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ORIGINAL RESEARCH ARTICLE

Enhancing physicochemical properties of rice straw for reinforcement in composite using combined Ultrasound-Plasma treatment

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ABSTRACT

Rice straw waste holds considerable potential as a bio-reinforcement material in polymer composites; however, effective utilization requires surface modification to enhance fiber–matrix interfacial adhesion. This study investigates environmentally friendly surface modification methods, including sodium carbonate treatment with ultrasound (SNCO5%), plasma treatment (PT), and their combination (SNCO5%-PT). Comprehensive characterizations were performed, including surface morphology, O/C ratio, silica content, functional group analysis, and X-ray diffraction. The SNCO5%-PT treatment resulted in the highest silica enrichment and O/C ratio, with increases of 4.5 and 4.75 times, respectively, compared to the untreated sample (NT). FTIR analysis revealed intensified absorption peaks in SNCO5%-PT, particularly at wavenumbers associated with hydroxyl groups and absorbed water in cellulose. XRD analysis further confirmed that SNCO5%-PT achieved the largest crystallite size and highest crystallinity index among all treatments. These findings suggest that the combined treatment enhances the physicochemical properties of rice straw. Future work should focus on evaluating the mechanical performance of treated fibers in polyethylene composite systems, as well as assessing interfacial adhesion and durability under application-relevant conditions.

Keywords: composite; fiber; plasma treatment; rice straw; ultrasound treatment

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1. Introduction

Indonesia was ranked as the fourth-largest producer of rice worldwide. According to BPS- statistics Indonesia data, the paddy harvest area in 2023 will be approximately 10.20 million, with rice production of about 30.90 million tons. Rice production produces agricultural waste such as straw, husk, ash, bran, and broken rice[1]. Rice straw is the biggest waste from rice production, with an amount of around 50 million tons per year. Presently, approximately 20% of rice straw is put to use in practical ways, like making paper, fertilizers, animal feed, and biofuels[2]. Unused rice straw is either burned or disposed of. This is certainly not a good approach because it could lead to other issues. Emissions of greenhouse gases can arise from burning rice straw[3]. Improper management of rice straw landfilling contributes to in-creased greenhouse gas emissions[4]. There have been several initiatives to boost the use of rice straw in various industrial sectors. Prior research has examined possible applications of rice straw for the production of bioenergy. The other potential usage of rice straw was as bio-fiber in composite. The use of rice straw as fiber in composites is interesting to study and explore. Embracing these uses can contribute to sustainable rice straw management and the development of circular economies[5].

One important consideration in the fabrication of composite is the interfacial adhesion between matrix and fibers. However, natural fibers contain wax, water, and free hydroxyl groups, which can inhibit the bonds formed between the fiber and polymer, thereby reducing the performance of composite products. The adhesion between natural fibers and the matrix can be improved using chemical, physical, or biological treatments. Chemical treatments usually used for natural fiber are alkaline treatment, silane treatment, and acid hydrolysis[6]. Physical treatment is carried out using plasma, ultrasonic, and steam explosion. Meanwhile, biological treatment is usually carried out enzymatically or through fermentation. Among these treatments, chemical treatment is the most frequently used method. The choice of chemicals used must be considered because some chemicals used to treat natural fiber are typically toxic, more expensive, and pose greater environmental risks[7]. Biological treatments offer a more environmentally friendly approach compared to traditional chemical methods, as they often involve the use of harmless enzymes or microorganisms. However, it depends on field conditions and the time is relatively long, so it is less stable, difficult to control, and is an obstacle in industries that require high productivity. This encourages the need to implement strategies in the form of methods that are simpler, faster, environmentally friendly, and sustainable. This encourages the need to implement strategies in the form of methods that are simpler, faster, environmentally friendly, and sustainable.

Environmentally conscious strategies for modifying the surface of natural fibers have increasingly emphasized the use of physical methods, such as ultrasonic treatment and plasma activation, particularly when coupled with safer alkaline agents like sodium carbonate. Ultrasonic treatment operates through the generation of high-frequency acoustic waves, which induce cavitation phenomena within a liquid medium. The collapse of cavitation bubbles in proximity to rice straw fibers induces localized mechanical disruption on the fiber surface. This process not only increases surface roughness thereby enhancing interfacial surface are but also facilitates the removal of surface waxes and impurities, exposing more chemically active sites conducive to improved fiber–matrix adhesion[8]. Meanwhile, plasma treatment, especially under low-pressure cold plasma conditions, induces micro-etching and topographical alterations on the fiber surface, further promoting mechanical interlocking with polymer matrices. When these two techniques are integrated, the resulting hybrid approach offers notable benefits in terms of energy efficiency and environmental sustainability. Compared to conventional chemical modification processes, this combination significantly reduces the reliance on hazardous reagents and minimizes the generation of toxic byproducts. Nevertheless, the optimization of this dual-treatment method requires careful regulation of gas inputs and process parameters to ensure consistency, efficacy, and long-term viability of the modified fibers for composite applications[9].

Several studies have been carried out on polymer/natural fiber composites using a cold plasma system. Flax fiber composites with high-density polyethylene (HDPE) and unsaturated polyester. In this study, treating the hemp fiber with cold plasma enhanced the adhesion strength of the unsaturated polyester/flax fiber composite interface[10]. Composites can made from recycled polyethylene (PE) with Kapok fiber. The results of this research show that kapok fiber that has been treated with plasma had better adhesion with the recycled PE as a matrix[11]. Wheat straw composites can made by urea formaldehyde (UF) resin[12]. Plasma treatment of wheat straw will increase the bonding strength of composite products. The above shows that cold plasma technology can be a viable, non-polluting alternative method to improve fiber/matrix interfacial adhesion in polymer composites. However, according to the literature survey, there are relatively few publications on the surface modification of rice straw using ultrasound with a safer salt or plasma treatment, especially for composite applications[13].

In recent years, agricultural biomass residues such as rice straw have been extensively investigated as natural reinforcements in polymer-based composites due to their abundance, renewability, and potential contribution to environmental sustainability[14]. However, their application has been constrained by inherent physicochemical limitations, including high silica content, inert surface morphology, and low crystallinity, which collectively hinder effective interfacial adhesion between the fibers and polymer matrices[15]. To address these challenges, various surface modification techniques have been introduced, particularly chemical treatments such as alkalization, acetylation, and the application of coupling agents[16]. In addition, physical approaches including plasma treatment and irradiation have been explored as more environmentally friendly alternatives, though their effectiveness has been shown to vary depending on fiber type and processing conditions. The potential of ultrasonic treatment for natural fibers has been explored in several studies, demonstrating improvements in dispersion, reduction in fiber diameter, and modification of internal structures through cavitation-induced effects[17]. Plasma treatment, on the other hand, has been reported to enhance surface cleanliness, increase surface energy, and introduce polar functional groups that promote better adhesion with polymer matrices[18]. Despite these advancements, the simultaneous application of both ultrasonic and plasma treatments to rice straw fibers remains scarcely investigated. This study proposes an innovative dual-treatment strategy combining ultrasonic and plasma techniques to engineer the morphological and chemical properties of rice straw fibers. This synergistic approach is designed to optimize interfacial compatibility with polymer matrices while avoiding the use of hazardous chemicals. A systematic investigation has been conducted to assess the impact of the combined treatment on silica content, crystallinity, surface functionalization, and the mechanical performance of the resulting composites. Through this method, a significant contribution is made to the advancement of sustainable bio-composite development, aligning with circular economy principles and emphasizing environmentally responsible processing strategies.

In the development of natural fiber-reinforced polymer composites, considerable attention has been given to improving interfacial bonding through various surface modification techniques. However, limited emphasis has been placed on the specific role of surface silica, particularly in silica-rich fibers such as rice straw. The inherent presence of silica in rice straw poses a complex challenge; while it may impede interfacial bonding due to its chemical inertness and rigidity, some reports suggest that under certain conditions, silica could enhance fiber–matrix compatibility by promoting surface wettability and mechanical anchoring[18]. These divergent perspectives underscore the absence of a coherent understanding of silica's function at the fiber–matrix interface. Moreover, most studies have evaluated surface modification efficacy without directly isolating the effects of silica enrichment or depletion. The influence of combined treatments such as alkaline extraction and plasma activation on surface silica morphology and its downstream impact on adhesion mechanisms has also remained largely unexplored. This indicates a critical gap in the literature regarding the interfacial dynamics introduced by silica modification, which is essential for tailoring natural fiber composites toward improved mechanical performance and thermal stability in sustainable material applications.

The aim of this study was to select the most effective methods for modifying the surface of rice straws. Plasma treatment, ultrasound treatment, and combined plasma-ultrasound treatment on rice straw were compared with untreated rice straw through morphology, O/C ratio analysis, and silica content on the surface with scanning electron microscope energy dispersive X-ray (SEM-EDX); functional group analysis with a Fourier transform infrared (FTIR) spectrometer; crystallinity index and crystallite using X-ray diffraction (XRD) analysis; and thermal characterization using a thermal gravimetric analyzer (TGA). Treated rice straws with the best performance and untreated rice straws are made into composites with PE and UP, and their mechanical properties are measured through the tensile strength test. It is expected that the environmentally friendly treatment applied to rice straw will significantly increase the value of the resulting composite.

2. Materials and methods

2.1. Material

The materials used in this study were rice straw, sodium carbonate (Na_2CO_3) Merck 1.06392.1000, distilled water, high-density polyethylene (HDPE), unsaturated polyester (UP). The rice straw used was a stem part from Tasikmalaya, Indonesia. The HDPE used was HD5301AA, with a Melt Flow Rate (190ºC/2.16 kg) of 0.1 g/10 min from PT. Lotte Chemical Titan Nusantara. We used YUKALAC as the UP, along with a type of catalyst known as Mepoksi.

2.2. Methods

Rice straw preparation

Dried rice straw was cut into pieces and then chopped using a crusher. The rice straw is then sieved using a 250 µm filter. Before moving on to the next step, we dry rice straw with a size of less than 250 microns in an 80°C oven for 24 hours to eliminate the water content.

Ultrasound Treatment

We added 10 grams of rice straw powder to a 250-ml Erlenmeyer flask, followed by adding 100 ml of Na_2CO_3 solution with concentrations of 5%. The mixture was then placed in an Elmasonic ultrasonic bath for 10 minutes at a temperature of 45 °C, using the digest mode. The rice straw powder was subsequently washed with distilled water until the wastewater from the washing process reached a neutral pH.

Plasma treatment

We placed a sample of rice straw powder in a low-pressure plasma chamber and activated the vacuum pump until the pressure -72 cmHg. We then initiated the plasma at a power 500 Watt and a frequency of 240-400 KHz for 30 minutes. After 30 minutes, we turned off the plasma, deactivated the vacuum, and removed the sample from the plasma chamber.

Preparation of Polyethylene/Rice Straw Composites

Dried polyethylene (PE) and dry rice straw powder were mixed in a weight ratio of 1:10. A twin-screw extruder processed the mixture at a screw speed of 60 rpm, with a temperature range from 40 °C to 190 °C across zones 1 to 8. The extrudate was then cooled using a pelletizer and formed into pellets. These pellets were subsequently shaped into specimens using a Collin P 300 P hot press at 185 °C, a pressure of 2 bar, and a duration of 15 minutes.

Preparation of UP/Rice Straw Composites

We combined dried rice straw powder and UP resin in a weight ratio of 1:10. The mixture was agitated at 300 rpm for one minute, after which we added a catalyst at a concentration of 1 wt% based on the total weight of the UP resin and rice straw powder mixture. The resulting mixture was then poured into a silicone mold that had been coated with wax. Drying was conducted at room temperature for 24 hours.

Morphology, O/C ratio analysis, and silica on the surface

We conducted morphology, O/C ratio analysis, and silica on the surface of rice straw powder using the Scanning Electron Microscope-Energy Dispersive X-ray (SEM-EDS) Jeol JSM-6519LA, following ASTM E 1508-12. Using carbon tape, we attached the rice straw powder to the holder until it filled its surface, approximately 1 cm by 1 cm, and then placed it in a vacuum evaporator to coat it with gold. The sample coated with gold was inserted into the SEM and observed at 500x magnification with a voltage of 20 kV.

Functional group analysis

We conducted a functional group analysis of rice straw powder at various treatments using a Fourier transform infrared (FTIR) spectrometer. We analyzed rice straw powder using the Attenuated Total Reflectance (ATR) method with a Hyperion 2000 FTIR spectrometer, covering a wavenumber range of 4000 cm^{-1} to 600 cm^{-1}. IR spectra are also used to calculate the relative crystallinity index ($CI_{relative}$). We calculate the relative crystallinity index ($CI_{relative}$) by comparing the peak area at the absorbance of approximately 1423 cm^{-1} for crystalline cellulose and 897 cm^{-1} for amorphous cellulose[19].

$$CI_{relative} = A_{1423}/ A_{897} \tag{1}$$

X-ray diffraction analysis

The PANalytical AERIS XRD with Cu-K± radiation at 15 mA and 40 kV was used to examine rice straw's phase composition with and without treatment. The dried sample was placed into a glass sample holder and scanned from 5° to 90° with a 10°/minute scan speed. The crystallinity index (CI) is calculated using the Segal equations as follows[20]:

$$CI = (I_{cr} – I_{am})/I_{cr} \times 100 \tag{2}$$

Where I_{cr} is the maximum diffraction intensity located at approximately $2\theta = 22.5°$ and corresponds to the crystalline region; I_{am} is the minimum diffraction intensity located at approximately $2\theta = 18°$ and corresponds to the amorphous region. The crystallite size is calculated using the Scherrer equation as follows: The crystallite size is calculated using the Scherrer equation as follows[21]:

$$D = (K \lambda) / (\beta \cos \theta) \tag{3}$$

This equation has four numbers: λ is the X-ray wavelength, β is the full width at the half maximum of the peak in radians, and θ is the angle at which the peak is diffracted. The Scherrer constant, K, has a value of 0.94 for a spherical crystal with cubic symmetry.

3. Results and discussion

3.1. Rice straw powder characterization

3.1.1. Morphology, O/C ratio analysis, and Silica on surface

SEM images of the surface of rice straw powder in various treatments are presented in Figure 1. Before treatment (**Figure 1(a)**), SEM images showed the smooth and uniform surface of untreated rice straw. Untreated rice straw is often coated with wax, moisture, and free hydroxyl groups that can inhibit effective bonding with polymer matrices. This weak interfacial adhesion can significantly diminish the mechanical performance of composite materials. After plasma treatment (**Figure 1(b)**), SEM images showed slight etching and roughening of the fiber surface, although this change did not significantly appear that the condition of the plasma used is less than optimal. After ultrasound treatment using sodium carbonate at a concentration of 5% (**Figure 1(c)**), SEM images showed increased fiber surface roughness post-treatment. The cavitation effect helps in removing waxy layers and other contaminants. After combined ultrasound-plasma treatment (**Figure 1(d)**), SEM images experienced the most significant changes in the surface of the rice straw. Previous study from Karim et.al, 2022 also reported that sodium carbonate treatment for 6 hours to Bamboo short fiber can cause the fiber surface to become clean and fiber roughness to increase. This is due to the removal of hemicellulose and lignin due to the alkali used[17]. The surface of rice straw becomes rougher and more porous than untreated rice straw. Surface roughness provides spots to anchored polymer and produces better interlocking[22].

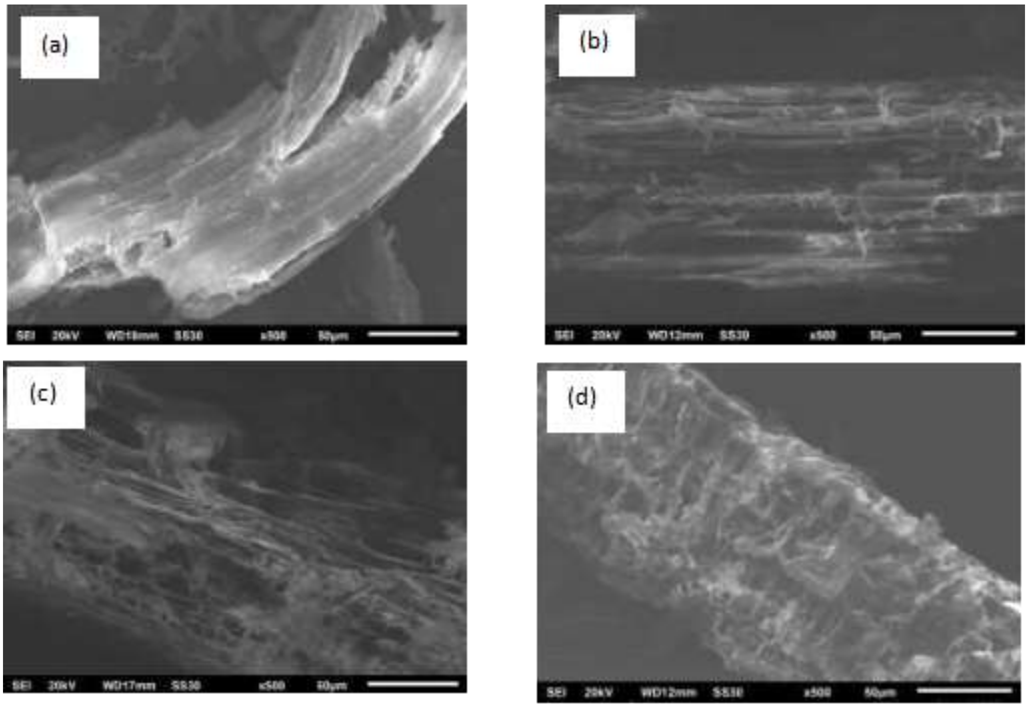

Figure 1. SEM images of rice straw powder: (a) NT, (b) PT, (c) SNCO5%, and (d) SNCO5%-PT.

The increase of Silica content on the outer surface of rice straw after plasma, sonication, and combined ultrasound-plasma treatments is due to the redistribution of silica from the inner to the outer layers of the straw. The treatment methods can disrupt the structure of the straw, leading to the release and migration of silica to the outer surface. **Table 1** shows that the SNCO5%-PT sample has the largest silica content on the outer surface among the others. The silica content in the SNCO5%-PT sample increased by 4.5 times compared to NT. The increase in silica content due to plasma treatment is following previous studies. Chen et al., 2019 reported that wheat straw outer surface treated with water vapor plasma increased Silica content by 8.3 times from 0.7 % to 5.8%. Thulluri et al., 2021 reported that deep eutectic-tetrahydrofuran treated rice straw could elevate silica content from 1.61% to 5.76%[23].

Table 1. Si content and O/C ratio on the outer surface of rice straw.

No.	Sample	% mass			O/C
		C	O	Si	
1	NT	89.00	3.65	2.11	0.04
2	PT	87.45	4.96	3.84	0.06
3	SNCO5%	88.00	5.90	4.78	0.07
4	SNCO5%-PT	73.42	13.69	11.64	0.19

Based on **Table 1**, the Oxygen to Carbon (O/C) ratio of rice straw powder in various treatments is presented in Table 1. It can be seen that the O/C ratio was affected by plasma treatment, leading to an increase in Oxygen and a decrease in Carbon. The SNCO5%-PT sample has the biggest O/C ratio among the others. The O/C ratio in the SNCO5%-PT sample increased by 4.75 times compared to NT, from 0.04 to 0.19. Several research groups showed a similar trend. Atmospheric pressure plasma treatment increased the O/C ratio in cotton fiber surfaces from 0.6 to 0.9. Flax fiber treated with plasma using argon and air increased the O/C ratio by 0.11 (from 0.36 to 0.47) and 0.19 (from 0.36 to 0.55), respectively. Helium plasma treatment of Ramie fabrics increased the O/C ratio from 0.58 to 0.64. Sisal fiber treated cold plasma with alkali has a greater O/C than treated with alkali, from 0.21 to 0.28. The O/C ratio can be affected by the introduction of oxygen functional groups on the surface of materials. This can impact the surface and mechanical properties of the material, as well as its adhesion to other substances. The increase in the O/C ratio after plasma treatment is indicative of the introduction of oxygen-based functional groups on the surface of the material, which can have various effects on its properties[24].

3.1.2. Functional group analysis

The FTIR spectra of rice straw with various treatments are shown in **Figure 2**.

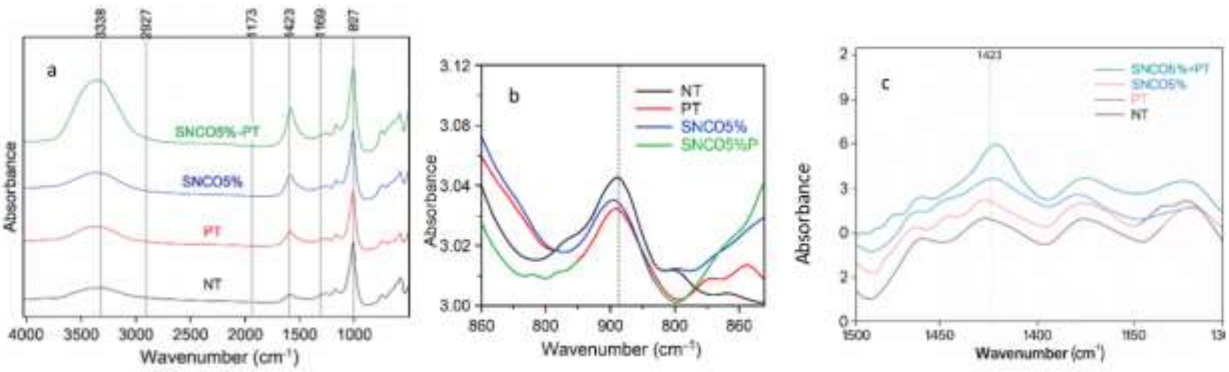

Figure 2. FTIR spectra of Rice straw with various treatments (a) IR spectra for functional group determination, IR spectra for analysis degree of relative CI at amorphous cellulose (b) and crystalline cellulose (c).

Based on **Figure 2**, FTIR spectra of NT, PT, SNCO5%, and SNCO5%-PT showed similar peaks, but SNCO5%-PT had more intensity peaks in wavenumber around 3384 cm-1, around 1634 cm-1, and several others. A peak at 3384 cm 1corresponds to OH stretching from cellulose of rice straw. A peak at 1634 cm-1 is probably associated with absorbed water in crystalline cellulose. 2927 cm-1 is C-H stretching vibration from CH in cellulose and hemicellulose components. 1723 cm-1 is carbonyl (C=O) stretching of hemicellulose and lignin. The peak at the wavenumber 1423 cm-1 is the CH_2 scissoring vibration, a crystalline region of crystalline. The peak at 1160 cm-1 indicates the C-O-C vibration of ether in cellulose. The peak at wavenumber 1057 cm-1 is the C-O stretching. The peak at the wavenumber of 897 cm-1 is a monosaccharide glycosidic bond; an amorphous region of cellulose[25]. The relative crystallinity index can be seen by comparing the peak area at absorbance of around 1423 cm-1 for crystalline cellulose and 897 cm-1 for amorphous cellulose. The findings of this investigation indicate that the NT, PT, SNCO5%, and SNCO5%-PT samples had relative crystallinity index values of 0.53, 0.54, 1.01, and 2.77, respectively. SNCO5%-PT had the highest relative crystallinity index value of all the samples. The relative crystallinity index from FTIR data is constrained and intended solely for qualitative comparison. XRD is the method used to assess the crystallinity index precisely[26].

3.1.3. X-ray diffraction analysis

The X-ray diffraction profile of untreated and treated rice straw can be seen in **Figure 3**. The diffraction peaks of samples appeared in a lattice plane of 200 at 2θ = 21.9°, a lattice plane of 110 at 2θ = 15.7°, and a lattice plane 004 at 34.6°. This XRD pattern is accordance with previous studies. The peaks correspond to cellulose-I structure. Compared to untreated rice straw, the intensity of the lattice plane of 200 in treated rice straw increased due to a well-organized cellulose chain after treatment. The significant increase in peaks in the lattice plane could also be due to exfoliation of previously hidden active sites[27].

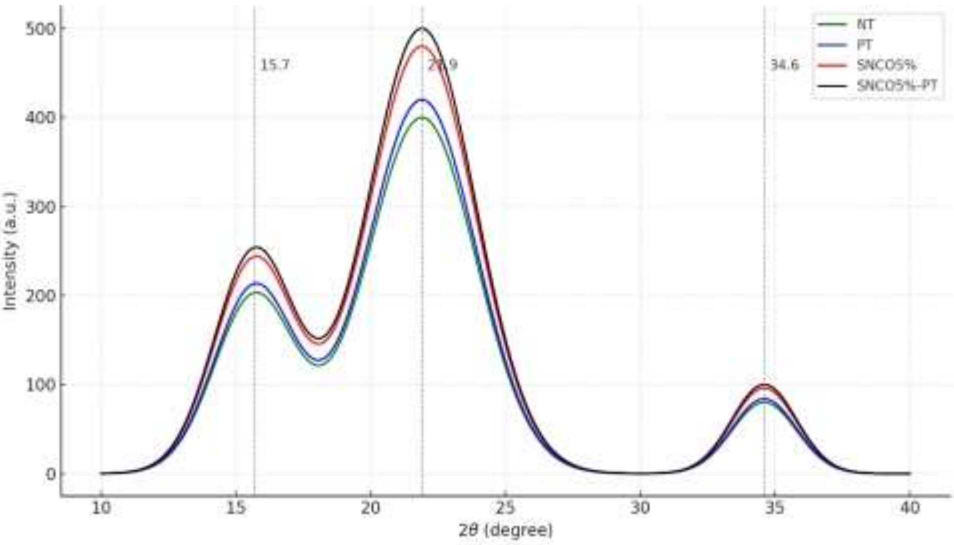

Figure 3. X-ray Diffraction pattern of rice straw with various treatments.

Crystalline index (CI) of untreated and treated rice straw was calculated with Segal equation and can be seen in **Table 2**.

Table 2. The crystalline index and crystallite of rice straw with various treatments.

Sample	CI (%)	Crystallite (nm)
NT	44.89	1.43
PT	45.29	2.34
SNCO5%	47.30	2.66
SNCO5%-PT	47.63	3.34

Based on **Table 2**, The SNCO5%-PT sample had the highest CI value, namely 47.63%, an increase only of 6.10% compared to NT. This increase in CI value is as in previous researches. Panigrahi et.al, 2022 reported that yard waste treated with Laccase enzyme increased the CI value by 25.7%, from 27.15% to 34.13%. Dos Santos, 2022 reported that Sisal fiber treated with sodium bicarbonate can increase the CI value by 4.58%, from 60.75% to 63.53%. Sisal fiber treated with sodium carbonate can increase the CI value up to 15.52%, from 60.75% to 70.18%. Boonsommbuti et.al., 2018 reported that alkaline, acid, and ionic liquid by microwave irradiation pretreated rice straw could increase crystallinity, with CI value prepositioned from 13.6% to 35.42%[28]. Coconut waste treated with 5% NaOH increased the CI value by 61.35% from 20% to 32.27%[29]. Rice straw treated by boiled sulfuric acid solution had a CI value between 55.4% and 61.2%. This CI value increased significantly compared to untreated rice straw which was only 23.6%[24]. However, contradictory results were also found by previous studies. Panigrahi et.al., 2022 also reported that yard waste treated with thermo-chemo-sonic experienced a decrease in the CI value of 2.80%, from 45.57% to 44.29%[30]. Delignification of Parthenium hysterophorus biomasa with alkaline using mechanical agitation and ultrasound. Both preteated biomass with mechanical agitation and ultrasound experienced a reduction in CI of 18.23% and 19.98%, respectively. Jisha et.al., 2022 also reported similar results. Ionothermal treatment of rice straw slightly reduces the CI value compared to untreated rice straw, from 46.34% to 46.22%. Decrease in the CI value is most likely because cellulose experiences depolymerization, forming short chains[31].

Table 2 also shows the crystallite size of untreated and treated rice straw. Untreated rice straw (NT) has the lowest crystallite value compared to other samples. Treatment of rice straw can increase crystallite size and the highest crystallite size value in ultrasonic treatment with a combined sodium carbonate and plasma (SNCO5%-PT). The increase on crystallite size may be caused incorporation of cellulose as consequent of the removal lignin and hemicellulose[32]. These results are in accordance with previous researches. Ningthoujam et.al., 2023 reported that sodium hydroxide treated rice straw experienced an increase in crystallite size by 13.28%, from 2.71 nm to 3.07 nm[33]. Panigrahi et.al., 2022 reported lignocellulosic waste treated by thermos-chemo-sonic as well as Laccase enzyme increased crystallite size from 0.39 nm to 0.47 nm and 0.36 nm to 0.43 nm, respectively. Dry torrefaction on corn cobs had a crystallite size of 6.51 nm – 8.12 nm. This value range is greater than untreated corn cobs, namely: 5.77 nm. Crystallite size of rice straw treated by boiled sulfuric acid solution had a value between 18.66 nm to 22.39 nm. This crystallite is larger than unthread rice straw, namely: 18.46 nm[34]. However, a study from Jisha et. Al., 2022 displays contradictory results. Ionothermal treatment of rice straw actually reduces the crystallite size from 3.20 nm to the range 0.56 nm – 2.16 nm.

3.2. Mechanical analysis

The mechanical characterization revealed that surface treatments significantly influenced the tensile strength and maximum strain of the rice straw fiber-reinforced composites.

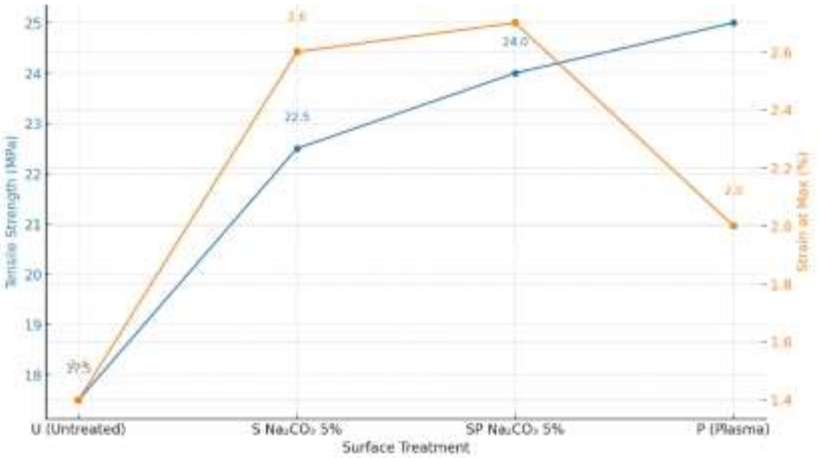

Figure 4. Effect of surface treatments on tensile properties of rice straw fiber composites.

Figure 4 illustrated that the untreated fibers (U) exhibited the lowest tensile strength, recorded at 17.5 MPa, with a maximum strain of 1.4%. This result is attributed to insufficient interfacial adhesion due to the presence of surface impurities and the absence of functional groups facilitating matrix bonding. A notable enhancement was observed when fibers were treated with a 5% Na_2CO_3 solution (S), where the tensile strength increased to 22.5 MPa and the strain at break reached 2.6%. This improvement is believed to result from the effective removal of amorphous substances and the exposure of hydroxyl groups, which enhanced matrix-fiber interactions. Further improvement was achieved through the combined ultrasound-Na_2CO_3 treatment (SP), with values of 24.0 MPa for tensile strength and 2.7% for strain. The synergistic effects of cavitation from ultrasound and chemical modification from Na_2CO_3 are assumed to have contributed to fiber defibrillation, increased surface roughness, and improved interfacial bonding, leading to superior mechanical performance[17]. In contrast, plasma-treated fibers (P) exhibited the highest tensile strength at 25.0 MPa, although the corresponding strain decreased to 2.0%. This indicates that plasma treatment may have increased surface activation and stiffness, but at the expense of ductility. The reduction in elongation is likely associated with enhanced crystallinity and crosslinking density introduced by the plasma exposure[35]. The combined ultrasound-alkaline treatment (SP Na_2CO_3 5%) is considered the most effective strategy for simultaneously improving strength and ductility. This finding underscores the potential of integrated physical-chemical surface treatments for optimizing the mechanical behavior of natural fiber composites within sustainable materials engineering.

3.3. Thermal gravimetric analysis

Thermal stability of the treated fibers was evaluated based on the residual mass at 700°C as derived from TGA data.

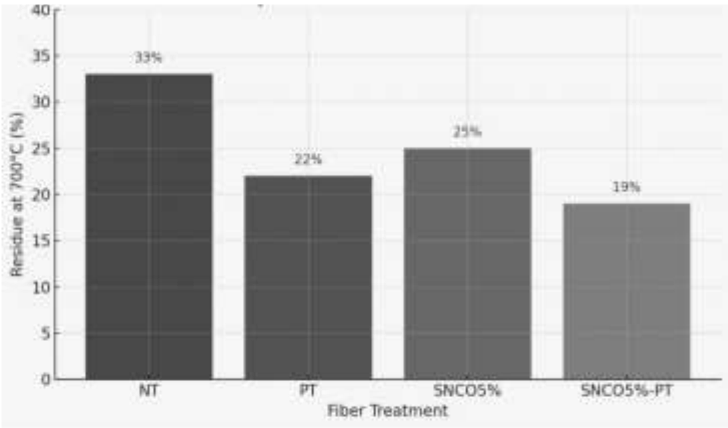

Figure 5. Thermal Stability of Treated Fibers Based on TGA Residue.

Based on **Figure 5**, the untreated fiber (NT) exhibited the highest residue at approximately 33%, indicating a high content of thermally stable inorganic components such as silica, which are commonly associated with lignocellulosic agricultural waste. In contrast, the sample treated with Na_2CO_3 and plasma sequentially (SNCO5%-PT) showed the lowest residue of 19%, suggesting an effective removal of thermally resistant impurities and enhanced decomposition of organic constituents. The Na_2CO_3-only treated fiber (SNCO5%) retained 25% of its mass, implying partial removal of silica and moderate thermal degradation behavior. Meanwhile, the plasma-treated sample (PT) showed a further reduction in residue to 22%, possibly due to enhanced surface etching and the oxidation of carbonaceous materials facilitated by the high-energy plasma environment. These findings suggest that the SNCO5%-PT treatment not only modifies the surface chemistry but also enhances the thermal degradability of the biomass, making it more compatible for thermoset or thermoplastic composite applications where thermal resistance and decomposition behavior are critical design parameters.

4. Conclusion

The effects of plasma, ultrasound (sonication), and combined ultrasound-plasma (SNCO5%-PT) treatments on rice straw powder were systematically examined in terms of morphology, functional group composition, and crystallinity. Untreated rice straw exhibited a smooth, waxy surface that hindered interaction with polymer matrices. Plasma treatment induced minor etching, while sonication using sodium carbonate (SNCO5%) contributed to increased surface roughness and partial removal of hemicellulose and lignin. The combined SNCO5%-PT treatment yielded the most notable surface modification, characterized by higher porosity and roughness, suggesting potential for improved polymer interlocking. Additionally, a substantial increase in surface silica content (approximately 4.5-fold) and O/C ratio (from 0.04 to 0.19) was observed, implying enhanced surface polarity and the presence of oxygenated functional groups. FTIR results showed stronger absorption bands associated with hydroxyl (-OH) and carbonyl (C=O) groups, while the relative crystallinity index (RCI) improved from 0.53 to 2.77. XRD analysis also confirmed an increase in crystallinity and crystallite size following SNCO5%-PT treatment. Although these physicochemical enhancements are promising, further mechanical testing and interface analysis are required to conclusively determine the extent of improvement in composite performance. Therefore, the results of this study are best interpreted as a preliminary indication of the potential of combined physical treatments to improve fiber-matrix compatibility in bio-based composites.

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Conflict of interest

The authors declare no conflict of interest.

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