

## EVALUATION OF TREATMENT PERFORMANCE OF DIFFERENT METHODS ON WHEAT STRAW COMPOSITES

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### Highlights

- Treatment methods are essential to ensure compatibility with the matrix
- The morphological form of the roughness after treatment affects wetting and mechanical interlocking
- Since microwave breeding is an energy-based process, it fulfills its application purpose through many molecular mechanisms

Article Info	Abstract
<b>Article History:</b> Received: May 25, 2024  Accepted: June 26, 2024	The objective of this research is to evaluate the impact of both traditional and innovative techniques on the qualities of wheat straw, as well as the mechanical performance of epoxy composites which use wheat straw as a reinforcing material. Two different methods were used for treatment: Alkali treatment (AIT) was carried out using NaOH at concentration of 5% w/v for 1 hour at 25°C, Microwave irradiation (MwT) was performed at 300 W for 20 minutes. It was found that Alkali and microwave treatment both changed the surface morphology of wheat straw. Microwave treatment provided further improvement in the mechanical properties of the composite materials, enabling a 37% increase in tensile strength and 62% increase in flexural strength. Microwave reclamation, which is cleaner and performs better than chemical reclamation, shows promising performance for more sensitive material application areas.
<b>Keywords:</b> Wheat straw; Microwave; Alkali; Composite	

## Buğday Sapı Kompozitlerde Farklı Yöntemlerin Islah Performanslarının Değerlendirilmesi

Makale Bilgileri	Öz
<b>Makale tarihçesi:</b> Geliş: 25 Mayıs 2024  Kabul: 26 Haziran 2024	Bu araştırmanın amacı hem geleneksel hem de yenilikçi tekniklerin buğday samanının nitelikleri üzerindeki etkisini ve buğday samanını takviye malzemesi olarak kullanan epoksi kompozitlerin mekanik performansını değerlendirmektir. İşlem için iki farklı yöntem kullanılmıştır: Alkali işlem (AIT) 25°C'de 1 saat süreyle %5 w/v konsantrasyonda NaOH kullanılarak, Mikrodalga ışınlama (MwT) ise 20 dakika süreyle 300 W'da gerçekleştirilmiştir. Alkali ve mikrodalga işlemlerinin her ikisinin de buğday samanının yüzey morfolojisini değiştirdiği bulunmuştur. Mikrodalga işlemi, kompozit malzemelerin mekanik özelliklerinde daha fazla iyileşme sağlayarak çekme mukavemetinde %37 ve eğilme mukavemetinde %62 artış sağlamıştır. Kimyasal ıslaktan daha temiz olan ve daha iyi performans gösteren mikrodalga ıslahı, daha hassas malzeme uygulama alanları için umut verici bir performans göstermektedir.
<b>Anahtar Kelimeler:</b> Buğday sapı; Mikrodalga; Alkali; Kompozit	

## 1. Introduction

The growing concern for the environment has necessitated a shift in perspective when it comes to engineering materials and their design. There is now a renewed interest in natural materials that are obtained from safe, renewable, and sustainable sources, as they show great potential in meeting industrial demands. These materials can be used in their natural form, or in forms such as powders, fibers, straws, or husks that are obtained as harvest residues. Lignocellulosic natural resources, in particular, offer exceptional advantages over traditional synthetic materials, owing to their inherent properties such as light weight, low cost, renewability, and sustainability. Among these, cereal straw has emerged as a highly promising natural ingredient, which is widely used as waste biomass in various industrial applications, owing to its impressive characteristics.

Wheat straw is a by-product of wheat production in agriculture. In Turkey, for instance, the production of wheat in 2016 amounted to 20.6 million tons, which, considering the wheat-to-wheat straw ratio of 3:1 (Talebniya, Karakashev, Angelidaki, 2010), results in approximately 7.4 million tons of wheat straw produced at the end of the process. While most of the wheat straw is utilized as animal feed, the remaining amount is either left on the ground or burned. Unfortunately, burning the straw leads to environmental pollution and reduced soil productivity. In recent years, several studies have been conducted to address these issues and increase the utilization of this value-added by-product in various industrial and technical applications.

Recent studies have highlighted the potential use of wheat straw as a natural reinforcement in polymer-based composite production, owing to its lightweight, low cost, and relatively high thermal stability. However, wheat straw contains different components such as lignin, silica, carbohydrates, wax, silica, cutin,

and some lipophilic substances, both on its interior and exterior surfaces. This structural difference between the two surfaces of wheat straw presents a significant challenge in composite production, as it creates incompatibility between the straw and polymer matrix (Xu, Minzhi, Xiaoyan, 2017). The lipophilic substances on the exterior surface cause strong adhesion forces and superficial adherence to the matrix, while the hydrophilic heteropolysaccharides containing hydroxyl (OH) and carboxylic acid (COOH) groups lead to weak adhesion forces and poor interfacial bonding (Mengeloglu, Karakus, 2008; Vaisanen et al., 2016). These factors inhibit the penetration of the hydrophobic matrix into the cellular structure of the wheat straw, leading to the formation of micro void spaces and insufficiently wet regions within the composite, resulting in impaired stress transfer from the matrix to wheat straw. Moreover, the compatibility between wheat straws and polymer matrices is not only influenced by interfacial attraction, but also by surface properties such as porosity, roughness, hydrophilicity/hydrophobicity, and functional groups (Xu, 2017). In addition, large-scale composite production with wheat straw for outdoor applications can be challenging due to their tendency to absorb moisture and poor dimensional stability caused by micro-cracking of the matrix around the swollen reinforcements.

In composite material manufacturing and various industrial applications (such as biofuel production, component extraction, or reinforcement), numerous physical (Zou, Shah, Yiqi, 2010), thermal, and chemical treatment methods have been employed to address the incompatibility stemming from the substrates present in wheat straw (both on the surface and within the biomass). These treatment applications aim to enhance compatibility between the reinforcement and the matrix by altering the proportion of structural components in the reinforcement or by introducing functional groups on the surface, as well as

by creating a suitable topographical texture. Chemical treatments, ion bombardments (plasma), microwave irradiation (as reviewed), and heat treatment play integral roles in this process. They function by either reacting with intrinsic hydroxyl groups to eliminate them or by forming new functional groups with a greater bonding tendency to the matrix material. These reactions also lead to a reduction in the proportions of wax, lignin, and hemicellulose present in the natural structure. Consequently, changes in surface characteristics, including topography, hydrophilicity, and surface free energy, become inevitable. In this context, the enhancement of mechanical interlocking between the matrix and the reinforcement becomes apparent. This is primarily attributed to the rougher surfaces resulting from the removal of irregularities or the formation of new functional groups through interactions and reactions.

Treatment mechanisms act in various ways on the reinforcement structure. Chemical treatments, such as alkali treatment, result in the partial removal of hemicellulose, lignin, and pectin content by dissolving the primary cell wall of the fiber structure. This process increases the number of potential bond-forming reactions due to the lack of OH groups on the fiber surface (Alemdar, Mohini, 2008; Huner et al. 2017). Alkaline treatment also enhances the amorphous cellulose content on the fiber surface, exposing short-length crystals through cellulose depolymerization. This exposure facilitates interactions between the OH groups, imparting hydrophilicity to the cellulose molecule and enhancing its interaction with alkali chemical agents (Mohanty, Misra, Drzal, 2001). Subsequently, substitution reactions form fiber-cell-O-Na groups between cellulose molecular chains, reducing hydrophilic OH groups and increasing the wettability between the fiber and the hydrophobic matrix (Kabir, wang, Lau, Cardona, 2012, Väisänen et al. 2016). Microwave irradiation treatment, predominantly used as a pre-treatment method to improve fermentation (Li et al, 2016) and biofuel

efficiency (Satpathy et al, 2014), acts as a heating process in the treatment of natural reinforcement materials (Sgriccia, Hawley, 2007). Depending on microwave power and processing time, it can have various effects on wheat straw. Microwave irradiation induces weight loss by removing water, low-molecular-weight organic volatiles, and pyrolyzing internal straw (Sgriccia, 2007 and Zhao, 2014). Additionally, it enhances hydrophobic characteristics, reducing moisture uptake and decreasing atomic H/C and O/C ratios associated with hydrophilicity due to volatile removal (Satpathy et al, 2014). Cross-linking occurs through covalent interactions between polymer molecular chains and the reinforcement surface. Notably, the physical, chemical, or irradiation treatment studies can lead to different directions in terms of surface hydrophilicity (more hydrophilic or more hydrophobic), depending on the intensity and application time. Physically, the treatment expands the microporous structure of the straw surface, increasing the interaction (wetting) area of the interface with the matrix material. Overall, these changes contribute to improvements in mechanical properties such as internal bond strength, modulus of elasticity, and modulus of rupture (Li et al, 2016 and Xu et al, 2017]. It is noteworthy that no research has compared the effects of different treatment methods under the same study. There is a need for studies that broaden the perspective on both the methods used and the utilization of wheat straw as reinforcement in composite production.

This study was carried out in order to investigate the effects of the treatment with simple chemical, microwave irradiation on the wheat straw and the reflections of these changes on the epoxy based composite material. It has been noticed that quite a few studies have been conducted using different treatments to improve the physical and mechanical properties of the wheat straw. The results of this study will contribute to remedy deficiencies by providing detailed information.

## 2. Materials and Method

### 2.1 Materials

Wheat straw was sourced from the Thrace region in Turkey and subjected to natural drying under sunlight for one month to eliminate surface water post-harvest in 2019. The resulting dry matter content of the wheat straw was 94.2 wt%. Before treatment, the wheat straws were precision-cut to sizes of 10-12 cm using a razor blade and then further dried under vacuum conditions at 70 °C for 24 hours. The polymer matrix utilized in the study was MGS LR 160 (Hexion), a commercial epoxy resin obtained from Dost Kimya Ltd. The technical characteristics of the epoxy resin, as provided by the manufacturer, are outlined in our previous study [reference]. Additionally, MGS LH 160 (Epikure) served as the curing agent in the epoxy, applied at a concentration of 0.40 w/w. The epoxy's properties, as per the manufacturer's data sheet, include a density of 1.17 g/cm<sup>3</sup>, viscosity of 800 MPas, tensile strength of 55 MPa, tensile modulus of 13 GPa, flexural strength of 125 MPa, and flexural modulus of 15 GPa. For alkaline treatments, sodium hydroxide (NaOH) and acetic acid (CH<sub>3</sub>COOH, glacial, ≥ 99.85%) were procured from Sigma-Aldrich (St. Louis, MO, USA). High purity water, with a conductivity less than 0.5 μS cm<sup>-1</sup>, was obtained from a PURELAB® Option-Q (ELGA LabWater, UK) unit.

### 2.2 Alkali treatment

The wheat straws underwent alkalization by exposure to 5% w/v NaOH aqueous solution. To facilitate this process, the straws were immersed in NaOH solutions for 1 hour at 25±1 °C to eliminate irregularities. Subsequently, the treated straws were thoroughly cleansed with distilled water and neutralized using an acetic acid solution (10 mL in 1 L water). This was followed by multiple washes with fresh distilled water until complete removal of NaOH. Finally, the straws were dried in an oven at 80 °C for 5 hours and then cooled in a desiccator. The alkali-treated wheat straws, denoted by specific codes in Table 1, were securely stored in a vacuum-sealed plastic bag to prevent atmospheric moisture contamination. This precaution was taken in anticipation of composite preparation and subsequent analyses.

### 2.3 Microwave treatment

The wheat straws were processed using a Kenwood Microwave Oven (Model MW587, London, UK) in a household setting, operating at a power intensity ranging from 0 to 1400 W and a frequency of 2.45 GHz. This apparatus is equipped with a glass turntable plate, a digital power control panel, and a time control panel. Initially, the samples were positioned at the center of the glass turntable plate. Subsequently, the microwave power was set to 300 W and the straws were exposed for 20 minutes. Following the microwave treatment, the treated samples (refer to Table 1) were stored using the previously mentioned method.

**Table 1.** Sample codes for the untreated and treated wheat straws

Untreated wheat straws		Alkali treated wheat straws			Microwave treated wheat straws		
Int.	Ext.	NaOH concent. (%)	Int.	Ext.	Exposure power-time	Int.	Ext.
Unt-IWS	Unt-EWS	5	5AIT-IWS	5AIT-EWS	300W-20min	300:20MwT-IWS	300:20MwT-EWS

Int: Interior surface, Ext: Exterior surface

## 2.4 Preparation of Composite Samples

Composite samples were fabricated employing the hand lay-up technique with vacuum assistance. The epoxy resin was meticulously prepared by blending it with the recommended curing agent at a manufacturer-specified ratio of 100:32. To ensure optimal homogeneity, the mixture underwent thorough stirring with a glass rod, eliminating any potential bubbling, and was further subjected to 30 minutes of sonication. The molds, featuring dimensions in accordance with ASTM D 3039 and ASTM D 790 standards, were constructed using glass and transparent PMMA. To prevent adhesion, a mold release agent was applied, and each straw was carefully immersed in the epoxy, with placement into the mold facilitated by hand and forceps to ensure comprehensive wetting. The fiber volume fraction ( $V_f$ ) for all composites consistently fell within the range of 0.45–0.47. Subsequently, the molds were subjected to a vacuum, facilitated by a bag, for a duration of 6 hours under pressure. The ensuing step involved post-curing at 50°C for an additional 6 hours. Wheat straw reinforced epoxy composites were denoted by codes in Table 2.

**Table 2.** Sample codes for the untreated and treated wheat straws reinforced composites

Untreated wheat straws r-comp.	Alkali treated wheat straws r-comp.	Microwave treated wheat straws r-comp.
UWSc	AITWSc	MWTWSc

## 2.5 Water absorption test

The water absorption test was conducted in accordance with ASTM D-570-98. The test specimens were rectangular bars measuring 76.2 mm in length, 25.4 mm in width, and 3.2 mm in thickness. The dry weights of the samples ( $W_d$ ) were determined using a precise balance (Sartorius, Model Ed 224s, Gottingen, Germany, accuracy  $\pm 0.0001$ ) at room temperature. Subsequently, the samples were immersed in a water bath (Memmert, Model WBN 22, Germany) set at

temperatures of 25 and 90 °C for a duration of 24 hours. Weight measurements were recorded at 1, 2, 3, 4, 5, 6, 12, and 24 hours for each sample. At regular intervals, the samples were removed from the water, and any excess water on the surfaces was carefully wiped off using blotting paper. The wet weights ( $W_w$ ) were then measured. The percentage of water absorption was determined by calculating the weight difference using Equation (1). The results were reported as the average of three replicate measurements. Additionally, the normalized water absorption ( $WA/WA_0$ ) was defined as the ratio of water absorption values to the initial values of water absorption

$$\text{Water absorption (WA, \%)} = \frac{W_w - W_d}{W_w} \times 100 \quad (1)$$

Where,  $W_d$  is the initial dry weight of the samples (g) and  $W_w$  is the weight of the samples after exposure to water (g).

## 2.6 Atomic Force Microscopy and Scanning Electron Microscopy

The surface topographies of the untreated and treated wheat straws were investigated by atomic force microscopy (AFM, Nano Magnetics Instruments, Ankara, Turkey) using in contact mode. The roughness of the samples was quantified as the root mean square ( $S_q$ ), derived from an average of five independent measurements over a scan area of 10  $\mu\text{m} \times 10 \mu\text{m}$ . The silicon nitride ( $\text{Si}_3\text{N}_4$ ) probe employed in the analysis featured a curvature radius of 10 nm, as specified by the manufacturer. A cantilever spring constant ( $k$ ) of approximately 0.02 N/m was selected due to the high force gradient in air. The calibration of cantilever spring constants was performed using a reference cantilever with a precisely controlled force constant. The scan rate in the force channel was set at 6 Hz.

The morphology of the composite surfaces and the fractured composite samples were examined using a Quanta™ FEG 250 scanning electron microscope (Oregon, USA) with an acceleration voltage of 2 kV and a large field low vacuum SED (LFD) detector.

## 2.7 Fourier transform infrared spectroscopy (FTIR)-attenuated total reflectance (ATR)

The differences in chemical composition of untreated and treated wheat straws were examined with Fourier transform infrared (FTIR) spectrophotometer (Perkin-Elmer spectrum BX, Perkin- Elmer, Woodbridge, Canada) equipped with a horizontal attenuated total reflectance (ATR) system (Perkin-Elmer spectrum BX, Perkin- Elmer, Woodbridge, Canada). All spectra were recorded in a range of 4000–400  $\text{cm}^{-1}$  at a resolution of 4  $\text{cm}^{-1}$  with 64 scans.

## 2.8 Mechanical Characterization

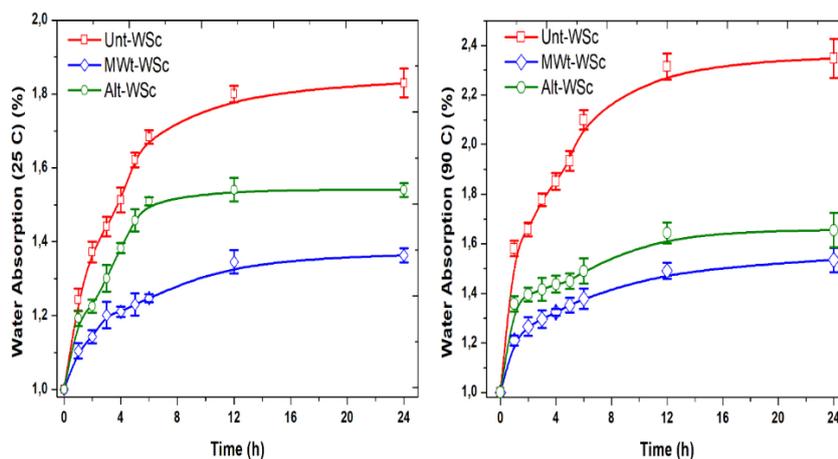
Mechanical properties of untreated and treated wheat straw reinforced composites were determined by tensile and bending tests. Mechanical tests were performed with an Instron Model 8501 (UK) (5 kN). The tensile test was carried out according to the ASTM D638 method at a crossing speed of 5 mm/min and a gauge length of 50 mm. The flexural test was carried out at 5 mm/min in accordance with ASTM D790 method. Three samples of each sample were tested for tensile and bending tests and the average results reported.

## 3. RESULTS and DISCUSSION

### 3.1 Water absorption results

Understanding and manipulating the polarity fraction of cellulosic materials can be crucial in designing materials with specific water absorption properties for diverse applications ranging from textiles and packaging to biomedical devices. It involves a balance between hydrophilic and hydrophobic components to achieve the desired performance characteristics. Higher polarity fraction generally leads to increased hydrophilicity (gaffar, 2016 and Gaffar, Mizi, 2017). Within the scope of the study, the water absorption test results of composite samples containing treated and untreated wheat straws are shown in Figure 1.

Hydrophilic groups, such as hydroxyl (-OH) groups in cellulose, can form hydrogen bonds with water molecules, making the material more capable of absorbing water. Additionally, wheat straw has a porous structure, and its fibers create a network that can absorb and retain water.



**Figure 1.** Water absorption results 25°C (left) and 90°C (right)

When the test results were analyzed, it was observed that the water absorption tendency exhibited high values in untreated wheat straw reinforced composites. In contrast, composites reinforced with alkaline and microwave-treated wheat straw displayed lower water

absorption values, consistent with similar studies (Li et, 2016 and Mittal, Shishir,2017). The water absorption mechanism initially penetrates the structure through capillary action, followed by the accumulation of water molecules that bond with the reinforcement. The

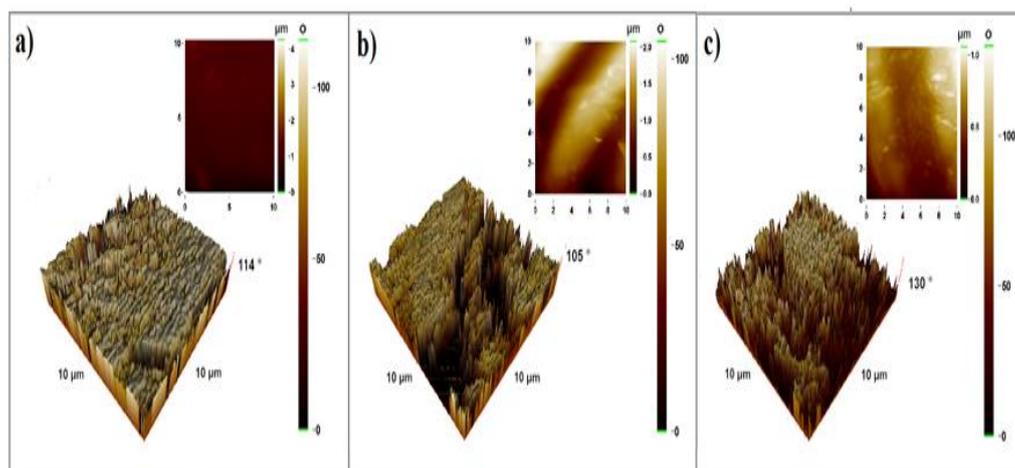
alkaline treatment method prevents the aforementioned uptake and accumulation by reducing free OH groups. It can be argued that the decrease in water absorption value is attributed to the fact that alkaline treatment provides better wetting with the epoxy matrix by altering the surface topography, thereby reducing micro voids and the possible capillary effect, and subsequently reducing water transmission within the structure.

### 3.2 AFM and SEM results

The morphological and free surface energy changes on wheat straws after the treatment process were examined by atomic force microscopy. AFM examinations of

untreated and treated interior and exterior wheat straws are shown in Figure 2 and 3.

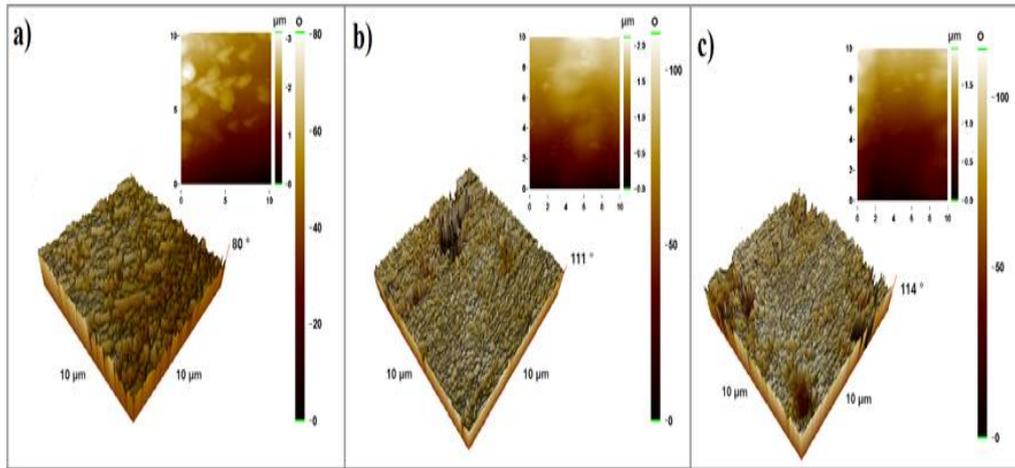
The interior and exterior surface roughness values of untreated wheat stalks were 0.176 and 0.149, respectively. While a significant roughness change was observed on the interior surface of the wheat stalk after alkali treatment, it was determined that the roughness change efficiency was low on the exterior surface ( $R_a=0.150 \mu\text{m}$ ,  $R_{\text{rms}}=0.190 \mu\text{m}$ ). This can be attributed to the difference in the chemical structure of the interior and exterior surfaces. Especially the excess of lipophilic chemical substances, cutin and wax on the exterior surface may have resisted alkaline treatment (Mohanty,2001).



**Figure 2.** Exterior surfaces phase and amplitude (front view) images of the untreated (a), alkali treated (b), microwave treated (c)

Microwave treatment shows its effect on surface roughness through molecular changes such as dehydration, crystallinity change or hydrolysis by means of interaction with polar groups (-OH). A

change of 65% and 182% was observed in the untreated interior and exterior surface roughness, respectively. It can be suggested that it creates more active sites in terms of wettability.



**Figure 3.** Interior surfaces phase and amplitude (front view) images of the untreated (a), alkali treated (b), microwave treated (c)

The existing literature has demonstrated that both alkaline and microwave treatment methods induce morphological changes on the surface of wheat straws, as well as alterations in the surface energy due to the molecular changes they elicit (Sgriccia,2007 and Li et al,2016). While alkaline treatment removes irregularities such as lignin, pectin, and wax from the wheat straw surface, it also causes a decrease in the free hydroxyl (OH) groups of cellulose in the primary wall of the stem (Li et al,2007 and Liu et al,2017 ). The roughness values of the interior and exterior surfaces after alkali treatment are presented in Table 3. As shown, there is a remarkable increase in the roughness values (Gadelmawla et al,2002). However, it would be

cautious to also consider the skewness and kurtosis values presented in the table when evaluating the surface morphology. The skewness and kurtosis values obtained in AFM contact mode measurements are also presented in Table 3. Roughness values provide a superficial assessment of the post-treatment surface morphology. In addition, the skewness/kurtosis values, the forms of the surface shapes that occur after reclamation, i.e. the formation of peaks/valleys or sharp/oval ends, determine both the wettability and the quality of the mechanical interlocking between the matrix and the surface.

**Table 3.** Surface roughness parameters of untreated and APPIJ treated wheat straw

Sample	Coefficient			
	Average ( $R_a$ )( $\mu\text{m}$ )	Root Mean Square ( $R_{rms}$ )( $\mu\text{m}$ )	Skewness ( $R_{sk}$ )	Kurtosis ( $R_{ku}$ )
UntIWS	0.176±0.03	0.191±0.02	-0.281±0.03	3.890±0.03
UntEWS	0.149±0.01	0.156±0.01	-0.173±0.02	3.418±0.03
5AIT-IWS	0.390±0.43	0.460±0.22	0.167±0.17	1.118±0.41
5AIT-EWS	0.150±0.21	0.190±0.24	0.155±0.19	1.614±0.33
300:20MwT-IWS	0.290±0.26	0.400±0.31	0.139±0.27	1.314±0.15
300:20MwT-EWS	0.420±0.34	0.490±0.25	0.118±0.36	1.959±0.52

Negative skewness values on the untreated wheat straw surface indicate crack-like formations, while kurtosis values greater than 3 indicate leptocurtic-like sharp ("pointy") surface formations. It can be concluded that the skewness value turned positive after both treatment processes and the crack-like formations decreased or were filled with various substances by plastering during the process. Kurtosis values were observed to be below 3 (Gadelmawla et al,2002. This indicates platykurtic surface formations, i.e. valley-like shape formations with reduced number of sharp edges.

AFM surface examinations suggest that the treatment process is beneficial for wettability and the resulting surface shapes are shaped in a way that helps the mechanical interlocking with the matrix.

SEM images to examine the surface roughness are shown in Figure 4 and 5. On the surface of the untreated wheat straw, the roughness ratio and the wax, pectin, etc. substances on the surface were clearly observed (Figure 4a, 5a).

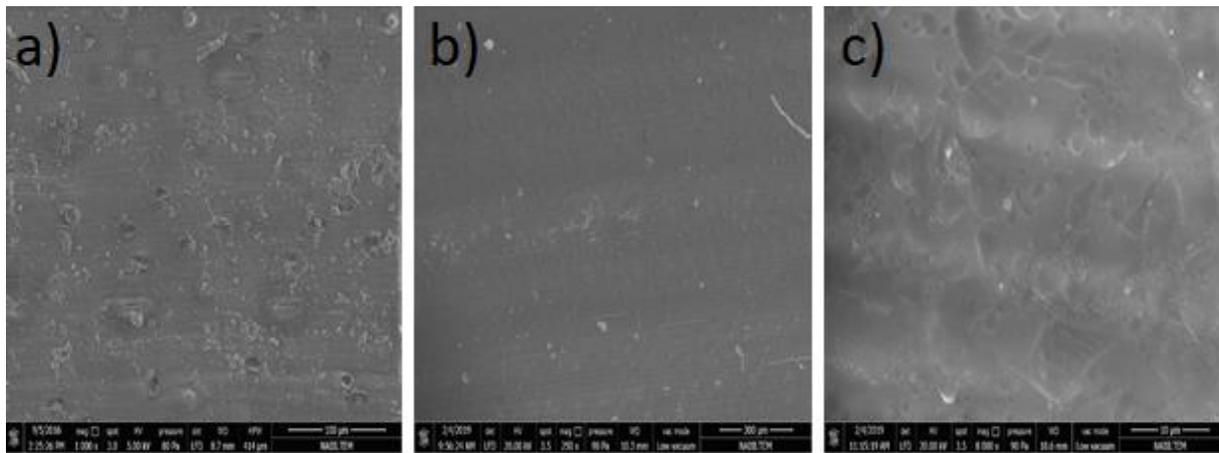


Figure 4. Exterior surface of untreated (a), alkali treated (b), microwave treated (c) wheat straws

Morphological differences between the interior and exterior surface are also evident. A clean structure was observed on the exterior surface after alkali treatment. On the interior surface, more pronounced surface contours appeared.

Microwave treatment, on the other hand, caused a rougher "shrunk" structure on the exterior surface compared to the alkaline one. On the interior surface, it caused a more distinct surface as in alkaline.

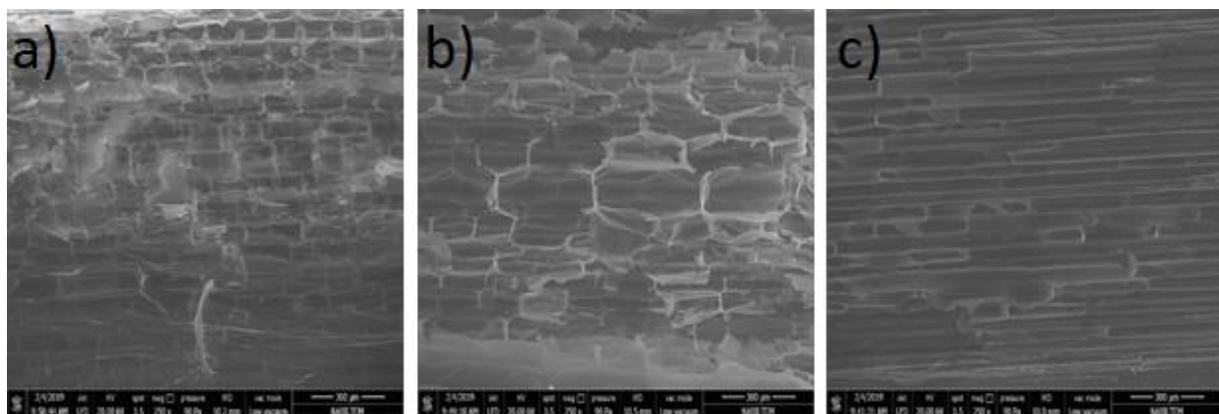
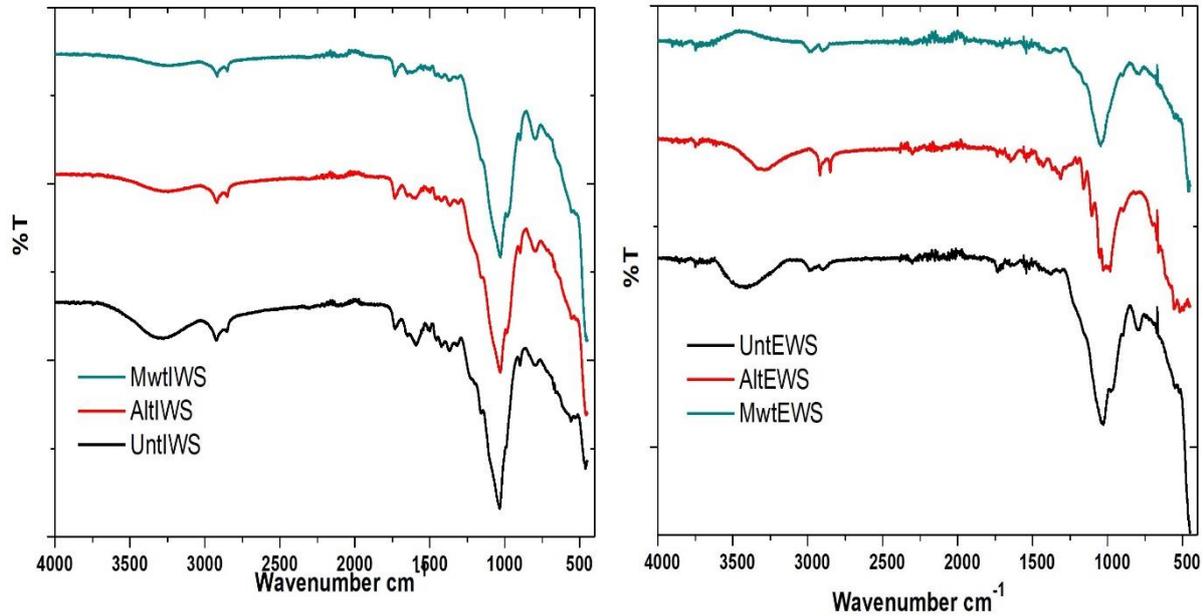


Figure 5. Interior surface of untreated (a), alkali treated(b), microwave treated (c) wheat straws

### 3.3 FTIR results

The results of the FTIR analysis to investigate the effect of the treatment on the molecular structure of the

interior and exterior surface of the wheat straw are shown in Figure 6.



**Figure 6.** FTIR results of interior (left) and exterior (right) surfaces of wheat straws

The stretching vibration absorption peak located around  $3400\text{ cm}^{-1}$  of the inner and outer surface corresponds to the aliphatic components of cellulose, lignin and wax of the -OH group present in the structure (Kabir et al, 2012). While the intensity of this peak decreases after alkali treatment on the interior surface, it can be stated that the effectiveness is more limited on the exterior surface. On the other hand, it can be claimed from the changes of the mentioned peak of the interior and exterior surfaces that microwave treatment plays a more effective role in the removal of -OH groups.

The peak located at  $2920\text{ cm}^{-1}$  is a characteristic peak originating from the stretching vibration CH group in hemicellulose and cellulose (Li et al ,2007; Talebnia et al, 2010 and Li et al, 2016). The C=C peak located at  $1507\text{ cm}^{-1}$  is the stretching of the aromatic rings of lignin. Partial fragmentation of lignin after reclamation caused a change in this peak. Peaks located around  $1000\text{ -}1300\text{ cm}^{-1}$  are due to Si-O and O-Si-O stretching.

Especially on the exterior surface, the intensity of the peaks in this region decreased or disappeared after treatment, which can be attributed to the degradation of the silicon layer.

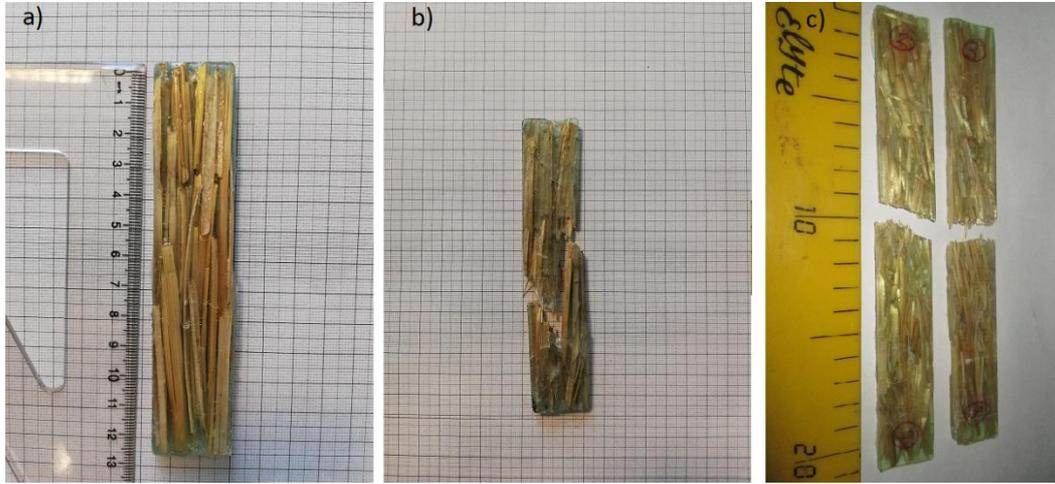
At  $870\text{ cm}^{-1}$ , the C-O peak typical of cellulose has decreased in intensity or disappeared, especially on the outer surface. In particular, the alkali treatment caused a change in the molecular bonds of the cellulose on the outer surface.

As a general evaluation, it can be suggested that both alkali and microwave partially/completely removed some components from the interior and exterior surfaces, and the effects on the interior and exterior surfaces varied depending on the difference in structural components. Surface roughness as well as possible molecular changes affect the wettability and bonding abilities between the matrix and the reinforcement, which in turn determine the overall physical/mechanical properties of the composite.

### 3.4 Mechanical test results

Tensile and flexure tests were carried out to investigate the effect of the treatment process on the mechanical

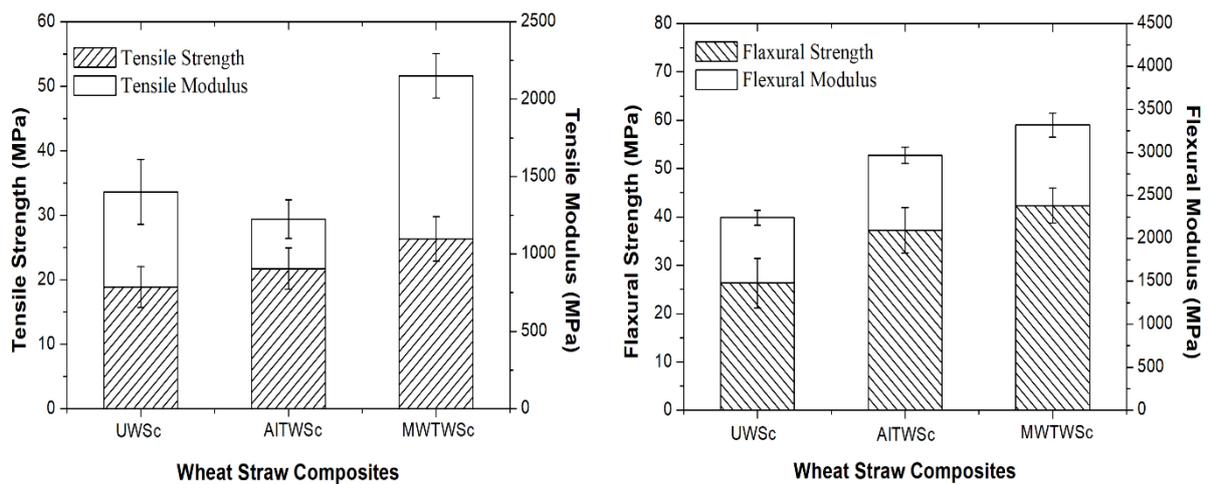
properties of the epoxy composites produced and Figure 7 shows the pre and post-test images of the samples. The results of tensile and flexural tests are shown as bar graphs in Figure 8.



**Figure 7.** Wheat straw reinforced composites a) tensile test sample, b) broken tensile test sample, c) flexural test samples

A tensile strength value of approximately  $19 \pm 3.15$  MPa was observed in composites containing untreated wheat straw. Alkali treated specimens showed an improvement of 15% and microwave treated specimens showed an improvement of 37%. This was attributed to surface treatment leading to better wetting and increased mechanical interlocking between the

reinforcement and the matrix, which is in line with similar studies (bolcu et al,2022). Considering the tensile modulus, it can be argued that alkaline treatment causes embrittlement on the straw and although it shows an increase in strength, its ability to absorb energy decreases.



**Figure 8.** Tensile test results of wheat straw composites (left), Flexural test results (right)

The flexural strength and modulus of the composites containing untreated wheat straw were determined as 26 and 2240 MPa, respectively. While the flexural strength of the composites with alkali treated wheat straw increased by 42%, the increase in flexural strength in the composites with microwave treated wheat straw was 62%.

The increase in flexural modulus in alkali treated samples was measured as 32%. This increase was 48% in microwave treated samples. (Bolcu et al,2022 and mittal et al ,2017) When the flexural test results are compared with similar studies, the differences can be attributed to the physical form of the wheat straw used in the composites or the differences in parameters such as processing time and chemical ratio in the treatment process.

#### 4. Conclusions

Untreated and alkali and microwave treated wheat straws were used in the production of composites using epoxy matrix.

When the effect of alkali treatment on the surface was examined, it was determined that the effectiveness was less on the outer surface. It caused a roughness change of approximately 162% on the inner surface.

Microwave treatment caused both roughness change and the resulting surface forms were found to be more appropriate in terms of wettability and mechanical interlocking.

At the molecular level, it was revealed with the help of FTIR that both treatment methods removed the free -OH groups in the wheat straw structure. It can be argued that microwave only helps more wettability with its effect on surface energy change and molecular bonding.

Mechanical test results revealed that both treatment processes were effective. Considering the strength values, microwave-treated wheat straw reinforced

composites performed better than alkaline-treated composites.

It can be argued that the effectiveness of the microwave treatment process contributes more to the process when it is desired to increase the wettability and mechanical bonding performance of the reinforcement without weight loss and without deteriorating the structural strength of the reinforcement during the treatment process.

#### Conflict of Interest

Author declare that there are no conflicts of interest.

#### References

- Alemdar, Ayse, and Mohini Sain. 2008a. "Bio composites from Wheat Straw Nanofibers: Morphology, Thermal and Mechanical Properties." *Composites Science and Technology* 68(2):557–65. doi: 10.1016/j.compscitech.2007.05.044.
- Bolcu, Dumitru, Marius Marinel Stănescu, and Cosmin Mihai Mirițoiu. 2022. "Some Mechanical Properties of Composite Materials with Chopped Wheat Straw Reinforcer and Hybrid Matrix." *Polymers* 14(15). doi: 10.3390/polym14153175.
- Gadelmawla, E. S., M. M. Koura, T. M. A. Maksoud, I. M. Elewa, and H. H. Soliman. n.d. "Roughness Parameters" *Journal of Materials Processing Technology* 123 (2002) 133±145
- Ghaffar, Seyed Hamidreza. 2016. *Aggregated Understanding of Characteristics of Wheat Straw Node and Internode with Their Interfacial Bonding Mechanisms.*
- Ghaffar, Seyed Hamidreza, and Mizi Fan. 2017a. "An Aggregated Understanding of Physicochemical Properties and Surface Functionalities of Wheat Straw Node and Internode." *Industrial Crops and*

- Products 95:207–15. doi: 10.1016/j.indcrop.2016.10.045.
- Kabir, M. M., H. Wang, K. T. Lau, and F. Cardona. 2012a. “Chemical Treatments on Plant-Based Natural Fibre Reinforced Polymer Composites: An Overview.” *Composites Part B: Engineering* 43(7):2883–92. doi: 10.1016/j.compositesb.2012.04.053.
- Li, Hongqiang, Yongshui Qu, Yongqing Yang, Senlin Chang, and Jian Xu. 2016. “Microwave Irradiation - A Green and Efficient Way to Pretreat Biomass.” *Bioresource Technology* 199:34–41.
- Li, Xue, Lope G. Tabil, and Satyanarayan Panigrahi. 2007. “Chemical Treatments of Natural Fiber for Use in Natural Fiber-Reinforced Composites: A Review.” *Journal of Polymers and the Environment* 15(1):25–33.
- Liu, Qi, Yun Lu, Mario Aguedo, Nicolas Jacquet, Canbin Ouyang, Wenqing He, Changrong Yan, Wenbo Bai, Rui Guo, Dorothée Goffin, Jiqing Song, and Aurore Richel. 2017. “Isolation of High-Purity Cellulose Nanofibers from Wheat Straw through the Combined Environmentally Friendly Methods of Steam Explosion, Microwave-Assisted Hydrolysis, and Microfluidization.” *ACS Sustainable Chemistry and Engineering* 5(7):6183–91. doi: 10.1021/acssuschemeng.7b01108.
- Mengelloglu, Fatih, and Kadir Karakus. 2008. “Thermal Degradation, Mechanical Properties and Morphology of Wheat Straw Flour Filled Recycled Thermoplastic Composites.” *Sensors* 8:500–519.
- Mittal, Varun, and Shishir Sinha. 2017. “Study the Effect of Fiber Loading and Alkali Treatment on the Mechanical and Water Absorption Properties of Wheat Straw Fiber-Reinforced Epoxy Composites.” *Science and Engineering of Composite Materials* 24(5):731–38. doi: 10.1515/secm-2015-0441.
- Mohanty, A. K., M. Misra, and L. T. Drzal. 2001. “Surface Modifications of Natural Fibers and Performance of the Resulting Biocomposites: An Overview.” *Composite Interfaces* 8(5):313–43. doi: 10.1163/156855401753255422.
- Satpathy, Sangram Kishor, Lope G. Tabil, Venkatesh Meda, Satya Narayana Naik, and Rajendra Prasad. 2014. “Torrefaction of Wheat and Barley Straw after Microwave Heating.” *Fuel* 124:269–78. doi: 10.1016/j.fuel.2014.01.102.
- Sgriccia, Nikki, and M. C. Hawley. 2007. “Thermal, Morphological, and Electrical Characterization of Microwave Processed Natural Fiber Composites.” *Composites Science and Technology* 67(9):1986–91. doi: 10.1016/j.compscitech.2006.07.031.
- Talebniya, Farid, Dimitar Karakashev, and Irini Angelidaki. 2010. “Production of Bioethanol from Wheat Straw: An Overview on Pretreatment, Hydrolysis and Fermentation.” *Bioresource Technology* 101(13):4744–53. doi: 10.1016/j.biortech.2009.11.080.
- Väisänen, Taneli, Antti Haapala, Reijo Lappalainen, and Laura Tomppo. 2016. “Utilization of Agricultural and Forest Industry Waste and Residues in Natural Fiber-Polymer Composites: A Review.” *Waste Management* 54:62–73.
- Zou, Yi, Shah Huda, and Yiqi Yang. 2010. “Lightweight Composites from Long Wheat Straw and Polypropylene Web.” *Bioresource Technology* 101(6):2026–33. doi: 10.1016/j.biortech.2009.10.042.