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Development of flame retarded composite fibreboard for building applications using oil palm residue

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ABSTRACT: Residential housing is a critical aspect of human living and in developing countries this is a mirage due to high cost of building materials. In order to meet the needs for affordable housing with low cost materials as well as meet required fire safety standards, this research developed flame retarded fibreboards with oil palm residue reinforced in polyester resin, incorporating 0, 12 and 18% flame retardant loading using hand lay-up compression moulding. The fibreboards were tested for impact, thermal and flammability properties. Based on experiments, it was found that 12% aluminum tri-hydroxide fibreboard meets the impact and thermal limitations while the 18% hybrid formulation meets the required fire safety standard for building interior applications which will benefit rural dwellers in Nigeria and in similar climes around the world seeking to substitute conventional materials with the advantage of low cost, easy to process, biodegradable, environmentally benign and flame retarded composite material.

KEYWORDS: Composite; Characterization; Fibre reinforcement; Polymer; Filler

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RESUMEN: *Desarrollo de tableros de fibras de material compuesto con retardo a la llama utilizando residuos de aceite de palma, para aplicaciones de construcción*. La vivienda residencial es un aspecto crítico de la vida humana y en los países en desarrollo esto es un espejismo debido al elevado coste de los materiales de construcción. Con el fin de satisfacer las necesidades de viviendas asequibles con materiales de bajo costo así como cumplir con las normas de seguridad contra incendios, esta investigación desarrolló tableros de fibra ignífugos con residuos de aceite de palma reforzados con resina de poliéster, que incorporan una carga retardante de llama de 0, 12 y 18% utilizando la colocación manual y moldeado por compresión. Los tableros de fibra se ensayaron para determinar sus propiedades de impacto, térmicas e inflamabilidad. Basándonos en los experimentos realizados, se encontró que el tablero de fibra de tri-hidróxido de aluminio al 12% cumple con el impacto y las limitaciones térmicas, mientras que la formulación híbrida al 18% cumple con el estándar de seguridad contra incendios requerido para la construcción de aplicaciones interiores que beneficiará a los habitantes rurales de Nigeria y de climas similares en todo el mundo, buscando sustituir los materiales convencionales con la ventaja de un material compuesto de bajo costo, facilidad de procesamiento, biodegradabilidad, medio ambientalmente adecuado e ignífugo.

PALABRAS CLAVE: Composite; Caracterización; Refuerzo de fibras; Polímero; Filler

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1. INTRODUCTION

Developing countries around the world like Nigeria with a current population growth rate index of about 2.5% in the last five years (1) is being faced with challenges of affordable housing resulting from high cost of building materials. Today, most Nigeria's as a result of low income live in ramshackle buildings that are dehumanizing especially in remote areas where it is difficult to transport building materials couple with their high transportation cost. In order to address these challenges, researches have shown that conventional materials used in buildings that is for ceilings and walls, floors, doors etc. can be substituted with agricultural waste reinforced in polymers composites with similar properties to lower cost (2, 3). Nigeria is rich in abundant agricultural products and natural fibres such as coconut, banana, sugarcane, sisal, oil palm and wood sawdust when processed can generate huge waste in disposal sites for burning, and this can constitute health risk. It is evident from various researches conducted on the mechanical properties and thermal behavior that natural fibres reinforced polymer composites can be suitable for the development of building materials (4-7).

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 fibreboards to meet our building needs when the fibres are used as reinforcement in polymers. 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 in the researchers' review paper on flammability of flame retarded natural fibre composites (8) shows that oil palm fibre 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 fibreboard to meet required fire safety standards for various building applications, flame retardants (FR) are usually incorporated during fabrication.

Flame retarded composite fibreboard can be manufactured either by incorporating halogenated based or halogenated free types of flame retardants as well as a lignocellulosic fibre bonded by polymer matrix to obtain lightweight fibreboards through different processing techniques (9, 10). The fibreboards can be used in building applications to delay the start and spread of fire during an advent of fire outbreak. Recent studies show that halogenated free FR such as aluminum trihydroxide (ATH) and ammonium polyphosphate (APP) are considered the most favourable flame retardant in polymers because they are greener, highly effective and of low toxicity (11-14) when compared to over 150 chemical compounds available which flame retardants can be derived. Studies by various authors (15-17) have shown that the addition of aluminium tri-hydroxide in polymers when decomposed during combustion releases water vapour which dilutes the combustible gases of the polymer and at the same time form aluminum oxide (Al_2O_3) which acts as a barrier to the mass transfer of heat. On the other hand, ammonium polyphosphate is said to promote intumescent char layer which acts as physical barrier to slow the mass transfer of heat. Furthermore, ammonium polyphosphate may be detrimental to the physical and mechanical properties of the composite (6, 18) as well as increased smoke generation (19, 20). Although some research works have been developed from industrial and agricultural waste to produce particleboards and ceiling boards by various researchers in Nigeria (21-24), the study of their fire behavior has not been given the desired attention. Hence, this research will benefit building engineers in Nigeria and in other similar climes seeking to substitute conventional materials where their pressure is much with a more low cost, easy to process; biodegradable, environmentally benign and flame retarded composite material. In order to understand how effective the flame retardant is on the fire behaviour and subsequently improve on the reaction to fire properties to meet required fire safety standards for building application, this paper developed and examined oil palm composite fibreboard incorporating ATH, APP with Gum Arabic powder a new intumescent flame retardant and their hybrids.

2. EXPERIMENTAL APPROACH

2.1. Materials

The oil palm fibre used in the fabrication of the composite fibreboard was sourced within the University of Nigeria, Nsukka community. Flame retardants fillers consisting of aluminium tri-hydroxide ($Al_2(OH)_3$) of particle size 10μm and ammonium polyphosphate ($(NH4PO3)n(OH)_2$) a white-free flowing powder soluble in g/100ml of H_2O with average particle size of 15μm, n-Hexane, litmus paper, distilled water and the polyester resin used were supplied by Joe Chem ventures. Gum Arabic purchased in the Northern part of Nigeria was further processed into a fine powder of particle size 300 μm used as a binder and source of carbohydrate to formulate new flame retardant specie. Unsaturated polyester resin (UPR) was cured with 2% methyl ethyl ketone peroxide (MEKP) as catalyst and 1% cobalt (Co) as accelerator. All chemicals were used without further purification. Note that the exact chemical structure of UPR was note supplied by the manufacturer.

2.2. Fabrication of the Oil Palm Composite Fibreboard

Oil palm fibre (OPF) as received (Figure 1a) 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 (25) and then soaked in n-Hexane overnight to complete leaching and further remove impurities. The fibres were then treated with 5% (NaOH) solution for about 2hrs to avoid fibre damage as displayed in (Figure 1b) to improve the compatibility with the polyester resin. Afterwards, the fibres were washed with distilled water until blue litmus paper turned red which indicates that excess concentration of NaOH have been neutralized and then sun dried for 3 days to remove moisture content as shown in (Figure 1c). The ready to use fibres in (Figure 1d) were used to fabricate the fibreboard using hand lay-up compression moulding technique as shown in (Figure 1e). The required quantities of the reinforcement and polyester resin used to produce the fibreboards was obtained using mass fraction model (26) through equations (Eq. [1] to [3]) as shown below. Four (4) fibreboards were produced and investigated (Figure 1f). Table 1 illustrates the composition of the various oil palm fibre composite (OPFC) fibreboard produced including the percentages of the added flame retardants.

$$X_x + X_y + X_z \qquad [1]$$

Where, X is the Mass of the various constituents x, y, and z and the mass fraction can be obtained through the relationship denoted in Eq.[2] and Eq. [3].

$$V_f = \frac{M_f \rho_c}{\rho_f} \qquad [2]$$

or

$$M_f = \left(\frac{m_f}{m_f + m_{f2} + m_m} \right) \qquad [3]$$

V_f = volume fraction of fibre, M_f = mass fraction of fibre, ρ_c = density of the various constituents x, y and z (g/cm^3), ρ_f = density of the fiber and m_{f}, $_{f2}$, m = measured mass of fibre (s) and matrix respectively.

TABLE 1. Formulation of flame retardant loadings in the fabricated composite panels

Specimen ID	OFF/Resin Ration (wt. %)	% of FR Formulations*	
		ATH	APP/GAP (2:1)
0%OPFC Fibreboard	10/90	-	-
12%ATH	10/90	12	-
12%APP-GAP	10/90	12	-
18%ATH/APP-GAP	10/90	9	-

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

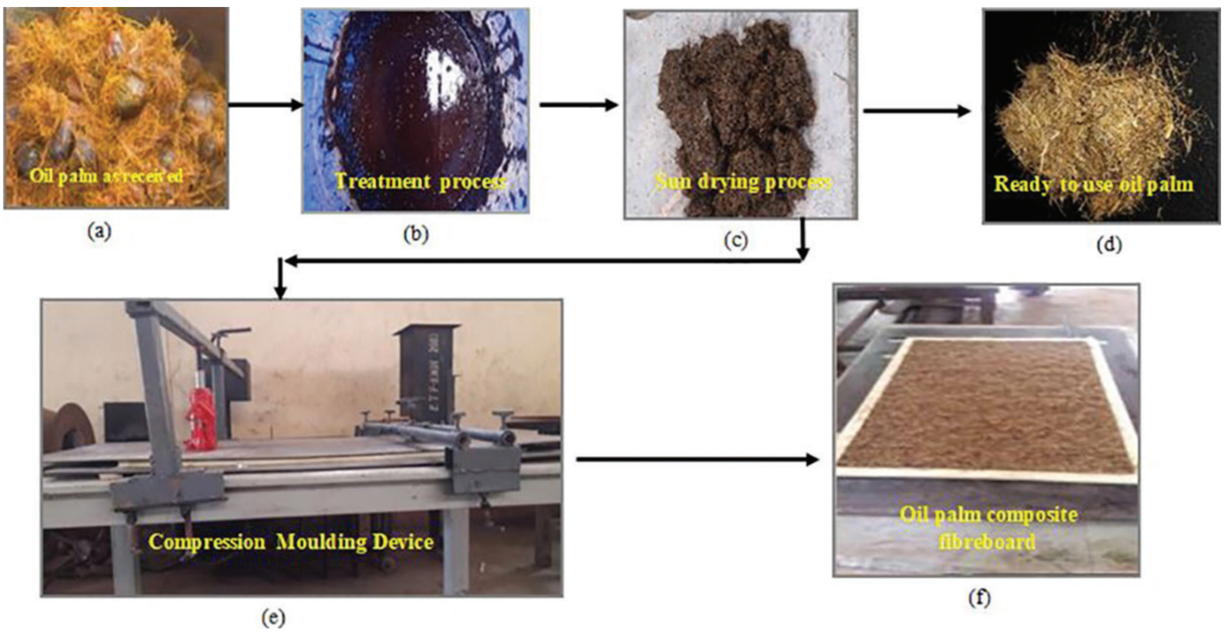

FIGURE 1. Fabrication process of the oil palm compose fibreboard using hand Lay-Up Compression Moulding.

2.3. Impact Strength

Izod impact test was performed on the fabricated composite fibreboard specimens using an impact tester in accordance with ASTM D 256 standards. From the fibreboard the IS dimension (100x10x10) mm was obtained. Prior to mounting on the test machine, the test specimen was notched to a depth of 2mm with a v-shaped hand file. The notched test specimen was then mounted on the impact-testing machine, which is operated to impact a blow to fracture the specimen at the opposite end of the notch by releasing the suspended handle of the pendulum swing. The impact strength measured as the absorbed before fracture was then read off on the calibrated scale. The specimens were repeated three (3) times and an average absorbed energy value recorded.

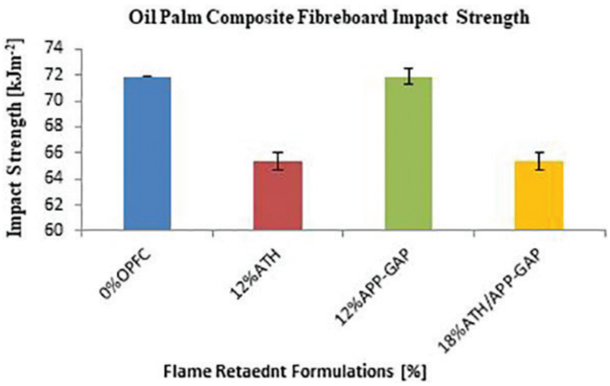

FIGURE 2. Graphs showing the influence of the various FR formulations on the impact strength of the OP composite fibreboard.

2.4. Thermo-gravimetric Analysis

TGA analysis of fibreboard was conducted using thermos-gravimetric analysis (TGA/DSC 1; Mettler Toledo, UKBRC, Edinburgh UK). A 5mg samples were first heated for 10min at 105^0C under nitrogen gas (N_2) to determine moisture content; the temperature was then raised at 25^0C min^{-1} to 900^0C where it remained for a further 10min to determine volatile matter content. Finally, air was introduced to the system combusting the sample (also at 900^0C) for 20 minutes in order to determine the ash content.

2.5. Flammability testing of the Fibreboard

The cone calorimeter apparatus was used to study the flammability (fire reaction properties) of the fabricated OPFC fibreboard according to ASTM E 1354. The specimens (100mm x 100mm x10mm) cut from the fibreboard were wrapped in aluminium foil; along the side and bottom to reduce heat losses as specified in the standard. The specimens were exposed to $50kwm^{-2}$ heat flux horizontal orientation. During testing, the time to ignition, heat release rate, mass loss rate, residual mass, smoke and toxic emissions were obtained. The test specimens were repeated severally and a sensible result was adopted for this study.

3. RESULTS AND DISCUSSIONS

3.1. Impact Strength

The results obtained for the impact strength as shown in (Figure 2) reveals that the absorbed energy of the OPFC fibreboard with FR did not show any improvement. In fact, the addition of $OPFC_{12\%ATH}$ and $OPFC_{18\%ATH/APP\text{-}GAP}$ in the fibreboard slightly deteriorated the amount of absorbed energy from $71.9kJ/m^2$ to $65.4kJ/m^2$ (9% decrease) compared to the $OPFC_{0\%}$. A similar trend in decrease has also been reported by Redwan et al (13) using different ATH content empty fruit reinforced/middle density board. It was however observed that the addition of $OPFC_{12\%APP\text{-}GAP}$ in the fibreboard maintained the same absorbed energy with the $OPFC_{0\%}$. This results agrees with the IS report by Nikmatin et al (27). In this study, the presence of GAP probably played a significant role in enhancing the fibreboard compactness and thus facilitated the transfer of stresses between the fibre, PR and the FRs during blow. The reason for the decrease in impact could be a non-uniform dispersion coupled with agglomerated FR particles which provides locations of stress concentrations, thus provides sites for crack initiations as reported by Subasinghe et al (28).

3.2. Thermal Behaviour of the Fibreboard

In Table 2, the parameters describing the thermal response of the OPFC are presented which was derived from the thermogravimetric (TGA) and derivative of TG (DTG) curves as shown in Figure 3. From the sigmoidal shaped curves, it reveals that the OPFC is typical single weight loss step degradation. The initial degradation process of the fibreboards was noted at 200^0C, indicative of the loss of water vapour from the fibres as well as confirms the hydrophilic nature of the fibres (29, 30). As the heating continues the fibreboard began to lose weight until it reaches the actual degradation of the fibreboards around the onset decomposition temperature (T_0) before degrading sharply noted at the peak DTG. The overall degradation process reveals that the $OPFC_{12\%ATH}$ addition in the fibreboard exhibited a better thermal stability as the T_0 reached 376^0C from 371^0C and the peak DTG reached 421.93^0C from 409.82^0C compared to the $OPFC_{0\%}$. Similar reports for the increase in thermal stability with ATH in oil palm empty fruit fibre reinforced epoxy was found in the work of Khalili et al (7). The increase in thermal stability agrees with the findings of Happarachchi (31), attributed to the endothermic dehydration and the subsequent release of 35% of H_2O crystallization into the gas phase which led to the formation of a ceramic layer of γ-Al_2O_3. It was observed that the $OPFC_{18\%ATH/AP-GAP}$ hybrid formulations gained in weight loss and char residue which stood at 83.3% and 15.46% compared to the $OPFC_{0\%}$ at 92.7% and 6.15% respectively. This suggests a slower decomposition and significantly enhanced thermal stability as well as a superior advantage in terms of their flame retardancy. ATH and APP upon reaction could be responsible for the high char formation as reported in the work of (32).

3.3. Flammability properties

The fire risk of the fibreboard was assessed through its time to ignition (Tig) response which is responsible for the fibreboard flame spread.

Table 2. Results data on thermal stability and degradation of oil palm fibre composite

Specimen I. D	T_0 (°C)	WL (%)	T_{DTG} peak (°C)	RC (%)
0%OPFC Fibreboard	371.15	92.7	409.82	6.15
12%ATH	376.25	88.18	421.93	10.62
12%APP-GAP	352.28	87.84	412.70	10.53
18%ATH/ APP-GAP	354.59	83.30	415.27	15.46

Table 3, shows the various ignition times obtained in the cone calorimeter at $50kWm^{-2}$. It reveals that the $OPFC_{12\%ATH}$ and $OPFC_{18\%ATH/APP-GAP}$ fibreboards delayed longer the release of combustible volatiles. The combined effect of tri-hydroxide (ATH) and ammonium polyphosphate and Gum Arabic powder (APP-GAP) could be responsible for the delay in Tig which is attributed to strictly the thermal decomposition of ATH into (Al_2O_3 and H_2O) since water vapour dilutes the volatile gases being formed. This may have cooled the gases and delay Tig. Another reason could be that APP-GAP dehydrates the fibreboard upon heating to form char that prevents the release of combustible volatiles.

The HRR profile of the OPFC obtained from cone calorimeter are depicted in (Figure 4). The heat release rate (HRR) is a key property in evaluating the fibreboards limits to fire safety. From the heat release rate curves it is clearly seen that shortly after ignition, a sharp rise and then a sudden decline was observed for all the fibreboard types which indicate the activities of the combustible products during combustion. It was observed that the $OPFC_{0\%}$ exhibited a double peak HRR, indicative of a high release of combustible rate probably caused by cracks in the char structure of the underlying composite fibreboard substrate as reported by Schartel and Braun (33). When flame retardant was added to the fibreboard it reveals a broader appearance which stayed at a lower profile throughout the burning process, indicative of the flame retardant interaction with the combustible products. In (Table 3) it was found that the addition of $OPFC_{18\%ATH/AP-GAP}$ followed

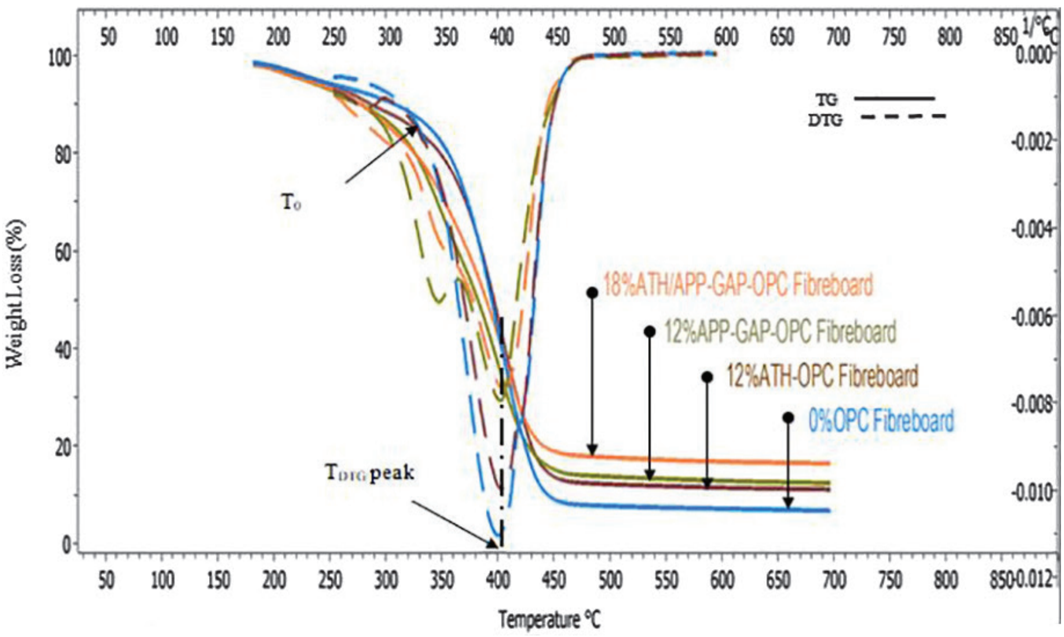

Figure 3. Thermogravimetric analysis and derivative of thermogravimetric curves of various flame retardant formulations in OPFC fibreboard.

TABLE 3. Summary of Flammability Properties of OPFC Panel obtained in the cone calorimeter

Specimen I. D	T_{ig} (S)	HRRavg (kW/m^2)	HRRp (MJ/m^2)	SMLRavg (gs^{-1}/m^{-2})	Rm (wt. %)
0%OPFC Fibreboard	17	150.2	265.5	12.7	8.2
12%ATH	21	67.6	136.5	6.9	11.9
12%APP-GAP	11	93.4	158.3	9.2	14.9
18%ATH/APP-GAP	20	55.8	86.6	7.1	54.6

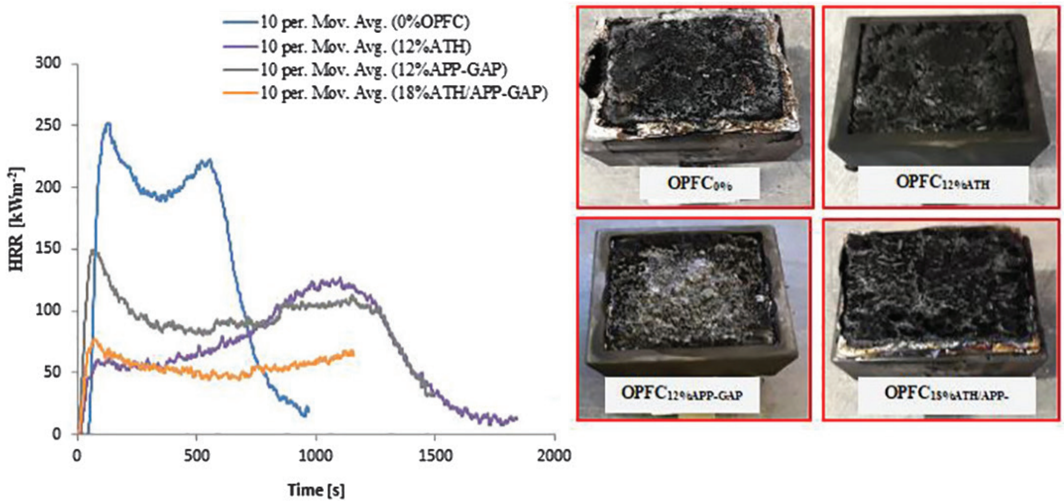

FIGURE 4. Comparison of $OPCF_{0\%}$ heat release rate (HRR) curves with FR formulations at $50kW/m^2$: Photographs show macroscopic images at the end of test.

by $OPFC_{12\%ATH}$ flame retardants in the fibreboard exhibited an outstanding performance as the peak HRR and average HRR indicating the intensity of fire and the contribution to sustained fire respectively were remarkably reduced to $86.6kWm^{-2}$ and $136.5kWm^{-2}$ from $265.5kWm^{-2}$ and $55.8kWm^{-2}$ and $67.6kWm^{-2}$ from $150.2kWm^{-2}$ respectively compared to $OPFC_{0\%}$. This could be elucidated by the effect of ATH and APP-GAP characteristic mechanism. Aluminium tri-hydroxide acts to trap the formed combustible products during the burning process, releasing water during dehydration and restricting access of oxygen to the fibreboard substrate. This agrees with the HRR profile found in the report of Nikolaeva and Karki et al (34). On the other hand, APP-GAP inhibits the mass and volatile transfer between the condense phase and gas phase once a char layer is formed, thus it contributes to the reduction in heat release rate.

The MLR of the fibreboard which is a measure of the dehydration reactions and pyrolysis was examined and presented as shown in (Table 3). It can be deduced from the specific MLR on average (SMLRavg) that $OPFC_{12\%ATH}$ followed by $OPFC_{18\%ATH/APP-GAP}$ were the lowest at 6.9 and 7.1 $gs^{-1}s^{-2}$ respectively, indicative of slow decomposing fibreboard compared to $OPFC_{0\%}$ which led to an enhanced residual mass (Rm) and suggest a good flame retardancy. In fact, the greater the decrease in SMLRavg, the better the reduction in the peak HRR was observed, this has also been confirmed by other authors (34-36). The combustion residue displayed for the fibreboards on the right hand side of (Figure 4) after combustion further reveals that $OPFC_{12\%ATH}$ left a mass of smoother black char and thin cracks indicating a better restrain of the combustion volatiles while the $OPFC_{18\%ATH/APP-GAP}$ with a fully covered, uniformly coherent and compact char residue left a mixture of gray and black mass of char indicating a rich carbonaceous char formation and the reason it attained the lowest peak HRR ($86.6kWm^{-2}$) and higher amount of Rm (54.6%). This implies that both mass and heat transfer between condense phase and gas phase was restricted and consequently the underlying material protected from further combustion of the polymer pyrolysis.

3.4. Smoke and Gas Parameters

The importance of the smoke release accompanied by toxic gases is a critical aspect of human survival during a fire as inhalation is one of the greatest hazards to life; hence the knowledge of the smoke

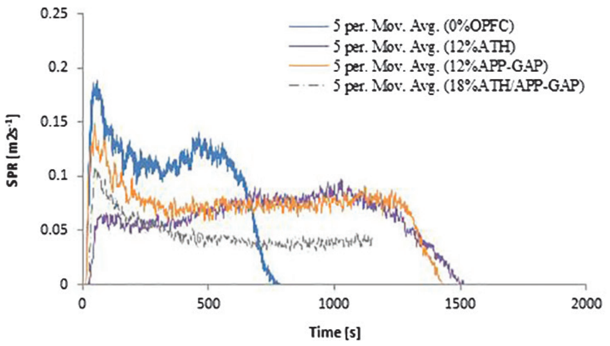

FIGURE 5. Comparison of smoke production rate curves of $OPCF_{0\%}$ with various FR formulations.

TABLE 4. Summary of Smoke and Gas Properties of OPFC Panel obtained in the Cone Calorimeter

Specimen I. D	SPRavg (m^2/s)	TSR (m^2/m^2)	av-SEA (m^2/Kg)	COP (Kg/Kg)
0%OPFC Fibreboard	0.08	2733.7	671.5	0.066
12%ATH	0.05	3604.8	613.6	0.069
12%APP-GAP	0.07	4644.6	780.5	0.036
18%ATH/ APP-GAP	0.05	2447.5	666.5	0.035

and gas release of the fibreboards is imperative. In (Figure 5), the smoke production rate (SPR) of the fibreboards as a function of time exhibits similar characteristics trend with those of the HRR curves and suggests that the release of smoke accompanied with toxic gases (Carbon-monoxide production, COP) as heat is evolved into the atmosphere. In (Table 4), it was observed that among the studied flame retardants in the fibreboard, the addition of $OPFC_{18\%ATH/APP\text{-}GAP}$ in the fibreboard caused a positive impact on the smoke and gas properties. $OPFC_{18\%ATH/APP\text{-}GAP}$ maintained the same SPRavg with the $OPFC_{0\%}$ at $0.05m^2s^{-1}$ while the total smoke released (TSR) was suppressed to $2447.5m^2m^{-2}$ from $3604.8m^2m^{-2}$ (32% decreases). From the carbon-monoxide production data, it shows that the entire flame retardants presence in the fibreboard suppressed the COP with $OPFC_{12\%APP\text{-}GAP}$ the least at 0.029Kg/Kg followed by $OPFC_{18\%ATH/APP\text{-}GAP}$ at 0.35Kg/Kg from 0.069Kg/Kg representing 58% and 49.2% respectively. The fibreboards however show that the av-SEA did not improve the entire FR studied. This could be attributed to the longer burning time of the FR fibreboard compared to the $OPFC_{0\%}$.

4. CONCLUSIONS

This research has shown that flame retarded fibreboards from oil palm residue reinforced in polyester resin can be fabricated using hand lay-up compression moulding. The results obtained from experimental observations shows that the addition of $OPFC_{12\%ATH}$ exhibited the most favourable IS of $71.9kJm^{-2}$ as it did not deteriorate compared to $OPFC_{0\%}$ which can meet interior furnishing in building. The thermal behaviour of the $OPFC_{12\%ATH}$ also improved slightly by 5.1^0C, indicating the limitation in use. In contrast, the $OPFC_{18\%ATH/APP\text{-}GAP}$ exhibited outstanding performance in flammability properties indicating low level of fire intensity and contribution to sustained fire of the fibreboards. The HRRp, HRRavg, SMLRavg and Rm improved by 67.4%, 62.8%, 44.1% and 54.6% respectively while smoke and toxic emission were suppressed better by the $OPCF_{12\%APP\text{-}GAP}$ and $OPFC_{18\%ATH/APP\text{-}GAP}$ formulations. With the main objective of this research, the fibreboards with $OPFC_{18\%ATH/APP\text{-}GAP}$ is the most suitable to meet required fire safety standards for interior building applications.

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