



DETERMINATION OF THE SUITABILITY OF OIL PALM FIBRE COMPOSITE FOR BUILDING APPLICATIONS BY CONE CALORIMETER AND THERMAL ANALYSIS

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ABSTRACT

The high yielding of oil palm fibre reinforced composite (OPFC) to fire has necessitated research to improve and develop fire retardants (FR) to mitigate the spread of fire. Researchers relied on Flame Retardants (FR) classified as either halogenated or non-halogenated based FR to improve the performance of composites with emphasis on flammability properties (FP). The main object of this paper is to evaluate the effect of six non-halogenated FR species in OPFC to meet required fire safety standards for building purposes. The six FR species comprising aluminum tri-hydroxide (ATH), ammonium polyphosphate (APP), Gum Arabic powder (GAP) and carbon black (CB) were processed with OPFC at 0, 15 and 18% loading ratio using hand lay-up compression moulding technique. Specimens cut from the OPFC panels were tested for flammability and thermal properties using thermogravimetric analysis (TGA/DSC Metlar Toledo) and cone calorimeter apparatus respectively. The result obtained for thermal analysis shows that the panel was thermally stable at 391.60C before degradation began compared to those without FR while peak flammability properties obtained for heat released rates, mass loss rates and smoke production rates showed the OPFC panels rapid fire response were significantly reduced respectively by 67.4%, 50.9% and 37.5% compared to those without FR. It can be concluded that the hybrid FR comprising APP-GAP showed a stable char structure during fire and thus prevented the escape of combustible volatiles which reduced the peak FP values of the OPFC panels. These flammability properties could be said to meet required fire safety standards for building applications.

Keywords: ammonium polyphosphate, flammability properties, flame retardants, gum arabic, oil palm, fibre, thermal stability

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INTRODUCTION

In the world of composites, fibres obtained from natural sources are becoming very attractive for non-structural building applications probably because of their huge benefits which includes but not limited to low-cost, abundant availability, lightweight, environmental benign and biodegradability as reported by Layth *et al.*, (2015). Natural fibres (NF) such as jute, sisal, hemp, flax, kenaf, sugarcane, banana, oil palm, coir, wood etc. are naturally grown. They consist of organic constituents which make up cellulose, hemicellulose and lignin and are usually called lignocellulosic or cellulosic fibres. NF derives its strength and rigidity from cellulose that is semi-crystalline polysaccharide in nature. Cellulose-based fibres obtained from plants are broadly used in polymer composites due to their abundance and renewability within a short time when compared to animal or mineral sources as reported by Layth *et al.*, (2015).

Oil Palm (*Elaeis guineensis*) with abundant production rate in Nigeria is regarded as huge amount of lignocellulosic waste and un-utilized. The waste from oil palm constitutes environmental nuisance which can be readily turned into valued-added products such as oil palm fibre reinforced polymer composite (OPFC) panels to meet various building needs. Oil palm fibre (OPF) is hard and tough but comes with a great challenge to overcome, their high susceptibility to flame when exposed to heat. The cellulosic content at 65% in OPF as reported by Suoware *et al.*, (2017) on flammability of flame retarded natural fibre composites shows that OPF when compared to other fibres will generate higher flammability risk. In addition, it is important to note that the polymer matrix is the primary source of flammable volatiles that consist of a complex mixture of gases and solid particulates from incomplete combustion. Hence, to reduce the flammability risk of the oil palm composite panels to meet current fire safety standards for various building applications, flame retardants (FR) are usually added to the panels during fabrication.

Flame retarded OPFC panels can be manufactured either by incorporating halogenated based or halogenated free of FR as well as a lignocellulosic fibre bonded by polymer matrix to obtain lightweight panels through different processing techniques as reported by Marjavaara *et al.*, (2009) and Virk *et al.*, (2012). The panels can be used for building interiors to delay the start and spread of fire. Recent studies have shown that FR such as aluminum trihydroxide (ATH) and ammonium polyphosphate (APP) are considered the most favourable FR in polymers because they are greener, highly effective and of low toxicity as reported by Subastinghe *et al.*, (2014), Yeonhae *et al.*, (2013), Suoware *et al.*, (2017) and Arjmandi. Kim *et al.*, (2014) studied the effect of a commercially available ATH on the flammability and smoke intensity properties of kenaf and oil palm fibre reinforced composites and obtained promising results. Ertugrul *et al.*, (2017) studied the effect of APP in combination with graphite in FR wood-polypropylene composite, the flammability properties (FP) obtained also showed some level of improvements. Other reports by Amina and Hassabo (2015), Gaelle *et al.*, (2013), Ricciardi *et al.*, (2012) and Hapuarachch (2009) also show that the combination of FR can achieve appreciable level of flame retardation.

The study of the fire behavior of OPFC panels has not been given the desired attention as seen from research works carried out using industrial and agricultural waste to produce particleboards and ceiling boards in Nigeria by Ikubanni *et al.*, (2018), Obam (2010) and Suoware *et al.*, (2019). In order to understand how effective, the FR on the fire behaviour and subsequently improve on the reaction to fire properties to meet required fire safety standards for building application. The APP was modified with gum Arabic powder (GAP) to form new intumescent FR species

and then hybridized with ATH along with a synergist carbon black (CB). The main objective in this paper is to develop and examine the FR effect on the flammability and thermal behaviour of oil palm composite panel.

MATERIALS AND METHODS

The testing of the OPFC panels was done at the Fire laboratory, University of Edinburgh, Scotland from January 2017 to April, 2017. First the samples; oil palm fibre were washed with n-Hexane to further remove impurities and treated with 5%NaOH to improve the compatibility of the fibres with the polyester resin. These were then sun dried for 3 days to remove moisture content. A wooden mould measuring (887.5 x 510 x 10) mm was used to prepare the test specimens for different test protocols according to ASTM E 1354. The internal area of the mould was applied with Vaseline for easy removal before oil palm fibres (OPF) were randomly inserted into the mould. A paste of FR poured over the OPF comprised of ATH with a chemical formula $[Al_2(OH)_3]$ of particle size $10\mu m$, APP with a chemical formula $[(NH_4PO_3)_n(OH)_2]$ a white-free flowing powder soluble in g/100ml of H_2O with average particle size of $15\mu m$, gum Arabic (GAP) and carbon black (CB). Six FR species as shown in table 1 were added to the total amount of polyester resin at 0, 12, 15 and 18% loading ratio. The OPFC panels were produced using hand lay-up compression moulding technique and cured at room temperature. The required quantities of the fibres and polyester resin used to produce the panels were obtained as shown in Equ.1.

$$V_f = \frac{M_f / \rho_m}{(M_f / \rho_m) + (1 - M_f) \rho_m} \quad (1)$$

Table 1: Formulation of flame retardant loadings in the fabricated composite panels

Specimen ID	OPF/Resin Ratio (wt. %)	% of FR Formulations*		
		ATH	APP/GAP (2:1)	CB
OPFC _{0%}	10/90	-	-	-
X ₀ :12%ATH	10/90	12	-	-
Y ₀ :12%APP-GAP	10/90	12	-	-
Z ₀ :15%ATH/CB	10/90	9	-	6
X ₁ :15%APP-GAP/CB	10/90	-	9	6
Y ₁ :18%ATH/APP-GAP	10/90	9	9	-
Z ₁ :18%ATH/APP-GAP/CB	10/90	9	6	3

*Formulation of flame retardant specified in percentage relative to the total amount of resin

[ATH: Aluminium Tri-hydroxide, APP: Ammonium polyphosphate, GAP: Gum Arabic powder, CB: Carbon black]

EXPERIMENTAL METHODS

Specimens cut from the OPFC were tested for thermal stability using thermogravimetric analysis (TGA/DSC 1; Mettler Toledo analyzer) at the University of Edinburgh as well as for flammability properties using the cone calorimeter apparatus by PL thermal sciences located in the fire laboratory of university of Edinburgh, Scotland.

THERMAL ANALYSIS

5g of OPFC samples were collected and then heated for 10min at 105°C under nitrogen gas (N₂) to determine moisture content; the temperature was then raised at 25°C min⁻¹ to 900°C where it remained for a further 10min to determine volatile matter content. Finally, air was introduced to the system combusting the sample (also at 900°C) for 20 minutes 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.

CONE CALORIMETER

The specimen were also tested for flammability properties such as ignition time (T_{ig}), peak HRR (HRR_p), average HRR (HRR_{avg}), total heat released (THR), effective heat of combustion (avg-E_{hc}), mass loss rate (MLR_p), specific MLR (SMLR_{avg}), residual mass (R_m), smoke production rate (SPR_{avg}), total smoke released (TSR), specific extinction area (avg-SEA), carbon monoxide production (COP) using the cone calorimeter apparatus. The specimens (100 x 100 x 10) mm was wrapped in aluminum foil; along the side and bottom to reduce heat losses. The specimens were inserted in a sample holder and then placed on the load cell which measures the specimen mass loss in real time as combustion takes place. The specimens were exposed in the horizontal orientation at heat flux of 50kW/m². An electric truncated cone shaped heater causes thermal decomposition and release pyrolysis products from the specimen surface. The height between the cone heater and the surface of the specimen is maintained at 25mm. Ignition is produced by an intermediate piloted spark igniter located above the specimen and removed when the pyrolysis products ignites. The process was repeated multiple times and a sensible result was recorded.

RESULTS

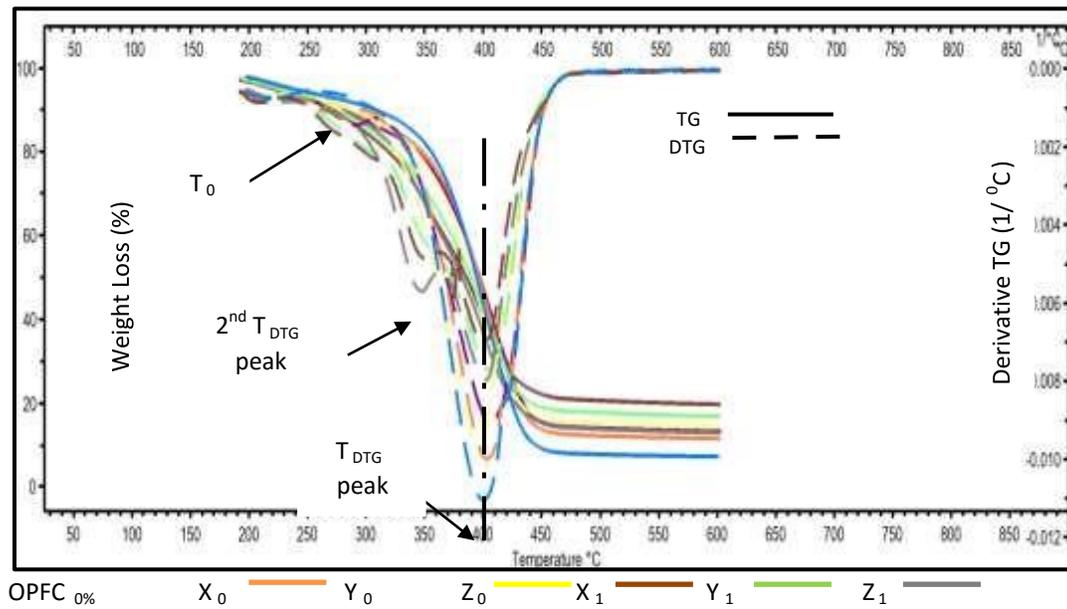


Figure 1: Thermogravimetric analysis (TGA) and derivative of thermogravimetric (DTG) curves of various flame retardant formulations in OPFC panel

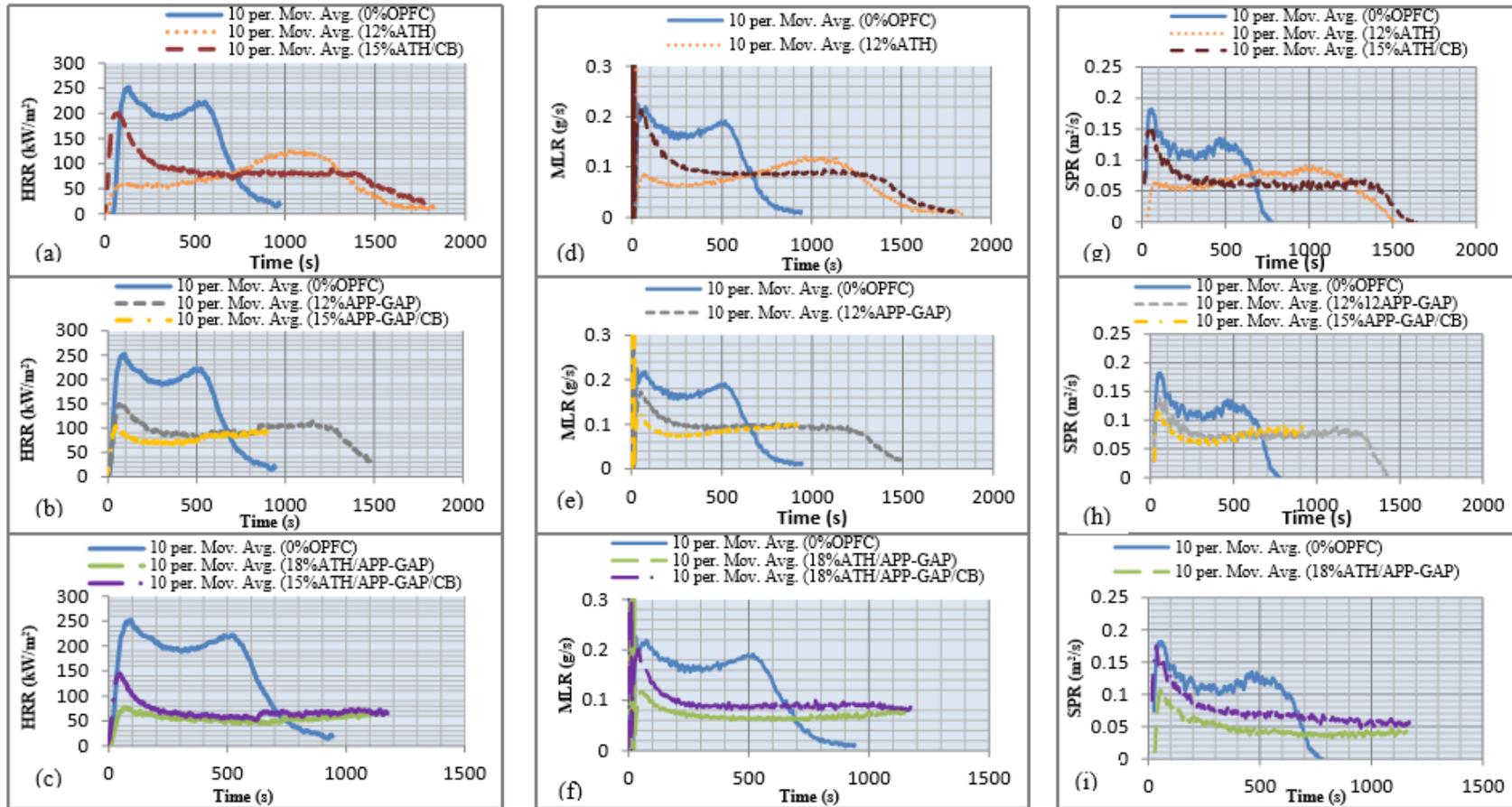


Figure 2: Comparison of the OPFC_{0%} panel with the FR formulation panels: (a-c) Heat release Rate, HRR (d-f) Mass Loss rate, MLR and (g-i) Smoke Production Rate, SPR at 50kW/m² Heat Flux

DISCUSSIONS

THERMAL STABILITY

In Fig 1, the sigmodal shaped TGA curves revealed the FR exhibited a typical single WL degradation. It also shows that the X_0 and X_I species in the OPFC panel exhibited better thermal stability as initial decomposition (T_0) began respectively around 376.3°C and 391.6°C which improved by 5.1°C and 20.48°C respectively higher when compared to the OPFC_{0%} panel at 371°C whereas the other FR deteriorated. This improvement could be caused by the constituents of oil palm fibre comprising cellulose of 65% and lignin of 29%. Lignin decompose early to form char that slow thermal decomposition. The Y_0 and Y_I panels exhibited the least thermal stability however implies that the FR decomposed early which is an indication of a good flame retardant as its early decomposition could trap the escape of combustible volatiles longer. The initial WL through to the end of the decomposition process falls within the temperature of (371°C-442°C) which agrees with the study of other researchers Dhandapani *et al.*, (2016) and Aisyah *et al.*, (2019) on OPEFB reinforced in bio- based polyester composite. Further thermal decomposition beyond T_0 to 900°C indicating the end of the test showed the X_I formulation of APP-GAP in the OPFC panel produced the highest char residue of 17.4% compared to OPFC_{0%} panel at 6.15%. The X_0 and X_I panels were fully degraded around 421.9°C and 426.9°C respectively higher than those without FR at 409.8°C.

FLAMMABILITY PROPERTIES

The HRR, MLR and SPR profile depicted in (Fig 2) shows that shortly after ignition, a sharp rise and then a sudden decline were observed this indicates the activities of combustibles gases during combustion. The broader appearance which stayed at a lower profile throughout the burning process suggests that the FR is interacting with the combustible products by oxidation starvation. This is also a typical characteristic of a char residue-forming material and confirms the role the lignocellulosic content the fibre played in the formation of char during combustion as reported by Mohammed *et al.*, (2020) and Haitang *et al.*, (2020). Besides, the last part of the HRR curves represents glowing combustion of the char residue at solid state interface (where volatiles have been burnt out) as reported by White *et al.*, (2011). It was observed that Y_I in the OPFC panel exhibited outstanding performance and consistency as it enhanced the entire flammability properties (FP). The FP were Tig = 20s, HRR_p = 86.6kW/m², HRR_{avg} = 55.8kW/m², THR = 36.7MJ/m², MLR_p = 0.131g/s, SMLR_{avg} = 7.1gs⁻¹, Rm = 54.6%, avg-Ehc = 8MJ/Kg, SPR_{avg} = 0.05m²/s, TSR = 2447.7m²/m², avg-SEA = 666.5m²/Kg and COP = 0.035Kg/Kg more those without FR (OPFC_{0%}) at Tig = 17s, HRR_p = 265.5kW/m², HRR_{avg} = 150.2kW/m², THR = 49.6MJ/m², MLR_p = 0.267g/s, SMLR_{avg} = 14.4gs⁻¹, Rm = 8.2%, avg-Ehc = 12.7MJ/Kg, SPR_{avg} = 0.08m²/s, TSR = 2733.7m²/m², avgSEA = 671.5m²/Kg and COP = 0.066Kg/Kg. this indicates the hybrid FR was effective in reducing the release of combustible volatiles and could meet current fire safety standards for building applications.

CONCLUSIONS

From the thermal stability analysis, it can be concluded that the panels did not exhibit any significant change when FR were added as early degradation was observed. However, the Y_I panel exhibited outstanding performance in delaying the early start of decomposition due to the decomposition mechanism of ATH/APP-GAP. The panel was

thermally stable at 391.6°C before degradation began compared to OPFC_{0%} at 371.2°C and was fully degraded at 426.9°C compared to OPFC_{0%} at 409.8°C. At 900°C which signifies the end of the test, the highest char residues were observed for X_I at 17.4% more than those without FR, confirming APP-GAP to be a good flame retardant. From the FP, it concludes that the Y_I panel slowed better the combustion process leading to outstanding performance of the entire flammability properties that could meet current fire safety standards. The peak HRR observed for Y_I panel indicates the intensity of fire and how grows was drastically reduced by 67.4% and the average HRR which is a more reliable measure of contribution to sustained fire was reduced by 62.8%.

ACKNOWLEDGEMENT

The authors would like to appreciate Tertiary Educational Trust fund (Tet fund), Nigeria through grant approval. African Centre of Excellence, ACE-SPED, University of Nigeria, Nsukka for their support and the management of Federal Polytechnic Ekowe for its travel approval and the technical staff of the Rushbrook Fire facility, John Mul Building, University of Edinburgh, Scotland United Kingdom.

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