

Effects of nano-sized boron nitride (BN) reinforcement in expandable graphite based in-tumescent fire retardant coating

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Abstract. The purpose of in-tumescent fire retardant coating (IFRC) is to protect substrate from fire attack by limiting heat transfer. A range of coating formulations have been prepared using Bisphenol A epoxy resin BE-188 and polyamide solidifier H-2310 as two-part binder, ammonium polyphosphate (APP) as acid source, melamine (MEL) as the blowing agent, expandable graphite (EG) as carbon source and nano-boron nitride (BN) as inorganic nano filler. The filler was used to improve the performances of the APP-EG-MEL coating. The effects of nano-BN on the char morphology and thermal degradation were investigated by fire test, thermo gravimetric analysis (TGA), Fourier transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS) and field emission scanning electron microscopy (FESEM). The results showed that by substituting or reinforcing of 4% weight percentage of nano-BN, residual weight of the char increases by 23.82% compared to APP-EG-MEL coating without filler. Higher carbon content was obtained in the char and a more compact char was produced. The results indicated that nano-BN could be used as a filler to improve thermal stability of the APP-EG-MEL coating.

1. Introduction

Fire can result in loss of lives and property. It can be controlled using fire protection system classified into two: active and passive. Active systems such as fire extinguishers, fire alarms and water sprinklers are commonly installed. However, passive systems such as fire-rated doors and fire retardant coatings are often overlooked and disregarded. Their purpose is to contain fire, delay building collapse and provide safe evacuation of occupants, and to limit the destructive effects of fire [1].

Steel is a strong material and the need to protect it may not be apparent. The exposure of steel structural members to high temperature results in buckling leading to structural collapse when it reaches 500°C, its critical temperature [2]. To ensure the safety of steel structure during fire, its temperature must be maintained below the critical temperature. This can be achieved by using intumescent fire retardant coatings [3].

Intumescent is defined as the swelling up of certain substances when they are heated and it comes from the Latin word 'intumescere' which means 'to swell up' [4]. The coating comprises of three main ingredients: an acid source, a carbon source and a gas source [5]. Past researches commonly used ammonium polyphosphate, pentaerythritol and melamine [6]. When the temperature rises in a fire, intumescent fire retardant coating swells many times of its original thickness to form carbonaceous foam charred layer that limits heat transfer to the substrate [7].

The expansion of the charred layer often depends on the coating ingredients as well as the ratio of carbon, nitrogen and phosphorus atoms in the compound [8]. The char layer is the key parameter to



ensure the coating provides full protection of substrate. The relationship between flammability and charring process of intumescent coating explained by Joseph and Ebdon [9] is as follows: the higher the amount of residual char after combustion, the lower the amount of combustible material to propagate the flame thus increasing the flame retardancy of the material. Existing intumescent fire retardant coatings reported that the char layer would damage easily by the turbulence during flame spread. The residual weight of the coating is also low as it is easily oxidized at high temperature [10].

Several studies [11-13] have indicated the usage of fillers to improve the performances of the coatings. Incorporating nano-sized fillers specifically into coatings can greatly enhance its fire resistance and anti-oxidation properties [14]. Boron compounds are one of the widely used fire retardant fillers for example: borax, zinc borate, barium metaborate and ammonium fluoroborate. These type of compound are non-toxic to human and act as fire suppressant during condensed and vapour phase [15].

A study by Jimenez, Duquesne and Bourbigot stated that borates and boric acid are well established as flame retardants; and addition of boric acid to the epoxy-based intumescent fire retardant has improved its performance by increasing the time of failure. Furthermore, the formation of boronphosphate during the degradation of boric acid and ammonium polyphosphate together has been suggested to provide superior mechanical resistance of the char and also promotes the adhesion between char and substrates [16].

Boron nitride (BN) is an inorganic boron compound that demonstrates excellent chemical and thermal stability, hence indicates it can be used as a filler to improve the performance of intumescent fire retardant coatings [17]. Nowadays, it has been utilized in many applications specifically in polymer composites [18], refractory materials [19] and coatings. Boron nitride will act as a thermal insulator for the underlying unburned coating when the temperature reaches above 600°C [19]. This will result in mass loss suppression and stabilizes the resulting char [20] thus increasing the fire performance of intumescent coating.

The objective of this study were to investigate the performance of intumescent fire retardant coating reinforced with nano-BN by determining its optimum thermal performance, residual weight, functional group of the char and char morphological structure of intumescent fire retardant coating.

2. Materials and methods

Both Bisphenol A epoxy resin BE-188 and polyamide solidifier H-2310 were obtained from McGrowth Chemical Sdn. Bhd., Malaysia. Ammonium polyphosphate was supplied by Clariant (Malaysia) Sdn. Bhd., Malaysia. Melamine, expandable graphite, and boric acid were purchased from Sigma-Aldrich (M) Sdn. Bhd., Malaysia. Boron nitride was supplied by Xuzhou Jiechuang New Material Technology Co., Ltd., China. Structural steel A36M was obtained from TSA Industries (Ipoh) Sdn. Bhd. Malaysia.

2.1. Coating preparation

All intumescent ingredients were weighed according to their respective weight percentages as in Table 1 and pulverized using ROCKLABS Bench Top Ring Mill for 60 sec. Binder and intumescent ingredients were then mixed using a shear mixer at 40 rpm for 30 min. The coatings were applied on sandblasted structural steel plate of 50x50x1.5mm and cured for 24 hours at room temperature before characterization tests.

Table 1. The weight % of materials in intumescent fire retardant coating.

Formulation	EG	APP	MEL	BA	Epoxy	Hardener	BN
BN-0	5.85	11.76	5.76	11.5	43.42	21.71	0
BN-1	5.85	11.76	5.76	11.5	42.75	21.38	1
BN-2	5.85	11.76	5.76	11.5	42.09	21.04	2
BN-3	5.85	11.76	5.76	11.5	41.42	20.71	3
BN-4	5.85	11.76	5.76	11.5	40.75	20.38	4
BN-5	5.85	11.76	5.76	11.5	40.09	20.04	5

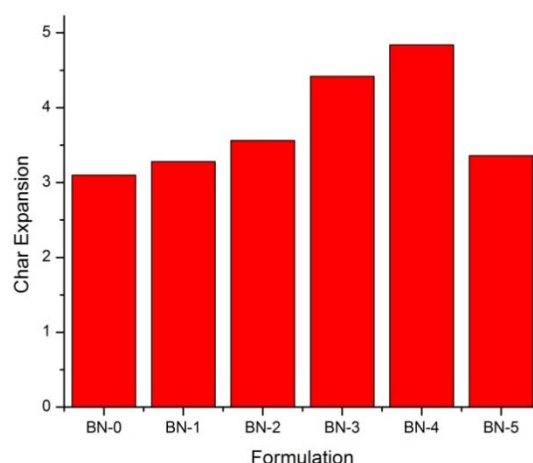
2.2. Characterization techniques

Fire test was carried out using Bunsen burner for 60 min and the distance between Bunsen burner and coating was set at 7cm. The coating's backside temperature was recorded using three thermocouples type K with miniature connector and Anritsu Data Logger AM-8000K. Thermal gravimetric analysis (TGA) of intumescent fire retardant coating samples was performed on Pyris 1 TGA (Perkin Elmer) at 10°C/min under nitrogen over temperature range of 25°C–800°C. Fourier transform of Infra-red Radiation (FTIR) for functional groups of char residual were determined by Spectrum One (Perkin Elmer) in range of 4000–400cm⁻¹. X-ray photoelectron spectroscopy (XPS) of char residual was performed using K-alpha XPS System (Thermo Scientific) with 14.0 kV of voltage and 300 W of power. The char layer was characterized by FESEM using SUPRA 55VP (Carl Zeiss AG) to physically observe its morphological structure.

3. Results and discussion

3.1. Analysis of char expansion

Furnace test was performed to burn the coating samples using CWF 13/5 Standard Chamber Furnace (Carbolite) at 6°C/min and held at 500°C for 1 h. The initial thickness of the coating was measured using Positector 6000 FS1 (DeFelsko). The char thickness after furnace test was obtained using digital Verniercaliper.

**Figure 1.** Expansion of intumescent coating after furnace test

The char expansion results are as follows: 3.1 (BN-0), 3.28 (BN-1), 3.56 (BN-2), 4.42 (BN-3), 4.84 (BN-4), and 3.36 (BN-5) times, from their original coating thickness as shown in Figure 1. Meanwhile Figure 2 shows the resulting char of BN-1, BN-2, BN-3, BN-4 and BN-5 after the furnace test. By comparing formulation BN-4 to BN-0, BN-1, BN-2, BN-3 and BN-5, BN-4 gives better expansion.

This is because of higher weight percentage of BN while APP, melamine, EG and boric acid used are kept constant in all formulations.

The role of APP, EG, melamine and boric acid are as acid source, carbon source, blowing agent and additive, respectively. Both boric acid and boron nitride will decompose into boric oxide that contributes to high residue and resulting in high amount of char [21]. Additionally, both boron compounds act as adhesion agent to provide strong bonding between the substrate and the char [22].

Initially the formulation expanded uniformly, but with the progressing fire over time, the remaining boron nitride continue to balance the expansion [17]. In previous studies, Duquesne et al. stated that a certain degree of expansion is required to get high thermal performance, but the highest expansion does not necessarily providing the highest efficiency [5]. Intumescent coating reinforced with boron nitride has moderate char expansion but it can limit heat transfer to the substrate effectively. With the presence of boron nitride, the resulting char does not contain any appalling cracks or holes that can cause heat to penetrate towards the substrate. Even though boron nitride used in BN-5 is the highest, it cannot contribute to higher expansion. This indicates that in any formulation with reinforced fillers, there is certain limitation in weight percentage where it provides optimum results [16].

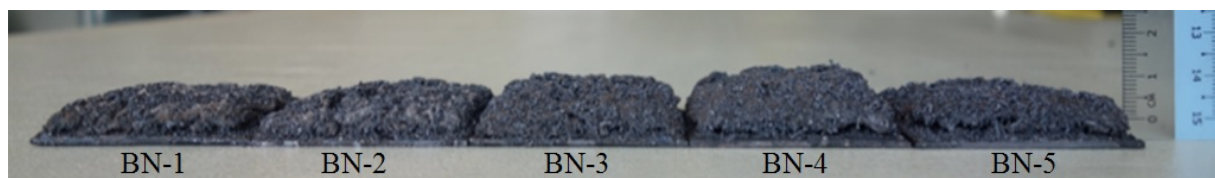


Figure 2. Physical char expansion comparison of BN-1, BN-2, BN-3, BN-4 and BN-5 from left to right respectively, after furnace test

3.2. Analysis of thermal insulation

Fire test was carried out using Bunsen burner for 60 min and the distance between Bunsen burner and coating was set at 7cm. The coating's backside temperature was recorded using three thermocouples type K with miniature connector and Compact Thermologger AM-8000K (Anritsu Meter). Time versus substrate temperature curve for heat shielding/insulation is illustrated in Figure 3.

Six samples of coating with different compositions of intumescent ingredients were burnt using portable Bunsen burner for 60 minutes. The substrate temperature of BN-0 after 30 minutes is 232°C, and after 60 minutes the temperature reached 261°C, respectively as reported in Figure 3. BN-0 contains no boron nitride and the char structure could not sustain the fire for 60 minutes as there were no further reaction products which can trim down the substrate temperature.

Figure 4 (a, b, c, and f) shows the char photograph of BN-0, BN-1, BN-2 and BN-5 with cracked char surface. The substrate temperature of BN-1, BN-2 and BN-5 was 234°C, 178°C and 193°C after 60 minutes respectively. The temperature at 60 minutes for BN-1, BN-2 and BN-5 is 27°C, 83°C and 68°C less than BN-0 respectively. Figure 4(b, c, and f) shows that the surface of BN-1, BN-2 and BN-5 char is also cracked and oxidized.

Figure 4 (d, e) shows a better char surface of BN-3 and BN-4 char compared to BN-0, BN-1, BN-2 and BN-5. The substrate temperature of BN-3 and BN-4 formulation reached 148°C and 145°C after 30 minutes and 162°C and 138°C after 60 minutes. This substrate temperature is significantly lesser than BN-0, BN-1, BN-2 and BN-5 after 60 minutes. Figure 2(d) of char photograph of burnt BN-3 formulation shows the existence of cracks on the surface. The cracks gradually increased the substrate temperature to 162°C after 60 minutes. In general, the addition of boron nitride increases adhesion of char to substrate [17]. From the literature [7], it was reported that when APP and boric compound were combined into the resin; the temperature of steel substrate reached 400°C in 29.5 minutes in contrast to 4 minutes when epoxy resin was used alone. Increasing the wt% of boron nitride results in heat shielding effects increases as shown in Figure 4 and surpasses the performance of coating without the usage of boron nitride as filler.

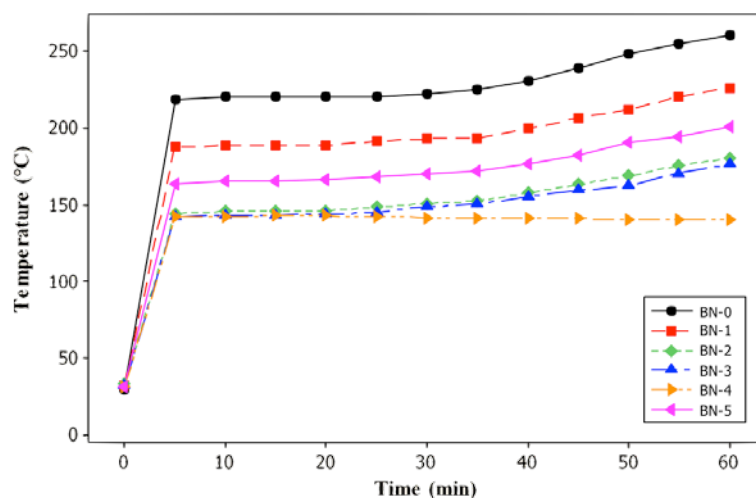


Figure 3. Time and substrate temperature curve of intumescent fire retardant coating

3.3. Analysis of char morphology

The char layer was characterized by FESEM using SUPRA 55VP (Carl Zeiss AG) to physically observe its morphological structure. The outer surface micrographs of char layer from furnace test of BN-0, BN-1, BN-2, BN-3, BN-4 and BN-5 are shown in Figure. 5 (a, b, c, d, e, f) respectively. It was noted that the morphological structure of the outer surface of the char layers were compact with tiny holes and cracks. Large crack surface and voids can be seen from BN-0, BN-1, BN-2 and BN-3. Char layer with large cracks could not provide good fire protection as heat can pass through char thus penetrating the substrate [7]. The char layer of BN-4 and BN-5 shows more refined compact surfaces with no crack and small voids that sized less than 50 μm . The compact outer surface char structure can provide resistance and limit heat transfer to the substrate hence protecting the substrate against fire. Thus, both well-expanded char and defined char structure are very crucial to general fire resistance properties of coatings [11].

Although nano filler was not incorporated in BN-0, it also formed bubbles but the distribution is quite uneven and there were some crack in the structure. Therefore, char morphological structure of BN-4 and BN-5 with 4% and 5% nano-BN respectively are better than BN-0 without nano filler. The char morphology of BN-4 formulation in Figure 5 (e) shows a thick and porous inner surface with the bubbles formation which is formed due to emission of gaseous compounds during the fire test. These gaseous compounds are produced from the thermal degradation of the coating during fire test [23].

3.4. Functional groups in char

FTIR for functional groups of char residual were determined by Spectrum One (Perkin Elmer) in range of 4000–400 cm^{-1} . The FTIR spectra of BN-0, BN-1, BN-2, BN-3, BN-4 and BN-5 char are presented in Figure. 6. The FTIR spectra of BN-0 char shows two regions of absorption. In the 1st region, the three bands at 545, 620, 925 and 1085 cm^{-1} are attributed to ν (B–O–H) stretching mode, ν (C–H) stretching mode, δ (B–O–H) deformation bond, and ν (B–O) stretching bond, respectively [24]. In the 2nd region above 2100 cm^{-1} , two weak bending peaks occurred at 2257 and 3215 cm^{-1} which represent the weak stretching vibrations of (–CH₃–CH₂–) and O–H groups due to cured epoxy resin.

The FTIR spectrum of BN-1 char shows three regions of absorption. In the low frequency side, at 494 cm^{-1} of the spectrum is attributed to fourth regions of ν is PO₄ [25]. The two broad bands centered on 1151 and 970 cm^{-1} are characteristic of phosphate anion [24, 26, 27]. In the region from 1380 – 1700 cm^{-1} , the peaks at 1633 and 1402 cm^{-1} are due to amino groups (NH₂) and ring(C,N), respectively. In the 3rd region above 2100 cm^{-1} , one stretching peak at 2627 cm^{-1} vibrations of

(-CH₃-CH₂-) and two weak bending peaks at 3154 and 3450 cm⁻¹ are identified the O-H groups. The spectra of BN-2, BN-3, BN-4 and BN-5 look almost similar as BN-1 because the ingredients and testing environment during fire test is the same. The difference in the formulations is in the amount of boron nitride.

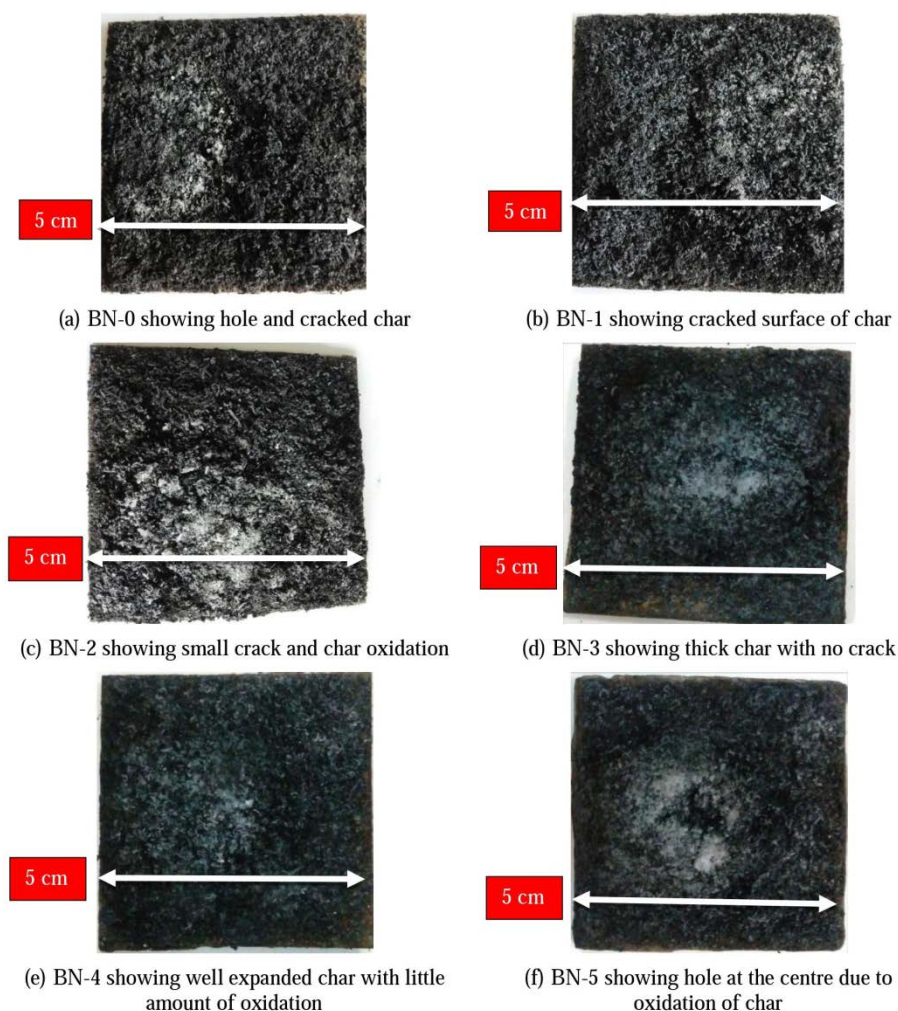


Figure 4. Char photographs (a, b, c, d, e, f) after fire test

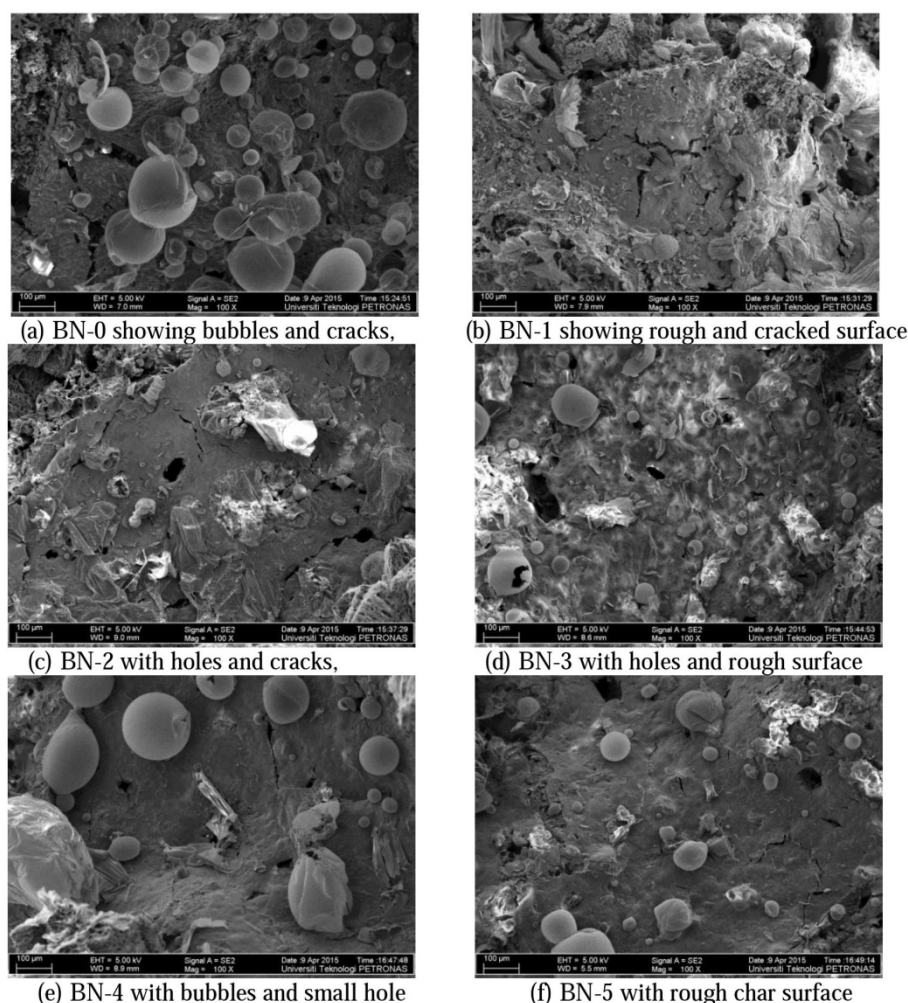


Figure 5.FESEM images (a, b, c, d, e, f) showed the char morphology of intumescent fire retardant coating

In the Figure 6, the B-O-P bending motions are observed at 613, 618, 644 cm^{-1} [25, 26] and O-P-O are observed at 548, 537 and 539 cm^{-1} , respectively. The peak from 548-537 cm^{-1} in B_2O_3 is assigned to $\delta(\text{O-B-O})$ mode. The two bands in the range of 975-824 cm^{-1} and 1089-1185 cm^{-1} are the stretching mode of $\nu(\text{B-O-P})$ [24]. The bending vibrations observed at 1412, 1436 and 1446 cm^{-1} of CH_2 or CH_3 are due to epoxy binder, while the $\text{C}\equiv\text{C}$ stretching vibration at 1630 cm^{-1} is due to epoxy binder and APP derivative in the char. In the second region around 2257-2348 cm^{-1} a strong stretching vibration characteristic of $\text{C}=\text{N}$ is due to polyamide hardener. In the region above 2400 cm^{-1} , a single broad peak at 2495-2895 cm^{-1} is a weak bending peak due to the bending vibration of $\text{CH}_3\text{-CH}_2$ and a double peak at 3213, 3405, 3467 cm^{-1} is identified as the weak bending peaks of O-H group. Both types of peak originate from the epoxy binder. The functional group of residual char compounds is identified by FTIR spectroscopy which revealed that the char contains functional groups of borophosphate, phosphate oxide, boron oxide, $-\text{CH}_3\text{-CH}_2$ and aromatic compounds obtained from the reaction between EG, APP, boric acid, melamine, boron nitride and cured epoxy resin during the fire test.

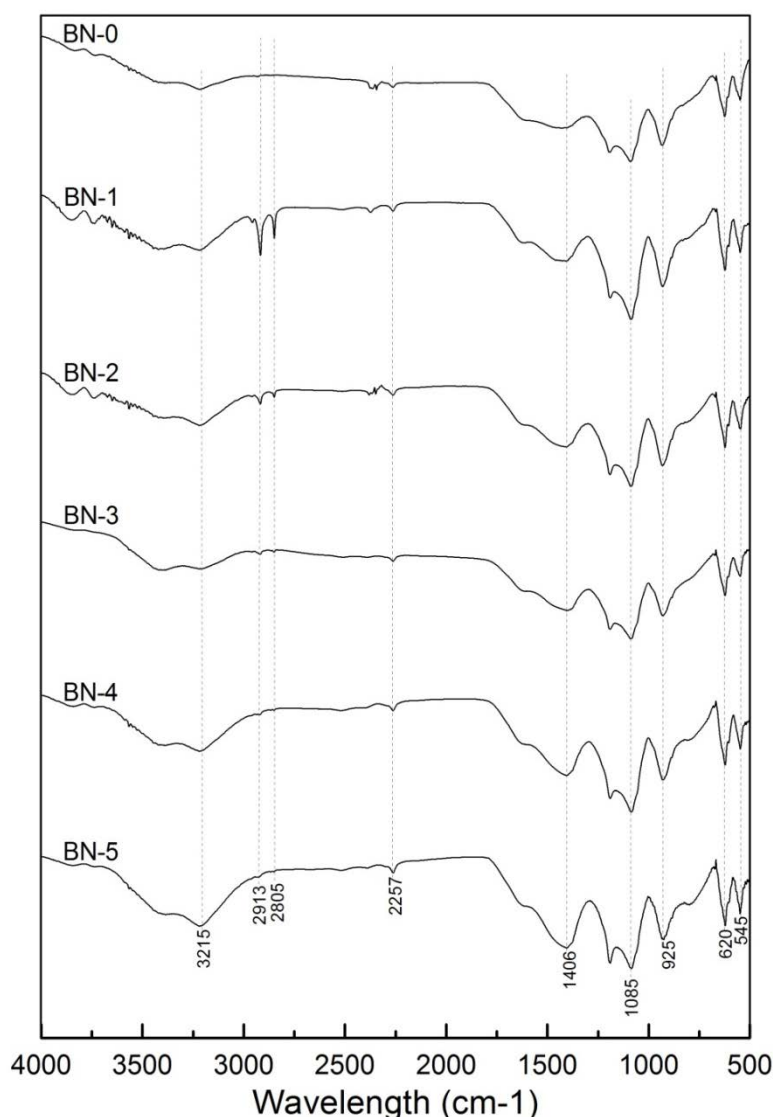


Figure 6. FTIR spectrum (a, b, c, d, e, f) showed the functional groups of residual char

3.5. Elemental analysis of char

XPS of char residual was performed using K-alpha XPS System (Thermo Scientific) with 14.0 kV of voltage and 300 W of power. XPS analysis provides detailed information for the elemental composition of charred intumescent fire retardant coating. Table 2 lists elemental contents of the char residue of BN-0, BN-4 and BN-5 after burned in the furnace. Their C1s spectra is shown in Figure 7 (a, b, c) respectively. The amount weight percentage of element C in char residue of BN-5 surpasses BN-0 and BN-4 by 18.04% and 10.18% respectively. Higher carbon content generally indicates higher level of char accumulation and higher level of cross-link [20]. This means there are more atoms and cross-link per unit area in char residue of the coating when boron nitride is incorporated. This will result in stronger and more compact char.

The peak from C1s spectra at around 285 eV is assigned to C–H and C–C in aliphatic and aromatic species in the sample while the peak at around 288.2 eV is ascribed to C–O bonds [19]. It can be seen that C1s peak intensity of BN-5 is higher compared to BN-0 and BN-4. The area of 285 eV in BN-5 increases from 18.46% and 33.73% to 47.81% compared to BN-0 and BN-4 respectively. The

result further shows that nano-BN increases the carbon accumulation in the coating which is favorable to its fire performance.

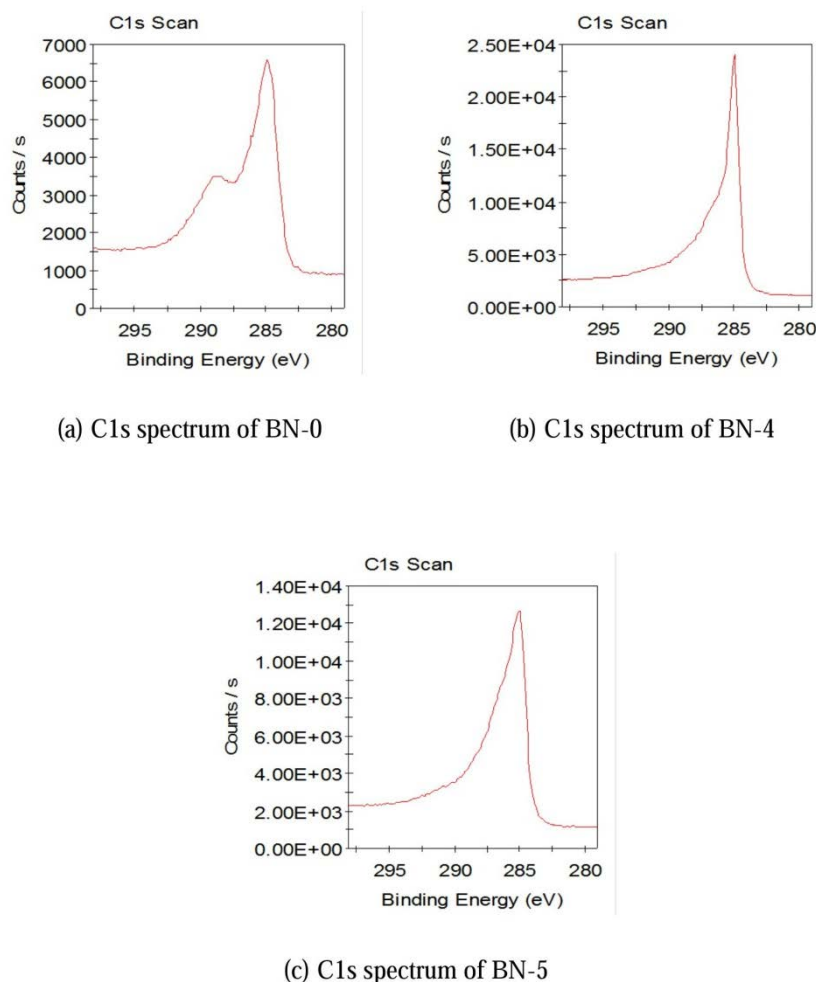


Figure 7. C1s XPS spectra (a, b, c) from char residue of intumescent fire retardant coatings

3.6. Analysis of char residual weight

TGA of intumescent fire retardant coating samples was performed on Pyris 1 TGA (Perkin Elmer) at 10°C/min under nitrogen over temperature range of 25°C–800°C. The TG curves of the intumescent fire retardant coatings are shown in Figure 8. It shows the final residual weight of the coatings before and after nano-BN was incorporated. The residual weight of each formulation at 800°C is: 34.64 wt% (BN-0), 37.85 wt% (BN-1), 39.62 wt% (BN-2), 41.38 wt% (BN-3), 44.50 wt% (BN-4) and 40.02 wt% (BN-5). The higher weight percentage of boron nitride contributed to an increase in the residual weight of BN-1 to BN-5 compared to BN-0. The TG curve shows that increasing the weight percentage of boron nitride increases the total residual weight. The residual weight of BN-4 formulation is 44.50%, which are 22.15 percent higher than that of BN-0. Based on these results, it is possible that boron nitride alongside boric acid are main components in the final protective layer developed in the char when the intumescent coating is exposed to fire.

The TG curves have four decomposition stages: stability stage (29 – 200 °C), formation of char layer (200 – 350 °C), char loss stage (350 – 550 °C) and inorganic layer formation stage (550 – 800 °C). The curves suggested there was little difference between the intumescent fire retardant coating without

filler and with nano-BN in the first and second stage. In third stage however, BN-4 and BN-5 with 4% and 5% nano-BN respectively shown little weight loss compared to other coatings. This suggested that nano-BN leads to the formation of a more thermally stable coating when it is incorporated at right weight percentage. In the first stage, weight loss was very small due to decomposition of volatile solvent and softening of the binder; and between 100–200 °C, boric acid degraded into metaboric acid and boron oxide [6]. During second stage, char began to form from the reaction of APP, EG and MEL. At 250 °C, APP began to decompose releasing ammonia and water and EG began to decompose releasing carbon dioxide [7]. MEL decomposed at 200–375 °C eliminating ammonia to further blows the charring layer [8] formed between APP, EG and boric acid. In the third stage, significant weight loss occurred due to further decomposition of APP into polyphosphoric and metaphosphoric acid [9]. In the final stage, only non-combusted char layer and inorganic substances were remained.

Nano fillers have superior surface-to-volume ratio but they are extremely difficult to disperse uniformly [10]. Thus, the weight percentage of nano fillers plays an important role in enhancing the intumescent fire retardant coating.

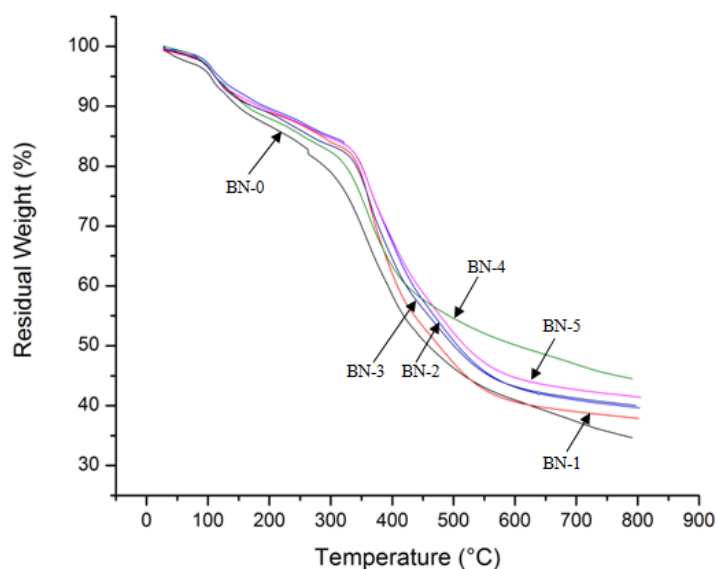


Figure 8. TGA of intumescent fire retardant coating with various percentages of boron nitride.

4. Conclusion

The effect of nano-boron nitride on thermal degradation of intumescent fire retardant coating were studied. Results show that by incorporating small quantities of nano-BN; it improves fire retardant properties of the intumescent fire retardant coating. This is evident from the measurement of substrate temperature which shows that the substrate temperature has been lowered by various degrees. The formulations BN-4 and BN-3 have shown better results on the substrate temperature, 138°C and respectively after 60 minute fire test. SEM results show improvements in the structure of residue char flakes when the wt% of boron nitride was increased. The TGA results demonstrate higher residual weight of intumescent fire retardant coating when nano-BN was introduced in the coating. As the weight percentage of nano-BN increased from 1%, 2%, 3%, 4% and 5%; the residual weight was 37.85%, 39.62%, and 41.38%, 44.50% and 40.02% respectively. This concluded that the increase of nano-BN will increase the percentage residual weight of the coating. XPS results show that nano-BN increases the carbon content and increases the C1s spectrum intensity. This will be beneficial in enhancing the strength of the char layer and improving the structure of the char layer itself. Thus, nano-BN as a filler could enhance the performance of intumescent fire retardant coating.

5. References

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