International Journal of Recent Technology and Engineering (IJRTE)
ISSN: 2277-3878, Volume-7, Issue-6S5, April 2019

The Influence of Fiber Length and Concentration on the Thermal Properties of Basalt Fiber Reinforced Intumescent Coating

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Abstract: The effect of different length and concentration of basalt fibers have been studied in this research which effects the thermal properties of intumescent coating. The investigated intumescent coating formulations, containing ammonium polyphosphate (APP), melamine (MEL), boric acid (BA), expandable graphite (EG), epoxy resin (NPEL-128) and polyamide amine hardener (H-2310) were reinforced with 0-1 wt.% of basalt fibers of each 6 mm and 12 mm. The effects of the basalt fiber's length on the char expansion and thermal stability of the intumescent coating, as well as on the char morphology and composition were studied. The char expansion was analyzed using a Carbolite furnace. Field emission scanning electron microscopy (FESEM) and Thermal stability test were carried out using thermogravimetric analysis (TGA). Furthermore, the morphological study confirmed that long fibers (12 mm) of basalt fibers form a network which protects the underlying steel substrate at elevated temperature in the event of fire. Elemental analysis showed that ICF-B1-12 has enhanced carbon content of 63.1% in the residual char. Thermogravimetric analysis results showed that BF reinforced formulations; ICF-B1-12 has 34% of residual weight at 800ºC which is more thermally stable among all formulations.

***Index Terms*: Keywords: Intumescent coating, Basalt Fibers, Thermal stability test, Char expansion, Char thickness.**

I. INTRODUCTION

Intumescent coatings are famous all over the world and used as passive fire protection materials that form a protective barrier in the event of fire and thereby control the heat and mass loss between the structure and the heat source. There are various advantages of this fire protection material compared to other types of materials; it can be applied easily onto substrates like polymer, textile, wood, steel, etc. [1]-[4]. Intumescent coating can be synthesized in different colors to give an aesthetic appearance to the structure. The coating is usually thin and light in weight. The thickness of the coating is decided on the type of the fire exposure. For example, in case of cellulosic and hydrocarbon fire, the intumescent coating thickness is in the range of 0.2 mm to 3 mm. The intumescent coating expands from 5 to 100 times the original thickness of the coating [5].

There are three main ingredients linked with each other in binder, which on exposure to heat formed an expanded char at high temperature. These active ingredients are commonly: a carbon source (e.g. pentaerythritol or expandable graphite), a blowing agent (e.g. melamine) and an acid source (e.g. boric acid and/or ammonium polyphosphate) [6]. These ingredients have some disadvantages being organic compounds. These ingredients undergo exothermic reactions which decrease the insulative efficiency of the system. The structure integrity of the char in some cases is low because of the mechanical load induced by the fire. Moreover, various toxic gases are released which are undesirable to the environment [7]. Various fillers have been used to improve the properties of intumescent coatings [8], [9]. However, the char oxidation and detachment at high temperature is a big issue which needs to be solved. Moreover, thick layer of coating is needed which increases the cost.

A novel filler to improve char strength is basalt fiber (BF), which is produced from basalt rocks at 1400oC. BF has diameter that varies from 9 to 13 µm. These fibers show strong mechanical and chemical properties with excellent corrosion resistance. The BFs are mostly used in the automotive and aerospace industries to fireproof textiles as well as in chemical industries to fabricate pressure vessels. The synergistic effects of BFs with aluminum trihydrate has been studied with promising results. The impact of BF's dispersion on the thermal properties of IC appears to be a crucial point, which was investigated in article previous paper [10].

Due to the limited bibliographic data on that subject, the influence of different lengths (short and long fibers) and concentration of BFs on the thermal properties of intumescent coating has not been studied so far. This paper discusses the effects of the expanded char structure on the effective thermal conductivity of the novel BF reinforced intumescent coating formulations. The morphology of the intumescent char was studied using FESEM, the thermal degradation of the ICs was analyzed using TGA technique and the elemental study was taken out to see the char composition in a quantitative way.

Revised Manuscript Received on December 22, 2018.

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II. METHODOLOGY/MATERIALS

A. Materials

The steel substrate was mild steel S355 (100 × 100 × 1.5 mm3 and 50 × 50 × 1.5 mm3) which was supplied from TSA industries, Malaysia. The plates were first sand blasted to remove oil and other impurities from the surface, producing a rough surface which increase the adhesion of the coating onto the steel plate. The binders used in this study were epoxy resin (NPEL-128) and polyamide amine (PAA) (H-2310) as the hardener both were supplied from WWRC Malaysia Sdn Bhd. Expandable graphite (EG) was used as physical expansion agent, supplied from Mc-Growth Chemical Malaysia Sdn Bhd. Ammonium polyphosphate (APP) was selected as acid source and blowing agent, purchased from Clariant Malaysia Sdn Bhd, and boric acid as an additive was supplied from Merck Malaysia Sdn Bhd. The basalt fibers strands (BF), in two different lengths of 6 mm and 12 mm and each fiber has 14 µm diameter were used as reinforcing filler, imported from JN Technologies Pvt Ltd. Fig. 1 shows the length and diameter of the BF used in this study.

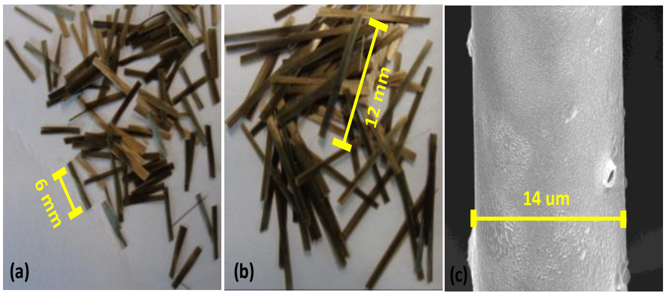

Fig. 1. Basalt fibers used in the research (a) length 6mm, (b) length 12 mm, (c) diameter 14 µm

B. Preparation of Intumescent Formulations

The different materials according to their weight percentages were weighed on an analytical balance (AB204-S, Mettler Toledo). Table I shows the intumescent formulations used in the research. The weighed materials were ground and mixed in a shear mixer with rotational speed of 40rpm at 25oC for 30 minutes. The synthesized coating formulations were applied onto the steel plate. The samples were cured for 7 days at 25oC and the dried samples have a thickness of 2 ± 0.3 mm.

Table I: The formulations of intumescent coating

Sample ID.	Ammonium polyphosphate	Expandable graphite	Melamine	Boric acid	Epoxy resin	Hardener	Basalt fibers	
							6 mm	12 mm
ICF-B0	11.76	5.8	5.76	11.5	43.45	21.73	-	-
ICF-B0.5-6	11.76	5.8	5.76	11.5	43.12	21.56	0.5	-
ICF-B1-6	11.76	5.8	5.76	11.5	42.79	21.40	1	-
ICF-B0.5-12	11.76	5.8	5.76	11.5	42.46	21.23	-	0.5
ICF-B1-12	11.76	5.8	5.76	11.5	42.13	21.07	-	1

III. CHARACTERIZATION

A. Char Expansion Test

To study the expansion and cell structure of the char, the samples (50×50×1.5 mm3) were heated in a Carbolite furnace (CWF 13/13) which has a volume of 13 liters from 30-1300°C. The temperature of the furnace was increased from 25oC until 500oC with a heating rate of 20oC/min for complete combustion to achieve complete char, holding the temperature at 500oC for 60 minutes, followed by cooling at ambient temperature. After the furnace test, the samples are proceeded for char expansion measurement. The total heating cycle of the furnace is shown in Fig. 2. It consists of three main steps. At the first step, the temperature is raised to 500oC with 20oC/min which results the melting of coating and char formation. The second step is the dwell time, holding the temperature at 500oC for 60 minutes. In the third step, the cooling process proceeds at room temperature.

The intumescent factor (I) is defined to be the maximum char thickness after the furnace test to the maximum coating thickness [11].

$$I = d2-d0/d1-d0 \qquad (1)$$

where d2 denotes the char thickness, d1 denotes the coating thickness, and the d0 denotes the uncoated steel substrate thickness. The intumescent factor is affected by the heating type, heating rate, initial temperature of the furnace at which the samples were placed in the furnace.

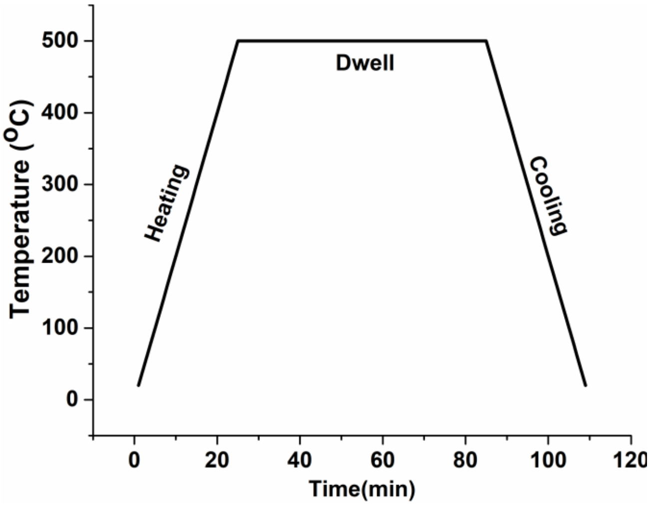

Fig. 2. Furnace Heating Cycle

B. FESEM Analysis of the Char

The char after the furnace test was studied and analyzed by the FESEM SUPRA 55 VP (Carl Zeiss, equipped with INCAx-act X-ray Detector, Oxford Instruments). The main purpose of the FESEM is to see the fibers alignment in the char and the char structure whether contains any cracks or dislocation. The Energy Dispersive X-ray spectroscopy (EDS) investigation was also carried out to see different elements and their composition in the char.

C. Thermal Stability Test

Thermal stability test was performed using thermogravimetric analysis on Pyris 1 TGA (Perkin Elmer) using 5mg sample in powder form, at 10oC/min under nitrogen, operated at a temperature range of 25-800oC. This analysis helps us to study the thermal degradation and mass loss rate with respect to time and temperature.

IV. RESULTS AND DISCUSSION

First of all, the prepared coated samples of (50×50×1.5 mm3) dimensions were heated in a Carbolite furnace for 60 minutes. The samples were expanded freely in the furnace and converted on to char. Fig. 3 shows the intumescent factor of all the formulations used in this study. The char expansion of the ICF-B0 was 3.83 which was the lowest among all the formulations because cracks were produced on the surface of the char during combustion process. The resulted char is fully oxidized leaving behind the weak char [10], [12]-[14].

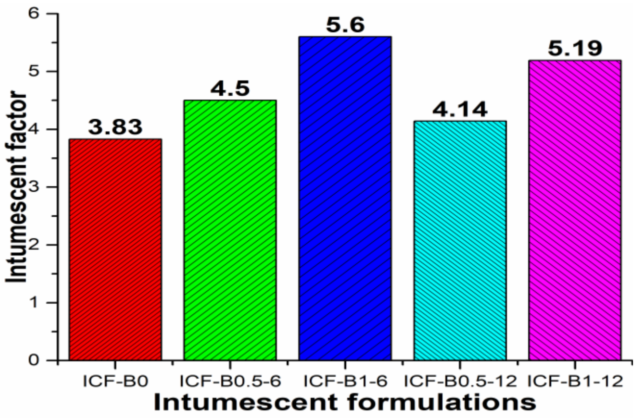

Fig. 3. Intumescent factor of ICF formulations

The char expansion was improved by adding basalt fibers in the coating. ICF-B1-6 has highest intumescent factor of 5.6 among all the formulations. This char expansion is further studied in the FESEM analysis.

Fig. 4 shows the microstructures of intumescent formulations. Fig. 4 (a) shows the formulations without the addition of the basalt fibers. The char contains cracks and deep holes which are resulted from the escape of gases during the combustion process. The gases are CO2, NH3 and SO2, respectively. These holes and cracks act as the heat carriers which supply the heat from the source to the substrate and the substrate temperature subsequently rises. Moreover, the char is fragile and fluffy.

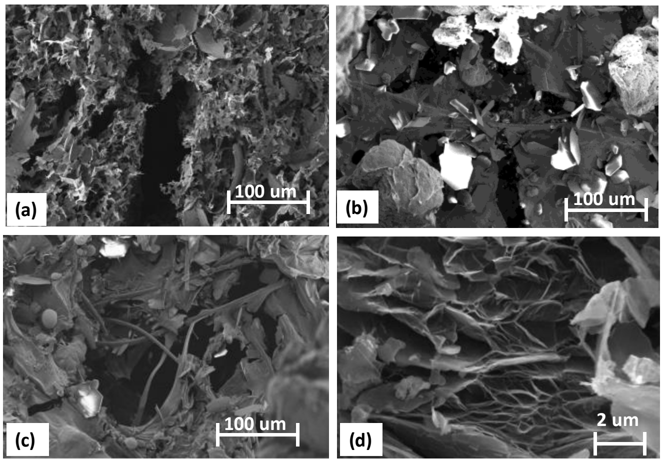

Fig. 4. Micrograph of (a) ICF-B0, (b) ICF-B1-6, (c) ICF-B1-12, (d) Cell structure of ICF-B1-12

When the basalt fibers are added in the formulation, those deep cracks and holes were covered with the network of basalt fibers which act as a bridges to avoid the char from cracking and also as heat shield and avoid the heat transfer from the source to the substrate as shown in Fig. 4 (b) and (c). The strongest fibers network is seen in ICF-B1-12 which contains 12 mm fibers as shown in Fig. 4 (c). These long fibers create a much stronger network as compared to the short fibers which gives strong network but though unable to defend the coating cracks from opening.

Fig. 4 (d) shows the cell structure of graphite in ICF-B1-12 formulation which is honey comb like structure that confirms the presence of the unburnt graphite in the char [15].

The EDS analysis in Fig. 5 shows that the antioxidation property which confirms the availability of the unburnt carbon in the char after combustion. The O/C ratio is the lowest in case of ICF-B1-12. This shows that the fibers in the formulations do not permit the char to be oxidized which improves the strength of the char.

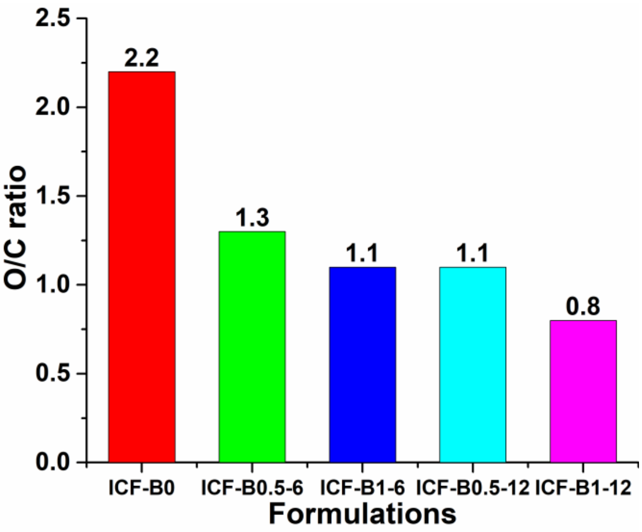

Fig. 5. O/C ratio of intumescent char

The thermal stability of the intumescent formulations was investigated by TGA in nitrogen atmosphere. Fig. 6 shows the TGA graph in which ICF-B0 has the lowest residual weight of 27% among all the formulations. It means that the intumescent cannot withstand at high temperature due to the absence of the fibers. The incorporation of basalt fibers in the formulation enchanced the thermal stability and the residual weight increased with the increase in weight percentage of BF. The residual weight of ICF-B1-6 was 31% which is higher than ICF-B0 but the highest residual weight of ICF-B1-12 has 34% which is the highest among all formulations. The reason is that the long fibers form a very stable and strong bonds with the binders which may resist at higher temperature. Moreover, basalt fibers contain Si-O groups which is itself heat resistant at higher temperature and prevents the intumescent coating from degradation.

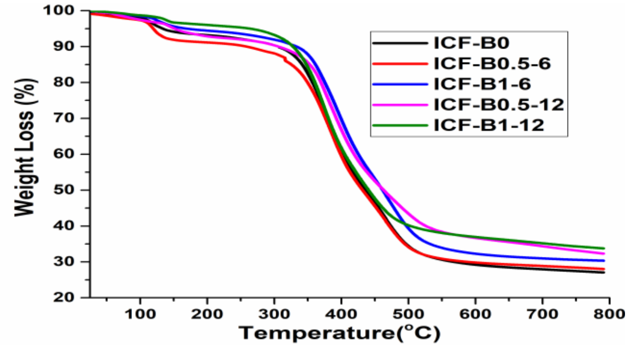

Fig. 6. TGA curves of intumescent formulations

The DTG curves are shown in Fig. 7. The DTG curves of the intumescent formulations show five main degradation steps. These degradation steps are: 110-120oC, 345-360oC, 390-410oC and 480-500oC. The first phase at 115-118oC correspond to the degradation of boric acid and formation of products like boron phosphate and boron oxide [10], [16]. There is a small curve seen at 315-320oC which may correspond to the dehydration and decomposition of Melamine, APP and EG to form melamine phosphate and release NH3 and N2. The EG starts decomposing at temperature range of 377-380oC which yields CO2 and SO2, respectively [17], [18]. This decomposition shifts to a higher temperature range when the formulations were incorporated with long basalt fibers as in case of ICF-B1-12. This ultimately, slow down the decomposition of the intumescent coating during burning. However, this degradation temperature is low in case of ICF-B0, because there were no fibers present as shown in FESEM analysis that may not withstand at high temperature.

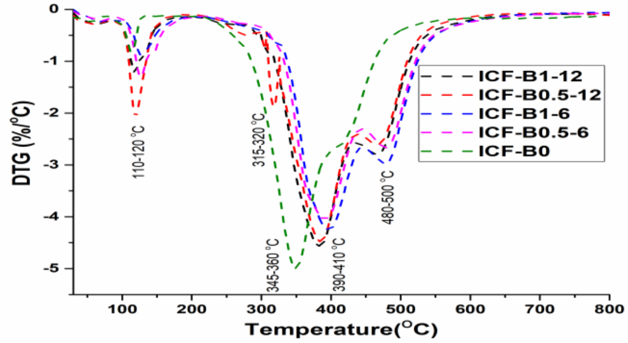

Fig. 7. DTG curves of intumescent formulations with temperature

V. CONCLUSION

This study displayed the effect of basalt fibers length on the thermal properties of intumescent coating and char. The FESEM results showed that ICF-B0 were consisted of cracks and deep holes that decrease the thermal insulation. From the EDS analysis that showed the high O/C ratio for the control formulations among formulations. This O/C ratio was lowered for ICF-B1-12 has 0.8 value, which means that introducing long fibers in the formulations improved the char anti-oxidation property which limited the oxygen presence in the surrounding which ultimately lowers the fire spread. ICF-B1-12 forms dense char and the residual weight was enhanced considerably to provide good thermal stability for the char at higher temperature. Well dispersed basalt fibers form a shield in the coating that minimize the flow of heat from the flame to the substrate.

VI. ACKNOWLEDGEMENT:

This work was financed by the Yayasan Universiti Teknologi PETRONAS (YUTP) award No. (0153AA-E30) and Ministry of Higher Education under the FRGS grant award No. (0153AB-K91). The authors also appreciate the guidance by the Centre for Advanced and Functional Materials, Institute of Sustainable Buildings, UTP.

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