Flammability of self-extinguishing kenaf/ABS nanoclays composite for aircraft secondary structure

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Abstract. This study investigates the flammability properties of kenaf fiber reinforced acrylonitrile butadiene styrene (ABS) with nanoclays composites. Natural fiber is one of the potential materials to be used with thermoplastic as a composite due to its attractive properties such as lightweight and strong. In this paper, flammability properties of this material are evaluated through Underwriters Laboratory 94 Horizontal Burning (UL94 HB), which has been conducted for both controlled and uncontrolled conditions, smoke density and limiting oxygen index tests (LOI). These flammability tests are in compliance with the Federal Aviation Regulation (FAR) requirement. The results from UL94 HB and smoke density tests show that the presence of nanoclays with effective composition of kenaf fiber reinforced ABS has enhanced the burning characteristics of the material by hindering propagation of flame spread over the surface of the material through char formation. Consequently, this decreases the burning rate and produces low amount of smoke during burning. On contrary, through LOI test, this material requires less oxygen to burn when exposed to fire, which hinders the enhancement of burning characteristics. This is due to burning mechanism exhibited by nanoclays that catalyzes barrier formation and flame propagation rate over the surface of the biocomposite material. Overall, these experimental results suggest that this biocomposite material is capable of self-extinguishing and possesses effective fire extinction. The observed novel synergism from the result obtained is promising to be implemented in secondary structures of aircraft with significant benefits such as cost-effective, lightweight and biodegradable self-extinguishing biocomposite.

1. Introduction
In recent years, the novel approach of using natural fibre reinforced thermoplastic material is receiving increased attention in many applications such as housing, automotive, aerospace and other low cost, high volume applications [1]. Hence natural fibre composites are emerging as realistic alternatives to synthetic fibre composites. This transformation is due to attractive properties exhibited by the natural composites fibre such as lightweight, environmental friendly, vast source of production, and reasonable strength and stiffness [2, 3]. However, knowledge on flammability properties of natural fibre reinforced thermoplastic material has not been broadly researched. Flammability is defined as the reaction of particles in order to
produce fire processes of a material, and this reaction can be classified into several different phases such as ignitability, flame spread, heat release, and resulting products from fire process [4]. Since fire issue is becoming the major concern in aerospace industry, a new biocomposite material that has the capability to act as a self-extinguisher is being popularly explored by researchers. Attention in reducing the fire risk and hazard to both human and structures is leading to the development of newer composite materials that can reduce fire accidents [5]. Flammability is a serious issue in aerospace industry applications, which needs a meticulous attention particularly in the area of structures since small or confined space is exposed to fire hazard [6]. Fire risk in aviation sector has been identified ever since the Wright’s brother built the aircraft. Looking back to past occurrences, the first fatal accident in aviation occurred due to an uncontrolled in-flight fire [7, 8]. Even though continuous improvement on fire safety of aircraft such as airworthiness requirement, manufacturing and maintenance standards, design of protective and emergency equipment, and improved procedures and training for flight crew and cabin crew has been taken, in-flight fire and smoke still occur in transport aircraft [7]. Aviation history proves that, apart from the loss of control, controlled flight into terrain and specific component failure, aircraft fire accidents is one of the top four leading fatalities [8]. Statistic shows that from 1990 to 2010, 18 major in-flight fire accidents occurred that lead to 423 fatalities [9].

There are different types of flammability testing techniques for investigating flammability properties of a material. However, aerospace sectors are subjected to strict rules and regulation when it comes to safety matter. The requirement for interior application must satisfies the FAR Part 25 Airworthiness Standards in Transport Category Airplane Subpart D regulation for interior application, which are FAR 25.853 (fire protection for compartment interiors) and FAR 25.856 (fire protection for thermal and acoustic insulation materials) [10]. Besides that, according to the requirements for biomaterials and biodegradable materials TA11-01-0, the material must conform to FAR 25.853 and FAR 25.856 for interior application [11]. Therefore, flammability testing techniques that comply with the standard requirement of FAR are the Underwriters Laboratory 94 horizontal burning (UL94 HB), smoke density and limiting oxygen index tests (LOI). These fire testing techniques evaluate various burning characteristics of biocomposites by considering the targeted applications. The burning characteristics that are being evaluated in these tests include burning rates, flame propagation, flame drips, smoke production and oxygen concentration [12].

A study has stated that kenaf fiber is one of the commemorate constituents of natural fibre reinforced plastic composites in Malaysia [13]. The physical property of kenaf, which is in long fibre form, enhances the mechanical properties and this becomes an advantage as it can be used in many industrial applications such as insulators seals [14]. However, since kenaf fibre constitutes of cellulose, hemicellulose and lignin, it is highly flammable and has a poor fire resistance due to the poor compatibility of hydrophobic polymer matrices [3-14]. Recently, kenaf has been used in conjunction with various matrices like polypropylene (PP), polyethylene (PE), high density polyethylene (HDPE), polylactic acid (PLA), polyvinyl chloride (PVC), phenol formaldehyde (PF), polyester and epoxy(EP) [15]. Due to high demands on plastic industry for petroleum-based material production, a vast investigation and research study is carried out on plastic composite including ABS [3-16]. ABS is a type of engineered polymers with an advantage of higher mechanical and thermal properties. When pure ABS is exposed to fire, it is easily burned with a luminous yellow flame, producing black soothe and smoke, and continues to burn even after removal of the ignition fire source [17]. Table 1 shows flammability data of polymeric materials based on LOI and UL 94 rating.

The major responsibility of flame retardant agent is to prevent, cease or minimize the combustion of a material. There are many types of flame retardant including phosphorus, halogen, silicon, nanometric particles and mineral additives. In this present work, nanoclay is being used as flame retardant and it can be classified as nanometric particles. Bonati [18] stated that as flame retardant agent, nanoclays possessed the capability to inhibit or stop the polymer combustion as it can either act physically such as by cooling through formation of a protective layer or chemically such as reaction in the condensed or gas phase.
Chigwada [19] researched the effects of fire behavior on ABS, PE and epoxy with incorporation of the nanoclays through cone calorimeter. The result showed that with nanoclay, the maximum heat release rate decreased while the ignition time increased during fire. On the other hand, Saba [20] concluded that there are two main flame retardant mechanisms in ABS polymer and kenaf with nanoclays composites. First mechanism is the formation of a barrier against heat and volatiles through the migration of the nanoclays towards the surface of the material. The second mechanism is the char formation between the surface of the material and the flame [21, 22]. Furthermore, Gacitua [22] stated that a small amount of nanoclays incorporation with the biocomposites can improve the flammability properties of biocomposite. Therefore, it can be considered as high-potential filler material for improving burning characteristic of biocomposite. It is thus concluded that addition of small amount of nanoclay into the polymer matrix can improve the flammability properties of biocomposites.

Table 1. Flammability data of polymeric material based on LOI and UL 94 rating

| Polymer | LOI (%) | UL 94 Rating |
|---------|---------|--------------|
| ABS     | 18      | HB           |
| PE      | 17      | HB           |
| PVC     | 45      | V            |
| PP      | 17      | HB           |
| EP      | 19      | HB           |

HB = Horizontal Burning, V = Vertical

The aim of this paper is to investigate the flammability properties of kenaf fiber reinforced acrylonitrile butadiene styrene (ABS) with nanoclays composites. The flammability properties of this material were evaluated through standard flammability testing such as Underwriters Laboratory 94 horizontal burning (UL94 HB), which was conducted for controlled and uncontrolled conditions, smoke density and limiting oxygen index tests (LOI). The performance of the material during UL 94 HB test in controlled condition was compared to that in uncontrolled condition. Results from three types of fire tests are presented.

2. Methodology

2.1. Materials and preparation
Kenaf fiber used in this study was composed of kenaf core and bast fibre obtained from fresh whole stem supplied by National Kenaf and Tobacco Board, Malaysia [24]. Meanwhile, ABS was supplied by Toray Plastics (Malaysia) with specific melt flow rate of 25 g/10 min at 220°C/10 kg. Both ABS and kenaf were pulverized with apparent density of 0.51 g/cm³ and 0.14 g/cm³, respectively, in accordance to the ISO 60:1977. Prior to extrusion, 1 phr of ULTRA-PLAST TP01 was added during the compounding process using a co-rotating twin screw extruder with twin screw speed of 5 Hz [23]. Table 2 shows the fabricated material composition.

C-100 was set as the control formulation while C-50/50 was the optimized formulation for the material in terms of mechanical properties such as tensile strength (TS), flexural strength (FS), Young’s modulus (YM) and flexural modulus (FM) as reported in [23]. This optimized formulation was added with 1% and 3% of nanoclays, respectively, in order to investigate the effect on the burning property of the material. On the other hand, C-90/10 was selected as part of the investigation compared to other composition due to its lowest value of TS, YM and FM. Although it showed the highest FS value, there was no remarkable differences compared to the C-50/50. Table 3 shows the material compositions that are being used in this flammability test.
Table 2. Material composition and code

| Composition | Code |
|-------------|------|
| ABS (%)     | Kenaf (%) |
| 100         | 0     | C-100  |
| 90          | 10    | C-90/10|
| 80          | 20    | C-80/20|
| 70          | 30    | C-70/30|
| 60          | 40    | C-60/40|
| 50          | 50    | C-50/50|

Table 3. Material composition

| Composition | Nano Clay Filler (%) |
|-------------|---------------------|
| ABS (%)     | Kenaf (%)           |
| 100         | 0                   | -     |
| 90          | 10                  | -     |
| 50          | 50                  | -     |

Optimized Material Composition

| ABS (%) | Kenaf (%) | Nano Clay Filler (%) |
|---------|-----------|----------------------|
| 50      | 50        | 1                    |
| 50      | 50        | 3                    |

2.2. UL94 horizontal burning

This flammability test is to serve as a preliminary indication of their acceptability with respect to flammability for a particular application. This method determines the burning rate, dripping time, and flame propagation.

2.2.1 Controlled condition. The burning rates of all samples were investigated by UL94 HB test according to the UL94 standards. The experimental setup with the dimensions for the test is as shown in Figure 1. Flammability test was conducted for all five samples. The specimen was clamped on the retort stand and flame was applied with transverse axis inclined at 45 ±2 degrees to the free end of specimen for 30 ±1 seconds without changing its position. The burner was then removed after 30 ±1 seconds, or as soon as the combustion at the front of the specimen reaches the 25 mm mark [24]. The timing device started when the combustion front reached the 25 mm mark and time taken for the flame to propagate to the benchmark was measured. The sample was classified as HB rating if the burning rate did not exceed 40 mm per minutes or it self-extinguished before 100 mm benchmark [24].

2.2.2. Uncontrolled condition. The main purpose of this test is to verify the burning characteristics of the material in real life application. In real life fire incident, the conditions are random, uncontrolled and the source of fire can even be anything. Therefore, it is important to analyze the differences between the material's performance in uncontrolled and standard controlled flammability tests. Generally, similar procedure from the standard control test was applied to this uncontrolled test except for the conditions and source of fire. This test was conducted in open environment with random conditions in which the relative humidity, air temperature and wind speed were subjected to the environmental condition during the experimental work. Apart from that, the source of fire used in this test was the Piezo-igniter cassette gas and a handmade fume cupboard was used as the test chamber during the flammability testing. The test chamber is shown Figure 2.

2.3. Smoke Density

The purpose of the smoke density test is to observe the visibility level through smoke generation during combustion, especially in confined spaces. The specimens were tested within a 300 x 300 x 790 mm test chamber [25]. The test chamber was instrumented with a light source, a photoelectric cell and also a meter to horizontally measure the light absorption across the 300 mm light beam path that will reflect on the amount of smoke produced [25]. This is shown in Figure 3. If the material dripped excessively, a second burner was introduced into the test chamber. The second burner was positioned in the test chamber such that its flame was directed towards the center of the steel collection tray.
2.4. **Limiting oxygen index**

The purpose of limiting oxygen index (LOI) test is to measure the minimum concentration of oxygen, in which is expressed in volume percentage that is required in order to support combustion process or flame propagation [26]. The schematic representation of the LOI test is shown in Figure 4. The specimen was placed in the holder at the center of the base of the test column. The flow valves were adjusted to obtain the desired initial oxygen concentration and the total flow rate. The oxygen and nitrogen flowed into the dispersion chamber and through the glass bead bed, hence the gases were mixed and dispersed evenly over the cross section of the test column [26]. The specimen was ignited such that the entire top tip of the specimen was burning like a candle. A gas flame at the end of a tube with small orifice was used to ignite the specimen. The samples were tested with a small pilot flame to find the minimum oxygen concentration required to just sustain the combustion of the sample. The result is expressed as shown in the Equation 1 [26]:

\[ n = \frac{100 \times O_2}{O_2 + N_2} \% \]  

(1)

![Figure 1. UL94 horizontal burning test experimental set-up [24]](image1)

![Figure 2. Handmade fume chamber](image2)

![Figure 3. Layout for smoke density test chamber photometer [25]](image3)

![Figure 4. Schematic representation of a limiting oxygen index (LOI) test setup [26]](image4)
3. Result and discussion

3.1. UL94 horizontal burning

From Figure 5 and Figure 6, it can be concluded that burning rate was decreasing with increasing kenaf fibre loading. This can be seen through samples ABS/KF 90/10 and ABS/KF 50/50. This result was due to the rapid formation of char layer by kenaf fibre that prevented heat and volatiles from penetrating the flammable zones. Pure ABS and ABS/KF 50/50 with 3% obtained the value of 19.67 and 19, respectively, for controlled condition and the value of 25.28 and 20.33 for uncontrolled condition. Overall, all samples could be classified as HB since all test specimens did not have burning rate exceeding 40 mm per minute.

From the analysis of graphs above, relatively large percentage difference can be seen between ABS/KF 90/10 and ABS/KF 50/50 1%. This is due to the presence of nanoclays in ABS/KF 50/50. Nanoclays play a role as flame retardant in which it changes the decomposition path of ABS matrix. It promotes reduction in viscosity or diluting the flame volatile when exposed to flame. This process increases the formation of a protective char barrier towards the surface of the material. This barrier is created through a roundabout path of migration of nanoclays. It will resist the oxygen diffusion or volatile flames from source of fire from entering into the material. The data suggests that the mechanism of nanoclays as flame retardant is exhibited in two different ways, which are through heat release reduction and also prevention of volatile flame dripping. Besides that, both graphs highlight that the burning rate for sample ABS/KF 90/10 showed the highest burning rate and this proves that addition of kenaf fibre, which serves as source of fuel, will increase the flammability properties of the material. Kenaf fibre is highly flammable because it is made up of cellulose, hemicellulose and lignin that affect the decomposition of the material constituent at different temperatures. Furthermore, it is hydrophilic that results in a poor compatibility with hydrophobic polymer matrices and has a poor fire resistance [3].

As stated previously, UL 94 horizontal burning test were conducted in two different conditions, which were controlled and uncontrolled conditions. The aim of this approach was to observe the difference in the flammability properties of the materials that were subjected to controlled environment and uncontrolled environment. Here, the uncontrolled conditions represented the real fire scenario. Based on the percentage differences that are tabulated in Table 4, it shows that the burning rates for the uncontrolled conditions are slightly higher than the controlled condition. This might be due to possible effects from the environmental conditions that may promote increased burning rate. However, the trend of the data for both controlled and uncontrolled conditions is similar. The propagation of flame spread was captured during the flammability testing and the visual observation is explained in Table 5.
Table 4. Percentage difference between controlled and uncontrolled conditions

| Samples          | UL 94 Horizontal Burning Rates (Controlled Condition) % | UL 94 Horizontal Burning Rates (Uncontrolled Condition) % | Percentage Difference % |
|------------------|------------------------------------------------------|----------------------------------------------------------|--------------------------|
| Pure ABS         | 19.67                                                | 25.28                                                    | 28.52                    |
| ABS/KF 90/10     | 37.33                                                | 39.47                                                    | 5.73                     |
| ABS/KF 50/50     | 18.33                                                | 21.71                                                    | 18.44                    |
| ABS/KF 50/50 1%  | 12.33                                                | 14.85                                                    | 20.44                    |
| ABS/KF 50/50 3%  | 19.00                                                | 20.33                                                    | 7.00                     |

Table 5. Physical observation on the flame propagation

Pure ABS
This sample ignited at a fast rate and a rapid spread of fire could be seen through the captured images. During the burning process, dripping and melting of the ABS polymer could be observed at a shorter time interval. It was noted that the dripping mass of volatile flame burned vigorously and produced black soothe.

ABS/Kenaf 90/10
This sample ignited at a faster rate than the pure ABS and a rapid spread of fire could be seen through the captured images. During the burning process, dripping and melting of the ABS/KF could be observed at shorter time interval. The dripping mass of volatile flame burned more vigorously than the pure ABS until the material's end. Smoke production was also higher during the burning and there was less black soothe production.

ABS/Kenaf 50/50
This sample ignited at a slow rate but a rapid spread of fire could be seen through the captured images. During the burning process, dripping and melting of the ABS/KF could be observed but at a delayed time interval. It was noted that the dripping mass of volatile flame extinguished once it dropped and a little ash could be seen.
Table 5. Physical observation on the flame propagation (continued)

|                  | ABS/Kenaf/Nanoclay 50/50 (1%) |
|------------------|--------------------------------|
| This sample      | ignited at the slowest rate    |
|                  | with the slowest spread of fire,|
|                  | which could be seen from the   |
|                  | captured images. During the    |
|                  | burning process, at the initial|
|                  | stage, the material did not    |
|                  | drip and melt. Instead it      |
|                  | turned out as ash before it    |
|                  | fell down. This mechanism of   |
|                  | the ABS/KF could be observed   |
|                  | in the left image. Less amount|
|                  | of smoke production could be   |
|                  | seen and no black soot was     |
|                  | produced.                     |

|                  | ABS/Kenaf/Nanoclay 50/50 (3%) |
|------------------|--------------------------------|
| This sample      | ignited at a slower rate with  |
|                  | a slower spread of fire, which |
|                  | could be seen through the      |
|                  | captured images. During the    |
|                  | burning, from the initial stage|
|                  | until the end, the material    |
|                  | did not drip and melt. Instead,| |
|                  | it turned out as ash before it |
|                  | finally fell down. This        |
|                  | mechanism of the ABS/KF could  |
|                  | be observed in both images      |
|                  | below. Less amount of smoke    |
|                  | production could be seen and   |
|                  | no black soot was produced.    |

3.2. Smoke density

Figure 7 shows that the presence of kenaf fibre increases the smoke density rating. Pure ABS recorded a rating of 82. On the other hand, ABS/KF 90/10 recorded the highest rating of 96, which is a 17.07% rise in value compared to the pure ABS. This is due to the characteristics of kenaf fibre that promote smoke production through char formation when exposed to flame. However, looking into the trend in Figure 7 for samples 3, 4 and 5, it shows that the smoke density rating for sample 3 slightly decreased with increasing kenaf fibre loading. The rating value recorded by ABS/KF 50/50 was 93. Apparently, with the presence of nanoclays, ABS/KF 50/50 1% and 3% recorded the same rating value of 91, which was slightly lower than the optimized composition ABS/KF 50/50 (or a 2.15% value drop to be exact). Hence the addition of a small amount of nanoclays resulted in decrement of smoke density rating.

The rate of production of flammable volatiles mass and also the degree of conversion of the volatiles to smoke for ABS/KF 90/10 were higher. Therefore, it had a higher density of smoke. On the other hand, for the pure ABS, the mass of flammable volatiles released during combustion and the degree of smoke generation were lower. Thus the density of smoke of pure ABS was the lowest. Typically, thermoplastics such as ABS have a low smoke emission when exposed to fire as they yield more char and black soot formation. ABS/KF 50/50 1% and 3% recorded an optimum smoke density along with the optimized composition ABS/KF 50/50. This is because higher fiber contents and the presence of nanoclays in the material reduce the smoke generation. This result suggests that the presence of organic material in the material composition promotes smoke generation.
3.3. Limiting oxygen index

Figure 8 shows that the volume of oxygen is increased with increasing kenaf fibre loading. The highest LOI value was obtained by ABS/KF 50/50, which was 14. Meanwhile, the lowest value was obtained by pure ABS, which was 11. LOI value obtained by ABS/KF 90/10 was 12. Apparently, with the presence of nanoclays, ABS/KF 50/50 1% obtained LOI value of 13 with 7.1% drop in value compared to optimized composition ABS/KF 50/50. However, for ABS/KF 50/50 3%, there was no change in volume. It could be seen that LOI value for ABS/KF 50/50 3% was higher compared to ABS/KF 50/50 1%. This might due to the melting and dripping of material during the test that caused the specimen to extinguish, thus mislead the LOI values [3]. Overall, an addition of small amount of nanoclays resulted in decreased LOI value.

Looking through the standard requirement for LOI, materials with LOI value less than 21 percent are defined as combustible material. In contrast, materials that have LOI value of more than 21 percent are defined as self-extinguishing. This is because, based on standard air composition, the oxygen level in air is 21 percent and thus materials with more than this value are not able to support the combustion at ambient temperature without an external source of fire. However, the flammability data obtained in this experiment does not correlate with the ‘real fire scenario’ [27]. This is because the flammability measurement was obtained at room temperature. LOI values decrease with increasing temperature, says under intense fire condition, in which materials with high LOI values may burn without extinguishing itself. Besides, if the materials self-extinguish through melting or dripping, this can mislead the decision for LOI values [2-4]. Therefore, this method cannot be considered as a fast rule in determining self-extinguishing property.

By making a comparison for the result obtained from the experimental test and standard evaluation for LOI, it can be concluded that all five samples are classified as highly combustible. However, this can be justified through the limitation applied to LOI for nanocomposites. The presence of nanoclays in ABS/KF 50/50 decreases the LOI values due to two factors related to the burning mechanism. The first factor is the increased flame propagation rate over the surface of the specimen when it is exposed to flame. Moreover, the second factor is the barrier formation by the nanoclays from the inside of the material that suppresses the flame spread.

4. Conclusion

Flammability properties of kenaf fibre reinforced ABS with nanoclays are evaluated in this study. It can be concluded that the presence of nanoclays in kenaf fibre reinforced ABS helps to enhance the burning characteristics of the material when exposed to source of fire. Flammability studies are carried out through three tests: UL 94 horizontal burning, smoke density and limiting oxygen index. The results suggest that this material possesses low burning rate, slow flame propagation, low amount of smoke generation and requires less oxygen concentration to support the burning process (subject to limitation). Hence, an ideal
formulation of ABS/KF 50/50 with 1% nanoclays is applicable for industrial usage, especially in confined spaces in aircraft as it can protect the underlying structure from risk of fire due to its capability as self-extinguishing material. For the better understanding of the burning mechanism, further investigation is required to develop correlation between these three tests in order to reach an optimized conclusion. Future work will be emphasized on developing a systematic approach for predicting fire behavior in real fire scenario by varying parameters such as relative humidity, temperature and wind speed.

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