Characterization of an Eco-friendly Gypsum Composite Board Using Agricultural Fibers (Rice Straw)

Ali Hassanpoor Tichi,[a,*] and Amin Khatiri [b]

In this research, a novel mineral-based composite board was developed using gypsum as a mineral binder and rice straw as a readily available agro-based resource. The study involved two key phases: Phase 1: The preliminary assessment of rice straw-gypsum composite involved integrating different ratios of rice straw into gypsum to examine the influence of rice straw integration on the composite board's performance. The specific proportions used were 90:10%, 80:20%, and 70:30% for rice straw to gypsum. Phase 2: Reinforcement with bacterial nanocellulose fibers. In the subsequent phase, gypsum board composites containing 10%, 20%, and 30% rice straw were further enhanced by the addition of bacterial nanocellulose fibers at 1% and 3% levels. The results indicated a significant influence of rice straw incorporation on the physical and mechanical properties of the panels. The composite boards with 3% bacterial nanocellulose fiber gel exhibited the highest mechanical performance, with values of 13.5 MPa for modulus of rupture, 4650 MPa for modulus of elasticity, and 0.79 MPa for Internal Bond. The study revealed that the adverse effects of rice straw substitution on the mechanical properties and thickness swelling of the panels could be mitigated to a certain extent by incorporating nanocellulose fibers.

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Keywords: Gypsum; Bacterial nanocellulose fiber; Rice straw; Agricultural residues; Panels

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INTRODUCTION

Wood and wood-based composites (WBCs) have several extraordinary features, such as high strength-to-weight ratio (specific strength), high mechanical strength, thermal insulation, electrical insulation, high flexibility in design, renewability, recyclability, and more importantly sustainability and availability. They also cover a vast spectrum of applications, such as building construction, interior design, exterior design, cabinetry, and furniture making, which have made them an integral part of mankind's evolution and play an important role in their well-being and comfort. Consequently, over the past decades as a response to consumer demand, the global production capacity of WBCs has remarkably risen from 25 million m^3 in 1961 to 400 million m^3 in 2021, which ended in doubling (2.5 billion m^3 in 1961 to 4 billion m^3 in 2021) the global consumption of wood-based raw materials (FAO 2023). In countries like Iran, the wood supply is much worse due to the tripling of production volume in the last decade (more than 3 million m3) and ongoing forest conservation plans (Heidarlou *et al.* 2023). This significant rise in the utilization of solid wood has put the forest – as the main supplier of woody raw material – into the sustainability challenge, opening strong competition for providing solid wood, especially fresh wood chips between WBC manufacturers. Furthermore, the significant rule of wood as the main raw material in the biofuel and biorefinery industries has amplified this competition, resulting in the drastic and undesirable rise in solid wood price.

In recent decades, significant research efforts have been dedicated to exploring alternative biomass resources as a response to evolving challenges in tandem with shifts in forest management strategies. These efforts focus on leveraging resources such as annual plants, lignocellulosic wastes, recovered cellulose fibers, and agricultural residues. One notable application of these resources is in the substitution of synthetic fibers with natural fibers to enhance the sustainability of concrete and mortar structures. Natural fibers, including sisal, flax, hemp, bamboo, and coir, present distinct advantages over synthetic alternatives due to their cost-efficiency, lightweight characteristics, and abundance as renewable resources, thereby contributing to the development of sustainable and eco-friendly materials (Van Nguyen and Mangat 2020). Rice, being one of the top three globally cultivated crops, generates a substantial by-product in the form of rice straw. Various methods exist for managing rice straw, including its use as livestock feed, on-site burning, or incorporation into the soil, each with its own set of benefits and drawbacks. While burning in the field is a cost-effective approach, it contributes to air pollution through CO_2 emissions (Van Nguyen and Mangat 2020; Haque *et al.* 2022). The current methods of rice straw processing and utilization have significant environmental implications due to the release of greenhouse gases such as CO_2, N_2O, and CH_4. These emissions contribute to climate change and have a critical impact on the environment. Moreover, these processes do not fully harness the potential of rice straw materials, resulting in a significant loss of valuable resources (Le *et al*. 2022). Upon examination of rice straw's potential as a natural fiber source, it becomes evident that there are promising possibilities for its utilization in strengthening cementitious mortar, gypsum, and concrete structures, thereby opening new avenues for sustainable construction materials. This utilization not only addresses waste management concerns, but it also showcases the high reinforcing potential of rice straw fibers in construction materials (Chen *et al.* 2018). It was shown that the above-ground crop stalks, such as rice, because of their similarity to wood in chemical and morphological aspects, are known as valuable agro-based residue and have gained a broad interest from researchers to be investigated. Rice straw, a type of lignocellulosic biomass, stands out due to its distinct chemical composition. Besides the typical components of lignocellulosic biomass, rice straw contains a significant amount of silica, which is absorbed from the soil through the polymerization of silicic acid. This unique interaction between silica and the cellulose and lignin components sets rice straw apart from other biomass materials (Bhattacharya and Mandal 2018). Specifically, rice straw is known as one of the most cellulose-rich biomass (more than 45%) with low content of lignin and hemicellulose (about 20% each) (Nguyen *et al.* 2018). The usage of rice straw has been studied as a sustainable alternative raw material resource for forest products to decrease the supplies of woody raw material, and to participate in agro-based waste management. However, despite all the aforementioned issues, it is reported that a large amount of high-potential agricultural residues including rice straw are currently burnt in the field without any specific purpose. As a result, burning agricultural residues delivers a huge amount of highly toxic and harmful pollutants and particles to the atmosphere, which can cause the incurable debilitating disease known as Silicosis for human beings and contributes to the climate change issue. However, parts of these agricultural residues account for livestock feed and domestic fuel, or are left in the field for better retention of soil nutrients.

The growing awareness of environmental degradation has led to a pressing need for sustainable practices. Biodegradable polymers are a promising solution to this ecological crisis, offering both a replacement for synthetic polymers and opportunities for innovative material development. One notable example of such a material is bacterial nanocellulose (BNC), a polymer synthesized by bacteria (Taubert *et al.* 2019; Volova *et al.* 2021; Skiba *et al.* 2023).

Bacterial cellulose (BC), like cellulose in general, is a polymer composed of glucopyranose monomers connected by β-1,4-glycoside bonds. This unique biopolymer is synthesized extracellularly by certain bacteria, including *Acetobacter* and *Gluconacetobacter*. The structure of BC is similar to that of plant cellulose, but its degree of polymerization, purity, and crystallinity is higher. With the advancement of fermentation and nanomaterials, BC is drawing more and more attention from researchers. Its research scope was extended from fermentation process optimization to application in different fields. In the food industry, BC has been used as a thickener, food packaging material, and supplement for low-calorie food. It has been used as an adsorbent for leaked crude oil and toxic substances. In the pharmaceutical industry, it has been used as wound antibacterial dressing and drug excipient. In the paper industry, it has been used as a material for the production of flexure-durable paper and other specialty paper (Xu *et al.* 2022). Bacterial nanocellulose (BNC) exhibits remarkable versatility, serving as both a matrix and a reinforcement. Additionally, its properties can be tailored through both in situ and ex situ modifications (Moniri *et al.* 2017; Stumpf *et al.* 2018).

Bacterial cellulose exhibits numerous eco-friendly attributes, primarily due to its exceptional purity, which necessitates less energy for purification compared to plant cellulose (Klemm *et al.* 2005). During the fermentation process, microorganisms either move freely within the media or are attached to cellulose fibers (Dufresne *et al.* 1997), resulting in a highly swollen gel structure. The key attributes of bacterial cellulose include its exceptional mechanical strength, purity, crystallinity, and water content (Klemm *et al.* 2005). The microfibrils of bacterial cellulose are organized in a three-dimensional nanofibrillar network, allowing for the retention of water within the thin, highly hydrophilic, porous structure. The wet or dry nanobacterial cellulose structure features numerous pores, making it suitable for various applications. The supramolecular arrangement of cellulose molecules, stabilized by interchain and intrachain hydrogen bonds, renders bacterial cellulose insoluble in both water and common organic solvents, as well as resistant to enzymatic and chemical hydrolysis (Römling 2002). Furthermore, the considerable mechanical strength of bacterial cellulose is attributed to the linear chains of cellulose and strong cohesion between macromolecules (Klemm *et al.* 2006). Mechanically, bacterial cellulose fibers exhibit a uniform material property, unaffected by variations in diameter (Ganesh *et al.* 2005). The crystallinity of bacterial cellulose is approximately 80%, influenced by the specific culture conditions, including the composition of the growth medium and the production process (static or agitated) (Jung *et al.* 2010; Trovatti *et al.* 2011). The production of eco-friendly products is gaining significance; in this context, microbial pathways for nanocellulose production offer distinct advantages (Varghese *et al.* 2019). Bacterial cellulose possesses unique physical and mechanical properties that set it apart from other biomaterials, as previously discussed. It also features ultrafine fiber networks with variable geometry pores, the ability to mold into diverse structures, and a wide range of chemistry and physics that can be modified (Stumpf *et al.* 2018). Due to these properties, bacterial cellulose has been shown to be a versatile biopolymer with extensive possibilities of applications since its discovery (Silva 2019a, b). Recent studies have demonstrated the breadth of bacterial cellulose applications across various sectors, including food (Azeredo *et al.* 2019), biomedical (Hobzova *et al.* 2018), and cosmetic industries (Stasiak-Rozanska and Ploska 2018). Besides the significance of pure bacterial cellulose, it also excels in the area of nanocomposites (Rai *et al.* 2019). The interest in using bacterial cellulose for nanocomposite applications stems from the high crystallinity and mechanical resistance conferred by its naturally nanosized three-dimensional network (Nascimento 2018a,b).

Gypsum composite boards, because of their outstanding characteristics as environmentally friendly building materials, such as energy saving, sound insulation, thermal insulation, low price, decoration capability, availability, constructability, and easy production, were among the most frequently used materials in civil engineering and have been widely employed in residential construction as roof or wall sheathing (Guna *et al.* 2021). Furthermore, plasterboards are commonly used in construction for thermal insulation and air purification, but high-purity natural gypsum products are essential in wallboard manufacturing due to the detrimental impact of impurities on gypsum properties (Bouzit *et al.* 2019). However, gypsum board composites have been used without any additional reinforcements or additives. *i.e*., in the form of plasterboard regarding their inadequate impact resistance, brittleness, and heaviness for some building applications. The most frequent additives used in gypsum boards are natural fibers (Hernández-Olivares *et al.* 1992; Li *et al.* 2003; Dalmay *et al.* 2010; Liu *et al.* 2012; Ramezani *et al.* 2012; Boccarusso *et al.* 2020; Tichi *et al.* 2020; Singh *et al.* 2022; Tichi *et al.* 2022), such as flax, sisal, rice, jute, wheat, hemp, maize, sunflower, cotton, barely, and wood fiber, mineral particles, synthetic fibers (Eve *et al.* 2002; Martias *et al.* 2014), including polyamide, and glass fiber, and polymers (El-Maghraby *et al.* 2007). Rice straw, a plentiful agro-based residue, presents a promising opportunity for integration into gypsum composite boards to address various challenges and enhance their performance as construction materials. The composition of rice fiber is notable, with cellulose, hemicelluloses, lignin, and wax comprising significant proportions of 41 to 57%, 33%, 8 to 19%, and 8% to 38%, respectively (Aladejana *et al.* 2020). This blend of components in rice straw, including cellulose, hemicellulose, lignin, ash, and other minor elements, offers a unique combination for composite board production. Specifically, rice straw contains cellulose (32 to 38.6%), hemicellulose (35.7 to 19.7%), lignin (22.3 to 19.5%), and ash (10 to 17%). Through leveraging these properties, the incorporation of rice straw into gypsum composite boards can lead to improved structural integrity and sustainability in construction applications (Goodman 2020).

In the study conducted by Kaya *et al.* (2020), it was observed that the replacement of pine particles in the gypsum matrix at levels ranging from 10% to 50% had a negative impact on the modulus of rupture, modulus of elasticity, internal bonding strength, thickness swelling, and water absorption, indicating a decline in the composite's overall performance. Another study showed that reinforcing the gypsum composite board with wooden fibers at 1% to 8% levels can significantly reduce the flexural properties and compressive strength of the composite boards (Hošťálková *et al.* 2019). Another study carried out by Vavřínová *et al.* (2022), investigated the combined effect of three gypsum types (Class I, II, and III) with wheat straw at levels of 0%, 2.5%, and 5%, and examined in relation to the mechanical and thermal performance of the composite boards. The findings of the study revealed that an increase in the wheat straw ratio within the binder matrix resulted in a decline in flexural and compressive strengths, as well as a reduction in thermal conductivity, suggesting a compromise in the overall performance of the composite. Nevertheless, the authors mentioned that the reduced mechanical properties of the composite made it unsuitable where high-strength plasterboard is needed but it is still suitable for interior applications. It is worth mentioning that the literature stated the negative effect of incorporation of lignocellulosic additives in the gypsum matrix in large part is attributed to the poor dispersion of wood fibers in the gypsum matrix, chemical incompatibility between the inorganic binder and wood fibers, and lack of sufficient mechanical interlocking due to the smooth surface of lignocellulosic additives.

Contrarily, the study conducted by Şahin and Demir (2019b) revealed that the incorporation of a waste cellulosic source, such as secondary fibers from waste papers, could have a favorable impact on the mechanical properties of gypsum board composites. The results of the investigation indicated that the particle size and their distribution within the binder matrix are two of the most influential factors in the mechanical performance of the composite boards, suggesting a potential for enhancing the composite's strength and durability.

Tichi *et al.* (2020, 2022) and Tichi and Razavi (2023) in their research on the reinforcing effect of nanoparticles (wollastonite and cellulose) in the gypsum-lignocellulosic matrix showed that the nanoparticles due to their specific surface area are more compatible with inorganic matrix compared to the agro-waste particles and can effectively improve the mechanical strength of the composite boards to some extent.

Given the unique characteristics of materials at the nanoscale, nanoparticles exhibit enhanced compatibility with the mineral binder matrix, offering potential to mitigate the negative impact of lignocellulosic additives within the matrix. Consequently, this research explored the impact of incorporating rice straw particles into the gypsum matrix initially, followed by assessing the reinforcing influence of nanocellulose particles on the physical, mechanical, and thermal attributes of the composite boards. This evaluation aimed to determine the suitability of the investigated composite as a viable building material.

EXPERIMENTAL

Materials and Methods

Rice straw

The rice straw required for the study was sourced from an agricultural field in Babol, Mazandaran, Iran, where rice is an important crop and is cultivated in substantial quantities annually, resulting in a large volume of straw as a byproduct. Following the collection, the straw was transported to the wood and paper science and technology laboratory at the Technical and Vocational University (TVU) of Mazandaran for further processing and preparation. First, the leaves and rachis were separated from the stem, and the rice straw was then thoroughly washed using deionized water to remove the debris and impurities. Next, the rice straw was air-dried to a moisture content of below 15%. After that, the clean air-dried straws were chopped into 15- to 30-mm-long strips (Fig. 1). Finally, the prepared straws were kept in plastic bags to prevent moisture exchange prior to the application.

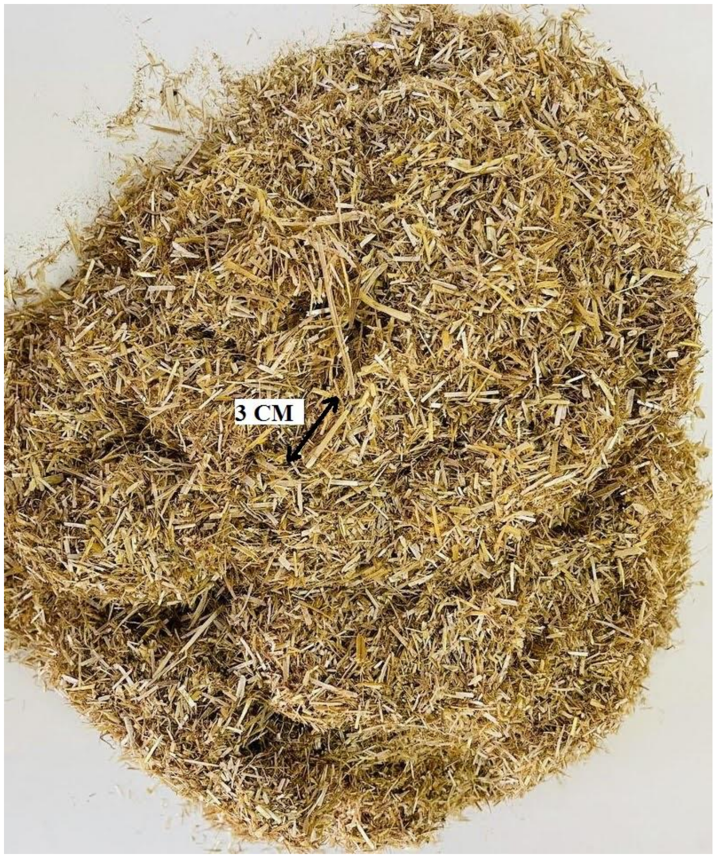

Fig. 1. Rice straw used in this study

Plaster

The commercial grade hemihydrate of calcium sulfate ($CaSO_4$. 0.5 H_2O), which is used as a constructional building material in Iran (building plaster), was employed as a bonding agent in the present study. The physicomechanical (Table 1) and chemical (Table 2) characteristics of gypsum powder described by the manufacturer (Omid company, Semnan, Iran) are listed as blow.

Table 1. Physicomechanical Characteristics of Gypsum Powder

Property	Content (%)
CaO	36.19
MgO	1.73
SiO_2	0.99
Fe_2O_3	0.31
Al_2O_3	0.24
NaCl	0.05
CO_2	0.41
SO_3	55.34
$CaSO_4$	91.85
Hydration water	4.56

*$CaSO_4$ is a partial combination of SO_3 and CaO. Because of this, the numbers in the table exceed 100%.

Table 2. Chemical Composition of Gypsum Powder

Whiteness (%)	Minimum Final Setting Time (min)	Maximum Final Setting Time (min)	Minimum Flexural Strength (MPa)	Minimum Compressive Strength (MPa)
92.2	8 to 10	22 to 25	3.07	8.29

Bacterial nanocellulose fiber (BNCF)

The nanocellulose fiber was kindly provided by Nano Novin Polymer Company, Sari, Iran. The nanoparticles were synthesized through bacterial synthesis in *Acetobacter xylinus* aqueous cultivation media for 2 weeks having a purity of ≤ 99% and fiber diameter of 30 to 50 nm (Fig. 2). Additional information on BNCF characteristics and features can be found elsewhere (Tichi and Razavi 2023).

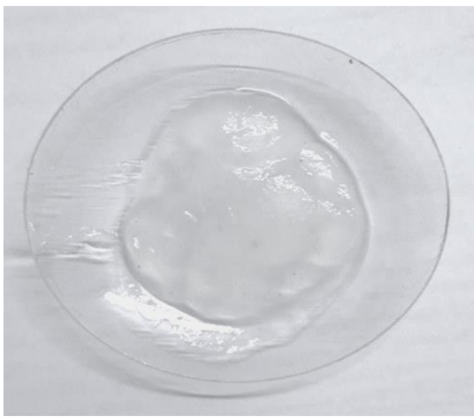

Fig. 2. Bacterial nanocellulose fiber used in this research

Sample preparation

In the present study, the plaster-type gypsum and rice straw were adopted as the binder and filler, respectively. The preparation process of experimental panels was performed at standard laboratory conditions. First, the bacterial nanocellulose fiber (BCNF) was prepared by diluting to 1% using distilled water. After that, the gypsum powder and chopped rice straw were homogeneously mixed in a laboratory-type blender (2000 rpm) at a certain gypsum-to-straw admixture ratio of 90:10, 80:20, and 70:30, respectively. Then the prepared BCNF suspension was gradually added to the mixture, accounting that the gypsum and water ratio was kept constant at 0.8 at all treatment levels (Table 3). The mixture was adequately blended for 20 min to obtain a homogeneous and well-dispersed mixture (Tichi and Razavi 2023). Next, the mixture was poured onto the steel mold with dimensions of 400 mm × 400 mm × 150 mm (length × width × height) that was covered with wax paper. The authors tried to perfectly distribute the mixture throughout the mold to achieve a uniform density profile as much as possible. Afterward, the mat was cold-pressed using an aluminum plate and carpentry clamps to the target thickness of 16 mm. The mats were retained under pressure for 2 days for curing (Fig. 3). After cold pressing, all panels were conditioned (23 ± 2°C temperature and 50 ± 5% humidity) for seven days prior to cutting and testing. Three panels were made as replicates for each variable, then their physical, mechanical, thermal, and chemical properties were examined. Also, the process of making rice straw and plaster composite board is shown in (Fig 4).

Fig. 3. Board made of rice straw and gypsum

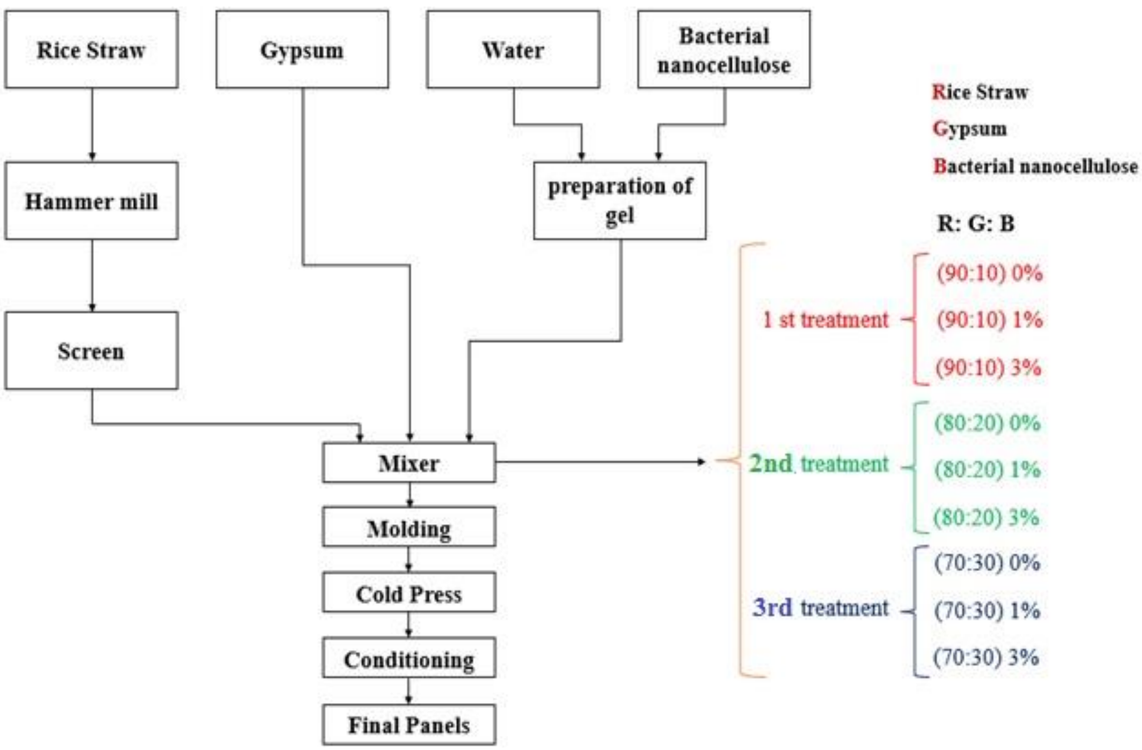

Fig. 4. The process of making rice straw and gypsum composite board

Mechanical properties

Modulus of rupture (MOR), modulus of elasticity (MOE), and internal bonding strength tests were conducted based on the descriptions in the DIN EN 634-1 (1995) and DIN EN 634-2 (1995) standards. The mechanical tests were performed using a universal mechanical tester (STM-600, Santam, Tehran, Iran).

Table 3. Experimental Design

Sample ID	Gypsum Ratio (%)	Rice Straw Ratio (%)	Bacterial Nanocellulose Fiber (%)	Panel Density (Kg/m^3)
A	90	10	0	1033
B			1	1120
C			3	1200
D	80	20	0	900
E			1	1000
F			3	1080
G	70	30	0	800
H			1	8800
I			3	9100

Thickness swelling

The thickness swelling after 2 and 24-h submersion of samples in distilled water was determined according to EN 317 (1993) to assess the short-term and long-term hygroscopic behavior of composites. Based on the standard description, after conditioning, the thickness of test specimens with a nominal dimension of 50 mm × 50 mm × 16 mm were precisely measured in the middle of samples using a digital caliper to an accuracy of ± 0.01 mm, then the samples were vertically immersed in distilled water. After each immersion time (2h-and-24-h), the thickness of samples was measured and the thickness swelling value was calculated according to Eq. 1,

$$G_t = \frac{t_2 - t_1}{t_1} \times 100 \tag{1}$$

where G_t is expressed as thickness swelling (%), t_2 and t_1 represent the sample thickness (mm) after immersion time (2 h and 24 h), and the initial thickness of samples before immersion (mm), respectively.

Three samples from each panel were taken and an average of 9 samples were reported for each variable (n = 9).

Mass loss (Fire retardant)

The mass loss (fire performance) was performed on samples with dimensions of 250 mm × 90 mm × 16 mm according to ISO 11925-3 (2020). Six samples were examined from each panel and an average of 18 samples were recorded for each variable (n = 18). The test specimens were fixed on the holder with the flame source adjusted at a 30 mm distance and 45° from the sample. The ignition process was performed for 30 s on samples and the mass loss was calculated using the following Eq. 2,

$$ML = \frac{W_2 - W_1}{W_1} \times 100 \tag{2}$$

where ML represents the mass loss (%) and W_2 and W_1 are the weight of the samples after exposure and the initial weight of samples before exposure (g), respectively.

Density

The apparent density was also measured based on the ratio of mass-to-volume for samples with nominal dimensions of 50 mm × 50 mm × 16 mm following EN 323 (1993). A digital caliper with an accuracy of 0.01 mm and a digital balance with an accuracy of

0.001 g were adapted to record the volume and mass of the samples, respectively. The density of samples was calculated based on Eq. 3. Three experimental samples were taken from each panel as replicates and a total of 9 measurements were reported for each variable (n = 9). Equation 3 is as follows,

$$\rho = \frac{m}{b_1 \times b_2 \times t} \times 10^6 \tag{3}$$

where ρ is the density (Kg/m^3), m is weight (g), b_1 and b_2 are the length and width of the samples (mm), and t is the thickness of samples (mm).

Fourier transform infrared spectroscopy (FTIR)

In the research, Fourier transform infrared spectroscopy (FTIR) analysis was conducted under ambient conditions to identify and characterize the chemical groups present in the gypsum composite board (Spectrum two, Perkinelmer, Japan). The KBr pellet (1:1000 w/w) method was employed to collect the spectra in the wavenumber range of 400 to 4000 cm^{-1} with a resolution of 2 cm^{-1} in transmittance mode. This analytical technique allowed for the identification and quantification of the functional groups and chemical bonds in the composite, thereby providing valuable insights into the chemical composition and structure of the material.

Scanning electron microscopy (SEM)

In the study, the microstructure of the failure surface in the gypsum composite board was analyzed using scanning electron microscopy (SEM). A small sample (10 mm × 10 mm) was carefully extracted from the failure surface and prepared for SEM analysis by mounting it on an aluminum holder. The sample was then subjected to gold sputtering (AIS2100; Seron Technology, South Korea) in a vacuum chamber at a current of 2 mA for a duration of 2 min to enhance its conductivity and improve the image quality. Subsequently, the sample was observed and imaged using the SEM, which provided detailed insights into the microstructure and morphology of the failure surface, thereby shedding light on the mechanisms of failure and the underlying factors that contribute to the material's performance and durability.

Statistical analysis

The Statistical Package for the Social Sciences (SPSS) software (Dell Inc., Round Rock, TX, USA) was employed to conduct statistical analysis on the data obtained from the gypsum composite board samples. The analysis involved utilizing one-way analysis of variance (ANOVA) to compare the data sets, with a significance level set at $P < 0.05$. This statistical approach allowed for a comprehensive evaluation of the experimental results, enabling the identification of significant differences and relationships within the data, thus providing valuable insights into the performance and characteristics of the composite materials.

RESULTS AND DISCUSSION

Infrared Spectroscopy (FTIR)

The FTIR analysis spectra are depicted in Fig. 5. The FTIR technique is extensively utilized for identifying surface functional groups in solid materials. In the FTIR analysis, the spectral bands observed between 3544 to 3406 cm^{-1} are attributed to the vibrational modes of -OH groups in α-cellulose and the robust bonding interactions present in gypsum, as documented in previous studies (Prasad *et al.* 2005). The bands identified within the range of 2800 to 2964 cm^{-1} correspond to the stretching vibrations of -CH bonds in the CH_2 and CH_3 groups. Additionally, the spectral region between 2000 to 2150 cm^{-1} exhibits characteristic C-OH stretching vibrations associated with cellulose structures. This thorough analysis using FTIR spectroscopy offers significant insights into the molecular composition and bonding properties of the composite materials being studied, enabling a deeper understanding of their structural features and chemical interactions (Yalcin 2022).

The analysis of waves in the FTIR spectra reveals the presence of Ca-O and Mg-O bonds at the 850-980 cm^{-1} range (Yalçin and Kaya 2022), while weak bonds and SiO_2 symmetrical and asymmetrical bonds are observed at 1040, 884, 525, and 480 cm^{-1} (Ağan *et al.* 2006). The band at 3538-3455 cm^{-1}, attributed to the -OH group, exhibits strong vibrations due to water in the structure. Generally, peaks in the range of 1030-1060 cm^{-1} and 1145-1162 cm^{-1} are assigned to C-O and C-O-C stretching in polysaccharides, specifically cellulose and hemicellulose. The bands at 3492, 3407, 3245, 1684, and 1621 cm^{-1} are attributed to the stretching vibration of the functional groups of the O–H bands (Jeong *et al.* 2017).

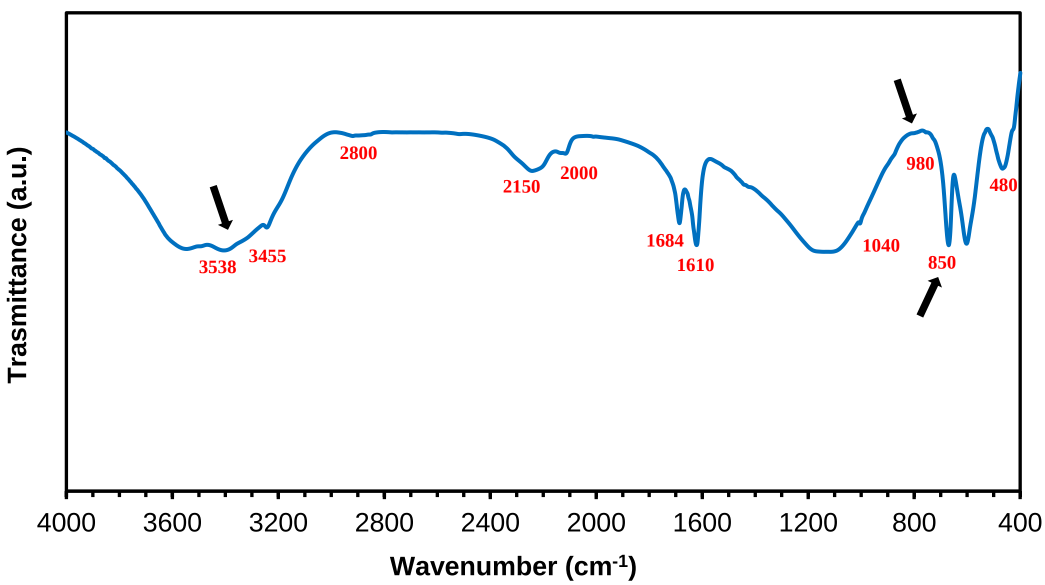

Fig. 5. Fourier transform infrared spectra of rice straw, gypsum, and bacterial nanocellulose fiber (BNCF)

Mechanical Characteristics of Gypsum Composite Board

The statistical analysis indicates that both independent and interactive variables significantly impacted the bending strength, modulus of elasticity, and internal strength of the specimens at a 95% confidence level. The mechanical resistance levels of the composite boards varied, as depicted in Figs. 6, 7, and 8, highlighting the diverse responses of the materials to different factors and interactions. The highest mechanical performance was attributed to boards comprising 3% bacterial nanocellulose fiber (BNCF) gel and 10% rice straw with 90% gypsum, demonstrating respective values of 13.5 MPa for modulus of rupture, 4650 MPa for modulus of elasticity, and 0.79 MPa for internal bonding strength. Conversely, the lowest mechanical resistance was observed in boards lacking nanocellulose and consisting of 30% rice straw with 70% gypsum. These findings underscore the enhanced mechanical properties, including bending strength, modulus of elasticity, and internal strength, associated with the incorporation of bacterial nanocellulose fiber (BNCF) gel in the composite boards. The data from the sources clearly indicate that integrating straw fibers into composite materials can substantially influence the mechanical properties of the final products. It was observed that as the proportion of straw in the samples rose, there was a noticeable reduction in both flexural strength and compressive strength, underscoring the importance of carefully considering the fiber content for achieving desired mechanical performance in the composite materials (Vavřínová *et al.* 2022). The decrease in strength observed in the composite materials with increasing straw fiber content can be attributed to the fibers' greater compatibility with gypsum compared to particles, leading to improved internal bonding strengths (FPL 1987). Because the compressive strength of plaster is higher than the compressive strength of rice straw, this is the reason why the modulus of rupture was greatly reduced by adding rice straw in the composite, but on the other hand, the modulus of rupture was increased by adding bacterial nanocellulose fiber (BNCF) to the matrix, as shown in Fig. 6. The incorporation of lower specific gravity materials with higher specific gravity/strength properties into the gypsum matrix can modify the rigid structure of gypsum, enhancing its strength properties. The arrangement and compatibility of particles or fibers with gypsum matrices significantly impact the strength of composite panels (Şahin and Demir 2019a). Furthermore, the interaction between gypsum and additives, facilitated by effective wetting of the additives' surfaces by gypsum, can result in improved strength properties and dimensional stability of the composite materials. The smooth surface of crushed straw stalks may contribute to the observed decrease in flexural and compressive strength. Bouasker *et al.* (2014) further emphasize the importance of these factors in determining the mechanical properties of composite materials. The roughness of straw fibers plays a crucial role in determining the mechanical strength of straw fiber materials, particularly affecting the pull-out resistance of fibers and their adhesion to gypsum. Studies have shown that higher roughness is necessary to enhance adhesion to gypsum. Some research has indicated that the low strength values obtained from certain composites render them unsuitable for structural applications (Wei and Meyer 2014; Aladejana *et al.* 2020; Ismail *et al.* 2020). Conversely, Yang *et al.* (2020) demonstrated that an increasing proportion of straw reinforcement leads to a decrease in modulus of rupture, modulus of elasticity, internal strength, and mass loss. The decrease in strength with higher straw content can be attributed to increased intergranular porosity and reduced binder content. Additionally, the smooth surface of crushed straw stalks may contribute to this reduction in strength. Each surface of rice straw has surface roughness. This surface structure is basically caused by the inherent structure of the cell. In addition to this, processing with various machining operations (hammer mill) generally causes an irregular surface structure. Therefore, when a liquid comes into contact with the surface, the inherent structure of the straw surface and the irregularity of the straw surface caused by machining are combined and may cause the liquid to move along the surface by capillary forces. Utilizing straw stalks, rice, corn, or other natural fibers with various binders can be advantageous in regions where these materials are abundant, potentially reducing manufacturing costs by incorporating inexpensive and renewable fillers (Vavřínová *et al.* 2022). The morphology of cellulose not only influences gypsum crystal formation but also impacts the microstructure and interlocking mode of gypsum crystals, subsequently affecting total porosity and mechanical properties of the composite material (Nindiyasari *et al.* 2016).

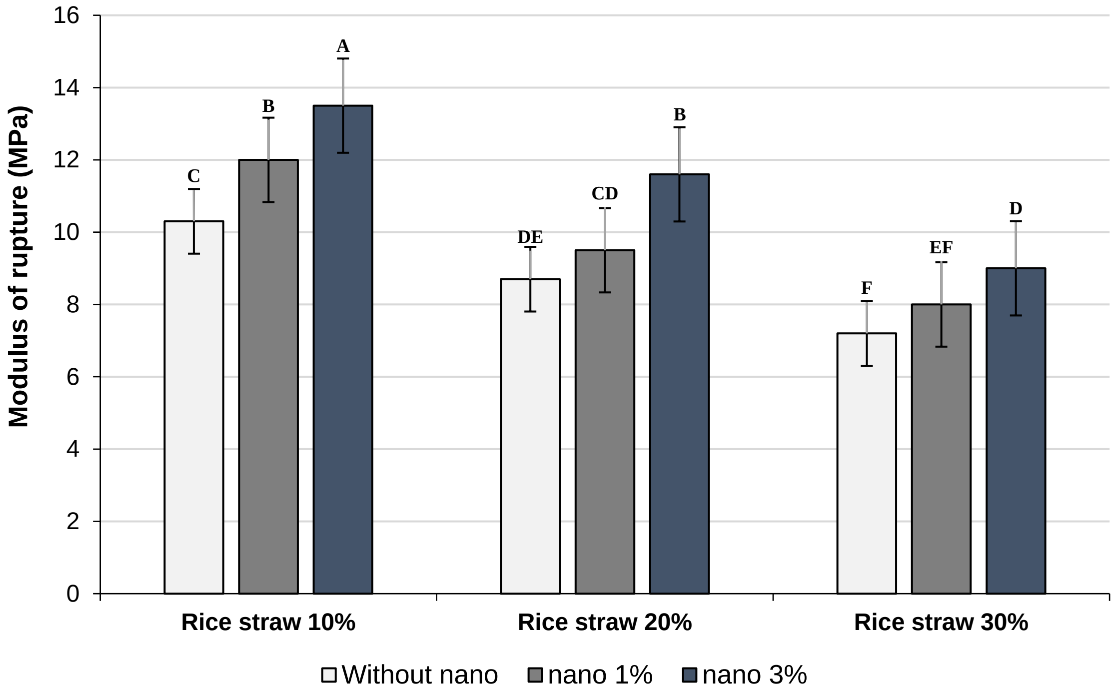

Fig. 6. Effect of rice straw mix ratio, gypsum, and bacterial nanocellulose fiber (BNCF) on modulus of rupture of composites

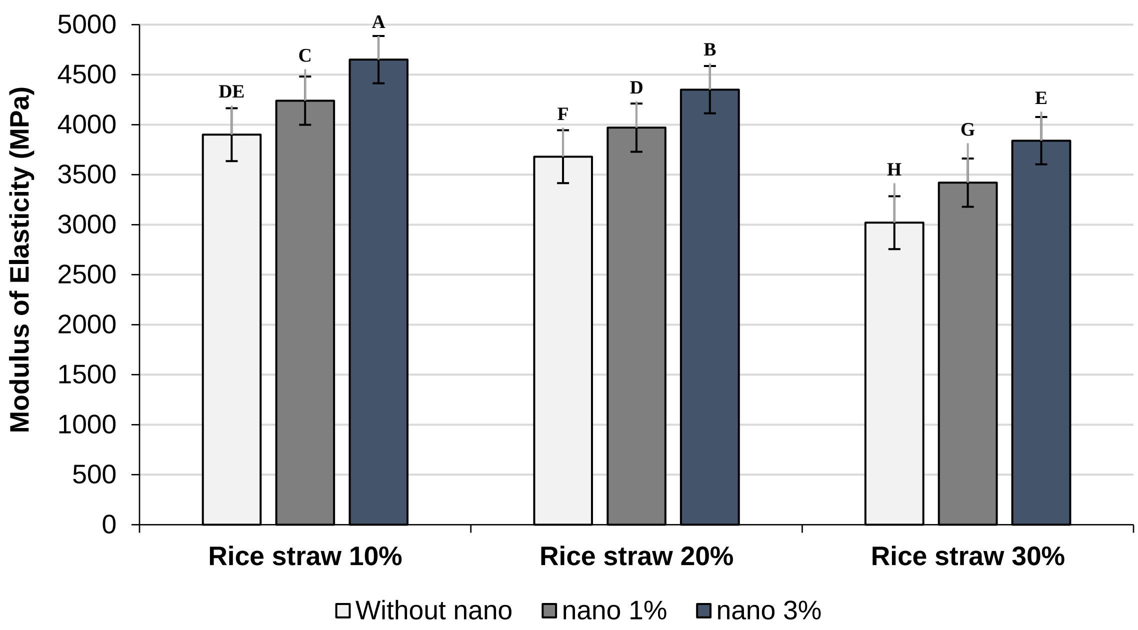

Fig. 7. Effect of rice straw mix ratio, gypsum, and BNCF on modulus of elasticity of composites

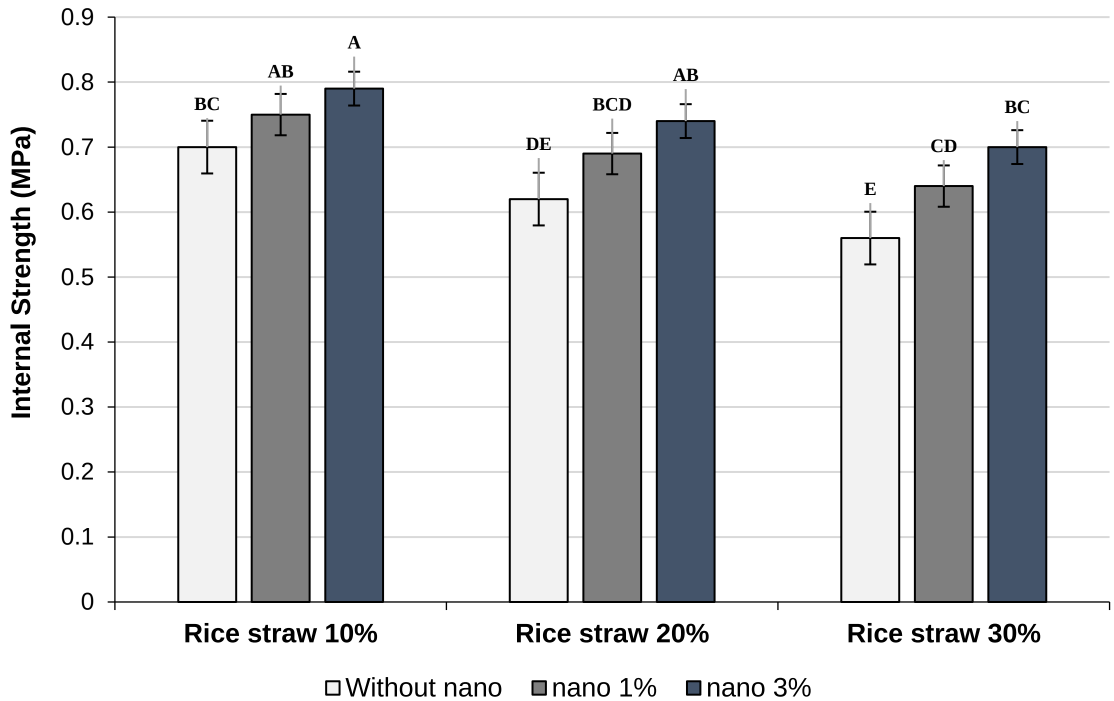

Fig. 8. Effect of rice straw mix ratio, gypsum, and BNCF on internal strength of composites

Stress concentrations provided by wood fibers in the gypsum matrix can lead to crack initiation and propagation at the interface, highlighting the importance of optimizing the volume fraction of fibers to matrix in composite design. The limited mechanical interlocking observed between gypsum and straw particles is attributed to the wax coating on straw hindering strong mechanical bonding with mineral binders (Ramezani *et al.* 2012). The presence of hydrophobic substances, such as wax in straw, compared to bagasse, may result in panels with higher tensile strength and lower internal bonding when straw content exceeds 50%, potentially due to the weaker bond strength of straw with gypsum (Nazerian and Kamyab 2013).

Physical Characteristics of Gypsum Composite Board

The boards with a blend of 10% rice straw, 90% gypsum, and 3% bacterial BNCF gel exhibited the least thickness swelling after 2 and 24 h of water immersion, measuring 2.75% and 3.34% respectively (Fig. 9). Increasing the bacterial nanocellulose fiber gel content from 0% to 3% in rice straw compositions of 10%, 20%, and 30% led to density increments of 16.5%, 20%, and 13.75% respectively (Fig. 10). Notably, boards containing 3% nanocellulose gel showed reduced thickness swelling compared to those without nanocellulose. The even dispersion of nanocellulose throughout the gypsum matrix and rice straw in BNCF-incorporated boards facilitated improved compaction, reducing water penetration, and minimizing thickness swelling. However, the hydrophilic nature of rice straw may compromise the dimensional stability of the boards, potentially due to increased hydroxyl group availability with higher fiber proportions in the composite. The introduction of bacterial nanocellulose fiber reduced the fire resistance of the boards due to its low heat transfer and organic composition. The higher surface energy of the cellulose nanogel enhanced the bonding between rice straw and gypsum, strengthening the surface and filling voids within the board, thereby increasing board density. The composite material demonstrated excellent flame resistance. These findings align with previous research by Tichi and Razavi (2023). As the agro-waste content rose from 0 to 25 wt%, the gypsum samples' density decreased, rendering it suitable for lightweight applications like non-load bearing walls.

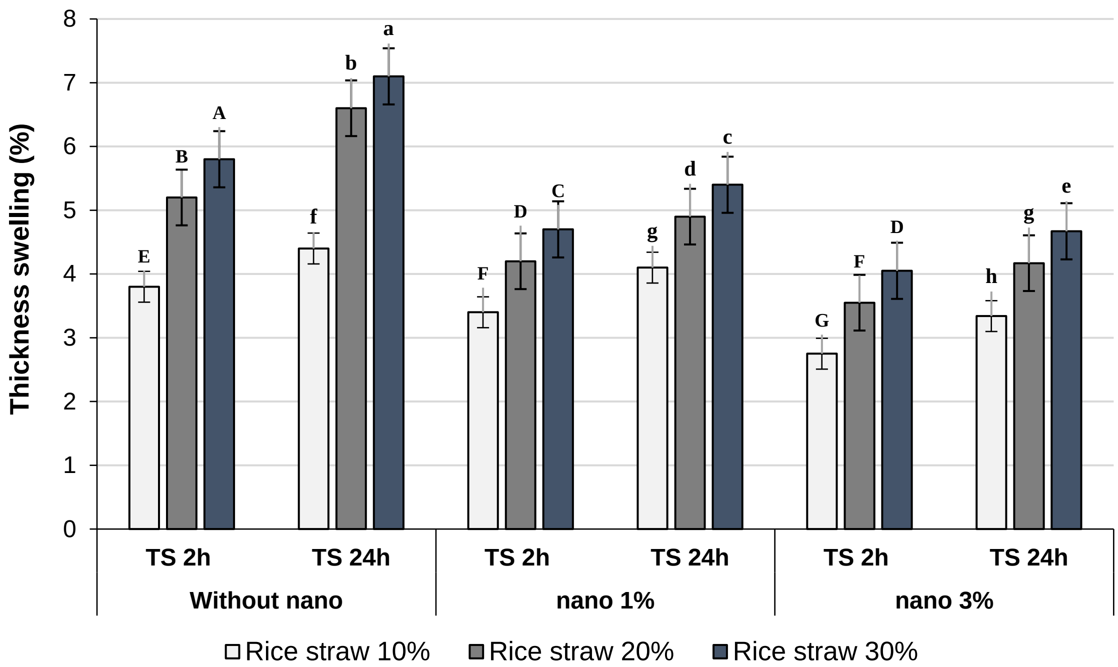

Fig. 9. Effect of rice straw mix ratio, gypsum, and BNCF on thickness swelling after 2 h and 24 h immersion in water of composites

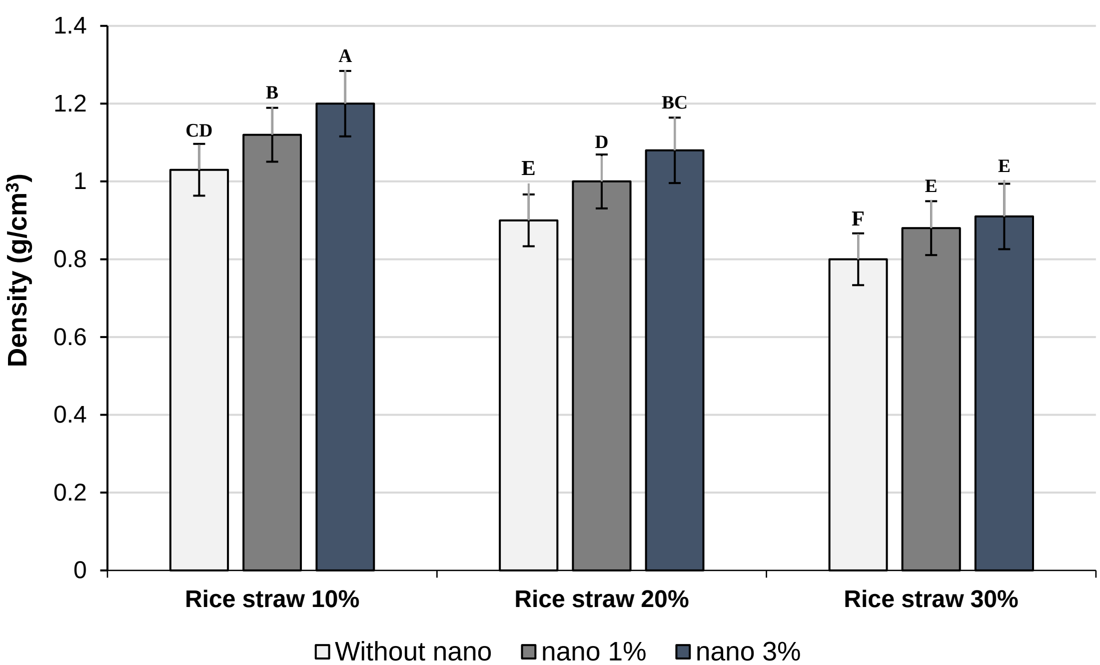

Fig. 10. Effect of rice straw mix ratio, gypsum, and BNCF on density of composites

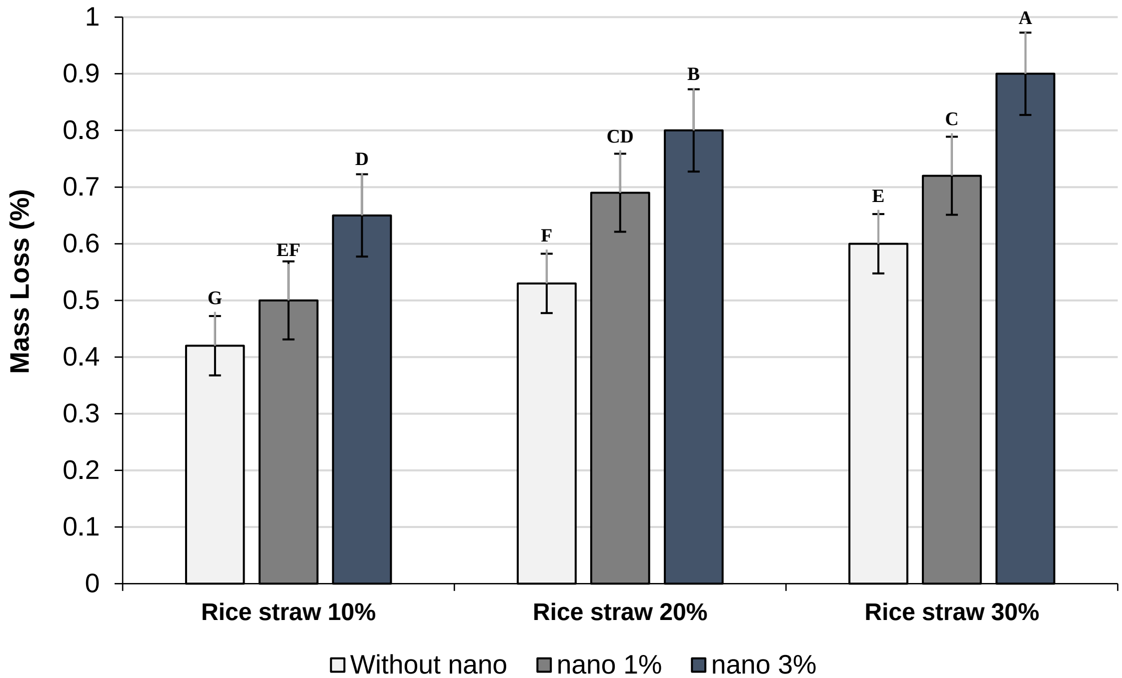

Fig. 11. Effect of rice straw mix ratio, gypsum, and BNCF on mass loss of composites

Gypsum's high water absorption capacity, exacerbated by rice straw incorporation due to straw's porous nature, is a notable drawback (Singh *et al.* 2022). During gypsum hydration, fibers bond with hydrates. Optimal bonding occurs with reduced fiber content and increased dehydrated crystals in wood-gypsum materials, leading to reduced thickness swelling (Ramezani *et al.* 2012). Lower bonding strength between straw particles and gypsum results in greater spring back after 24 h of water immersion due to weaker internal bonds (Nazerian and Kamyab 2013). Epidermic cells on the cross-section's outermost surface are coated with a thin layer of wax.

Also, in Table 4, the mechanical properties including flexural strength (MOR), modulus of elasticity (MOE) and internal bond strength (IB) and physical properties including thickness swelling, density and mass loss) of the composite board made of rice straw and gypsum with and without the addition of bacterial cellulose nanofibers are presented.

Table 4. Mechanical and Physical Properties of Composite Board Made of Rice Straw and Gypsum

Sample	MOR (MPa)	MOE (MPa)	IB (MPa)	TS 24h (%)	D g/cm^3	ML (%)
A	10.30	3900	0.70	4.40	1.03	0.42
B	12.00	4240	0.75	4.10	1.12	0.50
C	13.50	4650	0.79	3.34	1.20	0.65
D	8.70	3680	0.62	6.60	0.90	0.53
E	9.50	3970	0.69	4.90	1.00	0.69
F	11.60	4350	0.74	4.17	1.08	0.80
G	7.20	3020	0.56	7.10	0.80	0.60
H	8.00	3420	0.64	5.40	0.88	0.72
I	9.00	3840	0.70	4.67	0.91	0.90

Microscopic Scanning

Microscopic analyses were conducted to elucidate the mechanisms underlying the enhancement of mechanical properties in gypsum-rice straw composites. Microstructural images of samples with and without BNCF were obtained at 500 µm magnifications using SEM (Fig. 12). The examination of gypsum-rice straw crystal morphology revealed that the gypsum crystals were arranged irregularly and were needle-like in length (Fig. 12(a)). The interaction at the fiber-matrix interface is critical in determining the composite's strength, facilitating stress transfer from the gypsum matrix to the fibers across the interface during loading. Furthermore, the incorporation of bacterial nanocellulose fiber significantly enhances the bond strength, resulting in a substantial improvement in the surface roughness of rice straw (Fig. 12(a)).

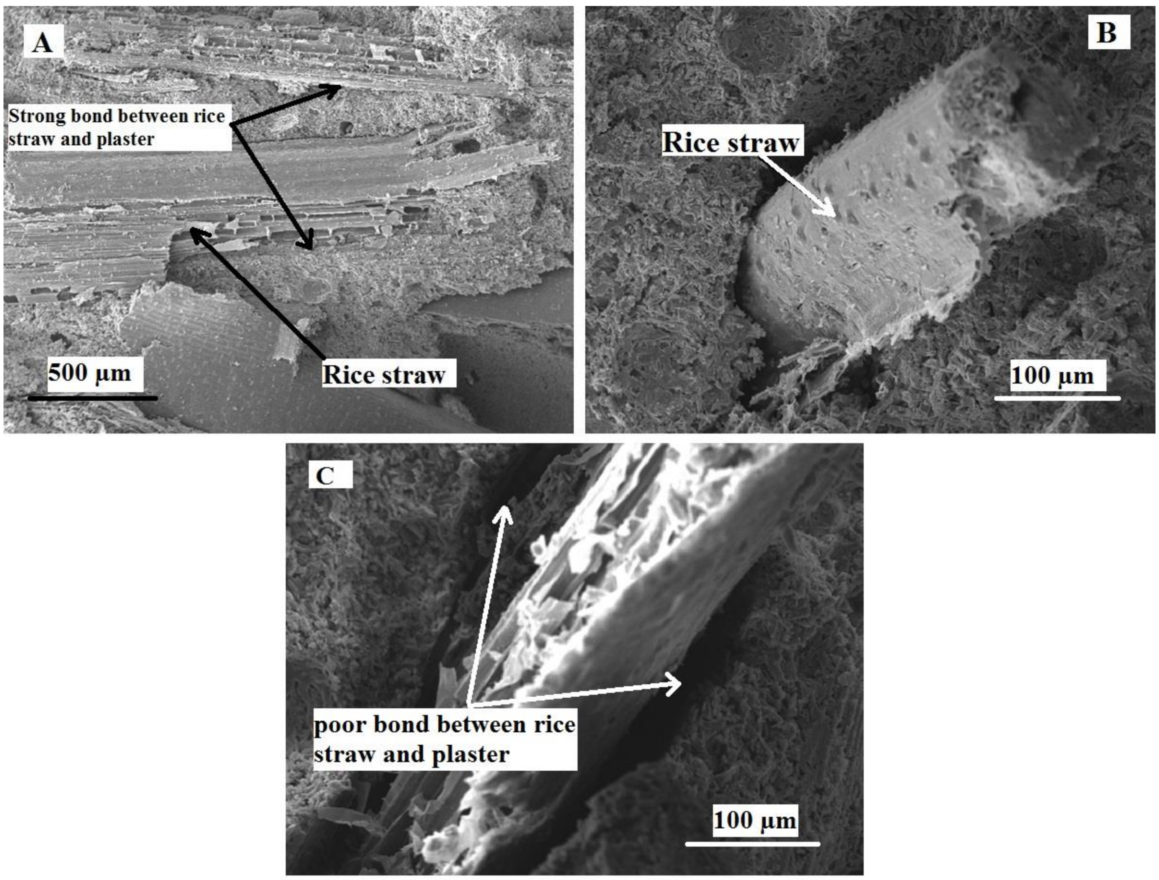

Fig. 12. SEM images of refractive surfaces, A: Boards containing 3% bacterial nanocellulose fiber, B: Boards containing 1% bacterial nanocellulose, and C; Boards without- bacterial nanocellulose (c)

Additionally, the findings suggest that water is anticipated to evaporate from the plaster, potentially enhancing fire resistance. This outcome is attributed to the evaporation process, which can reduce the plaster's combustible content. Furthermore, the gradual incorporation of bacterial cellulose nanofibers contributes significantly to this feature, owing to the inherent mechanical properties of these fibers. The addition of these nanomaterials induces mechanical entanglement and improved cohesion within the matrix, which, in turn, enhances fire resistance by reducing the material's flammability and increasing its thermal stability. Excess water in a gypsum rice straw mat needed to be excluded by drying, which led to many voids being generated (Amer *et al.* 2016; Yue *et al.* 2021). In Fig. 12, the incremental addition of bacterial nanocellulose fiber from 0% to 3% enhanced the adhesion between rice straw and gypsum, potentially resulting in increased mechanical and physical resilience of the boards. The microscopic images further demonstrate that BNCF promotes cohesion, resulting in a stronger and more secure bond between rice straw and gypsum. This process effectively fills voids within the board, enhancing dimensional stability and increasing the density of the composite panels. The improved cohesion and void filling contribute to the overall mechanical properties and durability of the composite materials.

In Fig. 12-C, the lack of bacterial nanocellulose fiber in the boards is evident, resulting in the formation of significant voids and a weakened bond between rice straw and gypsum.

Fibers extending from the fractured cement paste surface indicate a pull-out failure mechanism, suggesting that these fibers acted as crack-bridging elements within the cement paste. In the cement paste batches containing bacterial cellulose (BC), the visible fiber diameter was approximately 60 nm, while in cellulose nanofiber (CNF) batches, it measured around 30 nm. The fiber distribution appeared uniform throughout the matrix (Haque *et al.* 2022). A challenge associated with incorporating natural fibers in cementitious composites is the weak bond resulting from the hydrophilic nature of these fibers. The failure at the interface between natural fibers and the cement matrix under loading is attributed to inadequate chemical and physical interactions between the two components (Van Nguyen and Mangat 2020).

CONCLUSIONS

1. The increase in mechanical properties, including the modulus of rupture (MOR), modulus of elasticity (MOE), and internal bond strength, exhibits a positive correlation with the increasing concentration of bacterial nanocellulose (BNCF) in the composite board. This improvement can be attributed to the optimized density and uniform distribution of particles within the board, influenced by hydration temperature. Notably, the composition yielding the most favorable mechanical properties consists of 10% rice straw, 90% gypsum, and 3% bacterial nanocellulose fiber. The results are consistent with strong interfacial adhesion between the bacterial cellulose and the gypsum.
2. The board thickness swelling was 2.75% and 3.34% in 2 and 24 h, respectively, in the board made of 10% rice straw, 90% gypsum, and 3% BNCF. With the addition of bacterial nanocellulose fibers from 1 to 3%, the thickness swelling of the boards decreased. The is because of the higher density of the bacterial nanocellulose fibers. Because the density of nanocellulose is higher than gypsum, and in this case, with the increase of nanocellulose, the density of the board increases, and the thickness swelling decreases.
3. The addition of BNCF resulted in a significant decrease in the fire-retardant properties of the board. The reduced thermal conductivity of nanocellulose led to lower heat transfer, thereby diminishing the fire resistance of the gypsum composite boards. In contrast, the increase in rice straw content within the boards led to a reduction in overall

board density, which resulted in increased void formation. This phenomenon primarily contributed to the higher mass loss observed in the boards.

4. Finally, the composite board made of rice straw and gypsum using bacterial cellulose nanofibers can be used in indoor applications because it complies with DIN, EN and ISO standards according to the results obtained.
5. Ultimately, it is anticipated that the incorporation of a reinforcement into a composite will yield substantial enhancements, particularly with regard to the modulus. By leveraging agricultural waste and combining it with mineral materials such as cement and plaster, this approach can significantly contribute to the sustainability of forest resources and building applications. Notably, the present study demonstrates that even a modest addition of 1 to 3% bacterial cellulose nanofibers can have a profound impact on enhancing mechanical resistance, which can be highly valuable in practical applications.

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