VOL. 11, NO. 20, OCTOBER 2016 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

www.arpnjournals.com

CHARACTERIZATION AND STUDY OF CHAR PERFORMANCE OF GLASS WOOL AND ROCKWOOL HYBRID FIBRE REINFORCED INTUMESCENT COATINGS

N. Amir, A. N. A. Jalil and F. Ahmad
Mechanical Engineering Department, Universiti Teknologi Petronas, Bandar Seri Iskandar, Perak, Malaysia
E-Mail: norlailiamir@petronas.com.my

ABSTRACT

Intumescent fire retardant coating is widely used in the industry for protecting steel from high temperature and fire incident. The coating's application can be found in the oil and gas industries especially in the offshore structures due to its ability to withstand high temperature and severe weather. However, char strength of the existing intumescent coating can be further improved by fibre reinforcement. Previously, various researches on intumescent coating were conducted using single fibre with limited study on the hybrid fibre reinforced intumescent coating. The project examined the effects of adding hybrid fibres, exploring the mechanical and char characteristics in increasing the fire performance of the coating. The study was focused on adding glass wool fibre (G) and Rockwool fibre (R) as the hybrid reinforcement using two fibre lengths; 6 mm and 12 mm, and at 10% by weight percentage with three combinations of 3:7, 5:5 and 7:3. The investigation involved determination of potential fire resistance of the hybrid fibre reinforced intumescent coatings at various compositions and lengths of the fibres. The properties and performance of the coatings were based on the chars developed by the coatings in the furnace test. The coatings and their chars were characterized using X-Ray Fluorescence (XRF) and Scanning Electron Microscope (SEM). Finally, the chars produced were tested for strength performance test. In summary increased in the Rockwool fibre from 3% to 7% in the hybrid fibres content had produced higher char expansion (by 15.7%), lower weight loss (by 22.8%) as well as better char performance against loading (by 37%).

Keywords: hybrid fibre, intumescent, characterization, fire protection, and steel.

INTRODUCTION

Over the years, intumescent coating technology has contributed to the development of fire protecting system in many ways. Application of intumescent coatings as the fire protection system for building materials has currently, shoot up throughout the world for example in the offshore industry [1] and has also been considered for aircraft structural component [2]. Most construction materials that are currently in use worldwide, such as steel or wood are unable to withstand high temperatures. The materials require protective layer or coating to improve resistance towards high temperature surrounding. When the materials in a high temperature surrounding, intumescent coatings are regularly used to improve the material's fire performance.

Conventional intumescent coating formulation having basic intumescent ingredients usually produced soft char, which is weak and low in strength. The coating's performance is then improved by adding fillers [3-4]. Recently, there are several studies about the effects of using fibre to increase materials fire performance [5-7]. A fibre reinforced intumescent coating generally showed more compact cell structure and offered higher strength property [5]. Intumescent coating having hybrid fibres have been experimented, which provided enhancement to the fire protection of steel substrate [8].

Intumescent coating as passive fire protection of structural materials can maintain its ability to protect the materials during a fire incident only if it possess sufficient strength in its degraded form i.e. char. Thus, mechanical testing is required for the coating as well as its residue after it is burned. Thus, significant contributions of the article are the development of hybrid fibres reinforced intumescent coatings and investigation of the effects of fibre length and composition of glass wool and Rockwool fibres to the fire resistance and mechanical properties of intumescent coating.

MATERIALS AND METHODS

Materials

Eight intumescent formulations; SR10, LR10, SG7R3, SG3R7, SG5R5, LG7R3, LG3R7 and LG5R5 have been prepared and coated on to mild steel substrates. Materials used in these formulations include Bisphenol A epoxy resin, polyamide amine, ammonium polyphosphate (APP), expandable graphite (EG), melamine (MEL), boric acid (BA), glass wool fibre and Rockwool fibre. Two fibre lengths were selected, 6 mm (short fibre, S) and 12 mm (long fibre, L). The complete formulations are available in our previous publication [8], where in the current research, EG was used as carbon source to replace pentaerythritol.

Apparatus and procedure

Preparing the coating. The process started with weighing the materials based on coating formulations. APP, MEL and BA were mixed together with hybrid fibres as required based on the formulations and grinded for one minute. Then, EG was added to the mixture and slowly mixed or stirred until uniformly dispersed. The epoxy was then added into the formulation and stirred using electric mixer with a speed of 40 rpm for 30 minutes. After 30 minutes, the hardener was mixed together to the coating formulation and stirred at the same speed for 40 minutes until homogeneous coating was formed. The coating was evenly applied on the 5cm x 5cm and 10cm x 10cm sandblasted mild steel plates. The coating samples were left to dry at room temperature.

Furnace fire test. The average thickness for dry coating samples before the 400 ℃ test was 7.47 mm, while for the 800 ℃ test was 5.95mm. For this test, the samples were put inside Carbolite electric furnace, model CWF 1300 and burnt to maximum temperatures of 400℃ or 800 ℃. After reaching its maximum temperature, furnace temperature was maintained for one hour duration. Then, furnace temperature was reduced gradually until room temperature before the samples were taken out.

Char strength performance test. Ten loading discs were used to prepare varying loads i.e. 1 N, 2 N, 5N and 10N. The test setup was based on our previous reported research [9]. Loading discs were put on top of char incrementally until the char collapse or no more changes in the char height.

Analytical experiments. Two analytical approaches were used in this research; Scanning Electron Miscroscopy (SEM) using Phenom Pro X for morphological study and X-Ray Fluorescence (XRF) for elemental analysis for both coating and char materials.

RESULTS DISCUSSIONS

Morphology of hybrid fibre reinforced coating

Three coating samples that have fully cured were examined under SEM for the microstructure of the hybrid fibre reinforced intumescent coating. The coatings chosen were from the formulations having long, fibre length of 12 mm i.e. LR10 (single Rockwool fibre), LG3R7 and LR5G5. All samples were analyzed under the magnification of 1000x and 3000x for the cross sectional area and top view, respectively.

Figure-1 shows the cross sectional area of the sample LR10. It can be seen that Rockwool fibres were homogeneously well dispersed with the other coating materials. Almost all fibres are horizontally positioned and facing one direction. There are many micro voids present in the coating; the voids help to trap air and oxygen and prevent them from transferring to the other parts of the coating to promote fire propagation. This make intumescent coating became insulating layer and restricts fire from reaching the protected materials.

Figure-2 and Figure-3 show the cross section area views for LG3R7 and LG5R5 coatings, which contain hybrid fibres; Rockwool fibre and glass wool fibre; the difference lies on the composition of the fibres used in the coating formulations. In both micrographs, hybrid fibres were randomly dispersed, also laid horizontally due to gravity and facing single direction. The larger fibres in the figures with diameter around 16-18 μm were the glass wool, while the smaller fibres were the Rockwool, having diameter of 6 - 8 μm. Presence of micro voids surrounded by the fibres can be seen on both of the figures, indicating potential of fire protection characteristics to the coatings.

Hybrid fibre reinforced Char's expansion

There are changes in thickness of the coating after furnace test, due to the coating swelling reaction against fire in producing the char. Table-1 and Table-2 show coating expansion after 400 ℃ and 800 ℃ furnace test, respectively.

In this study only short fibre reinforced intumescent coatings (either single or hybrid fibres) were considered. Based on Figure-4 the 400℃ sample, SG5R5 had the highest char expansion with 585% and followed by samples SG3R7, SG7R3 and SR10 with the expansions of 502%, 374% and 359%, respectively. Addition of glass fibre and Rockwool fibre at equal weight percentage to form hybrid fibre reinforcement has greatly improved char expansion to maximum of 226%.

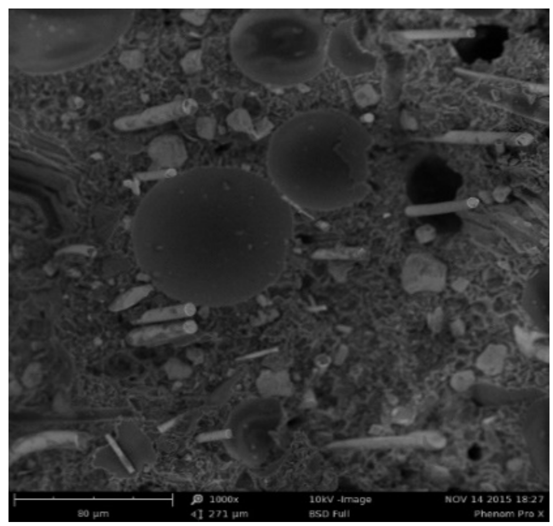

Figure-1. Cross section area, intumescent coating with only long Rockwool fibre at 10wt% content.

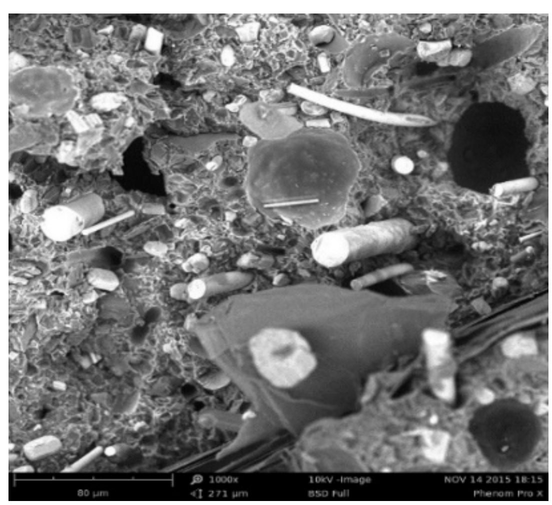

Figure-2. Cross section area, LG3R7hybrid fibre-coating.

ARPN Journal of Engineering and Applied Sciences

www.arpnjournals.com

Figure-3. Cross section area, LG5R5 hybrid fibre-coating.

Table-1. Intumescent coating expansion at 400 ^{o}C.

Samples	Thickness(mm)		Expansion (%)
	Before	After	
SR10– Formulation 10% Rockwool of 6mm length	5.75	20.66	359.30
SG3R7– Formulation 3% Glass wool 7% Rockwool of 6mm length	10.51	52.79	502.28
SG7R3 – Formulation 7% Glass wool 3% Rockwool 6mm length	7.21	26.94	373.64
SG5R5 – Formulation 5% Glass wool 5% Rockwool of 6mm length	6.42	37.61	585.82

Table-2. Intumescent coating expansion at 800 ℃.

Samples	Thickness(mm)		Expansion (%)
	Before	After	
SR10– Formulation 10% Rockwool of 6mm length	5.80	20.56	354.41
SG3R7– Formulation 3% Glass wool 7% Rockwool of 6mm length	7.39	23.95	324.08
SG7R3 – Formulation 7% Glass wool 3% Rockwool 6mm length	9.19	34.46	374.97
SG5R5 – Formulation 5% Glass wool 5% Rockwool of 6mm length	6.77	24.26	358.35

For the samples under 800 ^{o}C furnace test, the highest char expansion belonged to the sample SG3R7 and followed by SG7R3 with 15.7% difference. This showed that the highest char expansion was among the samples with hybrid fibre reinforcement.

Char amount or coating weight loss or residue weight

In furnace test, intumescent coating samples expanded and formed chars. At the same time the coating experienced loss in weight due to the burning of coating materials as well as degradation due to high temperature. In general, lower temperature exposure resulted in smaller weight loss to the coatings as evidence in Figure-5.

Sample SG7R3 demonstrated the most weight losses up to 22.4% (recorded weight loss was 10.33 g) and 28.0% (14.56 g) under 400 ℃ and 800 °C furnace test, respectively. This was followed by SG3R7 with weight losses of 17.1% (8.74 g) and 22.8% (10.6 g) under 400 ℃ and 800 °C furnace test, respectively. These indirectly implied that with increasing content of Rockwool fibre and at decreasing amount of glass wool fibre in the coating, the loss of materials' weight at high temperature can be reduced. Therefore, when the hybrid fibres was

VOL. 11, NO. 20, OCTOBER 2016 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

www.arpnjournals.com

replaced with 100% Rockwool fibre only in SR10, the weight losses decreased to 3.9% (1.52 g) and 19.0% (7.48 g) under 400 °C and 800 °C furnace test, respectively.

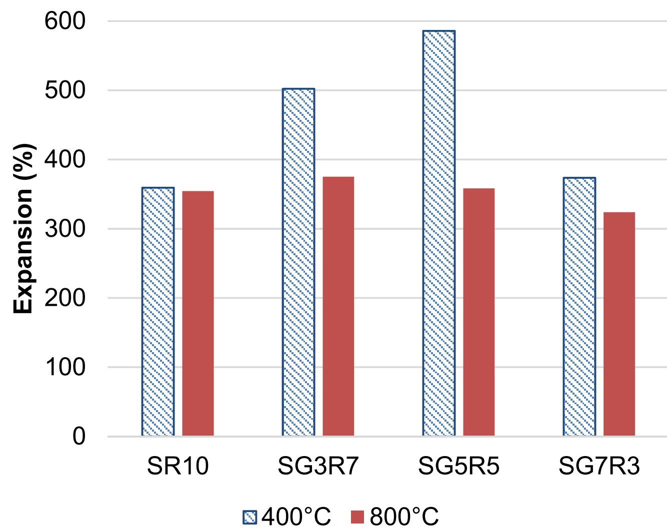

Figure-4. Char expansion percentage after 400 °C and 800 °C furnace test.

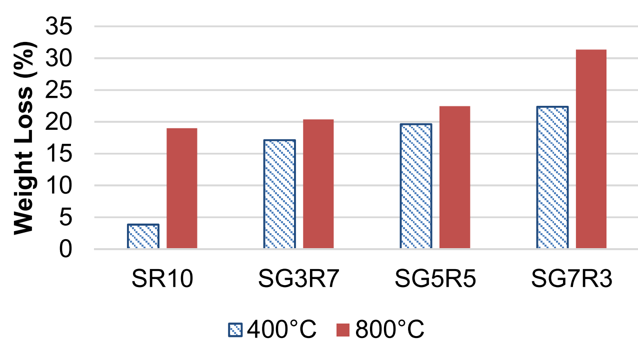

Figure-5. Weight loss after 400 °C and 800 °C furnace test.

Hybrid fibre reinforced intumescent coating's char strength performance

The char strength performance test was conducted to assess the strength of the char in sustaining external load, which is critical in protecting the steel structure against fire forces.

Figure-6 and Figure-7 plot the char thickness against load sustained for samples from 800°C and 400°C furnace test, respectively. Based on these two graphs, the 800 °C chars demonstrated lower strength when compared to those from 400 °C fire test. This can be attributed to more materials loss or severe degradation experienced by the former at higher temperature. Thus, char samples from the 800°Cfire test were loaded up to 10N or mass of 1000g for the strength performance test, while the chars from the 400 °C furnace test were tested until 40N.

Comparing the char strength performance between intumescent coating having either long or short hybrid fibres after 800 °C furnace test, it can be seen that the thickness of char with short hybrid fibres decreased lesser than the chars with using the long hybrid fibre. As an example, SG3R7 experienced thickness reduction at 37.4% only as compared to LG3R7 at 55.6%. This tell that higher aspect ratio of the fibre gave better char strength performance.

On another note, regardless of the fibre length, increase in Rockwool fibre percentage in the hybrid coating formulation had increase the char strength performance as shown by lower change in the char thickness. That is why for intumescent coatings having 7% glass wool fibre and only 3% Rockwool fibre resulted in lower performance i.e. SG7R3 at 74.4% in char thickness reduction after continuous loading with mass up to 1000 g. This implies that Rockwool fibre is more resistant to fire i.e. lower degradation at high temperature, than glass wool fibre. Similar trend was also true for the chars from the lower temperature furnace test as in Figure 7. The flatter, the plot is, the better is the char's strength performance.

 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

It can be deduced that an intact char layer will trap air between its hybrid fibre strengthened honeycomb structures, thus reduce heat transfer from the coating side that is exposed to heat source to the protected material.

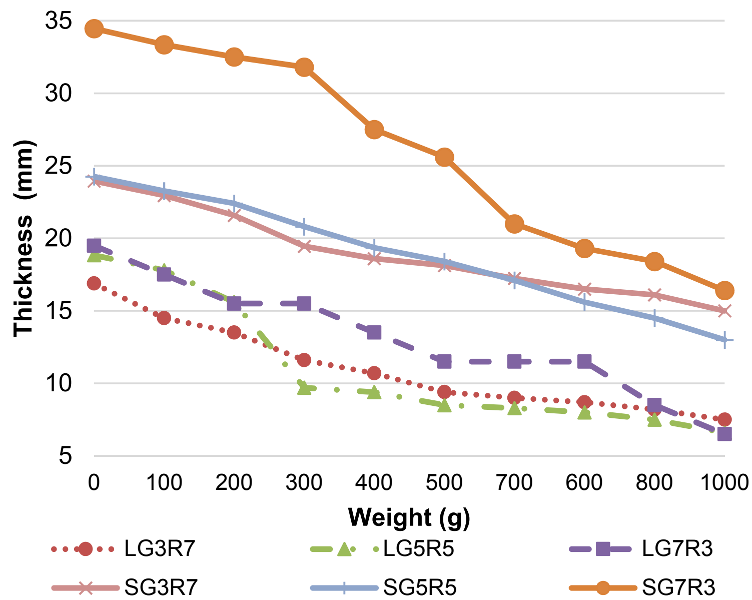

Figure-6. Char thickness reductions against increasing loads up to 10 N for various fibre reinforced intumescent coatings' char from 800 °C furnace test.

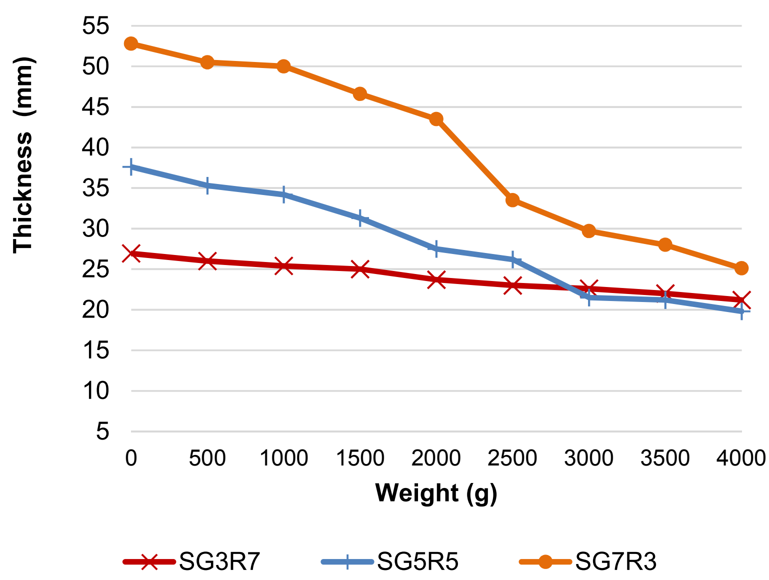

Figure-7. Char thickness reductions against increasing loads up to 40 N for short fibre reinforced intumescent coatings'char from 400 °C furnace test.

Hybrid fibre reinforced intumescent coating's charmorphology and elemental composition

Figures 8 and 9 shows the SEM micrograph of LG3R7 and LG5R5 chars, respectively. Fibrous, honeycomb-like and worm-like, multicellular layers are typical structure for expandable graphite [10]. The formulation with higher Rockwool fibre than glass wool fibre (LG3R7) produced a denser char layers with many tiny voids, while the formulation having equal fibre loading between the hybrid fibres (LG5R5) showed char networks with bigger voids indicating a softer structure.

ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

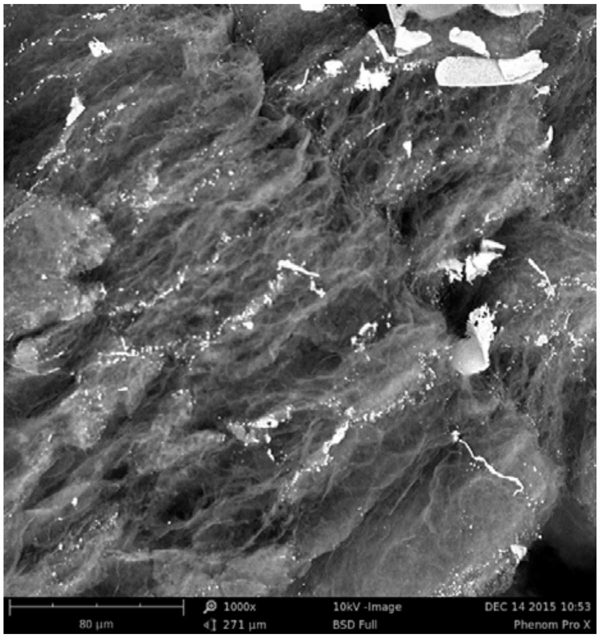

Figure-8. SEM image for LG3R7, hybrid fibre reinforced intumescent coatings' char from 800 ℃ furnace test.

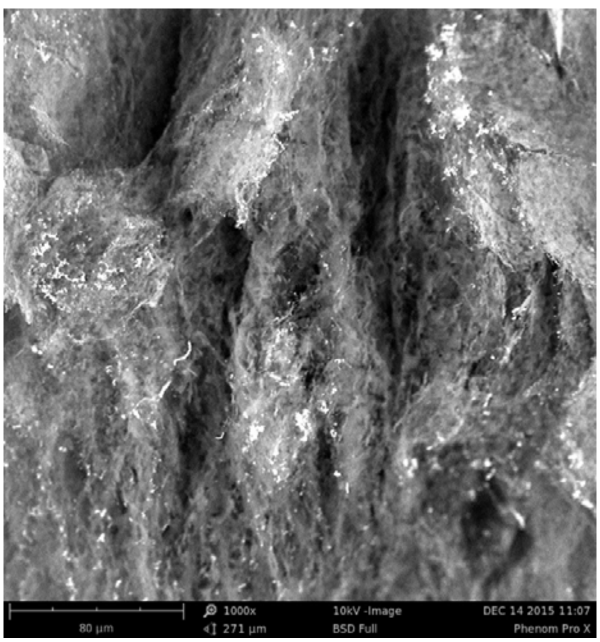

Figure-9. SEM image for LG5R5, hybrid fibre reinforced intumescent coatings' char from 800 ℃ furnace test.

Two hybrid fibre reinforced intumescent coatings were chosen for XRF analysis; LG3R7 and LG5R5, which are the competing coatings as discussed in the earlier experiments. For each formulation, the coatings and their char samples from 800 °C furnace test were analyzed for elemental information and compared as in Table-3.

As expected both coating and char samples contains high Phosphorus (P) element, followed by Calcium (Ca) and Silicon (Si). There were slight changes in the composition percentage of these elements before and after furnace test. For the top performing, LG3R7 formulation the composition percentage of P and Ca increased after the burning and possibly attributed to presence of fire retardant hybrid fibres at optimum percentage to preserve these elements disintegrating from the coating layer in the fire test.

Table-3. XRF analysis for LG3R7 and LG5R5 samples.

Components	Composition (%)			
	LG3R7		LG5R5	
	Coating	Char	Coating	Char
P	27	32	29.6	31.5
Ca	25.2	28.4	27	25.5
Si	8.2	13.2	7.09	14.6
Fe	12	15.1	15.7	12.8
K	2.45	3.12	2.52	2.30
Al	1.41	2.32	1.39	1.91
Ti	1.19	1.26	1.36	0.853
Cr	0.953	1.31	0.863	-
Mg	0.695	0.639	0.553	0.594
Mn	0.361	0.433	0.438	0.158
Ni	0.207	0.169	0.233	0.155
Zn	2.79	0.120	0.187	0.152
As	0.139	0.0676	0.0969	0.123
Sr	0.136	0.125	0.137	0.102
Cu	0.124	0.0569	0.156	0.0600
Rb	0.0586	0.0438	0.0788	0.0402
S	12	0.599	7.71	0.368
Cl	4.71	0.398	4.79	-
Na	-	0.478	-	0.508
V	-	0.0122	-	0.0453
Ru	0.140	-	-	-
Ba	-	0.149	-	-
Zr	0.0893	0.478	-	-
Mo	0.0563	-	0.0857	-

CONCLUSIONS

In this research, the effects of hybrid fibre reinforcement mainly by glass wool fibre and Rockwool into intumescent coating for potential uses as passive fire protection of mild steel were analyzed and discussed. Firstly, in this research the morphology examination showed that short and long hybrid fibres have been successfully dispersed randomly in the coating with proper mixing. On the common characteristics of intumescent coating such as char expansion and weight loss as well as char strength performance, a linear relationship with the content of short hybrid fibres was observed. Increase in the Rockwool fibre from 3% to 7% in the hybrid fibres content had produced higher char expansion (by 15.7%), lower weight loss (by 22.8%) as well as better char performance against loading (by 37%). The char layers with micro-voids trap air between the hybrid fibre strengthened honeycomb structures, thus reduce heat transfer from the coating side (that is exposed to heat source) to the protected material. Further elemental analysis the coatings and their chars also showed greater fire resistant elements were retained in the SG3R7.

ACKNOWLEDGEMENT

The authors were grateful for the funding received from the Ministry of Higher Education (MOHE) Malaysiaunder the Fundamental Research Grant Scheme (FRGS).

REFERENCES

[1] T. A. Roberts, L. C. Shirvill, K. Waterton, and I. Buckland 2010. Fire Resistance of Passive Fire Protection Coatings after Long-Term Weathering. Process Safety and Environmental Protection. 88: 1-19.

[2] H. T. Akshatha, A. Rinku and R. Gopalakrishnan 2014. Design, Development and Validation of Fire Protection Scheme for the Engine Firewalls and Cowling of a Typical Light Transport Aircraft. ARPN Journal of Engineering and Applied Sciences. 9(4): 415-418.

[3] J.-w. Gu, G.-c. Zhang, S.-l. Dong, Q.-y. Zhang, J. Kong 2007. Study on preparation and fire-retardant mechanism analysis of intumescent flame-retardant coatings. Surface and Coatings Technology. 201(2007): 7835-7841.

[4] M. C. Yew, N. H. RamliSulong, M. K. Yew, M. A. Amalina and M. R. Johan 2015. Influences of flame-retardant fillers on fire protection and mechanical properties of intumescent coatings. Progress in Organic Coatings. 78: 59-66.

[5] N. Amir, F. Ahmad and P. S. M. Megat-Yusoff 2012. Mechanisms of Char Strengthening in the Fibre Reinforced Intumescent Coatings. Journal of Applied Sciences. 12(23): 2459-2463.

[6] N. Amir, F. Ahmad and P. S. M. Megat-Yusoff 2011. Study on the Fibre Reinforced Epoxy-based Intumescent Coating Formulations and their Char Characteristics. Journal of Applied Sciences. 11(10): 1678-1687.

[7] N. Amir, F. Ahmad and P.S.M. Megat-Yusoff 2011. Characterization of Inorganic Non-Metallic Fibre Reinforced Epoxy-Based Intumescent Coatings (FRIC) and Chartex 7. Engineering e-Transaction. 2(6): 143-151.

[8] N. Amir, M. H. Roslan and F. Ahmad 2015. Fire Resistance of Glass Wool and Rockwool Hybrid Fibre Reinforced Intumescent Coating. Journal of Advanced Research in Materials Science. 1(12): 24-32.

[9] N. Amir, F. Ahmad and P. S. M. Megat-Yusoff 2013. Char Strength of Wool Fibre Reinforced Epoxy-Based Intumescent Coatings (FRIC). Advanced Materials Research 626(2013): 504-508.

[10] B.Gardelle, S. Duquesne, P. Vandereecken and S. Bourbigot 2013. Characterization of the Carbonization Process of Expandable Graphite/Silicone Formulations in a Simulated Fire. Polymer Degradation and Stability. 98(5): 1052-1063.