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Effects of compressing parameters and $Mg(OH)_2$ content on mechanical properties and flame retardancy of rice straw-fibre reinforced composites

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Abstract:

Rice straw fibre was utilized for unidirectional (UD) composites. In this study, the effects of compression temperature, duration, pressure, and fibre volume fraction on the mechanical properties of composites were investigated, respectively. The composite with optimal mechanical properties was prepared at a temperature of 180°C, pressure of 125 kg.cm^{-2} for 10 min, and at a fibre volume fraction of 40%. $Mg(OH)_2$ was found to be an appropriate additive to enhance the flame retardancy of the composite. Interestingly, this agent also improved the mechanical and thermal insulation properties of the obtained composite.

Keywords: **flame retardancy, poly(vinyl chloride), rice straw, thermal insulation.**

Classification number: **2.3**

Introduction

Rice straw is a typical agricultural residue that represents about 45% of the rice production volume [1], and is abundant in rice-growing countries such as India and Vietnam. Although rice straw possesses the ability to become a value-added by-product [2], it has been poorly treated. Traditionally, rice straw can be utilized as an animal feed or fuel source, but it is mainly incinerated and discarded back to the field resulting in not only a waste of resources but also environmental pollution [3]. Therefore, an appropriate method to utilize rice straw is needed in order to prevent this seasonal environmental pollution. In recent years, plenty of studies have been conducted in order to put rice straw in use. In particular, rice straw has been considered as a promising source to produce bioethanol via fermentation [4], agriculture compost [5], biodegradable fibre, or mostly as a composite reinforcement [6]. However, most of the aforementioned studies require rice straw to be chemically modified, which is expensive, time consuming, and may be harmful to the environment due to the use of chemicals.

In this study, rice straw fibres were used not only as fillers but also reinforcement materials for a poly(vinyl chloride) (PVC) matrix. Moreover, the high silica content and porous structure of rice straw fibres are considered to be an advantage aiming to improve the flame retardancy of the material. Among other kinds of polymers, PVC was chosen based on its unique ability to be processed at low temperatures. Besides, the significant amount of chlorine in PVC can retard fires from both starting and spreading [3]. To further increase the flame retardancy of the composite, $Mg(OH)_2$ was added to the components. Among the other inorganic flame retardants, $Mg(OH)_2$ was chosen due to the ability to endothermically decompose into MgO and release water vapor [7]. As the heat is absorbed by the decomposition reaction, the flame ignition of the associated components is delayed. At the same time, MgO acts as a barrier to prevent further interaction between the combustible components and oxygen. Besides, the released vapor water can not only cool down the combustion area but also partially dilute the combustible gases. Moreover, the flame retardant additive $Mg(OH)_2$ can be guaranteed to withstand the composite fabrication process since it starts to decompose at around 300-340°C. After the composites were produced, their stability, mechanical and thermal properties, and flammability were investigated for selective formulation purposes.

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Materials and methods

Materials

Rice straw was obtained directly from a field in the Hong Dan district, Bac Lieu province. $Mg(OH)_2$ was purchased from the CEMACO company (Vietnam). Commercial PVC powder was provided by the TPC Vina company with properties as shown in Table 1.

Table 1. Properties of PVC powder.

Property	Value
Density	1.45-1.50 g.cm^{-3}
Tensile strength	500-700 kg.cm^{-2}
Flexural strength	800-1,200 kg.cm^{-2}
Compressive strength	800-1,600 kg.cm^{-2}
Modulus of Elasticity	4,000-10,000 kg.cm^{-2}
Elongation at break	10-25%
Coefficient of linear expansion	0.00006-0.00007
Thermal conductivity	3.8-4*10^{-4} cal.(cm.s.°C)$^{-1}$

Rice straw mat preparation

After collecting raw rice straw from the field, the flower and leaf were removed and only the body was used. The body was then cleaned with water, oven-dried to expel moisture, disentangled, and cut into pieces 3-4 cm in length. Finally, a mould of evenly distributed rice straw was hot compressed by using a hydraulic press machine (PAN STONE P-100-PCD, Taiwan) under different conditions to form unidirectional fibre mats. The optimum conditions for fibre mat preparation were at a temperature of 130^{o}C and a pressure of 50 kg.cm^{-2} for 60 s. Under the mentioned conditions, lignin plays the role of adhesive in the rice straw-fibre mat, which is called a preform.

PVC film preparation

To investigate the optimum condition to prepare the PVC film, PVC powder was hot compressed under various conditions by using a hydraulic press machine (PAN STONE P-100-PCD, Taiwan) as well. The optimum conditions for PVC film preparation were carried out at 180^{o}C and a pressure of 100 kg.cm^{-2} for 30 s. After that, in order to increase the flame retardancy of the material, a film comprising PVC and $Mg(OH)_2$ with various ratios was fabricated by following the optimum condition.

Composite preparation

To prepare the rice straw composite, the prepreg was firstly prepared by compressing one layer of rice straw mat sandwiched by two layers of PVC film. Then, the final composite was made by pressing the prepregs between the hot plates, followed by decreasing the pressure to cool down the composite. Finally, all of the excess amounts of rice straw were removed from the composite. In this study, the effects of compressing temperature, duration, pressure, fibre ratio, and $Mg(OH)_2$ content on the mechanical properties of the obtained composites were successively investigated. Moreover, the effects of $Mg(OH)_2$ content on thermal properties and flammability were studied.

Measurements of mechanical properties

Tensile strength: tensile strength property was determined by using the Zwick/Roell BDO - FB050TN testing machine following the ASTM D638-04 standard, in which the rectangle specimens of 115x15x3 mm were used, and the number of tests was five.

Flexural strength: flexural strength measurements have been carried out by using the Zwick/Roell BDO - FB050TN testing machine in accordance with ASTM D790M/84. The total number of tests was five and the used sample dimensions were 45x15x3 mm.

Impact strength: impact strength measurements were conducted by using the Zwick/Roell BPI – 50COMC testing machine according to ASTM D 256-04 standard. Five replicates were carried out with a sample size of 65x15x3 mm and the 45° V-notch located at the position of 20% width.

Thermal insulation test: the thermal insulation test was conducted in the system consisting of two chambers, as shown in Fig. 1. The sample is placed in between two chambers. When heat is continuously provided into chamber 1 by the heat source, the temperature differential is determined by measuring the temperature at chamber 1 and chamber 2 for different periods.

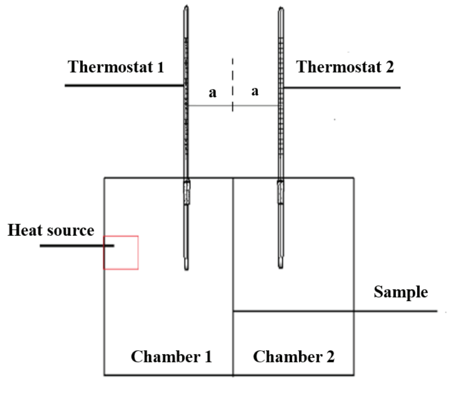

Fig. 1. Thermal insulation test setting.

Combustion test: the combustion tests were performed with respect to HB, HB45, HB75 standards (Fig. 2A), V0, V1, V2 standards (Fig. 2B), and 5VA, 5VB standards (Fig. 2C).

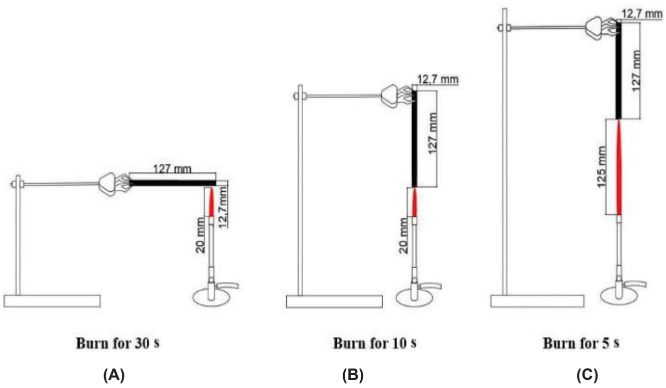

Fig. 2. Flame retardancy tests regarding (A) HB, HB45, HB75 standards, (B) V0, V1, V2 standards, and (C) 5VA, 5VB standards.

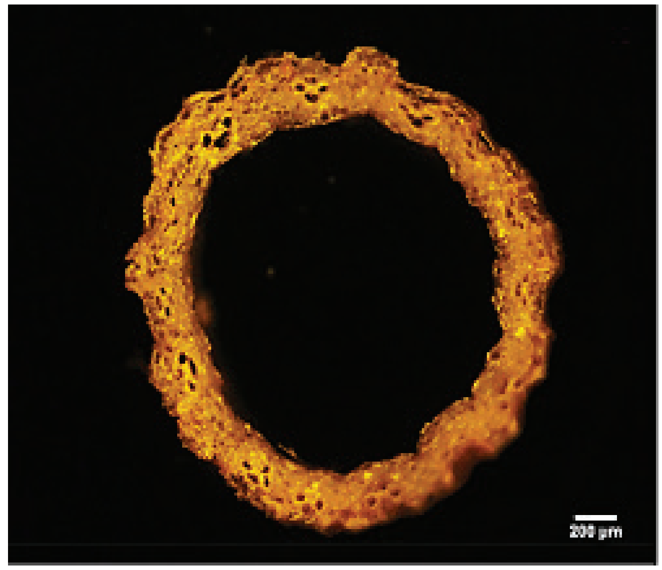

Fig. 3. All-in-focus image of rice straw with porous structure.

Results and discussion

Structure of extracted rice straw fibres

By using SEM and chemical composition determination, rice straw fibres, as well as other lignocellulosic fibres, are found to be mainly constituted of cellulose, hemicellulose, and lignin [6, 8, 9]. As a microscopic object can be better visualized by its best-focused image [10] or all-in-focused image [11], the obtained rice straw fibres were imaged by using an optical microscope (Nikon, ECLIPSE LV100POL, Japan) that was equipped with an autofocusing module [12] to create the best-focused image for better visibility of the fibre (Fig. 3).

Effects of compressing temperature on the mechanical properties of composite

To evaluate the effects of compressing temperature on the formation of the rice straw composite, mechanical tests (e.g., tensile strength, flexural strength, and impact strength) for the compression temperature variations (from 150 to 190^{o}C) were conducted while the compression duration, pressure, and rice straw ratio were fixed at 20 min, 100 kg.cm^{-2}, and 50%, respectively. However, the composite at a pressing temperature of 150^{o}C was separated into layers and the one at a pressing temperature of 190^{o}C was burned, so those samples were unable to be tested.

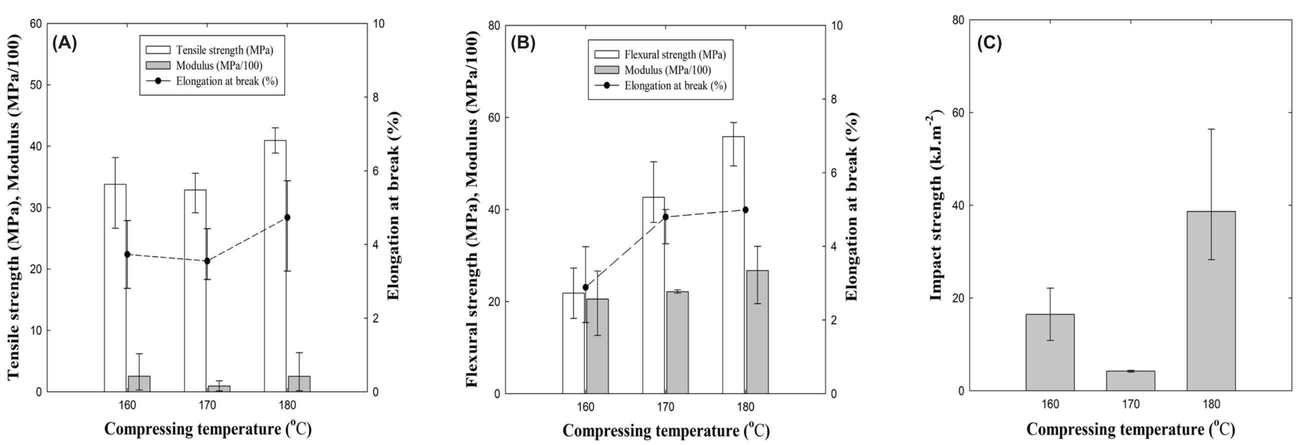

Fig. 4. Effect of compressing temperature on (A) tensile strength, (B) flexural strength, and (C) impact strength of rice straw composite.

The effects of the compression temperature on the tensile strength, flexural strength, and impact strength are shown in Fig. 4. There was a common trend observed of the mechanical properties of the rice straw composite regarding the pressing temperature. Specifically, as the pressing temperature increased, the mechanical strengths also increased, as well as the elastic moduli and the elongation at break. However, the tensile strength and the impact strength fluctuated as the pressing temperature went up, which is likely caused by the poor dispersion of the rice straw in the composite. The results show that the tensile strength, which represents the strength of the material, sharply rose approximately by 17.5% when the temperature was increased from 160°C (σ=33.79x10^{-2} MPa) to 180°C (σ=40.39x10^{-2} MPa). Also, the elongation at break, which represents the ductility of the material, expanded from 3.73 to 4.73% while the elastic modulus remained unchanged. In case of the flexural strength, which is defined as the maximum amount of force a material can bear without breaking or permanently deforming, as the pressing temperature increased, the flexural strength and elongation at break values significantly increased to approximately 61 and 42%, respectively. Besides, the elastic modulus was slightly changed from 20.56x10^{-2} MPa at 160°C to 26.74x10^{-2} MPa at 180°C. The rise of the tensile and flexural strength indicates that the interfacial adhesion between the rice straw mat and PVC increases with the increment in the compressing temperature. However, the impact strength, which corresponds to the capability of the material to resist a sudden stress or force, first declined as the compression temperature was raised from 160 to 170°C. Then, it bounced back to peak at 180°C (E=38.66 kJ.m^{-2}). This behaviour could be explained by the fact that at the low compression temperature of 160°C, the PVC was not completely melted resulting in plastic lumps in the composite. When the compression temperature was raised to 170°C, the polymer was completely melted but did not fully interact with the rice straw mat. Hence, the impact strength fell off. Eventually, 180°C was chosen as an adequate compression temperature to fabricate the composite. At this temperature, the heat effectively melted the PVC but did not burn the fibre leading to an increment in the interfacial adhesion between the rice straw mat and PVC.

Effects of compression duration on the mechanical properties of composite

In an attempt to analyse the effect of the compression duration on the rice straw composite formation, the composites were prepared with the compression duration varies from 2 to 25 min, while the pressing temperature, pressure, and rice straw ratio were fixed at 180°C, 100 kg.cm^{-2}, and 50%, respectively. However, as the pressing duration reached 25 min, the composite was burned. Therefore, the mechanical strength tests were conducted for the composites which were pressed for 2 to 20 min.

Figure 5 illustrates the mechanical strength (e.g., tensile strength, flexural strength, and impact strength) of the rice straw composite subjected to different compression durations. While the tensile strength, flexural strength, elastic moduli, and the elongation at break show the same declining tendencies; the impact strength demonstrated the opposite trend in which the impact strength increased when the compression duration rose. As the compression duration increased from 2 to 20 min, both the tensile strength and flexural strength showed similar trends in which these were increased (2 to 5 min) then were strongly declined to 30.46 and

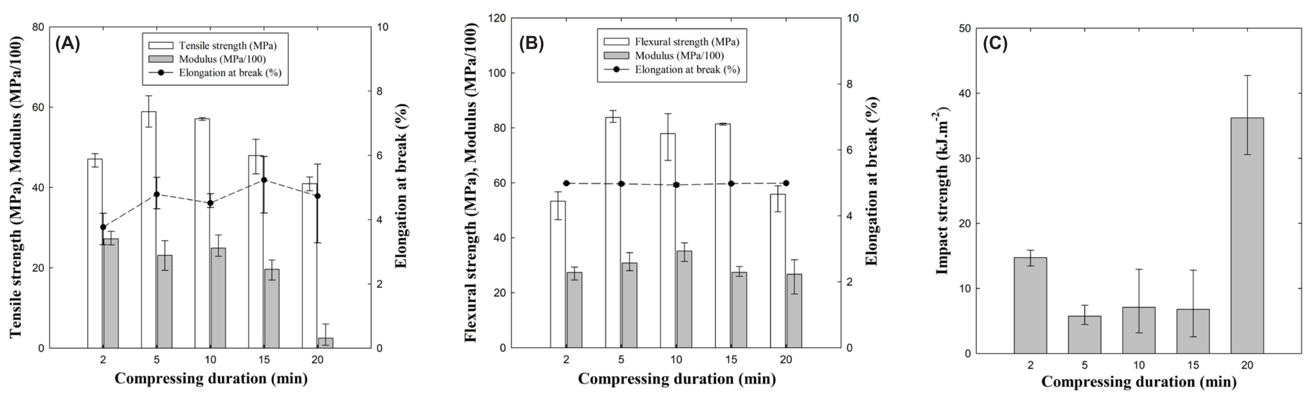

Fig. 5. Effect of pressing duration on (A) tensile strength, (B) flexural strength, and (C) impact strength of rice straw composite.

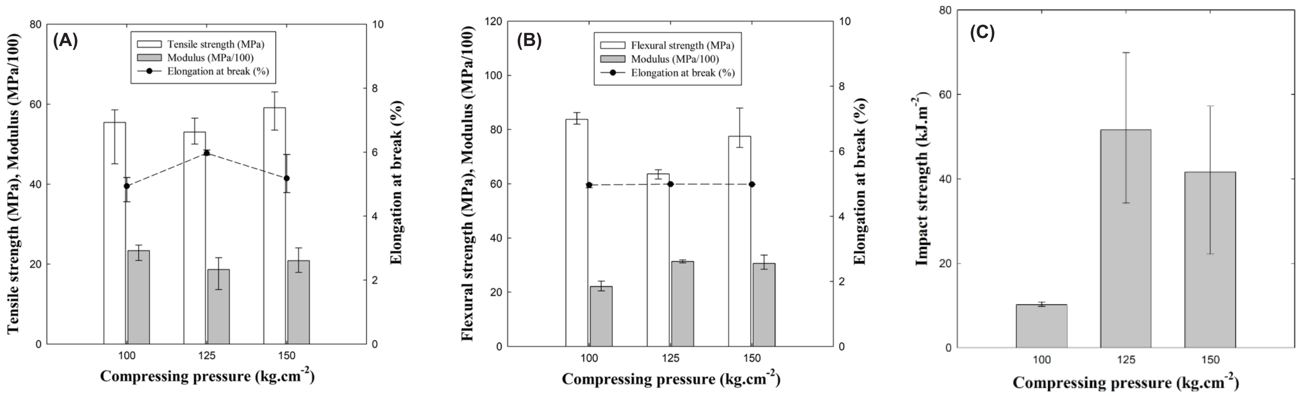

Fig. 6. Effect of compression pressure on (A) tensile strength, (B) flexural strength, and (C) impact strength of rice straw composite.

33.37%, respectively. In addition, the elastic modulus for both the tensile strength and flexural strength peaked at a compression duration of 10 min, after that the elastic moduli started to decrease. This behaviour implies that 10 min is a sufficient compression duration for the rice straw composite fabrication since below which the PVC was not sufficiently melted resulting in an enhancement of the interfacial adhesion between PVC and the fibres and above which the rice straw started to deform. In contrast to the tensile and flexural strength, the value for impact strength peaked at 20 min compression time. At which, the matrix of PVC and fibres was the strongest. However, as the composite was targeted to be used as a construction material in which the elastic moduli of the tensile strength and the flexural strength were prioritized over the impact strength, 10 min was chosen as an adequate compression duration. At this compression duration, the composite possessed the highest flexibility and sufficient hardness.

Effects of compression pressure on the mechanical properties of composite

The effect of compression pressure on the preparation of the rice straw composite was evaluated by fixing the pressing conditions at 180°C for 20 min and rice straw ratio at 50% with the variation of compression pressure ranging from 50 to 150 kg.cm^{-2}. Nevertheless, the composites were separated into layers when the compression pressure below 75 kg.cm^{-2} was applied, and those samples were unable to be tested. The effect of compression pressure on the mechanical properties of the rice straw composite can be seen in Fig. 6. As can be seen in the figure, there is almost no distinct difference in tensile strength when the compression pressure was increased. Nevertheless, at the compression pressure of 125 kg.cm^{-2}, the material possessed the highest values for both impact and flexural strength modulus indicating sufficient interfacial adhesion between PVC and the rice straw. When a stronger compression pressure was applied (e.g., 150 kg.cm^{-2}), the rice straw tended to displace to the sides leading to a slight fall in impact strength. In addition, since the material was targeted to be used as a construction material for ceilings, the flexural strength modulus was the key factor of material and thus the compression pressure of 125 kg.cm^{-2} was chosen as the optimum condition. At this pressure, the consumed energy is lower than at a compression pressure at 150 kg.cm^{-2} but the material still possessed adequate mechanical properties.

Effects of rice straw fibre on the mechanical properties of composite

As the compression conditions were determined, the effect of the rice straw ratio on the mechanical properties of the composite was measured with a variety of rice straw ratios (20 to 60%). However, when the rice straw ratio was 60%, the amount of polymer (PVC) was not sufficient to create the composite. Therefore, the sample with the rice straw ratio of 60% was unqualified to be tested.

Figure 7 shows the effect of rice straw ratio on the tensile, flexural, and impact strength of the composite. Predictably, adding rice straw into the formulation significantly influenced the mechanical strength of the composite. As the former ratio rose, the latter proportionally declined. Importantly, when the rice straw ratio was increased from 40 to 50%, the elongation at break values for both tensile strength and flexural strength sharply decreased emphasizing a reduction in the ductility of the material. In the case of impact strength, at low rice

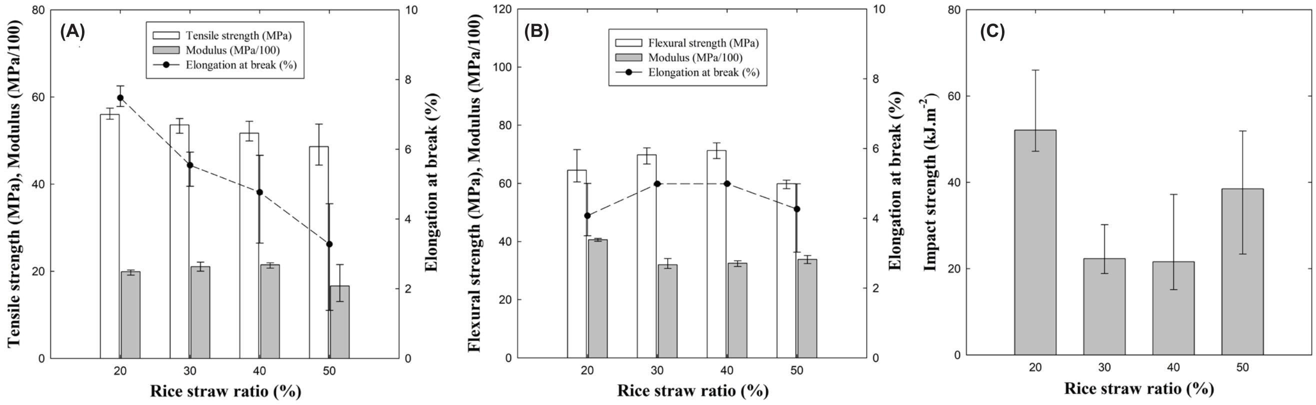

Fig. 7. Effect of rice straw ratio on (A) tensile strength, (B) flexural strength, and (C) impact strength of rice straw composite.

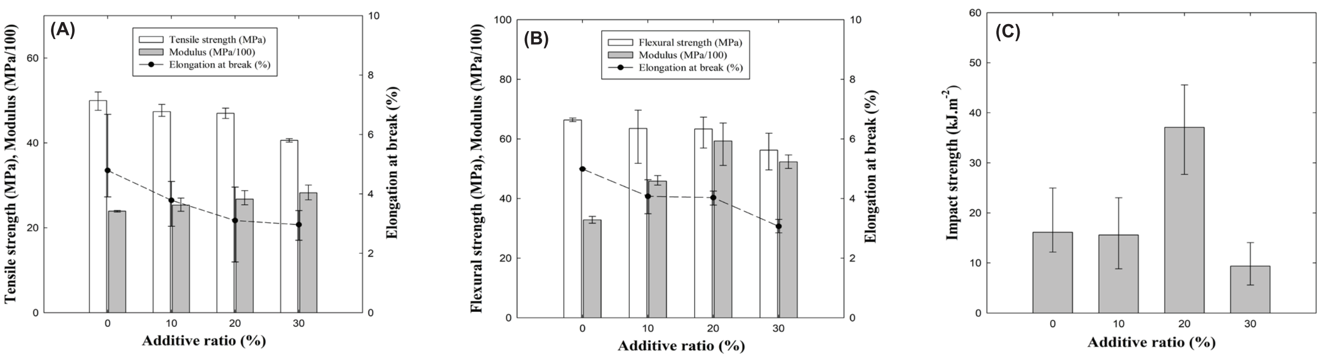

Fig. 8. Effect of additive on (A) tensile strength, (B) flexural strength, and (C) impact strength of rice straw composite.

straw content (e.g., 20%), PVC may have completely filled in the voids between the fibres and formed a homogeneous matrix. This matrix was weakened as the rice straw content increased. However, as the ratio of rice straw reached 50%, the value of impact strength rose again as the fibre became dominant in the composite. Eventually, a rice straw ratio of 40% was chosen to fabricate the composite since that composite consisted of the largest amount of rice straw while still meeting the mechanical standards for use as a construction material.

Effects of additive on the mechanical properties of composite

To analyse the effect of the addition of an additive on the rice straw composite formation, which is used to enhance the flame retardancy of the composite, the composites with a rice straw ratio of 40% were prepared at 180°C and 125 kg.cm^{-2} for 10 min with amounts of $Mg(OH)_2$ additive varying from 0 to 30 wt%.

In order to improve the flame retardancy ability of the composite, $Mg(OH)_2$ was added to the composite fabrication process. The effect of the flame-retardant additive on the mechanical properties of the composite can be seen in Fig. 8. When the additive powder was added into the mixture, it could fill into the voids created by the rice straw and PVC leading to an increment in the tensile and flexural strength moduli, as well as the impact strength. In particular, at a ratio of 20% additive, the impact strength reached its highest value of 31.1 kJ.m^{-2}, which is approximately 2.5 and 4 times higher than that value at 10% and 30% additive, respectively. When the additive content was increased further to 30% in PVC, the material started to become weak as the excess amount of $Mg(OH)_2$ hindered the interaction between the PVC and rice straw. Therefore, 20% of $Mg(OH)_2$ in PVC was chosen as the optimum ratio to be added to the rice straw composite.

Thermal insulation ability

Figure 9 reveals the thermal insulation ability of the rice straw composite. Clearly, the composite shows the ability to insulate heat even in the absence of a flame retardant additive (e.g. $Mg(OH)_2$). When heat is continuously provided to the system, the heat is immediately transferred from chamber 1 to chamber 2

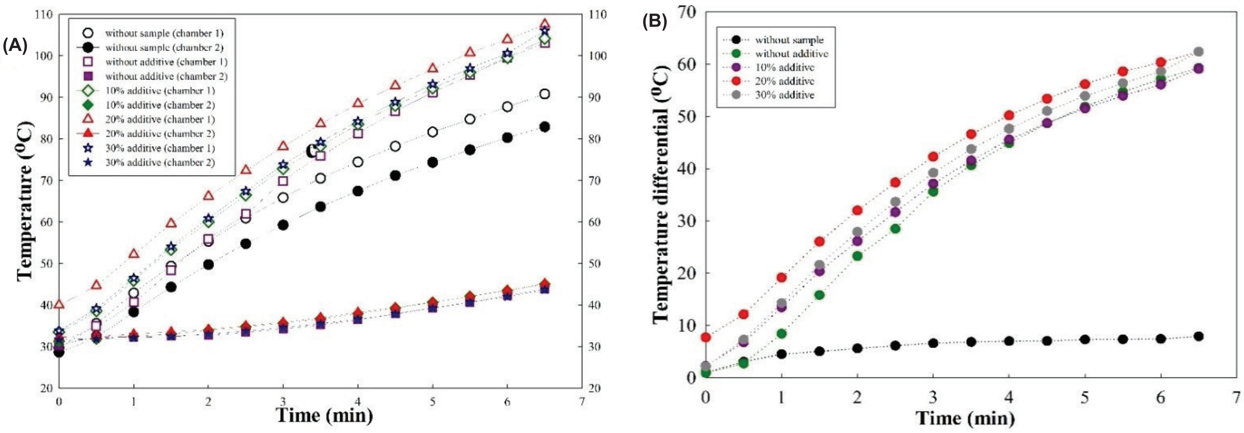

Fig. 9. Thermal insulation ability of the rice straw composite.

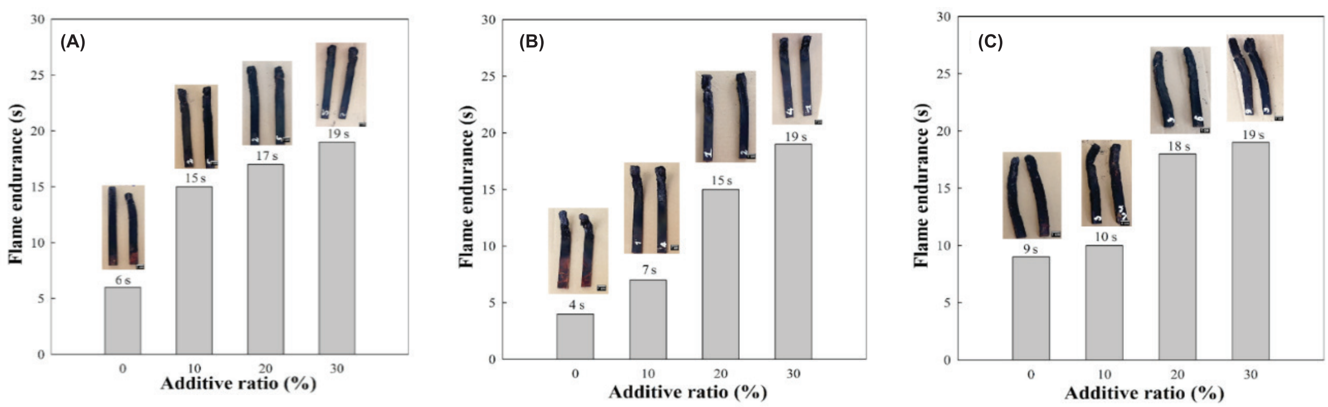

Fig. 10. Combustion tests regarding to (A) HB, HB45, HB75, (B) V0, V1, V2, and (C) 5VA, 5VB standards.

without any obstacles so that the temperature differential between the two chambers was negligible. When a rice straw composite was placed in between chamber 1 and chamber 2, the heat at chamber 1 is insulated leading to a noticeable temperature differential. Consequently, the thermal insulation ability of the rice straw is proven. Moreover, when a flame retardant chemical (e.g. $Mg(OH)_2$) was incorporated into the formulation, the ability of the rice straw composite to insulate heat improved further. It is worth noting that the ratio of 20 wt% $Mg(OH)_2$ in PVC shows the best result, which corresponds to the mechanical properties.

Flame retardancy ability

Figure 10 (A-C) represent the results of the horizontal, vertical, and 5V combustion tests, respectively. In general, as the flame-retardant additive ratio increased, the flame endurance, which is defined as the burning duration the specimen could bear before starting to deform, also increased for all three types of combustion tests. When the specimen was horizontally combusted by the 20 mm flame for 30 s, the specimen burned independently to the specimen thickness. Therefore, one could rate it as HB. In the case of the combustion tests regarding the V0, V1, V2 standards, in which the specimen was combusted vertically by the 20 mm flame for 10 s, the specimen self-extinguished within 10 s without dropping. Hence, one could rate it as V0. Finally, when the specimen was subjected to 5VA, 5VB combustion tests, the specimen could be classified as 5VB since it was not burned with flaming and glowing combustion, as well as dripping did not occur.

Conclusions

UD rice straw fibre-reinforced PVC composites were successfully prepared by a common compression moulding method. Composites at a fibre volume fraction of 40% prepared at a temperature at 180°C and pressure of 125 $kg.cm^{-2}$ for 10 min possessed the best possible mechanical properties. It was found that the flame-retardant additive $Mg(OH)_2$ did not only enhance the tensile strength and flexural strength moduli, but also improved the ability to insulate heat and retard a flame. The obtained composites with 20 wt% $Mg(OH)_2$ in PVC showed the best result in mechanical properties as well as thermal insulation ability. Moreover, the flame endurance of the specimen was rated as HB, V0, and 5VB standards.

COMPETING INTERESTS

The authors declare that there is no conflict of interest regarding the publication of this article.

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