CHAPTER 4

RESULTS AND DISCUSSION

4.1 Chapter overview

In the current chapter, the results obtained from the physical, chemical and thermal tests are presented and analysed. The synthesis and characterization of the intumescent coating formulations before and after fire test will be discussed including the results of char expansion, char morphology, residual weight, heat insulation test and weathering test. The results obtained from the experiments will be compared with those reported by other researchers.

4.2 Coatings and their intumescence behaviour

In this study the effect of various compositions on char expansion, morphology of the intumescent coating has been investigated and are presented in the followings.

4.2.1 Intumescent coating based on one ingredient with binder

Four types of ingredients were used such as EG, APP, melamine boric acid. Each ingredient was added to the formulation to study its effect on char expansion and char morphology with epoxy and hardener. The four formulations have been tabulated in Table 3.9 in chapter 3. The coated samples were cured for 5-7 days at room temperature and tested at 500^{o}C in the carbolite furnace to examine the char expansion and char morphology.

4.2.2 Effect of EG on char expansion and char morphology of IF1-1

IF1-1 contains expandable graphite 14.28 wt% with epoxy and hardener 57.14, and 28.57 wt%, respectively. After the fire test, the physical appearance of IF1-1 coating that insulates the substrate due to oxidation of EG is as shown in Table 4.2. Char expansion due to oxidation of EG was 7.18 times, while the physical appearance of the char was hard. Some bubbles were seen on the surface of the char which show dehydration of water during the fire test. The SEM micrographs as shown in Figure 4.1(a, b) show a smooth outer surface of IF1-1 after the fire test which indicates linkage of graphite with epoxy and hardener. The inner surface of char shows the graphite flakes which are considered as the barrier that hinders heat penetration to the substrate.

Table 4.1: Intumescent coating based on one ingredient before furnace test

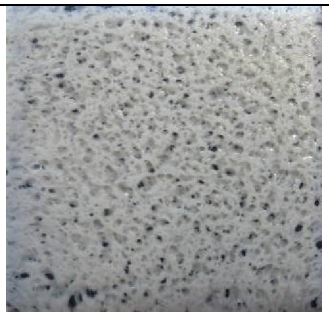
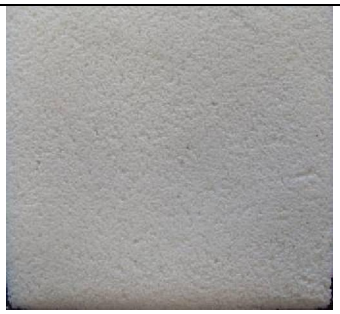
	Formulation IF1-1		**Formulation IF1-2**
Physical appearance			
Observations	– Less viscous – Easy to stir with mixer – Easy to apply with brush – Touch dry after 7 day	–	– Less viscous – Slightly yellowish – Severe coagulation of solid particles after a while – Touch dry after 8 days
	Formulation IF1-3		**Formulation IF1-4**
Physical appearance			
Observations	– Less viscous – Severe coagulation of solid particles after a while – Touch dry after 7 days		– Less viscous Touch dry after 6 days

Table 4.2: Intumescent coating based on one ingredient after fire test

	Formulation IF1-1	Formulation IF1-2
Physical appearance		
Observations	– Average coating thickness: 1.5mm – Average char thickness: 10.78mm – Expansion: 7.18 times – Weak char – small void in middle	– Average coating thickness: 2.0mm – Average char thickness: 17.42mm – Expansion: 8.71times – Weak char – Large void in middle
	Formulation IF1-3	**Formulation IF1-4**
Physical appearance		
Observations	– Average coating thickness: 1.0mm – Average char thickness: 2.1mm – Expansion: 2.1 times – Small void in middle – Cracked surface	– Average coating thickness: 2.37mm – Average char thickness: 4.8mm – Expansion: 2.02 times – large void inside char – Small bubbles on the surface

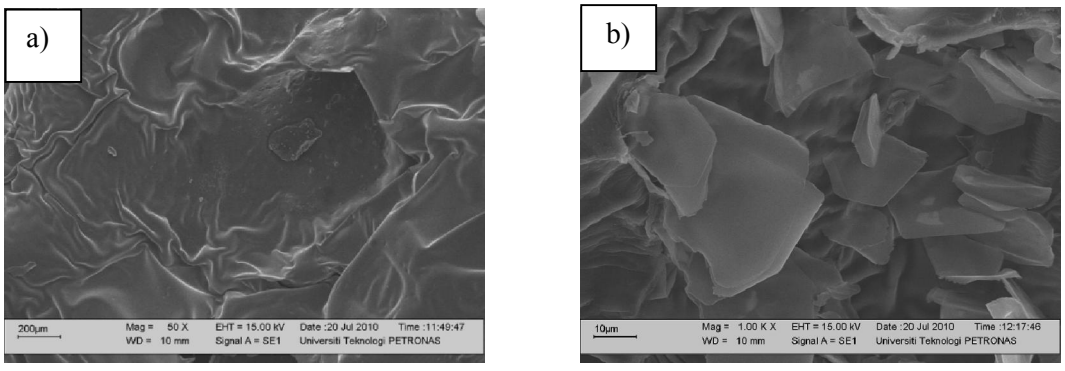
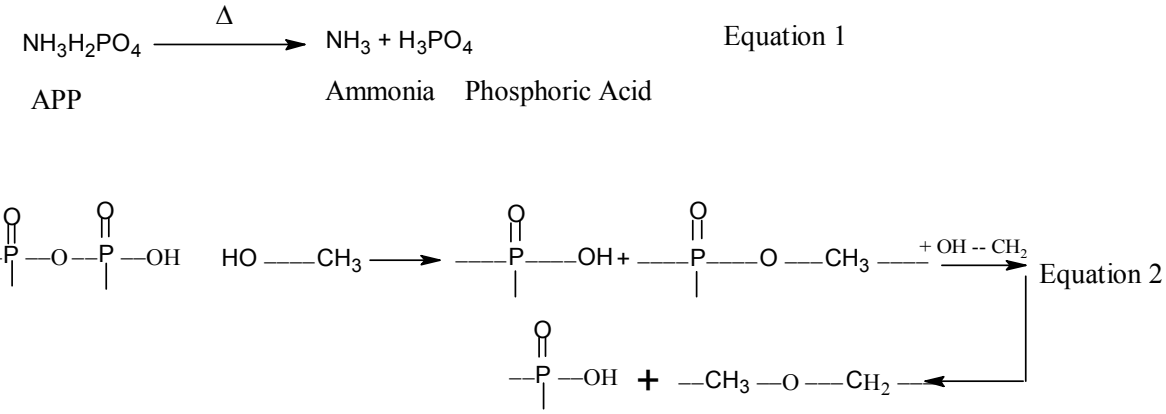


Figure 4.1: a) IF1-1 Smooth surface of the char of IF1-1

b) Microstructure of the inner surface of char IF1-1 showing flakes of graphite after burning at 500^{o}C

4.2.3 Effect of APP on char expansion and char morphology of IF1-2

IF1-2 contains 14.28 wt % APP and the rest is epoxy and hardener 57.14 wt % and 28.57 wt% respectively. After the fire test, this formulation insulated the substrate; expansion of this coating was 8.62 times which is 0.21 percent higher than IF1-1. IF1-2 coating was not bonded well with the steel substrate as APP is an acid source which initiates the chemical reaction of intumescent ingredients to form a carbonaceous char. There were no bubbles on the surface of IF2-1 char as shown in Table 4.2. From the physical appearance of char shown in Table 4.2 and SEM image, it is clear that the outer surface of IF1-2 char is very smooth as shown in Figure 4.2. The smooth carboneous char may be due to decomposition of APP and further reaction of polyphosphoric acid with rich polyhydric carbon source. The following chemical reaction as given in equation 1 and 2 may have occurred during the fire.



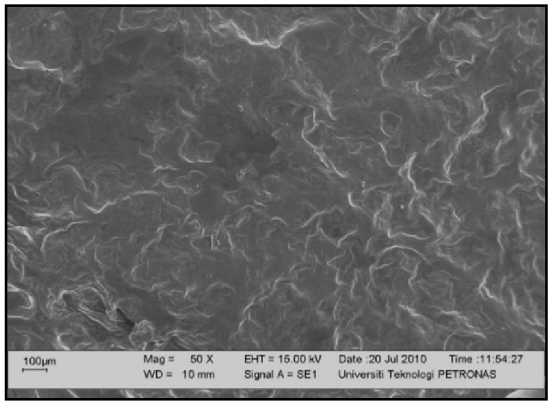


Figure 4.2: Outer surface of IF1-2 char, smooth surface represents uniform char layer on the substrate.

4.2.4 Effect of melamine on char expansion and char morphology of IF1-2

IF1-3 contains 14.28 wt% of melamine and the rest is epoxy and hardener 57.14 wt% and 28.57 wt% respectively. The expansion of char is 2.1times. As can be observed from the xxx in Table 4.2, IF1-3 did not expand well. There are cracks and holes observed on the surface of char; these are considered as due to decomposition of melamine into its derivates after 200^{o}C, releasing ammonia gas which produces holes on the surface of char. The cracks and holes can be observed in SEM image of IF1-3 char shown in Figure 4.3. These holes and cracks allow direct penetration of heat to the substrate. Therefore, melamine does not contribute towards protecting the steel substrate from fire. The decomposition reaction of melamine is explained in equation 3 below.

$$\underset{\text{Melamine}}{CH_3H_6N_6} \xrightarrow{-NH_3} \underset{\text{Melam}}{CH_3H_9N_{11}} \xrightarrow{-NH_3} \underset{\text{Melem}}{CH_3H_6N_{10}} \xrightarrow{-NH_3} \underset{\text{Melon}}{CH_3H_3N_9} \qquad \text{Equation 3}$$

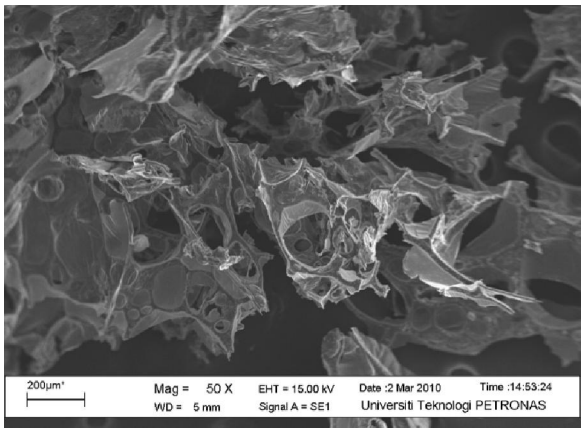


Figure 4.3: Represents the cracks and holes in the char of IF1-3

4.2.5 Effect of boric acid on char expansion and char morphology of IF1-4

IF1-4 contains 14.28 wt % of boric acid and the rest is epoxy and hardener, 57.14 wt % and 28.57 wt% respectively. Its expansion is 2.02 times which is lower than 225, 331, and 3.96 percent compared to IF1-1, IF1-2 and IF1-3, respectively. The physical appearance of char is as shown in Table 4.2. As evidenced from the SEM micrograph in Figure 4.4, the char that insulated the substrate have cracks and bubbles on its surface. During the fire test, when the temperature reached 120-140^{o}C the boric

acid decomposed into meta boric acid. After further temperature increase, it was converted into boron oxide at 140-200^{o}C and the final protecting layer of char was produced on the substrate.

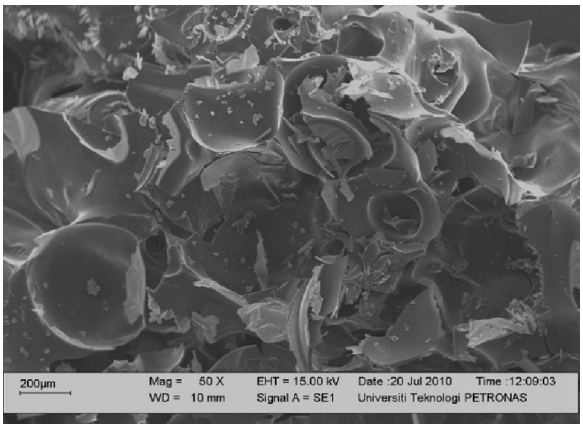


Figure 4.4: Cracks on the outer surface of boric acid with epoxy and hardener of IF1-4 coating

4.2.6 Conclusion

The above results have shown the individual effect of incorporating EG, APP, melamine and boric acid in intumescent formulations, with epoxy and hardener, in terms of physical appearance, expansion and morphology of char. The incorporation of APP in the formulation has resulted in enhanced and uniform expansion due to the reaction of with rich polyhydric carbon source. SEM micrograph of IF1-1 shows flaky appearance inside the char which indicates the presence of graphite in the char that acts as a fire retardant. The role of melamine as blowing agent alone did not yield significant char expansion. However, its role is further investigated with the incorporation of two intumescent ingredients. Boric acid, even though, shows minimum expansion but the flakes of boron oxide insulate the steel substrate.

4.3 Effect of two ingredients on intumescent behaviour

As listed in Table 3.10, six formulations were prepared to investigate the effect of two ingredients with epoxy and hardener on expansion, morphology, composition of char residue, and thermal degradation behaviour of coating. The coating formulations

were cured at room temperature for 6-8 days. The physical appearance and observation of coating are presented below in Table 4.3. Then the coatings were burned at 500^{o}C; the physical appearance and observation of the resulting char are shown in Table 4.3.

Table 4.3: Intumescent coating based on two ingredients before furnace test

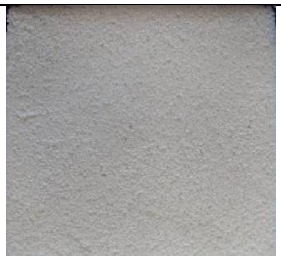
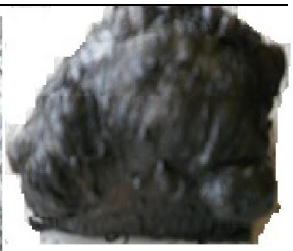
	Formulation IF2-1	**Formulation IF2-2**	**Formulation IF2-3**
Physical appearance			
Observations	– Less viscous – Easy to stir with mixer – Easy to apply with brush – Touch dry after 7 day	– Less viscous – Touch dry after 6 days	– Less viscous – Slightly yellowish – Severe coagulation of solid particles after a while – Touch dry after 8 days
	Formulation IF2-4	**Formulation IF2-5**	**Formulation IF2-6**
Physical appearance			
Appearance	– Less viscous – Severe coagulation of solid particles after a while – Touch dry after 7 days	– Less viscous – Severe coagulation of solid particles after a while – Touch dry after 7 days	– Less viscous – Severe coagulation of solid particles after a while – Touch dry after 7 days

Table 4.4: Intumescent coating based on two ingredients after furnace test

	Formulation IF2-1	**Formulation IF2-2**	**Formulation IF2-3**
Physical appearance			
Observation	– Ave coating thickness: 1.70mm	–Ave coating thickness: 1.65mm	– Ave. coating thickness: 1.35mm

	– Ave char thickness: 17.25mm – Expansion: 10.15 times	– Ave char thickness: 8.41mm – Expansion: 5.10times	– Ave char thickness: 14.1mm – Expansion: 10.5 times
	– Weak char – Large void in middle	– Weak char – Large void in middle	– strong char – Small void in middle
	Formulation IF2-4	**Formulation IF2-5**	**Formulation IF2-6**
Physical appearance			
Observation	– Ave coating thickness: 1.3mm – Ave char thickness: 5mm – Expansion: 3.8 times – Strong char – No void in middle	– Ave coating thickness: 1.5mm – Ave char thickness: 20mm – Expansion: 13.3 times – Weak char – No void in middle	– Ave coating thickness: 1.85mm – Ave char thickness: 13.6mm – Expansion: 7.35 times – Weak char – Large void in middle

4.3.1 Effect of EG and APP on char expansion and char morphology of IF2-1

The composition of IF2-1 coating consists of EG 6.66%, APP 13.13%, epoxy and hardener 53.3, and 26.66% by weight respectively. The char expansion of IF2-1 formulation is 10.15 times from the original coating thickness. Cracks, as shown in the photograph in Table 4.4, appeared on the surface of char after burning at 500^{o}C.. Char insulated the whole substrate while the surface of the char was rough with very small bubbles on its outer surface due to the release of CO_2 and N_2 from oxidation and decomposition of EG and APP, respectively. The cracks on the top surface can be clearly observed from the SEM micrograph of IF2-1 char as shown in Figure 4.5.

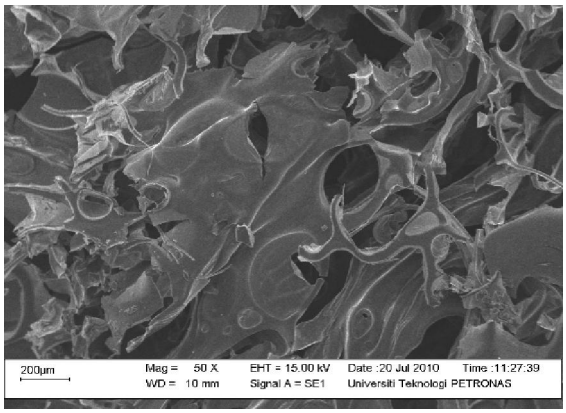


Figure 4.5: Rough and cracked surface of IF2-1 char

4.3.2 Effect of EG and melamine on char expansion and char morphology of IF2-2

The IF2-2 coating contains EG 6.66%, melamine 13.13%, epoxy 53.3% and hardener 26.66% by weight respectively. The IF2-2 coating shows cracks on the char surface and has an expansion of 5.10 times of the original coating thickness as illustrated in the photograph in Table 4.4. The expansion performance of IF2-2 formulation that contains both EG and melamine has improved considerably compared to when melamine was used alone as observed from IF1-3. However, cracks are observed on the char surface as shown in Table 4.3. As EG oxidizes at 350^{o}C, CO_2 gas is released; at this temperature also melamine decomposes into its derivatives producing excess amount of ammonia. Cracks were formed on the char as the gases escaped. Figure 4.6 shows the cracked surface with holes created inside the char. Effectively, the substrate was not fully protected from fire due to poor adhesion of char to substrate.

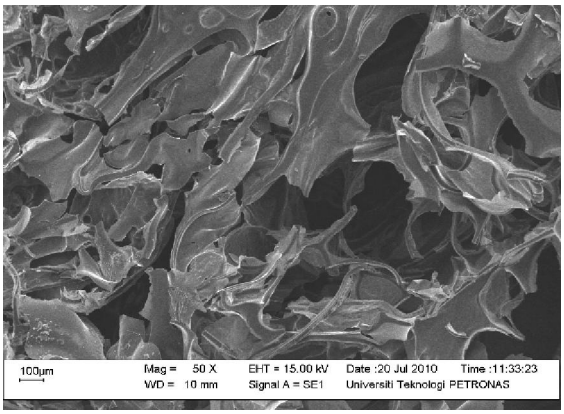


Figure 4.6: Rough and cracked surface of IF2-2

4.3.3 Effect of EG and boric acid on char expansion and char morphology of IF2-3

The IF2-3 coating contains 6.66 wt% of EG, boric acid 12.33 wt %, epoxy 53.33 wt% and hardener 26.66 wt%. The char expansion is 10.5 times and this is considered as good performance of the coating. The good adhesion of IF2-3 char to substrate is due to the addition of boric acid. Boric acid forms a hard glass with the release of

water and it can play the role as blowing agent by providing a “glue” to hold the char together and provides structural integrity to the char with the substrate [11]. The physical appearance of IF2-3 char is shown in Table 4.4.

A comparison of IF2-3 against IF2-1 and IF2-2, shows that IF 2-3 has the higher expansion performance by 3.34 and is 105.8 percent better than those coatings. The higher expansion and better adhesion of IF2-3 are most likely due to the release of CO_2 by EG that contributes to improved char expansion [129], and the decomposition of boric acid to boron oxide at 140-200^{o}C to form the final char residue [37], respectively. SEM micrographs show cracks and holes on the outer surface of the char as observed in Figure 4.7 (a), while the inner surface shows thick char residue which protects the substrate from fire as shown in Figure 4.7 (b).

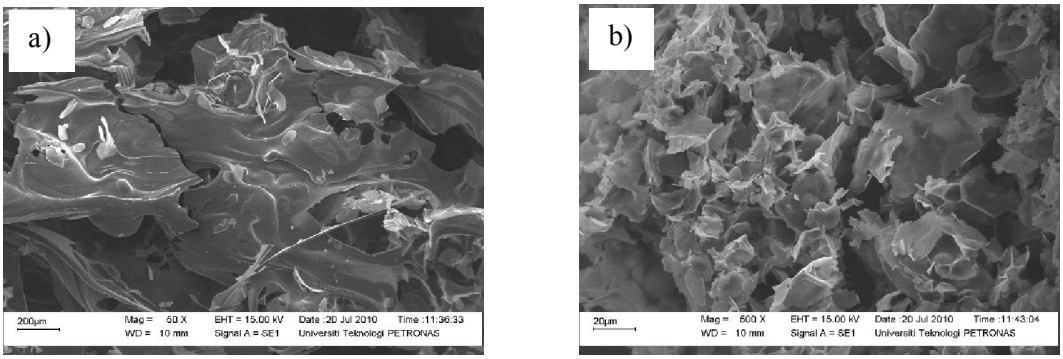


Figure 4.7: a) Rough and cracked top char surface of IF2-3 b) Inner side showed thick char with flakes of IF2-3

4.3.4 Effect of APP and boric acid on char expansion and char morphology of IF2-4

The formulation IF2-4 contains equal amount of APP and boric acid i.e. 12.5 wt%, 50 wt% epoxy and 25wt% hardener. The physical appearance of IF2-4 char shows a crack on the surface as illustrated in Table 4.4; there are small bubbles on the surface of IF2-4 due to emission of gases during fire test. The combination of APP and boric acid did not give a better result compared to when boric acid was used alone (IF1-4). The char expansion performance is 3.8 times which is 167, 197 and 34.2 percent less than IF2-1, IF2-2 and IF3-3, respectively. This formulation is not considered good in terms of char expansion, so the char morphology is not examined.

4.3.5 Effect of APP and melamine on char expansion and char morphology of IF2-5

The IF2-5 coating is composed of 12.5wt% APP, 12.5wt% melamine, 50 wt% epoxy, and 25wt% hardener. The expansion performance of this coating after fire test at 500^{o}C is 13.3 times. The IF2-5 char fully insulates the substrate as shown in Table 4.4. From Figure 4.8, a very smooth surface of the char after fire test is observed. APP is an acid source that reacts with epoxy to form a carboneous char while melamine is a blowing agent that releases N_2 and NH_3 gases [75, 130, 131]. These gases resulted in char expansion up to 11.2 times of the original coating thickness. The char expansion performance of IF2-5 is 30, 160.7, 26.6 and 263 percent better than IF2-1, IF2-2, IF2-3, and IF2-4, respectively.

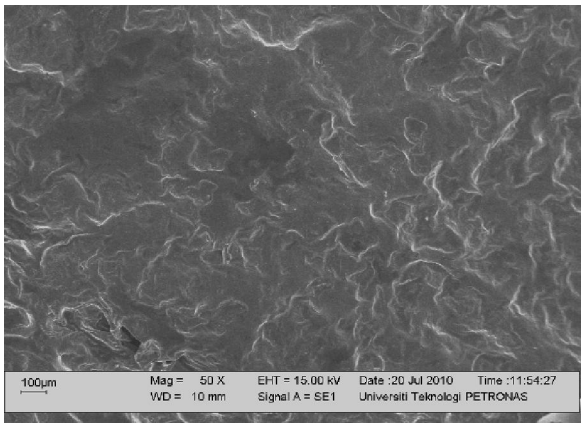


Figure 4.8: Smooth outer surface of IF2-5

4.3.6 Effect of melamine and boric acid on char expansion and char morphology IF2-6

The IF2-6 formulation contains melamine, boric acid, epoxy and hardener at 12.5wt%, 12.5wt%, 50wt% and 25wt%, respectively. The expansion of IF2-6 char is 7.35 times. The char was detached from the substrate and some cracks were observed on the surface as shown in Table 4.4. These observations are attributed to the presence of boric acid and melamine. Boric acid formed a glassy surface of boron oxide at 140-200^{o}C which increases char adhesion to the substrate [129]. While the decomposition of melamine released NH_3 and N_2 gases [75] during the fire test which resulted

expansion of char showed a weak adhesion with the substrate Figure 4.9 shows the micrograph of the outer layer of IF2-6 coating surface.

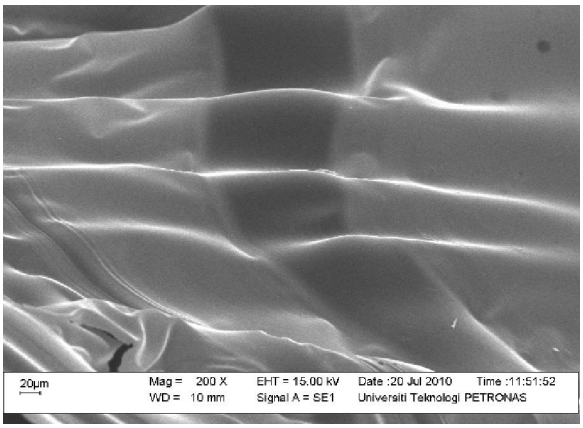


Figure 4.9: Charred layers outer surface of IF2-6

4.3.7 Char functional groups of ICFs based on two ingredients

FTIR used to determine the functional groups present in the residual char of two ingredients ICFs. FTIR spectra for coatings IF2-2, IF2-3 and IF2-4 are illustrated in Figures 4.10 to 4-13, respectively. The FTIR spectrum of IF2-2 char represents functional groups of residual char of melamine, epoxy and hardener as shown in Figure 4.10. A weak band at 1402cm^{-1} represents the CH_2 or CH_3 vibration due to the presence of melamine and polyamide hardener, 1588cm^{-1} represents the amino group NH_2 from melamine, 2384 and 2595cm^{-1} are weak stretching vibrations which represent the C=N [131]. The most pronounced variational features of melamine are shown as intense peaks at 1593-1598cm^{-1} corresponding to scissoring and ring, NH_2 stretching and ring side-chain CN stretching respectively [130, 131]. NH_2 observed at 992-1000cm^{-1} and stretching vibrations of asymmetric N-H at 3684-3791cm^{-1} are due to the gas phase of melamine [132].

Figure 4.11 shows a very clear spectrum of IF2-3, B-O-P bending motion is at 635cm^{-1}. It is confirmed from literature that the bending motion of B-O-P occurs at 635am^{-1} [133]. The O-P-O in borophosphate appeared at around 588-545 cm^{-1} and 1185-900 cm^{-1} represent the region of tetrahedral BO_4 [133, 134]. At 1449cm^{-1}, the peak represents CH_2 bending vibrations due to epoxy resin, while 2257 cm^{-1} and

2510 cm^{-1} show the weak stretching and bending vibrations of C=N due to the presence of polyamide hardener in the formulation. N-H bond stretching shown at 3223 cm^{-1} is from cured epoxy resin [135].

Figure 4.12 shows a typical FTIR absorption spectrum collected from residual char of IF2-4. B-O-P bending motion is observed at 626cm^{-1} and O-P-O in borophosphate appears at around 588-545 cm^{-1}. The peak at 545.8 cm^{-1} is assigned to δ(O–B–O) mode due to the presence of boron oxide in the char (B_2O_3) [133]. The two bands at 923 cm^{-1} and 1083cm^{-1} are ν(B–O–P) stretching mode of borophosphate in the char [134, 136]. The C≡N stretching vibration at 1615cm^{-1} is due to the presence of polyamide hardener. The band at 2346cm^{-1} is designated as B–H terminal bonds [137]. Small peaks in the region of 2850–2950cm^{-1} were also recorded. These peaks are assigned to an internal vibration mode of B–B internal icosahedral of the α-boron primitive cell [134]. A weak peak at 2933cm^{-1} is assigned to CH_2 stretching band and another at 3421cm^{-1} is assigned to OH; these two peaks correspond to carbon-rich polyhydric epoxy resin [135].

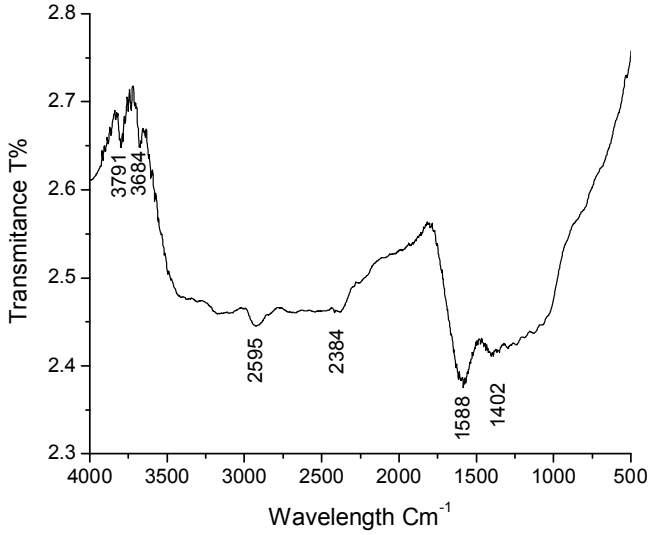


Figure 4.10: FTIR spectrum of IF2-2 residual char

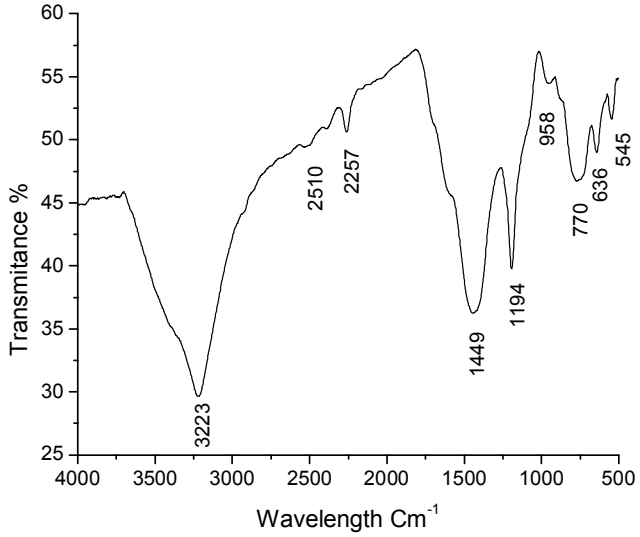


Figure 4.11: FTIR spectrum of IF2-3 residual char

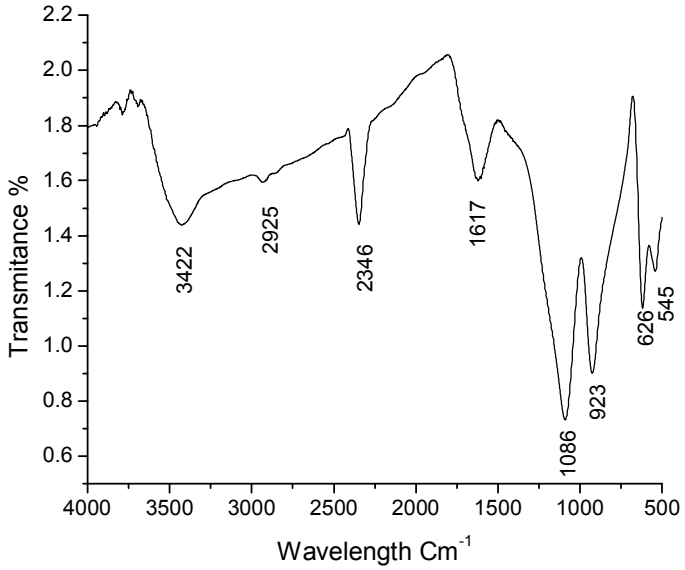


Figure 4.12: FTIR spectrum of IF2-4 residual char

4.3.8 Residual weight and derivative of thermogravimetric analysis (DTGA) of ICF based on two ingredients

The thermogravimetric characterizations of EG, boric acid, APP, melamine, epoxy and hardener are shown in Figures 4.13 to 4.16 respectively. From TG curve in Figure 4-13 it can be seen that there is no weight loss of APP before 290^{o}C. In the

range of 290-500°C, the residual weight was 40% due to decomposition of APP into phosphoric acid and the release of NH_3 and H_2O. In the range of 500-700°C, the residual weight was 19%, which is due to further decomposition of phosphoric acid into poly phosphoric acid and in the later stages it was converted to poly metaphosphoric acid [68]. Around 790°C, the residual weight left was 12%; during this time, the poly metaphosphoric acid was vaporized [11].

Figure 4-13 represents the two steps degradation mechanism of boric acid. The residual weight was 70% over the range of 100-140°C during the first step. The temperature range of the second step was 140–200°C with the residual weight 55% of the initial mass [12]. The curve of the boric acid indicates that there was no more weight loss after the temperature range 140–200°C.

The first step of degradation of boric acid involves the removal of water and conversion into metaboric acid. The second step corresponds to the dehydration of HBO_2 into boron oxide (B_2O_3) [12].

TG curve of EG is presented in Figure 4.13; the residual weight of EG is 87.48 wt% after 800°C. EG graphite starts to oxidize in the range of 200°C to 400°C, releasing CO_2 and H_2O. In Figure 4.14, it is shown that melamine had completely decomposed below 370°C and the residual weight of melamine is zero percent. The decomposition released NH_3 which caused gentle reduction of oxygen concentration [138]. Figure 4.15 shows the residual weight of bisphenol A epoxy resin BE-188 (BPA) after 500°C which is 4.12%, while the residual weight of ACR Hardener H-2310 polyamide amine is 1.33% after 500°C. The epoxy resin started to degrade after 300°C and complete degradation was achieved at about 460°C. Between 300 to 460°C the epoxy resin lost 90% of its mass [12, 139].

TG curves in Figure 4.16 explain the residual weight of the intumescent coating formulations based on two ingredients. The residual weight of char formed by each coating formulation at 800°C is 14.84 (IF2-1), 13.49 (IF2-2), 23.5 (IF2-3), 24.97 (IF2-4), 20.92 (IF2-5) and 15.94% (IF2-6). The residual weight (percentage) of IF2 coating formulations at different temperature zones is illustrated in Table 4.5.

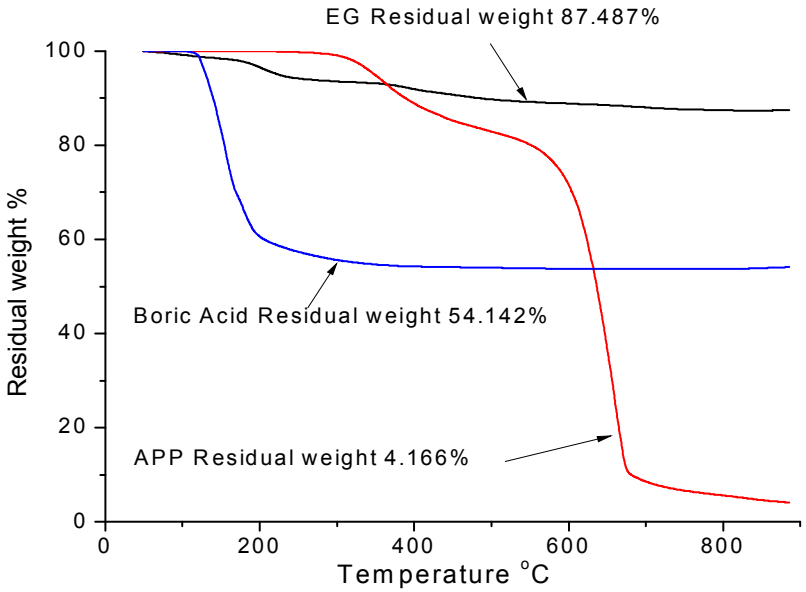


Figure 4.13: TGA of APP, EG and Boric Acid

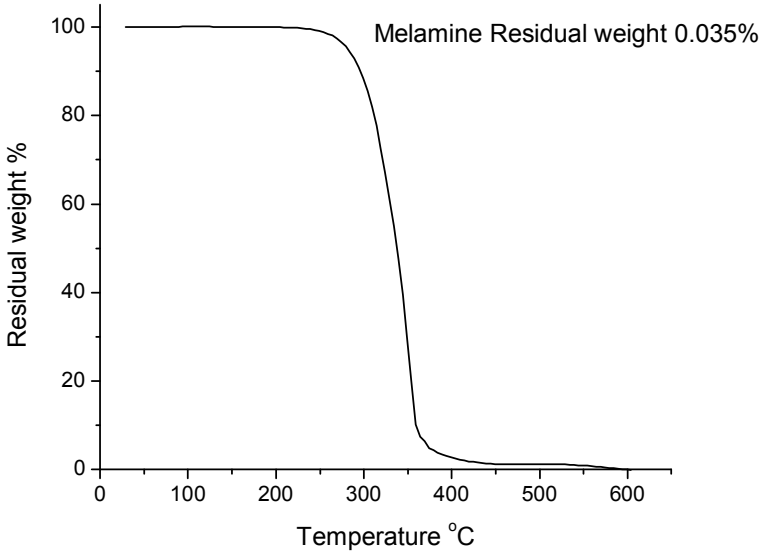


Figure 4.14: TGA of Melamine

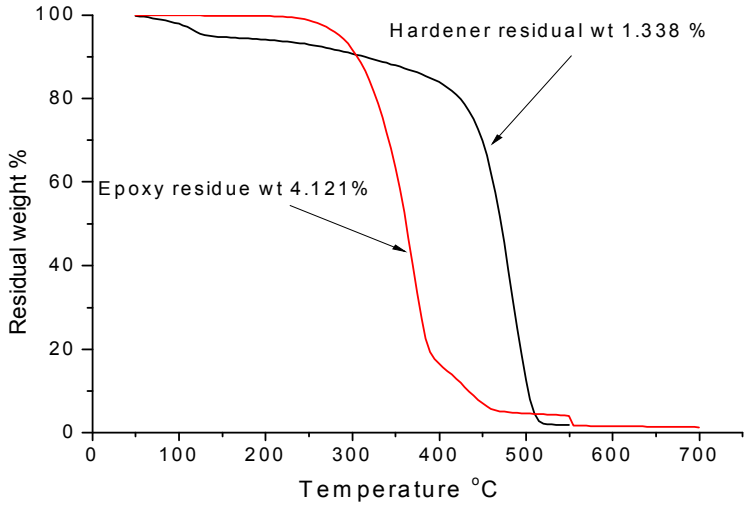


Figure 4.15: TGA of Epoxy and Hardener

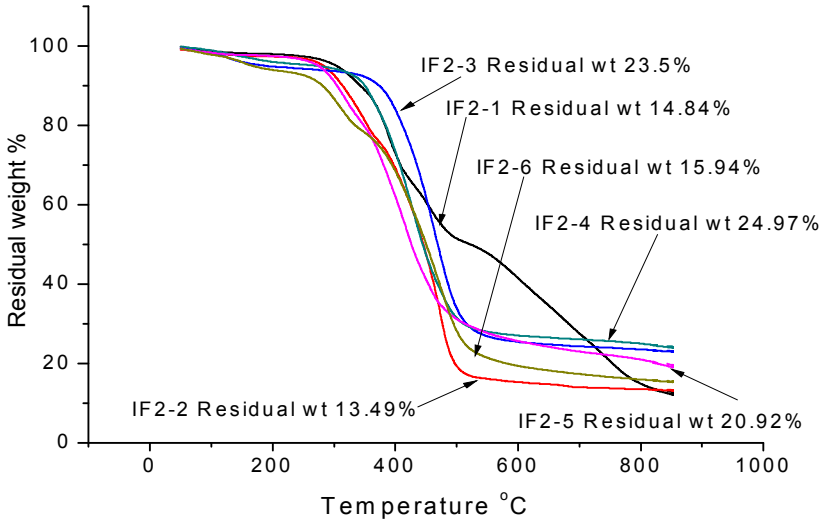


Figure 4.16: TGA of IF2 coating formulations

Table 4.5: Residual weight percent of IF2 at different temperature regions

Formulation No	Residual wt% 0-200°C	Residual wt% 200-350°C	Residual wt% 350-500°C	Residual wt% 500-800°C
IF2-1	98	88.9	49	14.8
IF2-2	97.3	81.2	74.6	13.49
IF2-3	94.8	90	67.2	23.5
IF2-4	96	87	31.4	24.9
IF2-5	96	89.1	31.4	20.9
IF2-6	93.98	78.2	28.16	15.9

As indicated in Table 4.5 and Figure 4.17, the residual weight of IF2-1 was 98% in the range of 0-200^{o}C attributed to the removal of hydroxide group from the cured epoxy resin. In the range of 200-350^{o}C, it decreased to 88.9% as the expandable graphite was being oxidized, and CO_2 and H_2O were released. In the range of 350-500^{o}C, the residual weight was 49% due to the decomposition of APP and the release of NH_3 gas and H_2O. In the range of 500-700^{o}C, the residual weight was 27.4% contributed by the release of phosphoric acid, poly phosphoric acid and poly metaphosphoric acid [11]. At 800^{o}C, the residual weight was 14.8%; during this time, poly metaphosphoric acid was vaporized [11].

The derivative of thermo gravimetricanalysis (DTGA) curves for IF2-1 is presented in Figure 4.18. DTGA curves show six steps of thermal degradation of IF2-1 at 101^{o}C, 340^{o}C, 384^{o}C, 451^{o}C, 597^{o}C and 753^{o}C. The first curve at 101^{o}C represents the degradation of cured epoxy, the 2nd curve at 340^{o} shows the oxidation of EG with the release of CO_2 and H_2O. The 3rd curve shows the degradation of APP and epoxy releasing NH_3 and H_2O respectively at 380^{o}C. The 4th curve represents the degradation of ACR polyamide hardener at 451^{o}C.

The 5th curve states further decomposition of APP into phosphoric acid, poly phosphoric acid and poly meta phosphoric acid. Poly metaphosphoric acid was further decomposed at 753^{o}C as indicated by the 6th curve of IF2-1 DTGA shown in Figure 4.18. In Figure 4.19, TGA curve illustrates the degradation of IF2-2 coating formulation. In the range of 0-200^{o}C, the residual weight was to 97.3% due to the removal of hydroxyl group from cured epoxy while in the range of 200-350^{o}C the residual weight was reduced to 81.2% due to the release of NH_3 gas from melamine. The residual weight was decreased to 77.77% in the range of 350-380^{o}C due to the oxidation of EG with the release of CO_2 and H_2O. In the range of 380-490^{o}C the residual weight was 74.6%, due to the degradation of epoxy and polyamide hardener which produces CO_2 and H_2O.

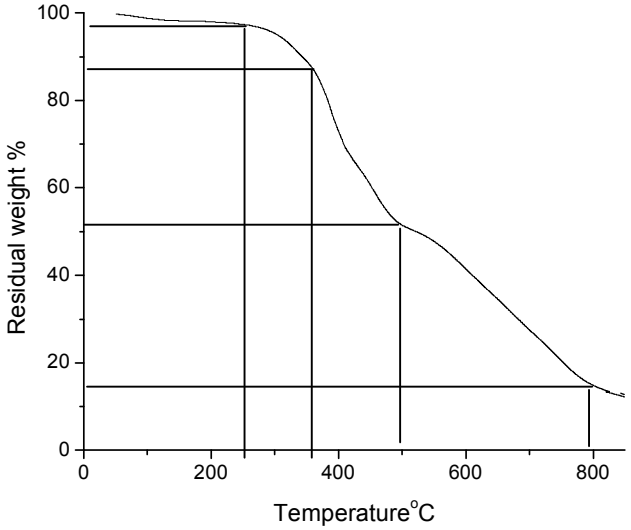


Figure 4.17: TGA of IF2-1coating showing residual weight 14.8% at 800°C

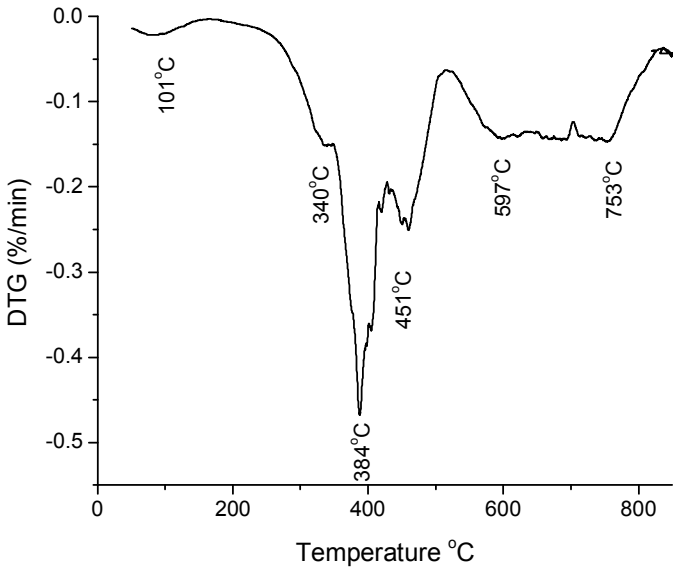


Figure 4.18: DTGA curve of IF2-1 showing six steps degradation

The DTGA curve in Figure 4.20 is showing three points of degradation. The first derivative curve shows the first degradation of cured epoxy resin at 104°C, while the second curve illustrated at 374°C shows the release of gases by melamine and EG. The 3rd curve at 476°C shows the degradation of epoxy and polyamide hardener.

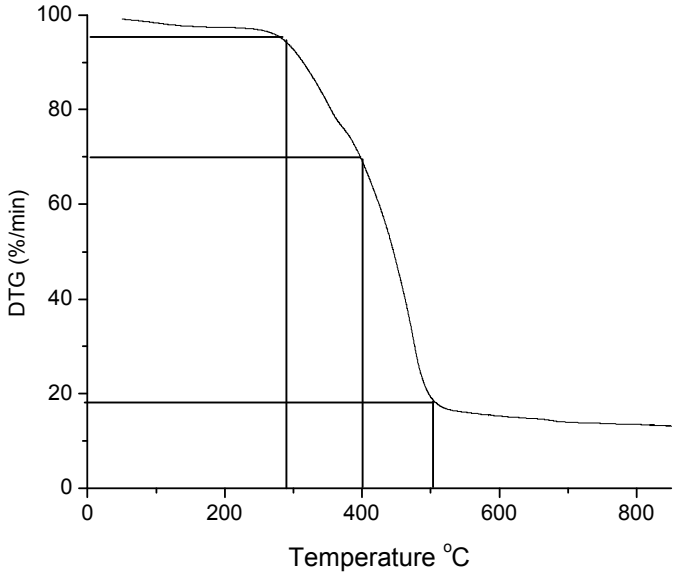


Figure 4.19: TGA of IF2-2 coating showing residual weight of 13.49% at 800°C

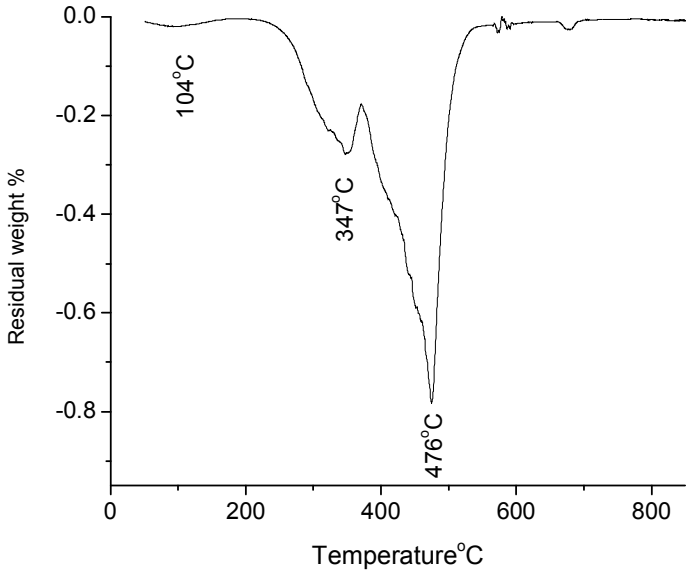


Figure 4.20: DTGA curve of IF2-2 coating showing three steps degradation

The TGA curve of IF2-3 in Figure 4.21 is showing three steps of degradation. In the range of 0-200°C, the residual weight was 94.8% as H_2O was released from the cured epoxy resin, and boric acid decomposed into meta boric acid at 100-140°C. In the 2nd temperature range of 200-370°C, the residual weight decreased to 90%. In this region, EG was oxidized with the release of CO_2 and H_2O. In the range of 370-510° C, the residual weight decreased to 67.2% due to the degradation of BPA cured epoxy

resin. The DTGA of IF2-3 in Figure 4.22 shows two degradation curves; the first curve that appears at 127°C is due to decomposition of boric acid into meta boric acid and release of H_2O from polyamide hardener. The 2nd curve showing maximum degradation of IF2-3 at 458°C represents oxidation and degradation of EG, epoxy and hardener.

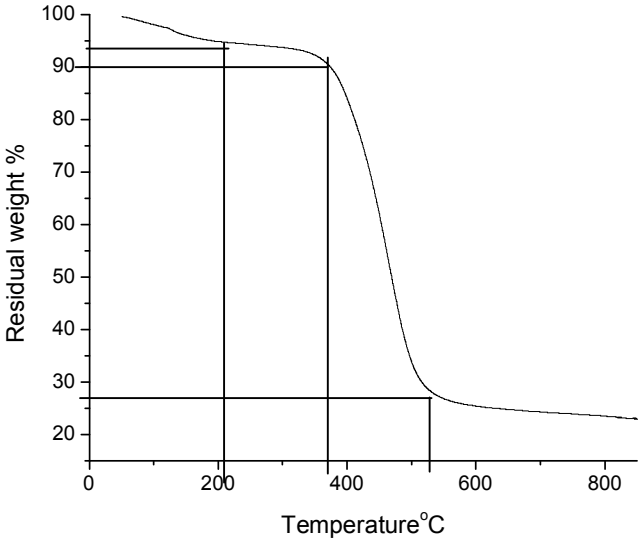


Figure 4.21: TGA curve of IF2-3 coating showing residual weight 23.5% at 800°C

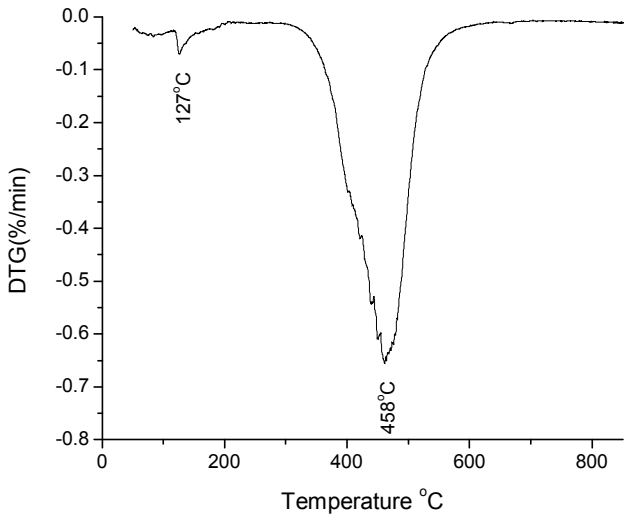


Figure 4.22: DTGA of IF2-3 coating showing two steps degradation

As illustrated in Figure 4.23, TGA of IF2-4 coating shows three steps of thermal degradation. In the range of 0-200°C, the residual weight was 96%; this weight lost is attributed to two events, firstly H_2O being released from cured epoxy resin and,

secondly the decomposition of boric acid into meta boric acid. The residual weight was 87% in the range of 200-360^{o}C due to decomposition of APP releasing H_2O and NH_3 gas. In the range of 360-500^{o}C, APP decomposed into polyphosphoric acid, and meta phosphoric acid, and epoxy and hardener decomposed into CO_2 and H_2O; these reduced the residual weight to 31.4%. At this stage, it might be possible that APP reacted with boric acid to form a thermally stable compound assumed as borophosphate [11]. The TGA results are confirmed by DTGA curves illustrated in Figure 4.24 which indicates five steps of thermal degradation at 129, 169, 362, 436 and 481^{o}C. The first degradation at 129^{o}C indicates degradation of cured epoxy, and conversion of boric acid into meta boric acid; while the second degradation at 169^{o}C is the conversion of meta boric acid into boron oxide. The decomposition of BPA epoxy resin and APP occurred at 362^{o}C and the degradation of polyamide hardener occurred at 481^{o}C.

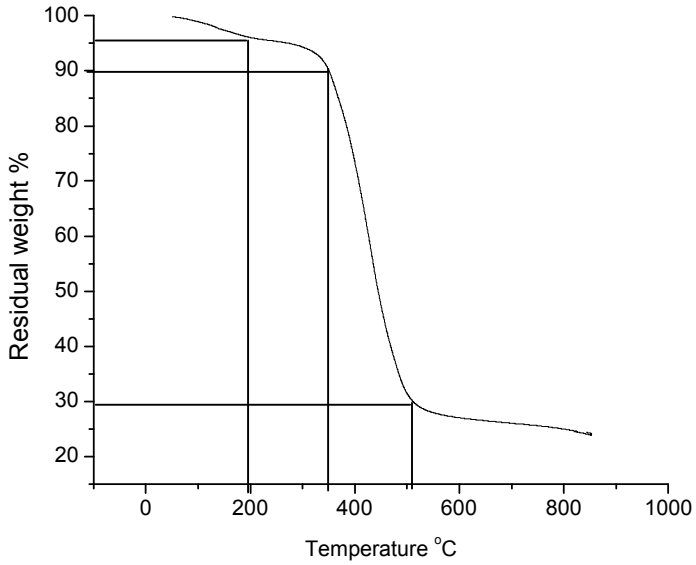


Figure 4.23: TGA of IF2-4 coating showing residual weight 24.9% at 800^{o}C

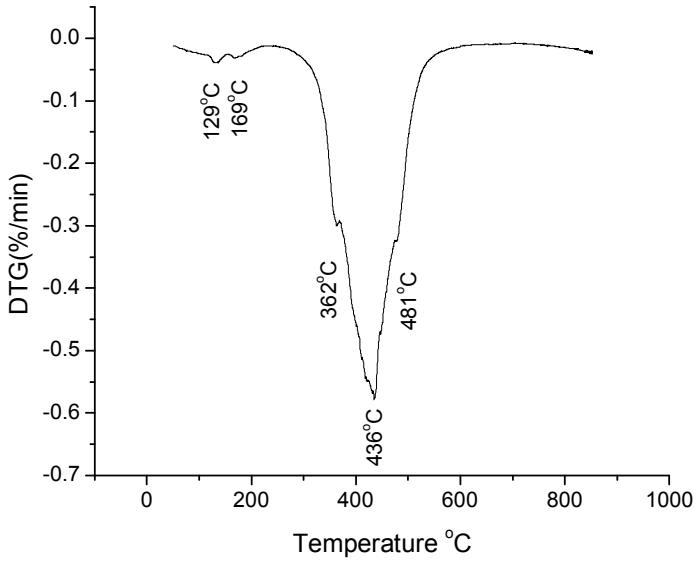


Figure 4.24: DTGA of IF2-4 coating showing five steps degradation

TGA curve of IF2-5 with residual weight 20.92% at 800°C is illustrated in Figure 4.25. The residual weight was 96% in the range of 0-200°C due to release of water from cured epoxy. In the range of 200-350°C, the residual weight was 89.1% as the melamine decomposed into its derivatives with the release of NH_3 gas. In the range of 350-500°C, decomposition of melamine derivatives, APP, BPA epoxy and polyamide hardener occurred and the residual weight was reduced 31.4%. The DTGA curve confirms four steps of thermal degradations of IF2-5 as shown in Figure 4.26. The first degradation occurred at 124°C due to cured epoxy, the second degradation happened at 317°C as melamine and APP decomposed with the release of NH_3 gas. Third degradation occurred at 422°C due to decomposition of epoxy while fourth degradation occurred at 456°C due to decomposition of polyamide hardener.

TGA of IF2-6 coating formulation is shown in Figure 4.27; the residual weight at 800°C is 15%. After the decomposition of cured epoxy and boric acid in the range of 0-200°C, the residual weight was 93.98%. In the second step of degradation that occurred between 200-350°C involving decomposition of melamine into its derivatives with the release of NH_3 gas, the residual weight was reduced to 78.2%. . The residual weight was further reduced to 28.16% due to decomposition of melamine derivatives, epoxy resin and polyamide hardener in the range of 350-500°C.

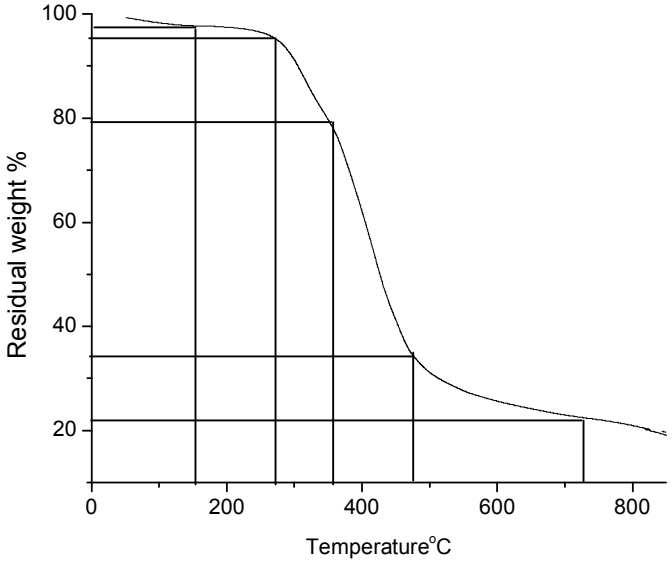


Figure 4.25: TGA of IF2-5 coating showing residual weight 20.92% at 800°C

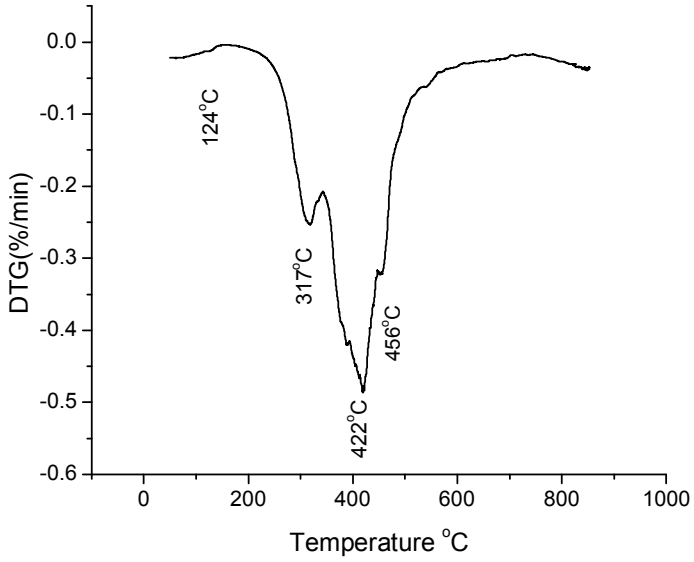


Figure 4.26: DTGA curve of IF2-5 coating showing four steps degradation

The DTGA curve confirms the degradation points of IF2-6 at 127, 149, 311 and 472°C stated in Figure 4.28. The first curve shows the cured epoxy released H_2O and boric acid convert into meta boric acid with the release of H_2O at 127°C. The 2nd curve shows the degradation of meta boric acid into boron oxide with H_2O release. B_2O_3 is a very hard glass, the crystals begin to break down at 300◦C, and a series of suboxides are produced with partial melting until full fusion is reached at 700◦C. Such

a thermally stable compound is interesting to use in an intumescent system because obtaining a high residue means that the amount of remaining char will be high. Moreover, due to the presence of boron oxide, the char is expected to show good mechanical resistance. The 3rd curve represents the decomposition of melamine into its derivatives at 311°C with the release of ammonia gas, while the decomposition of epoxy resin and polyamide hardener is indicated by the 4th degradation curve.

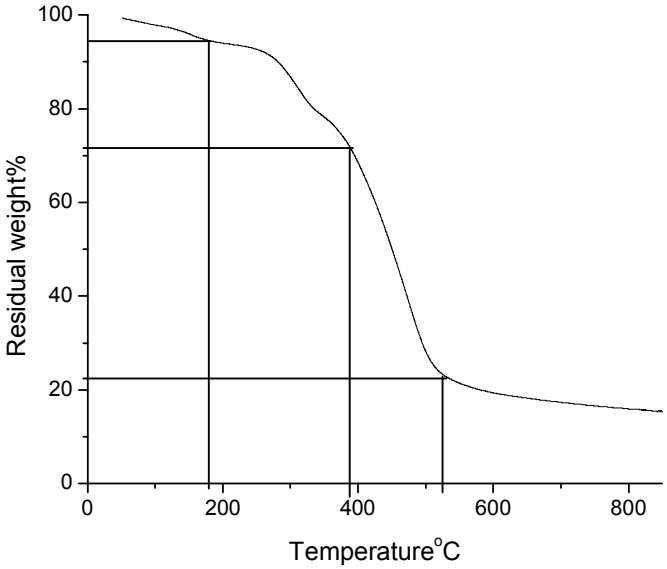


Figure 4.27: TGA of IF2-6 coating showing residual weight 15.94% at 800°C

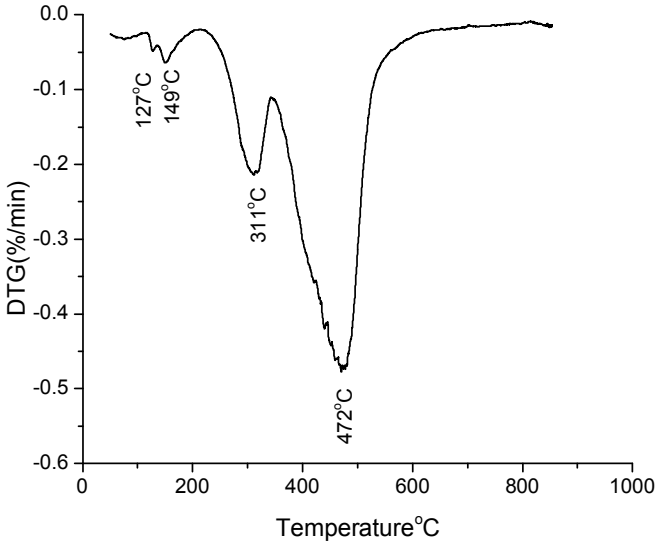


Figure 4.28: DTGA of IF2-6 coating showing three steps degradation

4.3.9 Conclusion

This investigation has been carried out with the objective to optimize the formulation which has best char expansion, morphology and residual weight. The above results have shown the behaviour of char expansion and char morphology. In general, the combination of two intumescent ingredients show better results in terms of char expansion performance and morphology compared to one intumescent ingredient. Among the six formulations (IF2-1, IF2-2, IF2-3 IF2-4, IF 2-5 and IF2-6), the formulation IF2-5 containing APP and melamine show the best char expansion performance i.e.13.3 times. In the case of char morphology, IF2-3 has the best char structure (flaky) compared to other formulations. FTIR analysis of IF2-2 and IF2-3 show presence of the functional groups such as B-O-P, O-P-O and O-B-O in the char residue which hindered heat penetration to the substrate. The residual weight of IF2-4 was 24.9%, which has shown the best residual weight among the formulations with two types of binder ingredient. IF2-5 shows better results in terms of char expansion with smooth char structure, but the residual weight is 19 percent less than IF2-4. On the other hand, IF2-3 formulation shows better char structure, but the char expansion is 21 percent less than IF2-5 and 176 percent higher than IF2-4. Whilst, IF2-4 shows better residual weight of 24.9% as boric acid and APP reaction product is borophosphate, but its char expansion is only 3.8 times which is 167, 34, 176, 250 and 93% lower than IF2-1, IF2-2, IF2-3, IF2-5 and IF2-6, respectively.

4.4 The effect of three ingredients on char expansion, morphology, composition of char and residual weight of IF3 ICFs

This study was carried out to investigate the effect of three ingredients with epoxy and hardener on char expansion, morphology, composition of residual char, presence of functional groups in char residue, and residual weight of char. Four formulations were prepared and the compositions have listed in Table 3.11, chapter 3.

Table 4.6: Intumescent coating based on three ingredients before furnace test

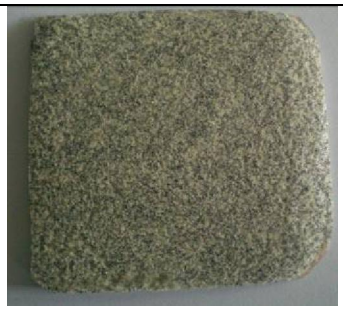
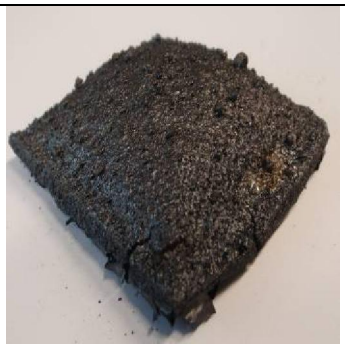
	Formulation IF3-1	**Formulation IF3-2**
Physical appearance		
Observations	Less viscous Easy to stir with mixer Easy to apply with brush Touch dry after 7 day	Less viscous Easy to stir with mixer Easy to apply with brush Touch dry after 7 days
	Formulation IF3-3	**Formulation IF3-4**
Physical appearance		
Observations	Less viscous Slightly yellowish Severe coagulation of solid particles after a while Touch dry after 7 days	Less viscous Severe coagulation of solid particles after a while Touch dry after 7 days

Table 4.7: Intumescent coating based on three ingredients after furnace test

	Formulation IF3-1	**Formulation IF3-2**
Physical appearance		
Observations	– Ave coating thickness: 2.0mm – Ave char thickness: 4.1mm	– Ave coating thickness: 1.65mm – Ave char thickness: 5.85mm

	– Expansion: 2.05times – Soft char – Cracks on the outer surface	– Expansion: 3.5 times – Hard char – Small bubbles on surface
	Formulation IF3-3	**Formulation IF3-4**
Physical appearance		
Observations	– Ave coating thickness: 2.0mm – Ave char thickness: 5.15mm – Expansion: 2.57 times – Hard char – Small void in middle – Small bubbles on surface	– Ave coating thickness: 2.0mm – Ave char thickness: 25mm – Expansion: 12.43 times – Weak char – Large void in middle

4.4.1 Effect of three ingredients on char expansion and char morphology of IF3-1 formulation

IF3-1coating contains 11.11% of APP, 11.11% of melamine, 11.11% of boric acid, 44.44% of epoxy and 22.22% of hardener. Based on the physical appearance of IF3-1 as shown in Table 4.7, it is evident that the char insulated the steel substrate after the fire test at 500^{o}C. The char expansion of IF3-1 is 2 times from its original coating thickness. Figure 4.29 shows a rough surface texture of IF3-1 char and presence of cracks on the surface. The char could not sustain its integrity during expansion due to its melamine content as melamine released gaseous N_2 during the fire test when the temperature reached around 280^{o}C, thus the rough and cracked char structure.

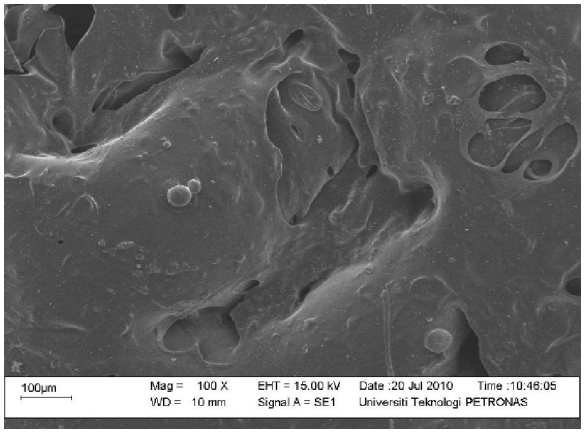


Figure 4.29: SEM micrograph showing rough and cracked surface of IF3-1 char

4.4.2 Effect of three ingredients on char expansion and char morphology of IF3-2 formulation

The IF3-2 char insulated the substrate, as shown in Table 4.7. The coating contains 5.88% of EG, 11.76% of melamine, 11.76% of boric acid, 47.05% of epoxy and 23.52% of hardener as stated in Table 3.11. In the absence of APP (acid source) in IF3-2 coating, the expansion of char is 3.5 times, and the char insulation is better than IF3-1 char. The better char insulation property is attributed to APP which initiates a long chain chemical reaction with rich polyhydric carbon source to form carboneous char[105]. In the absence of long chain reaction, the expansion of char is reduced. The SEM image in Figure 4.30 shows a smooth surface of IF3-2 as EG has a hexagonal structure which created a smooth char structure during fire test and protected the substrate from heat.

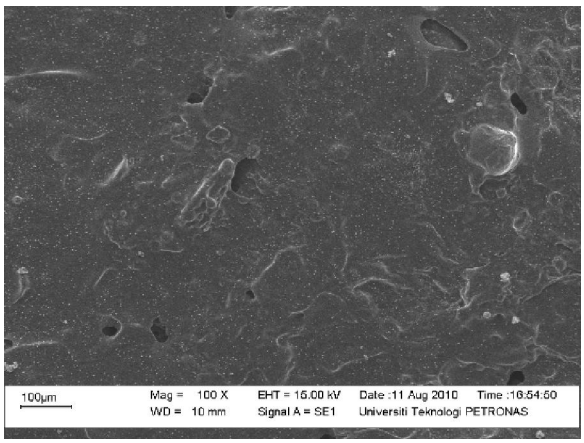


Figure 4.30: SEM micrograph showing smooth surface of IF3-2 char

4.4.3 Effect of three ingredients on char expansion and char morphology of IF3-3 formulation

This formulation contains 5.88% of EG, 11.76% of APP, 11.76% of boric acid, 47.05% of epoxy and 23.52% of hardener. The physical appearance and observations show that the char fully insulates the substrate. The char expanded 2.57 times. As discussed earlier, boric acid decomposed to boron oxide at 140-200^{o}C which further reacted with APP to form borophosphate. Boron oxide is a hard glassy material which hinders char expansion of the coating formulation. The SEM micrograph in Figure 4.31 shows a mixture of rough and smooth char surface.

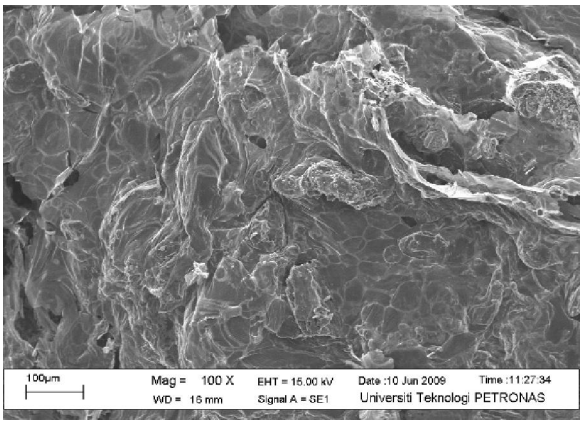


Figure 4.31: SEM micrograph showing rough and smooth surface of IF3-3 char

4.4.4 Effect of three ingredients on char expansion and char morphology of IF3-4 formulation

In this formulation, char expansion of IF3-4 coating is 12.43 times. The physical appearance of IF3-4 char shows a weak char and the presence of large voids inside the char; char adhesion with the substrate is weak as there is no reaction product which sustained the char integrity. As discussed previously, boric acid forms a glassy surface of boron oxide with char and substrate, so due to the absence of boric acid weak char and poor adhesion is observed in IF3-4. From Figure 4.32, the SEM image shows a smooth outer surface of the IF3-4 char. In this coating composition, all three ingredients (EG, APP, melamine) contributed to the expansion and smooth surface of the carboneous char.

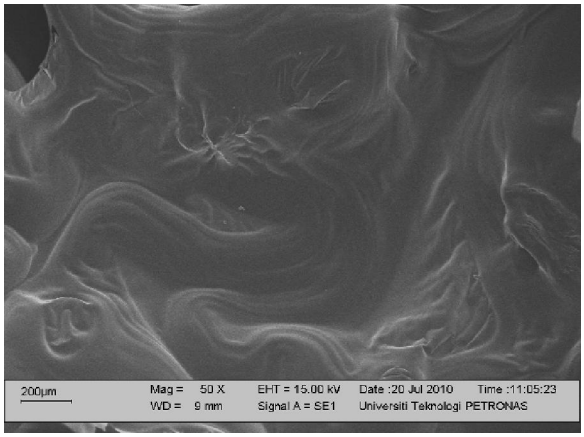


Figure 4.32: SEM micrograph showing smooth outer surface of IF3-4 char

4.4.5 Char composition of three ingredients based ICFs

When the char layer is progressively oxidized at high temperature, barely a few vague carbon and inorganic materials remained in the carboneous char. The inorganic materials become an important protecting shield at later stage of fire, when the temperature is higher than 600^{o}C [67]. The residual char of IF3-1, IF3-2, IF3-3 and IF3-4 were analyzed by X-ray diffraction using EVA software; the XRD spectra are shown in Figures 4.33, 4.34 and 4.35. The XRD spectra of IF3-1 and IF3-2 char are presented in Figure 4.33.The spectrum of IF3-1 shows the presence of borophosphate (JCPDS card no 34-0132) at 2θ = 24.5^{o} with d spacing value 3.61 and boron oxide (JCPDS card no 06-0297) at 2θ = 40^{o} with d spacing value 2.25, respectively. The spectrum of IF3-2 shows the presence of graphite only (JCPDS card no 12-0212) at 2θ = 26^{o} with d spacing 3.37. It is clear from both spectra that due to the absence of EG in IF3-1 formulation, the char residue contains borophosphate and boron oxide while in the absence of APP in IF3-2 coating the char contains graphite. These show that there is a chemical reaction between boron oxide and APP to form borophosphate, which is final char protective residue.

The results of XRD show that the reaction between boric acid with APP and O_2 enhanced the performance of anti-oxidation of the intumescent fire retardant coating due to presence of borophosphate and boron oxide which act as protective char layer on the steel substrate.

Figure 4.34 presents the XRD spectrum of IF3-3, which shows the presence of borophosphate, graphite and boron oxide at θ values of 24.5, 26, and 40 with d spacing values of 3.6, 3.47 and 2.25, respectively. This shows that APP reacted with boron oxide to form borophosphate.

Figure 4.35 shows the XRD spectrum of IF3-4, which shows the existence of graphite at $2\theta = 26^{o}$ and d spacing value of 3.47. From this result, it is clear that without boric acid, APP decomposed into polyphosphoric acid and no further reaction occurred between 400°C to 600°C and after 600°C all phosphorous is lost [11]. Similarly, in IF3-2 coating without APP, boric acid decomposed into boron oxide. These account for the graphite peak observed in the spectra of IF3-2 and IF3-4.

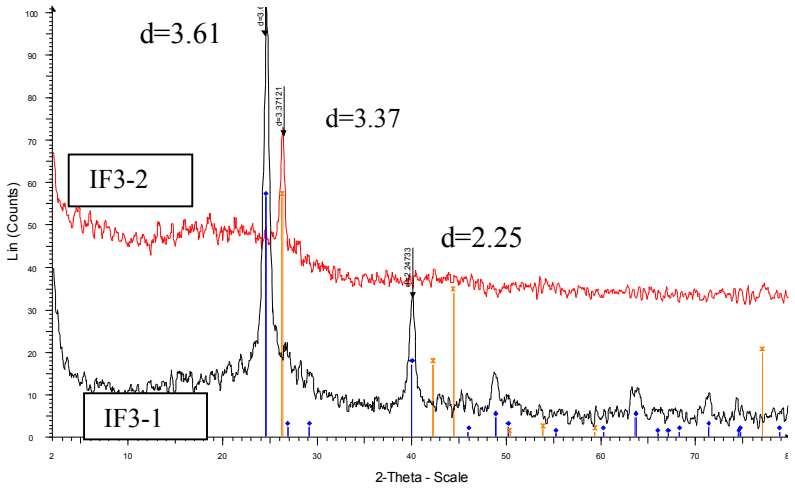


Figure 4.33: X-RD spectra of IF3-1 and IF3-2 char

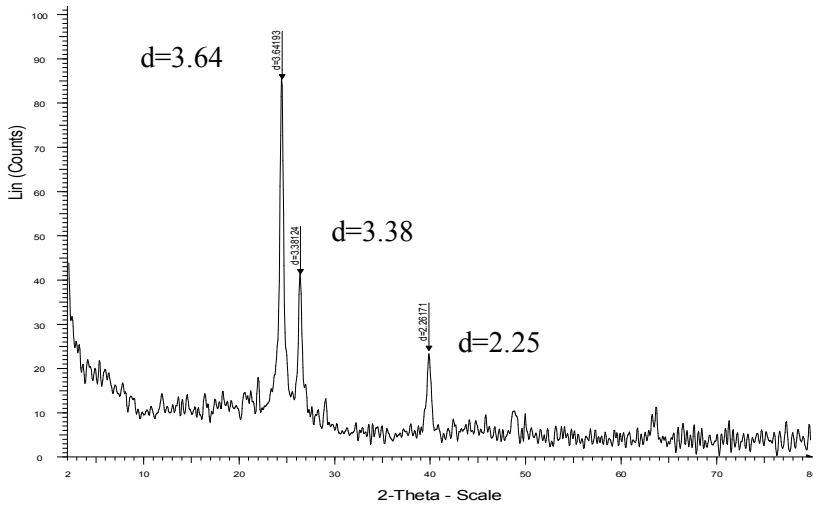


Figure 4.34: XRD spectrum of IF3-3 char

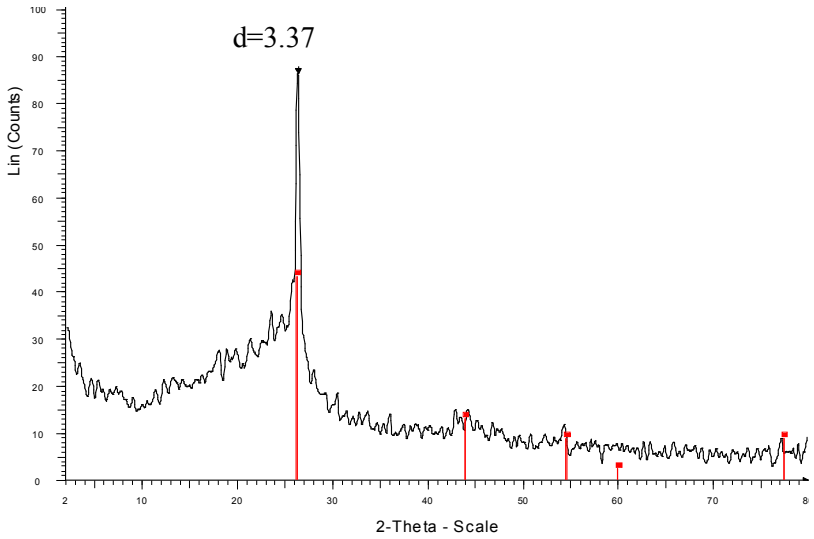


Figure 4.35: XRD spectrum of IF3-4 char

4.4.6 Char functional groups of three ingredients based ICFs

The residues of char have been analyzed using spectroscopic tools (XRD, FTIR, and TGA) to determine the mechanism of interaction between EG, APP, melamine, boric acid, epoxy and hardener. The FTIR spectra of residual chars of IF3-1, IF3-2, IF3-3 and IF3-4 are shown in Figures 4.36 to 4.39, respectively. The FTIR spectrum of IF3-1 char residue which contains APP, melamine, boric acid with epoxy and hardener is shown in Figure 4.36. This spectrum shows B-O-P bending motions at 602cm^{-1} and O-P-O in borophosphate which appeared at around 1185-900cm^{-1} represents the region of tetrahedral BO_4 [133, 134, 136, 137, 140]. A weak band at 1402cm^{-1} represents the CH_2 or CH_3 vibration due to melamine and poly amide hardener, and the peak at 1588cm^{-1} is assigned to amino group NH_2 in melamine and APP [141]. The peak at 1622cm-1 shows the C=C stretching vibration, while the peaks at 2394 and 2922cm^{-1} indicate a weak stretching vibrations due to the presence of C=N ascribed to polyamide hardener. The O-H bond stretching that appeared at 3416cm^{-1} is due to epoxy resin [135].

Figure 4.37 shows the FTIR spectrum of IF3-2 char. Very weak bending vibrations are observed at 723 and 1029cm^{-1} of O-P-O ascribed to APP in the formulation. The stretching vibration at 1418cm^{-1} characteristic of CH_2 or CH_3

vibration is ascribed to melamine and epoxy resin. The weak vibration at 2921 cm^{-1} of C=N, 3219 cm^{-1} of N-H and 3394 cm^{-1} of O-H group presents in the char is due to decomposition of melamine and cured epoxy [135].

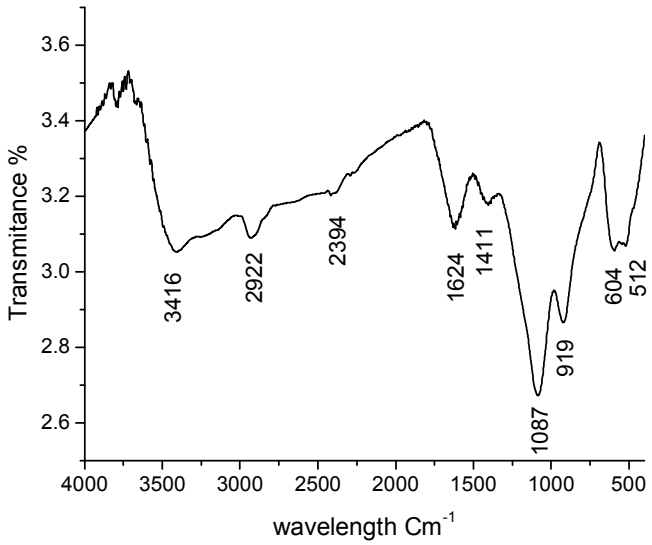


Figure 4.36: FTIR spectrum of IF3-1char

Figure 4.38 shows the spectrum of IF3-3 formulation. B-O-P bending motions at 624cm^{-1} and O-P-O in borophosphate appeared at around 588-545cm^{-1}. The peak at 541cm^{-1} in B_2O_3 is assigned to δ(O–B–O) mode. The two bands at 927cm^{-1} and 1089 cm^{-1} are identified as ν(B–O–P) stretching mode [134]. The peak at 1089cm^{-1} is assigned to B–O–H stretching mode [136]. A very weak bending vibration at 1457cm^{-1} either a CH_2 or CH_3 vibration is ascribed to epoxy resin. A C≡N stretching vibration at 1615cm^{-1} is attributed to cured epoxy resin that contained poly amide hardener [135].

The spectrum of IF3-4 char is shown in Figure 4.39. The peaks representing O-P-O in phosphate appeared at 975cm^{-1} and 1152cm^{-1}. The peak at 1402cm^{-1} representing CH_2 or CH_3 vibration is due to decomposition of melamine and poly amide hardener, the peak at 1622 cm^{-1} shows the C=C stretching vibration while the peak at 2355cm^{-1} is identified as a strong stretching vibration due to C=N in the polyamide hardener .

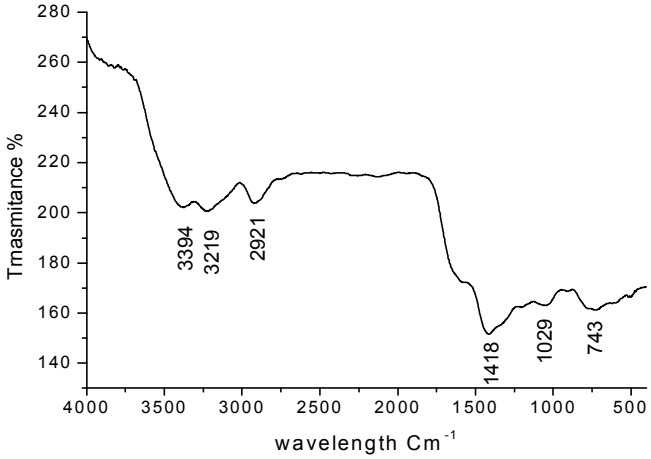


Figure 4.37: FTIR spectrum of IF3-2 char

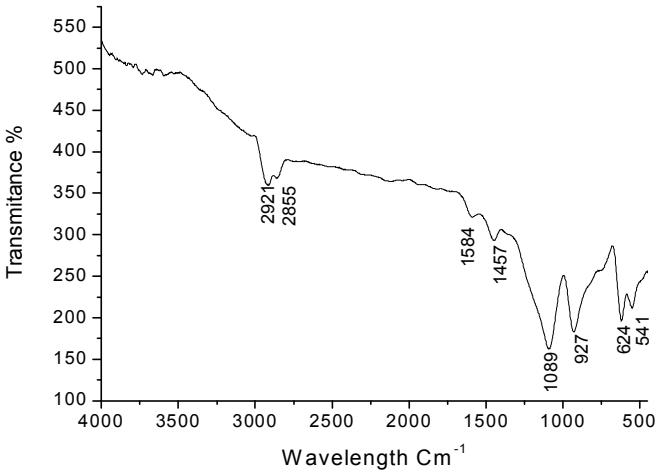


Figure 4.38: FTIR spectrum of IF3-3 char

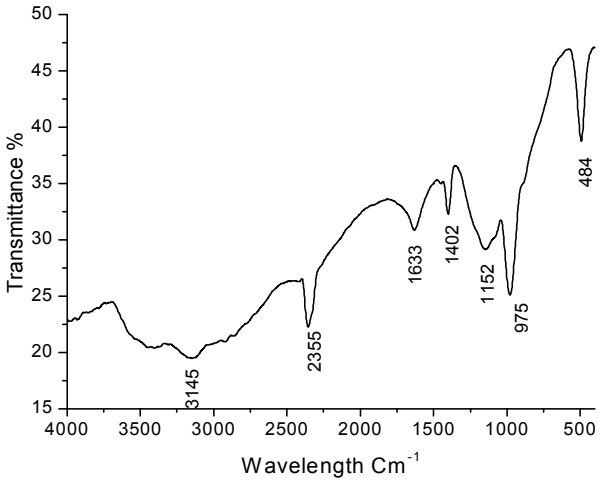


Figure 4.39: FTIR spectrum of IF3-4 char

4.4.7 Residual weight and DTGA of three ingredients based ICFs

TG curves in Figure 4.40 explain the residual weight of IF3 coating formulations. The residual weight of char formed by each coating formulation at 800°C is 22.11% (IF3-1), 20.33% (IF3-2), 30.98% (IF3-3) and 24.54% (IF3-4). Coating formulations based on three ingredients show better residual weight compared to coatings based on two ingredients. . The IF2-4 has a maximum residual weight of 24.97%, while IF3-3 has a residual weight of 30.98%. IF2-4 contains 12.5% APP and 12.5% boric acid with 50% epoxy and 25% hardener, while IF3-3 contains 5.88% EG, 11.76% APP, boric acid 11.76%, 47.05% epoxy and 23.25% hardener. A comparison of the compositions of IF3-3 and IF2-4, and their residual weights, suggests that the addition of 5.88 wt% of EG in IF3-3 coating increased the residual weight of IF 3-3 by 24%.

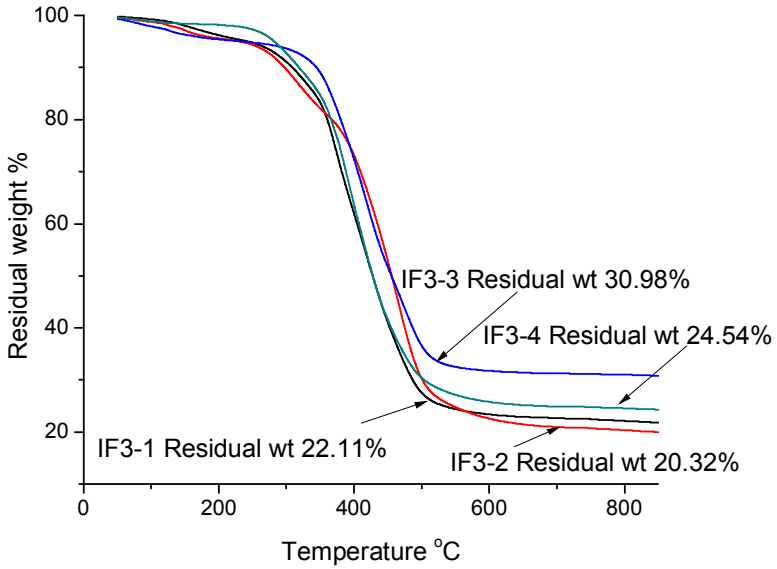


Figure 4.40: TGA curves of IF3 coating formulations

Figure 4.41 shows the TGA curve of IF3-1 coating. In the range of 0-200°C the residual weight was 96.2%; the weight loss is mainly due to the release of H_2O from cured epoxy resin and boric acid. The decomposition of APP in the range of 200-350°C releasing gaseous NH_3 and H_2O reduced the residual weight to 83.35%. The residual weight was 27.53% in the range of 350-500°C due to the decomposition of APP into polyphosphoric acid and meta phosphoric acid, and the degradation of epoxy and hardener into CO_2 and H_2O. In the range of 500-800°C it was reduced

further to 22.11%. As IF2-5 coating was modified by adding 11.76% boric acid to form IF3-1, the residual weight of char of IF3-1 increased by 5.6 percent. Boric acid reacted with APP to form borophosphate in the residual char which increased the residual weight by 1.19%.

The DTGA curve of IF3-1 char is shown in Figure 4.42 with five steps of thermal degradation at 147, 334, 372, 427 and 470^{o}C, while IF2-5 coating has 4 steps of thermal degradation as shown by the DTGA curve illustrated in Figure 4.17(b). The first degradation curve of IF3-1 at 147^{o}C shows the degradation of cured epoxy resin and dehydration of boric acid t into meta boric acid, while the second degradation at 334^{o}C is declared as the decomposition of melamine. At the temperatures 373^{o}C established the decomposition of melamine derivatives, the degradation of reaction product of APP and melamine such as melamine phosphate and cured epoxy resin occurred at 470^{o}C.

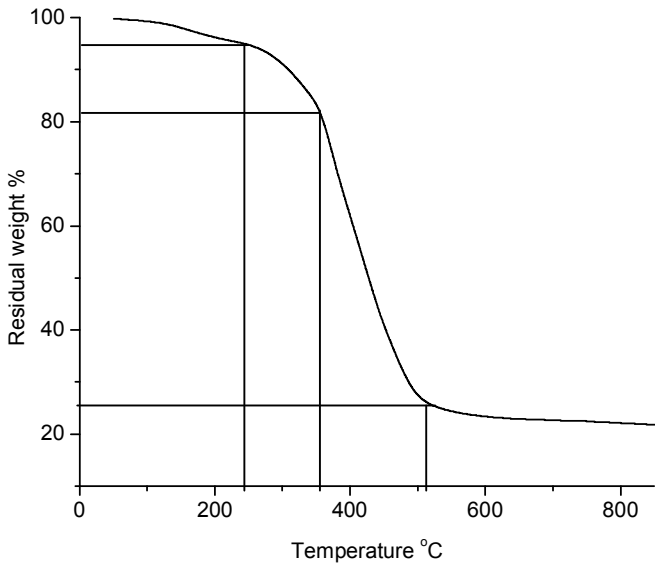


Figure 4.41: TGA of IF3-1 coating showing residual weight 22.11% at 800^{o}C

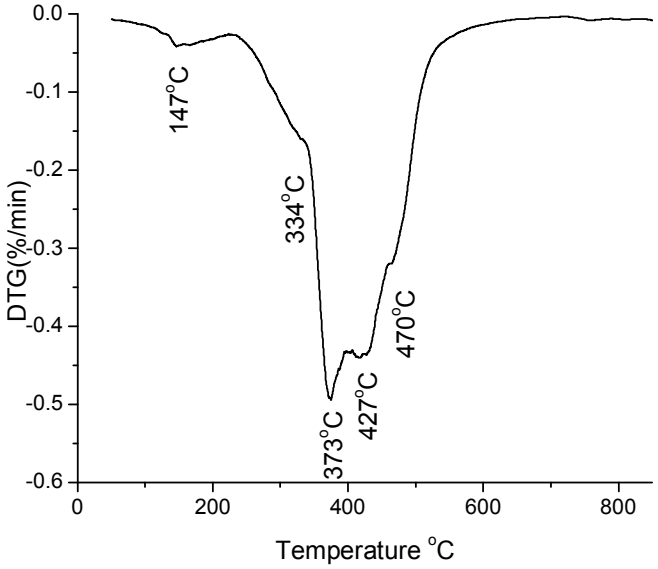


Figure 4.42: DTGA of IF3-1 coating showing four steps degradation

Figure 4.43 shows the TGA curve of IF3-2 coating. In the range of 0-200°C the residual weight was 96.5% due to the release of H_2O from cured epoxy resin and boric acid. In the range of 200-350°C, melamine decomposed into its derivatives with the release of NH_3 gas, this bring down the residual weight to 82.29%. The decomposition of EG and degradation of epoxy and hardener into CO_2 and H_2O in the range of 350-500°C reduced the residual weight to 30.23%. The final residual weight at 800°C was 20.34%. The IF2-1 coating was modified by adding 11.76% boric acid to form IF3-2; residual weight of IF3-2 is increased by 37%. The DTGA curve is illustrated in Figure 4.44 with four steps of thermal degradation at 118°C, 149°C, 308°C, and 496°C. The degradation of cured epoxy resin and conversion of boric acid into meta boric acid occurred at 118°C, while the second degradation at 149°C affirmed the dehydration of meta boric acid into boron oxide. The 3rd degradation at 308°C proved the decomposition of melamine. The fourth curve at 496°C shows the decomposition of BPA epoxy resin, derivative of melamine, and polyamide hardener.

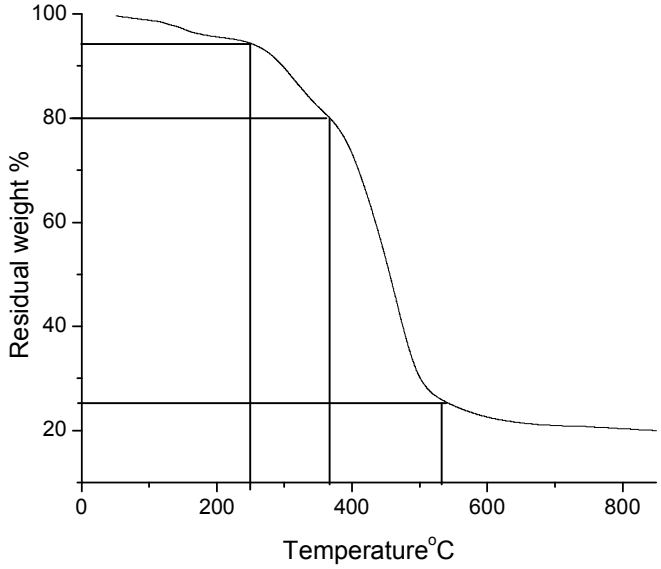


Figure 4.43: TGA of IF3-2 coating showing residual weight 20.32% at 800°C

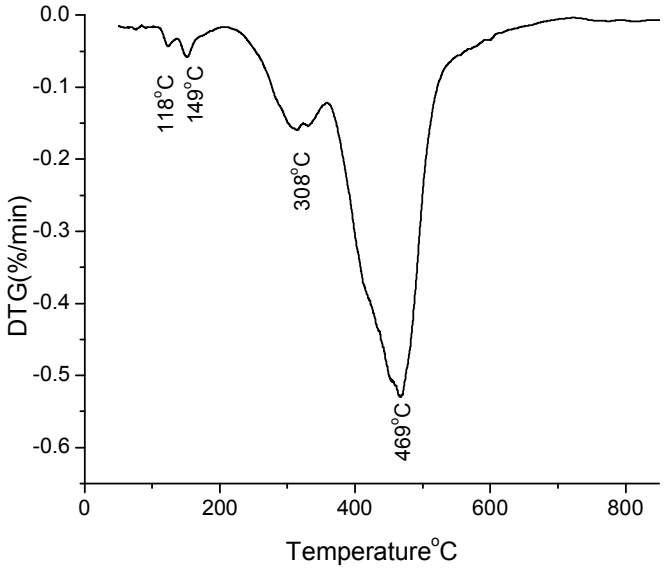


Figure 4.44: DTGA of IF3-2 coating showing four steps degradation

Figure 4.45 shows the TGA curve of IF3-3 coating. In the range of 0-200°C the residual weight was 95.4%. As H_2O was released from cured epoxy resin and boric acid, the residual weight decreased to 89.21% in the range of 200-350°C. The residual

weight was 36.52% after the decomposition of APP, EG and degradation of epoxy and hardener into NH_3, CO_2 and H_2O in the range of 350-500°C. The final residual weight at 800°C was 31%. The IF2-3 coating was modified by adding 11.76% of APP to form IF3-3 which caused an increase of residual weight from 23.5% (IF 2-3) to 30.98% (IF3-2). The DTGA curve is illustrated in Figure 4.46 with three steps of thermal degradation at 126, 413 and 470°C. The first degradation at 126°C shows the dehydration of cured epoxy resin and boric acid. The 2nd degradation at 413°C shows the decomposition of EG and APP. The 3rd curve at 470°C shows the decomposition of BPA epoxy resin, derivative of APP, and polyamide hardener.

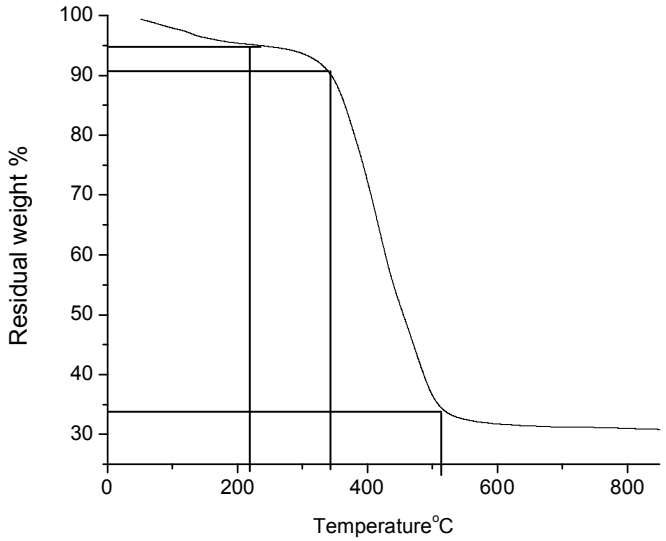


Figure 4.45: TGA of IF3-3 coating showing residual weight 31% at 800°C

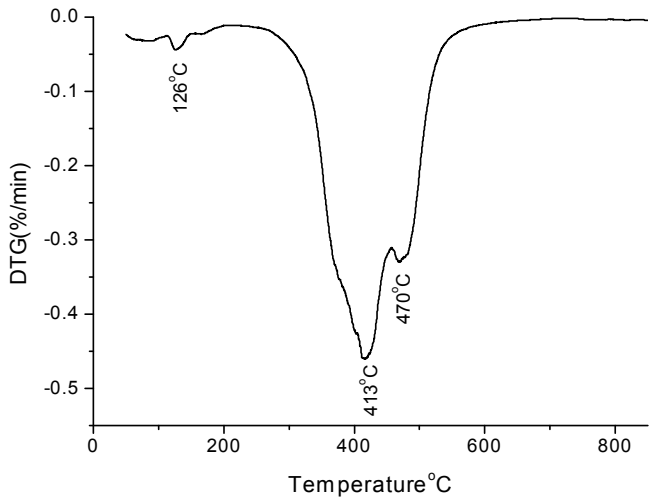


Figure 4.46: TGA of IF3-3 coating showing three steps degradation

Figure 4.47 shows the TGA curve of IF3-4. In the range of 0-200^{o}C, H_2O was released from cured epoxy resin; the residual weight was 98.2%.. The decomposition of melamine occurred in the range of 200-350^{o}C reducing the residual weight to 84.91%. The residual weight was decreased to 30.37% due to the decomposition of APP, EG and degradation of epoxy and hardener into NH_3, CO_2 and H_2O in the range of 350-500^{o}C. The final residual weight at 800 ^{o}C was 24.56% as further weight loss occurred due to decomposition of the derivative of melamine and APP in the range of 500-800^{o}C. The IF2-2 coating was modified by adding 11.76% of APP acid to form IF3-4 coating; the residual weight increased from 13.49% of IF2-2 to 24.5% of IF3-4. It is believed that APP reacted with melamine to form melamine phosphate to increase the residual char. The DTGA curve of IF3-4, illustrated in Figure 4.48, shows three steps of thermal degradation at 110^{o}C, 311^{o}C and 390^{o}C. The first degradation at 110^{o}C is the dehydration of cured epoxy resin. The 2nd degradation at 311^{o}C is the decomposition of melamine and the 3rd curve at 390^{o}C is the decomposition of EG, APP, BPA epoxy resin and polyamide hardener.

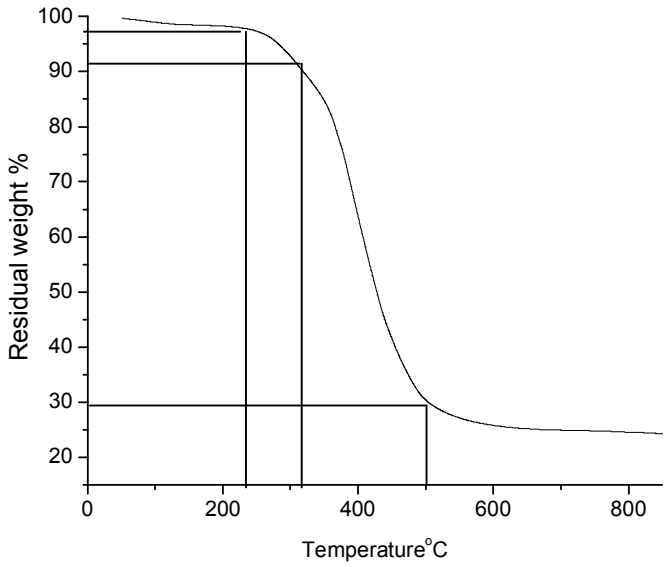


Figure 4.47: TGA of IF3-4 coating showing residual weight 24.54% at 800^{o}C

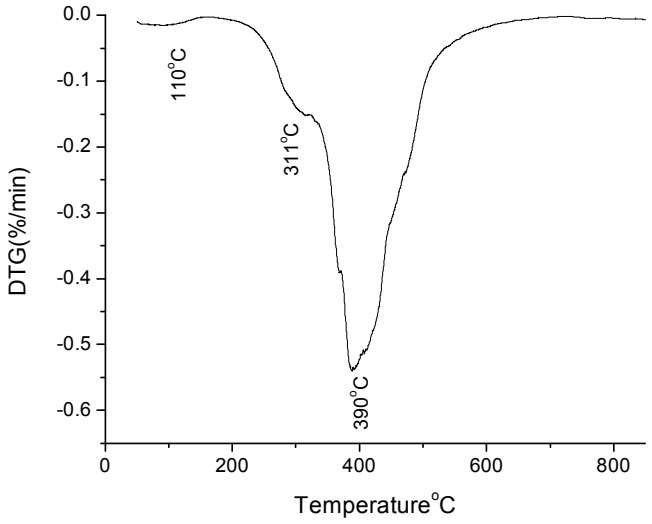


Figure 4.48: DTGA of IF3-4 coating showing three steps degradation

The residual weight of IF3 formulations is compared in Table 4.8. The lowest residual weight was 20.34% of IF3-2 coating. The IF 3-2 coating does not contain APP; APP is an acid source that will strongly form poly phosphoric acid above 200°C. This material does not undergo additional residual weight loss below 600°C, but above this temperature an azotropic P_4O_{11}-H_2O boils and phosphorous lost from the system loss is due to evolving NH_3 [11]. The 2nd lowest residual weight is IF3-1 formulation as it does not contain EG. EG is a carbon source and it contributes to char expansion by the formation of hexagonal network. EG oxidizes above 350°C and evolves CO_2 and H_2O, and the residual weight was 87.48% at 800°C as discussed earlier in Figure 4.12(a). The IF4-3 coating has the 3rd lowest residual weight. IF 4-3 coating does not contain boric acid which forms a glassy char surface and helps stick together the char and substrate. The highest residual weight was obtained from IF3-3 coating due to the presence of APP and boric acid that formed borophosphate and boron oxide to increase the residual weight.

Table 4.8: Residual weight of IF3 at different temperature regions

Formulation No	Residual wt % 0-200°C	Residual wt % 200-350°C	Residual wt % 350-500°C	Residual wt % 500-800°C
IF3-1	96.20	83.35	27.53	22.11
IF3-2	95.60	82.29	30.23	20.34
IF3-3	95.60	89.21	36.52	31.00
IF3-4	98.20	84.91	30.37	24.56

4.4.8 Conclusion

The above results have shown the behaviour of three intumescent fire retardant coating in terms of char expansion, morphology, char composition, functional groups in char residue and residual weight%. The best char expansion 12.43 times was obtained from IF3-4 however, this coating formulation has weak char and voids were present inside the char. IF3-3 shows better results of char morphology, char residue (31%), and its char composition contains graphite, borophosphate and boron oxide. From this investigation, it can be deduced that three ingredients coating gave better results compared to two ingredients, with epoxy and hardener, in the terms of char morphology and residual weight. The following Table 4.9 shows the comparison between two and three ingredients.

Table 4.9: Char expansion, morphology and residual weight of two and three ingredients ICFs

Coating no	Char expansion (times)	Char morphology	Residual weight%
IF2-1	10.15	Cracked surface	14.84
IF2-2	5.10	Cracked surface	13.49
IF2-3	10.5	Flaky char	23.53
IF2-4	3.8	Smooth char	24.97
IF2-5	13.3	--------	20.92
IF2-6	7.35	Charred layer	15.94
IF3-1	2.05	Rough and cracks	22.11
IF3-2	3.5	Smooth surface	20.32
IF3-3	2.57	Charred layered	30.98
IF3-4	12.43	Smooth surface	24.54

4.5 Effect of particle sizes of expandable graphite on char expansion and char morphology of ICFs

The main objective of this study to select a suitable particle size from 63µm, 150µm, 212µm and 300µm of expandable graphite particle size can give enhanced