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Flexural and Flammability Characteristics of Woven Jute Fabric Reinforced Vinyl Ester Treated with Ammonium Polyphosphate

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Abstract
The work aims to determine the optimum fire-retardant treatment for the jute/vinyl ester composites on the composites' flexural and flammability characteristics. Ammonium polyphosphate deposition in composite samples increased the thickness and weight, which led to an increase in their sample densities. The deposition of 10% ammonium polyphosphate (APP) resulted in the highest flexural strength. However, APP significantly increased the flexural modulus of all samples to the untreated sample. In terms of flammability properties, the deposition of APP increased composites' performance against fire. Incorporating 10% of APP provides good flexural and fire-retardancy properties for the woven jute/vinyl ester composites.

Keywords: jute; composite; vinyl ester; fire-retardant

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1.0 Introduction
There has been an interest in researchers on natural fibre-reinforced composites for the last three decades due to their potential such as possible overall cost reduction, production of multifunctional components, reduction in weight, and reduction in production time (Alsubari et al., 2021; Bachtiar, Kurkowiak, Yan, Kasal, & Kolb, 2019; Chapple & Anandjiwala, 2010; Jariwala & Jain, 2019; Kim, Dutta, & Bhattacharyya, 2018; Misnon, Islam, Epaarachchi, & Lau, 2014). However, due to the limitations in mechanical and other properties, the applications of these materials are mainly found in non-bearing parts. To expand their application to the targeted applications such as in infrastructure construction and automotive industries, another issue that should be solved for these materials to remain relevant is their ability to inhibit fire (Bachtiar et al., 2019; Kandola, Mistik, Pornwannachai, & Anand, 2018; Kim et al., 2018).

There are many works done on the enhancement of natural fibre composite's fire-retardancy (Alongi, Carosio, & Malucelli, 2012; Arjmandi, Ismail, Hassan, & Bakar, 2017; Bakar, Ishak, Taib, Rozman, & Jani, 2010; Boccarusso et al., 2016; Gopinath, Kumar, & Elayaperumal, 2014; Sain, Park, Suhara, & Law, 2004; Shukor, Hassan, Islam, Mokhtar, & Hasan, 2014; Xu et al., 2002; Zhang et al., 2012). The faster, more practical and efficient way to attain flame retardancy is by using additives that can disrupt the process during a particular stage of the burning process (Sain et al., 2004). APP is a chemical that can increase the composite absolute stability and flame-resistance properties and is not harmful to health like the other phosphorus-based additives (Boccarusso et al., 2016). There are five fundamental steps in the burning process: ignition, combustion, heating, propagation, and decomposition. The presence of APP in composite material systems could disrupt the burning even at the earliest stage, which makes the APP become an effective chemical for composite materials.

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This work aimed to determine the optimum formulae in producing fire-retardant woven jute/vinyl ester composite, focusing on the effect of APP incorporation in the composite system on flexural properties. The investigation was also expanded to the burning or flammability characteristic of fabricated composites to analyse their safety for application fields.

2.0 Materials and Method

2.1 Materials

Yeo Hup Kee Sdn supplied commercial plain weave structure jute fabric. Bhd. Vinyl ester resin (SH 7000), methyl ethyl ketone peroxide (MEKP), sodium hydroxide (NaOH), and acetic acid were purchased from BT Science Sdn. Bhd. Ammonium polyphosphate (Exolit ® AP 435) was purchased from DKSH Malaysia Sdn. Bhd.

2.2 Fabrication of Woven Jute Fabric/Vinyl Ester Composites

The woven jute fabric was treated with alkali and fire-retardant to enhance its fabric-resin adhesion and flammability. For the alkalisation, the fabric was immersed in a 2% concentration of NaOH for 2 hours before it was neutralised with acetic acid. The concentration of NaOH was determined to give the optimum effect regarding physical and mechanical properties during preliminary analysis. Fire-retardant treatment was imposed during the composite fabrication.

The vinyl ester resin was prepared by mixing five different percentages of APP on the ratio 95:5, 90:10, 85:15, 80:20, and 75:25 (resin: APP) before MEKP was added into the mixture with the ratio of 1:44 (MEKP: resin) by weight. The prepared mixture was then applied on four fabric layers (with the dimension of 350 × 350 mm) for each layer by employing the hand lay-up technique. The mixture (wet fabrics) was laid between thick glass plates (400 × 400 × 100 mm in dimension). This assembly was then compressed with a weight (20 kg) placed on top of this mixture to ensure it fully spread on the fabric and remove the excess resin. This assembly was left to cure at room temperature for 24 hours before the post-curing process took place in an oven for four hours at 80°C. The samples were named and abbreviated according to the amount of APP content in the samples, which are JC0 (control sample), JC5, JC10, JC15, JC20 and JC25.

2.2 Testing Methods

ASTM D792 standard method was followed to determine the relative density of the fabricated composite samples. The constituent contents of the sample, such as fibre volume fraction and matrix content volume percentage of composite. The testing was conducted by ASTM: D3171 test method II.

A tensile test was conducted by ASTM D638 Type II. The tensile load was applied at a constant displacement rate of 2 mm/min on the composites coupon with the dimension of 185 × 20 × 4 mm, and at least 5 specimens were tested. Meanwhile, the test was conducted for flexural properties to determine the flexural properties of the composites according to ASTM: D790 (three-point bending). The specimen dimension used for this test is 100 × 12 × 4 mm. The load was applied at a constant crosshead speed of 2 mm/min. The samples were monitored for tensile and flexural tests until fibre rupture occurred.

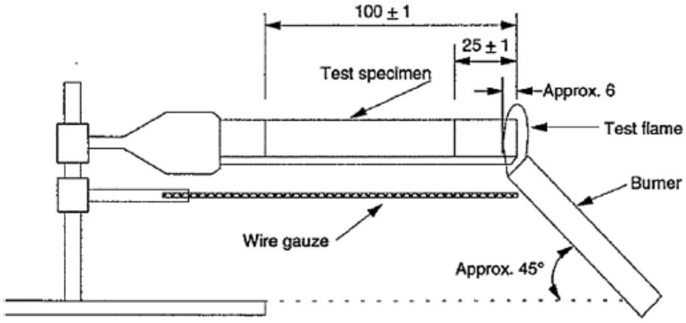

Fig. 1 Test fixture for the burning test by ASTM D635

Flammability properties were investigated using a burning testing method according to ASTM D635. This fire-test response test method compares the relative linear rate of burning composites in the form of a rectangular specimen in the horizontal position. The test setup is shown in Fig. 1. The results of burning testing are intended to serve as a preliminary indication of their acceptability concerning flammability. Each sample's specimen is cut into 125 ± 5 mm long by 13.0 ± 0.5 mm wide and more than 3.0 mm thick. A bar specimen of the fabricated composite material to be tested is supported horizontally at one end. The free end is exposed to a specified gas flame for 30 s. The time and extent of burning are measured and reported if the specimen does not burn 100 mm. An average linear burning rate (V), in millimetres per minute, is reported for material if it burns to the 100 mm mark from the ignited end and calculated based on the following equation:

$$V = 60L/t \tag{1}$$

Where:
V = the rate of burning for the material.
L = the burned length, in millimetres. If the flame front reached the 100-mm reference mark, L = 75.
t = the time in seconds.

A scanning electron microscope (SEM) instrument was used for morphological analysis to observe the physical appearance and fracture on the surface of the composites.

3.0 Results and Discussion

3.1 Flame-Retardant Treatment for Woven Jute Fabric/Vinyl Ester Composite

The physical properties of fabricated composites were measured in terms of their thickness, density, fibre volume fraction and resin volume fraction, as seen in Table 1. The control sample (JC0) showed the highest fibre volume fraction (31.46%) among the other samples. Whilst other samples possessed lower, the trend decreased as the amount of APP treatment was increased. However, the vinyl ester volume fraction showed an inverse trend to fibre volume with the highest point by sample JC25 (80.89%), as shown in Table 1. This is due to the increment of APP deposited on the composite material, which increases the thickness and weight of fabricated composites. A similar reason is applied to the increment trend of sample densities. Justification from the cross-section view in Fig. 2(b-d) at 100μm magnification of the FESEM micrograph, clearly visible the fibre, matrix and APP particle at the JC5, JC20 and JC25 in comparison with Fig. 2(a) where at the resin part the surface was clear and smooth.

Table 1. Physical properties of woven jute/vinyl ester composites

Sample	Density (g/m3)	Thickness (mm)	Fibre volume fraction (%)	Matrix volume fraction (%)
JC0	1.213	3.23	31.46	67.60
JC5	1.24	3.80	26.16	74.44
JC10	1.24	3.85	25.82	74.79
JC15	1.24	3.9	25.49	75.12
JC20	1.29	3.97	24.07	79.64
JC25	1.297	4.08	23.29	80.89

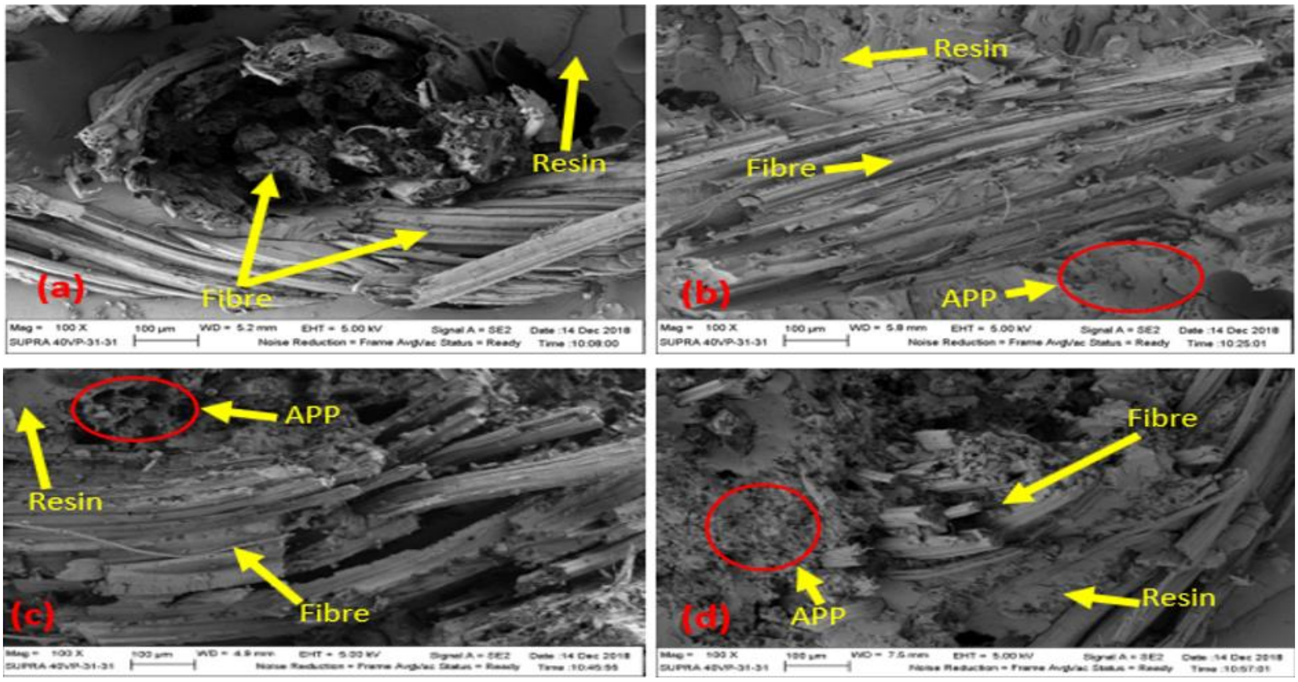

Fig. 2: Cross-section view; FESEM micrographs of tensile fracture (a) JC, (b) JC5, (c) JC20 and (d) JC25 samples

In addition, EDX, as the chemical analysis, was also used to investigate the elemental analysis to analyse the presence of fire-retardant elements in several samples. Fig. 3 shows that the treated sample consisted of four main elements: carbon (C), oxygen (0), phosphorus (P) and aurum (AU). Nevertheless, aurum(gold) was exempted from the discussion because it is an external element used to coat the sample for conductivity and avoid charging effect purposes. EDX results in Table 2 show each element's weight percentages (wt%) in all composite samples. The C and O elements were the major elements in the X-ray emission spectrum as they theoretically followed the main element in the reinforcement matrix. The main element of JC0 contains carbon and oxygen, with weight percentages of 39.57% and 12.90%. Meanwhile, for treated samples JC10 and JC20, the phosphorus increased from 1.02% to 1.66% as the amount of APP increased. The presence of a higher phosphorus element should promote better thermal stability and flammability properties for fabricated composite samples.

Meanwhile, for the JC5 sample, traces of phosphorus elements were not found in the EDX analysis. This could be a quantification error as the sample volume for EDX analysis is small compared to the large samples produced. This case might be an aggregation of APP particles in the JC, which obstructed the mixture's dispersion and thus reduced the JC sample's homogeneity.

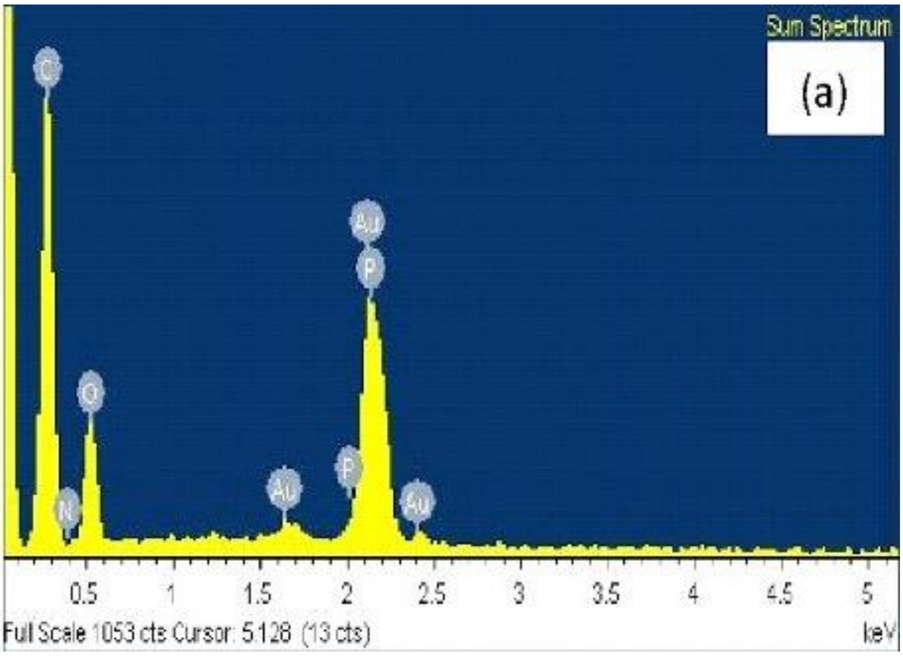

Fig. 3: Typical EDX analysis on the woven jute/vinyl ester composite

Table 2. Energy-dispersive X-ray analysis of woven jute/vinyl ester composites

Sample	Weight percentage (%)				
	C	O	P	AU	Total
JC0	39.57	12.90	-	47.53	100
JC5	36.43	11.52	-	52.05	100
JC10	37.73	14.20	1.02	47.02	100
JC20	39.03	11.57	1.66	47.74	100

3.2 Flexural Strength Properties of Woven Jute Fabric/Vinyl Ester Composite

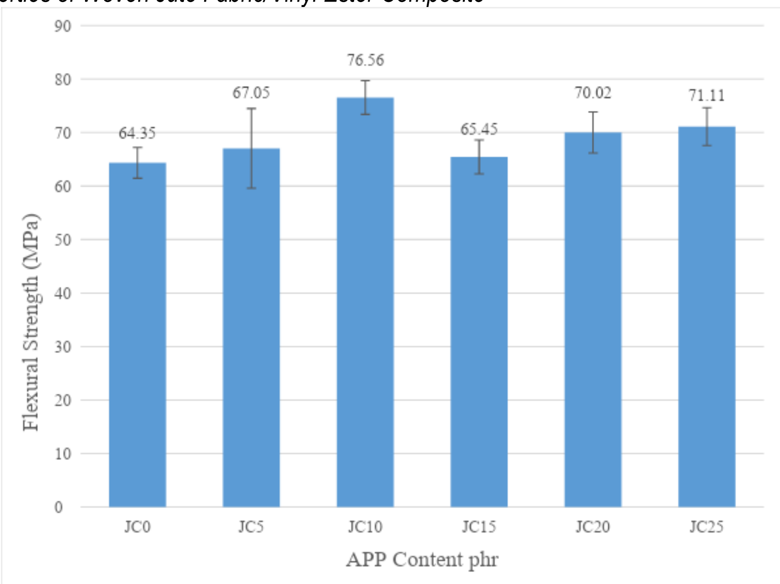

Fig. 4: Flexural strength of woven jute/vinyl ester composites

Fig. 4 shows the flexural strength of untreated sample (JC0) and treated samples with different incorporation content of APP. It can be seen that there is some improvement in the flexural strength of treated composite samples compared to untreated. In addition, JC10 showed the highest flexural strength compared to other samples. This might be due to the excellent interface adhesion and low-stress concentration in the polymer composites (Arjmandi et al., 2017); however, as the APP content increases from 10% to 25%, the flexural strength of the composites decreases. Similar results were reported by Arjmandi et al. (2017) and Zhang et al. (2012), that indicated one of the reasons for a decrement in flexural strength is due to the filler agglomeration that usually occurs at high content, which could initiate the fracture of the composites possibly by the acts of internal stress concentrator points.

Based on Fig. 5, composites incorporating APP contents showed better flexural modulus than the control sample. The incorporation of APP enhanced the stiffness of the composite, which was contributed by the presence of an immobilised or partially immobilised polymer phase due to the high stiffness of the phosphorus layers of APP. An increase in the amount of APP from 5% to 10% was observed to increase the flexural modulus of the composites. However, the flexural modulus declined with APP content from 15% to 25%. It could be that high APP content causes the lack of homogeneity present within the composites. Agglomeration of filler particles under high APP content obstructs the dispersion of the mixture and thus reduces the homogeneity of the composite. Besides, high APP content possibly disrupted the interfacial adhesion between vinyl ester resin and jute fibres, decreasing the flexural modulus. Arjmandi et al. (2017) observed a similar trend in their study, where the effect of the APP content on kenaf/polypropylene and rice husk/polypropylene composites was investigated. Shukor et al. (2014), in their study of the effect of APP content on the alkali, treated kenaf fibre-filled PLA biocomposites, also found similar findings. As shown in Fig. 15, the right amount of APP content would enhance and improve the flexural modulus, while the excess amount of APP would deteriorate the properties of the composite.

The surface morphology of treated and untreated jute composites in the cross-section view was observed and studied under FESEM. Fig. 2(a-d) shows clear visible fibres, matrix and APP particles at 100μm magnification. As shown in Fig. 2(b-d), the micrographs showed less aggregate of APP particles in the JC10 sample. They displayed a smoother surface, resulting in less formation of voids and cavities than the JC20 sample. The presence of the voids and cavities is getting higher because aggregates of APP particles in the composites lead to the formation of micro-cracked and initiate the crack within the matrix, weakening the internal strength of the composite. This explains that the deterioration of flexural strength is much higher as APP content increases on the composite samples. A poor fibre-matrix adhesion on the samples can also be seen in Fig. 2(b-d). There are traces of matrix mixture sticking on the fibre surface, reducing the composite's strength. Similar results were reported by Arjmandi et al. (2017) and Bakar et al. (2010). Meanwhile, the claims mentioned above were aligned with Gopinath et al. (2014), stating that the significant factors for reduction in reinforced composite strength are fibre-matrix adhesion, air voids, dispersion and orientation of fibre and fibre agglomeration.

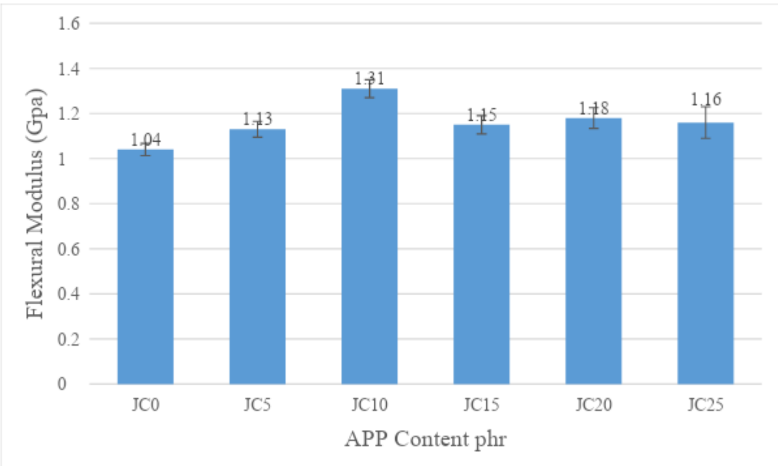

Fig. 5: Flexural modulus of woven jute/vinyl ester composites

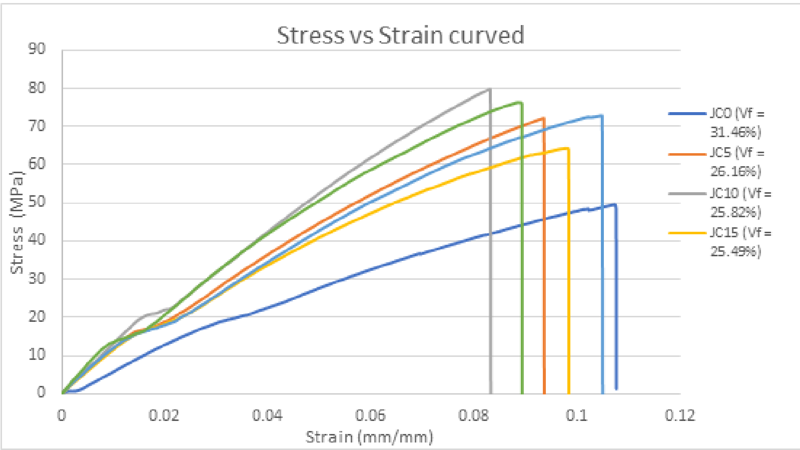

Fig. 6: Stress-strain behaviour of woven jute/vinyl ester composites

The uniaxial flexural stress-strain behaviours for the jute vinyl ester/APP composites are shown in Fig. 6**.** As can be seen, the trend showed trilinear behaviour by each composite sample, which went upward before completely rupturing. The control composite's initial stiffness was lower than the treated composite. This can be related to the lower fibre volume fraction of the treated samples than the untreated sample. The treated composites' matrix volume fraction and thickness increase as the fibre volume fraction decreases. Thus, the initial stiffness of treated composite samples was expected to be higher than that of untreated samples. The strength of the composite decreased because, at the fibre's surface, the matrix initiated the crack and weakened as the load increased. However, the strength of the composites then continually increased as the fibres supported and provided the strength for the composites as the load increased until the sample was fully ruptured.

3.2 Burning/Flammability Properties of Woven Jute Fabric/Vinyl Ester Composite

Burning characteristics of fabricated samples were investigated following ASTM D635, and the results were tabulated in Table 3. In this test, 30s-time exposure of a small flame was set and exposed within the 25mm mark on the composite specimens. Only the JC0 sample was burned entirely among all the samples, as shown in Fig. 7(a). The time taken was 235.06s for the flame to spread and reach 100mm marks. While the burning process progressed, it was also observed that the sample was burned in yellowish-orange flame, produced thick black smoke and released a pungent smell like burnt plastic mixed with paper, which might correspond to the mixture of the jute fabric and vinyl ester resin. The measured linear burning rate of this sample was 19.17 mm/min.

After the test, a massive amount of ash was produced, and only a slight residue was left behind this sample, indicating that the JC0 sample has poor flame retardancy performance. The JC5 sample was partially burned with yellowish-orange black smoke where the flame spread reached 25mm marks but did not reach 100mm marks. Besides, the sample shape was maintained when the test finished due to char formation that prevented the flame from spreading and disintegrating the composition into ash. The burnt part of the sample became brittle in the charred area, as shown in Fig. 7(b). As the content of APP increases from 10% to 25%, it produces less and thin smoke while the abilities of the composite samples to retard the flame from spreading keep increasing Fig. 7(c-d). This is proven by the failure of the flame to reach even the first mark, as shown in Table 3. Hence, it can be concluded that the increase in APP content improves the flame retardancy of the composite sample. When exposed to high temperatures, APP releases phosphoric acid and non-flammable gases, producing intermolecular dehydration and charring. Many oligomers and small molecular species are produced by acid from the pyrolysis of APP catalysts –C–O– bond breaking. This action retards the burning process, especially in the ignition, combustion and heating stages. (Alongi et al., 2012; Chapple & Anandjiwala, 2010; Horrocks, 2011; Xu et al., 2002).

Table 3. Results of the burning test of woven jute/vinyl ester composites

Sample	1st mark (25 mm)	2nd mark (<100mm)	3rd mark (100 mm)	Elapsed time (s)	Liner burning rate (mm/min)
JC0	/	/	/	235.05	19.17
JC5	/	/	X	113.35	X
JC10	X	X	X	X	X
JC15	X	X	X	X	X
JC20	X	X	X	X	X
JC25	X	X	X	X	X

X indicated that the expected event had not happened

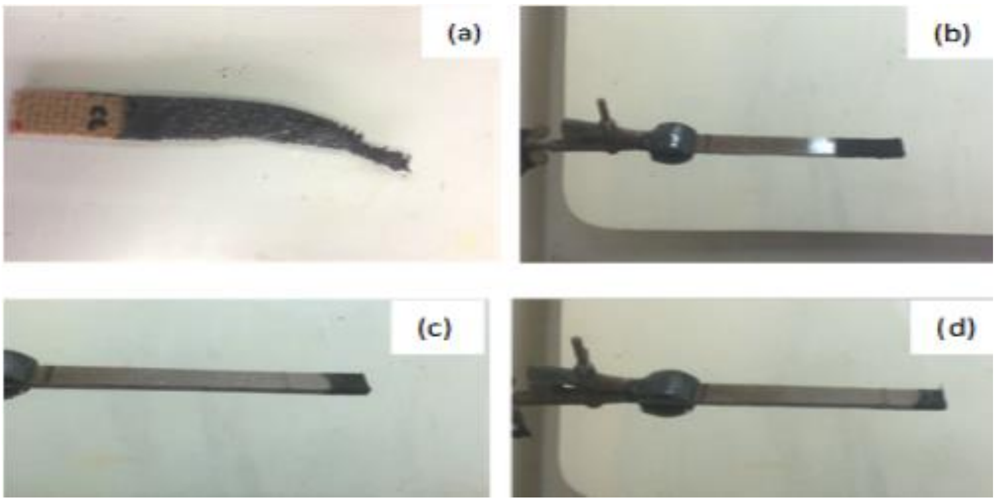

Fig. 7: Image of sample; (a) JC0, (b) JC5, (c) JC10 and (d) JC20 after the burning test

4.0 Conclusion

In this study, woven jute/vinyl ester composites were treated with different amounts of APP were fabricated. Applying APP increased the fabricated samples' thickness, weight and density. Regarding flexural properties, the deposition of 10% APP resulted in the highest flexural strength of the samples. Lower and above percentages than 10% deposition resulted in lower flexural strength. However, APP significantly increased the flexural modulus of all samples compared to the untreated sample, which is attributed to the presence of an immobilised or partially immobilised polymer phase due to the high stiffness of the phosphorus layers of APP. In terms of flammability properties, incorporating APP in fabricated samples improved fire-retardancy properties. The deposition of APP of more than 5% in composite systems exhibited excellent performance against thermal and fire. None of the APP-treated samples burned and did not reach the first mark when exposed to a flame source for the 30s. Thus, the APP treatment was highly effective in enhancing and improving the thermal and flammability properties of natural fibre reinforced polymer composites. Scrutinising all the results, incorporating 10% of APP is the optimum value, providing good flexural and fire-retardancy properties for the woven jute/vinyl ester composites; it is safe for low-load bearing applications.

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Paper Contribution to Related Field of Study
The paper contributes to the study field of textile materials, composite materials, mechanical properties characterisation and fire retardancy characterisation.

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