefully crafted using a specially designed handloom for cost-effective fabrication. Five different composites were developed, each incorporating various chemical treatments of banana fibers. The fibers underwent treatments with alkaline, peroxide, sodium chlorite, stearic acid, and permanganate, alongside with immersion in a pH 3 medium. These treatments aimed to enhance stress transfer across the composite interfaces and prevent moisture absorption, which can weaken the bond between fibers and the matrix, leading to structural weaknesses and reduced durability. A critical focus was modifying the hydrophilicity of banana fiber to resist water penetration and minimize dimensional changes, crucial for maintaining structural integrity in humid environments. The samples showed diffusion constants (n) less than 0.5, indicating Fickian diffusion (Case I). Sodium chlorite and stearic acid treatments improved the mechanical strength of banana fibers and reduced moisture absorption, which is significant for structural applications in industries like automotive and construction, where durability and resistance to environmental factors are crucial.

Keywords: Chemical treatment, banana fiber composite, swelling thickness, diffusion mechanism, *FEA.*

1. Introduction

Natural fiber-reinforced epoxy composites (*NFREC*) have gained significant attention due to their outstanding mechanical properties, including low density, lightweight nature, stiffness, biodegradability, and cost-effectiveness [1]. Despite their advantages, natural fibers have limitations such as high moisture absorption due to their structural components (cellulose, hemicellulose, lignin, pectin, and waxy substances), which results in poor adhesion with matrix materials [2]. To improve fiber-matrix compatibility and reduce moisture absorption, chemical treatments are essential. These treatments introduce functional groups that modify the fiber composition, enhancing adhesion and overall performance [3]. Sodium hydroxide (NaOH) treatment removes hemicellulose, lignin, pectin, wax, and oils, cleans the fiber surface, and decreases fiber diameter, enhancing bonding with the matrix and improving moisture resistance [4, 5]. Sodium chlorite ($NaClO_2$) bleaches fibers in acidic solutions, forming chlorine dioxide (ClO_2) that removes lignin and hydroxyl groups, increasing hydrophobicity and flexibility while reducing stiffness [4, 5]. Potassium permanganate ($KMnO_4$) generates Mn^{3+} ions that react with cellulose, enhancing bonding with the matrix and thermal stability [4]. Peroxide treatment promotes polyethylene grafting on fibers, improving adhesion, reducing moisture absorption, and enhancing thermal stability. Stearic acid reacts with fiber hydroxyl groups, improving water resistance by removing non-crystalline components and increasing fibrillation [6]. While hybrid composites with various fibers have been extensively studied, banana

fiber in epoxy composites has not been explored. This study fills this gap by evaluating banana fiber composites in an acidic medium (pH 3), fabricating five types with different chemical treatments, and analyzing swelling, water absorption, and fiber-matrix interactions under both dry and pH 3 conditions. The main objectives of this study are to investigate the types of chemical treatments that improve the mechanical strength and the kinetics of diffusion in an acidic medium (pH 3).

2. Materials, Chemical Treatments, Fabrication, and Aging Process

This research highlights the use of unidirectional banana fiber mats as reinforcement, combined with an epoxy resin matrix (Lapox B-11 resin and K-6 hardener). The customization of fiber mats through handloom weaving and the emphasis on chemical treatments to enhance fiber-matrix adhesion are crucial aspects. These steps are likely aimed at improving mechanical properties such as tensile strength, impact resistance, and durability of the composites. Alkaline treatment involved soaking fibers in 5% NaOH for 48 hours to improve wettability and remove impurities, while peroxide treatment included a 3% NaOH pre-treatment for 3 hours, followed by immersion in 6% benzoyl peroxide at 60°C for 30 minutes. Sodium chlorite treatment used a 5% NaOH pre-soak for 1 hour and subsequent exposure to $NaClO_2$ at 70°C for 24 hours. Stearic acid treatment immersed fibers in 4% stearic acid dissolved in ethanol at 90°C for 1 hour. Permanganate treatment incorporated sequential steps: 5% NaOH for 3 hours, 4% acetic acid for 2 hours, and 0.5% $KMnO_4$ for 30 minutes. After the treatment, fibers were thoroughly washed with water to remove any residual chemicals, dirt, or impurities from the treatment phase. After washing, the fibers were sun-dried, ensuring that any moisture content was reduced to avoid interference during composite fabrication. Each composite comprised four fabric layers with an epoxy resin and hardener matrix in an 11:1 weight ratio [3, 6-7]. To study moisture effects, a pH 3 solution prepared with 99% glacial acetic acid, stabilized for 24 hours before sample immersion. FEA was employed to simulate tensile and flexural tests providing efficient validation of experimental data.

2.1 Moisture Absorption Test

Studies on chemically treated banana fiber-reinforced epoxy composites examined moisture absorption and swelling in corrosive conditions (pH-3). The test samples were prepared per the ASTM D570 standard, soaked for 48 days, and swelling thickness was measured using a digital caliper. A 4-digit digital scale with 0.0001g accuracy was used to measure weight changes at room temperature 30°C.

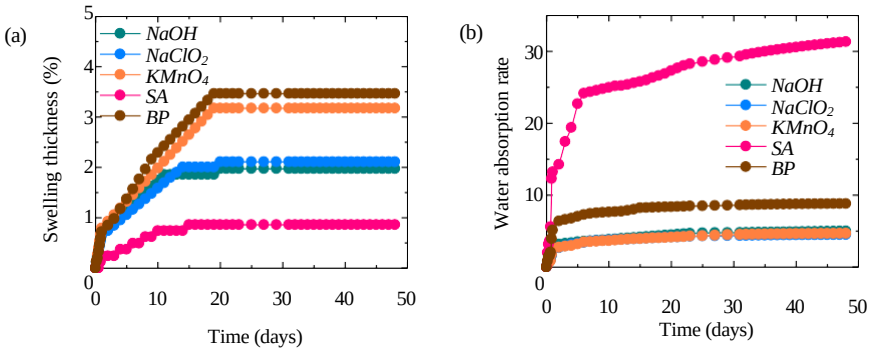


Fig. 1 Water absorption behavior: (a) swelling thickness; (b) moisture absorption rate

The swelling behavior of banana fiber composites varies with chemical treatments, which is illustrated in Fig. 1(a). BP and $KMnO_4$ cause the highest swelling due to increased water absorption. $NaClO_2$ and NaOH show moderate swelling, affecting the fiber-matrix bond, and causing moderate moisture uptake. SA leads to minimal swelling by forming a hydrophobic barrier that reduces moisture absorption. The highest moisture absorption was seen in SA treated samples due to increased fiber hydrophilicity. BP strengthens the fiber-matrix bond without greatly affecting moisture absorption. Sodium hydroxide (NaOH) had the lowest moisture absorption, likely due to chemical degradation of the fiber structure.

2.2 Kinetics of Diffusion Mechanism

Fick's law of diffusion is one of the most significant laws that describe the kinetics of the water absorption mechanism. There are three different kinds of diffusion behavior (Case I, Case II, and Case III) [8, 9]. From a theoretical point of view, the shape of the absorption curve, which can be represented by equation;

$$\frac{M_t}{M_m} = Kt^n \quad \text{(i)}$$

In moisture diffusion, M_t represents moisture content at time t, and M_m is the moisture content at saturation. k is a constant reflecting sample-water interaction, while n indicates the diffusion mode. For Fickian diffusion (Case I), $n = 0.5$; for Case II, $n = 1$ (or $n > 1$ for super Case II); and for anomalous diffusion (Case III), $0.5 < n < 1$. The values of n and k are derived from the slope and intercept of a log plot of M_t/M_m versus t using experimental data according to equation (ii)

$$\log \frac{M_t}{M_m} = \log k + n \log t \quad \text{(ii)}$$

Diffusion coefficient (D) can be calculated from the initial linear portion of the water absorption curve (i.e., the slope of M_t vs. $t^{1/2}$, h^{-1}) [5]. The values of n and k were determined by linear regression analysis and are given in Table 1.

$$D = \frac{\pi}{t} \times \left(\frac{M_t \times h}{4\, M_s}\right)^2 \text{ Where } n < 0.5 \quad \text{(iii)}$$

$$D = \frac{h^2}{t} \times \left(-\frac{1}{7.3} \times ln\left(1 - \frac{M_t}{M_m}\right)\right)^{\frac{1}{7.5}} \text{ Where } n > 0.5 \quad \text{(iv)}$$

The diffusion behavior of chemically treated banana fiber composites varies by treatment. NaOH treated composite shows the lowest diffusion coefficient, and slowest moisture absorption due to partial fiber degradation. SA treatment has the highest diffusion coefficient and rapid moisture absorption despite its hydrophobicity. All samples exhibit anomalous diffusion (Case III, $0.5 < n < 1$), where hydrophobic treatments can paradoxically increase water absorption due to surface modifications, while oxidizing agents alter water interaction. Strong bonding, as in BP, limits water entry, whereas NaOH degrades fibers, reducing permeability.

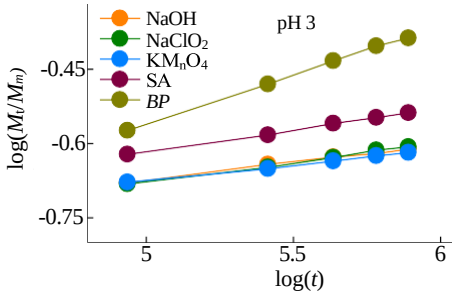


Fig. 2 log (M_t/M_m) vs. log (t) curve of fabricated composites immersed in pH 3

Table 1. Kinetics of diffusion of NaOH, $NaClO_2$, $KMnO_4$, BP, and SA treated banana samples

Treatment type	D (m^2/s)	n	k	R^2
NaOH	2.81649×10^{-11}	0.07	1.03	0.98203
$NaClO_2$	3.37549×10^{-11}	0.081	1.082	0.991
$KMnO_4$	2.00727×10^{-11}	0.06	0.99	0.935
BP	2.90389×10^{-11}	0.087	1.05	0.987
SA	3.37526×10^{-11}	0.19	1.54	0.972

2.3 Tensile Test

Dumbbell-shaped specimens were used following the ASTM D638-01 standard. The tests were conducted using an Instron Testometric M500-100CT universal testing machine (UTM), equipped with a 100 kN load cell. A pretension of 0.001 N was applied, and the cross-head speed was maintained at 10 mm/min. Key tensile properties were automatically generated by the WinTestTM software. These tests were performed both before and after an aging process. Dry, unaged specimens served as control samples. Additionally, specimens of each composite type were soaked in pH 3 for over 150 days, and then tested to evaluate the impact of corrosion on the tensile properties. Finite element analysis (*FEA*) is a valuable tool for analyzing elastic behavior, predicting key mechanical properties, and validating experimental findings efficiently. In this study, *FEM* was performed using ABAQUS software to evaluate mechanical properties and compare the results with experimental data.

Figure 3 shows the stress vs. strain curve of chemically treated samples, both by experiments and by *FEM*. SA treated sample exhibits the highest stress at peak (43.043 N/mm²), indicating better mechanical strength for control samples. Stress significantly drops in wet conditions. K. G. Raghavendra and R. Rajkumar et al. reported tensile strength 30.5 MPa for SA treatment [10]. Turjo et al. demonstrated that the tensile strength was 26.77 MPa for bananas in dry conditions [2]. P. T. R. Swain et al. and S. Biswas et al. reported tensile strength 29.8 MPa (for NaOH-treated banana fiber composites) [11]. Stress for NaOH treated samples decreases to 22.011 MPa from 37.501 MPa in wet conditions. This drop is due to moisture weakening the fiber and matrix interface, reducing the composite's ability to withstand stress. Strain at peak is generally higher in dry conditions, showing that the composites can stretch more before breaking. SA, for example, has the highest strain (2.36%) in dry conditions. However, strain at peak decreases in wet conditions, with the composite becoming more brittle and less capable of stretching before failure. Young's modulus is higher in dry conditions as shown in Fig. 3(f), which signifies that the composite is stiffer. For instance, $NaClO_2$ shows the highest Young's modulus (7015.446 MPa). It

decreases in wet conditions. This reduction is due to moisture absorption, which affects the fiber-matrix bonding and overall rigidity of the composite. Elongation at break is generally higher in dry conditions compared to wet conditions. For instance, SA shows the highest elongation at break (2.743 mm) in dry conditions, indicating enhanced flexibility in the dry state. Break decreases in wet conditions. This decrease can be attributed to the absorption of moisture, which affects fiber-matrix adhesion and overall composite integrity. Table 2 shows the summaries of tensile properties of NaOH, $NaClO_2$, $KMnO_4$, BP, and SA treated banana samples.

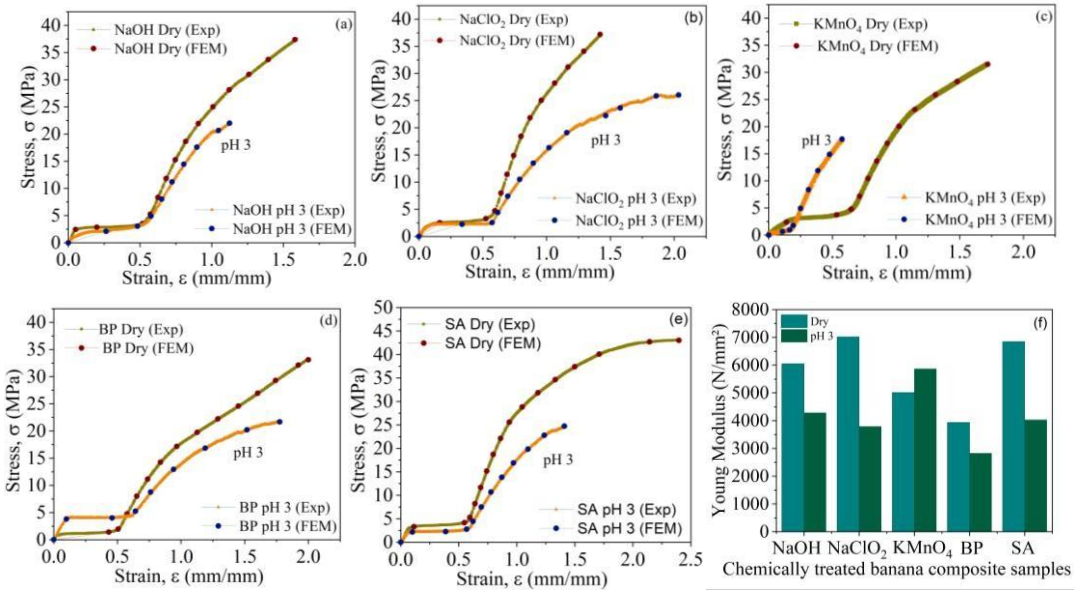


Fig. 3 Tensile stress vs. strain curve of chemically treated dry and wet specimens (Experimental and *FEM*)

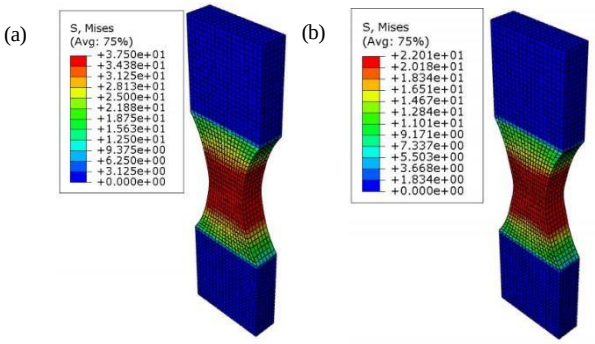


Fig. 4 Tensile strength analysis by *FEM* of NaOH chemically treated sample: (a) dry; (b) wet

Table 2. Tensile properties of NaOH, $NaClO_2$, $KMnO_4$, BP, and SA treated banana samples

Treatment type	Elong.@ break (mm)		Stress @ peak (MPa)		Elong. @ peak (mm)		Strain @ peak (%)		Young's modulus (MPa)	
	Dry	pH 3	Dry	pH 3	Dry	pH 3	Dry	pH 3	Dry	pH 3
NaOH	1.641	1.187	37.501	22.011	1.627	1.15	1.593	1.125	6047.616	4281.799
$NaClO_2$	1.443	2.082	37.181	26.037	1.443	2.068	1.429	2.039	7015.446	3787.994
$KMnO_4$	1.71	0.581	31.508	17.687	1.71	0.581	1.713	0.576	5010.319	5867.044
BP	2.006	1.917	33.107	21.683	2.006	1.785	1.992	1.775	3935.014	2824.589
SA	2.743	1.753	43.043	24.734	2.381	1.448	2.36	1.418	6844.043	4027.991

2.4 Flexural Test

Three-point bending tests assessed flexural properties following the ASTM D790. Testing used a Tinius Olsen 25ST UTM with a 60 mm span, 10 mm/min cross-head speed, and 5 N pre-load. Maximum deflection was set at 35 mm. Figure 5 illustrates the flexural stress vs. strain curves of chemically treated composites. The $NaClO_2$-treated sample exhibited the highest ultimate stress (72.67 MPa), while Stearic Acid (SA) had the lowest (50.02 MPa). R. N. Das et al. reported flexural strength 65.5 MPa for $NaClO_2$ treated banana fiber composites [12]. In wet conditions, $NaClO_2$ still had the highest ultimate stress (70.61 MPa), and SA the lowest (39.68 MPa). The maximum MOR was also in dry $NaClO_2$ (72.7 MPa), decreasing in wet conditions, with $NaClO_2$ at 70.6 MPa and SA at 39.7 MPa. Moisture absorption reduced mechanical properties, but NaOH and $NaClO_2$ treatments improved fiber bonding and matrix interaction. NaOH-treated composites had the highest stiffness (1760 N-m), while SA

had the lowest (725 N-m). Wet samples showed decreased stiffness, with $NaClO_2$-treated composites highest in dry conditions (1390 N-m) and SA lowest when wet (559 N-m). Strength was highest in $NaClO_2$ (211 N-m) and Benzoyl Peroxide (139 N-m), with SA the lowest (94.9 N-m), and these values also decreased in wet conditions. $NaClO_2$ and NaOH treatments improved stiffness and strength, though less effective in wet conditions due to water-induced degradation. Istiak et al. also found the similar result with 5% (w/v) NaOH chemically treated JUCO fiber composites [7]. Table 3 illustrates the summaries of flexural properties of NaOH, $NaClO_2$, $KMnO_4$, BP, and SA treated banana samples.

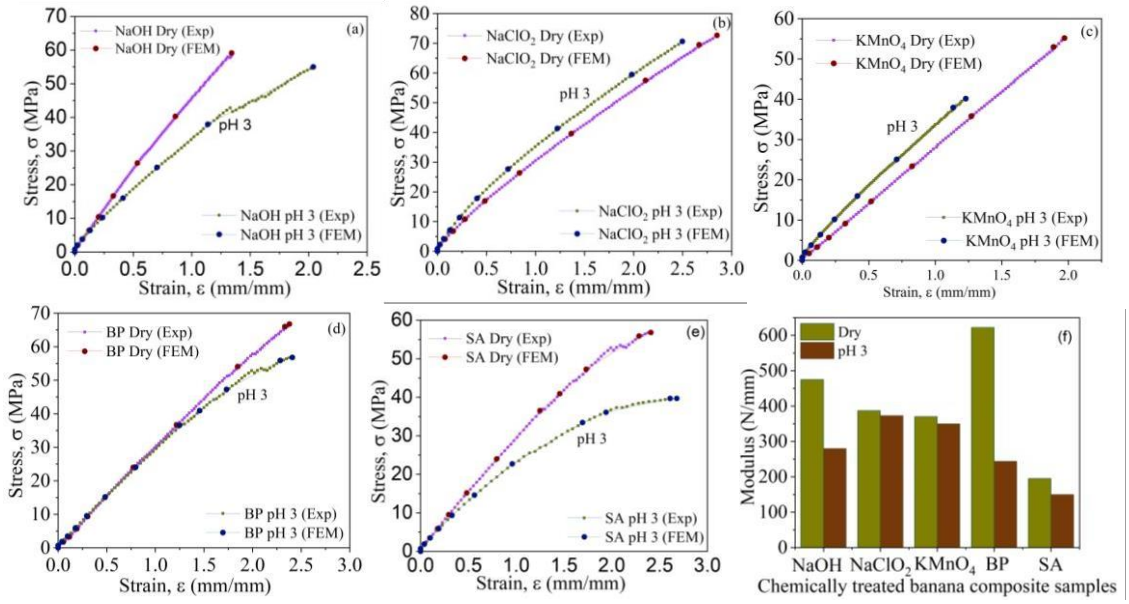


Fig. 5 Flexural stress vs. strain curve of chemically treated dry and wet specimens (Experimental and *FEM*)

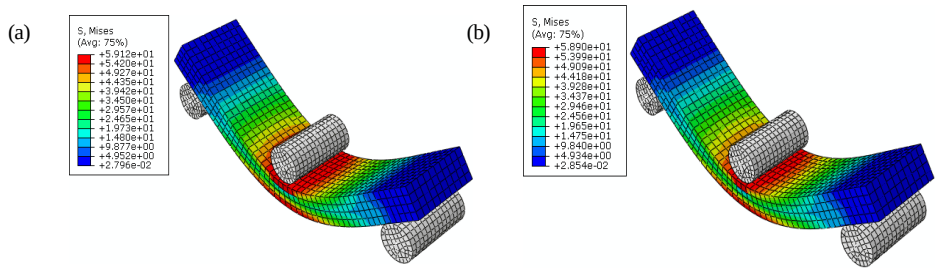


Fig. 6 Flexural strength analysis by *FEM* of NaOH chemically treated sample: (a) dry; (b) wet

Table 3. Flexural properties of NaOH, $NaClO_2$, $KMnO_4$, BP, and SA treated banana samples

Treatment type	Modulus (N/mm^2)		Ultimate Force (N)		Ultimate Stress (MPa)		MOR (MPa)		EI (Stiffness)	
	Dry	pH 3	Dry	pH 3	Dry	pH 3	Dry	pH 3	Dry	pH 3
NaOH	475	280	790.4	729.91	59.12	58.9	59.1	58.9	1760	1040
NaClO2	387	373	1192.21	1068.41	72.67	70.61	72.7	70.6	1440	1390
KMnO4	370	350	697.7	555.66	55.16	40.13	55.2	40.1	1380	1300
BP	622	244	1044.26	645.36	66.74	56.81	66.7	56.8	976	908
SA	195	150	535.85	397.4	50.02	39.68	50	39.7	725	559

2.5 Impact Test

Charpy impact testing was conducted on banana fiber-reinforced composites according to the ASTM D6110. In dry conditions, BP achieved the highest impact energy (18 J) due to strong covalent bonding, while $NaClO_2$ showed the lowest impact energy (15 J) due to fiber brittleness from delignification. $KMnO_4$ treatment followed with a high impact energy of 17 J, enhancing fiber-matrix adhesion through oxidation. $NaClO_2$ had the lowest impact energy (15 J) due to increased fiber brittleness. V. N. Rao et al. reported impact strength 13.8 J for SA treatment [13]. In terms of impact strength, $KMnO_4$ provided the highest and $NaClO_2$ with the lowest. V. K. Singh et al. found impact strength 11.6 J/cm² for $NaClO_2$ treatment [14].

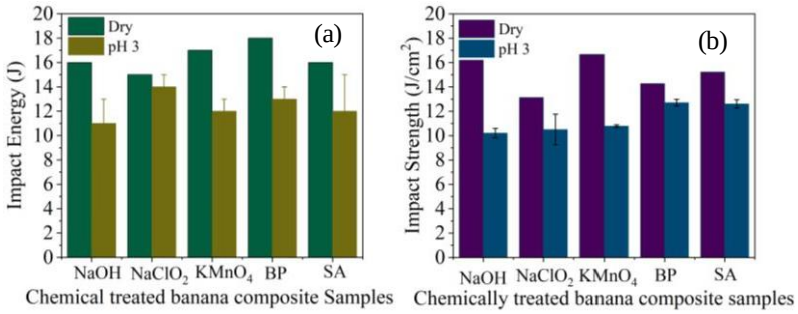


Fig. 7 Comparison of (a) impact energy, (b) impact strength of dry and wet samples

3. Conclusion

The following conclusions can be made from this experimental analysis -

- BP and $KMnO_4$ cause the highest swelling, indicating that enhancing water absorption and weakening the composite structure, and SA treated sample indicates the greatest water absorption capacity.
- SA treated composites exhibit the highest diffusion coefficient and the most rapid moisture absorption, driven by surface hydrophilicity despite its hydrophobic nature. However, SA treated sample exhibits the highest stress at peak in tensile test indicating better mechanical strength in the dry state.
- The highest ultimate stress and MOR was observed in the $NaClO_2$ treated sample due to improve fiber bonding and matrix interaction
- The highest impact energy under dry environment found from BP due to strong covalent bonding and free radicals that initiate cross-linking reactions.
- $NaClO_2$ removes lignin from the fiber, making it more brittle and reducing its impact resistance.

4. References

1. G. I. Williams and R. P. Wool, "Composites from natural fibers and soy oil resins," *Applied Composite Materials*, vol. 7, no. 5, pp. 421–432, 2000. doi: 10.1023/A:1008292903025.
2. S M K S. Turjo, M. F. Hossain, M. S. Rana, Md Al-Mamun and M. S. Ferdous, "Durability and mechanical characterization of unidirectional jute/banana and synthetic fiber reinforcement epoxy composite," *Hybrid Advances*, vol. 6, p. 100232, June. 2024, doi: 10.1016/j.hybadv.2024.100232.
3. M. Abdelmouleh, S. Boufi, M. N. Belgacem, and A. Dufresne, "Short natural-fibre reinforced polyethylene and natural rubber composites: Effect of silane coupling agents and fibres loading," *Composites Science and Technology*, vol. 67, no. 9, pp. 1627–1639, 2007. doi: 10.1016/j.compscitech.2006.07.009.
4. X. Li, L. G. Tabil, and S. Panigrahi, "Chemical treatments of natural fiber for use in natural fiber-reinforced composites: A review," *Journal of Polymer Environmental*, vol. 15, no. 1, pp. 25–33, 2007. doi: 10.1007/s10924-006-0042-3.
5. Istiak Ahmed, Md Foisal Hossain, Muhammed Sohel Rana, Md Shafiul Ferdous, "Mechanical Characterization and Diffusion Analysis of Jute, JUCO, and Banana Fiber Reinforced Epoxy Composites", Hybrid Advances, vol. 7, p. 100291, 2024.
6. S. A. Paul, K. Joseph, G. D. G. Mathew, L. A. Pothen, and S. Thomas, "Influence of polarity parameters on the mechanical properties of composites from polypropylene fiber and short banana fiber," *Composites: Part A*, vol. 41, pp. 1380–1387, 2010.
7. S. M. R., 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," *Carbohydrate Polymers*, vol. 207, pp. 108–121, Nov. 2019. Available: https://doi.org/10.1016/j.carbpol.2018.11.083.
8. S. M. K. S. Turjo, M. F. Hossain, M. S. Rana, and M. S. Ferdous, "Mechanical characterization and corrosive impacts of natural fiber reinforced composites: An experimental and numerical approach," *Polymer Testing*, vol. 125, 108108, Jun. 2023. Available: https://doi.org/10.1016/j.polymertesting.2023.108108.
9. M. S. Alvy, M. F. Hossain, M. S. Rana, M. M. Rahman, M. S. Ferdous, Influence of stacking sequences of woven jute-carbon hybrid composites: diffusion mechanism and mechanical characterization, *Heliyon*, vol. 10, pp. e36632, 2024.
10. K. G. Raghavendra and R. Rajkumar, "Influence of Stearic Acid Surface Treatment on Mechanical Properties of Banana Fiber Reinforced Composites," Journal of Applied Polymer Science, vol. 136, no. 24, pp. 1-7, 2019. DOI: 10.1002/app.47812.
11. P. T. R. Swain and S. Biswas, "Mechanical Properties of Surface-Modified Natural Fiber Reinforced Epoxy Composites," *Journal of Composite Materials*, vol. 51, no. 26, pp. 3909-3922, Dec. 2017. DOI: 10.1177/0021998317697012.
12. R. N. Das, M. Venkatesh, and A. K. Singh, "Chemical Treatment and Flexural Properties of Banana Fiber Composites," *Materials Research Express*, vol. 6, no. 4, pp. 11-17, 2020. DOI: 10.1088/2053-1591/ab30c2.
13. V. N. Rao and P. T. Swain, "Effect of Stearic Acid Treatment on Impact Resistance of Banana Fiber Composites," *International Journal of Polymer Science*, vol. 2020, no. 1, pp. 1-7, 2020. DOI: 10.1155/2020/3412819.
14. V. K. Singh, N. S. Das, and R. K. Yadav, "Effect of Sodium Chlorite on the Mechanical Properties of Banana Fiber Composites," *Journal of Composite Materials*, vol. 55, no. 5, pp. 561-567, 2021. DOI: 10.1177/0021998320970020.