



Fabrication of Oil Palm Fibre Polymer Composite Panel: Impact of Hybridized Flame Retardant Formulations on Thermo-Mechanical Properties

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Abstract

Various oil palm fibre reinforced polyester composite (OPFPC) panels has been fabricated with different FR formulations at increasing percentage loading using hand lay-up compression moulding technique. The effects of aluminium hydroxide (ATH), ammonium polyphosphate (APP), their hybridization and synergism with carbon black (CB) on the mechanical and thermal properties have been investigated. The results obtained showed that 12% ATH inclusion in the OPFPC enhanced the tensile strength (TS), tensile modulus (TM) and flexural strength (FS) relative to 0%OPFPC panel by 16.2%, 5.9% and 71.2%, respectively. The inclusion of 15%APP-GAP/CB decreased in TS and FS by 18.8% and 20.8% respectively while 18%ATH/APP-GAP/CB hybrid formulation decreased the most by 28.1% in TM showing poor performance in the presence of CB. TGA results reveals that the inclusion of 12%ATH and 15%APP-GAP/CB improved the thermal degradation relative to the 0%OPFPC by 5.1°C and 20.48°C respectively while other FR formulations exhibited a decrease. Char residue at 900°C which signifies the end of the test increased significantly for all the FR formulations with 15%APP-GAP/CB observed as the highest at 17.47% and 12%APP-GAP as the least at 10.53%. The inclusion of 12%ATH in the OPFPC panel suggest a better mechanical and thermal stability while the inclusion of 15%APP-GAP/CB suggest a better flame retardancy owing to the highest char formation.

Keywords: Hybridization; Mechanical properties; Oil palm fibre; Polymer; Thermal properties

Introduction

The quest for high-performance materials made from natural sources such as lignocellulosic based fibres obtained from plants 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]. Oil palm fibre (OPF) is a form of lignocellulosic residue in abundant waste which constitutes environmental nuisance. It can be harnessed as reinforcement in polymeric composites for the production of various components credited to several authors working on the suitability of OPF polymer composite [4,5]. OPF derives its strength and rigidity like any other natural fibres such as jute, sisal, flax, banana, coir, wood etc. from cellulose that is semi-crystalline polysaccharide in nature. However, their high susceptibility to flames due to the presence of cellulosic content in the fibres and the polymer, which are combustible in nature has limited their broad use especially in areas such as the aerospace, boats and ground transportation where there fire behavior is unknown. 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.

Quite a number of FRs has been studied in natural fibre polymer materials which can delay the start of a fire and even mitigate its spread [6-8]. 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 [9,10]. However, recent study by Khalili et al. [6] 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 Subastinthe and Bhattacharyya [7] agree 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 is not studied quite extensively. Hence, this paper is aimed at formulating ATH, a new intumescent FR with g/Gum Arabic powder (GAP) and their hybrids in OPFPC at different FR loadings and analyzes the effect on mechanical and thermal properties using universal testing machine and TGA respectively. This would provide an insight on the effect of ATH and APP hybrids in OPFPC.

Experimental Methods

Materials

Oil palm fibres used in this work were obtained from neighbouring communities in Nsukka, Southern part of Nigeria. Polyester resin and other reagents such as methyl ethyl ketone peroxide, cobalt, aluminium hydroxide (ATH), and ammonium Polyphosphate (APP), NaOH, n-Hexane and carbon black (CB) were all locally sourced and purchased from within Enugu State, southern Nigeria. GAP was purchased from Adamawa market in the Northern part of (Nigeria).

Preparation of the OPFPC Panel

OPF obtained were first extracted by washing with hot water to remove the remaining residual oil retained during oil extraction, a

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Received July 25, 2018; Accepted July 31, 2018; Published August 11, 2018

Citation: Suoware TO, Edelugo SO (2018) Fabrication of Oil Palm Fibre Polymer Composite Panel: Impact of Hybridized Flame Retardant Formulations on Thermo-Mechanical Properties. J Material Sci Eng 7: 472. doi: [10.4172/2169-0022.1000472](https://doi.org/10.4172/2169-0022.1000472)

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method proposed by Vijaya et al. [11] 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 as shown in Figure 1. 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 [12].

Tensile testing

Samples cut from the OPFC panel (Figure 1) 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 position 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.

Flexural testing

Flexural test was also performed with the universal testing machine (Hounsfield model No.8898), using a 3-point bending method according to ASTM D790 procedure. 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.

Thermogravimetric analysis

The analysis of all the samples was carried out using thermogravimetric analysis (TGA/DSC 1; Mettler Toledo, UKBRC,

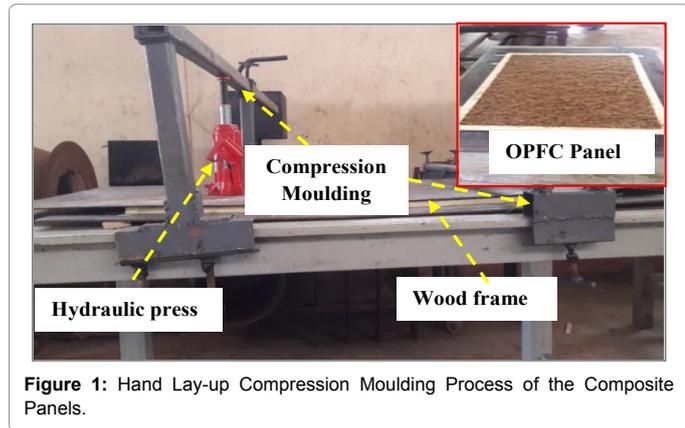


Figure 1: Hand Lay-up Compression Moulding Process of the Composite Panels.

| Specimen I.D | OPF/Resin Ratio (wt. %) | % of FR* | | |
|--------------------|----------------------------|----------|-----------|----|
| | | 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 |

*Formulation of flame retardant species specified relative to the total quantity of resin.

Table 1: Experimental Design of the Flame Retardant OPF Composite Panels.

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 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.

Results and Discussions

Mechanical properties

Hybrid-FR formulations at different percentage loadings and 10% fixed OPF loading was used to fabricate the composite panels. Reports by Beg et al. [13] showed that at 10 wt. % fibre content, maximum tensile strength can be obtained with empty fruit bunch fibre. The influence of the FR formulations on the mechanical properties was then evaluated and the results on average are presented in Table 2.

Tensile Properties: From Table 2, it can be seen that the effect of 12%ATH inclusion in the OPFC panel is positive, indicating an improvement of tensile properties relative to % OPFC panel. Contrary to this trend, the addition of the intumescent FR and hybridized FR formulations showed negative which implies a decrease in tensile properties but improved in TM for 15%APP-GAP/CB and 18%ATH/APP-GAP. The graphs showed in Figure 2a decreased in TS with increasing IFR and hybrid FR formulations while Figure 2b exhibited an intermediate behaviour which implies that the FR formulations did not follow any particular other of FR increase. The decrease caused by the presence of APP is expected as it agrees with the report of Bocz et al. [14]. Similar trend have been reported for OPFC as seen in the work of Khalili et al. [6], Redwan et al. [15] and Norzali et al. [16]. 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 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. The enhanced TS observed 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. The decrease in the tensile properties is expected and agrees with the reports of Subasinghe and Bhattacharyya [7], and Shukor et al. [17] on the detriments of FR to composites.

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 Figure 3. 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 the FR-OPFC panels could be explained as having a better covalent bonding

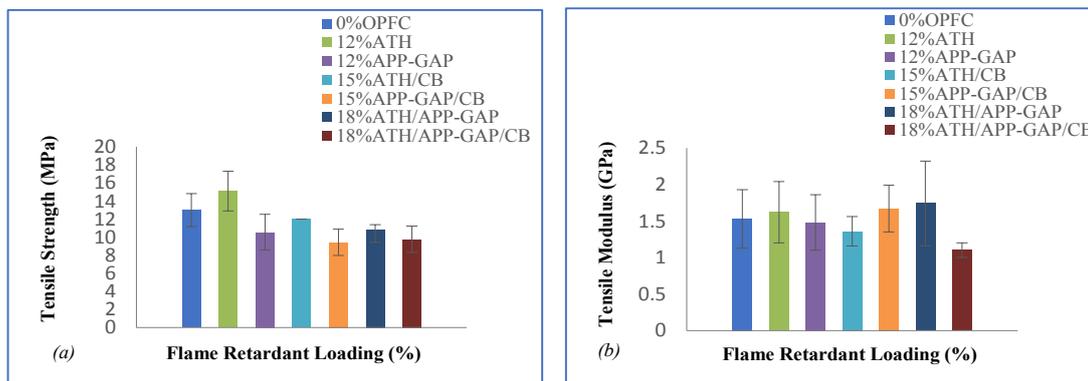


Figure 2: Graphs showing the influence of the various FR formulations in OPFC panel: (a) tensile properties (b) tensile modulus.

| Specimen I.D | T.S | Effect | T.M | Effect | F.S | Effect |
|-------------------|--------------|--------|-------------|--------|---------------|--------|
| | (Mpa) | (%) | (Mpa) | (%) | (Gpa) | (%) |
| 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 |

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

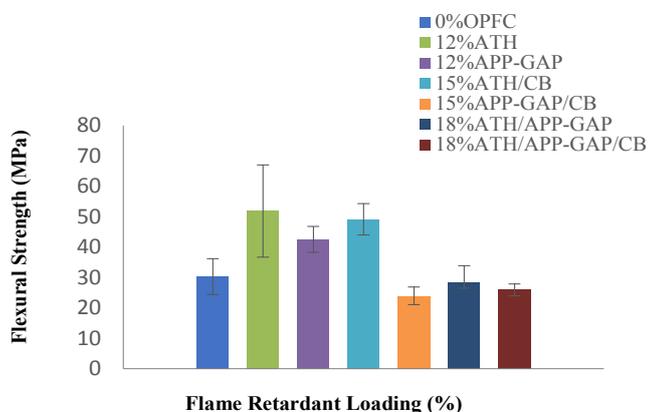


Figure 3: Flexural strength data for OPFC panels at varying flame retardant loadings.

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 [18-20]. On the hand, 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.

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 Figure 4, it showed that the overall thermal

decomposition of the OPFC panel began around 150°C, indicating loss of water from the fibres credited to several authors [21-23]. A sharp drop within the range of 200°C to 350°C indicates the loss of hemicellulose followed by pyrolysis of cellulose within a temperature range of 350°C to 450°C. The degradation of lignin stretches within a long temperature range (150°C to 900°C) which leaves solid charred residue at the end of the test. 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. [24] which suggest that the presence of OPEFB enhances thermal stability of polymer composites. When 12%ATH and 15%APP-GAP/CB was 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

| Specimen I.D | T.S | TE | WL | T _{DTG peak} | Char residue |
|-------------------|--------|--------|-------|-----------------------|--------------|
| | (°C) | (°C) | (°C) | (°C) | (°C) |
| 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 |

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

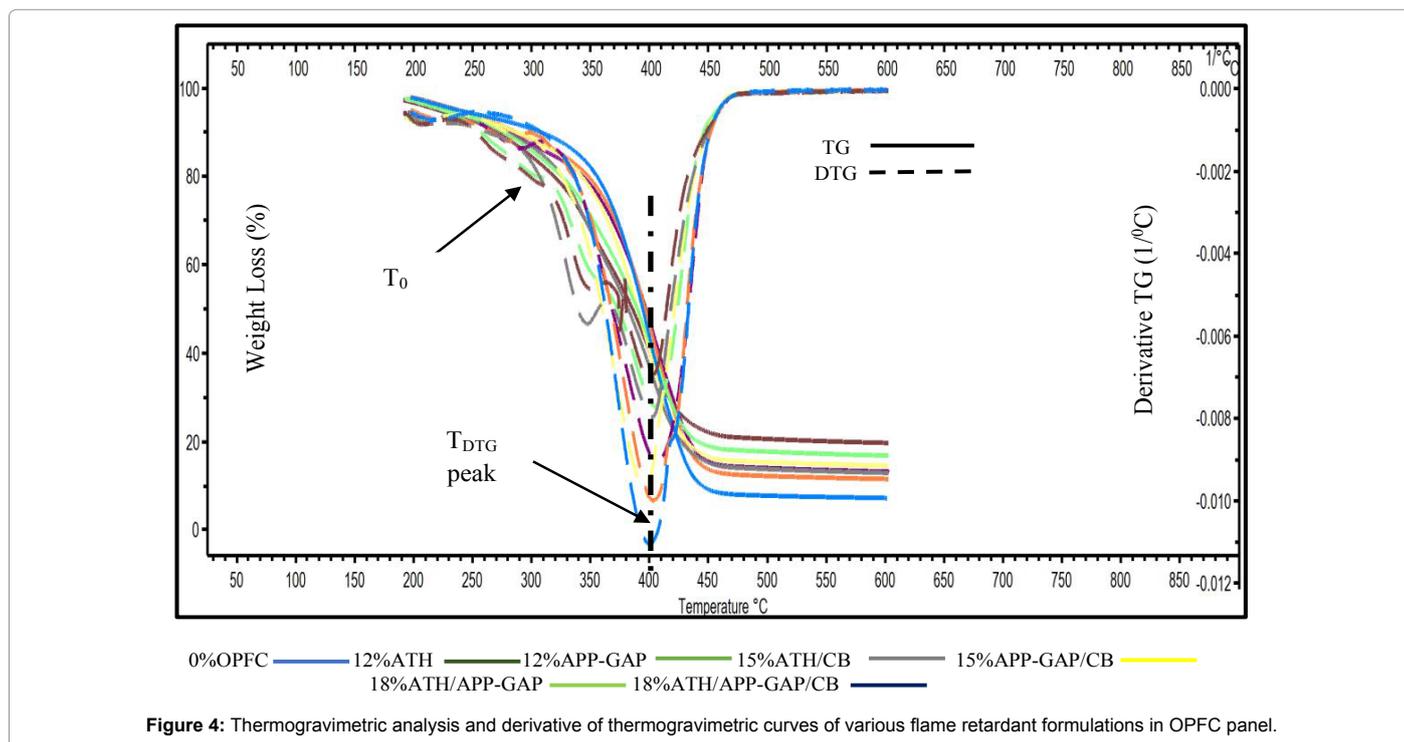


Figure 4: Thermogravimetric analysis and derivative of thermogravimetric curves of various flame retardant formulations in OPFC panel.

water vapour due to its endothermicity and converts the thermal stable aluminium oxide that demonstrates very high melting temperature [25]. On the hand, the presence of APP could be attributed to the findings by Alam et al. [26]. 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 Figure 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 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) biocomposties was found in the work of Siyamak et al. [27] and with IFR in kenaf fibre reinforced polypropylene by Subasthinghe and Bhattacharyya [7]. 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 (Figure 5). 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. [28] and Le Bras et al. [29]. Also, the loss of absorbed and structural water in GAP [30] may be have been involved in the early degradation.

Conclusions

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

1. ATH at 12% loading in the OPFC panel enhanced in TS, TM and FS with maximum values at 15.11 MPa, 1.62 MPa and 51.86 GPa respectively. This is attributed to the interfacial adhesion of the ATH and PR as well as having great affinity in PR.
2. Further increase from 12% to 18% FR loadings and their hybrid formulations decreased in TS and increased in TM for 12%APP-GAP, 15%ATH/CB and 18%ATH/APP-GAP/CB. The FS exhibited a decrease for 15%APP-GAP/CB, 18%ATH/APP-GAP and 18%ATH/APP-GAP/CB while an increase for

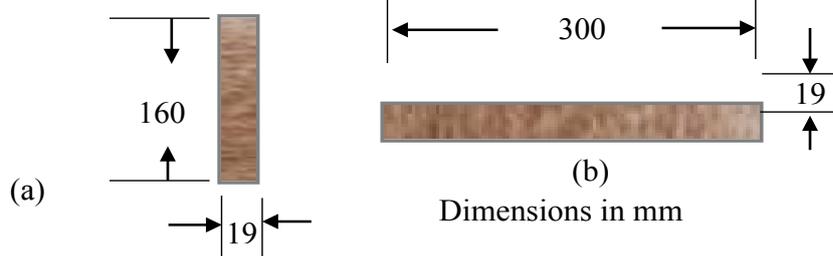


Figure 5: (a) Tensile measurement (b) Flexural measurement.

12%ATH, 12%APP-GAP and 15%ATH/CB was observed, indicating that the addition of the synergist CB and hybrid formulations decreased the overall mechanical properties with increase in FR loadings.

3. TG and DTG analysis showed that 12%ATH and 15%APP-GAP/CB exhibited improved thermal stability when compared to 0%OPFC. The reason is because ATH dehydrates endothermically upon heating and the presence of CB in APP-GAP restricts the mobility of the PR to become slower.
4. TG and DTG analysis showed that 12%APP-GAP, 15%ATH/CB, 18%ATH/APP-GAP and 18%ATH/APP-GAP/CB were less thermally stable when compared to 0%OPFC, indicating poor performance of the hybrid formulations at increase FR loadings. This could be attributed to the nature of APP upon thermal degradation which is responsible for the premature destruction of the PR caused by water hydrolysis.
5. The inclusion of the FR formulations improved significantly the char residue at the end of the TGA test with the highest char formation observed at 15%APP-GAP/CB formulation and suggests a better flame retardancy.

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