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Research Article

Enhancing the Performance of High-Loaded Coconut Shell Biocomposites with NaOH Treatment

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ABSTRACT

The immense environmental concerns and the need for sustainable materials have driven extensive research into the development of biocomposites. This paper, therefore, investigates the effects of alkali (NaOH) concentration on coconut shell particles and how it influences the physical, mechanical, and thermal characteristics of the resulting biocomposites. Coconut shell particles were soaked in NaOH solutions at varying concentrations of 0%, 5%, 10%, 15%, and 20%, with soaking times of 4 and 8 hours. Coconut shell particles were analyzed using X-ray diffraction (XRD), X-ray fluorescence (XRF), and Fourier Transform Infrared Spectroscopy (FTIR), both before and after NaOH treatment. The biocomposite boards were fabricated using a press method, applying a load of 9 tons for 30 minutes. The coconut shell particles were mixed with epoxy resin at a ratio of 80 vol.% coconut shell particles (high-loaded biocomposite) and 20 vol.% epoxy resin. The physical, mechanical, and thermal properties of the biocomposite were evaluated. The universal testing machine, thermogravimetric analysis, and scanning electron microscope (SEM) were utilized to characterize the biocomposite samples. The results indicated a significant improvement in the performance of the biocomposites following the treatment of coconut shell particles with a 20% NaOH solution for 8 hours. The thickness, swelling, and porosity decreased from 10.9% and 9.5% to 2.8% and 2.5%, respectively. The modulus of rupture and modulus of elasticity increased from 8.34 MPa and 764 MPa to 19.22 MPa and 4447 MPa, respectively. The biocomposite became more thermally stable. The above points imply that the biocomposite material underwent property modifications due to the alkaline treatment of the coconut shells, thereby improving the compatibility and interaction between the coconut shell particles and the epoxy resin.

1. Introduction

Due to increased awareness of environmental deterioration and the sustainable use of resources, there has been a major paradigm shift in materials science, particularly in biocomposites, which are replacing traditional petrochemical-based composite materials. The use of natural resources as matrix materials and reinforcements has opened promising avenues for reduced petrochemical consumption, lower carbon emissions, lighter, lower-density materials, and greater ease of biodegradability, thereby eliminating non-biodegradable waste to a large extent [1-3]. However, the use of natural

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resources as reinforcements in biocomposites still faces certain limitations, such as unaltered mechanical properties and the lack of standardized processing techniques for natural resources [4]. As part of improving interfacial properties in such composites, following certain recent advancements concerning interfacial properties enhancement in composites [5], it has been found that the addition of nano-reinforced materials like Multi-Walled Carbon Nanotubes (MWCNTs) assists in improving mechanical properties as well as wear resistance characteristics of polymer composites by enhancing interfacial properties in the epoxy matrix.

The never-ending quest for innovation in sustainable biocomposite development offers numerous opportunities across various engineering disciplines. An important approach to developing biocomposites is the use of natural fibers as reinforcements. This not only results in reduced weight and lower material costs of the composites, but it also serves as a potential substitute for synthetic reinforcement materials such as glass, carbon, and Kevlar [6]. Various natural fibers, such as jute, bamboo, ramie, oil palm, and chicken feathers, as well as hybrid combinations, have been reported to exhibit improved mechanical and morphological properties [7-14]. In particular, coconut shell particles are a highly promising lignocellulosic filler material that is abundant, inexpensive, and readily available [15]. They are highly available and have extremely low cost; hence, these fibers are appropriate for mass production. Despite their numerous desirable attributes, their natural hydrophilic behavior leads to low adhesion to hydrophobic polymers [6]; thus, there is a strong need to develop modification approaches to optimize their mechanical properties [16]. Natural fiber-reinforced composites are hindered by their low compatibility levels [17].

To overcome this problem, many surface modification methods have been developed. Of these methods, alkaline treatment with sodium hydroxide (NaOH) has been widely used [18-20]. This process reduces hemicellulose and lignin content, increasing surface roughness and creating sites for improved mechanical and chemical interlocking between the fiber and the polymer surfaces.

Indonesia and the Philippines are among the largest global coconut-producing countries, accounting for about 67% of global crude coconut oil exports [21, 22]. Despite the large-scale production, a tremendous amount of coconut shell waste is produced. The use of coconut shells as a new material for making biocomposites is the optimal approach for reducing waste and conserving natural resources. The use of coconut shells, including their coir fibers, in biocomposite boards has been demonstrated in previous studies [23-28].

However, most previous studies still have several limitations. Many works were performed at a single NaOH concentration, without investigating a wide range of concentrations or variable immersion times [23, 28, 29]. Several studies have placed greater emphasis on mechanical performance at the expense of chemical and structural modifications that take place during alkali treatment [30, 31]. Systematic investigations that relate the chemical modification parameters, such as NaOH concentration and immersion time, to the physical, mechanical, and thermal properties of biocomposites in an integrated approach are still few [32-34]. For example, Meftah evaluated only one NaOH concentration, excluding consideration of immersion time and examination by FTIR and XRD analyses, which are important for assessing chemical modification issues [35]. On the other hand, Livingston focused solely on mechanical properties, without evaluating the impact of alkali treatment parameters on microstructural and thermal properties [36]. Most studies also used relatively low loadings of coconut shell filler, typically less than 55% [23, 29, 33, 36], whereas a high biomass content is highly sought after in biocentric, sustainable development of biocomposites.

In light of the above, the insufficiency of similar works implies that the mutual influence of the alkali treatment process and the properties of coconut shell biocomposites containing a large quantity of fillers is, as yet, unclear. Up to now, there have been no systematic studies addressing the influence of NaOH concentration and immersion time as controlling factors in defining the physical, mechanical, and thermal properties of coconut shell biocomposites containing 80% filler. In this respect, the current research highlights the novelty of the concept, as it will offer a detailed analysis of the mutual influence of the aforementioned factors and the properties of coconut shell biocomposites containing 80% filler, which have, to date, never been reported in the literature. This approach offers new insights into the optimization of chemically modified, high-loaded lignocellulosic biocomposites.

NaOH treatment has also been shown to significantly increase the maximum degradation temperature of coconut shell particles, reaching up to 434°C, which is much higher than that of untreated particles [29]. In addition to improved mechanical strength, NaOH treatment also reduces water absorption by removing hydrophilic components such as hemicellulose and lignin [30]. However, few studies have

correlated this with in-depth thermal stability analysis.

Based on the above background, this study aims to prepare high-loaded biocomposite (80 vol.% of fillers) and comprehensively evaluate the effect of NaOH concentration (0%, 5%, 10%, 15%, and 20%) and soaking time (4 and 8 hours) on the physical, mechanical, and thermal properties of the coconut shell particles biocomposite. Epoxy resin is selected as the matrix in the fabrication of biocomposite boards due to its excellent adhesion properties. Its high stiffness, thermal resistance, and resistance to softening make it particularly suitable as a binding agent for forming robust biocomposite structures [37, 38].

2. Materials and Methods

2.1. Materials

Dried coconut shells obtained from PT Indratma Sahitguna, Indonesia, were processed into fine particles (200 mesh powder) by grinding and sieving. Figure 1 shows the coconut shell powder after grinding and sieving. The sieving process was carried out to achieve a uniform particle size distribution, resulting in coconut shell powder with relatively homogeneous particle sizes.

Fig. 1. a) Coconut shells, b) Particles of coconut shell

Epoxy resin manufactured by the Avian Company was used as the matrix to fabricate the biocomposite. The resin and hardener were mixed in a 1:1 ratio. The curing time is about 8 hours. The density of epoxy resin is 1.15 – 1.20 g/cm^3.

Sodium Hydroxide (NaOH) in powder form was supplied by Merck. The NaOH solution was prepared by mixing distilled water with NaOH powder according to the desired concentrations (0, 5, 10, 15, 20%).

2.2. Alkali Treatment of Coconut Shell Particle

Coconut shell particles (CSP) were immersed in an alkaline NaOH solution with various concentrations (0, 5, 10, 15, and 20%) and immersion times (4 and 8 hours). The alkali treatment process of coconut shell is shown in Fig. 2. After the treatment, the particles were thoroughly rinsed and air-dried. Alkali treatment was employed as an effective approach to reduce particle surface tension while improving surface cleanliness. In addition, this process facilitates the removal of impurities adhered to the particle surfaces [39].

Fig. 2. Alkali treatment process of coconut shell particles through immersion in NaOH solution

2.3. Fabrication of Composite

The fabrication process for the coconut shell composite is shown in Fig. 3.

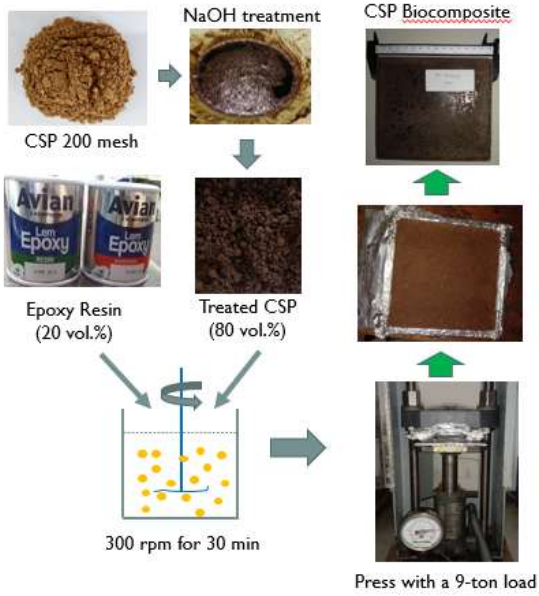

Fig. 3. The fabrication process of the coconut shell composite

Coconut shell particles were subjected to alkali treatment by immersion in NaOH solutions at 0%, 5%, 10%, 15%, and 20% for 4 and 8 h. After the treatment, the particles were filtered through a filter cloth to facilitate separation, then dried in an oven at 105 °C for 4 h until a stable moisture content was achieved. Biocomposite boards were fabricated by mixing 80 vol.% of the alkali-treated coconut shell particles with 20

vol.% epoxy resin. The epoxy resin used was a commercial product from Avian, with a resin-to-hardener ratio of 1:1. The mixture was then poured into aluminum foil-lined molds with dimensions of 150 mm × 150 mm × 10 mm, followed by compression molding using a hydraulic press under a load of 9 tons for 30 min at room temperature.

2.4. Characterization

The elemental content of coconut shell particles, both before and after NaOH treatment, was determined via X-ray Fluorescence (XRF). The composition and crystal structures were simultaneously analyzed by X-ray Diffraction (XRD) using a Shimadzu XRD-6000 (Shimadzu Corporation, Tokyo, Japan). To analyze the chemical reactions, Fourier Transform Infrared (FTIR) Spectroscopy was used to analyze the untreated and NaOH-treated coconut shell particles.

The physical properties investigated included density, porosity, and thickness swelling, in accordance with ASTM D571. Mechanical properties, specifically flexural strength (modulus of rupture) and flexural modulus (modulus of elasticity), were evaluated according to ASTM D790 using a Hung Ta Company (Taichung City, Taiwan) HT-2402 Universal Testing Machine (UTM). For thermal properties, thermal conductivity and Thermogravimetric Analysis (TGA, Shimadzu, type DTG–60, Kyoto, Japan) were performed. TGA was conducted under a nitrogen atmosphere from room temperature to 600°C at a heating rate of 2°C/minute. Thermal conductivity was measured in accordance with ASTM C177. The morphological characteristics of the fractured surfaces of the biocomposite samples, after flexural testing, were analyzed using a scanning electron microscope (SEM, Thermo Fisher Scientific, Waltham, MA, USA).

2.5. Statistical Analysis

A one-way analysis of variance (ANOVA) with Duncan's multiple range tests was conducted to test for homogeneity of variance and to determine the effect of NaOH treatment on the physical, mechanical, and thermal properties of the biocomposites. The statistical significance used in the analysis was 0.05 (5%). The calculation was performed using SPSS version 22.0 (IBM, Chicago, IL, USA).

3. Results and Discussion

3.1. Chemical Property

The coconut shell particles, before and after NaOH treatment, were examined by XRF, XRD, and FTIR. Table 1 displays the XRF data for the chemical compounds in coconut shell particles before and after treatment with various NaOH concentrations for 4 hours of soaking time.

Table 1. The chemical compound in the coconut shell particles after immersion in NaOH solution for 4 hours

Chemical Compound	NaOH Concentration				
	0 %	5 %	10 %	15 %	20 %
CaO (%)	42.0	70.5	71.0	65.9	59.9
Fe_2O_3 (%)	20.7	14.8	16.6	20.0	25.3
K_2O (%)	20.0	-	-	1.2	1.2
SiO_2 (%)	4.8	3.3	3.9	4.4	4.5
P_2O_5 (%)	5.3	3.9	3.5	3.5	3.8
CuO (%)	2.2	1.9	1.8	1.9	2.0

The significant increase in CaO content from 42.0% to 70.5% at 5% NaOH indicates an interaction between calcium compounds and alkaline treatment, likely due to leaching of lignocellulosic components, resulting in a relative enrichment of calcium or the formation of calcium-rich phases that are more readily detectable. Under basic conditions, NaOH dissolves alkali-labile components and promotes the precipitation of the $Ca(OH)_2$ phases on the particle surface. Fe_2O_3 shows a minimum at 5% NaOH, then increases with increasing NaOH concentration, reaching 25.3% at 20% NaOH. Therefore, this phenomenon reveals that Fe_2O_3 has very poor solubility in alkaline media and undergoes relative enrichment as the other components dissolve. K_2O is below the limit of detection at 5–10% NaOH; this indicates the high solubility of potassium compounds under basic conditions. However, it is again present within the range 15–20% NaOH at a constant low concentration of 1.2%, suggesting either ion redistribution or a shift in chemical equilibrium at higher alkali concentrations. Findings from this study are consistent with those of Bichang'a et al. [23], which demonstrated that alkaline treatment enhances the selective removal of lignin and hemicellulose to achieve surface purification. This can be related to NaOH treatment, which results in a relative enrichment of inorganic constituents such as CaO and Fe_2O_3 and a decreased content of alkaline-labile fractions, thereby increasing interfacial interactions in composite materials.

Table 2 presents the changes in the chemical composition of coconut shell particles after 8 hours of soaking. The trends established here and those for the 4-hour soaked particles differ only in the extent to which the alkali treatment has

influenced the composition. CaO is again the major compound, and its concentration has definitely increased, while the amount of K_2O has nearly reached zero. The increase in soaking time has increased the extent of the process's selectivity. Other compounds, such as Fe_2O_3, SiO_2, P_2O_5, and CuO, have helped to define the chemical composition.

Table 2. The chemical compound in the coconut shell particles after immersion in NaOH solution for 8 hours

Chemical Compound	NaOH Concentration				
	0 %	5 %	10 %	15 %	20 %
CaO (%)	42.0	67.5	70.1	68.2	73.2
Fe_2O_3 (%)	20.7	15.7	17.0	16.2	16.5
K_2O (%)	20.0	0.8	0.5	0.5	-
SiO_2 (%)	4.8	3.6	3.4	3.7	3.5
P_2O_5 (%)	5.3	3.3	3.1	3.1	2.8
CuO (%)	2.2	1.6	1.5	1.3	1.5

The XRD data for coconut shell particles, before and after treatment with various NaOH concentrations, are presented in Fig. 4 and Fig. 5 for soaking durations of 4 and 8 hours, respectively. The broad amorphous peaks were observed at 15.2 and 21.3 degrees. These peaks are related to lignocellulosic biomass (coconut shell), indicating the presence of cellulose, hemicellulose, and lignin [23]. The peak observed at 29.4 degrees is associated with K_2O. The intensity of this peak decreases as the NaOH concentration increases, indicating that the K_2O concentration decreases, which agrees with the XRF results.

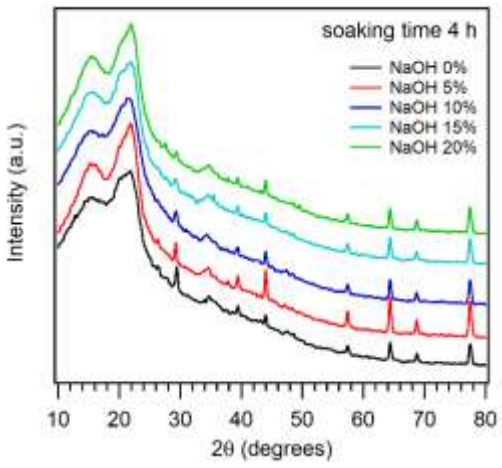

Fig.4. The XRD spectra of coconut shell particles after immersion in NaOH solution for 4 hours

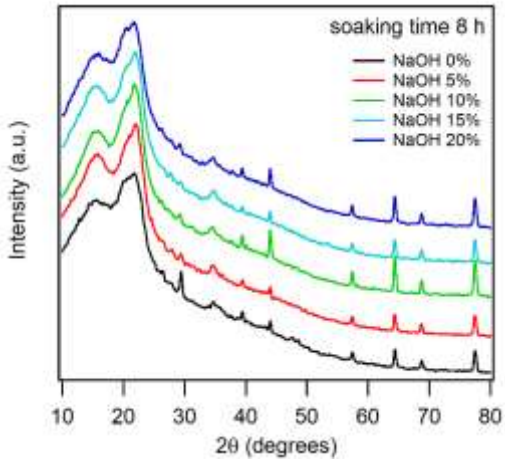

Fig. 5. The XRD spectra of coconut shell particles after immersion in NaOH solution for 8 hours

The other peaks observed are at 39.3 degrees (CuO), 43.8 degrees (Fe_2O_3), 57.4 degrees (Fe_2O_3), 64.2 degrees (CaO), 66.8 degrees (SiO_2), and 77.4 degrees (TiO_2). Similar peaks were observed for a soaking time of 8 hours.

FTIR data for coconut shell particles before and after NaOH treatment are presented in Fig. 6 (for 4-hour immersion) and Fig. 7 (for 8-hour immersion). Several characteristic peaks were observed. A broad peak at 3307 cm^{-1} signifies the O-H stretching vibrations of cellulose, hemicellulose, and lignin. The peak at 2910 cm^{-1} corresponds to aliphatic C-H stretching vibrations, indicative of C-H bonds present in biomass materials. Peaks around 2360 cm^{-1} are associated with carbonyl (C=O), hydroxyl (-OH), and aromatic C=C bonds, characteristic of lignocellulosic material. A peak at 2102 cm^{-1} is attributed to C≡C stretching vibrations from alkanes.

Furthermore, a peak at 1734 cm^{-1} indicates C=O stretching vibration, which is present in lignin [40]. The peak at 1599 cm^{-1} is typically linked to aromatic C=C stretching vibrations, common in lignocellulosic materials. A peak observed at 1246 cm^{-1} in FTIR spectra is typically attributed to C-N stretching vibrations associated with functional groups in hemicellulose and lignin. Similarly, a peak at 1053 cm^{-1} is associated with C-O stretching vibrations in hemicellulose [41].

Following NaOH treatment for both 4-hour and 8-hour soaking times, the disappearance of peaks at 1734 cm^{-1} and 1246 cm^{-1} was observed. These peaks are associated with hemicellulose and lignin, respectively, indicating that a portion of these components was successfully removed from the coconut shell particles after NaOH treatment. Although FTIR primarily shows the disappearance of characteristic peaks rather than the formation of new chemical bonds, the reduction of peaks associated with hemicellulose

and lignin, combined with the significant improvements in mechanical properties (MOR, MOE) and the enhanced interfacial adhesion observed in SEM images, strongly indicates effective chemical modification and improved filler-matrix interaction.

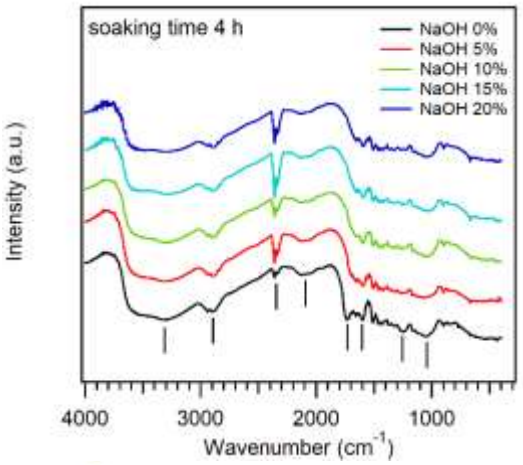

Fig. 6. The FTIR spectra of coconut shell particles after immersion in NaOH solution for 4 hours

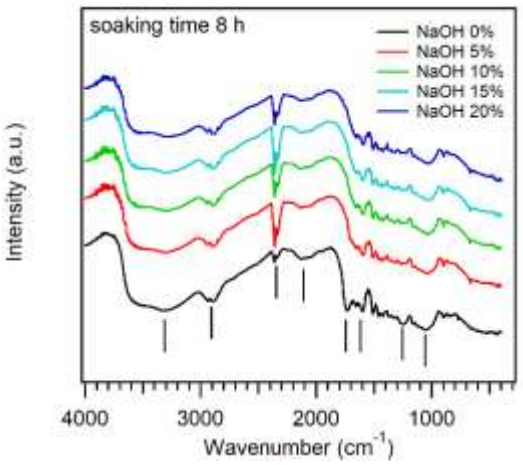

Fig. 7. The FTIR spectra of coconut shell particles after immersion in NaOH solution for 8 hours

3.2. Physical Properties

The physical properties of the coconut shell particle biocomposites were determined in this research using density, porosity, and thickness swelling tests. From the graph in Fig. 8, it is clear that the biocomposite density increased with increasing NaOH concentration for both soaking times of 4 hours and 8 hours.

The reason for this enhancement in density could be best ascribed to the effectiveness of the alkaline treatment in altering the lignocellulosic composition of the coconut shell micro-particles. NaOH treatment has been known to remove the amorphous portion of the lignocellulosic content, consisting of hemicellulose and some lignin. These regions of lower density are removed, and/or the microstructure of cellulose may densify, resulting in higher-density packing of the sample. This densification is again reinforced by the FTIR results (explained in the previous paragraph), where the characteristic peaks of hemicellulose (1734 cm^{-1}) and lignin (1246 cm^{-1}) vanish upon NaOH treatment. Moreover, the information in Fig. 8 shows the effects of soaking time on the resulting density values after different concentrations of NaOH treatment; in this case, soaking the samples for 8 hours is superior to 4 hours at all NaOH concentrations.

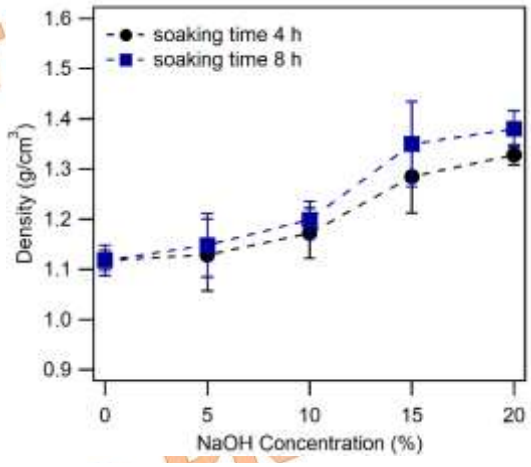

Fig. 8. Density of coconut shell biocomposite for different NaOH concentrations with immersion times of 4 hours and 8 hours.

The density change indicates that higher concentrations of NaOH are more effective at performing alkali treatments, thereby improving the material's overall physical properties. Alkalification has been observed to produce fiber surface topography that favors mechanical interlocking and thus improves fiber/matrix interfacial bonding [42]. Some authors have observed that amorphous content may increase and hydrogen bonding decrease with alkalification [2]. Density ranges of all biocomposite board samples made from coconut shells varied between 1.118 and 1.380 g/cm^3. This implies that, since their densities are above 0.8 g/cm^3, they are classified as HD biocomposites.

The porosity of the coconut shell biocomposite is shown in Fig. 9. Porosity of the biocomposite was measured to be around 9.5% before the treatment with NaOH concentration solution; hence, a significant number of voids or empty spaces in the biocomposite existed. It is noted that due to poor interfacial bonding between coconut shell particles and the epoxy resin, the presence of lignin, hemicellulose, and other impurities remains on the surface of coconut shell particles [43]. The least porosity was observed in the biocomposite sample treated with a 20% NaOH solution; its porosity was measured at 2.5%. For both immersion times of 8

hours and 4 hours, the porosity reduced considerably after soaking.

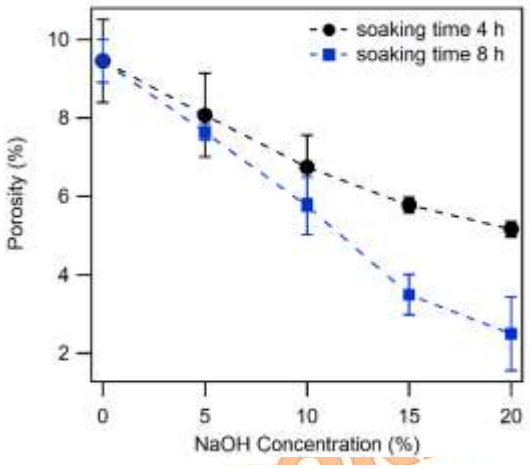

Fig. 9. Porosity of coconut shell biocomposite for different NaOH concentrations with immersion times of 4 hours and 8 hours

The large reduction in porosity as the NaOH molarity increases is an obvious indication of the alkali treatment's efficiency on the particle surface. The alkalization treatment is considered efficient for removing lignin, hemicelluloses, and other surface contaminants from the particles. The removal of these substances from the particle surfaces was confirmed by FTIR analysis, as discussed earlier. This makes the fiber surface cleaner, coarser, and more cellulose-exposed, resulting in better wetting properties of the resin and more successful interlocking. The improved interfacial bond between the fiber and the resin minimizes the porosity of the biocomposite board. The extended 8-hour soaking period effectively modified the particle surface, making the biocomposite generated even more nonporous. This highlights that both NaOH concentration and treatment duration are critical parameters in optimizing the physical properties, particularly porosity, of these biocomposites.

The thickness swelling of the coconut shell biocomposite for different concentrations of NaOH is shown in Fig. 10. Initially, before NaOH treatment, the thickness swelling of the biocomposite measures 10.9%. After 20% NaOH treatment and 8 hours of soaking, the thickness swelling of the biocomposite decreases to 2.8%.

Thickness swelling is primarily influenced by density and porosity; lower porosity reduces water absorption. The greater decrease in value at 4 hours indicates the influence of treatment time; excessive NaOH treatment may have caused some degradation, preventing further enhancement [44].

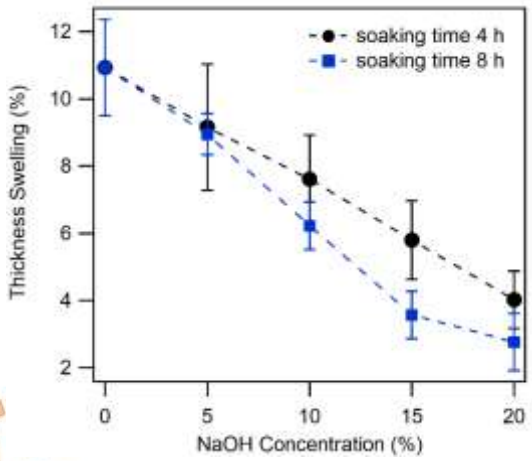

Fig. 10. Thickness swelling of coconut shell biocomposite for different NaOH concentrations with immersion times of 4 hours and 8 hours

The extent of thickness swelling, which is reduced further in the 4-hour treatment, clearly shows that the initial phase of the NaOH treatment is even more effective at removing the most hydrophilic and accessible portions of the lignocellulosic composition. However, the increase in treatment time to 8 hours, even though it results in higher density and improvements in all mechanical properties, is more closely related to overall densification and stress transfer. At longer treatment times, the NaOH treatment may inhibit the reduction in thickness swelling, and the mechanical properties will continue to progress.

From the results, a clear relationship has been established between the material's thickness swelling and its density and porosity. The porosity of the biocomposite material restricts the amount of water that enters the biocomposite board. The higher the amount of voids inside the material, the more the material can absorb water. This results in a high expansion or swelling of the material. Therefore, reducing voids in the material is key to improving the biocomposite's stability.

One of the most interesting observations is the greater thickness-swelling resistance of the sample with 4 hours of immersion compared to 8 hours. Based on the density results, it is anticipated that an enhanced densification effect with an immersion time of 8 hours would have contributed more to thickness swelling resistance than the sample with an immersion time of 4 hours. However, the fact that it is the reverse suggests that, at least during the early NaOH exposure period (4 hours), the most accessible hydrophilic components of the sample are removed extremely efficiently, resulting in relatively improved thickness-swelling resistance. Beyond 4 hours, even with an effective densification process, a complex mechanism involving NaOH interactions within the polymer

matrix may occur. This implies that as the NaOH concentration increases, material degradation or structural changes accelerate and become more pronounced over time. This is indeed expected, since higher NaOH concentrations are known to cause decomposition or reduced bonding strength within the polymer matrix [44], which could lead to a plateau or slight increase in swelling if the degrading process disrupts the overall integrity. However, this was not necessarily observed as an increase here.

Importantly, the results for thickness swelling were also compared with the ANSI standards, which require no more than a permissible swelling value of 8% [45]. From our results, we can see that the biocomposite boards treated with NaOH concentrations of 10%, 15%, and 20% met the required standards, as they did not exceed the permissible swelling value. This implies that the alkali treatment process conducted on the coconut shell particulates was effective, and the resultant biocomposite boards possessed the required dimensional stability. On the other hand, the biocomposite boards treated with 0% and 5% NaOH did not meet the required standards, as they exceeded the permissible limit.

The statistical analysis of the physical properties of the biocomposite with various NaOH concentrations has been conducted. For a 4-hour soaking time, the significant statistical values for density, porosity, and thickness swelling are 0.353, 0.110, and 0.828, respectively. All of these values are greater than 0.05, indicating that the variances of the density, porosity, and thickness swelling data across various NaOH concentrations are homogeneous. The calculated F values for density, porosity, and thickness swelling are 14.497, 30.121, and 11.809, respectively. The value of F theoretical (5%) for df1/df2 (4/10) is 3.48. The calculated F values exceed the theoretical F values, indicating that the NaOH concentration at 4 hours of soaking time significantly affects the biocomposite's physical properties. Similarly, for an 8-hour soaking time, the significant statistical values for density, porosity, and thickness swelling are 0.353, 0.646, and 0.163, respectively. The p-values are greater than 0.05, indicating that the variances of the density, porosity, and thickness swelling data across various NaOH concentrations are homogeneous. The calculated F values for density, porosity, and thickness swelling are 26.383, 63.033, and 52.044, respectively. The value of F theoretical (5%) for df1/df2 (4/10) is 3.48. The calculated F values exceed the theoretical F values, confirming that the NaOH concentration at an 8-hour soaking time also significantly affects the biocomposite's physical properties.

3.3. Mechanical Properties

Modulus of rupture (MOR) is a very important indicator of the bending strength of a material. The results in Fig. 11 clearly show a remarkable improvement in the MOR of coconut shell biocomposites for various concentrations of NaOH.

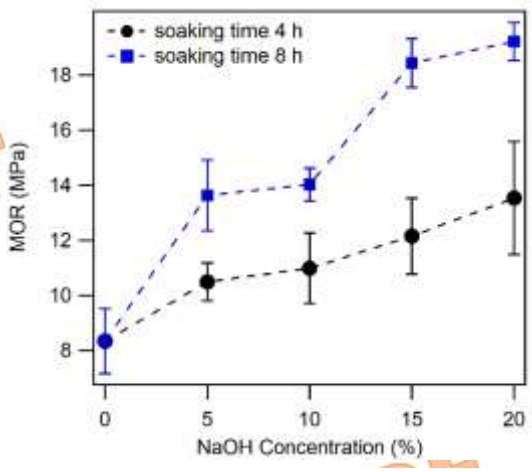

Fig. 11. MOR of coconut shell biocomposite for different NaOH concentrations with immersion times of 4 hours and 8 hours

Initially, without NaOH treatment, the biocomposite showed a bending strength of 8.34 MPa. The low strength in the untreated sample can be attributed to the presence of waxes, oils, and other surface impurities in raw coconut shell particles. According to [29], the presence of non-cellulosic substances creates a barrier layer that does not favor a strong bond between filler particles and epoxy resin. This can be identified as a point of weakness that often leads to stress concentration and, consequently, a reduced MOR.

After treatment with 20% NaOH for 4 hours, MOR values increased to 13.53 MPa, a remarkable rise. The graphical representations in Fig. 8 further clarify that MOR values increased with increasing NaOH concentration for both 4 and 8 hours of soaking time. This particular rise is a direct result of NaOH solutions' ability to alter the surface chemistry and morphology of coconut shells. NaOH solutions effectively remove lignin, hemicellulose, and waxes from coconut shells, yielding a rough surface with high reactivity of the cellulose content, thereby enhancing mechanical interlocking and adhesive interactions with the epoxy resin and resulting in a stronger composite structure.

Most importantly, the data showed that the MOR values of biocomposites treated with the 8-hour process were higher than those treated with the 4-hour process, as previously reported [30, 46]. This further supports the idea that the time required during alkali treatment is as important as the process itself for optimizing bond strength

at the interfacial boundaries. The longer the time, the better the removal of unwanted fractions and, subsequently, the better the fibrillation/etching of the surfaces, thus creating a larger interaction zone between the resin and the particle.

The performance of the biocomposite boards was assessed against the American National Standards Institute [45] for biocomposite boards. Our results indicate that the coconut shell biocomposite boards treated with 15% and 20% NaOH for 8 hours successfully met the requirements of the ANSI standard, classifying them as grade H-1. This indicates that they are useful for higher-strength applications. By contrast, the biocomposite boards treated with 0% NaOH were rated grade M-0, indicating very poor mechanical performance. This attests to the paramount importance of optimizing the NaOH treatment parameters for producing biocomposites with enhanced mechanical properties that meet the set standards.

Modulus of elasticity (MOE), which is the material property denoting the resistance to elastic deformation and material stiffness, was significantly affected by the NaOH treatment of coconut shell particles, as indicated in Fig. 12. For the untreated biocomposite containing 0% NaOH, the MOE was found to be 764.6 MPa, which was quite low. This is because the untreated samples exhibit lower stiffness, primarily due to the presence of lignin and other impurities on the surface of the coconut shell particles. This hydrophobic property, or the hindrance to resin flow, makes the coconut shell particles and epoxy unsuitable for effective interaction and wetting. Therefore, there is poor interfacial transfer of stress, resulting in the entire biocomposite being stiff.

The MOE of the biocomposite significantly increased after NaOH treatment. It measured 4097.2 MPa after 4 hours of immersion and further increased to 4446.7 MPa after 8 hours. The MOE of the biocomposite significantly increased after NaOH treatment. It measured 4097.2 MPa after 4 hours of immersion and further increased to 4446.7 MPa after 8 hours. The consistent upward trend in MOE with increasing NaOH concentration, as seen in Fig. 12, for both soaking durations, underscores the effectiveness of alkali treatment. Alkalization treatment alters the particle surface, primarily by removing amorphous components such as lignin and hemicellulose, along with other impurities. This increases surface roughness and diminishes the fibers' inherent hydrophilicity, thereby improving the wettability and adhesion of treated particles to the polymer matrix.

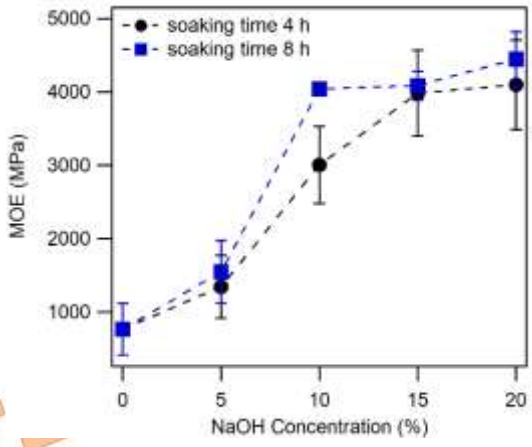

Fig. 12. MOE of coconut shell biocomposite for different NaOH concentrations with immersion times of 4 hours and 8 hours

A stronger interface, as realized in proper alkali treatment, enables greater stress transfer efficiency from the matrix to the reinforcement particles. This ensures equal stress distribution in the composite. Stress concentration in the vicinity of the fiber endings is, therefore, reduced, and as such, the composite stiffness is improved [29]. The 8-hour soaking time yielded an MoE value greater than that for the 4-hour soaking period. A longer soaking time ensures better surface treatment and, hence, a stiffer composite.

The statistical analysis of the mechanical properties of the biocomposite with various NaOH concentrations has also been conducted. For a 4-hour soaking time, the significant statistical values for MOR and MOE are 0.683 and 0.243, respectively. All of these values are greater than 0.05, indicating that the variances of MOR and MOE data across various NaOH concentrations (a 1-hour soaking time) are homogeneous. The calculated F values for MOR and MOE are 10.590 and 57.244, respectively. The value of F theoretical (5%) for df1/df2 (4/10) is 3.48. The calculated F values exceed the theoretical F value, indicating that the NaOH concentration at a 4-hour soaking time significantly affects the mechanical properties of the biocomposite. Similarly, for an 8-hour soaking time, the significant statistical values for MOR and MOE are 0.475 and 0.200, respectively. The p-values are greater than 0.05, indicating that the variances of MOR and MOE data across various NaOH concentrations are homogeneous. The calculated F values for MOR and MOE are 87.783 and 160.664, respectively. The value of F theoretical (5%) for df1/df2 (4/10) is 3.48. The calculated F values exceed the theoretical F values, confirming that the NaOH concentration at an 8-hour soaking time also significantly affects the mechanical properties (MOR and MOE) of the biocomposite.

Comparison of the experimental MOR and MOE results for the biocomposite boards with the ANSI 208.1-2009 standard demonstrates the effectiveness of the method. Biocomposite boards in this investigation complied with the ANSI standard for grade H-1 (after NaOH treatment).

3.4. Thermal Properties

Figures 13 and 14 show the TGA results for the biocomposite treated with different concentrations of NaOH solution at soaking times of 4 hours and 8 hours, respectively. The TGA graph of the coconut shell biocomposite shows a three-stage degradation pattern, as observed in lignocellulosic materials.

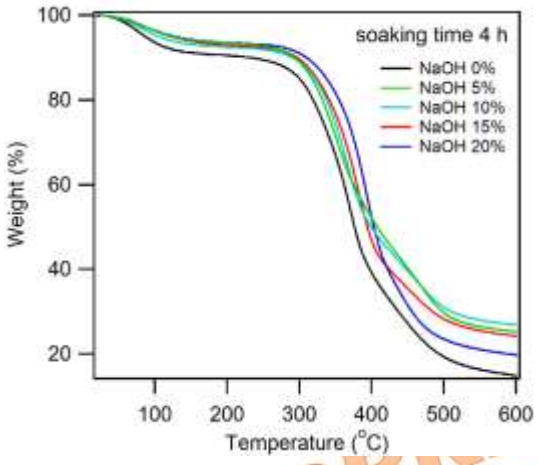

Fig. 13. TGA of coconut shell biocomposite for various NaOH concentrations with 4 h soaking time

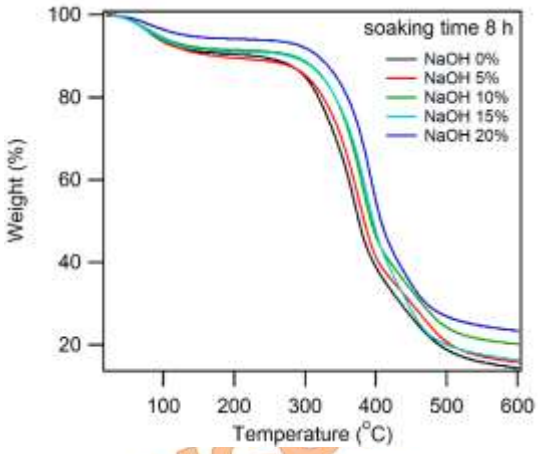

Fig. 14. TGA of coconut shell biocomposite for various NaOH concentrations with 8 h soaking time

The first stage of the biodegradation process, occurring between 80 and 150 °C, mainly relates to the evaporation of the absorbed moisture and the volatilization of other low-molecular-weight materials in the biocomposite. This stage emphasizes the loss of the non-structural, volatile fraction. The second and most pronounced stage of degradation happens between 300 °C and 450 °C. This is the main mass loss and is ascribed to the decomposition of the predominant components of the lignocellulosic material: hemicellulose, cellulose, and a fraction of the lignin present [47]. The amorphous hemicelluloses would be the first to undergo decomposition due to the low crystal density, followed by the more dense celluloses. The lignin, which is highly aromatic and compactly three-dimensional, also contributes during this phase. The third phase of degradation, spanning 450 °C to 500 °C, is primarily attributed to the subsequent and complete breakdown of lignin [48]. This is because lignin has a tough molecular structure that can withstand heat for longer, thereby contributing to its thermal stability at high temperatures. Between 550 °C and 600 °C, about 20% to 30% of the biocomposite remained as a residue. The main constituent of this residue is char, which emanates from incomplete carbonization of the lignocellulosic components; the contribution of lignin to its yield is important.

Most importantly, the TGA results indicate that, following NaOH treatment, the decomposition temperatures were higher for both soaking times (4 and 8 hours). This is a major observation. NaOH treatment is efficient at removing the amorphous portions of the materials, such as the hemicellulose, waxes, and the cellulose component of the lignin, which are generally less heat-stable than cellulose. Essentially, the NaOH-treated samples have cleaner cellulose, which is more ordered and dense. It will therefore take higher temperatures to decompose the NaOH-treated samples, as indicated by the higher decomposition temperatures observed after NaOH treatment. This indicates that the NaOH-treated samples can perform their operations at higher temperatures, thereby broadening the applications of the biocomposites.

The derivative thermogravimetry (DTG) of coconut shell biocomposites is displayed in Fig. 15 for a 4-hour soaking time and in Fig. 16 for an 8-hour soaking time. Before treatment, the coconut shell particles with NaOH, the Tonset and Tmax were 210 °C and 365 °C, respectively. As NaOH-treated coconut shell particles were heated, the Tonset increased to 240 °C for a 4-hour soaking time and 250 °C for an 8-hour soaking time at 20% concentration. The Tmax increased to 392 °C for a 4-hour soaking time and 394 °C for an 8-hour soaking time with 20% concentration. These results confirm that NaOH treatment increases the thermal stability of the biocomposites.

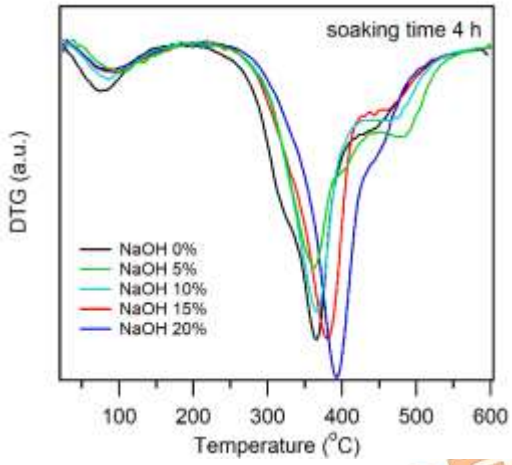

Fig. 15. DTG of coconut shell biocomposite for various NaOH concentrations with 4 h soaking time

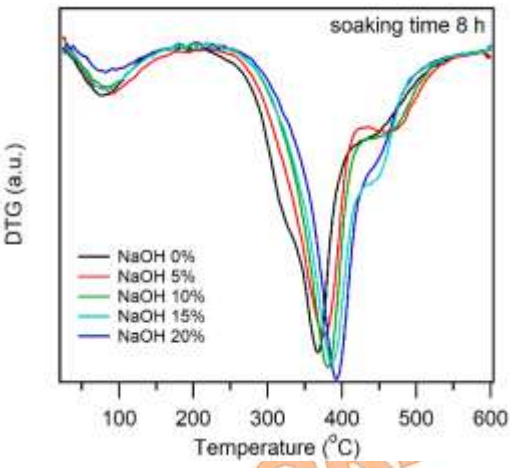

Fig. 16. DTG of coconut shell biocomposite for various NaOH concentrations with 8 h soaking time

The thermal conductivity of the coconut shell biocomposites, measured using the single plate method and presented in Fig. 17, shows a clear trend: NaOH treatment consistently increased the material's thermal conductivity. At the start, when there was no NaOH treatment, the thermal conductivity of the biocomposite was 0.0877 W/m K. This value is fairly low, as expected for untreated lignocellulosic composites, which contain air voids and whose natural components, such as lignin and hemicellulose, can behave like thermal barriers. However, after the NaOH treatment, a significant improvement in thermal conductivity was observed. For example, increasing exposure to 4 hours with 20% NaOH increased the thermal conductivity to 0.1062 W/m K. Further increasing the treatment time to 8 hours with 20% NaOH resulted in an even higher value, reaching 0.1231 W/m K.

The statistical analysis of the biocomposite's thermal conductivity at various NaOH concentrations has also been conducted. For a 4-hour soaking time, the significance of the thermal conductivity data is 0.695. The p-value is greater than 0.05, indicating that the variances of thermal conductivity data across various NaOH concentrations (a 4-hour soaking time) are homogeneous. The calculated F value for thermal conductivity is 0.872. The value of F theoretical (5%) for df1/df2 (4/10) is 3.48. The calculated F value is smaller than the theoretical F value, indicating that the NaOH concentration at a 4-hour soaking time does not significantly affect the biocomposite's thermal conductivity. This could be because of large standard deviations.

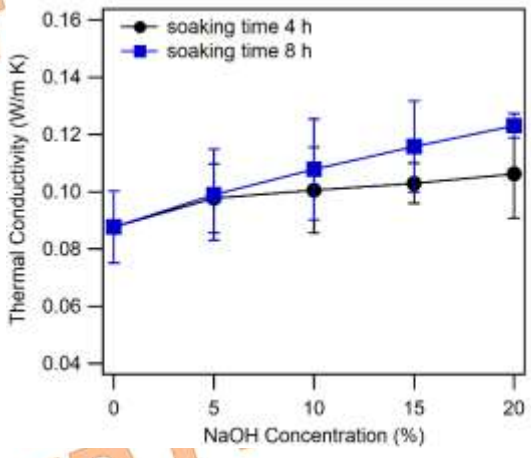

Fig. 17. Thermal conductivity of coconut shell biocomposite for different NaOH concentrations with immersion times of 4 hours and 8 hours

For an 8-hour soaking time, the significant statistical value of thermal conductivity is 0.341. The p-values are greater than 0.05, indicating that the variances of the thermal conductivity data across various NaOH concentrations are homogeneous. The calculated F value for thermal conductivity is 26.898. The value of F theoretical (5%) for df1/df2 (4/10) is 3.48. The calculated F value exceeds the theoretical F value, confirming that the NaOH concentration at an 8-hour soaking time significantly affects the biocomposite's thermal conductivity.

The increase in thermal conductivity is directly correlated with changes in the biocomposite's physical properties resulting from the NaOH treatment. As mentioned earlier, one of the most active agents in alkali treatment is the removal of amorphous components of hemicelluloses, lignin, and surface impurities such as waxes and oils from the cell wall. In this process, a denser, more compact material structure is achieved, as previously demonstrated by the density increase and reduced porosity discussed above. There are fewer internal voids, hence less trapped air, and a poor thermal conductor means a thermally more conductive material.

Moreover, the enhanced interfacial bonding of the treated coconut shell particles and the

polymer matrix increases the heat transfer. A stronger interface will enable more efficient heat propagation through the material, rather than being hindered by weak connections or discontinuities. The higher MOE and MOR values indirectly support this, as greater rigidity and structural integration generally improve thermal conduction.

Another indication of the trend of increasing thermal conductivity with both higher NaOH concentrations and longer soaking times is that the more the material is modified, the higher its thermal conductivity. This is because higher modifications result in greater removal of non-cellulosic content and denser biocomposites, thereby improving the biocomposite's heat conductivity.

3.5. Morphological Property

Scanning electron microscopy (SEM) results, obtained at a magnification of 5000x on coconut shell particle biocomposites, helped elucidate the internal structure and adhesive properties, paving the way for investigations of the physical and mechanical properties. This is shown in Fig. 18, which shows SEM micrographs (untreated and treated coconut shell particles using NaOH). This micrograph clearly shows the formation of voids (marked by arrows in Fig. 18), signifying the void formation in the biocomposite material. This is due to two main factors: trapped air during processing and poor particle-epoxy resin interaction, indicating the formation of voids during solidification.

We used ImageJ to evaluate porosity from SEM images quantitatively. The "Analyze Particles" feature was used to calculate the total pore area in each image. The porosity value was obtained by dividing the total pore area by the total image area, then multiplying by 100%. Before the coconut shell particles were treated with NaOH, the porosity of the biocomposite was 11.4%. After being treated with NaOH solution for 4 hours, the porosity values of the biocomposite were 8.9%, 8.2%, 6.8%, and 5.6% for NaOH concentrations of 5%, 10%, 15%, and 20%, respectively. Meanwhile, for an 8-hour immersion time, the porosity values of the biocomposite were 9.4%, 7.6%, 5.1%, and 4.0% for NaOH concentrations of 5%, 10%, 15%, and 20%, respectively. These results confirm that the porosity of the biocomposite decreases with increasing NaOH concentration, indicating that the bonding between the matrix and the filler improves.

Untreated coconut shell particles naturally contain lignin, hemicellulose, and other non-polar compounds that impede effective chemical interaction with the polar epoxy resin. As observed in the biocomposite boards without alkali treatment shown in Fig. 18 (a), a higher number of voids were present compared to the treated boards shown in Fig. 18 (b) – (i). This strongly suggests that the interfacial adhesion between the untreated coconut shell particles and the matrix was very weak [29]. The incompatibility between the filler's hydrophobic surface and the hydrophilic resin results in poor wetting and incomplete particle encapsulation, leading to numerous unbonded regions.

Further examination of the fracture surfaces via SEM for biocomposite boards treated with 5% and 10% NaOH for 4 hours revealed a less-than-optimal filler-matrix interface. It was clear that the filler was not well-bonded to the matrix. There was a buildup of adhesive and the formation of voids in other places because the distribution was not uniform. There seemed to be a lack of cohesion in the aggregates and the matrices. This resulted in an uneven interface, indicating that the samples' mechanical properties were low.

Conversely, the SEM micrographs of the fracture surface of biocomposite boards treated for only 8 hours with 20% NaOH solution (Fig. 18(i)) showed a much better microstructure. From the micrographs, it is clear that the biocomposite contains well-bonded, evenly distributed filler particles. This greater homogeneity is, in fact, the direct result of the higher density and other enhanced physical properties, including decreased porosity and a lower thickness swelling value. The greater density, in turn, is an indirect function of the lowered porosity and the reduced thickness swelling value, in which the NaOH alkali treatment greatly decreased the biocomposite porosity and, consequently, the biocomposite thickness swelling value by reducing its expansion.

Importantly, the microstructural refinement observed in the SEM images directly correlates with the striking improvement in the modulus of elasticity (MOE). Reduced interfacial voids and increased filler-matrix adhesion during alkali treatment would promote better stress transfer from the epoxy matrix to the coconut shell particles, thereby increasing stiffness. The better MOE values observed after an 8-hour alkali treatment period are therefore attributed to the effectiveness of developing a denser, more integrated interfacial network rather than to reduced hydrophilicity.

The importance of alkali treatment in achieving this enhanced microstructure cannot be overstated. According to the literature [44], NaOH is an effective means of dissolving the wax coating on the filler surface. Such treatment ensures not only the cleaning of the filler surfaces but also their roughening, an important

parameter for the mechanical locking of the filler with the matrix. In addition, the dissociation of hemicellulose and lignin in the cell walls of the filler causes an increase in the number of hydroxyl groups exposed at the cellulose surfaces, further improving their bonding with the epoxy matrix. These complementary roles of alkali treatment, involving mutual improvement of mechanical locking and subsequent enhanced bonding, ensure an improved filler-matrix interface, an important factor in achieving the enhanced physical and mechanical characteristics of treated biocomposites. This improved microstructure is associated with the observed improvements in physical (porosity and thickness-swell values), mechanical (flexural strength), and thermal stability of the biocomposites. These results agree with some recent studies [49, 50]. Generally, an improved filler-matrix interface containing improved bonding between filler and matrix ensures efficient stress transfer, an important factor in realizing the superior characteristics of the biocomposites.

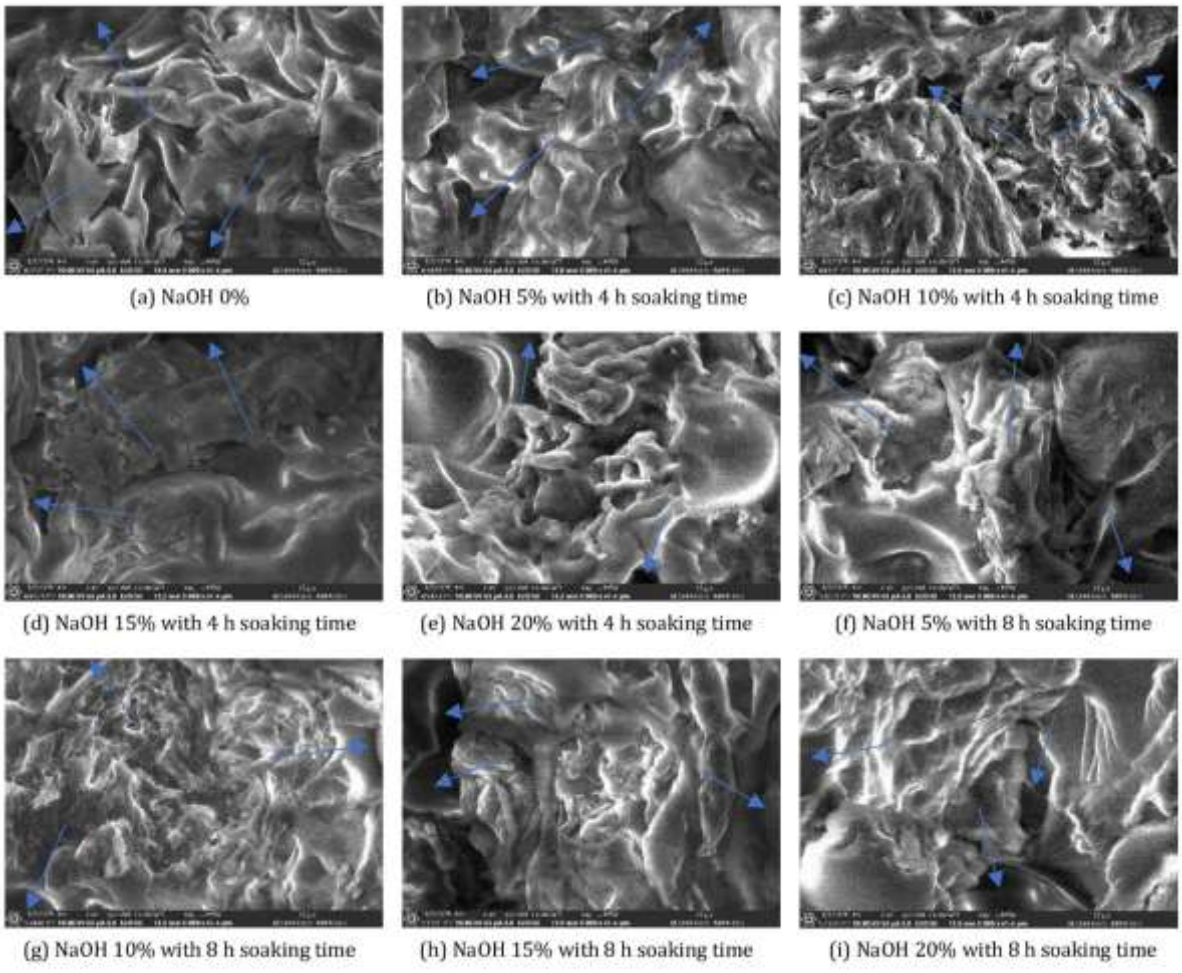

Fig. 18. SEM images of coconut shell biocomposites for various NaOH concentrations

4. Conclusions

In this work, the biocomposite reinforced with a high loading of coconut shell particles was successfully fabricated. This work demonstrates that the alkali, NaOH, treatment significantly improves the properties of coconut shell particle biocomposites. Chemical analyses by XRF and FTIR showed that NaOH was effective in altering the particle surface by reducing hemicellulose and lignin content and affecting inorganic elements such as CaO and K_2O. This creates a more compatible filler for the epoxy matrix. These chemical changes have directly translated into the superior physical, mechanical, and thermal performance-the biocomposites had increased density, reduced porosity and thickness swelling, significant improvements in modulus of rupture and modulus of elasticity, and enhanced thermal conductivity and stability. Further validation of these enhancements was provided by the SEM analysis, which showed a more uniform distribution of the filler material, reduced void content, and improved interfacial adhesion in the treated samples, notably at a 20% NaOH solution concentration and a soaking time of 8 hours. In the end, the optimal concentration

of NaOH solution and soaking time are crucial for producing high-quality coconut shell biocomposites.

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Conflicts of Interest

The author declares that there is no conflict of interest regarding the publication of this article.

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