Evaluation of Mechanical Properties and Hydrophilicity of Alkaline and Plasma Treated Abaca Fiber Epoxy Composite with Mineral Waste as Fillers

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Abstract: Natural fiber composites (NFC) are increasingly recognized for their sustainability and versatility. Abaca fiber is one of the prominent natural fibers that are sourced from the leaf sheaths of the abaca plant. This study provides an overview of the comparison of the mechanical properties and water uptake behavior of surface-modified N_2 plasma and alkali treated abaca fiber-reinforced epoxy composites with granite powder used as fillers. It mentions the use of varying filler content (0 wt.%, 10 wt.%, 20 wt.%, and 30 wt.%) in both the pre-treated composites and compares their properties. Plasma-treated granite filler samples showed superior tensile strength and flexural properties, despite a negative effect on impact properties. A lower water absorption is observed in plasma-treated composites with fillers compared to those without fillers. Overall, the potential of surface modification techniques with granite-based mineral fillers is apparent in improving the performance of N_2 plasma treated NFCs, thus expanding their applications across different industries aiming for the good mechanical hygroscopic properties.

Keywords: Abaca fiber, N_2 Plasma, Alkali treatment, Granite fillers, Mechanical properties, Hydrophilicity, Composite.

INTRODUCTION

Composites are a class of engineering materials consisting of a carrier matrix for reinforcing fibers, which impart strength and stiffness to the resulting material. Polymer matrix composites (PMCs) consist of organic matrix material. The insertion of reinforcements of higher tensile strength and modulus into a polymer matrix enables the enhancement of thermal and mechanical properties. These reinforcements can include a variety of synthetic fibers, such as carbon fibers, glass fibers, aramid fibers, and many more. These synthetic fibers can create long-term environmental concerns after they reach the end of their life cycle. Due to this, Sustainable, biodegradable materials have become a key consideration in material design. Natural fibers, formed through geological processes or from plant and animal bodies, have garnered significant interest due to their lifecycle superiority, biodegradability, low cost, and notable mechanical properties. The integration of natural fibers into PMCs represents a promising direction for developing environmentally friendly composite materials without compromising on performance.

Abaca fibers, among the strongest natural fibers available, surpass other commonly used fibers like cotton and jute in terms of strength and resistance to salinity [1, 2]. Abaca fibers typically contain 60.8–64 wt.% cellulose, 17.5–21 wt.% hemicelluloses, 12–15 wt.% lignin, and small proportions of fats, pectin, ash, and waxes. However, the presence of hydroxyl groups in natural fibers like abaca poses drawbacks in applications, such as rapid absorption and release of moisture, leading to poor wettability and interfacial bonding, particularly in moist conditions. Polarization properties can also lead to poor matrix compatibility, which results in bad interfacial bonding and lowered mechanical properties in comparison to synthetic fiber composites.

To address these challenges and improve interfacial bonding between fiber and matrix, surface modifications of fibers through suitable pretreatments are necessary [4]. Some of the chemical treatments include acetylation, etherification, and mercerization. Physical treatments like plasma treatment, corona treatment, and ultrasonication are being used to modify the natural fiber surface. Among these pretreatments, plasma treatment has emerged as an effective, promising, dry, and eco-friendly option [5, 6]. Plasma, as the fourth state of matter, is composed of a high concentration of reactive species capable of inducing physical and chemical changes on polymeric surfaces [7]. The surface modification by plasma treatment is a result of the sputtering effect at the fiber surface during the plasma bombardment of the material, which produces chemical modifications [7, 8]. Plasma treatment offers advantages over wet treatments by enabling surface property changes (up to the range of a few nanometers) rather than altering bulk properties. Plasma treatment may have several effects on the surface of the substrate, including cleaning, activation, grafting, etching, and polymerization. The plasma atmosphere comprises ions, radicals, free electrons,

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atoms, molecules, and other particles. Excited by radiofrequency electric fields, free electrons collide with neutral gas molecules, dissociating them into various reactive species. The interaction of these species with the substrate placed on the plasma bed results in physical and chemical surface modifications [8].

Reducing pressure in a plasma system using a vacuum pump allows for the generation of low-pressure plasma (LPP). This low pressure allows the generation of plasma at much lower temperatures, where the energy required to ionize does not translate into heat energy but still allows for the conditions to generate the plasma, which is usually referred to as cold plasma, which operates at lower temperatures. LPP treatment enhances mechanical properties by improving adhesion with the surrounding matrix [9]. Physical changes induced by LPP treatment, such as etched surfaces, have the potential to enhance bonding between fiber and matrix [9, 10]. Improved interfacial adhesion through plasma treatment leads to enhanced mechanical properties compared to their non-treated counterparts [10, 11]. N_2-LPP treatment reduces the hydrophilic nature of fibers and improves adhesion through physical surface etching [12, 13].

The final properties of the composite material also depend on the type of reinforcements and fillers used. N_2-plasma's higher affinity for moisture can be seen. Commonly used fillers such as calcium carbonate, talc, glass microspheres, carbon black, and silica nanoparticles play a crucial role in minimizing costs and enhancing performance. Fillers improve flexural strength by acting as barriers to crack propagation, with higher filler content corresponding to a higher crack resistance capacity of the material [17]. Granite fillers show great potential for composite cost reduction, mitigating the harmful effects of synthetic fibers, promoting eco-friendly production practices, and enhancing the tensile, flexural, and hydrophobic nature of the composite [19]. Granite fillers create an effective barrier against water penetration, which creates shielding. This barrier effect inhibits the movement of water molecules within the composite, leading to lower water absorption.

However, the literature lacks studies on granite powder-added plasma-pretreated Abaca fiber-reinforced epoxy composites. This study aims to develop a hybrid composite and compare it with conventional alkali-treated abaca fiber-reinforced epoxy resin with granite fillers. The investigated mechanical and physical properties of the hybrid composite demonstrate its potential for lightweight structural applications.

2. MATERIALS AND METHODS

2.1. Materials

In this study, the reinforcements used were abaca fibers sourced from Artipistolos, Campo de Criptana, Spain, with a density of 0.8 g/cm³. The fiber type was Regular Sinamay without Stiffening of 17x17 DPI, which was fabricated using a handmade method. A 4 wt.% solution of NaOH was supplied by Carl Roth GmbH, Karlsruhe, Germany. IN2 epoxy resin was purchased from Easy Composites. The fillers utilized in this study were obtained from eBay and later sieved to ≤0.75 µm. Detailed descriptions of the resin and granite powder are provided in the tables below.

Table 1: Properties of Granite Powder

Property	Value
Type	Natural ground Granite
Particle size	≤0.75 µm
density	2.85 g/cm^3

Table 2: Properties of Epoxy Resin

Property	Value
Tensile Strength	63.5 - 73.5 MPa
Tensile Modulus	2.6 - 3.8 GPa
Flexural Modulus	3.35 GPa
Density	1.5 g/cm^3
Viscosity (at 20°C)	200–450 mPa.s.

2.2. Alkali Treatment of the Fibers

Alkali treatment is the conventional and most popular method for treating natural fibers. It improves interfacial bonding between the fiber and the matrix material, leading to higher mechanical interlocking between the fibers and the matrix. This, in turn, directly enhances the mechanical properties of the resultant composite material [4, 11]. The alkaline solution reacts with the hydroxyl (-OH) groups present in the cellulose molecules of the fibers, causing a change in the physical structure of the fiber by disrupting the hydrogen bonds between the cellulose chains. Consequently, this process leads to alterations in the orientation of cellulose molecules along the fiber axis. In this study, one set of abaca fabrics were immersed in a 4% concentration of NaOH solution for 2 hours. Subsequently, the fabrics were removed from the solution and rinsed thoroughly in distilled water to ensure complete removal of the alkali solution and other impurities. Following rinsing, the fabrics were initially air-dried at room temperature for 24 hours, followed by further drying in an oven at 40°C for an

additional 12 hours to ensure complete removal of moisture.

2.3. Plasma Treatment of the Fibers

Another set of fabrics were prepared for plasma treatment. Before placing the fabric into the plasma chamber, it was completely dried in an oven at a temperature of 40°C for 2 to 3 hours. For the surface treatment of the abaca fabric, a low-pressure plasma system, PINK V6-G, was employed, as illustrated in Figure **1**. This model has a plasma generator that operates with a microwave power of 50–300 W, an excitation frequency of 2.45 GHz, and plasma chamber dimensions of 170 x 200 x 170 mm. The fabric was trimmed according to the dimensions of the plasma bed in the plasma chamber. N_2 was used as a purge gas. Preliminary investigations were conducted to optimize the plasma treatment parameters. Various combinations of pressure, power, treatment time, and gas flow rate were systematically tested to determine their effects on the surface properties of the fabrics. The selected parameters were then employed for the subsequent treatment of the fibers in the main experiments, as mentioned in Table **3**.

Table 3: Plasma Parameters

Specific Parameters	Value
Base pressure	50 Pa
Gas Flow rate; N_2	50 ml/min
Power	100 W
Process pressure	±50 Pa
Time	120 sec

The N_2 plasma comprises a significant concentration of active components, including N_2, N_2-, N_2+, N_3-, and N_3-. When these active components interact with the surface of the fabric, they induce changes of the surface properties. Consequently, the interaction results in the formation of free radicals such as NH_2, NO_3, NH_2-, and NH_2- on the fabric surface. The molecular interaction of plasma on the cellulose fiber like Abaca in presence of nitrogen gas is shown in the Figure **2** [12].

N_2 gas plasma is favored for its low ionization level and limited penetration depth. In the course of plasma treatment, a thin layer (typically a few nanometers) forms on the fiber surface, enhancing roughness, thus facilitating improved bonding between the fiber and matrix.

Figure 1: Pink V6-G Plasma Device.

2.4. Sample Preparation Using Hand-Layup (Assisted) Vacuum Baggage Process

Another Composite fabrication for both Alkaline and plasma pre-treated samples was carried out using the hand-layup (HL) assisted vacuum bagging technique, illustrated in Figure **3** [21]. Given the higher density of solid granite fillers compared to the epoxy matrix, ensuring proper dispersion of these fillers into the matrix was essential. The fabrication process involved creating a series of composite compositions with varying filler weight percentages: 0%, 10%, 20%, and 30%, while maintaining a constant fiber weight percentage of 20. This experimental setup was applied to both plasma-treated and alkali-treated samples. These compositions were labelled as 0GF_NaOH, 10GF_NaOH, 20GF_NaOH, 30GF_NaOH for Alkali treated samples and 0GF_PI, 10GF_PI, 20GF_PI, 30GF_PI for Plasma treated samples. Detailed weight percentages are shown in Table **4**. The aim was to investigate the effects of different filler concentrations on the properties of the composites and comparing the properties of both plasma and alkaline pre-treated samples. Initially, a mold release agent (Silicone spray) was applied to the glass mold. Abaca fabric was then positioned on the mold surface, followed by the

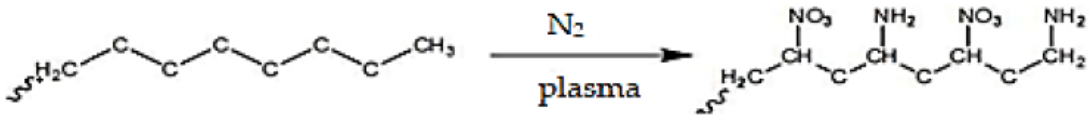

Figure 2: Chemical reaction on fabric surface with N_2 in plasma chamber.

application of a mixture of granite fillers and epoxy resin onto the fabric surface using a brush. Subsequently, a roller was employed with mild pressure to remove any trapped air and to squeeze out excess resin. This process was repeated for each layer, ensuring proper wetting of the fabric with the stone powder-filled resin, until the desired number of layers were stacked. The consolidated material was then covered with peel ply (a synthetic fabric aiding in demolding and ensuring proper surface finish) and a breather fabric (a relatively thick non-woven fabric providing an escape path for air evacuation and absorbing excess resin). The entire lay-up assembly was enclosed with a sealing bag, and the edges were sealed with silicone tape. Vacuum port was connected to the bag for air evacuation using a vacuum pump. Activating the pump evacuated all the air from the space between the vacuum bag and the mold, resulting in consolidation of the composite under a pressure of approximately -1 bar

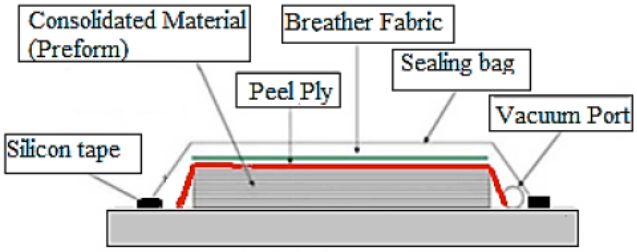

Figure 3: Schematic Diagram of hand-layup (assisted) vacuum baggage process [21].

Samples were maintained under vacuum until the curing process was completed. Studies have shown that samples consolidated under vacuum demonstrate superior mechanical properties compared to non-consolidated samples [14]. Vacuum bagging ensures uniform pressure distribution, resulting in consistent laminate thickness and quality throughout the composite. Additionally, it facilitates effective bonding between laminate layers, reducing voids and excess resin. This process significantly enhances the overall strength and performance of the resulting composite material. The laboratory setup of the vacuum baggage process is shown in Figure **4**. The specimen was allowed to cure for 72 hours to ensure proper composite hardening. After pre-curing in the vacuum bag, the specimen has gone through post-curing in an oven at 40°C for 6 hours. To prepare the sample for testing and characterization, the composite has been precisely cut into the required dimensions for each test setup. All the composite material compositions were made in this manner.

Volume fractions of Composite is calculated by using the formula below

$$Ø_f = \frac{w_f/\rho_f}{(w_f/\rho_f)+(w_m/\rho_m)+(w_{fi}/\rho_{fi})} \quad (1)$$

Where Ø f – fiber volume fraction, w_f—fiber weight fraction, ρ_f —fiber density, w_m – matrix weight fraction, ρ_m—matrix density, w_{fi}—filler weight fraction, ρ_{fi} —filler density.

Figure 4: Hand layup assisted vacuum Baggage setup.

The Weight percentages of each composition of both plasma treated and alkaline treated samples are given in the below Table **4**. Where the both composition like 0GF_NaOH and 0GF_Pl are written in single phrase; 0GF_NaOH/Pl.

2.5. Test Equipment and Test Parameters

2.5.1. Tensile Tests

Tensile tests were conducted to evaluate the behavior of the materials under tension. The tests were performed according to the DIN EN ISO 527 standard, utilizing a ZwickRoell universal testing machine. The crosshead speed was set to 5 mm/min. The Young's modulus was determined within the strain range of

Table 4: Composition of Alkali/Plasma Treated Samples in wt.-%

Designation	wt.-% of fiber	wt.-% of filler	wt.-% of matrix
0GF_NaOH/Pl	20	0	80
10GF_NaOH/Pl	20	10	70
20GF_NaOH/Pl	20	20	60
30GF_NaOH/Pl	20	30	50

0.05% to 0.25%. Five specimens of each composite material were tested, and the average values were recorded. Throughout the test, precise monitoring of the specimen's force and displacement allowed for the generation of comprehensive stress-strain curves. The specimens measured were of dimensions 120 × 10 × 5 mm.

2.5.2. Flexural Tests

The flexural test, also known as the 3-point bending test, involves placing a specimen on two support anvils, and a loading pin is used to apply force at the center of the sample to measure its properties. Flexural tests were conducted according to the DIN EN ISO 178 standard. The test setup included a 0.1 MPa preload and a flexure modulus of 2 mm/min. Parameters measured included flexural strength, flexural modulus. Five specimens were tested for each composition, and the average values were recorded. The dimensions of the measured samples were 80 × 10 × 5 mm.

2.5.3. Impact Tests

Impact testing of composites is conducted to assess their impact resistance by measuring the amount of energy absorbed during fracture. Impact resistance refers to the ability of a material to absorb energy under impact loads. Charpy impact tests were performed following the DIN EN ISO 179 test standard. Test parameters included an impact energy of 4 J and an impact velocity of 2.9 m/s. The dimensions of the unnotched samples were 80 mm × 10 mm × 5 mm

2.5.4. Water Absorption Test

This test was employed to determine the amount of water absorbed by the composite under specified environmental conditions. Water diffuses into the composites, ending up either in the matrix or at the fiber-matrix interphase. The amount of water present in the matrix must be different from that in the interphase. Consequently, a mismatch in volumetric expansion between the matrix and fiber interphase leads to the formation of localized stress and strain in the composites [15]. The experiments were conducted following the ASTM D 570-98 standard. The percentage of water absorption in a sample is calculated by equation (2). The test specimens are dried for 24 hours in an oven at 90°C. After cooling, the specimens are weighed using a digital weighing machine. Each weighed specimen is then submerged in a salt and Distilled water solution at 25°C for a duration of 24 hours. Subsequently, the submerged specimens are retrieved, their surfaces are wiped with a tissue, and then immediately reweighed to record any weight gain as shown in Figure **5**. This process is repeated for a total duration of 240 hours for all compositions of plasma and alkali treated.

$$\% \text{ of water absorption} = \frac{(w_2 - w_1)}{(w_1)} \times 100 \quad (2)$$

where w_2 = Weight of the wet sample and w_1 = weight of the dry sample.

3. RESULTS

3.1. Tensile Test

The tensile modulus and tensile strength of plasma- and alkali-treated samples with varying filler content are shown in Figures **6** and **7**, respectively. In terms of tensile modulus, the NaOH-treated samples exhibited better results than the plasma-treated samples. Initially, both samples without fillers showed almost the same tensile modulus. However, as the filler content increased from 10% to 20%, the modulus values increased from 3224 to 3941 MPa for alkaline-treated samples and from 2143 to 2691 MPa for plasma-treated samples. Remarkably, for both sample types, the tensile modulus value drastically decreased at 30% filler content.

Figure 5: Test specimens immersed in salt water. The weights of the samples were checked with a high-precision weighting scale.

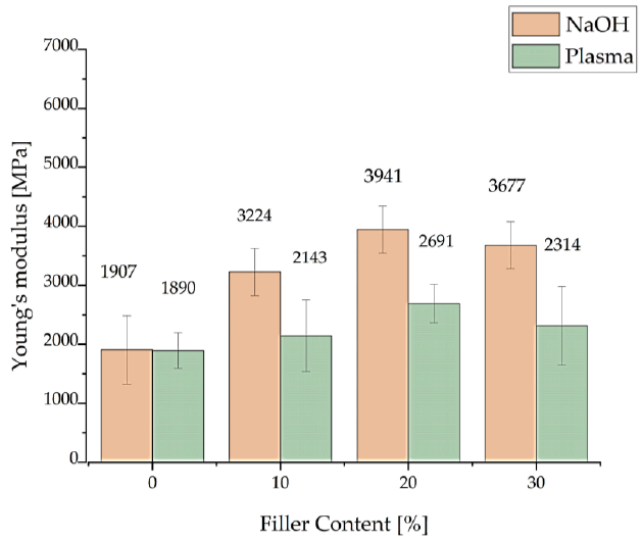

Figure 6: Comparison of Young´s modulus of NaOH and Plasma treated samples with varying Filler content.

In terms of tensile strength, plasma-treated samples demonstrated superior results compared to the alkali-treated samples. In the case of virgin composites without any fillers, the tensile strengths of NaOH and plasma-treated samples were 34 MPa and 43.9 MPa, respectively. The NaOH-treated samples exhibited an increase in strength (from 35 MPa to 43 MPa) as the filler percentage increased up to 30%, while the tensile strength of plasma-treated samples ranged from 44.7 MPa to 47 MPa at 10% and 20% filler content. However, at 30% granite fillers, they both showed a decreased value.

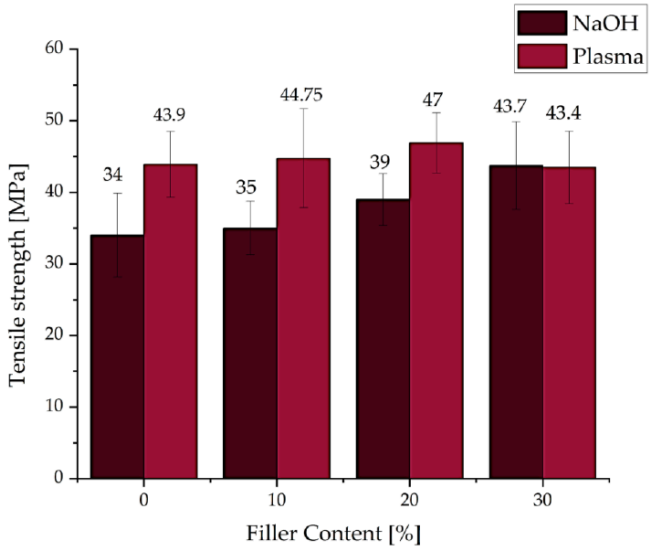

Figure 7: Comparison of Tensile Strength of NaOH and Plasma treated samples with varying filler content.

3.2. Flexural Test

Flexural modulus and strength exhibited similar trends. In the case of flexural modulus, NaOH-treated samples showed values ranging from 1620 to 2087 MPa for filler contents of 0%, 10%, and 20%, respectively. Meanwhile, Plasma-treated samples consistently demonstrated higher values, ranging from 2580 to 3288 MPa across the same filler content range. However, both pre-treatments exhibited a decrease in flexural modulus at the highest filler content among all samples, in Figure **8**. As depicted in Figure **9**, the flexural strength increased from 45 MPa to 57.18 MPa in Alkali-treated samples with filler content ranging from 0% to 20%. Similarly, in the Plasma Surface Pre-treated samples, the strength values ranged from 49 MPa to 57.8 MPa until 20%, demonstrating a consistent increase. However, a downward trend was observed in both pre-treatments beyond 20 wt.-% filler content in the composite material. Overall, Plasma pre-treatment exhibited better flexural strength values.

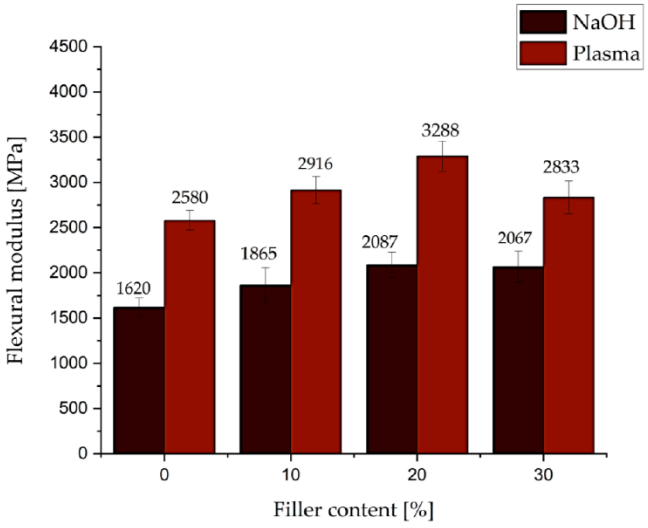

Figure 8: Comparison of Flexural Modulus of NaOH and Plasma treated samples with varying Filler content.

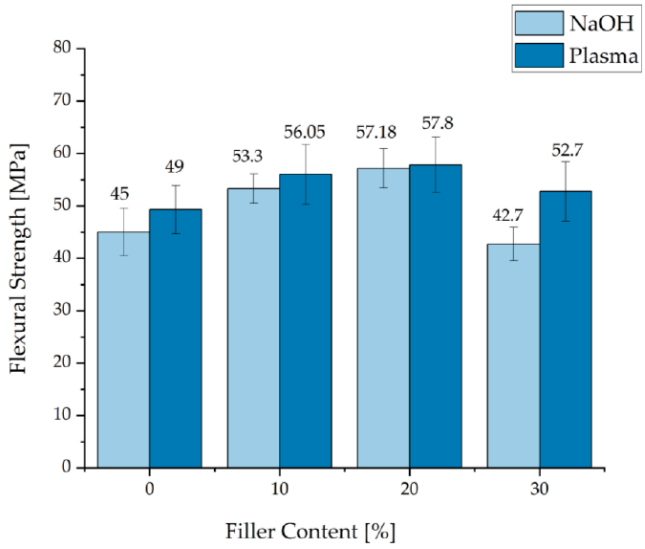

Figure 9: Comparison of Flexural Strength of NaOH and Plasma treated samples with varying Filler content.

3.3. Impact Test

Impact strength was measured in units of KJ/m². In both plasma and alkali samples, as the filler content

increased, the impact strength exhibited a decreasing trend. For the NaOH samples, the strength of the composite without fillers was 5.8, decreasing to 4.7 with 30% fillers. Similarly, in the composite with plasma treatment, the values decreased significantly from 5.9 to 4.9 with an increased filler wt.% as shown in Figure **10**.

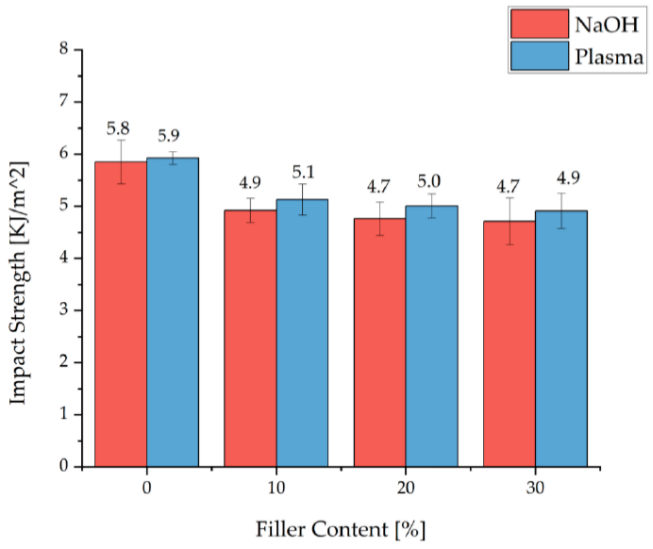

Figure 10: Comparison of Impact Strength of NaOH and Plasma treated samples with varying Filler content.

3.4. Hydrophilic Testing

In the water absorption test of Abaca fiber composite reinforced with granite fillers, Plasma treated samples exhibited a higher affinity for the salt solution compared to Alkaline samples, as shown in Figure **11** and Figure **12**. Interestingly, the composite fabricated without granite filler displayed a greater propensity for water absorption. The incorporation of granite filler content into the fiber-reinforced polymer composite resulted in a reduction of water absorption property from 10 to 30 wt. %. In the Initial 24hours PI samples absorbed more water and then this trend has been continued upto 240 hours. But when the comparison is drawn in between samples of varying filler content in both the treatments higher filler content shows lesser %. This reduction can be attributed to the uniform dispersion of granite stone powder within the composite. Additionally, the presence of porous holes in the composite influenced its absorption capacity, providing a preferential path for the solution. These pores likely formed during composite fabrication

4. DISCUSSION

Mechanical tests were conducted on abaca fiber pre-treated with nitrogen plasma and were compared to conventionally alkali-treated abaca fiber with dispersed granite fillers to study and compare the treatment effect. The results indicate that plasma-treated samples exhibited superior performance in terms of tensile strength and flexural properties compared to NaOH-treated samples. However, the tensile modulus and impact strength of plasma-treated samples were not as satisfactory, suggesting that several factors (fiber and filler morphology, filler distribution) may influence these conditions.

In alkali treatment, the effective removal of OH-groups from the fibers results in increased surface roughness [16]. This higher roughness parameter may contribute to the higher initial threshold observed during the tensile tests, leading to a higher modulus compared to plasma-treated samples. Chemical-treated samples demonstrated the ability to withstand higher stress levels over a certain amount of strain at the early stage of stress application to the composite materials.

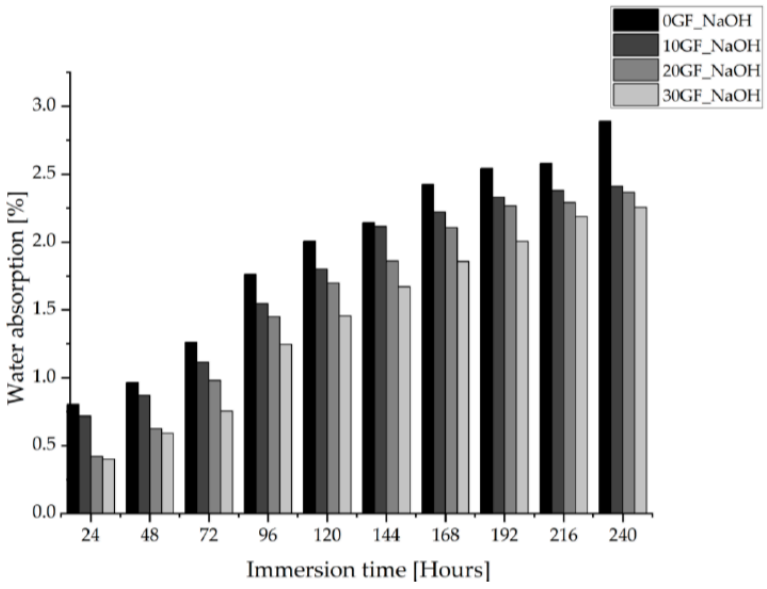

Figure 11: Comparison of water absorption % of NaOH treated samples with varying Filler content.

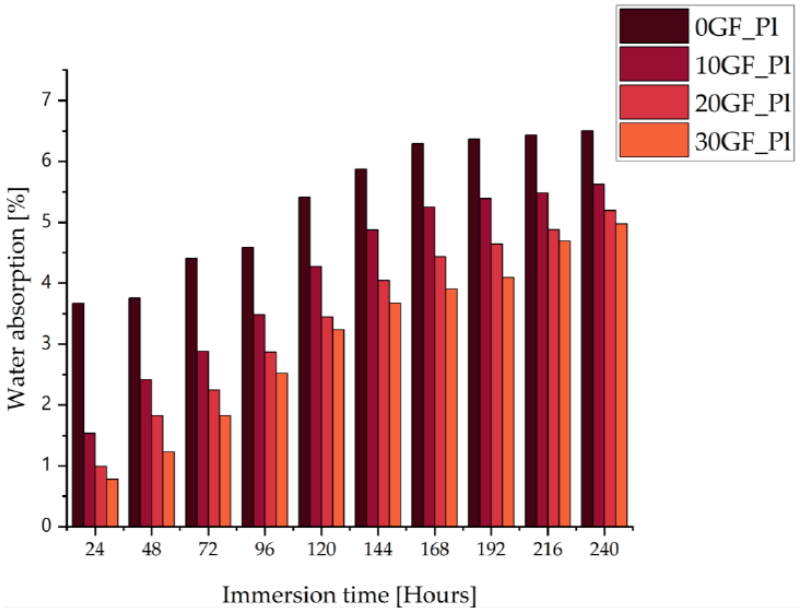

Figure 12: Comparison of water absorption % of Plasma surface treated samples with varying Filler content.

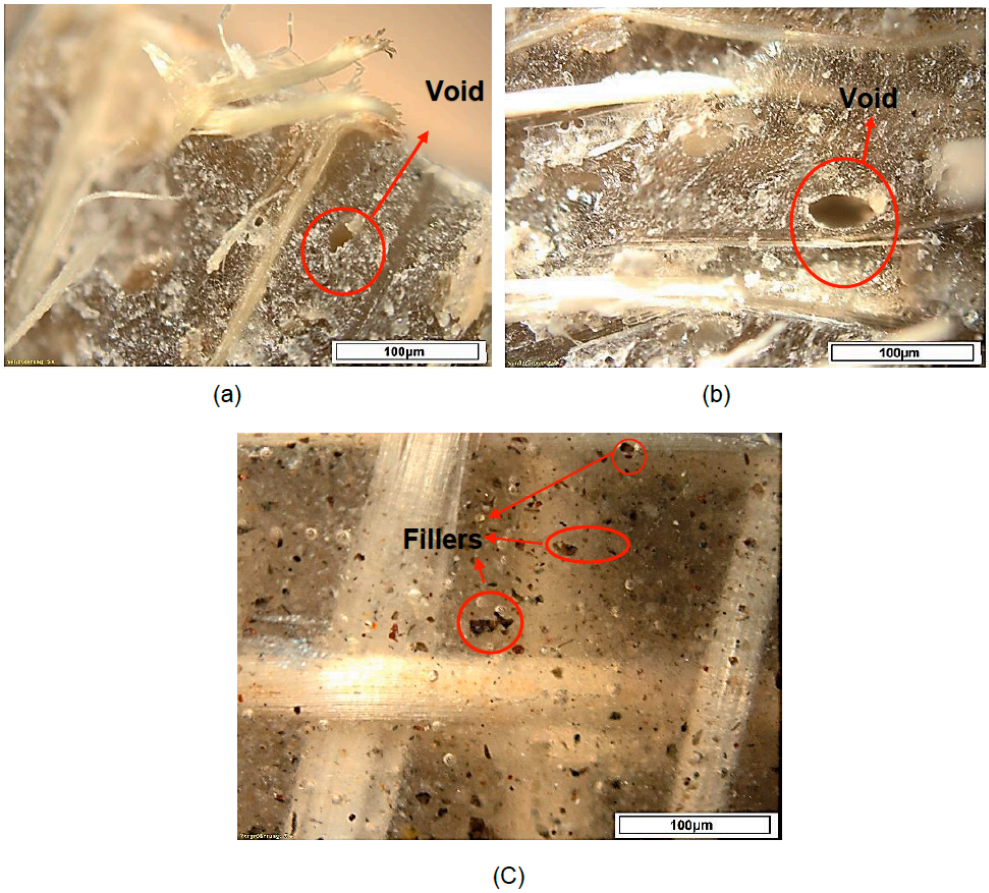

Figure 13: (**a**) Optical microscopy test image of 10GF_PI at 100 µm. (**b**) Optical microscopy test image of 20GF_PI at 100 µm. (**c**) Optical microscopy test image of 10GF_NaOH at 100 µm.

The observed lower Young's modulus in the plasma samples can be attributed to the presence of multiple layers, where the movement of a single fabric layer might affect overall material properties.

The impact strength decreased as the filler content increases, this can be attributed to the effect of fillers on the overall properties of the material. Weakly bonded fillers can create voids between interfaces as shown in Microscopic Image Figure **13 a**, **b**. limiting

proper stress transfer and leading to crack initiation and propagation, ultimately resulting in reduced energy absorption. Additionally, filler particle size and shape significantly influenced the impact properties of the composite, where the asperities in the filler may accumulate stress; leading to stress concentration [17] as shown in Figure **13 c**.

As described in [18], interfacial debonding occurs in composite materials when critical bending stress values are exceeded, leading to stress redistribution across subsequent layers until fracture occurs. The results indicate that initial layers are sufficiently saturated with resin due to the vacuum bagging process, ensuring proper compaction. Flexural properties depend on the stiffness, ultimate strain, and the stress intensity factor. Fillers served as crack resistance by acting as a barrier to crack propagation, thereby enhancing the material's ability to resist deformation. However, beyond a filler content limit of 20% (*i.e*., 30%), a decrease in properties were observed, likely attributable to filler particle accumulation and agglomeration [19].

In water absorption tests, a trend of increased hydrophobicity was observed with the addition of granite fillers in both the pre-treated samples, attributed to the uniform dispersion of fillers. Conversely, plasma-treated samples exhibited higher water absorption rates due to elevated surface polarity and surface energies resulting from the introduction of nitrogen functional groups, which enhanced their affinity for polar liquids, consequently reducing the contact angle. In this context, granite fillers proved to be effective to some extent in mitigating the overall hydrophilic nature of the composite material.

5. CONCLUSION

The investigation into N_2 LPP-treated samples with dispersed granite fillers within the matrix has revealed notable improvements in certain mechanical properties compared with the same composite compositions with the alkali-pre-treated samples. Nitrogen plasma treatment has proven effective in enhancing adhesion through the deposition of nitrile and amine-based compounds on the fiber surface. Although the tensile modulus and impact properties of the composite were not satisfactory, the nitrogen plasma treatment holds promise for further enhancing flexural properties.

The incorporation of granite fillers in this composite material has exerted significant influence, particularly up to 20% filler content, showing an increasing trend in tensile and flexural properties. Beyond this saturation point, fillers become closely packed, as observed with the 30% content, hindering movement under external stress and diminishing properties. Additionally, the impact properties have been negatively affected by filler size, shape, and improper bonding with the matrix material, highlighting the significant impact of filler morphology on impact properties.

The addition of granite fillers has a positive effect on the hygroscopic nature of the composite material, as observed in both samples. Consistent with expectations, nitrogen plasma treatment has exhibited a higher affinity for saltwater solutions, as evidenced by the results in samples with no filler content and also with the alkali

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CONFLICTS OF INTEREST

The authors declare no conflicts of interest.

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