



Research Article

Thermal, mechanical and flame spread properties of hybridized flame retardant in oil palm fibre-polyester panel



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Abstract

In order to mitigate the start and spread of flame of combustible materials, flame retardants (FR) are incorporated during processing that influences mechanical and thermal properties. In this paper six (6) FR species derived from aluminium hydroxide (ATH), ammonium polyphosphate with gum arabic powder (APP-GAP) a new FR, ATH/APP-GAP hybrid formulation and CB synergist at 0, 12, 15 and 18% loading ratio were developed in oil palm fibre reinforced polyester composites panel using hand lay-up compression moulding technique. The effect of the FR on the mechanical, thermal and flame spread properties were then evaluated. Flame spread results using radiant panel flame spread apparatus show hybrid flame retardant panel traveled less distance of 150 mm from 450 mm of the control without FR which represents a 67% decrease and indicative of a weak available flame spread energy that led to flame extinguishment. Mechanical properties obtained with the universal testing machine reveals improved tensile strength, tensile modulus and flexural strength in the case of the 12%ATH non-hybrid FR by 16.2%, 5.9% and 71.2% respectively, indicative of ATH having great affinity with the polyester resin and effective transfer of stresses. Thermogravimetric analysis reveals that the 12%ATH non-hybrid FR and the 15%APP-GAP/CB non-hybrid-synergist FR are more thermally stable before degradation began around 376 °C and 391 °C and fully degraded at 421 °C and 415 °C respectively. Char residue at 900 °C also confirms that the non-hybrid synergist residual mass at 17.47% indicative of a rich decomposition nature of APP-GAP–CB into a more stable char structure could be a good flame retardant. The results obtained indicates that the new concept of APP-GAP to formulate an hybridized FR and non-hybrid-synergist in oil palm fibre-polyester panel is feasible to meet the expectations of a good flame retardancy and thermal stability but do not support improved mechanical properties.

Keywords Hybridization · Mechanical properties · Oil palm fibre · Polyester · Thermal properties · Flame spread

1 Introduction

The quest for high-performance materials made from natural sources such as jute, sisal, hemp, flax, kenaf, sugarcane, banana, oil palm, coir, wood and their likes are naturally grown and consist of organic constituents that comprise of cellulose, hemicellulose and lignin and thus, can be called lignocellulosic or cellulosic fibres. Lignocellulosic based fibres is still very attractive in today's world of composites because it satisfies several purposes which includes but not limited to a reduction in resin cost,

biodegradability, lightweight, environmental benign, high stiffness and the ease in processing into finished products [1–3]. There is evidence that the production of composites materials by the addition of lignocellulosic based fibres taking into account its biodegradable and physical performance to polymers allows the improvement of mechanical and thermal properties. Thakur et al. [4] studied the mechanical and thermal behaviour of natural cellulosic fibres reinforced polymer composites. In their findings it shows that the fibres were successfully fabricated with useful mechanical properties. The

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authors [5] also showed that pine needles were used to prepare phenol formaldehyde resin, a novel polymer matrix. Analysis on thermal properties and polymer composites showed promising results.

Oil palm empty fruit bunch (OPEFB), generated from oil palm industry are regarded as huge amount of ligno-cellulosic waste and unutilized. These products normally cause major environmental pollution that can be readily turned into useful value-added products [6]. It can be harnessed as reinforcement in polymeric composites for the production of various components as reported by several authors working on the suitability of OPF polymer composite [7, 8]. OPF derives its strength and rigidity like any other natural fibres from cellulose that is semi-crystalline polysaccharide in nature. However, their high susceptibility to flames due to the presence of high cellulosic content in the fibres and polymer, which are combustible in nature, has limited their broad use especially in areas such as the aviation, marine and rail industries where stringent fire safety regulation do exist. Therefore, for OPFPC to meet stringent requirements for application in these sectors, incorporating flame retardants (FRs) during composite processing has become highly desirable as well as not affecting their mechanical and thermal properties.

There are quite a number of FRs that have been studied in nature fibre polymer materials which can delay the start of a fire and even mitigate its spread [9–11]. Among the studied FRs, aluminium tri-hydroxide (ATH) and ammonium polyphosphate (APP) are considered the most common because they are 'greener' and have a positive effect in reducing the burning rate of materials, reduce smoke production and resist the spread of flame [12, 13]. However, recent study by Khalili et al. [9] showed that the hybrid formulation of ATH and APP at low percentage loading exhibited deterioration in the mechanical and thermal degradation properties of empty fruit bunch fibre reinforced epoxy. Similar reports by Subasthinghe and Bhattacharyya [10] agrees with these findings. In their work, the authors observed that a decrease in thermal decomposition rate when APP was added into polypropylene/Kenaf composites but improved in mechanical properties. The effect of ATH and APP FR formulations or their hybridization in OPFPC has not studied quite extensively. Hence, this paper is aimed at formulating ATH, a new intumescent FR with Gum Arabic powder (GAP) and their hybrids in OPFPC at different FR loading proportion and analyzes the effect on mechanical, thermal and flame spread properties using universal testing machine, Thermogravimetric analysis (TGA) metler Toledo and radiant panel flame spread apparatus respectively. This would provide an insight on the effect of ATH and APP hybrids in OPFPC.

Table 1 Experimental design of the flame retardant OPF composite panels

Specimen I.D	OPF/resin ratio (wt%)	% of FR ^a		
		ATH	IFR (2:1)	CB
WSPC _{wo}	10/90	–	–	–
12%ATH	10/90	12	–	–
12%APP-GAP	10/90	12	–	–
15%ATH/CB	10/90	9	–	6
15%APP-GAP/CB	10/90	–	9	6
18%ATH/APP-GAP	10/90	9	9	–
18%ATH/APP-GAP/CB	10/90	9	6	3

^aFormulation of flame retardant species specified relative to the total quantity of resin

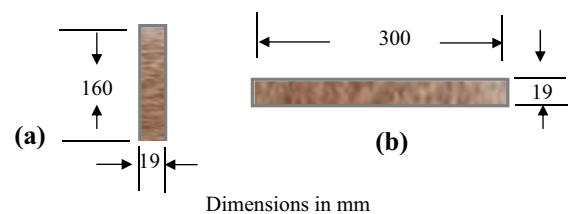


Fig. 1 a Tensile measurement, b flexural measurement

2 Experimental methods

2.1 Preparation of the OPFC Panel

OPF obtained were first extracted by washing with hot water to remove the remaining residual oil retained during oil extraction, a method proposed by Vijaya et al. [14] and then soaked in n-Hexane overnight to complete leaching and further remove impurities. The fibres were then treated with 5% (NaOH) solution to improve compatibility with the polyester resin (PR). Fabrication of the OPFC panels at different FR loadings was carried out using hand lay-up compression moulding. A pasty solution made up of the FR formulations as depicted in (Table 1) was cast into the mould containing evenly distributed OPF with no voids. The required quantity of OPF was obtained using mass fraction model developed by Ezema [15].

2.2 Tensile testing

Samples cut from the fabricated OPFC panel with dimensions shown in Fig. 1a were subjected to a static tensile test according to ASTM D 638 standards using a UTM (Hounsfield model No. 8898). The specimens were held

horizontally and the ends of the specimens were positioned in the mechanical grips. The speed to pull out the specimens was 2 mm/min at a temperature of 22 °C and humidity of 50% in all cases. The load cell rating is a 5000 N and the distance between the holders fixed at 40 mm. Stress–strain curves were plotted from the force–extension data obtained on a special graph during the tests and the tensile properties were determined. An average value was recorded after 3 tests were conducted.

2.3 Flexural testing

The samples were also subjected to a 3-point bending method using the universal testing machine (Hounsfield model No. 8898) according to ASTM D790 procedure to determine the flexural strength with dimensions shown in Fig. 1b. The specimens were placed vertically on a support span and the load is applied to the centre by the loading nose, producing three points bending at a specified rate. In each test, also 3 specimens were recorded and an average value recorded.

2.4 Thermogravimetric analysis

The analysis of all the specimen panels was carried out using thermogravimetric analysis (TGA/DSC 1; Mettler Toledo, UKBRC, Edinburgh, UK). Samples were first heated for 10 min at 105 °C under N₂ to determine moisture content; the temperature was then raised at 25 °C min⁻¹ to 900 °C where it remained for a further 10 min to determine volatile matter content. Finally, air was introduced to the system combusting the sample (also at 900 °C) for 20 min in order to determine the ash content. Fixed carbon is calculated on a weight percent basis by subtracting moisture, volatile matter and ash values from the original starting mass.

2.5 Flame spread travel

Surface flame spread test was performed on the specimens according to ASTM E 1321 standard procedure using a radiant panel flame spread apparatus. A (500 mm × 100 mm × 10 mm) specimen was slide between an aluminium frame with hinged front cover backed by a non-combustible board and a sample holder made of aluminium wire. Along the edge of the specimen holder there are every 25 mm marks to aid in recording the flame spread rate. Video recordings of the moving flame front position were captured in real time as shown in Fig. 2 for the flame front in order to derive the velocities. The test was repeated thrice for each specimen to verify the rate of surface flame spread and average value recorded.

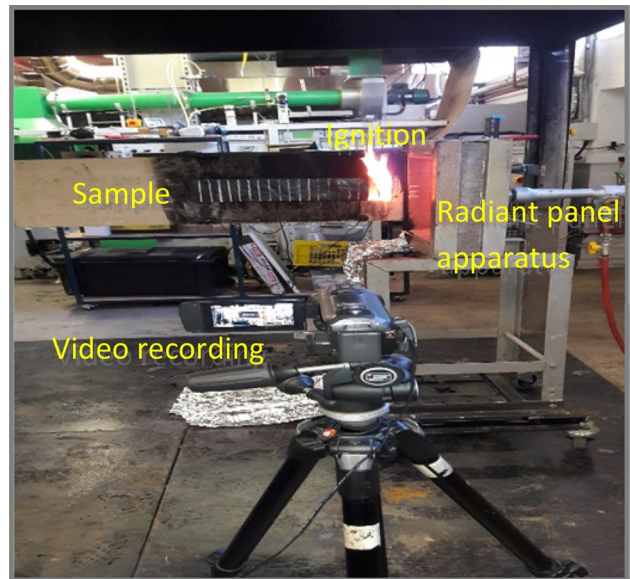


Fig. 2 Experimental set-up of the flame spread test

3 Results and discussion

3.1 Mechanical properties

At 10% fixed OPF loading used to fabricate the composite panels shows that maximum tensile strength can be obtained as reported by Beg et al. [16]. The influence of the FR formulations on the mechanical properties was then evaluated and the results on average presented in (Table 2).

3.1.1 Tensile properties

In (Table 2), it can be seen that the effect of 12%ATH inclusion in the OPFC panel is positive for TS and TM, indicating an improvement in tensile properties relative to %OPFC panel. The graph depicted in Fig. 3a further shows that the 12%ATH specimen enhanced better the TS among the FRs studied and could be attributed to the presence of ATH having great affinity in the PR which may have caused a transfer and distribution of stresses effectively. It further indicates that a proper interfacial adhesion exists between the PR, fibres and the ATH-FR formulations. Contrary to this, the addition of 15%APP-GAP/CB and 18%ATH/APP-GAP specimens as shown in Fig. 3b improved better in TM but decreased in TS. The presence of APP which caused a decrease as expected agrees with the report of Bocz et al. [17]. Similar trend have been reported for OPFC as seen in the work of Khalili et al. [9], Redwan et al [18] and Norzali et al. [19]. The reason can further be elucidated by the FR particles disturbing the load transfer from PR to OPF, resulting in the reduction in the tensile properties of OPFC

Table 2 Results on mechanical properties of oil palm fibre composite panels

Specimen I.D	T.S (MPa)	Effect (%)	T.M (MPa)	Effect (%)	F.S (GPa)	Effect (%)
0%OPFC	13.00 ± 1.84	–	1.53 ± 0.40	–	30.29 ± 5.87	–
12%ATH	15.11 ± 2.19	16.2	1.62 ± 0.42	5.9	51.86 ± 15.15	71.1
12%APP-GAP	10.56 ± 1.97	–18.8	1.48 ± 0.38	–3.3	42.58 ± 4.25	40.6
15%ATH/CB	12.00 ± 0.00	–7.7	1.36 ± 0.20	–11.1	49.13 ± 5.15	62.2
15%APP-GAP/CB	9.44 ± 1.45	–27.4	1.67 ± 0.32	9.2	24.00 ± 2.89	–20.8
18%ATH/APP-GAP	10.89 ± 0.49	–16.2	1.74 ± 0.58	13.7	28.38 ± 5.48	–6.3
18%ATH/APP-GAP/CB	9.78 ± 1.45	–24.8	1.10 ± 0.10	–28.1	25.93 ± 1.92	–14.4

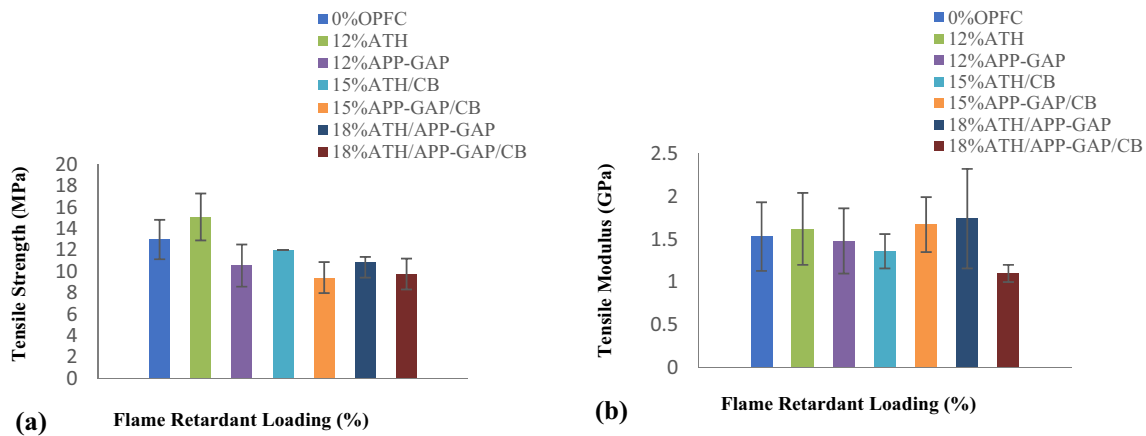


Fig. 3 Graphs showing the influence of the various FR formulations in OPFC panel: **a** tensile properties, **b** tensile modulus

panels. Besides, higher loadings of FR have been reported to enhance the microspaces in the FR-PR interfacial region, cause agglomeration due to interactions and incomplete interfacial wetting may lead to decreased stress transfer from PR to fibres resulting in decrease in tensile properties [20, 21].

3.1.2 Flexural strength

The ability of the studied FR formulations in OPFC panels to withstand bending forces applied perpendicularly to its longitudinal axis was evaluated and the results obtained on average are presented in Fig. 4. The effect of FR inclusion was positive for 12%ATH, 12%APP-GAP and 15%ATH/CB, indicating an enhanced FS. The increase in FS for 12%ATH is consistent as seen in the result obtained for TS. The reason could be that a strong intermolecular force between ATH and PR exists. The hybrid FR formulations showed negative values indicating a decrease in FS. In this paper, the increase in FR formulations in the OPFC panel from 12 to 15% maintained an increase in FS and then began to decrease with increase in FR formulations. It can be seen clearly that the FR formulation with 12%ATH showed the highest FS of 51.86 MPa among the composite panels studied. The overall increase in FS for

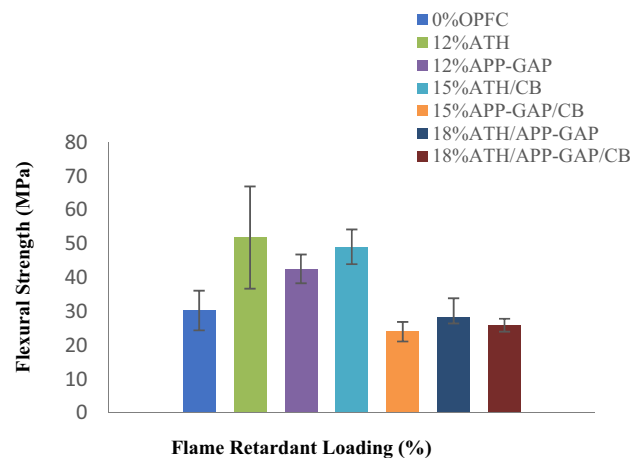


Fig. 4 Flexural strength data for OPFC panels at varying flame retardant loadings

the FR-OPFC panels could be explained as having a better covalent bonding at the interface of the fibre/PR/FR which provides improved properties of the composite panels relative to the 0%FR-OPFC and also suggest an efficient stress transfer from the PR to the Fibre/FR [22]. On the other and, the decrease in FS suggest incompatibility issues at high FR loadings and their hybridized formulations making it

difficult for the PR to infiltrate, hence it weakens the interfacial adhesion between fibre/PR/FR composite panels.

3.2 Thermogravimetric analysis

The thermal degradation of the FR formulations in the OPFC panel is an important aspect of its commercial significance. Thermographs i.e. thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) were employed to evaluate the effect of the FRs on the thermal response. The results obtained are tabulated in (Table 3). From the TGA curve depicted in Fig. 5, it shows that the overall thermal decomposition of the OPFC panel began around 150 °C, indicating loss of water from the fibres as well as confirms its hydrophilic nature [23–25]. A sharp drop within the range of 200–350 °C indicates the loss of hemicellulose followed by pyrolysis of cellulose within a temperature range of 350–450 °C. In addition lignin decomposes in a wide range of temperature (150–900 °C) and is said to be the greater part of char formation. This has also been reported by other authors on natural fibres [26, 27]. The onset decomposition temperature

(T_0) obtained in (Table 3) falls within the same range of OPEFB reinforced bio-based polyester composite studied by Dhandapani et al. [28] which suggest that the presence of OPEFB enhances thermal stability of polymer composites. When 12%ATH and 15%APP-GAP/CB were added into the OPFC panel, it was effective in delaying the T_0 by 5.1 °C and 18.9 °C respectively, exhibiting better thermal stability over other FR formulations which recorded less compared to 0%OPFC panel. This could be attributed to the presence of ATH when decomposed, releases water vapour due to its endothermicity and converts the thermal stable aluminium oxide that demonstrates very high melting temperature [29]. On the other hand, the presence of APP could be attributed to the findings by Alam et al. [30]. The presence of the FR formulations has shown to decrease the total weight loss resulting in less by amount of material consumed during heating as seen in (Table 3). From the DTG curves depicted in (Fig. 4), an endothermic T_{DTG} peak shifted to a higher temperature relative to 0%OPFC for all the FR formulations. The inclusion of 12%APP-GAP, 15%APP-GAP/CB and 18%ATH/APP-GAP formulations showed two endothermic T_{DTG} peaks

Table 3 Results data on thermal stability and degradation of oil palm fibre polyester composite

Specimen I.D	T_0 (°C)	T_E (°C)	WL (%)	T_{DTG} peak (°C)	Char residue (%)
0%FR-OPFC	371.15	442.21	92.70	409.82	6.15
12%ATH	376.25	443.86	88.18	421.93	10.62
12%APP-GAP	352.28	437.83	87.84	412.70	10.53
15%ATH/CB	366.61	435.81	85.24	409.90	13.65
15%APP-GAP/CB	391.63	403.08	80.85	426.92	17.47
18%ATH/APP-GAP	354.59	441.59	83.30	415.27	15.46
18%ATH/APP-GAP/CB	368.09	406.61	86.07	416.41	12.16

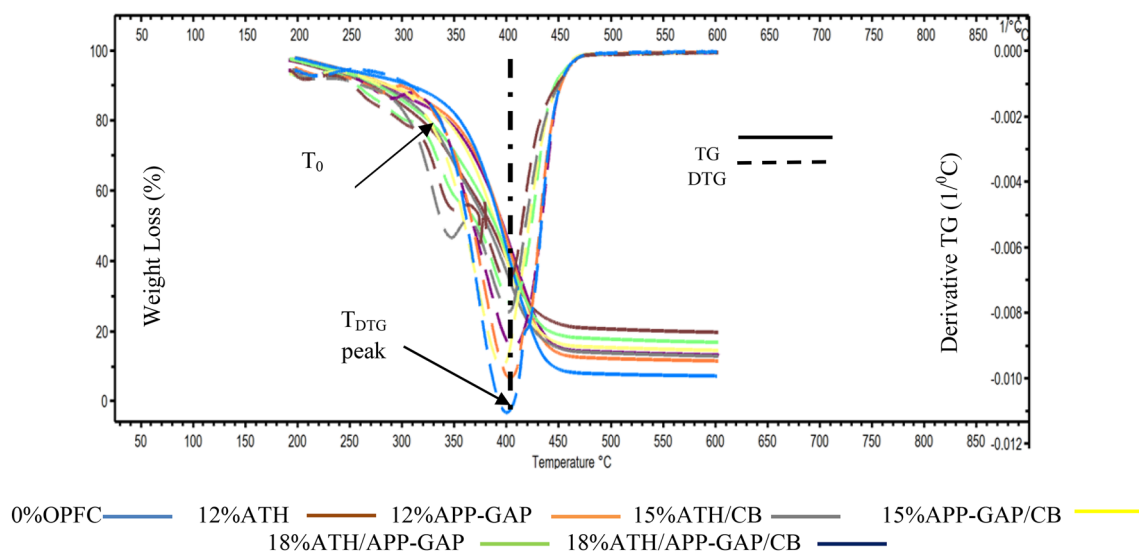


Fig. 5 Thermogravimetric analysis and derivative of thermogravimetric curves of various flame retardant formulations in OPFC panel

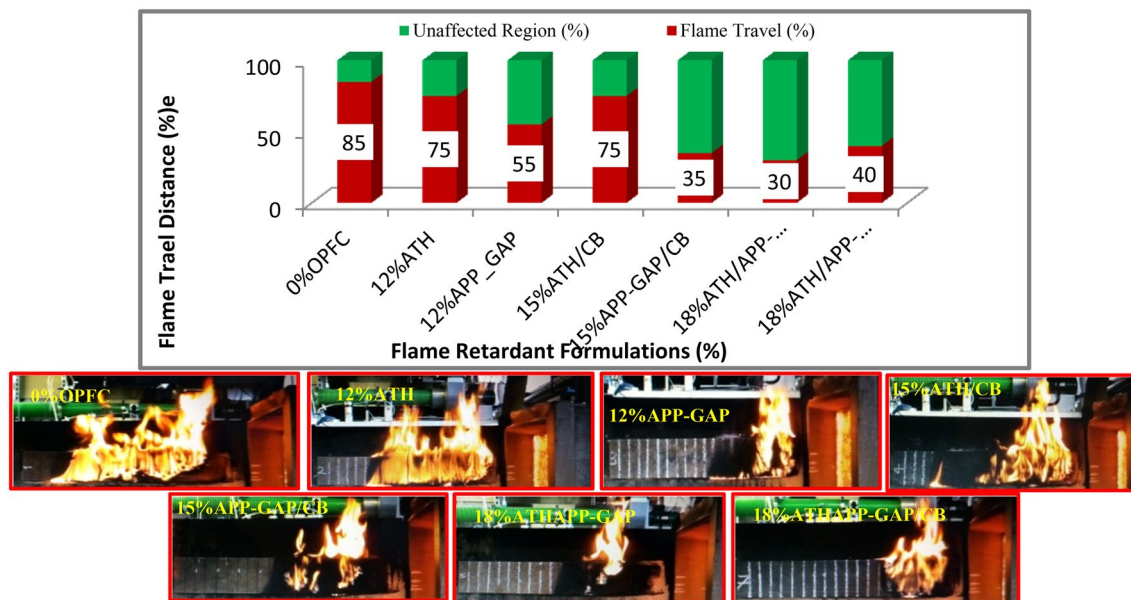


Fig. 6 Stages of flame front travel on the OPFC surfaces in real time showing affected and unaffected region

behaviour, indicating a two-step degradation process. The first peak was observed around 356.7 °C, 361.3 °C and 350.8 °C respectively which are below the 0%OPFC panel associated with the degradation of hemicellulose while in the second peak it is clearly seen as a shift to a higher temperature relative to 0%OPFC. Similar trend of double peaks with OPEFB fibre reinforced poly (butylene adipate-co-terephthalate) biocomposites was found in the work of Siyamak et al. [31] and with IFR in kenaf fibre reinforced polypropylene by Subastinghe and Bhattacharyya [10]. At 900 °C which indicates the end of the test, the FR formulations present in the OPFC panel formed an increased amount of char residue which suggests a slower decomposition and a barrier for the mass transfer of heat. Among the FRs studied, 15%APP-GAP/CB formed the highest char at 17.5% which signifies enhanced thermal stability as well as a superior advantage in terms of their flame retardancy. In this study, the decrease is consistent with the reports of Choh et al. [5] on negative effect of increasing FR loadings in composites. It can be further be elucidated by the water content in APP which upon thermal degradation is responsible for the premature destruction of the polymer caused by water hydrolysis, attributed to Reti et al. [32] and Le Bras et al. [33]. Also, the loss of absorbed and structural water in GAP [34] may be have been involved in the early degradation.

3.3 Flame spread travel

From the surface flame analysis, successive ignition as the flame travels along the composites surface was captured

by camera imaging and presented as shown in Fig. 6. It reveals that among the FRs in the OPFC panel, 18%ATH/APP-GAP travelled less distance of 150 mm (30% travel) before receding as opposed to the 0%OPFC panel which travelled a distance of 425 mm (85% travel) and showed a rich flame as it travels down the composite displaying sufficient heat transfer ahead of the flame which continue to spread along the surface. A similar behaviour was observed for 12%ATH and 15%ATH/CB specimens, indicating less effect of the treated fibre and the ATH/CB mechanism as it covered a distance of 375 mm (75% travel). Furthermore, the 12%APP-GAP, 18%ATH/APP-GAP/CB and 15%APP-GAP/CB specimens traveled less distances at 275 mm, 200 mm and 175 mm representing 55%, 45% and 35% respectively relative to 0%OPFC. This suggests that the combined effect of ATH and APP-GAP interfered with the forward heat transfer better and decreases the formation of combustible gases. Consequently, the addition ATH and APP-GAP to OPFC shows that the surface panels was starved of oxygen and thus recede flame travel which sometimes may lead to extinguishment. This is expected as the flame retardant mechanism acts both in the condensed and gas phases [35].

4 Conclusions

In this research paper, fabricated OPF panels with various FR formulations have been examined and the effect on the mechanical, thermal and flame spread properties concludes as follows:

1. Among the FRs studied in the OPFC panels, the 12%ATH specimen showed an overall improved mechanical property that is for tensile strength (TS), tensile modulus (TM) and flexural strength (FS) with maximum values at 15.11 MPa, 1.62 MPa and 51.86 GPa respectively. Further increase from 12 to 18% FR loadings and their hybrid formulations decreased in TS and increased in TM while the addition of the synergist carbon black decreased the FS behaviour.
2. Based on the findings on thermal behaviour of the OPFC panels, TGA and DTG analysis showed that 12%ATH and 15%APP-GAP/CB exhibited improved thermal stability when compared to 0%OPFC as ATH dehydrates endothermically upon heating and the presence of CB in APP-GAP restricts the mobility of the PR to become slower.
3. Radiant panel analysis reveals that the presence of 18%ATH/APP-GAP specimen in OPFC panel weakens the surface flame spread travel along the surfaces of the panel as opposed to the control specimen with rich flame displaying sufficient heat transfer.

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Compliance with ethical standards

Conflict of interest The corresponding author received a study Grant from TETFUND, Nigeria with Grant approval Reference No. (TETFUND/ES/AST&D/POLY/EKOWE/VOL.1) for further studies and solemnly declares that there are no conflicts of interest.

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