CHAPTER 5

CONCLUSION

5.1 Chapter Overview

This chapter summarizes the findings of this research by drawing the conclusion of overall results and recommendation for future works. The objectives on this research are reviewed and the achievement is drawn in this chapter.

5.2 Conclusion

This research was carried out with the main objective to develop a new intumescent fire retardant coating by using EG as the carbon source. The research has been approached by first investigating the effect of one, two and three ingredients on char expansion and char morphology. The research has been approached further by optimizing the particle size of EG based on char expansion and char morphology. Then the formulation with the optimized EG particle size was used to develop intumescent formulations using selected intumescent additives. The optimized intumescent formulation with higher char expansion, char morphology, minimum substrate temperature and high residual char weight percentage was obtained from IF5-BA-Mel. The optimized formulation was reinforced with three inorganic fillers (MWCNTs, kaolin clay, zirconium silicate) by using different weight percentage to study its chemical, physical, thermal and weathering properties. From the results, it can be seen that zirconium silicate has significant effect in intumescent coating. The following conclusions are drawn from the study:

1. The combination of two intumescent ingredients produces better results in term of char expansion performance and morphology compared to one intumescent ingredient with epoxy binder. Three formulations were optimized based on their best property namely IF2-5 for char expansion, IF2-3 for morphology and IF2-4 for residual weight. In case of char expansion IF2-5 shows the char expansion of 13.3 times, which is good compared to IF2-1, IF2-2, IF2-3 IF2-4 and IF2-6. In the case of char morphology, IF2-3 shows the better char structure compared to other formulations in the study of char morphology of two intumescent ingredients coatings. The residual weight of IF2-4 is 24.9% which is the best result for two ingredients intumescent coating.

2. Coating with three ingredients gave better results compared to two ingredients with epoxy and hardener in terms of char morphology and residual weight. The best char expansion of 12.43 times was obtained from IF3-4. IF3-3 shows better results of char morphology, char residue, and its char composition consists of graphite, borophosphate and boron oxide.

3. Three conclusions are drawn on the effect of particle size of EG on char expansion and char morphology. Increasing the particle size of EG increases the char expansion. Increasing the weight percent of EG in the intumescent coating formulation also increases char expansion. A formulation with 8.8wt% of EG in the intumescent coating formulations resulted in maximum char expansion for IF4-4-150µm is 9.38, and for IF4-4-212µm is 11.0 times. However, with 8.8 wt% of EG, the formulation with EG particle size of 300µm shows the best results in char expansion, 13.4 times from the original thickness of the coating. SEM images of char of the formulations 63, 150, 212 and 300µm EG particles show char morphologies containing cavities and cracks in the char structure. While in some structures, smooth surface without any cracks is observed, instead in these structures thick char flakes which form strong barrier between fire and substrate are observed. The 300µm EG char with flaky char structure has with the highest char expansion which provides thermal stability to the coatings. It is assumed that excess amount of EG,

above 10.8 wt%, causes the char to expand abruptly (causing raptures) with uniform network structure by affecting the top surface of the char.

4. Among the formulations with EG 300μm, the best optimized formulation is IF5-BA-Mel. This formulation has the minimum substrate temperature of 367^{o}C. The cellular char structure is good compact compared to others formulations studied for intumescent behaviour of individual ingredients. The XRD and FTIR show the composition of char residue, which contains graphite, borophosphate, sassolite and boron oxide which are effective for reducing heat penetration to the underlying substrate. The char residual weight of is 28.58% which is helpful to reduce the heat flow to the substrate.
5. Reinforcement of 0.4wt% of MWCNTs in IF5-BA-Mel formulation increased char expansion which is 50 percent better compared to IF5-BA-Mel. The substrate temperature of IF3-MWCNTs after 60 min was 273^{o}C which is 90^{o}C less than IF5-BA-Mel formulation. FESEM result shows that the structure of residual char is improved by increasing the wt% of MWCNTs, the char cracks are joined by MWCNTs. XRD and FTIR show the presence of boron oxide and borophosphate in the residual char. Thermal analysis shows that 0.4wt% MWCNTs enhanced the residual weight by 3 percentage higher than that of IF5-BA-Mel coating. MWCNTs is an additive that will create uniform protective layers on the upper and inner surface of char to insulate the underlying materials.
6. For kaolin clay reinforced formulations, char expansion increases with each increase in weight % of kaolin clay. IF5-KC gave char expansion of 15.42 times from its original coating thickness, which is 208 percent better than IF5-BA-Mel formulation. The substrate temperature of IF5-KC after 60 min was 257^{o}C, which is 110^{o}C temperature less than IF5-BA-Mel formulation. SEM results show that the structure of char residue is improved in terms of cracks and holes by increasing the wt% of Kaolin clay. XRD and FTIR show the presence of boron oxide, borophosphate, sassolite and kaolinite compounds in the residual char which are helpful to lessen the effect of fire to the substrate. TGA shows that 5wt% of kaolin clay enhanced the residual weight by 49 percent higher than that of IF5-BA-Mel formulation. DTA analysis shows that

the melting point of decomposed fire retardant additives increased by using 5wt% of kaolin clay. IF5-KC gave higher carbon content in the residual char compared to IF4-KC using XPS analysis.

7. For zirconium silicate reinforced formulations, expansion of the char was increased by increasing the weight % of zirconium silicate. IF5-ZS gave better char expansion 24.05 times with respect to the original coating thickness and is 390%, 226%, and 90% higher compared to IF5-BA-Mel, IF3-MWCNTs and IF5-KC formulations (with and without inorganic fillers), respectively. The substrate temperature of the IF5-ZS coating was 213^{o}C after 60 minute fire test using bunsen burner and this is 154^{o}C, 60^{o}, 44^{o}C less than IF5-BA-Mel, IF3-MWCNTs and IF5-KC formulations, respectively. FESEM results show the structure of residual char improves by zirconium silicate. XRD and FTIR show the presence of boron oxide, borophosphate, and zirconium silicate in the residual char. Thermal analysis shows that zirconium silicate enhanced the residual weight 33% higher than that of IF5-BA-Mel formulation. XPS results also show the composition of the char content; IF5-ZS have the high carbon (66%) content and lowest oxygen (20%) content in the residual char investigated. These compositions are favorable to char accumulation and anti-oxidation of char layer. The gaseous products of IF5-ZS are very much less compared to IF5-BA-Mel.

8. Weathering test shows that the coating colour was changed from slightly black to yellowish due to ultraviolet rays but the char structure is retained after the weathering test. Thus, the heat transfer rate is reduced using MWCNTs, kaolin clay and zirconium silicate giving a better intumescent effect which improves fire retardant performance. The weathering test show that IF5-ZS has better results in char expansion and char morphology. Expansion of the IF5-ZS char was decreased 1.79 times after the weathering test. The char expansion of IF5-ZS is 22.26 times with respect to the original coating thickness after the weathering test. SEM results show that the structure of residual char remains the same as the char structure before weathering test. Zirconium silicate forms a ceramic like protective barrier on the surface of the char to insulate the

substrate. Thus, the efficiency of heat transfer is reduced and intumescent effects are enhanced.

Based on the overall results, it can be concluded that zirconium silicate enhance the performance of intumescent coating due to its tetragonal layered structure which can give cellular char structure ceramic like protective layer in order to protect the underlying substrate. IF5-ZS give the best performance of intumescent behaviour in overall tests including char expansion, char structure, residual weight, heat shielding, gaseous products and weathering test.

From the overall results, it can be conclude that the objective of this research was achieved. This research is new in Malaysia which can be helpful to safety standards in Malaysia. Recommendation for future work was also proposed in this chapter.

5.3 Recommendations

It is recommended to carry out further analysis in the current work. These analysis are:

1. The measurement of compactness of the char has significant effect on flow of heat to substrate. Rheometer is suggested to be used to measure the strength/compactness of the char.
2. The synergistic effect of inorganic fillers on ICF can be further studied.

REFERENCES

[1] R. G. Gann and J. W. Gilman, "Flame Retardants: An Overview," in *Kirk-Othmer Encyclopedia of Chemical Technology*: John Wiley & Sons, Inc., 2000.

[2] G. K. Castle, "FIRE PROTECTION OF STRUCTURAL STEEL," *Loss Prev,* vol. 8, pp. 57-64, 1974.

[3] U. S. C, "Intumescent coatings in the service of fire protection," *Paintindia,* vol. 50, pp. 45-52, 2000.

[4] S. Duquesne*, et al.*, "Intumescent paints: fire protective coatings for metallic substrates," *Surface and Coatings Technology,* vol. 180-181, pp. 302-307, 2004.

[5] S. Posner, "In Survey and technical assessment of alternatives to decabromodiphenyl ether (decaBDE) in textile applications," Sundbyberg 2004.

[6] *The Economic Cost of Fire: Estimates for 2004April* Available: www.communities.gov.uk/documents/fire/pdf/144524.pdf

[7] S. Bourbigot and S. Duquesne, "Fire retardant polymers: recent developments and opportunities," *Journal of Materials Chemistry,* vol. 17, p. 2283, 2007.

[8] Available: http://www.bomba.gov.my/index.php.statistik-kebakaran-mengikutpunca retrieved on 17th January 2011.

[9] h. L. Vandersall, "Intumescent coating system, their development and chemistry," *Journal of Fire and Flammability,* vol. 2, p. 97, April 1971.

[10] G. Camino*, et al.*, "Intumescent fire-retardant systems," *Polymer Degradation and Stability,* vol. 23, pp. 359-376, 1989.

[11] M. Jimenez*, et al.*, "Intumescent fire protective coating: Toward a better understanding of their mechanism of action," *Thermochimica Acta,* vol. 449, pp. 16-26, 2006.

[12] M. Jimenez*, et al.*, "Characterization of the performance of an intumescent fire protective coating," *Surface and Coatings Technology,* vol. 201, pp. 979-987, 2006.

[13] D. G. Weldon, "Failure Analysis of Paints and Coatings," John Wiley & Sons, 2002.

[14] D. Drysdale, *An Introduction to Fire Dynamics, second Edition*: John Wiley & Sons 1999.

[15] J. G. Quintiere, *Principles of Fire Behavior*: Thomson Delmar Learning, 1998.

[16] P. J. Briggs, "Combustibility of plastics Frank L. Fire Van Nostrand Reinhold, New York, 1991. pp. ix+326, price £36.50. ISBN 0-442-23801-0," *Polymer International,* vol. 30, pp. 551-551, 1993.

[17] F. J. W. J. Labelschange, "Metal catalysed intumescent of polyhydroxyl compounds," Chemical Engineering, University of Pretoria-Labuschange, 2003.

[18] "Looking at synergy in flame-retardants," *Additives for Polymers,* vol. 1997, pp. 10-11, 1997.

[19] J. Green, "Mechanisms for flame retardancy and smoke suppression - A review," *Journal of Fire Sciences,* vol. 14, pp. 426-442, 1996.

[20] E. M. Pearce, "Flame retardants for polymer systems,," *Pure and Applied Chemistry,* vol. 58, pp. 925-930, 1986.

[21] J. Green, "A Review of Phosphorus- Containing Flame Retardants," *j fire sci,* vol. 14, pp. 353-366, 1996.

[22] J. Green, "A Phosphorus-Bromine Flame Retardant for Engineering Thermoplastics—A Review," *Journal of Fire Sciences,* vol. 12, pp. 388-408, July 1, 1994 1994.

[23] B. Serge and D. Sophie, "Intumescence-Based Fire Retardants," in *Fire Retardancy of Polymeric Materials, Second Edition*: CRC Press, 2009, pp. 129-162.

[24] J. Green, "25 Years of Flame Retarding Plastics," *Journal of Fire Sciences,* vol. 15, pp. 52-67, January 1, 1997 1997.

[25] Miller B., "Intumescents, FR Efficiency Pace Flame Retardant Gains," presented at the Plastics World, Dec 1996.

[26] R. a. L. K. Wolf, B.,, *Plastics, Additives in Ullman's Encyclopaedia of Industrial Chemistry*, 5th ed. vol. A20: VCH Verslag, Weinheim, 1992.

[27] G. L. Nelson, Wilkie, , "Fire Retardancy in 2001," presented at the Fire & Polymers, Materials and Solutions for Hazard Prevention ACS Symposium Series 797 2001.

[28] J. W. Gilman, "Flammability and thermal stability studies of polymer layered-silicate (clay) nanocomposites 1 This work was carried out by the National Institute of Standards and Technology (NIST), an agency of the U. S. government, and by statute is not subject to copyright in the United States. 1," *Applied Clay Science,* vol. 15, pp. 31-49, 1999.

[29] B. J, "Fire retardance in polymers: An introductory lecture," *Polymer Degradation and Stability,* vol. 23, pp. 313-325, 1989.

[30] L. Sergei V., "Introduction to Flame Retradancy and Polymer Flammability " in *Flame Retardant Polymer Nanocomposites*, A. B. M. a. C. A. Wilkie, Ed., New York: Copyright 2007 John Wiley & Sons, Inc., pp. 1-30.

[31] "<Flame retardant polymers 1986.PDF>."

[32] R. N. Rothon and P. R. Hornsby, "Flame retardant effects of magnesium hydroxide," *Polymer Degradation and Stability,* vol. 54, pp. 383-385, 1996.

[33] J. W. K. Gilman, T.; Harris, R. H., Jr.; Lomakin, S. M.; Lichtenhan, J. D.; Bolf, A.; Jones, P. , "Char Enhancing Approaches to Flame Retarding Polymers.," in *Chemistry and Technology of Polymer Additives*, M. Malden, Ak-Malaika, S.; Golovoy, A.; Wilkie, C. A.,, Ed.: Blackwell Science Inc, 1999, pp. p135-150.

[34] L. Delfosse, Baillet, C., Brault, A. and Brault, D. , "Combustion of ethylene-vinyl acetate copolymer filled with aluminium and magnesium hydroxides," *Polymer Degradation and Stability,* vol. 23, pp. p. 337-347, 1989.

[35] C. Baillet and L. Delfosse, "The combustion of polyolefins filled with metallic hydroxides and antimony trioxide," *Polymer Degradation and Stability,* vol. 30, pp. 89-99, 1990.

[36] I. Touval, *Antimony and other inorganic flame retardants in Kirk Othmer's Encyclopaedia of Chemical Technology*, 4th ed.: John Wiley and Sons, New York, 1993.

[37] C. Nyambo*, et al.*, "Thermal stability and flammability characteristics of ethylene vinyl acetate (EVA) composites blended with a phenyl phosphonate-intercalated layered double hydroxide (LDH), melamine polyphosphate and/or boric acid," *Polymer Degradation and Stability,* vol. 94, pp. 513-520, 2009.

[38] "Molybdenum and zinc flame retardants," *Plastics, Additives and Compounding,* vol. 5, p. 24.

[39] P. Georlette, Simons, J. and Costa, L. , "Halogen-Containing Fire-Retardant Compounds, in Fire Retardancy of Polymeric Materials,," C. A. Wilkie, Grand, A.F., Ed.: Marcel Dekker Inc., New York., 2000, pp. p. 245-284.

[40] A. G. Mack, "Flame Retardants, Halogenated," in *Kirk-Othmer Encyclopedia of Chemical Technology*: John Wiley & Sons, Inc., 2000.

[41] J. Green, " Phosphorous-Containing Flame Retardants, in Fire Retardancy of Polymeric Materials.," C. A. Wilkie, Grand, A.F, Ed., New York: Marcel Dekker Inc., 2000, pp. p. 147-170.

[42] J. F. Rakszawski and W. E. Parker, "The effect of group IIIA–VIA elements and their oxides on graphite oxidation," *Carbon,* vol. 2, pp. 53-63, 1964.

[43] E. D. Weil, "Flame Retardants, Phosphorus," in *Kirk-Othmer Encyclopedia of Chemical Technology*: John Wiley & Sons, Inc., 2000.

[44] E. D. Weil*, et al.*, "A systems approach to flame retardancy and comments on modes of action," *Polymer Degradation and Stability,* vol. 54, pp. 125-136.

[45] G. Camino*, et al.*, "Overview of fire retardant mechanisms," *Polymer Degradation and Stability,* vol. 33, pp. 131-154, 1991.

[46] S. V. Levchik and E. D. Weil, "A Review of Recent Progress in Phosphorus-based Flame Retardants," *Journal of Fire Sciences,* vol. 24, pp. 345-364, September 1, 2006 2006.

[47] P. Joseph, Ebdon, J.R., , Horrocks, A.R. and Price, D., " Recent developments in flame-retarding thermoplastics and thermosets, in Fire Retardant Materials," A. R. a. P. Horrocks, D.,(Eds.), Ed., Cambridge, U.K: Woohdhead Publishing Limited, 2000, pp. pp. 220-263.

[48] P. Kannan and K. Kishore, "Novel flame retardant polyphosphoramide esters," *Polymer,* vol. 33, pp. 418-422, 1992.

[49] T. J.H, "Methods for the fire protection of plastics and coatings by flame retardant and intumescent systems," *Progress in Organic Coatings,* vol. 11, pp. 41-69, 1983.

[50] A. Fina*, et al.*, "Thermal Behavior of Nanocomposites and Fire Testing Performance," in *Fire and Polymers V.* vol. 1013: American Chemical Society, 2009, pp. 10-24.

[51] A. R. Horrocks, "Flame-retardant Finishing of Textiles," *Review of Progress in Coloration and Related Topics,* vol. 16, pp. 62-101, 1986.

[52] P. Joseph, Ebdon, J.R., , "Phosphorus-Based Flame Retardants," in *Fire Retardancy of Polymeric Materials*, C. A. Wilkie, Morgan A.B., Ed.: Taylor & Francis Group, 2010, pp. pp.107-127.

[53] C. Caze, Devaux, E., Testard, G., and Reix, T., , "New intumescent systems: An answer to the flame retardant challenges in the textile industry," in *Fire Retardancy of Polymers: The Use of Intumescence*, M. Le Bars, Camino, G., Bourbigot, S., and Delobel, R. (Eds.), Ed., London,: Royal Society of Chemistry, 1998, pp. pp. 363-375.

[54] D. Price*, et al.*, "Flame retardance of poly(methyl methacrylate) modified with phosphorus-containing compounds," *Polymer Degradation and Stability,* vol. 77, pp. 227-233, 2002.

[55] J. Asrar*, et al.*, "Synthesis and characterization of a fire-retardant polyester: Copolymers of ethylene terephthalate and 2-carboxyethyl(phenylphosphinic)

acid," *Journal of Polymer Science Part A: Polymer Chemistry,* vol. 37, pp. 3119-3128, 1999.

[56] S. Bourbigot*, et al.*, "Recent Advances for Intumescent Polymers," *Macromolecular Materials and Engineering,* vol. 289, pp. 499-511, 2004.

[57] W. J. E. Sinclair; Michael John, "Fire retardant intumescent coating, ," USA Patent, 2007.

[58] G. Camino, R. Delobel, , *Intumescence, In: Fire Retardancy of Polymeric Materials*: Marcel Dekker, New York, 2000.

[59] S. R. Gottfried, NY), "Intumescent fire-retardant composition for high temperature and long duration protection," United States Patent, 1998.

[60] WEIL*, et al.*, *Melamine phosphates and pyrophosphates in flame-retardant coatings: old products with new potential* vol. 66. Blue Bell, PA, ETATS-UNIS: Federation of Societies for Coatings Technology, 1994.

[61] S. K. Suzuki; Keitaro, Shindo; Masuo "Melamine polymetaphosphate and process for its production," United States, Patent, 1999.

[62] J. W. a. D. C. B. Hanafin, "Low Density, Light Weight Intumescent Coating," 2000.

[63] B. S. Pollak. (2005) Fire Protective Coatings: An Overview. *Modern Steel Construction*.

[64] UL, "263; Standard for Safety for Fire Tests of Building Construction and Materials."

[65] S. Duquesne*, et al.*, "Thermoplastic resins for thin film intumescent coatings – towards a better understanding of their effect on intumescence efficiency," *Polymer Degradation and Stability,* vol. 88, pp. 63-69, 2005.

[66] Liu F., "Fire retardant coating composition " Patent, 2000.

[67] Z. Wang*, et al.*, "Influence of expandable graphite on fire resistance and water resistance of flame-retardant coatings," *Corrosion Science,* vol. 49, pp. 2237-2253, 2007.

[68] J.-w. Gu, *et al.*, "Study on preparation and fire-retardant mechanism analysis of intumescent flame-retardant coatings," *Surface and Coatings Technology,* vol. 201, pp. 7835-7841, 2007.

[69] H. Q. M. Pham, M. J. , Weinheim, , "Epoxy Resins“," in *Ullmann's Encyclopedia of Industrial Chemistry (online-version),* : Wiley-Intersciene, 2005.

[70] A. D. La Rosa, *et al.*, "An oxygen index evaluation of flammability on modified epoxy/polyester systems," *Polymer,* vol. 40, pp. 4093-4098, 1999.

[71] B. Biswas, *et al.*, "A quantitative study of carbon monoxide and carbon dioxide evolution during thermal degradation of flame retarded epoxy resins," *Polymer Degradation and Stability,* vol. 92, pp. 765-776, 2007.

[72] E. D. Weil and S. Levchik, "A Review of Current Flame Retardant Systems for Epoxy Resins," *Journal of Fire Sciences,* vol. 22, pp. 25-40, January 1, 2004 2004.

[73] S. Y. Lu, I. Hamerton, "Recent developments in the chemistry of halogen-free flame retardant polymers," *Progr. Polym. Sci.,* vol. Vol. 27, pp. 1661–1712, 2002.

[74] T. Saito, "Cyclic organophosphorus compounds and process for making them," 1972.

[75] Z. Wang, *et al.*, "Effect of nanoparticles on the improvement in fire-resistant and anti-ageing properties of flame-retardant coating," *Surface and Coatings Technology,* vol. 200, pp. 5706-5716, 2006.

[76] W. Liu, *et al.*, "Char-forming mechanism of a novel polymeric flame retardant with char agent," *Polymer Degradation and Stability,* vol. 92, pp. 1046-1052, 2007.

[77] A. R. Horrocks, *et al.*, "Complex char formation in flame retarded fibre-intumescent combinations: 1. Scanning electron microscopic studies," *Polymer,* vol. 37, pp. 3197-3206, 1996.

[78] B. K. Kandola and A. R. Horrocks, "Complex char formation in flame-retarded fiber/intumescent combinations: Physical and chemical nature of char," *Textile Research Journal,* vol. 69, pp. 374-381, 1999.

[79] G. Li*, et al.*, "An Investigation of the thermal degradation of the intumescent coating containing MoO3 and Fe2O3," *Surface and Coatings Technology,* vol. 202, pp. 3121-3128, 2008.

[80] C. J. Hilado, *Flammability Handbook for Plastics*: Technomic Publishing Co, 1998.

[81] X. Almeras*, et al.*, "Effect of fillers on the fire retardancy of intumescent polypropylene compounds," *Polymer Degradation and Stability,* vol. 82, pp. 325-331, 2003.

[82] G. F. Levchik*, et al.*, "Thermal behaviour of ammonium polyphosphate—inorganic compound mixtures. Part 1. Talc," *Thermochimica Acta,* vol. 239, pp. 41-49, 1994.

[83] C.-S. Chou*, et al.*, "Preparation and characterization of the intumescent fire retardant coating with a new flame retardant," *Advanced Powder Technology,* vol. 20, pp. 169-176, 2009.

[84] G. Li*, et al.*, "Effects of EG and MoSi2 on thermal degradation of intumescent coating," *Polymer Degradation and Stability,* vol. 92, pp. 569-579, 2007.

[85] D. Scharf*, et al.*, "Studies on flame retardant intumescent char: Part I," *Fire Safety Journal,* vol. 19, pp. 103-117, 1992.

[86] Y. Tang*, et al.*, "PA-6 and EVA alloy/clay nanocomposites as char forming agents in poly(propylene) intumescent formulations," *Polymers for Advanced Technologies,* vol. 16, pp. 338-343, 2005.

[87] F. Laoutid*, et al.*, "Intumescent mineral fire retardant systems in ethylene–vinyl acetate copolymer: Effect of silica particles on char cohesion," *Polymer Degradation and Stability,* vol. 91, pp. 2140-2145, 2006.

[88] A. R. Horrocks, K.B.Kandola S. A. Padbury,, "Interactions between Nanoclays and Flame Retardant Additives in Polyamide 6 and Polyamide 6.6 Films," in *From Fire Retardancy of Polymers: New Applications of Mineral*

Fillers, M. L. Bras, Charles A. Wilkie, S. Bourbigot Ed.: Royal Society of Chemistry;, 2005.

[89] B. Howell, "Thermal properties of compounds possessing both solid-phase and gas-phase flame retardant potential," *Journal of Thermal Analysis and Calorimetry,* vol. 89, pp. 373-377, 2007.

[90] J. W. S. M. S. Gillman, Mt. Airy, MD, 21771, US), "NEW NON-HALOGENATED FIRE RETARDANT FOR COMMODITY AND ENGINEERING POLYMERS," 1998.

[91] X. Q. Cao*, et al.*, "New double-ceramic-layer thermal barrier coatings based on zirconia–rare earth composite oxides," *Journal of the European Ceramic Society,* vol. 26, pp. 247-251, 2006.

[92] X.-Q. Liu*, et al.*, "Synthesis of organo-modified α-zirconium phosphate and its effect on the flame retardancy of IFR poly(lactic acid) systems," *Polymer Degradation and Stability,* vol. 96, pp. 771-777, 2011.

[93] B. Du and Z. Fang, "Effects of carbon nanotubes on the thermal stability and flame retardancy of intumescent flame-retarded polypropylene," *Polymer Degradation and Stability,* vol. 96, pp. 1725-1731, 2011.

[94] F. Gao*, et al.*, "A mechanistic study of fire retardancy of carbon nanotube/ethylene vinyl acetate copolymers and their clay composites," *Polymer Degradation and Stability,* vol. 89, pp. 559-564, 2005.

[95] T. D. Hapuarachchi and T. Peijs, "Multiwalled carbon nanotubes and sepiolite nanoclays as flame retardants for polylactide and its natural fibre reinforced composites," *Composites Part A: Applied Science and Manufacturing,* vol. 41, pp. 954-963, 2010.

[96] N. A. Isitman and C. Kaynak, "Nanoclay and carbon nanotubes as potential synergists of an organophosphorus flame-retardant in poly(methyl methacrylate)," *Polymer Degradation and Stability,* vol. 95, pp. 1523-1532, 2010.

[97] F. Samyn*, et al.*, "Characterisation of the dispersion in polymer flame retarded nanocomposites," *European Polymer Journal,* vol. 44, pp. 1631-1641, 2008.

[98] Z. Wang, *et al.*, "Fire and Corrosion Resistances of Intumescent Nano-coating Containing Nano-SiO2 in Salt Spray Condition," *Journal of Materials Science & Technology,* vol. 26, pp. 75-81, 2010.

[99] M. R. Schütz, *et al.*, "Intumescent-like behavior of polystyrene synthetic clay nanocomposites," *Polymer,* vol. 52, pp. 3288-3294, 2011.

[100] C. A. Florentina T., Wilkie, "Combination of Carbon Nanotubes with Fire Retardants: Thermal and Fire Properties of Polystyrene Nanocomposites," in *Fire Retardancy of Polymers: New Strategies and Mechanisms*, B. K. T Richard Hull, Kandola, Ed., 2009.

[101] I. Sridhar and K. Narayanan, "Processing and characterization of MWCNT reinforced aluminum matrix composites," *Journal of Materials Science,* vol. 44, pp. 1750-1756, 2009.

[102] L. Ye, *et al.*, "Synergistic effects and mechanism of multiwalled carbon nanotubes with magnesium hydroxide in halogen-free flame retardant EVA/MH/MWNT nanocomposites," *Polymer Degradation and Stability,* vol. 94, pp. 751-756, 2009.

[103] S. A. Bortz, and Wonneberger, B., , ""Review of Durability Testing in the United States and Europe Dimension"," in *Stone Cladding: Design, Construction, Evaluation and Repair*, K.R. Hoigard, Ed., ASTM International, West Conshohocken PA: STP 1394, 2000, pp. pp. 94-108.

[104] X. Almeras, *et al.*, "Artificial Weathering and Recycling Effect on Intumescent Polypropylenebased Blends," *Journal of Fire Sciences,* vol. 22, pp. 143-161, March 1, 2004 2004.

[105] Z. Wang, *et al.*, "An investigation into fire protection and water resistance of intumescent nano-coatings," *Surface and Coatings Technology,* vol. 201, pp. 1528-1535, 2006.

[106] R. Benrashid and G. L. Nelson, "Effect of Environmental Weathering on the Flammability Behavior of Polycarbonate Sheet," *Journal of Fire Sciences,* vol. 12, pp. 452-480, September 1, 1994 1994.

[107] X. Shi, *et al.*, "Concurrent physical aging and degradation of crosslinked coating systems in accelerated weathering," *Journal of Coatings Technology and Research,* vol. 5, pp. 299-309, 2008.

[108] X. Shi and S. Croll, "Thermal-induced recovery of small deformations and degradation defects on epoxy coating surface," *Journal of Coatings Technology and Research,* vol. 7, pp. 73-84, 2010.

[109] F. Deflorian, *et al.*, "Comparison of organic coating accelerated tests and natural weathering considering meteorological data," *Progress in Organic Coatings,* vol. 59, pp. 244-250, 2007.

[110] C. A. Harper, *Handbook of Building Materials For Fire Protection*: Mcgraw Hill, 2004.

[111] R. L. P. E. Brockenbrough, "Properties of Structural Steel and Effects of Steelmaking and Fabrication," in *Structural Steel Designer's Handbook*, F. S. M. Roger L.Brockenbrough, Ed.: McGraw Hill New York., 1999.

[112] American Insitute of School Instruction, "Specification of structural Steel Buildings," 2005.

[113] F. G. Charles A.Wilkie, Arthur, , *Fire Retardancy of Polymeric Materials*. 270 Madison Avenue, New York: Marcel Dekker, Inc, 2000.

[114] B. Johan, *Additives for Coatings*. Germany: Wiley-VCH, 2000.

[115] R. G, "Surface characterization of iron and steel prior to coating," *Progress in Organic Coatings,* vol. 15, pp. 125-148, 1987.

[116] A. C. Rouw, "Model epoxy powder coatings and their adhesion to steel," *Progress in Organic Coatings,* vol. 34, pp. 181-192, 1997.

[117] ASTM, "Standard test method for fire tests of building construction and materials," in *ASTM E-119, American Society for Testing and Materials*. Conshohocke,PA.

[118] Z. Wang, *et al.*, "Influence of nano-LDHs on char formation and fire-resistant properties of flame-retardant coating," *Progress in Organic Coatings,* vol. 53, pp. 29-37, 2005.

[119] G. Wang and J. Yang, "Influences of binder on fire protection and anticorrosion properties of intumescent fire resistive coating for steel structure," *Surface and Coatings Technology,* vol. 204, pp. 1186-1192, 2010.

[120] C. Xiling*, et al.*, "Preparation of lower-sulfur content and expandable graphite," *Carbon,* vol. 34, pp. 1599-1600, 1996.

[121] R. Xie*, et al.*, "Dynamic FTIR studies of thermo-oxidation of expandable graphite-based halogen-free flame retardant LLDPE blends," *Polymer Degradation and Stability,* vol. 72, pp. 313-321, 2001.

[122] S. Zhang and A. R. Horrocks, "A review of flame retardant polypropylene fibres," *Progress in Polymer Science,* vol. 28, pp. 1517-1538, 2003.

[123] S. A. A. Ramazani*, et al.*, "Investigation of flame retardancy and physical–mechanical properties of zinc borate and aluminum hydroxide propylene composites," *Materials & Design,* vol. 29, pp. 1051-1056, 2008.

[124] A. B. Shehata*, et al.*, "Kaolin modified with new resin–iron chelate as flame retardant system for polypropylene," *Journal of Applied Polymer Science,* vol. 92, pp. 3119-3125, 2004.

[125] E. P. Lenka VACULÍKOVÁ , Silvie VALLOVÁ and Ivan KOUTNÍK "Characterization and Differentiation of Kaolinites from Selected Czech Deposits Using Infrared spectroscopy and Differential Thermal Analysis " *Acta Geodyn. Geomater,* vol. Vol. 8,, pp. 59-67, 2011.

[126] NACE Standard, "Joint Surface Preparation Standard," vol. SSPC-SP NO. 10/NACE NO. 2: The Society for Protective Coatings, 2000.

[127] M. Zhang*, et al.*, "A new method to prepare flame retardant polymer composites," *Journal of Materials Processing Technology,* vol. 208, pp. 342-347, 2008.

[128] JIS, "Testing methods for paints. Part 7: long-period performance of film. Section 7: accelerated weathering and exposure to artificial radiation (exposure to filtered xenon-arc radiation)," in *JIS K5600-7-7*: Japanese Standards Association, Tokyo, 2008.

[129] C.-L. Chiang and S.-W. Hsu, "Novel epoxy/expandable graphite halogen-free flame retardant composites–preparation, characterization, and properties," *Journal of Polymer Research,* vol. 17, pp. 315-323, 2010.

[130] P. Ducrocq*, et al.*, "Interactions between chlorinated paraffins and melamine in intumescent paint—investing a way to suppress chlorinated paraffins from the formulations," *Progress in Organic Coatings,* vol. 57, pp. 430-438, 2006.

[131] B. A.I, "Thermal decomposition study of intumescent additives: Pentaerythritol phosphate and its blend with melamine phosphate," *Thermochimica Acta,* vol. 435, pp. 188-196, 2005.

[132] Y.-L. Wang*, et al.*, "IR spectroscopy and theoretical vibrational calculation of the melamine molecule," *Journal of the Chemical Society, Faraday Transactions,* vol. 93, pp. 3445-3451, 1997.

[133] H. Huang*, et al.*, "Preparation and characterization of superhard boron-suboxide films," *Journal of Vacuum Science & Technology A: Vacuum, Surfaces, and Films,* vol. 21, p. 1595, 2003.

[134] S. Yu*, et al.*, "Nanofilms with clusters of boron suboxide and their infrared absorption," *Solid State Communications,* vol. 115, pp. 307-311, 2000.

[135] J.-S. Wang*, et al.*, "Polyamide-enhanced flame retardancy of ammonium polyphosphate on epoxy resin," *Journal of Applied Polymer Science,* vol. 108, pp. 2644-2653, 2008.

[136] D. Music*, et al.*, "Synthesis and mechanical properties of boron suboxide thin films," *Journal of Vacuum Science & Technology A: Vacuum, Surfaces, and Films,* vol. 20, pp. 335-337, 2002.

[137] O. Moon*, et al.*, "Temperature effect on structural properties of boron oxide thin films deposited by MOCVD method," *Thin Solid Films,* vol. 464-465, pp. 164-169, 2004.

[138] A. R. Horrocks, "Developments in flame retardants for heat and fire resistant textiles - The role of char formation and intumescence," *Polymer Degradation and Stability,* vol. 54, pp. 143-154, 1996.

[139] H. A.Richard, "Developments in flame retardants for heat and fire resistant textiles—the role of char formation and intumescence," *Polymer Degradation and Stability,* vol. 54, pp. 143-154.

[140] Y. Shi, *et al.*, "Crystal Structure and Thermal Decomposition Studies of Barium Borophosphate, BaBPO5," *Journal of Solid State Chemistry,* vol. 135, pp. 43-51, 1998.

[141] L. Montagne, *et al.*, "Mechanism of polyphosphate gel formation in the Na2O-Al2O3-P2O5 system," *Journal of Non-Crystalline Solids,* vol. 155, pp. 115-121, 1993.

[142] V. Bhatnagar, *et al.*, "Study of the combustion of a fire retardant coating by thermal analysis and different complementary techniques," *Journal of Thermal Analysis and Calorimetry,* vol. 30, pp. 467-478, 1985.

[143] S. Bourbigot, *et al.*, "Carbonization mechanisms resulting from intumescence-part II. Association with an ethylene terpolymer and the ammonium polyphosphate-pentaerythritol fire retardant system," *Carbon,* vol. 33, pp. 283-294, 1995.

[144] S. Ullah, *et al.*, "A study of bonding mechanism of expandable graphite based intumescent coating on steel substrate," *Journal of Applied Sciences,* vol. 11, pp. 1630-1635, 2011.

[145] S.-H. Chiu and W.-K. Wang, "Dynamic flame retardancy of polypropylene filled with ammonium polyphosphate, pentaerythritol and melamine additives," *Polymer,* vol. 39, pp. 1951-1955, 1998.

[146] S. Bourbigot, *et al.*, "PA-6 clay nanocomposite hybrid as char forming agent in intumescent formulations," *Fire and Materials,* vol. 24, pp. 201-208, 2000.

[147] W. Sawodny, *et al.*, "Vibrational spectrum and assignment of normal vibrations of melamine," *The Journal of Chemical Physics,* vol. 45, pp. 3155-3156, 1966.

[148] B. K. Kandola, *et al.*, "Empirical and numerical approach for optimisation of fire and mechanical performance in fire-retardant glass-reinforced epoxy composites," *Fire Safety Journal,* vol. 43, pp. 11-23, 2008.

[149] L. Costa, *et al.*, "Mechanism of Thermal Degradation of Fire-Retardant Melamine Salts," in *Fire and Polymers*. vol. 425: American Chemical Society, 1990, pp. 211-238.

[150] Z. Wang, *et al.*, "Thermal degradation study of intumescent flame retardants by TG and FTIR: Melamine phosphate and its mixture with pentaerythritol," *Journal of Analytical and Applied Pyrolysis,* vol. 86, pp. 207-214, 2009.

[151] M. K. Baykal A. G. Gozel, "X-Ray Powder Diffraction and IR Study of Calcium Borophosphate, $CABPO_5$," *Turk Journal of Chemistry,* vol. 24, pp. 381-388, 2000.

[152] A. M. V. RAY L. FROST, "The dehydroxylation of the kaolinite clay minerals using infrared emission spectroscopy " *Clays and Clay Minerals,* vol. Vol. 44, pp. 635-651, 1996. .

[153] N. Saikia, "Characterization, beneficiation and utilization of a kaolinite clay from Assam, India," *Applied Clay Science,* vol. 24, pp. 93-103, 2003.

[154] Z. Wang, *et al.*, "Effect of acrylic polymer and nanocomposite with nano-SiO2 on thermal degradation and fire resistance of APP–DPER–MEL coating," *Polymer Degradation and Stability,* vol. 91, pp. 1937-1947, 2006.

[155] A. R. K. H. Sepehriana, M.K. Rofouei and S. Waqif Husaina "Non-thermal synthesis of mesoporous zirconium silicate and its characterization," *Journal of the Iranian Chemical Society,* vol. Vol. 3, pp. pp. 253-257, 2006.

[156] H. S, "Phosphorus flame retardants in thermoset resins," *Polymer Degradation and Stability,* vol. 64, pp. 427-431, 1999.

[157] Y.-L. Liu, *et al.*, "Novel thermosetting resins based on 4-(N-maleimido)phenylglycidylether: III. Studies on the thermal degradation kinetics and mechanisms of the cured resins," *Polymer Degradation and Stability,* vol. 86, pp. 135-145, 2004.

[158] M. Jimenez, *et al.*, "Multiscale Experimental Approach for Developing High-Performance Intumescent Coatings," *Industrial & Engineering Chemistry Research,* vol. 45, pp. 4500-4508, 2006/06/01 2006.

[159] Z.-y. Wang, *et al.*, "Fire-resistant effect of nanoclay on intumescent nanocomposite coatings," *Journal of Applied Polymer Science,* vol. 103, pp. 1681-1689, 2007.

[160] J. Wang and Y. Chen, "Flame-retardant Mechanism Resulting from an Intumescent System," *Journal of Fire Sciences,* vol. 23, pp. 55-74, January 1, 2005 2005.

[161] T. Olcese and C. Pagella, "Vitreous fillers in intumescent coatings," *Progress in Organic Coatings,* vol. 36, pp. 231-241, 1999.

[162] G. Bertelli, *et al.*, "Parameters affecting fire retardant effectiveness in intumescent systems," *Polymer Degradation and Stability,* vol. 25, pp. 277-292, 1989.

LIST OF PUBLICATIONS

Journals

1. Sami Ullah, Faiz Ahmed and P. S. M. Megat Yusoff. "The effect on Expansion and Thermal degradation of 63µm Expandable graphite on Intumescent fire retardant coating composition. Published in Res. J. Chem. Environ., Volume 15, No. (2) June (2011) Pages 944-951.

2. Sami Ullah, Faiz Ahmad and P.S.M. Megat-Yusoff. "Study of bonding mechanism of expandable graphite based intumescent coating". Published *in the Journal of Applied Sciences 11(9):1630-1635.*

3. Jesbains Kaur, Faiz Ahmad, P. S. M. Megat Yusoff and Sami Ullah. "The Study of Bonding Mechanism of Expandable Graphite Based Intumescent Coating. Published in Res. J. Chem. Environ., Volume 15, No. (2) June (2011) Pages 401-405.

4. Sami Ullah, Faiz Ahmed and P. S. M. Megat Yusoff. "Effect of boric acid with kaolin clay on thermal degradation of intumescent fire retardant coating". Journal of Applied Sciences Vol(11): 3645-3649.

5. Sami Ullah, Faiz Ahmed and P. S. M. Megat Yusoff. "Enhancing the Char Strength of Intumescent Fire Retardant Coating by using Carbon Nano Tubes". Accepted in Solid State Phenomena.

Conference proceedings

1. Sami Ullah, Faiz Ahmad and P.S.M. Megat-Yusoff. "Effect of kaolin clay on thermal degradation of intumescent fire retardant coating". Published in the proceeding of the Program for 24th Symposium of Malaysian Chemical Engineers / 1st International Conference on Process Engineering and Advanced Materials (ICPEAM) June 2010.

2. Sami Ullah, Faiz Ahmed and P. S. M. Megat Yusoff. "Enhancing the Char Resistant of Intumescent Fire Retardant Coating by using Carbon Nano Tubes". Published in the Proceeding of International Conference on Materials for Advanced Technologies, Singapore 26 Jun to 1 July 2011.

3. Sami Ullah, Faiz Ahmad and P.S.M. Megat-Yusoff. "Study of bonding mechanism of expandable graphite based intumescent coating". Published in the proceeding of the 2nd International Conference on Plant Equipment and Reliability (ICPER) June 2010.

Submitted Research Articles

1. Sami Ullah, Faiz Ahmed and P. S. M. Megat Yusoff. "Effect of Boric acid and Melamine on the Intumescent Fire retardant Coating composition for the fire protection of structural steel substrate". Under review to the Journal of Applied Polymer Science.

2. Sami Ullah, Faiz Ahmed and P. S. M. Megat Yusoff. "Effect of Zirconium silicate on expandable graphite based Intumescent Fire retardant Coating". Under review to the Polymer degradation and stability.

3. Sami Ullah, Faiz Ahmed and P. S. M. Megat Yusoff. Effect of Ammonium Polyphosphate and Boric acid on the thermal degradation of intumescent fire retardant coating. Under review to the Journal of Fire Sciences.

4. Sami Ullah, Faiz Ahmed and P. S. M. Megat Yusoff. Effect of EG and Ammonium Polyphosphate on the fire performance of intumescent fire retardant coating. Under review Journal of Fire Sciences.

Achievements in Local and International Exhibitions

1. Sami Ullah, Faiz Ahmad and P.S.M. Megat-Yusoff. "Development of intumescent fire retardant coating for the fire protection of structural steel". 25th Engineering Design Exhibition Universiti Teknologi PETRONAS 21-22 April 2010.

   i. Champion of Most Innovation Award.

   ii. Gold Medal

   iii. Best Presenter Award

   iv. Best Poster Award

2. Faiz Ahmad, Sami Ullah, P.S.M. Megat-Yusoff. "Efficient Intumescent Fire Retardant Coating. Malaysian Technology Expo (MTE) 2011, Convention Center Kuala Lumpur 17-18 February 2011.

   i. Gold Medal

3. Faiz Ahmad and Sami Ullah. Efficient Intumescent Fire Retardant Coating. Invention & New Product Exposition (INPEX) 2011 held on 15-17 June 2011. Monroeville Convention Center, Pittsburgh, Pennsylvania, USA

   i. Gold Medal

4. Sami Ullah and Faiz Ahmad P.S.M. Megat-Yusoff. "Intumescent Fire Retardant Coating for the Fire Protection of Steel Structures". 28th Science and Engineering Design Exhibition Universiti Teknologi PETRONAS 10-11 August 2011.

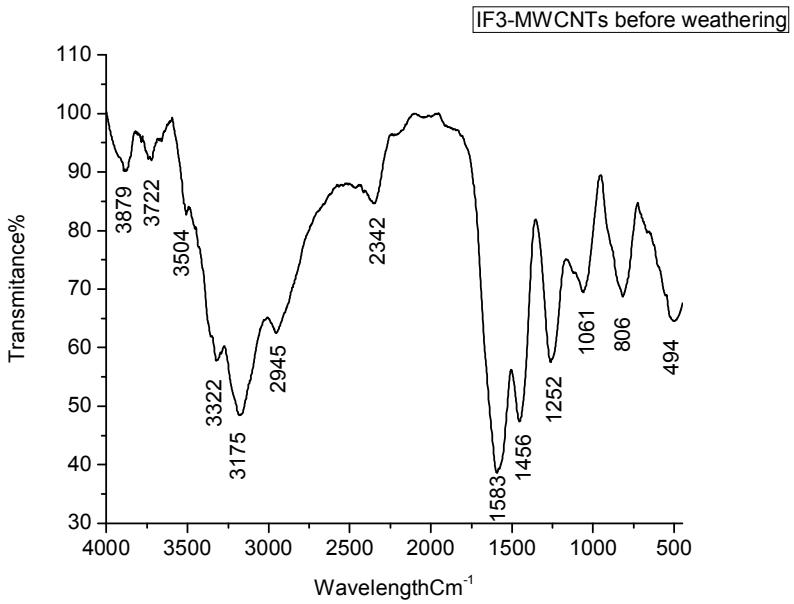
i. Gold Medal

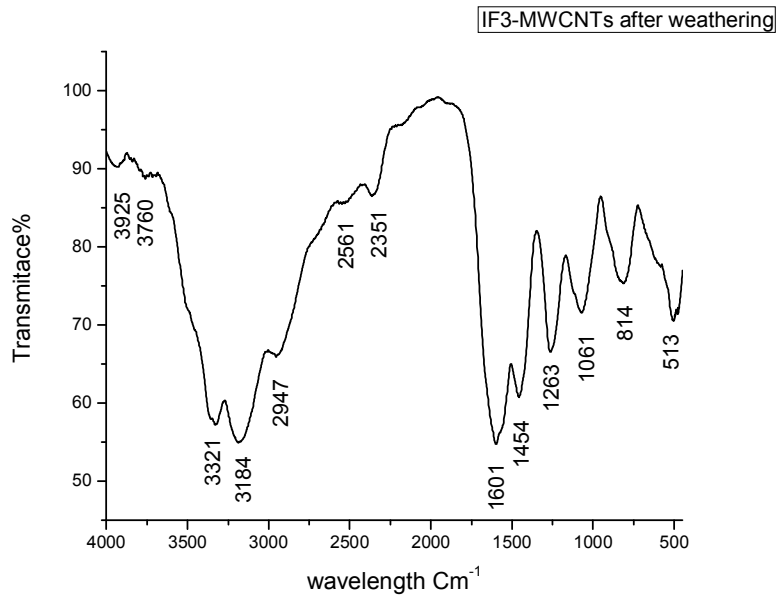
Patent

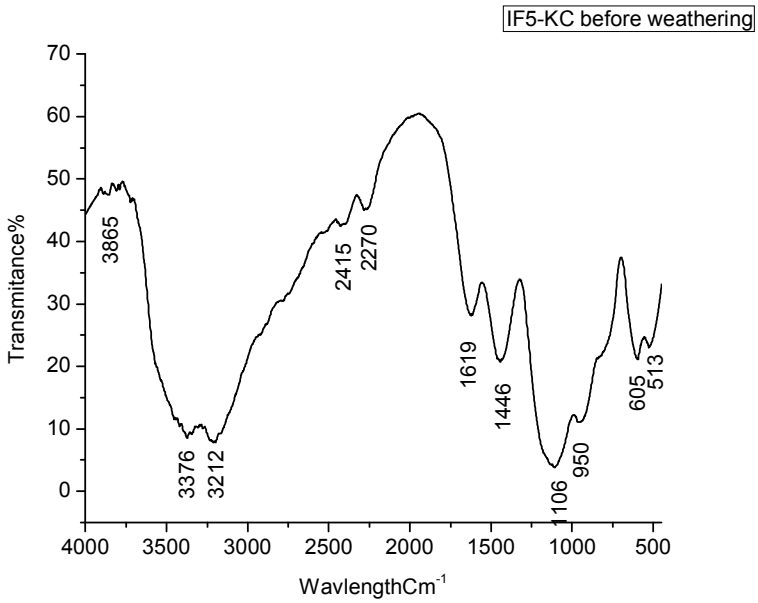
Development of New Intumescent Fire Retardant Coating, in progress under Universiti Teknologi PETRONAS, 09 November 2010.

APPENDIX A

FUNCTIONAL GROUPS OF REINFORCED ICF BEFORE AND AFTER WEATHERING TEST






IF5-KC before weathering
Transmitance%
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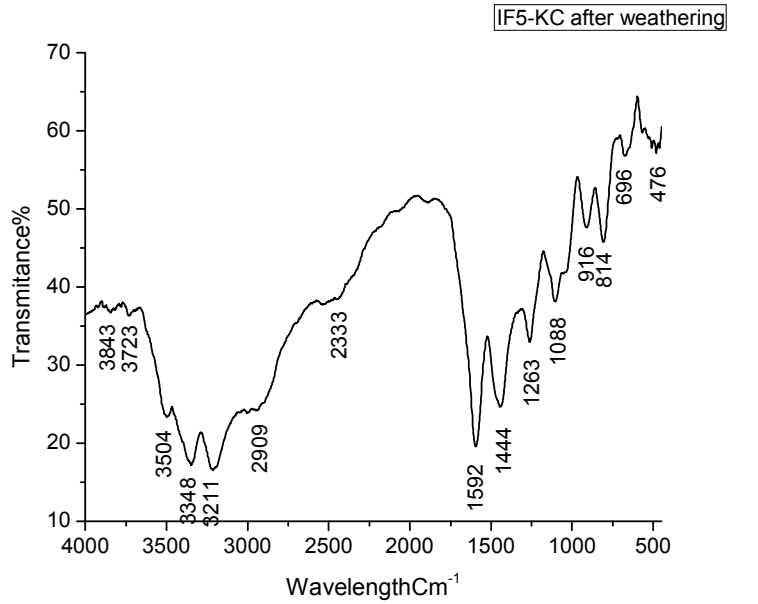
3212
2415
2270
1619
1446
1106
950
605
513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Transmitance%
WavelengthCm-1
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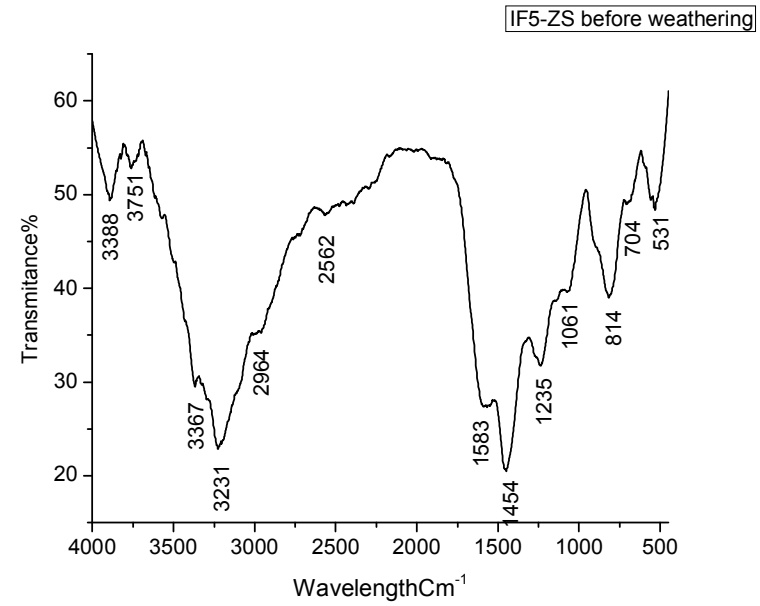
2333
1592
1444
1263
1088
916
814
696
476


IF5-ZS before weathering
Transmitance%
WavelengthCm-1
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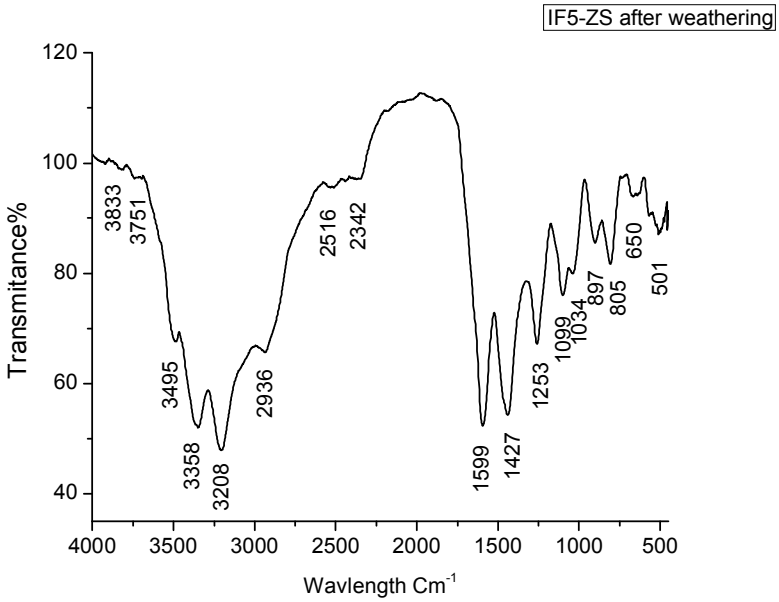
1583
1454
1235
1061
814
704
531


IF5-ZS after weathering
Transmitance%
Wavlength Cm-1
3833
3751
3495
3358
3208
2936
2516
2342
1599
1427
1253
1099
1034
897
805
650
501