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Influence of NaOH Treatment Duration on the Properties of High-Loaded Coconut Shell Particle Epoxy Biocomposites

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Abstract

The objective of this work is to study the effect of soaking time of coconut shell particles in 10% NaOH solution on the performance of the biocomposites. The soaking time was varied from 0, 2, 4, 6, and 8 hours. The size of the coconut shell particles used in this study was 200 mesh. The chemical characterization of coconut shell particles before and after treatment with NaOH was performed using FTIR, XRF, and XRD. The coconut shell biocomposites were prepared by using the press method, consisting of 80 vol.% coconut shell particles and 20 vol.% epoxy resin. The density, porosity, water absorption, thickness swelling, modulus of rupture, modulus of elasticity, and thermal decomposition of biocomposites were measured. The results show that the performance of the biocomposites is significantly affected by the immersion time of coconut shell particles in the NaOH solution. Further, it has been observed that the notable improvements in the properties occur after NaOH treatment of the fillers. SEM images showed a significant reduction in particle agglomeration and porosity after NaOH treatment of the fillers. This indicates that the NaOH treatment modified the surface characteristics of the coconut shell particles by removing lignin, which improved their compatibility and interaction with the epoxy resin.

Keywords: *alkaline treatment; biocomposite; coconut shell particles; mechanical property; physical property; soaking time; thermal property.*

Introduction

Biocomposites have emerged as one of the key domains of research due to their ability to replace conventional synthetic materials (Geetanjali and Singh, 2022; Mahmud et al., 2021; Santhosh et al., 2024; Siraj et al., 2024). Biocomposites apply natural materials as reinforcement, offering a wide range of benefits, not only technically relevant but also highly relevant from a sustainability point of view concerning the environment (Faruk et al., 2012; Hoque et al., 2021; Elfaleh et al., 2023). Biocomposites normally take up natural fibers such as coconut fiber, coconut shells, palm fibers, and so on, as reinforcement in polymer matrices. These materials are biodegradable, where they decompose naturally and do not produce waste that harms the environment. Biocomposites have also contributed to reducing the consumption of petrochemical-based feedstocks that are non-renewable and that further contribute to harming ecosystems. Many biocomposites reinforcing materials come from agricultural waste, such as straws from wheat, rice, soy, and canola (Ahmed et al., 2023; Mohanty et al., 2018; Pokharel et al., 2022). By utilizing this waste as a reinforcing material, biocomposites not only increase the economic value of previously worthless waste but also help reduce the problem of waste accumulation. Thus, in the fabrication of biocomposites, the composition of natural fillers should be more dominant than the polymer matrix. The biocomposite that has a more dominant composition of natural fiber (such as more than 50%) is called a high-loaded filler biocomposite.

A major limitation of biocomposites, however, is that their mechanical properties are generally inferior to those of their synthetic composite counterparts. Aravindh et al. (2022), McKay et al. (2024), and Yang et al. (2023) attributed this to the nature of the reinforcing natural fibers, e.g., coconut fiber, jute, or hemp. These fibers possess hydrophilic properties because of their high cellulose content, which causes them to readily absorb moisture from the environment. As a result, they tend to form weak interfacial bonds with hydrophobic polymer matrices such as polypropylene or epoxy resin. Poor adhesion at the fiber-matrix interface inhibits efficient stress transfer between fibers and the matrix, thus causing poor performance and durability in a composite material (Chegdani & Mansori, 2024; Mylsamy et al., 2024).

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Various chemical and physical treatment methods have been developed to improve the compatibility between polymer matrices and natural fibers. One of the most widely used methods is alkali treatment with sodium hydroxide (NaOH) (Jawalkar and Kant, 2021; Kudva et al., 2024; Samanth and Bhat, 2023). The advantages of this treatment are the reduction in hemicellulose and lignin content, with the elimination of impurities on the surface, and construction of a coarse fiber texture to provide an improved interface in cohesion with the matrix. In this respect, Patel et al. (2016) and Kondo et al. (2023) addressed the duration of immersion in the NaOH solution, which is an important parameter to investigate with regard to the optimization of biocomposite properties. Marzuki et al. (2017), Lou et al. (2013), Jayabal et al. (2012), and Faizi et al. (2017) also discussed the importance of the duration of NaOH solution soaking. Accordingly, Jayabal et al. (2012) showed the effect of NaOH immersion time on the properties of polyester-based coir fiber composites, and they reported the highest tensile and flexural strengths after 72 hours for a 5% NaOH solution. Similarly, a study by Marzuki et al. (2017) found that the treatment of kenaf fibers with NaOH solution influenced the composite mechanical properties. The optimum tensile strength and elastic modulus were achieved at a 6-hour immersion duration. Faizi et al. (2017) conducted research on the soaking time of oil palm empty fruit bunch fibers and concluded that the highest value of Young's modulus was achieved for 7 hours of treatment.

Coconut shells are a waste by-product of coconut production. Many tropical countries, including Indonesia, Malaysia, and the Philippines, generate large amounts of this waste each year, much of which is not effectively utilized. Using coconut shell particles as natural fillers in biocomposites is an efficient approach to converting waste into value-added products while reducing environmental hazards. According to Bledzki et al. (2010), coconut shell particles are promising biocomposite fillers due to their high contents of cellulose, hemicellulose, and lignin, at approximately 34%, 21%, and 27%, respectively. Coconut shell-based biocomposite studies have focused on low filler loadings usually below 50% as reviewed by Nadzri et al. (2022). For instance, Singh et al. (2013) studied the water absorption and mechanical properties of coconut shell particle-epoxy composites using 20, 30, and 40 wt.% filler content. Onwumere et al. (2019) developed coconut shell biocomposites bonded with epoxy adhesives and studied their mechanical and thermal properties at 5, 10, 15, and 20 wt.% filler loading. Udhayasankar et al. (2020) fabricated coconut shell-reinforced cardanol composites using 10, 20, 30, and 40 wt.% coconut shell fillers. However, it has been reported that when the coconut shell particle content exceeds 30%, the mechanical properties of the biocomposite decrease significantly (Singh et al. 2014). On the other hand, biocomposites should contain a higher proportion of natural fillers than the polymer matrix to achieve better sustainability.

There are several studies related to the NaOH treatment of coconut shell particle biocomposites. Onwumere et al. (2019) conducted a study on the influence of NaOH treatment on the mechanical and thermal properties of coconut shell epoxy composites. The biocomposites were formulated with 5, 10, 15, and 20 wt.% coconut shell loadings. The NaOH concentration used was 5% with a soaking time of 2 hours. The results of the study showed that the Ultimate Tensile Strength increased from 18 MPa to 23 MPa after the coconut shell was treated with NaOH. Udhayasankar et al. (2020) conducted a study on coconut shell-reinforced cardanol composites. The coconut shell particles were treated with 5% NaOH for a soaking time of 24 hours. They reported an improvement in the flexural, tensile, and impact strength of the composites after the NaOH treatment. However, the studies discussed above did not investigate the effect of soaking time. Natrayan et al. (2024) examined the mechanical properties of hybrid biocomposites using bamboo fiber, eggshell, and coconut shell particles treated with 5% NaOH for a soaking time of 24 hours. This previous research, however, did not consider how the NaOH treatment affected the properties of the composite.

Although there is much research on the use of coconut shells as a biocomposite filler, many gaps remain that need to be addressed. First, most studies have focused on low to moderate filler loadings. Research on coconut shell composites with dominant filler compositions remains very limited. Second, though NaOH treatment is widely recognized for enhancing fiber-matrix adhesion, the influence of varying soaking times on the properties of the high-loaded coconut shell composite has not yet been explored. Addressing these gaps, the present study examines the impact of the NaOH soaking time on the physical, mechanical, and thermal properties of high-load coconut shell epoxy resin biocomposites. Coconut shell is a natural, renewable, and biodegradable material. The application of it in large volumes reduces reliance on synthetic polymers, thereby contributing to eco-friendly composite developments. Previous studies have demonstrated the feasibility of applying coconut shell particles at 70 wt.% (Ismail et al., 2022) and 85 vol.% (Ismail et al., 2020a) composition. Therefore, a large amount of coconut shell particles (80 vol.%) was chosen as fillers in this work. Though previous studies, such as those by Onwumere et al. (2019) and Udhayasankar et al. (2020), have treated coconut shell particles using 5% NaOH, one glaring gap in the existing literature pertains to the effects of higher concentrations. No studies have yet investigated the implications of a 10% NaOH treatment on these particles. Therefore, this study employed a 10% NaOH solution to treat the coconut shell particles.

Methodology

The dried coconut shells in the form of 200-mesh particles were supplied by PT Indratma Sahitaguna Indonesia. Epoxy resin manufactured by the Avian Company was utilized as the matrix. The resin and hardener were mixed in a 1:1 ratio. Sodium Hydroxide (NaOH) in the form of powder was supplied by Merck.

Coconut shell particles were treated using NaOH solutions at 10% concentration. The soaking time of the coconut shell particles in the NaOH solution was varied for 0, 2, 4, 6, and 8 hours. After being treated with NaOH, the coconut shell particles were washed with water until the pH of the water used for washing reached a neutral pH. Then, the coconut shell particles were oven-dried at 105 °C.

Fluorescent X-ray (XRF) manufactured by PANalytical, Netherlands, was used to analyze the content in coconut shell particles before and after NaOH treatment. The measurement was carried out at State University of Malang, East Java, Indonesia. Furthermore, X-ray diffraction (XRD) manufactured by Shimadzu, Japan, type D6000, was also used to assess compound composition and crystalline nature of the particles for the same conditions. The XRD test was carried out at the Physics Department, Universitas Syiah Kuala, Banda Aceh, Indonesia. The chemical composition of coconut shell particles, both untreated and NaOH-treated, was analyzed using Fourier Transform Infrared spectroscopy manufactured by Shimadzu, Japan. The measurement was conducted at the Chemical Engineering Department, Universitas Syiah Kuala, Banda Aceh, Indonesia.

Coconut shell particles, both untreated (as a control) and those treated with NaOH and dried, were mixed with epoxy resin using a mixer until thoroughly blended. The composition of mixture consisted of 80 vol.% coconut shell particles and 20 vol.% epoxy resin for all samples. The biocomposite was fabricated using a pressing technique at room temperature with a mould size of 15 cm x 15 cm. Three samples were tested for each treatment condition. The average and standard deviation of the test results were then calculated. The standard deviation was used to create the error bars on the graph. Three samples were produced and tested for each treatment condition. The average and standard deviation of the test results were then calculated. The standard deviation was used to create the error bars on the graph. Some photos of coconut shell biocomposites made in this study are shown in Figure 1.

Figure 1 Photos of coconut shell biocomposites.

The physical properties of the biocomposite tested were density, porosity, thickness swelling, and water absorption. The mechanical properties assessed in this study included the modulus of rupture (MOR) and modulus of elasticity (MOE). The measurement was conducted at the Physics Department, Universitas Syiah Kuala, Banda Aceh, Indonesia using the universal testing machine manufactured by Hung Ta Company (Taiwan). The methods used for testing physical and mechanical properties have been reported in a previous publication (Ismail et al., 2020a).

Thermogravimetric analysis (TGA) using a Shimadzu DTG-60 (Japan) was used to evaluate the thermal properties of coconut shell biocomposites within a temperature range of 25 °C to 600 °C. The TGA measurement was conducted at the Lhokseumawe State Polytechnic in Lhokseumawe, Aceh Province, Indonesia. Additionally, a Thermo Fisher Scientific Scanning Electron Microscope (SEM) made in USA was utilized to analyze the morphology of the biocomposites. The SEM test was conducted at the Physics Department, Universitas Syiah Kuala, Banda Aceh, Indonesia. The sample used for the SEM test was the fracture surface sample from the mechanical property test.

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Results

X-Ray Fluorescent Data

The chemical compound content in coconut shell particles before and after being treated with NaOH was tested using X-Ray Fluorescent (XRF). The results are shown in Figure 2 and Table 1. The main compounds in the coconut shell particles were CaO of 49.7%, Fe_2O_3 of 21.0%, and K_2O of 15.1%. Previous studies also reported that the coconut shell contained CaO, Fe_2O_3, and K_2O (Ismail et al., 2020b; Leman et al., 2016). After coconut shell particles were treated with 10% NaOH with a soaking time of 2 hours, the intensity of K disappears, and the intensity of Fe reduces. While the intensity of Ca increases as shown in Figure 2. For a 2-hours immersion time, the CaO compound increased to 68.1%, and Fe_2O_3 decreased to 13.5%. However, the K_2O compound droped to 0%.

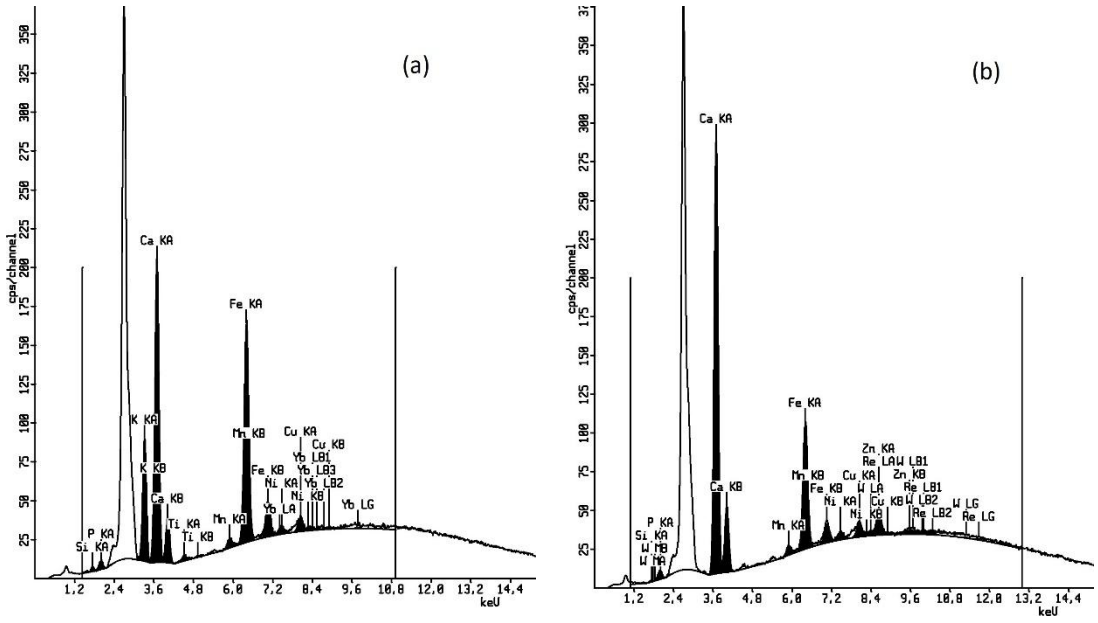

Figure 2 XRF data from the coconut shell particles: (a) before treatment, (b) after treatment with NaOH for 2-hour soaking time.

Table 1 Chemical compounds in coconut shell particles before and after treatment with 10% NaOH

Chemical Compound	0 h	2 h	4 h	6 h	8 h
CaO (%)	49.7	68.1	70.1	73.5	70.1
Fe_2O_3 (%)	21.0	13.5	16.8	14.3	17.9
K_2O (%)	15.1	0	0	0	0
SiO_2 (%)	4.9	3.0	0	0	3.6
P_2O_5 (%)	4.0	3.8	4.2	3.1	3.6
CuO (%)	1.4	1.6	1.9	1.6	1.7
Yb_2O_3 (%)	2.0	0	0	1.0	0
MnO (%)	0.95	1.0	1.2	1.1	2.1
TiO_2 (%)	1.0	0	0	0.8	0
NiO (%)	0.4	0.8	1.0	0.6	0.9
Re_2O_7 (%)	0	3.8	4.7	3.9	0
WO_3 (%)	0	2.2	0	0	0
ZnO (%)	0	1.6	0	0	0

X-Ray Diffraction Data

The X-ray diffraction (XRD) spectroscopy data illustrating the characteristics of coconut shell particles before and after NaOH treatment, with soaking durations ranging from 0 to 8 hours (0, 2, 4, 6, and 8 hours), are presented in Figure 3. The XRD results show that coconut shell particles have a mixture of crystalline and amorphous phases. The amorphous peaks are observed at 15.66 degrees and 22.08 degrees. These broad peaks are associated with cellulose, hemicellulose, and lignin (Bichang'a et al., 2024). The peak observed at 29.22 degrees designates the presence of K_2O. There are some other peaks observed that are MnO, CuO, Fe_2O_3, CaO, and TiO_2. Based on the XRF data, the K_2O reduces to 0% after

treatment (see Table 1). The XRD data (Figure 3) show that the K_2O peak is at 29 degrees. The intensity of this peak decreases significantly as the soaking time increases, indicating that the percentage of K_2O in the coconut shell decreases after NaOH treatment. However, the intensity of K_2O is still present, which means the K_2O is still present in a small amount in the coconut shell. This discrepancy is related to their sensitivity. XRF detects elemental composition (regardless of phase), while XRD detects crystalline structure and phase identification. Potassium hydroxide (KOH) is soluble in water. When the NaOH treatment is followed by washing or soaking in water, the KOH is likely to dissolve in water. Thus, the percentage of K_2O is reduced.

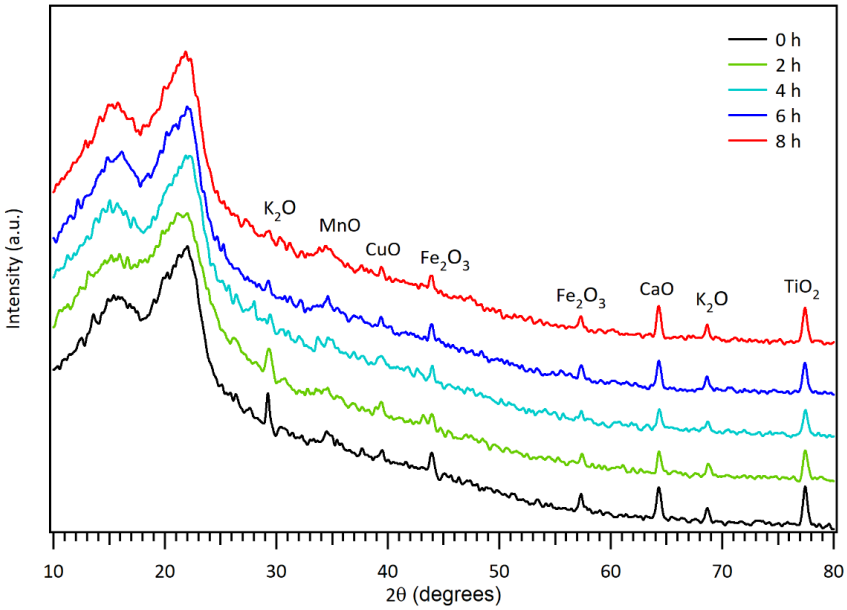

Figure 3 XRD data from the coconut shell particles before and after treatment at various NaOH soaking times.

Fourier Transform Infrared Spectroscopy Data

Fourier Transform Infrared Spectroscopy (FTIR) was employed to analyze modifications in the chemical composition of coconut shell particles following treatment with a NaOH solution. Figure 4 presents the FTIR results obtained from coconut shell particles that were soaked in NaOH solution for different lengths of time. For untreated coconut shell particles (0 h), the spectra of FTIR are displayed in black colour in Figure 4. There are several peaks observed, which are listed in Table 2. FTIR peaks at 3288.6 cm^{-1} (marked in red) of coconut shell particles indicated the presence of -OH stretch vibrations, i.e., the characteristics of natural compounds such as hemicellulose and lignin contained in coconut shells. The intensity of this peak decreased as the soaking time of coconut shell particles in the NaOH solution increased. The FTIR peaks at 2980.0 - 2831.5 cm^{-1} of the coconut shell particle indicated the C-H stretch vibration of the alkane group found in the coconut shells. The intensity of these peaks did not change with NaOH treatment. The FTIR peak at 2121.7 cm^{-1} was associated with the vibration of the stretch of triple bonds, such as C≡C (carbon-carbon), indicating the vibration of the stretch of the triple bond of carbon (C≡C) in alkane compounds. There are some other peaks observed as displayed in Figure 4 and Table 2 including N=C=S stretching, C=O stretching, C=C stretching, N-O stretching, C-H bending, N-O stretching, C-N stretching, CO-O-CO stretching, and C=C bending.

Following treatment of coconut shell particles with NaOH, two peaks at 1766.8 cm^{-1} and 3554.8 cm^{-1} became more pronounced. These peaks are C-H bending and O-H stretching, which are in the class of aromatic compounds and alcohols, respectively. Four peaks disappeared after being treated coconut shell particles with NaOH, namely at wave numbers 1247.9, 1369.5, 1450.0, and 1724.4 cm-1. The peaks at wave numbers 1247.9 and 1724.4 cm-1 (marked in red, Figure 4) were caused by the stretching vibrations of C-N and C=O, respectively. These peaks are the groups present in lignin and hemicellulose. Absorption at wavenumber 1369.5 cm-1 was caused by O-H bending, which is a phenol contained in coconut shell particles. The peak occurring at the wavenumber of 1450 cm-1 was caused by C-H bending, which is an alkane in coconut shell particles.

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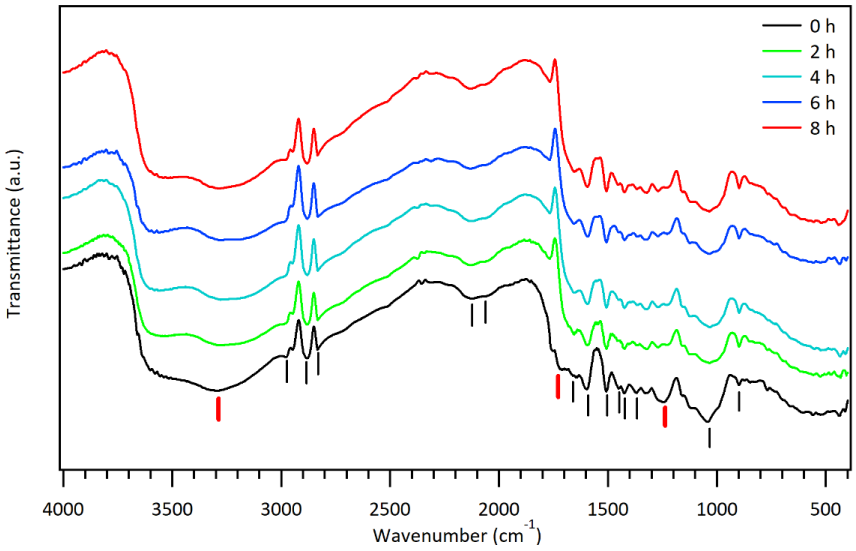

Figure 4 FTIR data from the coconut shell particles before and after treatment at various NaOH soaking times.

Table 2 FTIR data of Coconut Shell Particle Prior to NaOH Treatment

FTIR Results (wavenumber cm^{-1})	Interval Peak Position (wavenumber cm^{-1})	Class
3288.6	3550 - 3200	alcohol
2980.0	3000 - 2840	alkane
2881.7	3000 - 2840	alkane
2831.5	3000 - 2840	alkane
2121.7	2140 - 2100	alkane
2063.8	2140 - 1990	isothiocyanate
1724.4	1740 - 1720	aldehyde
1597.1	1650 - 1566	cyclic alkene
1508.3	1550 - 1500	nitro compound
1450.0	1450 - 1375	alkane
1423.5	1450 - 1375	alkane
1369.5	1372 - 1290	nitro compound
1247.9	1250 - 1020	amine
1043.5	1050 - 1040	anhydride
896.9	995 - 985	alkene
767.7	840 - 790	alkene

Density of Biocomposite

The density of the biocomposites made from coconut shell particles before and after NaOH treatment with varied soaking times (0, 2, 4, 6, 8 hours) is displayed in Figure 5. The results indicated that the density of the coconut shell biocomposites was influenced by the soaking time of the coconut shell particles in NaOH. For a soaking time of 0 hours (before NaOH treatment), the density of the biocomposites was 1.233 g/cm^3. The density value obtained from this study was slightly greater than the density value obtained from the previous study (Ismail et al., 2020a) due to the difference in the composition of coconut shell particles. In this study, the composition of coconut shell particles was 80%, whereas in the previous study by Ismail et al. (2020a), it was 85%. After treatment with 10% NaOH and a soaking time of 8 hours, the density increased to 1.365 g/cm^3.

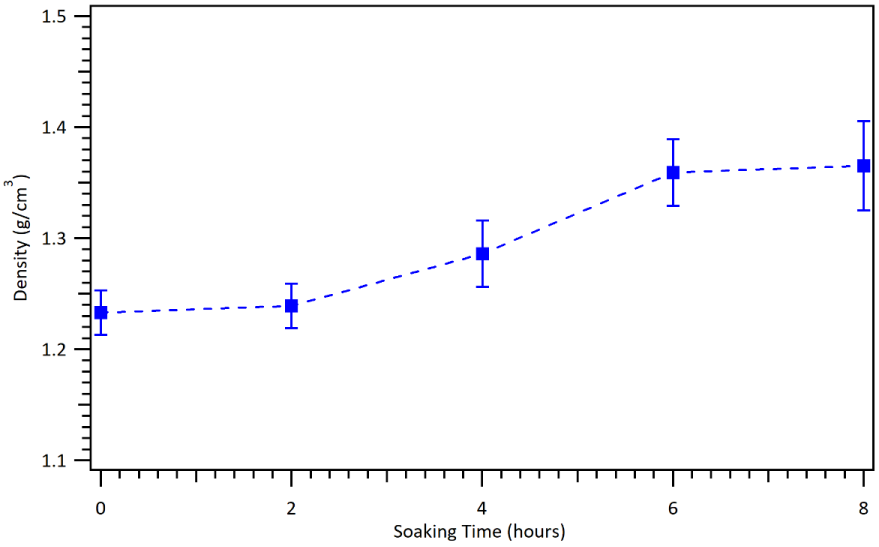

Figure 5 Density of biocomposites at various NaOH soaking times.

Porosity of Biocomposite

The porosity test results of coconut shell particle biocomposites before and after treatment with NaOH where the soaking time varied (0, 2, 4, 6, 8 hours) are shown in Figure 6. The results of the porosity test showed that the porosity of the coconut shell biocomposites was affected by the soaking time of the coconut shell particles in NaOH solution. For a 0-hour soaking time (without treatment), the porosity was 14.8%. The porosity value from this study was slightly lower than the porosity value from the previous study, which was 16.8% (Ismail et al., 2020a). The difference was due to the variation in coconut shell particles composition. After being treated with 10% NaOH with a soaking time of 8 hours, the porosity of the biocomposites decreased to 7.4%.

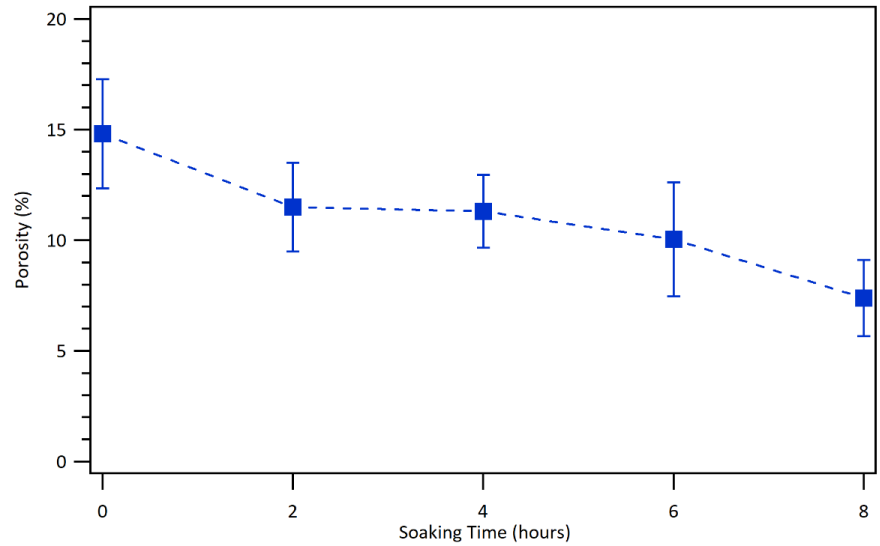

Figure 6 Porosity of biocomposites at various NaOH soaking times.

Water Absorption of Biocomposite

Data from the water absorption test results of the coconut shell particle biocomposites before and after treatment with NaOH where the soaking time varied (0, 2, 4, 6, 8 hours) are shown in Figure 7. Water absorption test data showed that the water absorption of coconut shell biocomposites was affected by the concentration of NaOH in treating coconut shell particles. For a 0-hour immersion time, the water absorption was 12.03%. After being treated with 10% NaOH with a soaking time of 8 hours, water absorption became 8.02%.

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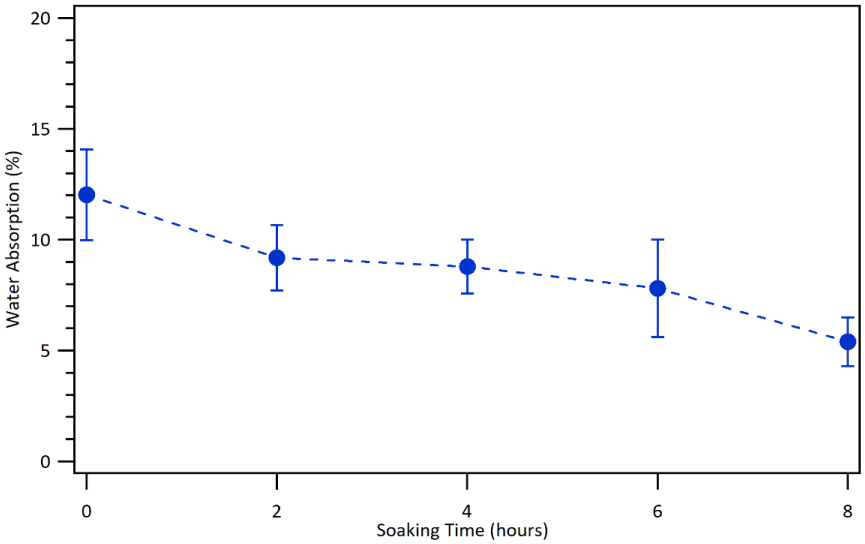

Figure 7 Water absorption of biocomposites at various NaOH soaking times

Thickness Swelling of Biocomposite

Data from the thickness swelling test results of coconut shell particle biocomposites before and after treatment with NaOH where the soaking time varied (0, 2, 4, 6, 8 hours), are shown in Figure 8.

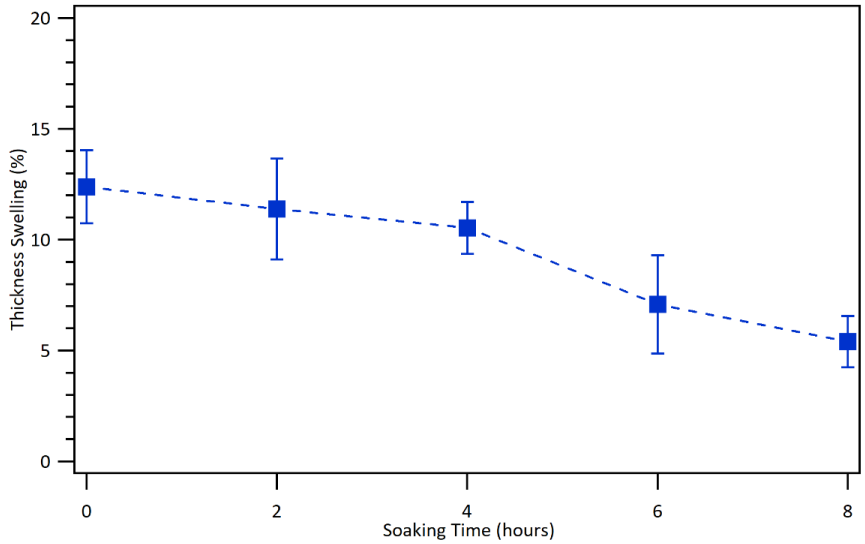

Figure 8 Thickness swelling of biocomposites at various NaOH soaking times.

The results showed that the thickness swelling of the coconut shell biocomposites was also influenced by NaOH treatment. For a 0-hour soaking time, the thickness swelling was 12.38%. After being treated with coconut shell particles with 10% NaOH with a soaking time of 8 hours, the thickness swelling of the biocomposite dropped to 5.39%.

Mechanical Property of Biocomposite

The mechanical properties of the coconut shell particle biocomposites have been measured. The Modulus of Rupture (MOR) is displayed in Figure 9 for the 10% NaOH treatment with variations in soaking time of 0, 2, 4, 6, and 8 hours. The MOR value was 26.48 MPa for 0 hours of soaking (without treatment). After the coconut shell particles were treated with NaOH for a soaking time of 2 hours, the MOR value increased to 29.42 MPa. The MOR value continued to increase, reaching 34 MPa for a soaking time of 8 hours.

The mechanical properties of the biocomposite, Bending Modulus of Elasticity (MOE), are shown in Figure 10 for the 10% NaOH treatment with a variation in the soaking time of 0, 2, 4, 6, and 8 hours. Before being treated with NaOH, the

MOE value of the biocomposite was 2.7 GPa. After treatment with 10% NaOH, the MOE value increased to 3.2 GPa for a 2-hour immersion time. The MOE value increased to 3.8 GPa for a 4-hour immersion time and 4.7 GPa for an 8-hour immersion time. The MOE value increased with the increase in the time of immersion of coconut shell particles in NaOH.

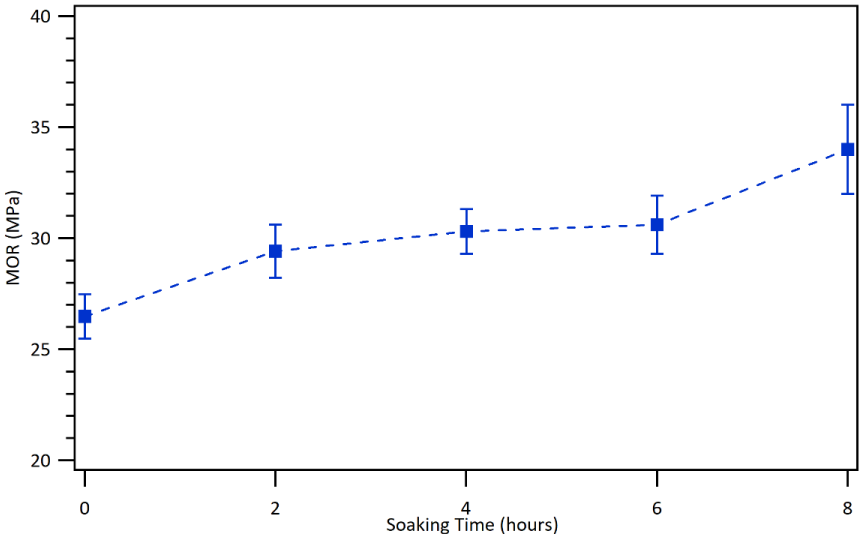

Figure 9 MOR of biocomposites at various NaOH soaking times.

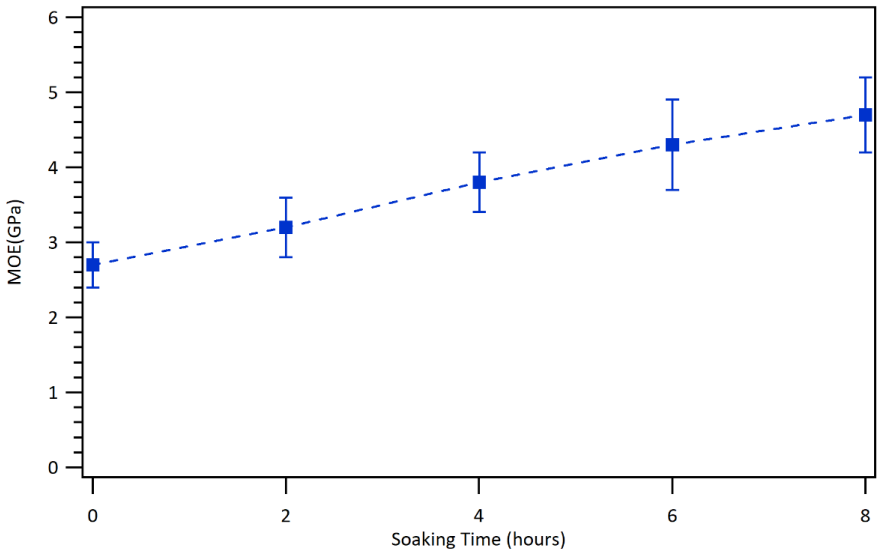

Figure 10 MOE of biocomposites at various NaOH soaking times.

Thermal Property of Biocomposite

The thermogravimetric analysis (TGA) and the differential thermogravimetry (DTG) test results of coconut shell biocomposites are shown in Figures 11 and 12 for the 10% NaOH treatment at various soaking times. The TGA and DTG results showed that three stages of degradation occur in the coconut shell biocomposite. The first stage of degradation occured at temperatures of 80 – 150 °C, caused by the moisture content and other substances in the biocomposites. The second stage of degradation occured at temperatures of 300 – 450 °C, due to hemicellulose, cellulose, and lignin (Yang et al., 2007). The third stage of degradation occured at temperatures of 450 – 500 °C, which is associated with the decomposition of lignin at higher temperatures (Brebu and Vasile, 2010). At temperatures of 550 – 600 °C, the biocomposite residue was around 20 – 25%.

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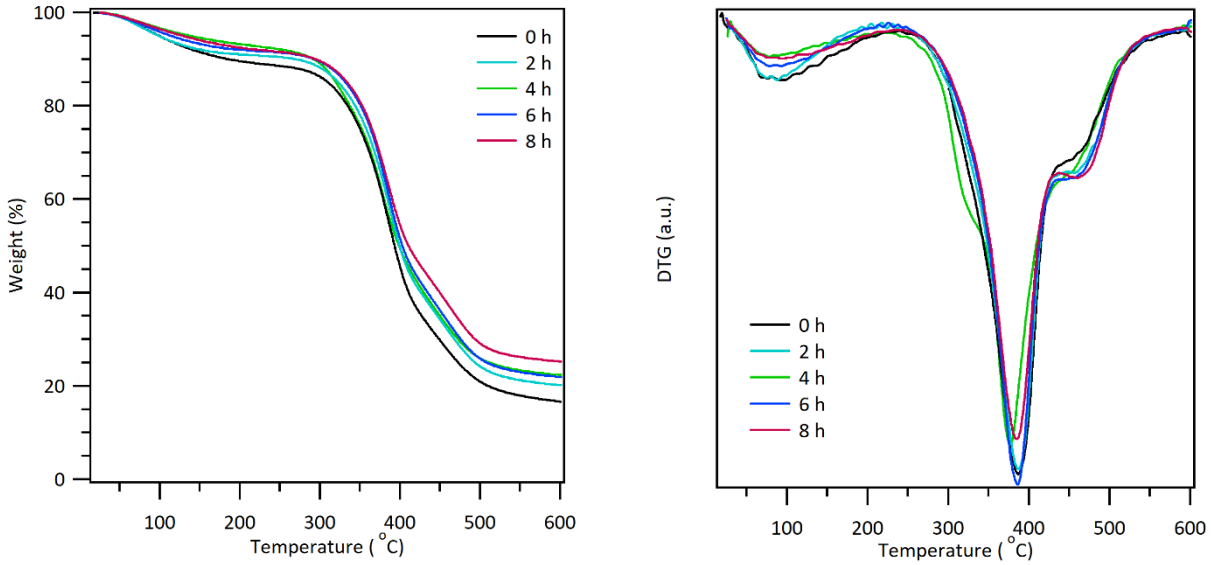

Figure 11 TGA (a) and DTG (b) spectra of biocomposites at various NaOH soaking times.

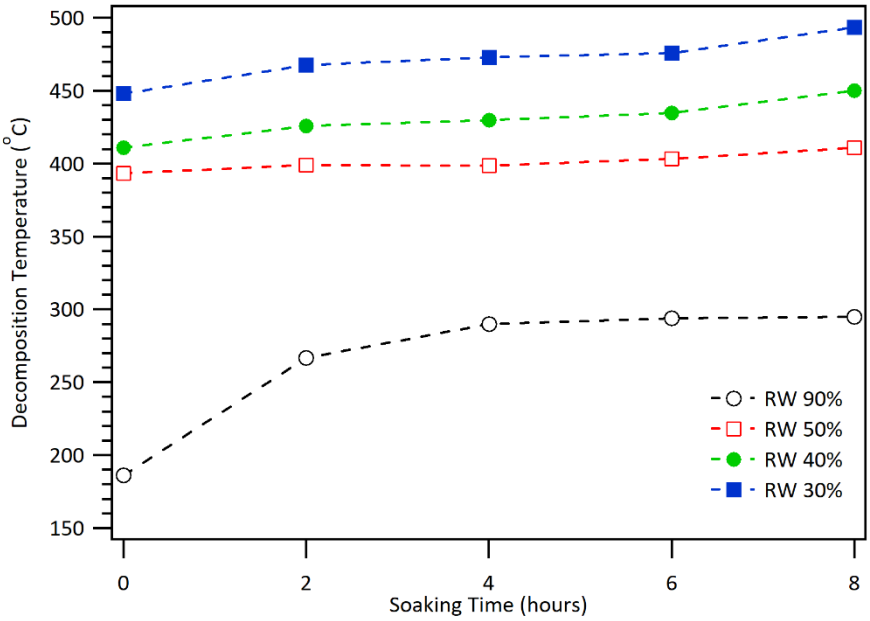

Figure 12 Decomposition temperature of biocomposite for remaining weight (RW) 30%, 40%, 50%, and 90% at various NaOH soaking times.

Morphology of Biocomposite

The SEM micrographs of the coconut shell biocomposites after the NaOH pretreatment, highlighting the influence of the treatment on its microstructure, are displayed in Figure 13. It is found that the filler-matrix interaction was demonstrably different as varying soaking times of the coconut shell particles in a 10% NaOH solution were employed. In the absence of any treatment (0 hours soaking time), there was significant particle agglomeration, with the coconut shell particles clumping together and inhibiting their uniform distribution within the epoxy resin matrix. Additionally, there was a large degree of porosity, indicating the presence of voids and incomplete wetting of the filler by the resin, which may compromise the structural integrity of the biocomposites. The application of treatment with a 10% NaOH solution for 2 hours substantially improved the morphology of the biocomposites. Indeed, both the extent of particle agglomeration and the level of porosity were significantly reduced, as seen from the SEM images. This suggests that the NaOH treatment effectively altered the surface characteristics of the coconut shell particles, by removing some lignin, thereby increasing their compatibility and interaction within the epoxy resin. The improved dispersion of the filler within

the matrix indeed pointed towards a more homogeneous structure, which is actually a pre-requisite for attaining the optimal performance in composite materials. Further refinement in the biocomposites' microstructure appeared to be achieved with increasing soaking time in the NaOH solution beyond 2 hours.

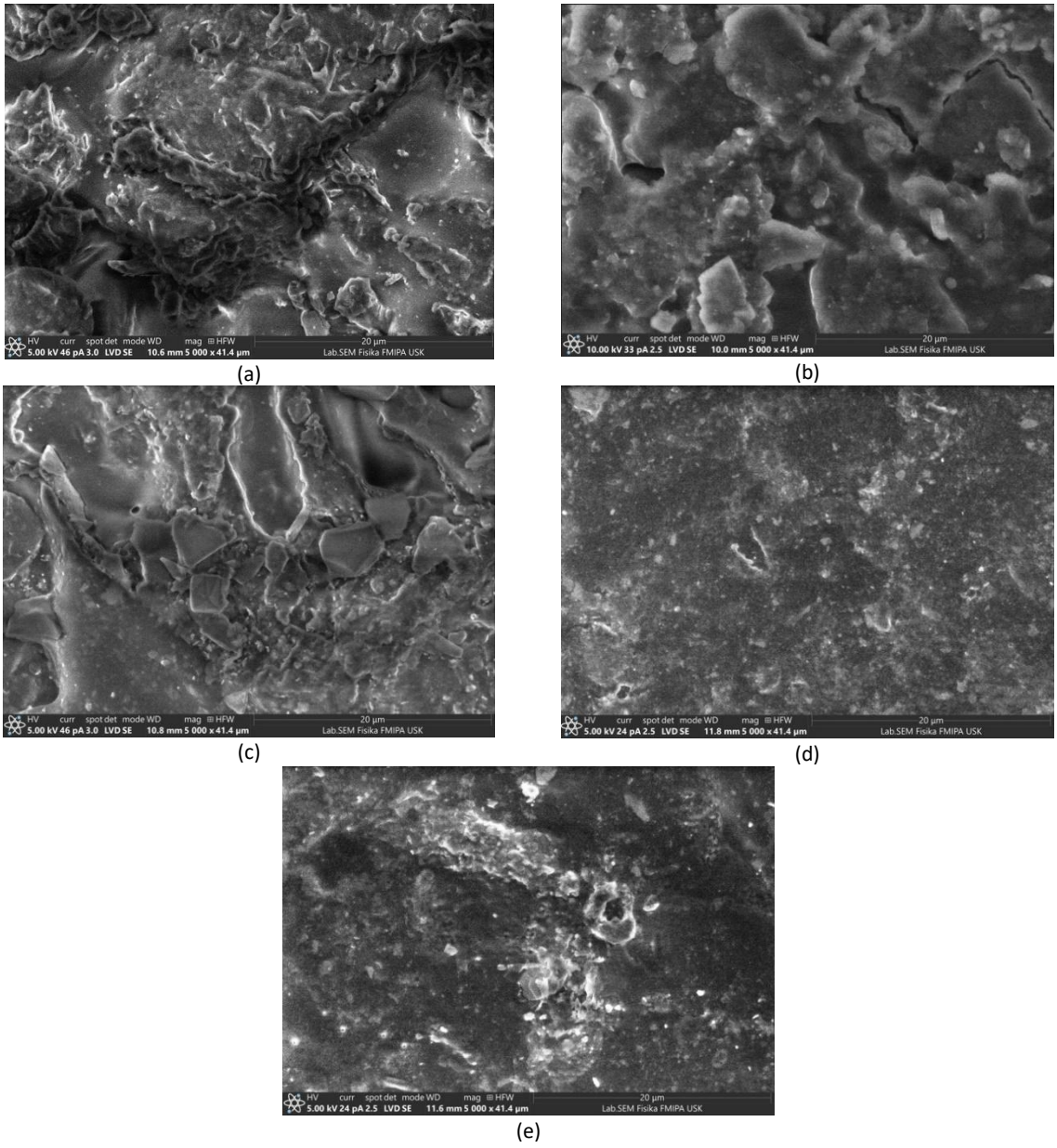

Figure 13 SEM images (magnification of 5000x) of biocomposites at various NaOH soaking times: (a) 0 h, (b) 2 h, (c) 4 h, (d) 6 h, and (e) 8 h.

Discussion

The content of compounds in coconut shell particles is affected by the soaking time. For a NaOH concentration of 10%, the CaO content was 68.1% (2-hour immersion time). It increased to 73.5% for a 6-hour immersion time. Likewise, the content of Fe_2O_3 is also affected by the soaking time of the filler in the NaOH solution. NaOH is an alkaline solution and acts as a saponifying agent. It dissolves and removes the waxy and fatty extractives present on the surface and within the pores of the coconut shell particles. This leads to a change in the composition of compounds within the coconut shell particles. It can be concluded that the content of compounds in coconut shell particles changes when treated with NaOH solution. As a result of this change in chemical composition, the physical and chemical properties of the treated coconut shell particles will be affected. As shown in the FTIR data above, the content of lignin and hemicellulose in coconut shell particles decreased after being treated with NaOH solution. These impact the properties of the biocomposites.

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The results indicate that NaOH functions to remove impurities, lignin, and hemicellulose from the surface of coconut shell particles. The increase in immersion time cleans the particle surface and makes it rougher, which enhances the bonding between particles and the epoxy matrix. This improves the density of the biocomposites. The immersion time of coconut shell particles in the NaOH solution affects the porosity of the biocomposite because it is associated with the chemical and physical processes on the surface and particle structure during treatment. NaOH solution dissolves the lignin, hemicellulose, and impurities from the fillers' surface, that is, from coconut shell particles in the present study. Therefore, the longer the soaking time, the higher the release of non-cellulose components. Removal of impurities reduces the prevalence of empty space or pores in biocomposites, making it denser and, therefore, less porous (Yew et al., 2019), ultimately decreasing the overall porosity in biocomposites. The sodium hydroxide solution treatment of coconut shell particles influenced the water uptake of the biocomposites due to the chemical and physical modification of the surface of the particles. The NaOH solution dissolves lignin, hemicellulose, and impurities present on the surface of coconut shell particles (Checol et al., 2025). Treatment with NaOH makes the surface of the particles more rugged and increases the contact area with the epoxy matrix. The improved bonding between particles and the matrix decreases the chances of the creation of voids/gaps that usually act as an entry route for water, hence decreasing the water uptake. Treatment with NaOH increased the reactivity of the particle surface towards epoxy matrices by forming strong chemical bonds. Strong bonds decrease the possibility of void creation in biocomposites, making it difficult for water to penetrate into the biocomposite, causing a decrease in thickness swelling.

The increase in MOR and MOE values in NaOH alkali-treated biocomposites took place by way of modification to the structure and physico-chemical properties of coconut shell particles acting as reinforcers within the resin matrix. According to Jawalkar et al. (2021) and Kondo et al. (2023), the removal of lignin and hemicellulose increases the load distribution between the resin matrix and the reinforcer. The surface of particles turns out to be cleaner, and with that, the mechanical and chemical adhesion with resin increases. The NaOH solution altered the surface morphology of the coconut shell particles into a coarser one. Surface roughness of coconut shell particles can lead to an improvement in mechanical bonding between the particles and the resin matrix, whereby the stress distribution becomes more evenly distributed along the material. This causes the mechanical properties of the biocomposites treated with NaOH-coconut shell particles to improve.

The highest MOR value obtained from this study was 34 MPa using an 8-hour soaking duration. This value is almost similar to the MOR value from the previous study conducted by Somashekhar et al. (2018), in which the MOR value was 40 MPa for 30 wt.% coconut shell particles and 70 wt.% epoxy resin. Durowaye et al. (2014) reported that the MOR value of the biocomposite was 15.78 MPa for a composition of 30 wt.% coconut shell particles and 70 wt.% polyester. Durowaye et al. (2014) also reported the MOE value of the biocomposite to be 1.9 GPa for a composition of 15 wt.% coconut shell particles and 85 wt.% polyester. It appears that the treatment of filler material with NaOH resulted in achieving excellent MOR and MOE for high-loaded fillers. According to the ANSI Standard (ANSI 208.1-2009), the minimum requirements for H-3 grade particleboard (the highest grade) are 21.1 MPa for MOR and 2.475 GPa for MOE (ANSI, 2009). The coconut shell biocomposite from this study showed 34 MPa for MOR and 4.7 GPa for MOE. This satisfies the requirements of ANSI standards for H-3-grade particleboard. Therefore, the biocomposite from this research has the potential to be used as commercial particleboard.

The TGA and DTG results indicate that the treatment of coconut shell particles with NaOH influences the biocomposite decomposition temperature. From Figure 12, it can be seen that when the coconut shell particles were soaked for an increasingly longer time in the NaOH solution, the decomposition temperature increased. Thus, at a remaining weight of 90% the decomposition temperature of the biocomposite was 186 °C, whereas after the coconut shell particle was treated with NaOH for 2 hrs of soaking, the decomposition temperature increased to 267 °C, and with 4 hrs of soaking, it increased further to 290 °C. A similar trend is observed from Figure 12 for other remaining weights such as 50%, 40%, and 30%. This is due to the fact that the NaOH treatment decreased the lignin and hemicellulose content in the coconut shell particles (reinforcement for composite). The bonding thus improved between the coconut shell particles and the epoxy resin, causing an increase in the thermal properties-degradation temperature-of the biocomposite.

The SEM analysis reveals a consistent trend of the mixture between the coconut shell filler particles and the epoxy resin matrix for better homogeneity. There is a better distribution of particles, with a minimum of pores, indicating an improvement in interfacial adhesion. This improved morphology-filler distribution homogeneity and reduced porosity, directly relates to the improvements in physical properties, such as reduced water absorption; mechanical properties, such as improved flexural strength; and thermal properties, such as increased thermal stability. This was observed in the biocomposite, as evident from other experimental results (Lawal et al., 2023; Nhuapeng et al., 2022). Indeed, this improved interfacial bonding allows efficient transfer of stress across the matrix to the filler, therefore, contribute to the performance of the biocomposite material.

Future Considerations

We have observed a definite relationship between immersion time of fillers in NaOH solution and the resulting physical, mechanical, and thermal properties of biocomposites. Nonetheless, further statistical analysis may be performed to establish the significance of such trends in future work.

NaOH treatment enhances fiber-matrix interactions by removing impurities, waxes, and lignin from the surfaces of natural fibers, hence increasing the availability of hydroxyl groups for chemical reactions. Natural fibers will always retain their hydrophilic nature; hence, they will tend to imbibe water. Prolonged exposure to water will result in the following effects of water seepage: i) fiber expansion, causing micro-cracking between the fiber and the matrix; ii) biodegradation and fungus growth, particularly for natural fibers located in humid climates; and iii) deterioration of fiber properties such as flexural strength and stiffness (Ang et al., 2023). Methods being developed to mitigate the effects include hydrophobic coating techniques, the use of silane coupling agents, and the combined use of natural and synthetic fibers.

Other than hydrolysis, another major factor affecting the durability of biocomposites treated with NaOH is ultra-violet, or UV, rays. This has been known to damage biocomposites in terms of reducing their durability. Photo-oxidation caused by exposure to UV rays has been known to damage both the matrix material and the fiber. The consequence of this damage was, therefore, discoloration, chalking, or embrittlement, which weakened its mechanical strength (Yousif and Haddad, 2013). Remedial measures must, therefore, be put in place to ensure that it resists damage caused by UV rays for it to have a longer lifespan.

In this regard, a combination of NaOH treatment and other modification methods, such as silane coupling agents, can be a promising approach to enhance performance in biocomposites (Sahai et al., 2022). The primary impact of NaOH treatment lies in eliminating lignin, hemicellulose, waxes, and impurities from the surfaces of plant fibers. This increases the surface roughness, while also exposing more hydroxyl groups that chemically activate fibers and, hence, improve fiber-matrix adhesion. The additional reinforcement of the interface is provided by covalent bonds between the fiber and the polymer matrix through the action of silane coupling agents. Silanes act as molecular bridges and enable efficient stress transfer across the interface. Silanes interact more effectively when applied subsequent to NaOH treatment, causing an increase in the number of active sites developed during the alkali pretreatment. The thermal stability, as evidenced from TGA, is also improved for silane-treated fibers due to reduced moisture absorption and improved encapsulation by the matrix (Yeh et al., 2015).

Scaling up the production of biocomposites from laboratory levels to fully-fledged industrial production has many opportunities for commercial success, but there are many technical difficulties along the way. In most cases, natural cellulosic fibers from plants have high variability in their characteristics related to climatic, harvesting, or post-harvest processing factors (e.g., climate, time of harvest, or processing methods, yet unknown or undetermined as of now (Darawsheh et al., 2022; Dayan et al., 2022). These variabilities can significantly undermine the performance characteristics or working life of the finalbiocomposites. There is also a need for large-scale laboratory production redesign for curing, mixing, or forming, all of which require immediate or pressing changes. Any scaling up of production from small laboratory production to large-scale, fully automated, or continuously running industrial production systems would naturally involve certain variabilities in terms of temperature, pressure, or mixing patterns, which can work against product uniformity or product value (Bumphenkiattikul et al., 2018).

Conclusion

This study concerned with the effects of NaOH treatment on coconut shell biocomposites. XRF and XRD tests indicated calcium (CaO), iron (Fe_2O_3), potassium (K_2O), and silicon (SiO_2) as major components in the coconut shells. After NaOH treatment, calcium increased significantly, and iron and potassium decreased, with a significant reduction in potassium. A reduction in lignin content after the NaOH treatment was further confirmed by FTIR results. The present work confirms that NaOH treatment enhanced the performance of coconut shell biocomposites to a large extent. With an increase in soaking time, density increased to 1.365 g/cm3, and consequently, porosity and water absorption decreased to 7.4% and 8.02%, respectively, and the mechanical strength improved accordingly, with a MOR of 34 MPa and MOE of 4.7 GPa. The obtained values indicate that our biocomposites met the commercial particleboard standards (ANSI 208.1-2009). The thermal stability improved after NaOH treatment, which was reflected in the increase of decomposition temperature of the biocomposites to 295 °C. Improvement in the filler dispersion and reduction in voids were observed in the SEM images after NaOH treatment. Overall, NaOH treatment improves the performance of coconut shell biocomposites. There are several areas in which this study may be continued in the future. First, treating the coconut shell particles with NaOH and silane to further improve the biocomposites' performance. Additionally, biocomposite

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products should be coated to prevent easy water absorption and mildew/fungal growth. Second, it is necessary to produce larger-sized samples (scaling up the fabrication of biocomposites from the laboratory to industrial level).

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Compliance with ethics guidelines

The authors declare they have no conflict of interest or financial conflicts to disclose.

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