Research on the fabrication of composite materials based on unsaturated polyester resin matrix and modified rice husk reinforcement

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ABSTRACT

In this study, rice husks (RHs) were utilized as fillers to create a reinforcing phase during the fabrication of composites based on an unsaturated polyester (UPE) resin matrix. The aim was to maintain the mechanical properties of the materials at a certain level and partially replace the amount of thermosetting resin used, thereby reducing negative environmental impacts. Sodium hydroxide (NaOH) and 3-methacryloxypropyltrimethoxysilane (MPS) were employed in the modification process of the RHs. The structure and morphology of the modified RHs were investigated using Fourier-transform infrared (FTIR) spectroscopy and field emission scanning electron microscopy (FESEM). Additionally, the mechanical properties of composite samples were evaluated according to international standards, such as ISO 527:2012, ISO 178:2019, ISO 604:2002, and ASTM D256. The results showed that the RHs sequentially modified by a NaOH solution and a MPS-dissolved mixture significantly improved the mechanical properties of the respective composites, compared to the composites with untreated RHs and RHs chemically modified by one solution. Specifically, compared to the reference samples formed by raw RHs, the best composite among ones with two-stage-treated RHs demonstrated flexural strength with an increase of 34.1% (reaching 45.1 MPa), compressive strength with a climb of 51.6% (reaching 33.8 MPa), tensile strength rising by 44.6% (at 34.3 MPa), and impact strength of 6.3 kJ/m² (18.9% higher). These results indicate that modified RHs can meet the research objectives and pave the way for further studies on RH-based composites.

Keywords: Rice husk; Natural fiber; UPE composite; 3-methacryloxypropyltrimethoxysilane; Mechanical properties.

1. INTRODUCTION

The potential of agricultural waste as raw material for composite production is significant and remains underutilized. In 2020, Vietnam's rice production reached 42.69 million tons, with RHs accounting for approximately 20–33% of the mass after milling [1]. Approximately 9 million tons of RHs are generated annually, with most being discarded into the environment. Only a small fraction of these husks are utilized for producing charcoal, activated carbon, or as fillers in construction materials. The majority of RHs are directly burned in fields, leading to air pollution and resulting in serious health and environmental issues [2, 3]. Therefore, utilizing the abundant and inexpensive RH waste for processing and production activities not only generates economic value but also reduces the environmental impacts associated with improper disposal of excess RHs.

RHs and natural fiber reinforcements, in general, exhibit the drawback of incompatibility with polymer matrices. This issue arises from the presence of lignin, wax, and crude fats in natural fibers, which impede the bonding between the matrix and the reinforcement. Appropriate treatments are required to remove these components from the surface of natural fiber reinforcements. Among these treatments, alkaline treatment is the most commonly used. Numerous studies have demonstrated that alkaline treatment alters and disrupts the hydrogen bonds in the cellulose structure, thereby increasing the roughness and surface area of the material.

Additionally, alkaline treatment helps to remove some of the lignin, hemicellulose, wax, and fats within the RH structure. The removal of these amorphous components is highly beneficial, as it reduces the surface tension of the RHs and enhances the interaction between the husks and the matrix. This improvement leads to increased Van der Waals forces and enhanced mechanical properties of the composite product [4-7]. RH, being the outer layer of rice, has a unique structure. After milling, RHs resemble small boats, which can make it challenging for them to be fully covered by the resin, potentially leading to the formation of bubbles in the final product. Therefore, grinding the husks into finer particles is necessary to ensure better coverage by the resin. Studies on the modification of RHs with alkaline treatments have investigated parameters such as NaOH concentration, reaction time, and reaction temperature. A 5% NaOH treatment at room temperature for 1 hour is commonly used due to its simplicity, as it does not require temperature control, offers a quick reaction time, and has a NaOH concentration that is suitable for modifying the husks without disrupting the cellulose bonds [7-10]. After modification, RHs are combined with various polymer matrices to create composite materials. These matrices can be either thermoplastic or thermosetting resins. However, thermoplastic resins are often less favored due to their inferior mechanical and thermal properties. Among them, polylactic acid is of particular interest due to its biodegradable nature, which has led to increased research focus on its application [7].

In addition to NaOH, silane-based compounds have been explored for their ability to modify natural fibers, showing some promising outcomes [11-13]. NaOH is considered an effective modifier for natural fibers; however, it does not alter the nature of the bonding between the matrix and the reinforcement, which remains physical (Van der Waals) bonding. In contrast, silane-based compounds have the potential to transform these interactions into covalent (chemical) bonds. Additionally, silanes improve the bonding between the reinforcement fibers and the matrix by increasing crosslinking levels and the surface area of the fibers. Among silane compounds, those with vinylsilane groups have been shown to be the most effective in bonding with unsaturated polyester (UPE) resin matrices [13-16]. Among these, 3-methacryloxypropyltrimethoxysilane (MPS) is considered the most suitable compound for use with UPE. The tensile strength and tensile modulus of fique fiber/UPE composites increase by up to 60% and 80%, respectively, when the fibers are treated with MPS [17]. The flexural strength of sisal/UPE composites can increase by up to 63% [18]. Dynamic Mechanical Analysis (DMA) indicates that treating banana fibers with MPS enhances the dynamic storage modulus, suggesting improved adhesion between the banana fibers and the polyester matrix [19, 20].

In this study, MPS is applied for the first time as a modifying agent for RHs to fabricate UPE-based composite materials. The enhancement of the interaction between RHs and the polyester matrix is achieved through the modification of the husks. The RHs were treated using three surface treatment methods: NaOH treatment, MPS treatment, and a combination of NaOH and MPS. The effects of these modifiers on the structure and morphology of the RHs were investigated before analyzing the mechanical properties of the composite materials. The chemical functional groups of the materials were characterized using Fourier-transform infrared spectroscopy (FTIR), while the structure and morphology of the RH samples were observed using scanning electron microscopy (FESEM). For the composite samples, mechanical properties were evaluated according to international standards (tensile strength ISO 527:2012, flexural strength ISO 178:2019, compressive strength ISO 604:2002, and impact strength Izod ASTM D256).

2. EXPERIMENT

2.1. Materials

UPE resin 2117 with a viscosity of 400-800 cps at 25 °C was obtained from China. Methyl ethyl ketone peroxide (MEKP), used as a hardener, was also procured from China. RHs, sourced from Ben Tre, Vietnam, were ground into a fine powder by a 3A608TM machine. Sodium

hydroxide (NaOH), in solid form from Xilong Scientific (China), was employed for chemical treatment. Additionally, MPS, commercially known as KH-570 and sourced from China, was used for further modification. Ethanol (99.5%), anti-stick wax (trade name Wax 8), and acetic acid were provided by Xilong Scientific (China).

2.2. Experimental

2.2.1. Research methodologies

Tensile strength was determined according to ISO 527 using a YM-H02 testing machine (Taiwan) with a pulling speed of 2 mm/min. Compressive strength was measured following ISO 604 standards using the Lloyd LR 30K machine (USA) with a speed of 2 mm/min. Flexural strength was assessed according to ISO 178 using the YM-C01 device (Taiwan) with a testing speed of 1.50 mm/min. Impact strength was evaluated according to ASTM D256 using the YM-J0301 machine (Taiwan). For morphological analysis, a TESCAN Mira 4 FESEM (Switzerland) was employed, operating at an accelerating voltage of 10 kV. FTIR spectra ranging from 500 to 4000 cm^{-1} were obtained using the Platinum ATR Alpha II spectrometer (Bruker, Germany).

2.2.2. Modification of RH

Preliminary treatment: The RH samples were initially ground using a 3A608TM mill to achieve a particle size of less than 0.5 mm. Subsequently, the ground RHs were washed with water three times to eliminate residual impurities. The cleaned husks were then dried at 60 °C for 12 hours. Such untreated RHs were marked as sample U.

Alkaline modification: The alkaline modification process was developed based on previous research on the modification of natural fibers [21-24]. 200 g RHs were initially immersed in a 5% NaOH solution at room temperature for 1 hour with vigorous stirring. The mass ratio of RHs to NaOH solution was 1:15. Following this, the treated RHs were removed, thoroughly rinsed with distilled water, and then neutralized with a few drops of acetic acid to counteract any residual NaOH. After neutralization, the husks were rinsed several additional times with distilled water. Then, RHs were then air-dried at room temperature for 48 hours, followed by drying in an oven at 60 °C for 12 hours. Such alkaline-treated RHs were marked as sample A.

Silane modification: A 5% solution of MPS was prepared in a mixed solvent of ethanol and water (volume ratio 1:1) and stirred for 3 hours. 200 g RHs were then added to the solution and stirred mechanically for 1 hour. The mass ratio of RHs to MPS solution was 1:15. After treatment, RHs were rinsed with distilled water, air-dried at room temperature for 48 hours, and then oven-dried at 60 °C for 12 hours. Such silane-treated RHs were marked as sample S.

Sequential modification (using both NaOH and MPS solutions): The ground RHs first underwent the alkaline modification mentioned above. Subsequently, such RHs experienced the described silane modification, but five different solutions with MPS concentrations of 1%, 2%, 3%, 4% and 5% were used. Such RH samples were denoted as AS1, AS2, AS3, AS4, and AS5, according to the MPS concentrations selected.

2.2.3. Fabrication of UPE-matrix composites reinforced with modified RH

To prepare the resin-RH composite, the required quantities of resin and RH were first measured using a plastic cup with 10% RH follow weight UPE. The mixture was then stirred using a mechanical stirrer for 40 minutes. Following this, the required amount of MEKP was measured and added to the resin-RH mixture with a pipette. The combined mixture was stirred for an additional 5 minutes. The mold must be thoroughly cleaned and wiped. A brush is then used to apply Wax 8 to the mold surfaces, creating a protective layer to prevent direct contact between the mold and the material. Next, the prepared resin-RH mixture is poured into the mold, and a glass stirrer is used to ensure the even distribution of the material within the mold. The mixture is allowed to rest for 30 minutes to facilitate partial curing of the resin. Once the surface of the resin

begins to solidify, a glass mold coated with Wax 8 is used to press down and smooth the composite surface. After 4 hours, the mold is carefully removed to extract the composite sample. The samples are then allowed to cure for an additional 7 days before being cut to the required dimensions for mechanical property testing according to standard procedures. The composite samples were named by adding "CP" before the code of the respective RH sample. They are CP-U, CP-A, CP-S, CP-AS1, CP-AS2, CP-AS3, CP-AS4 and CP-AS5.

3. RESULTS AND DISCUSSION

3.1. Functional group analysis

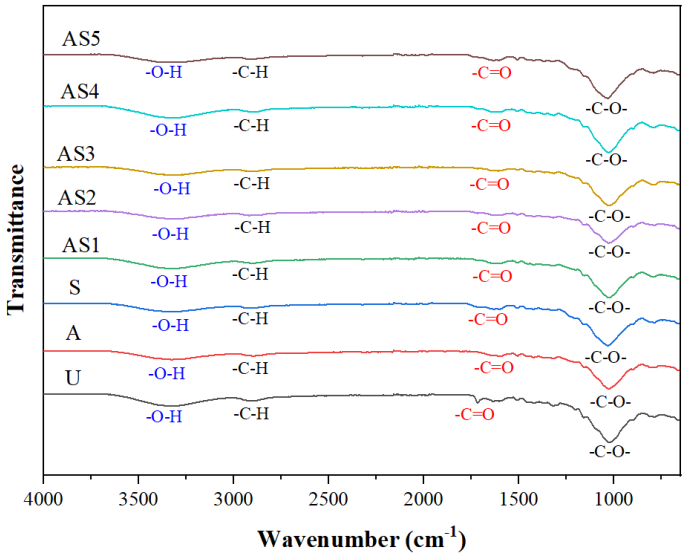

Figure 1. *FTIR analysis of RH samples.*

As shown in figure 1, a broad peak at 3340 cm^{-1} corresponds to the O-H stretching vibrations indicating the presence of hydrogen bonding. The peak at 2890 cm^{-1} is attributed to the C-H stretching vibrations of methyl and methylene groups. The peak at 1630 cm^{-1} represents the aromatic ring vibrations associated with lignin, while the peak at 1024 cm^{-1} corresponds to the asymmetric stretching vibrations of the C-O groups [25, 26].

Comparison between the untreated RH (sample U) and the NaOH-treated RH (sample A) reveals a noticeable decrease in the intensity of the peak at 1630 cm^{-1} [27], which is associated with lignin. This reduction indicates that NaOH treatment effectively removes a significant portion of lignin and non-polar impurities from the RH surface. This result supports the hypothesis that alkaline treatment cleanses the RH, enhancing its surface characteristics and potentially improving its compatibility with polymer matrices. The exposure of cellulose on the fiber surface due to the alkaline treatment increases the number of hydroxyl groups available for equation (1) [28].

$$NaOH + RH - OH \rightarrow Na - O - RH + H_2O \quad (1)$$

Comparing sample U with sample S reveals a reduction in the peak intensity at 3340 cm^{-1}, corresponding to the -OH groups. This decrease can be attributed to the reaction between silanol hydroxyl groups (Si-OH) and the hydroxyl groups of the RH (RH-OH), forming new -Si-O-C- linkages. This indicates that the modification of the RH with MPS has been successful.

During the hydrolysis process, alkoxy groups in silane compounds are converted into silanols according to equation (1). These silanols then react with the hydroxyl groups present on the RH fibers, forming covalent bonds between the husk and the silane according to equation (2) [11]. This reaction establishes strong covalent linkages, enhancing the adhesion and compatibility of the

RH with the polymer matrix.

$$R_1 - Si - (OR_2)_3 + 3\ H_2O \rightarrow R_1 - Si - (OH)_3 + 3\ R_2OH \quad (2)$$

$$R_1 - Si - (OH)_3 + RH - OH \rightarrow R_1 - Si(OH)_2 - O - RH + H_2O \quad (3)$$

The unsaturated groups presenting in MPS, upon reaction with peroxide, form chemical bonds with styrene. Such interactions create crosslinks between the RH and the UPE matrix, thereby establishing a robust connection and improving the overall adhesion and integration of the RH within the composite material [16].

Comparison between the untreated sample (U) and the samples treated with increasing concentrations of MPS (from 1% to 5%) reveals a decrease in the peak intensity at 3335.68 cm^{-1} in the treated samples (AS). This reduction is attributed to the reaction between the hydroxyl groups of silanol (Si-OH) and the hydroxyl groups of RH (RH-OH), resulting in the formation of new functional groups such as -Si-O-C-.

3.2. Structural morphology

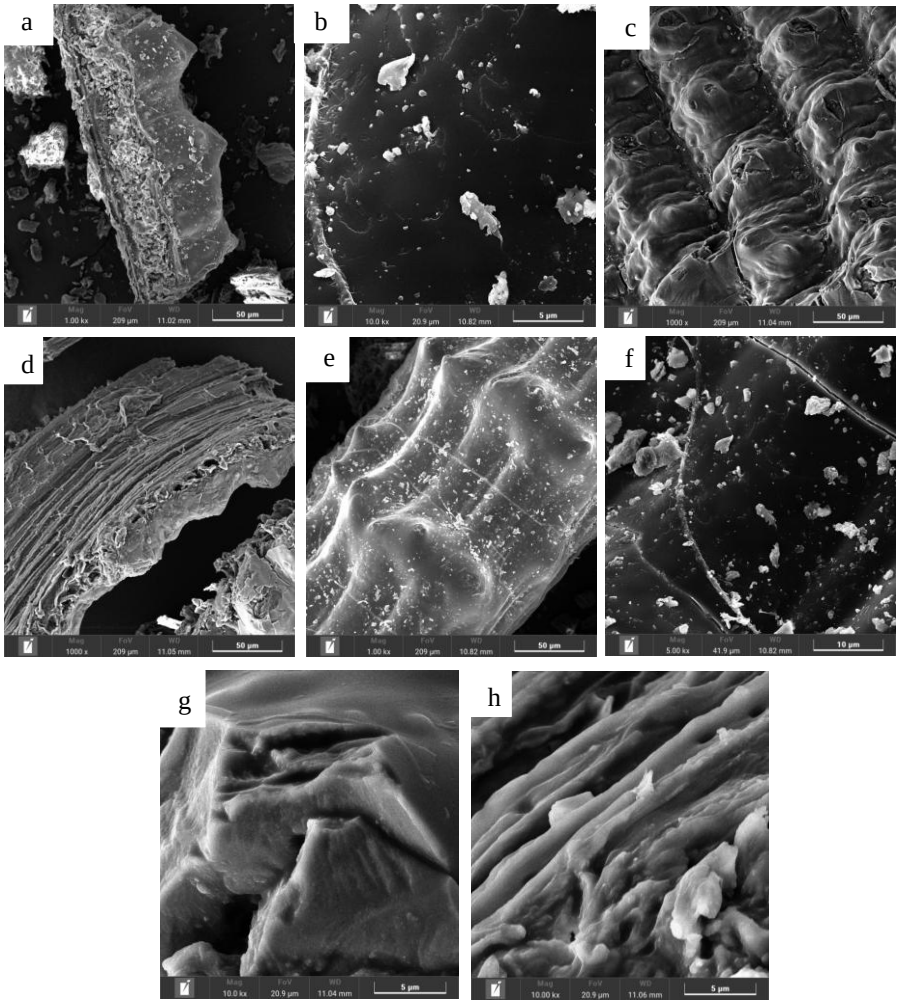

Figure 2. *FESEM images: untreated RH with (a) 1000x and (b) 10,000x magnification; (c) the outer surface and (d) inner structure of sample A; the surface structure of sample S with (e) 1,000x and (f) 5,000x magnification; (g) the outer surface and (h) the inner of sample AS5 with 10,000x magnification.*

The outer surface of the initial RHs (sample U) exhibits a pattern of ridges resembling mountainous terrain with protrusions, and the inner surface is relatively smooth. The cross-sectional thickness of the RH is approximately 40-50 μm (figures 2a and 2b), in accordance with the previous study [8].

The observed layers of lignin, wax, and grease in the RH contribute to binding cellulose and forming the husk's structure. However, this layer has a very low surface energy (hydrophobic nature). When in contact with UPE, this low-energy layer reduces the contact area between the RH and UPE, resulting in weaker Van Der Waals forces and consequently diminishing the mechanical properties of the composite material [5]. Additionally, this layer also covers the hydroxyl groups, hindering their ability to react with MPS.

The FESEM images indicate that NaOH effectively removes some lignin, wax, and other impurities from the surface of sample A (figures 2c and 2d). Firstly, it alters the husk's structure, increasing its surface area. Improved mechanical bonding between the husk and the matrix can be achieved with increased surface roughness or area.

The surface of sample S does not exhibit significant changes compared to untreated husks (figures 2e and 2f). This can be attributed to the fact that the 5% MPS solution does not possess the capability to dissolve contaminants such as lignin and wax on the surface of the RHs [13, 14, 16, 24]. The primary function of the 5% MPS treatment is to react with the hydroxyl groups on the RH surface, later forming chemical crosslinks with the UPE resin.

When RHs are treated using a combination of NaOH and 5% MPS, the surface of AS5 sample is quite similar to that observed with NaOH treatment alone. Impurities, dust, wax, and lignin are effectively removed. This combined treatment retains all the advantages of NaOH treatment. Additionally, the surface of the sample treated with both NaOH and MPS is smoother compared to that treated with NaOH alone (figures 2g and 2h). This can be attributed to the coating of the husk with MPS [6, 16].

3.3. Mechanical properties

All samples prepared for mechanical testing were cut and ground carefully to meet the standard requirements. In order to draw figure 3, each data point with an error bar was obtained by measuring 5 sample pieces of each composite.

The mechanical properties of CP-A composite (in which RHs were treated with NaOH only) exhibited notable improvements compared to those of CP-U samples. Specifically, the flexural strength increased from 33.6 MPa to 35.9 MPa, marking a 6.8% enhancement. The compressive strength saw a substantial rise from 22.3 MPa to 28.5 MPa, reflecting a 27.8% improvement. The tensile strength also improved significantly, increasing from 23.7 MPa to 30.7 MPa, which represents a 29.5% gain. Additionally, the impact strength increased from 5.3 kJ/m² to 5.6 kJ/m², indicating a 5.7% enhancement. The improvement in mechanical properties upon NaOH treatment is attributed to the disruption of hydrogen bonds within the cellulose network. This process results in increased surface roughness of the RH. The alkaline treatment also removes lignin, wax, and fats from the fiber surface, which directly impacts cellulose. It enhances both the amount of cellulose exposed on the fiber surface and the number of reactive sites available. The increased surface roughness induced by the alkaline treatment facilitates better mechanical interlocking [29]. Alkaline treatment is an effective and cost-efficient method for modifying the surface of natural fibers. This treatment results in changes to both the surface energy and the fiber morphology [30].

In comparison with CP-U samples, CP-S composite showed an increase in flexural strength from 33.6 MPa to 37.0 MPa, representing a 10.1% improvement. At the same time, the compressive strength of CP-S samples rose from 22.3 MPa to 29.1 MPa, a 30.5% increase, while the tensile strength enhanced from 23.7 MPa to 30.9 MPa, showing a 29.4% improvement (figure 3). Additionally, the impact strength of CP-S samples increased from 5.3 kJ/m² to 5.7 kJ/m²,

reflecting a 7.5% enhancement. In the presence of water, the alkoxy groups in silane undergo hydrolysis to form silanol groups. These silanol groups then react with the hydroxyl groups of cellulose, creating strong covalent bonds between the fibers and the silane. This reaction effectively reduces the number of surface hydroxyl groups on the cellulose fibers. The hydrocarbon chains of MPS on the fiber surface form a network of crosslinks with UPE. In addition to decreasing the surface hydroxyl groups of cellulose, MPS establishes chemical bridges between the RH and UPE, resulting in durable chemical bonds [31].

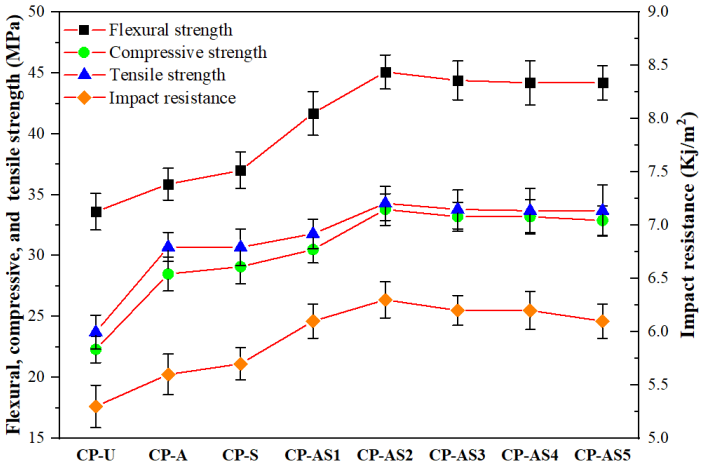

Figure 3. *The line graph with error bars illustrating the mechanical properties of UPE-matrix composites reinforced by different types of RHs.*

As for CP-AS composites (figure 3), the MPS concentration increasing from 1% to 2% leads to an enhancement in the mechanical properties. Specifically, for CP-AS2 samples compared to CP-AS1 samples, the flexural and compressive strength grow from 41.7 MPa to 45.1 MPa and from 30.5 MPa to 33.8 MPa, respectively. Similarly, the tensile and impact strength improve from 31.8 MPa to 34.3 MPa, and from 6.1 kJ/m² to 6.3 kJ/m², respectively. However, when the MPS concentration climbs further from 2% to 5%, the mechanical properties of respective composite materials (CP-AS3, CP-AS4 and CP-AS5) decline slightly. Specifically, their flexural and compressive strength drop slightly to 44.2 - 44.4 MPa and 32.9 - 33.2 MPa, respectively, whereas their tensile and impact strength reduces minorly in turn to 33.7 - 33.8 MPa and 6.1 - 6.2 kJ/m².

These positive results originate from the fact that the combination method integrates the advantages of both alkali and MPS treatments. Alkali treatment effectively removes lignin, wax, and fatty substances from the surface of the RH, which not only increases the surface roughness and expands the surface area but also exposes hydroxyl groups previously covered by wax and lignin, making them available for subsequent reaction with MPS. As a result, when treated with MPS, the number of hydroxyl groups significantly increases compared to untreated husk, leading to a higher number of covalent chemical bonds between the husk, MPS, and UPE. When the MPS concentration is increased from 1% to 2%, the mechanical properties improve markedly, implying that the bonding between the hydroxyl groups of the husk and MPS is strengthened. However, as the MPS concentration continues to rise to 5%, the mechanical properties insignificantly decrease. This reduction is mainly due to the limited number of hydroxyl groups on the husk, which can react with only a certain amount of MPS. In other words, treating RHs with higher MPS concentration (beyond 2%) does not improve the mechanical properties of resulting composites.

4. CONCLUSIONS

In this study, various chemical treatment methods for RHs were investigated, specifically alkali

treatment, MPS treatment, and a sequential combination of the two treatments. FESEM analysis revealed a roughened surface structure of the fibers after multiple chemical treatments. This roughened surface was effective in enhancing the mechanical bonding between the fibers and the resin, which subsequently improved the mechanical properties of the composite material. The combination of alkali and MPS treatments demonstrated superior performance compared to treatments with either alkali or silane alone. The study also showed that CP-AS2 composite treated with alkali and a 2% MPS solution exhibited the best mechanical properties. Specifically, the flexural strength and compressive strength of CP-AS2 reached 45.1 MPa and 33.8 MPa, compared to that of the U sample with only 33.6 MPa and 22.3 MPa, respectively. Similarly, the tensile strength and impact resistance of CP-AS2 improved and reached 44.6 MPa and 6.3 kJ/m², compared to that of the U sample with only 29.5 MPa and 6.3 kJ/m².

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TÓM TẮT

Nghiên cứu chế tạo vật liệu composite trên cơ sở nền nhựa polyester không no, cốt vỏ trấu biến tính

Trong nghiên cứu này, vỏ trấu (RHs) đã được sử dụng làm chất độn để tạo pha gia cường trong quá trình chế tạo vật liệu composite nền polyeste không no (UPE). Mục tiêu là duy trì cơ tính vật liệu ở một mức độ nhất định và thay thế một phần lượng nhựa nhiệt rắn sử dụng để hạn chế tác động tiêu cực đến môi trường. Trong đó, sodium hidroxide (NaOH) và 3-methacryloxypropyl trimethoxy silane (MPS) được sử dụng trong quá trình biến tính RHs. Cấu trúc và hình thái của RHs biến tính được khảo sát bằng quang phổ hồng ngoại biến đổi Fourier (FTIR) và kính hiển vi điện tử quét (FESEM). Bên cạnh đó, tính chất cơ học của các mẫu composite được đo theo các tiêu chuẩn quốc tế như ISO 527:-2012, ISO 178:2019, ISO 604:2002 và ASTM D256. Kết quả cho thấy, RHs biến tính lần lượt bằng dung dịch NaOH và dung dịch MPS đã cải thiện đáng kể các tính chất cơ lý của mẫu composite tương ứng. Cụ thể, so với mẫu tham chiếu tạo thành từ RHs thô, mẫu tốt nhất (trong số các composite với RHs được biến tính hai giai đoạn) đạt độ bền uốn tăng khoảng 34.1% (đạt 45.1 MPa), độ bền nén tăng khoảng 51.6% (đạt 33.8 MPa), độ bền kéo tăng khoảng 44.6% (đạt 34.3 MPa), độ bền va đập là 6.3 Kj/m^2 (cao hơn 18.9%). Các kết quả này cho thấy, RHs biến tính có thể đáp ứng các mục tiêu nghiên cứu đã đề ra, đồng thời tạo tiền đề cho các nghiên cứu tiếp theo về composite kết hợp vỏ trấu.

Từ khoá: Vỏ trấu; Sợi thiên nhiên; UPE composite; 3-methacryloxypropyl trimethoxy silane; Cơ tính.