Effects of Particle Size of Fumed Silica and Aluminium Trihydrate on Fire Performance of Intumescent Fire Retardant Coating

By

Shiva Sarvanan Pillai Subramaniam

Dissertation submitted in partial fulfillment of

the requirements for the

Bachelor of Engineering (Hons)

(Mechanical Engineering)

August 2012

Universiti Teknologi PETRONAS

Bandar Seri Iskandar

31750 Tronoh

Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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Approved by,

____

(Associate Professor Dr Faiz Ahmad)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

August 2012

CERTIFICATION OF ORIGINALITY

This is certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and the original work contained herein have not been undertaken or done by unspecified sources or person.

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(SHIVA SARVANAN PILLAI SUBRAMANIAM)

Abstract

Intumescent coating is a form of passive fire protection where paint is coated on the metal and later reacts chemically in fire. The coating expands to greater size forming char that protects the steelwork for a specified time period from the heat of fire thus elongate the time for the occupants to escape. In order to produce an efficient intumescent system, three ingredients are required; an inorganic acid, a carbon rich polyhydric material as char former (carbonific) and a blowing agent called a spufimic. Aluminium trihydrate and fumed silica are two fillers used widely in intumescent coatings composition and is proven to be efficient in thermal insulation and durability of char under excessive fire. Different types of formulations are prepared for the intumescent coating and each formulation is altered in the mixture of fumed silica and ATH under different particles sizes.

The particle size of both fumed silica and ATH are altered by grounding it into smaller sizes. The variation of surface area of both fumed silica and ATH will directly affect their performances in technical applications such as thickening efficiency, reaction with heat and the expansion rate of the intumescent coating.

The effects of the particle size of the fillers on fire performance and char expansion rate has been studied. The results obtained from the fire testing will be compared and analyzed in order to identify the best mixture for intumescent coating. Then 4 best samples which provided maximum char expansion will be chosen. Those samples will be analyzed using TGA and XRD to study its residual weight and composition of how percentage of fillers has on intumescent coating. Overall this study identified the best mixture of formulation of intumescent coating with high flame retardant characteristics towards heat/fire.

ACKNOWLEDGEMENTS

Firstly, I would like to express appreciation to UTP for providing laboratory equipments, facilities, related materials and funds for me to be able to conduct this project according to plan.

I would like to take this opportunity to thank my supervisor, Associate Professor Dr Faiz Ahmad for sharing his knowledge, experiences and guiding me all throughout this project. Dr Faiz Ahmad have been guiding for me in so many aspects such as providing the details on the experiments, the preparation needed to be done and in report writing.

Special thanks to Mechanical Engineering technicians for assisting and guiding me during the whole lab sessions. Finally, I am indebted to so many people who have been helping me during to completing this project, where their presences are vital in making this project successful. They are people of my respects who involve directly or indirectly throughout this project.

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CHAPTER 1: INTRODUCTION

1.1 Background of study

Fire safety has become a prime requirement of building regulations in many countries globally. The strength and load- bearing capacity of the steel decreases rapidly with increasing temperature, hence heating the steel structure of buildings above 500 Celcius may lead the building to fragile and collapse. In order to prevent these incidents, steel protection from fire becomes necessary. Intumescent coatings are widely used for ensuring effective flame retardant for structural concretes. Besides, various fillers are added in the coating and its fire retardant level is analyzed and tested [1].

The method of fire protection for building is known as passive fire protection which is intumescent fire retardant coating. Economic competitiveness and environmental concerns have been forcing the experts to come up with new formulation and approaches to improve the effective of mineral based coatings. The effectiveness of protection of substrate against fire depends on factors such as quality of the coating, the substrate's (steel) characteristics, and properties of the coating and possible hazards surrounding the building. Intumescent coating protect the steel by two methods; first it acts as a thermal barrier and secondly by react chemically with the heat when fire occurs [2].

Fumed silica is widely used as inorganic filler in intumescent coating and its mechanical behavior of silica composites has been for years under modification involving of the particle surface and polymer grafting [3]. In this article is says, great attention have been paid to polymeric composite with halogen free flame retardant like Aluminium Trihydrate (ATH) [4]. In order to utilise ATH effectively in intumescent coating, the dispersion and compatibility of the inorganic filler in polymer matrix are improved. Besides that, appropriate coupling agent and compatibiliser are selected in order to improve the mechanical properties of the ATH nanocomposites [5].

1.2 Problem statement

Most of the construction materials have been equipped with passive fire protection known as intumescent coatings. Intumescent fire retardant coating is a layer constructed which will act as a thermal barrier for construction materials in the event of fire hazard occurs. Traditional fire retardant are not adequate to cover large fire breakdown in the site or plant. The problem with the available coatings in the market are such as low thermal degradation of the substrate, loss of mechanical properties in the substrate and the release of toxic gases during the burning of halogenated flame retardant materials. In our research the efficiency of the coating will be improved by the altering the particle size of fumed silica and ATH. Subsequently its fire performance towards the intumescent fire retardant coating will be analyzed.

1.2.1 Problem Identification

Different inorganic fillers with different particle size react with intumescent coating ingredients thus resulting in different efficiency of the coating at the end.

1.2.2 Significant of Project

The purpose of this project is to study the effects of the particle sizes on fire performance on intumecent fire retardant coating with respect to use of different inorganic fillers(Fumed Silica and Aluminium Trihydrate,ATH). The reasons of choosing the two fillers (Fumed silica and ATH) are both posses' thermodynamic properties and ability to act effectively towards flame retardant. It will be tested with fire to analysis its heat shielding ability.

1.3 Objectives and Scope of Study

Objectives:

- Main objective of this study is to determine the effects of the particle size of fumed silica and aluminium trihydrate(ATH) on fire performance of the intumescent fire retardant coating. .In this study a range of formulation which includes the composition of expandable graphites, boric acid, ammonium polyphosphate (APP), melamine, fumed silica, aluminium trihydrate, epoxy and hardener are developed. This study will improve the thermal barrier and increase the thermal insulation of intumescent coating in case of fire. The particle sizes will be altered and varied in each formulation of the coating and the subsequent result will be analyzed.
- It is expected the grounded particle size of both fillers will provide better and stronger intumescent effect towards heat compared to non grounded

Scope of Study:

1. The percentage of aluminium trihydrate and fumed silica are used in the range of 0 to 2.5 % and 2.5% to 0% respectively. Two types of fire tests will be conducted upon the coating are applied to the specimens which are furnace test and burner test. The flammability characteristics and thermal degradation behavior of systems are studied under using thermogravimetric analysis (TGA). The morphological structure of the layer produced is analysed by using X-Ray diffraction (XRD).

2. An effective intumescent coating can be formed by implementing the accurate formulae and the formulae is later altered according to the particle sizes as it is part of the study as well.

1.4 Relevance of the Study

In this study, the effect of the particle sizes of the inorganic fillers on the efficiency of the intumescent coating is discussed. The study has relevant to the programme the author is undertaking, Mechanical Engineering. Under this programme there are several courses that contribute information to the author to equip hi with better understanding on the fillers. Among the courses are Introduction to Material Science, Engineering Materials, Manufacturing Technology, Failure Analysis and Advance Polymer Engineering.

1.5 Feasibility of the Project within the Scope and Time Frame

The first step in this project will be getting an introduction to the related topics by reading books, journals and research papers. Research will be done in order to understand better on the effects of particle size of fumed silica and ATH on fire performance of intumescent fire retardant coating. Each formulation is prepared and applied to the substrate/steel and dried. This process will take 1 month for the coating to dry completely as the coatings will be ensured fully dried and hardened on the steel. Once all the substrate is dried, it is then tested with fire using Bunsen burner and furnace. Lastly the burned substrates are studied and analyzed under TGA and XRD.

CHAPTER 2: LITERATURE REVIEW

From the journal "Thermal Degradation study of fire resistive coating containing melamine polyphospate and dipentaerythritol" by GuoJian Wong J.Y [6], intumescent fire resistive coatings have been as a common solution used as passive fire protection for steel structure in industry. In this study, general fire resistive coatings consist of three fire retardant additives; an acid source, a carbon source and a blowing agent which is bounded together by a polymer binder. Upon heating, the coating swells and forms an intumescent char layer

From the journal "Parameters Affecting Fire Retardant Effectiveness in Intumescent System" by GC Bertellia [7] which is related to fillers, the combustion behavior of an intumescent fire retardant filled with inorganic fillers is evaluated in relation to its foaming behavior and to the structure and thermal insulation properties of the char formed on burning. It is discovered in this study that by increasing the amount of filler, there is higher tendency to a decrease in swelling and loss of the foamed structure of the char while thermal insulation improves and fire retardant characteristics is worsen. On continuous burning the fillers decrease the rate of combustion.

The justification of choosing fumed silica and ATH are explained in the journal paper published by"Gabriele Landucci F.(2009). Desing and Testing of Innovative Materials For Passive Fire Protection" where inorganic materials like fumed silica and ATH are inert to flame impingement and keep the initial configuraion with excellent thermal resistance. The tendency of them to become brittle after continous heat exposure is high is explained in this article. On the other hand, organic based minerals in intumscent coating provide better mechanical properties but they can be only suitable to obtain ligth structures which is fragile and have to be replaced occasionally. Similarity between both organic and inorganic are they are subjected to thermal degradation due to fire impingement [8].

From the journal "Fire Mater" by B.Ostman, A-Voss,A-Hughes, PJ Havke and Q-Grexa, during the intumescent process, the binder become important due to two effects, it contributed to the char layer expansion and ensured the formation of foam structure. Acid source such as ammonium phosphate (APP) and carbon source, pentaerythritol(PER) in the coatings are very sensitive to corrosive substance such as water, acid and alkali [9].

The importance of particle sizes are reviewed in the journal entitled “Flame retardant mechanism of silica gel/silica.; Fire Mater, 24 (2000), pp. 277–289” which is reviewed by T. Kashiwagi, J.W. Gilman, K.M. Butler and R.H. Harris. It is explained in this journal, if the inorganic particles are too big then they will filter out on the carbon fiber fabric during composite manufacture or worse, create defect sites for micro cracking and mechanical failure. Therefore particle sizes of 5micrometer or smaller are needed and silica and alumina are both available in this particle size. Fumed silica was chosen due to known effects of reducing mass loss and helping to form char in some polymer systems under thermal decomposition temperature [10-13].

According to Alexander B. Morgana in his study of “Use of inorganic materials to enhance thermal stability and flammability behavior of a polyimide”, alumina represents an inert filler and bulk ceramic shield and so was chosen as a relatively inexpensive filler to do most of thermal protection and work in a potentially synergistic manner with the other nanofillers. In this study, alumina is calcined and finely grounded, and then it should have very little effect on polyimide processing since its typical surface chemistry (Al-OH) that would interact with polyimides will have been minimized. This point is important since nanomaterials used to produce polymer nanocomposites often increase resin viscosity to the point that the material is processing while providing thermal protection [14].

The importance of expandable graphite in intumescent coating is well explained by Bhagat VJ in his publication entitled “BEHAVIOUR OF EXPANDABLE GRAPHITE AS A FLAME”. According to the article, expendable graphite is manufactured using natural graphite flake. Natural graphite flake is a layered crystals consisting of sheets of carbon atoms. The atoms are tightly bound to each other within a layer, but the layer themselves are weakly held together. When expendable graphite is exposed to heat, it expands to more than 100 times its original volume and covers the entire burning surface by “worm” like structure of expanded graphite. Expended graphite acts as a char former and also as an insulating agent due to the formation of small air gaps between the graphite layers. It dramitacally reduces the heat release, mass loss, smoke generation and toxic gas emission. However, all expendable graphite does not act as flame retardant, only low temperature expendable graphite acs as flame retardant. The expansion must occur at "critical temperature" where decomposition, exothermal reaction and ignition occur spontaneously. This critical temperature range depends upon the chemical composition of polymer. Critical temperature in flexible polyurethane is 300˚ C to 500˚ C [15].

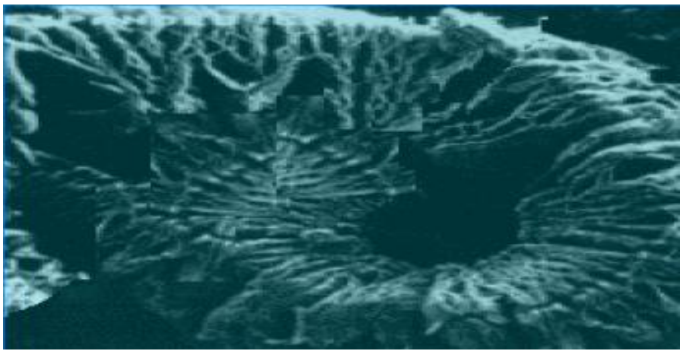


Figure 1: Worm like structure of Expandable Graphite [15]

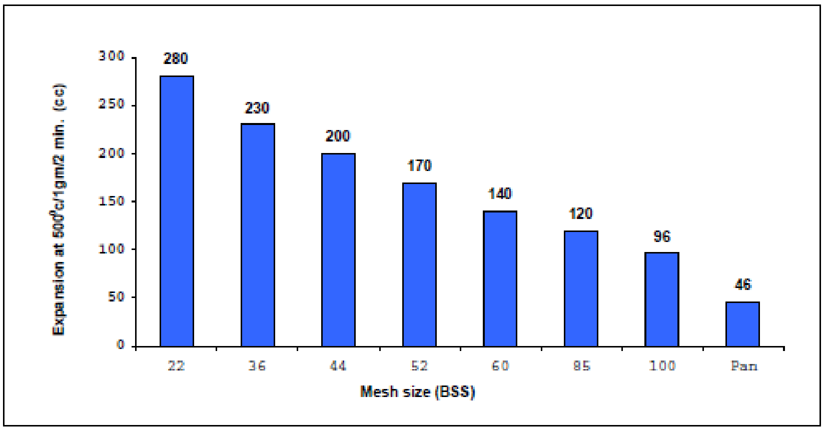


Figure 2: Expansion of expandable graphite with respect to various particle sizes [15]

For the study of nano coating, the journal entitled "Effect of nanoparticles on the improvement in fire-resistant and anti-ageing properties of flame-retardant coating" published by Wang Z has attracted a lot of attention as a simple and cost effective method of enhancing coating properties by the addition of a small amount of properly designed and dispersed nanometer fillers. However, studies on application of nano particles in ammonium polyphosphate-melamine-pentaerythritol coating are scarce in the literature, in this study; the reports are based on the preparation nd characterization of nano coating wit nano-LDHs and nano-TiO_2. Nano-LDHs are highly promising flame retardants since their application perspectives are connected with a unique fire resistant property. Nano LDHs absorb the heat and send out the H_20 and CO_2 when heated, which can lower the temperature of the substrate and enhance char structure. Nano-TiO_2 is very attractive in practical applications because of such advantages as permanent stability under UV exposure, high anti corrosion and chemical stability high temperature [16-17]. The effect of nano-LDHs and nano-TiO_2 on the improvement in fire resistant and anti ageing properties of the APP-Per-Mel coating is investigated using scanning electron microscopy (SEM), X-ray diffraction (XRD), differential thermal analysis (DTA), thermogravimetry analysis (TGA) and fire protection test. The DTA and TGA data show that the thermal decomposition of nano LDHs at 310˚ C – 430˚ C is helpful to the char formation of the APP-PER-MEL system at 300˚C – 440 C. The XRD and SEM data show that the thermal decomposition of nano-LDHs leads to the formation of an intercalated nanostructure of the char and mixed metal oxides (AL_2O_3 and $MgAl_2O_4$). The intercalated nano structure can improve the anti-oxidation property of the char structure [18].

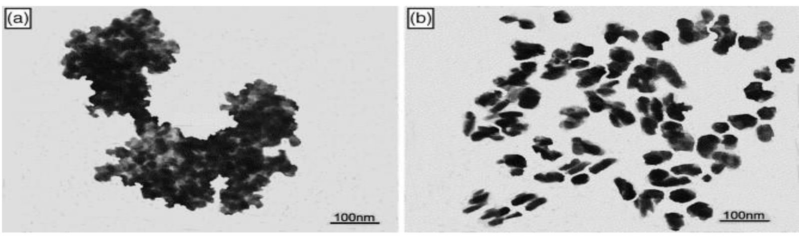


Figure 3: TEM (Transmission Electron Microscopy) image of unmodified nanoparticles (a) and nanoparticles modified by NJ-2 polymer dispersing agent (b) [18]

The importance of the fillers chosen can be seen from the study conducted by Lei Ye where from his article entitled “Synergistic Effects of Fumed Silica on Intumescent” the characteristics of fumed silica in intumescent coating are explained. It is investigated that fumed silica is usually used as an enhancing agent in thermoplastic polymer to increase the mechanical properties, such as tensile strength and toughness. In this study, the synergistic effect of fumed silica on combustion and flame retardant propertied is analyzed. It is found that only suitable amount of fumed silica can increase the thermal stability and leave more char residues after 500˚ C. Excessive amount of fumed silica could restrict the formation of the char layers and destroy the swelling behavior of intumescent layers which would deteriorate the flame retardant and thermal properties of the substrate [19].

Meanwhile, in the presence of aluminium trihydrate (ATH) the synergistic effect be augmented significantly. This can be reviewed in the publication released by A De Fenzo entitled “SYNERGISTIC EFFECTS OF ZINC BORATE AND ATH”. For ATH filler, the presence of the micro sized particle has a detrimental effect at lower concentrations compared with the neat epoxy system [20].

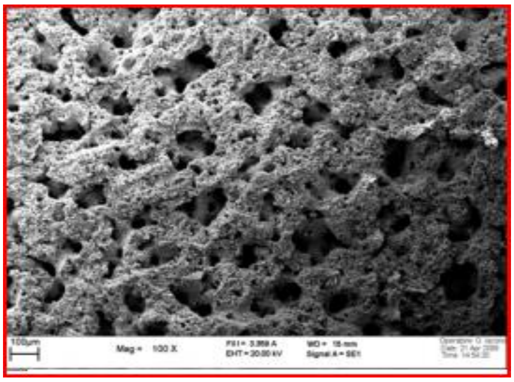


Figure 4: Granular porous like structure characterized by very small particles of additive along with some residual carbon from the resin for the ATH mixture [20]

CHAPTER 3: METHODOLOGY

3.1 Project Work

In order to achieve the objectives of this project, some researches had been done on some resources from books and technical papers. Different findings and methodologies are gathered from the research work of other researchers and to be included in this project. First and foremost, various journals and technical papers were read through the get the general understanding on the project. It is also needed to identify the objective of this project and to come up with planned schedule on executing the experiment. Diagram below explains the author's approach in executing the project effectively.

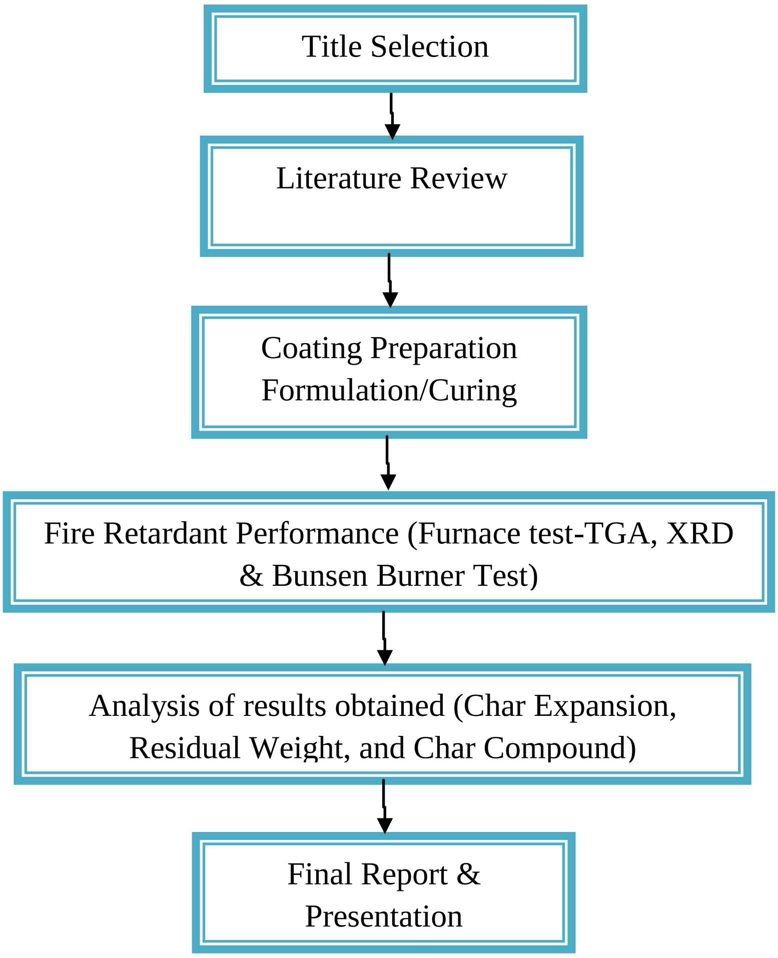


Figure 5: Project Activities Flow Chart

3.2 Project Activities

Details of Flow Chart:

i. **Title Selection :** An appropriate title is selected for Final Year Project
ii. **Preliminary Research/Literature Review:** Studies on related journals, textbooks and magazines to get better understanding of the Project chosen
iii. **Coating Preparation Formulation:** Formulation for the experiments are identified and applied to the substrates and let for natural curing. This process takes approximately 1 month.
iv. **Fire Retardant Performance:** Samples are burned under furnace and the expansion of the char is calculated. Besides, samples are tested with Bunsen Burner Test to study its Heat Shielding Effect. From the result obtained, 4 samples with maximum expansion and best heat shielding properties are further analyzed under TGA and XRD.
v. **Analysis of the result obtained:** The residual weight of the compound is obtained from TGA result and the from XRD analysis, important compound present can be identified to analysis better on char strengthening factor. From all the results and analysis done, recommendations are made from the formulation results which meet the objective of the study.
vi. **Final Report and Presentation:** A complete dissertation is submitted as per the guidelines requirement and a presentation is done on explaining the importance of this study with necessary recommendations.

The experiment to produce the coating is shown below. Once the coating is produced based on different formulation via two types of inorganic fillers (Fumed Silica and ALuminium Trihydrate), it is then coated onto steel plates. The steel plates used for 1 formulation is 5 pieces consist of 1 large piece (10cm X 10cm) and 4 small pieces (5cm X 5cm). These plates are left for ten to15 days at room temperature to ensure it dried fully. Substrate produced is now tested with Burner test and furnace heating. The results are recorded and are further analyzed via XRD and TGA microscope.

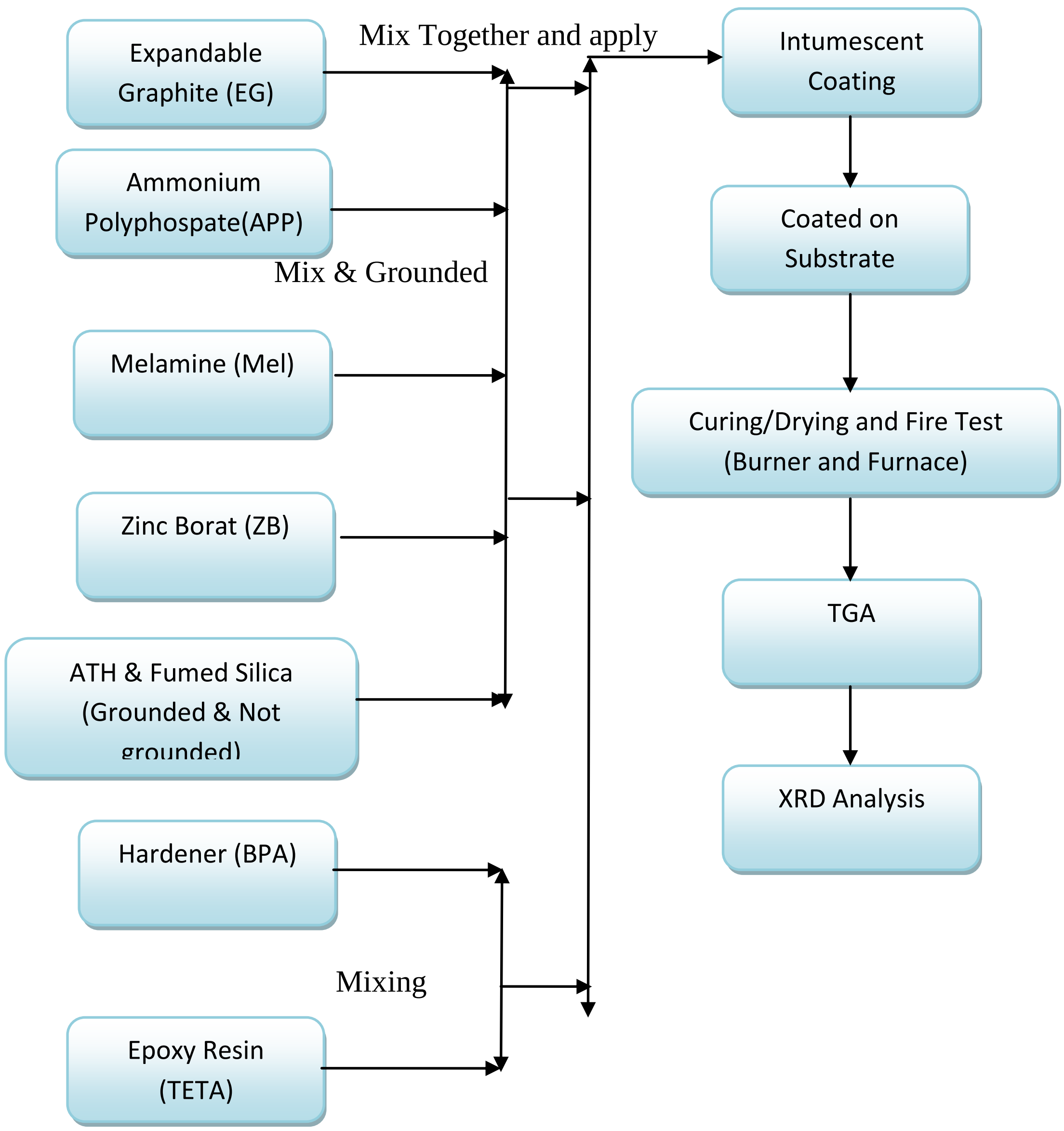


Figure 6: Experimental process overview upon developing Intumescent Coating

Formulations for sample preparation:

Table 1: Formulation of mixture for Intumescent coating developed (With ground the fillers)

Formulation	*EG (g)*	*APP(g)*	*MEL(g)*	*ZB(g)*	*ATH (g)*	*FS(g)*	*BPA(g)*	*TETA(g)*	*Total (g)*
1	**5.56**	**11.11**	**5.56**	**11.11**	**0**	**0**	**44.44**	**22.22**	**100.00**
2	**5.56**	**11.11**	**5.56**	**11.11**	**0.5**	**0.5**	**43.94**	**21.72**	**100.00**
3	**5.56**	**11.11**	**5.56**	**11.11**	**1.0**	**1.0**	**43.44**	**21.22**	**100.00**
4	**5.56**	**11.11**	**5.56**	**11.11**	**1.5**	**1.5**	**42.94**	**20.72**	**100.00**
5	**5.56**	**11.11**	**5.56**	**11.11**	**2.0**	**2.0**	**42.44**	**20.22**	**100.00**
6	**5.56**	**11.11**	**5.56**	**11.11**	**2.5**	**2.5**	**41.94**	**19.72**	**100.00**

Table 2: Formulation of mixture for Intumescent coating developed (Without ground the Fillers)

Formulation	*EG (g)*	*APP(g)*	*MEL(g)*	*ZB(g)*	*ATH (g)*	*FS(g)*	*BPA(g)*	*TETA(g)*	*Total (g)*
1	**5.56**	**11.11**	**5.56**	**11.11**	**0.5**	**0.5**	**43.94**	**21.72**	**100.00**
2	**5.56**	**11.11**	**5.56**	**11.11**	**1.0**	**1.0**	**43.44**	**21.22**	**100.00**
3	**5.56**	**11.11**	**5.56**	**11.11**	**1.5**	**1.5**	**42.94**	**20.72**	**100.00**
4	**5.56**	**11.11**	**5.56**	**11.11**	**2.0**	**2.0**	**42.44**	**20.22**	**100.00**

List of Ingredients required for Intumescent coating preparations:

EG	**Expandable Graphite**
APP	**Ammonium Polyphospate**
MEL	**Melamine**
ZB	**Boric Acid**

ATH	**Aluminium Trihydrate**
FS	**Fumed Silica**
BPA	**Epoxy Resin (Bisphenol A)**
TETA	**Tetraethylene-Tetamine (TETA)**

- **Particle size of fumed silica is 0.014micrometer**
- **Particle size of aluminium trihydrate is 1micrometer**

3.2.1 Procedure of preparing Intumescent Coating (Ground)

1. Using digital weighing machine, measure the weight of each materials and collect it in a container.
2. Expendable graphite is not included in the container.
3. Materials in the container are now grounded using grinder for a time of 60 seconds.
4. Grounded material is now mixed with expendable graphite and is collected in the container.
5. In a paper cup, both epoxy and hardener are mixed and via mixer it is mixed until a thick white layer is produced.
6. Simultaneously, the materials from the container are now poured in the paper cup containing epoxy and hardener and are mixed using mixer for a period of 15 minutes. The speed of mixer is fixed at 30 rpm.
7. Completed mixture is coated on steel plates with specific dimension and left for curing naturally under room temperature for approximately 30 days.

3.2.2 Procedure of preparing Intumescent Coating (Fillers Not Ground)

1. Using digital weighing machine, measure the weight of each materials and collect it in a container.
2. Expendable graphite, fumed silica and ATH are not included in the container.
3. Materials in the container are now grounded using grinder for a time of 60 seconds.
4. Grounded material is now mixed with expendable graphite, fumed silica and ATH and is collected in the container.
5. In a paper cup, both epoxy and hardener are mixed and via mixer it is mixed until a thick white layer is produced.
6. Simultaneously, the materials from the container are now poured in the paper cup containing epoxy and hardener and are mixed using mixer for a period of 15 minutes. The speed of mixer is fixed at 30 rpm.
7. Completed mixture is coated on steel plates with specific dimension and left for curing naturally under room temperature for approximately 30 days.

3.2.3 Furnace Test

Once all the coating has dried, it is then tested under Furnace at temperature of 500˚C for a period of 30 minutes. For furnace, the sample chosen is the small steel (5cm X 5cm). It is later let for dwelling and finally for cooling till it reaches room temperature, 25˚C. The char expansion/thickness for each sample is calculated by comparing the expansion to the original thickness of the samples. This test applies for both grounded and non grounded samples.

Figure 7: Furnace Test Machine used for char expansion

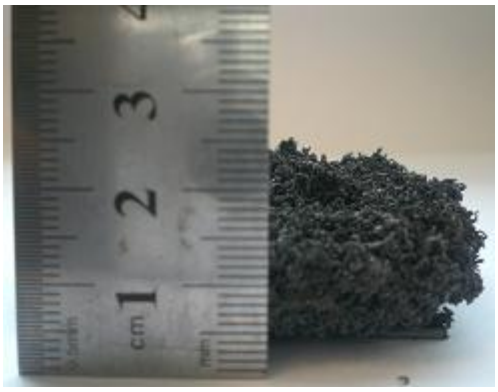


Figure 8: Char of samples after burned in furnace

3.2.4 Heat Shielding Effect:

For heat shielding effect, large sample (10cm X 10cm) is used to do the testing. Each sample are burned under Bunsen burner for a period of 40 minutes to 50 minutes and subsequently the readings are taken via data logger. During the testing, air sucker is ensured switched on to remove the chemical odor released from the burning. The samples which gives the minimum rise in the temperature reading is one with best heat shielding effect and this can be found from this test.

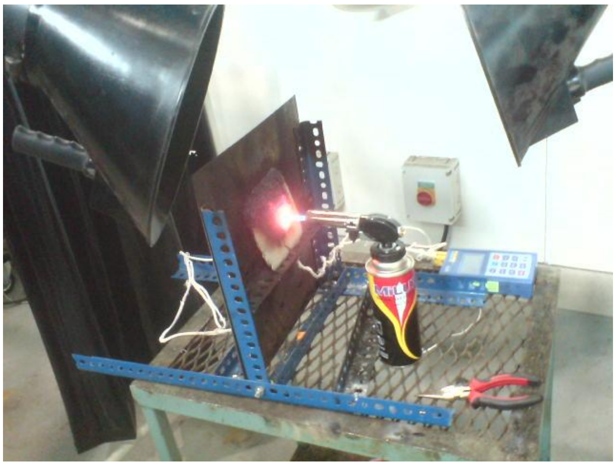


Figure 9: Heat shielding test arrangement

3.3 Gantt Chart and Key Milestone

No	Detail / Week	1	2	3	4	5	6	7	Mid Semester Break	8	9	10	11	12	13	14	15
1	Coatings are ensured dried and permanent. Fire test are conducted for all the samples	■	■	■	■	■	■	■									
2	Submission of Progress Report									★							
3	Analysis and detailed study of the burned samples through XRD and SEM									■	■	■	■	■			
4	Pre EDX												★				
5	Submission of Draft Report													★			
6	Submission of Dissertation (softbound)														★		
7	Submission of Technical Paper														★		
8	Oral Presentation															★	
9	Submission of Dissertation (hard bound)																★

Figure 10: Gantt chart for the second semester project implementation

■ Processes ★ Milestones

3.4 Tools required

- X-Ray Diffraction (XRD) and FTIR analysis - charring layer and their morphological structures
- Thermo gravimetric analysis (TGA) - thermal stability of coating.
- Bunsen burner
- Furnace
- Digital weighing machine
- Mixer
- Grinder
- Hand gloves, paper cups, spatula, plastic containers
- Data logger –AMS 850

CHAPTER 4: RESULTS AND DISCUSSIONS

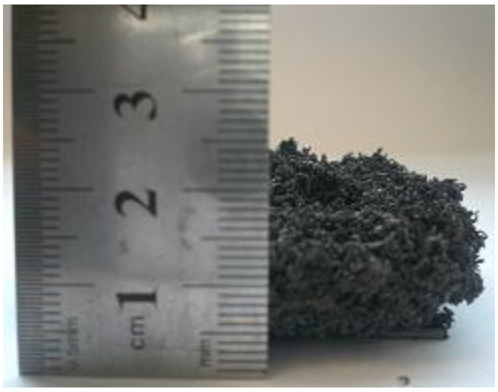
4.1 Measurement for coating with filler

Thickness of the coating onto the steel substrate is taken at four points on each side using venire caliper and the average reading is considered as the reading. Once the sample has gone through the furnace test, the expansion of char is measured by using a ruler. The measurement is marked at the highest point of the char expansion on each sample.

Measurement Technique: - **Sample taken from Formulation 5(2% of fillers ground)**

Average Thickness = (Thickness 1 + Thickness 2 + Thickness 3 + Thickness 4) / 4

Figure 11: Sample 5 of substrate with coating applied for 5cm X 5cm steel



Maximum Expansion = Peak expansion of Char

Figure 12: Sample 5 of char formed upon fire testing for substrate

4.2 Experiment Progress

Attached below are the images of the coatings successfully applied on each steel according to various formulations:

4.2.1 Samples with particle sizes of both fillers grounded:

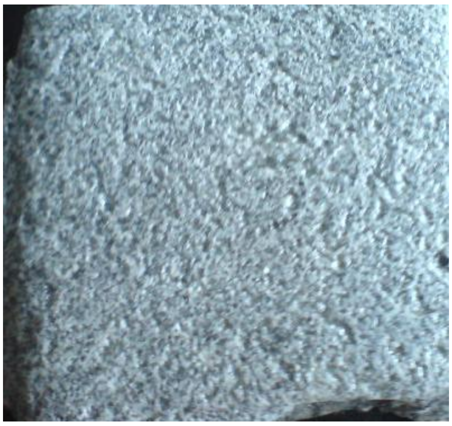


Figure 13: Coating applied for 1st formulation without fillers, (Sample 1)

Figure 14: Coating applied for 2nd formulation with fillers grounded (0.5 grams of ATH and fumed silica, Sample 2)

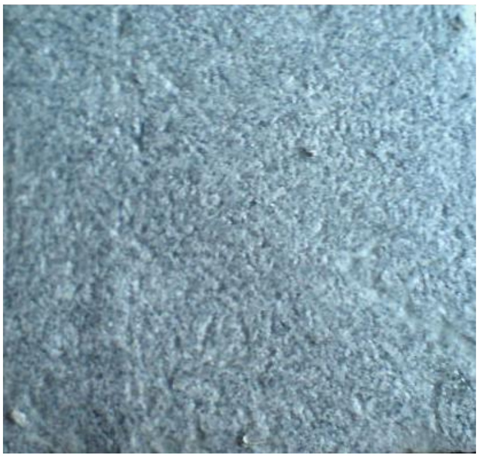


Figure 15: Coating applied for 3rd formulation with fillers grounded (1.0 gram of ATH and fumed silica, Sample 3)

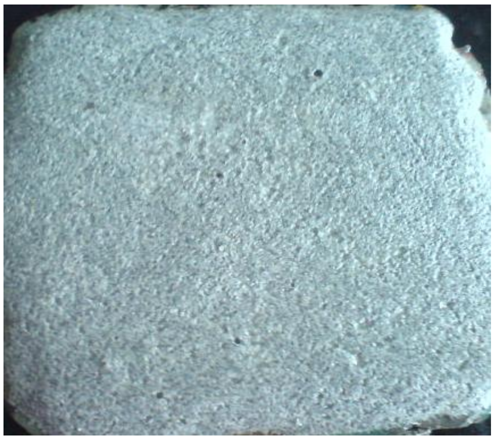


Figure 16: Coating applied for 4th formulation with fillers grounded (1.5 grams of ATH and fumed silica, Sample 4)

Figure 17: Coating applied for 5th formulation with fillers grounded (2.0 grams of ATH and fumed silica, Sample 5)

Figure 18: Coating applied for 6th formulation with fillers grounded (2.5 grams of ATH and fumed silica, Sample 6)

4.2.2Samples with particle sizes of both fillers without grounded:

Figure 19: Coating applied for 7th formulation with fillers not grounded (0.5 gram of ATH and fumed silica, Sample 7)

Figure 20: Coating applied for 8th formulation with fillers not grounded (1.0 gram of ATH and fumed silica, Sample 8)

Figure 21: Coating applied for 9th formulation with fillers not grounded (1.5 gram of ATH and fumed silica, Sample 9)

Figure 22: Coating applied for 10th formulation with fillers not grounded (2.0 gram of ATH and fumed silica, Sample 10)

Observations:

From the coating applied on the substrate, it is observed that the grounded fillers coatings are smoother. The surface roughness for non grounded fillers are high compared to the grounded. For both grounded fillers, as the percentage of ATH and Fumed Silica increases the surface roughness too increases. The hardness of the coating also increases as the amount of fillers increases. For non grounded filler, it was impossible/hard to apply the coating when the percentage of the fillers is increased to 2.5% as the coating formed doesn't stick on the steel. The curing time for the coating increases as the percentage of the fillers increases.

4.2.3.1 Furnace Test Results for ATH & Fumed Silica Substrates (Ground)

Table 3: Furnace Test Results for ATH & Fumed Silica Substrates (Ground) at 500 Celsius

Sample	Temperature	Detachability	Picture	Thickness (mm) Before	Thickness (mm) After	Expansion = Thickness (after) / Thickness (before)
Formulation 1: 0% ATH & Fumed Silica	**500 Degree Celsius**	**Detached**		2.14	15.2	7.10
Formulation 2: 0.5% ATH & Fumed Silica		**Detached**		2.43	16.25	6.69
Formulation 3: 1.0% ATH & Fumed Silica		**Detached**		2.33	20.35	8.73
Formulation 4: 1.5% ATH & Fumed Silica		**Detached**		2.65	23.61	8.91
Formulation 5: 2.0% ATH & Fumed Silica		**Detached**		2.70	24.88	9.21
Formulation 6: 2.5% ATH & Fumed Silica		**Detached**		3.28	24.68	7.52

4.2.3.2 Furnace Test Results for ATH & Fumed Silica Substrates (Not Ground)

Table 4: Furnace Test Results for ATH & Fumed Silica Substrates (Non Ground) at 500˚C

Sample	Temperature	Detachability	Picture	Thickness (mm)		Expansion
				Before	After	$= \frac{Thickness(after)}{Thickness(before)}$
Formulation 7: 0.5% ATH & Fumed Silica	500 Degree Celsius	Detached		3.10	25.23	8.14
Formulation 8: 1.0% ATH & Fumed Silica		Detached		3.18	30.03	9.43
Formulation 9: 1.5% ATH & Fumed Silica		Detached		3.73	32.98	8.84
Formulation 10: 2.0% ATH & Fumed Silica		Detached		3.55	30.35	8.55

$$\textbf{Char Expansion Calculation:} = \frac{\textbf{Thickness after (cm)}}{\textbf{Thickness before (cm)}}$$

$$\textbf{Example: Sample 1 Char Expansion} = \frac{\textbf{15.2}}{\textbf{2.14}}$$

$$= \textbf{7.10}$$

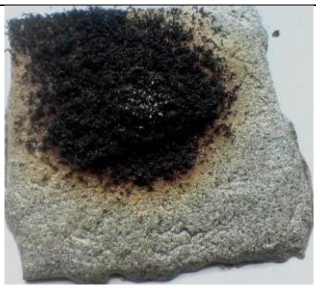
4.2.4 Bunsen Burner Test Results/Heat Shielding Results for ATH & Fumed Silica Substrates (Grounded)

Table 5: Bunsen Burner Test Results/Heat Shielding Results for ATH & Fumed Silica Substrate (Grounded)

Sample	Detachability	Picture	Thickness(mm) Before	Thickness(mm) After	Expansion = Thickness After / Before
Formulation 1: 0% ATH & Fumed Silica	Detachable		3.88	22.6	5.82
Formulation 2: 0.5% ATH & Fumed Silica	Detachable		4.10	24.3	5.93
Formulation 3: 1.0% ATH & Fumed Silica	Non Detachable		3.95	16.8	4.25
Formulation 4: 1.5% ATH & Fumed Silica	Detachable		3.15	19.8	6.29
Formulation 5: 2.0% ATH & Fumed Silica	Detachable		3.85	23.7	6.15
Formulation 6: 2.5% ATH & Fumed Silica	Non Detachable		3.78	18.4	4.87

4.2.5 Bunsen Burner Test Results/Heat Shielding Results for ATH & Fumed Silica Substrates (Non Grounded)

Table 6: Bunsen Burner Test Results/Heat Shielding Results for ATH & Fumed Silica Substrates (Non Grounded)

Sample	Note	Picture	Thickness(mm)		Expansion = Thickness After / Before
			Before	After	
Formulation 7: 0.5% ATH & Fumed Silica	Non Detachable		2.90	12.6	4.34
Formulation 8: 1.0% ATH & Fumed Silica	Non Detachable		3.10	15.7	5.06
Formulation 9: 1.5% ATH & Fumed Silica	Non Detachable		3.67	16.9	4.60
Formulation 10: 2.0% ATH & Fumed Silica	Non Detachable		3.50	13.30	3.80

4.2.6 Summary of Heat Shielding Effect Results

Sample 1	Time	Temperature(˚C)	Expansion	Final Temperature (˚C)
Formulation1 (0%ATH& Fumed Siica)	14.40	78	**5.82**	**86**
	14.50	122		
	15.00	108		
	15.15	105		
	15.30	86		

Sample 2	Time	Temperature(˚C)	Expansion	Final Temperature (˚C)
Formulation 2(0.5% ATH & Fumed Siica Grounded)	15.45	67	**5.93**	**77**
	15.55	79		
	16.05	122		
	16.20	98		
	16.35	77		

Sample 3	Time	Temperature(˚C)	Expansion	Final Temperature (˚C)
Formulation 3(1.0%ATH & Fumed Silica Grounded)	15.45	72	**4.25**	**92**
	15.55	88		
	16.05	127		
	16.20	112		
	16.35	92		

Sample 4	Time	Temperature(˚C)	Expansion	Final Temperature (˚C)
Formulation 4 (1.5% ATH & Fumed Siica Grounded)	15.43	82	**6.29**	**106**
	15.53	103		
	16.03	88		
	16.18	107		
	16.23	106		

Sample 5	Time	Temperature(˚C)	Expansion	Final Temperature (˚C)
Formulation 5 (2.0% ATH & Fumed Siica Grounded)	09.56	81	**6.15**	**64**
	10.06	120		
	10.16	71		
	10.31	61		
	10.46	64		

Sample 6	Time	Temperature(˚C)	Expansion	Final Temperature (˚C)
Formulation 6 (2.5% ATH & Fumed Siica Grounded)	09.56	71	**4.87**	**67**
	10.06	70		
	10.16	69		
	10.31	67		
	10.46	67		

Sample 7	Time	Temperature(˚C)	Expansion	Final Temperature (˚C)
Formulation 7 (0.5% ATH & Fumed Siica Non Grounded)	11.28	88	**4.34**	**78**
	11.38	116		
	11.48	102		
	11.58	82		
	12.08	78		

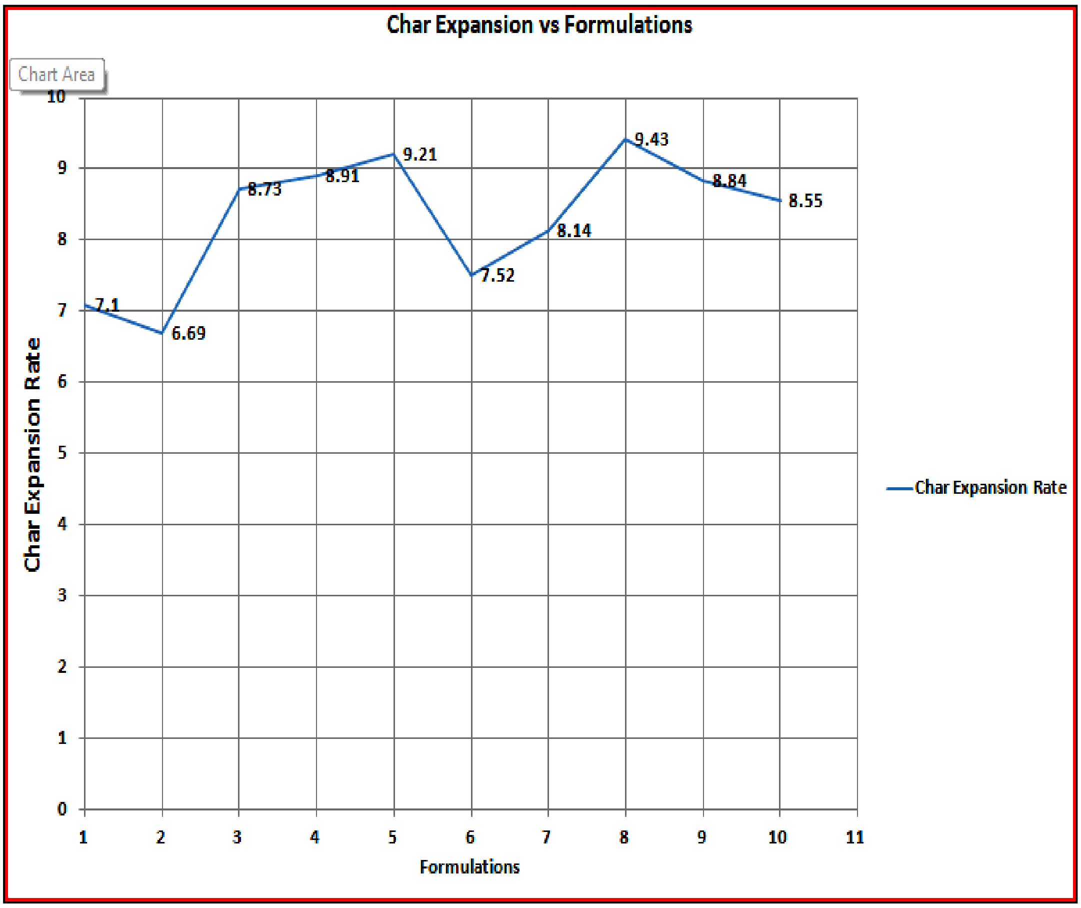
Sample 8	Time	Temperature(˚C)	Expansion	Final Temperature (˚C)
Formulation 8 (1.0% ATH & Fumed Silica Non Grounded)	15.06	76	**5.06**	**65**
	15.16	69		
	15.26	73		
	15.40	66		
	15.50	65		

Sample 9	Time	Temperature(˚C)	Expansion	Final Temperature (˚C)
Formulation (1.5% ATH & Fumed Siica Non Grounded)	15.45	70	**4.60**	**80**
	15.55	94		
	16.05	93		
	16.15	83		
	16.25	80		

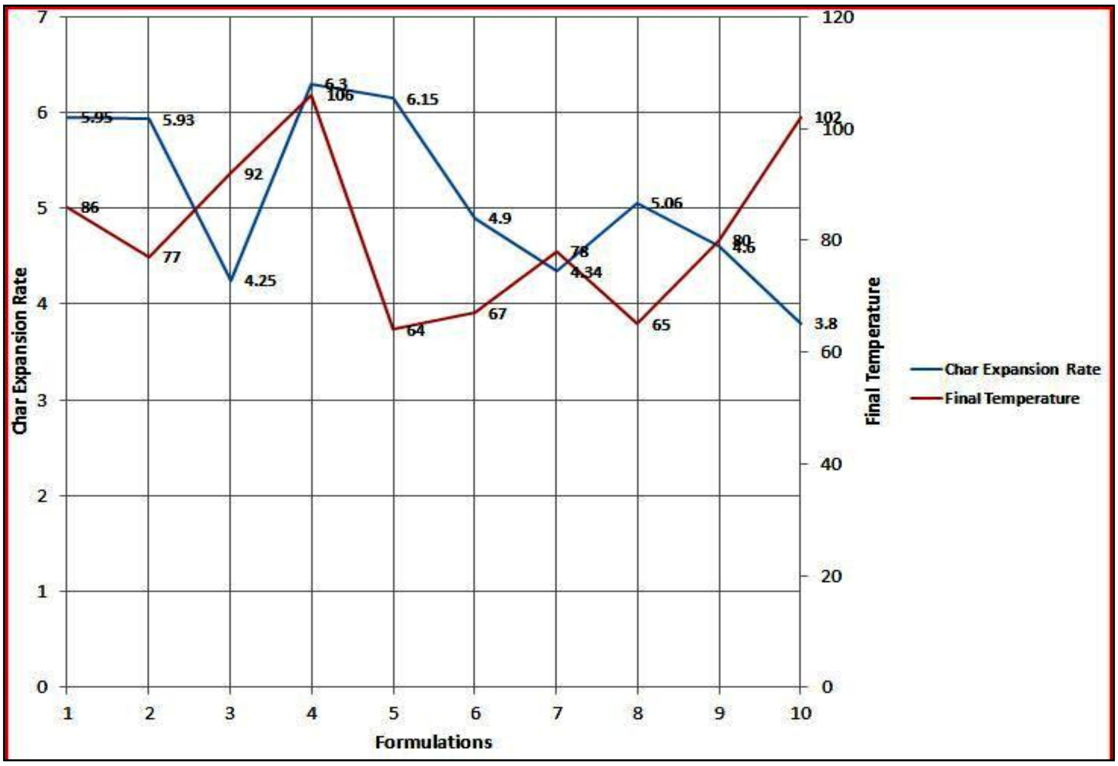
Sample 10	Time	Temperature(˚C)	Expansion	Final Temperature (˚C)
Formulation 10 (2.0% ATH & Fumed Siica Non Grounded)	16.00	44	**3.80**	**102**
	16.20	69		
	16.30	77		
	16.40	99		
	16.50	102		

The results obtained from Bunsen burner test is recorded using AMS 850 data logger and tabulated as above. From the results, it is observed the final temperature of the test range from 64˚C to 104˚C. The burning was done for approximately 40 to 50 minutes under room temperature with air suction is switched on to remove the toxic smell from the burning. All the samples shows increase in temperature for the first 20 to 25 minutes of the burning and later the temperature decreases once the char have been formed in the substrate.

Furnace Test Graph on Char Expansion Comparison:



Heat Shielding Effect Graph Comparison on Char Expansion and Final Temperature Comparison:



Discussion of Furnace test and Bunsen Burner test:

From the results tabulated and graph plotted above, it is sown that for furnace test formulation 1, formulation 4, formulation 5 and formulation 8 has the best char expansion rate. The reason of choosing these specific 4 formulations are for comparison in coherent with the project objective where 1 sample without filler (Formulation 1), 2 best samples from grounded fillers (Formulation 4&5) and 1best sample with fillers not grounded (Formulation 8) are selected. The char expansion for Bunsen burner test doesn't follows the results obtain from furnace test because of amount of coating applied on the substrate is not the same as the fillers increases in weight, the amount of coating also increases. Hence from the observation these 4 formulations are identified and are further analyzed with XRD and TGA method.

4.2.7 TGA Results

TGA result for Formulation 5 – 2.0% ATH and Fumed Silica grounded at 800˚C

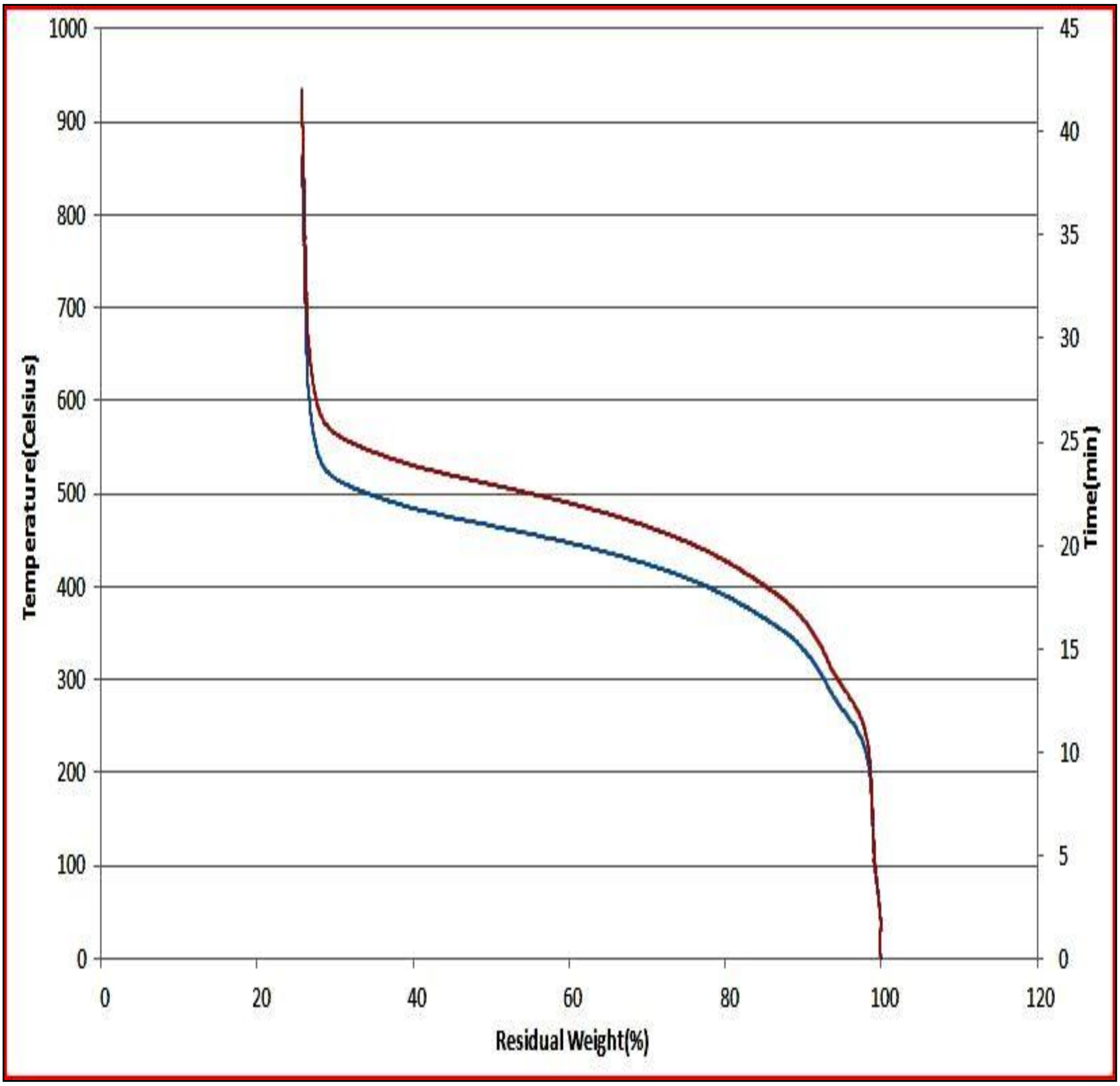


Figure 23: TGA result for Formulation 5 – 2.0% ATH and Fumed Silica grounded at 800˚C

Graph of Temperature (Celsius) vs. Residual Weight (%)

Graph of Time (min) vs. Residual Weight (%)

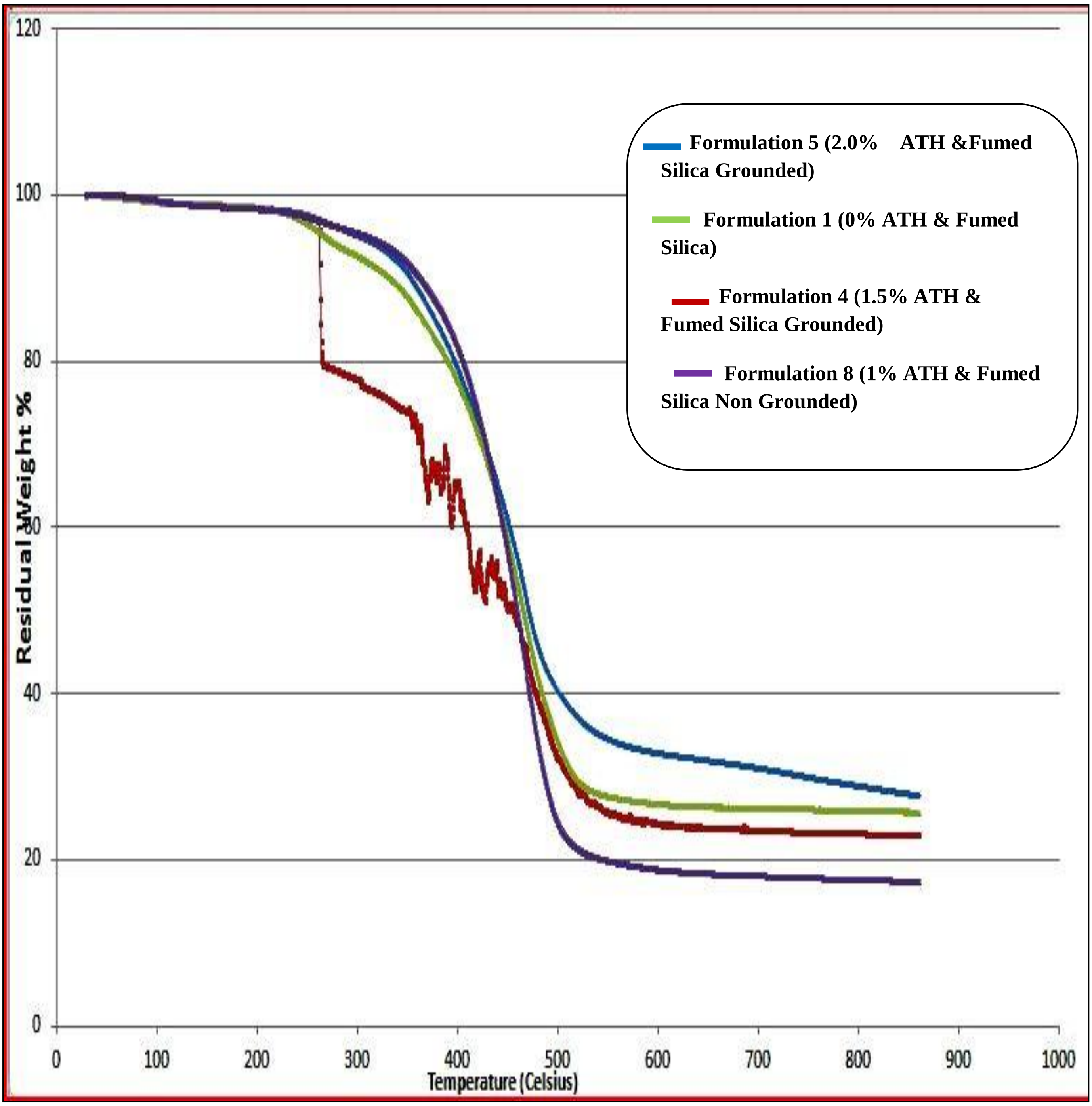


Figure 24: Combined graph of all TGA results with respect to residual weight (%) against Temperature ˚C

Summary of TGA results are tabulated in the table below :

Table 7: TGA Final Results

Formulation	Temperature(˚C)	Time (min)	Residual Weight left (%)
0% ATH & Fumed Silica	800˚C	42	22.443
1.5% ATH & Fumed Silica Grounded	800˚C	42	22.911
2.0% ATH & Fumed Silica Grounded	**800˚C**	**42**	**25.723**
1% ATH & Fumed Silica Non Grounded	800˚C	42	17.387

For thermo gravimetric analysis, the samples were taken from the coating applied where the excessive coating around the substrate are cut and collected in a plastic. TGA analysis was done at temperature of 800˚C for a period of 42 minutes for each sample. The results of each sample for TGA shows similar thermal degradation as the all the samples are composed of same materials to form the intumescent coating. However the residual weight increases as the percentage of the fillers increases. Besides that, the performance of the grounded formulation is better that the non grounded. From the graph shown below, Formulation 5 (2.0% ATH & Fumed Silica Grounded) has the maximum percentage of residual weight (25.723%) and thus gives the best intumescent effect compared to other formulations. The comparison among the graphs with respect to residual weight (%) can be seen in Figure 24.

Figure 25: Samples for TGA analysis

4.2.8 XRD Results

XRD result for Formulation 1 – 0% ATH and Fumed Silica at 500˚C

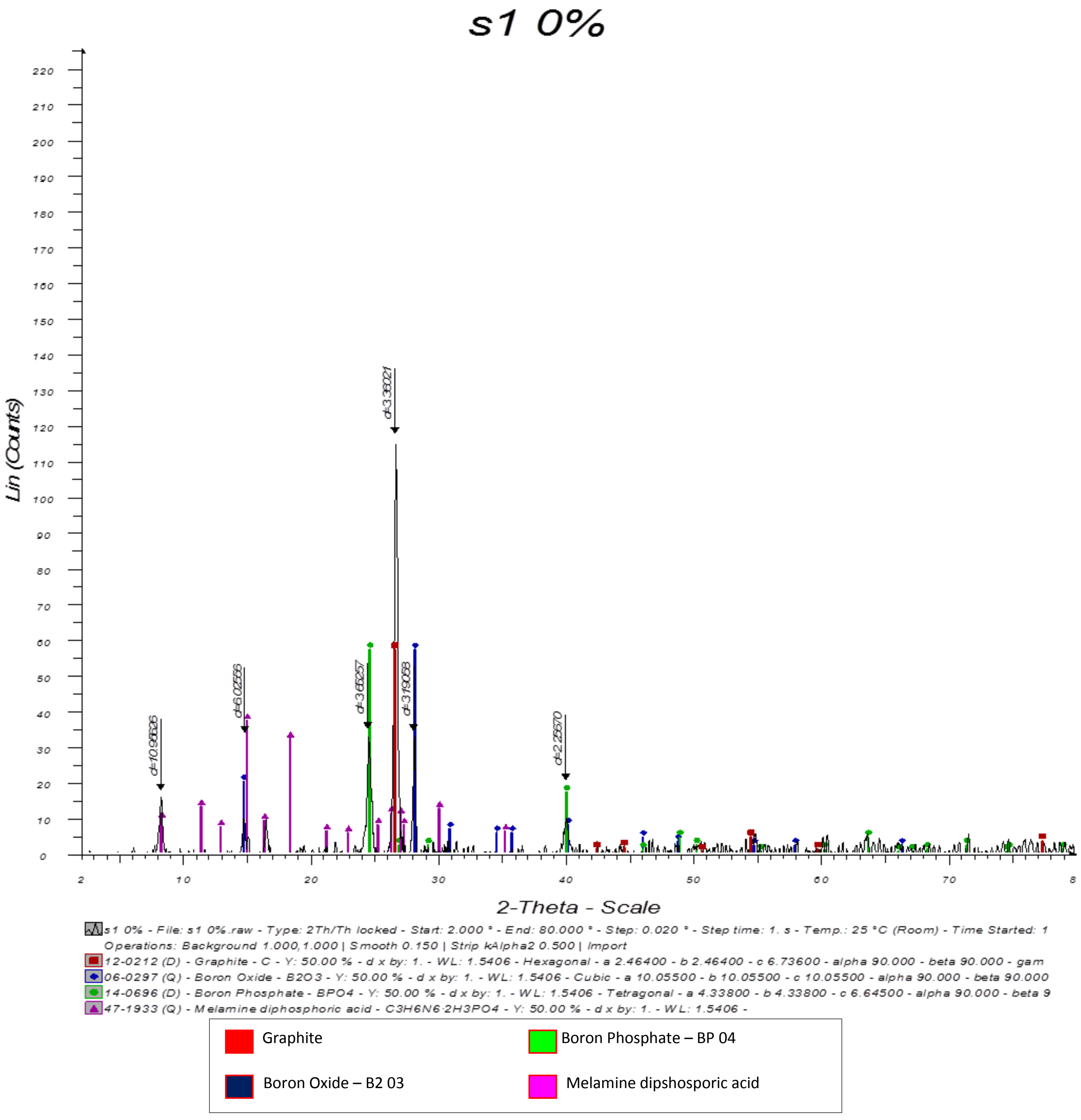


Figure 26: XRD curve for residue char for Formulation 1 – 0% ATH and Fumed Silica at 500 Celsius

XRD result for Formulation 4 – 1.5% ATH and Fumed Silica grounded at 500˚C

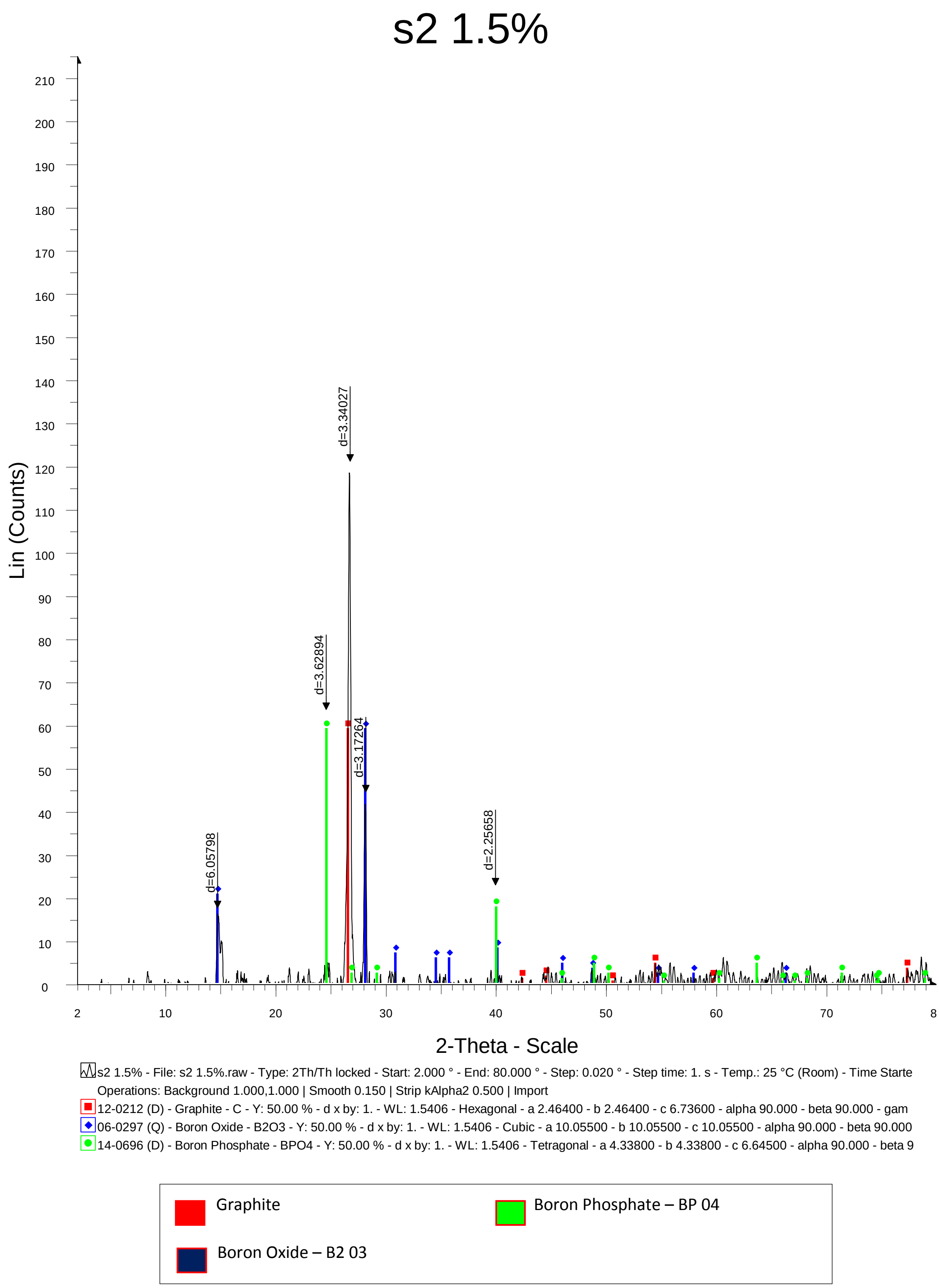


Figure 27: XRD curve for residue char for Formulation 4 – 1.5% ATH and Fumed Silica grounded at 500˚C

XRD result for Formulation 5 – 2.0% ATH and Fumed Silica grounded at 500˚C

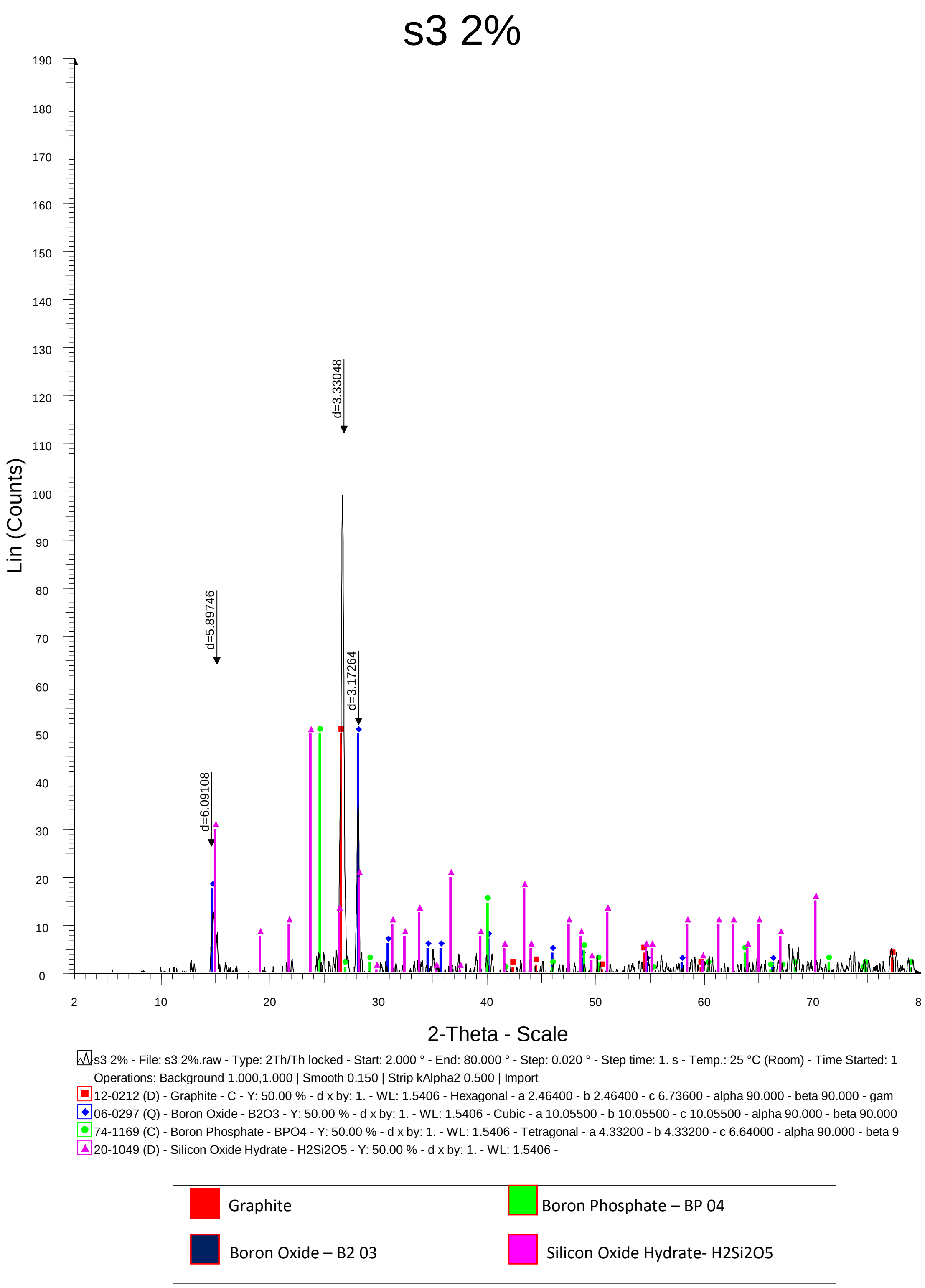


Figure 28: XRD curve for residue char for Formulation 5 – 2.0% ATH and Fumed Silica grounded at 500˚C

XRD result for Formulation 8 – 1.0% ATH and Fumed Silica non grounded at 500˚C

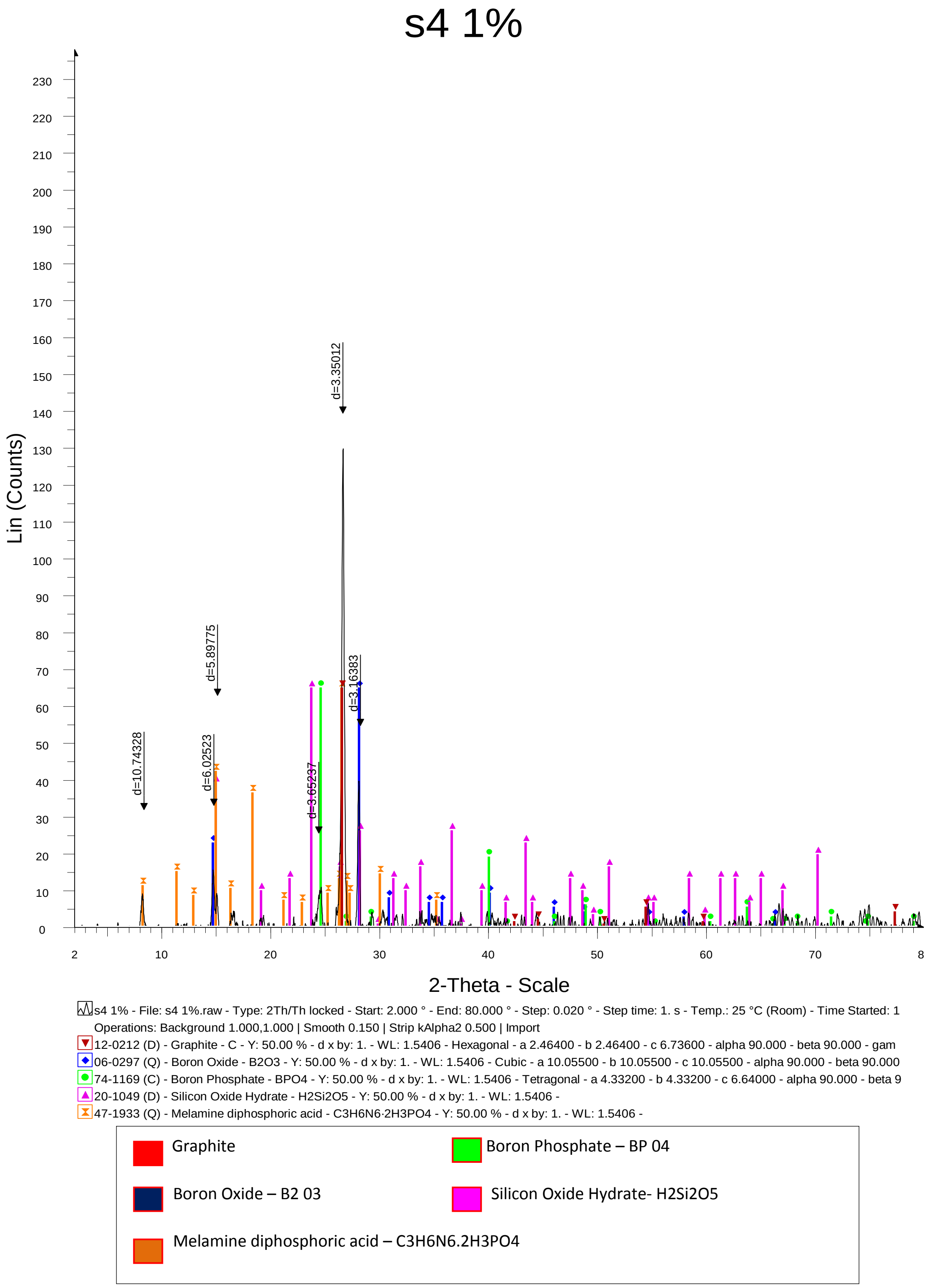


Figure 29: XRD curve for residue char for Formulation 8– 1.0% ATH and Fumed Silica non grounded at 500˚C

Summary of XRD Results:

XRD analysis was done to identify the compounds present in the char for chosen formulation samples. Each sample of the char are crushed into smooth powders and weighed to 10mg for the test. From the results obtained, every sample shows the present of graphite, boron oxide (B2O3) and boron phosphate (BPO4) and silicon oxide hydrate (H2Si2O5). Only sample 3, formulation 5 (2.0% ATH and fumed silica grounded) shows presence of silicon oxide hydrate. Besides, the last sample (1.0% ATH and fumed silica non grounded) shows the presence of Melamine diphosphoric acid – C3H6N6.2H3PO4. The presence of silicon oxide hydrate in sample 3 proves that the fillers in the coating have maximum char strength and fire protection properties. Besides that it is found that sample 3, S3 from formulation 5 gives the best result for XRD as the pattern of the line observed reflects the maximum compounds of the intumescent coating mixture.

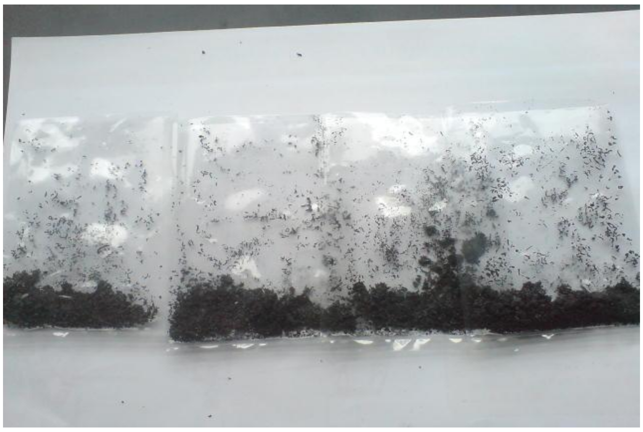


Figure 30: Char samples to be sent for XRD analysis

CHAPTER 5

5.1 CONCLUSION

This project has been successfully completed within the time frame given. Overall the objective and scope of study of this project have been met from the results and analysis obtained. As the study is based on particle sizes of inorganic fillers ATH & Fumed Silica, several factors must be taken into consideration such as wind, ambient temperature and climate as this will deviate in the final product of the coating. The tests done on the coatings are such as Furnace test, Bunsen burner test and analysis done are XRD and TGA analysis.

The manipulative factor for this project; the particle sizes of the fillers did affect the efficiency of the particle sizes as the grounded fillers are more resistance towards heat and produce better intumescent effect. The expansion of char from fire test, the final temperature absorbed from heat shielding effect, the residual weight (%) from TGA and morphological structure of the layer in XRD supports the initial theory that grounded particles gives better intumescent effect. Overall from the 10 formulations, in this study the author concludes that formulation 5 (2.0% ATH and Fumed Silica) in grounded composition yields the best intumescent effect out of all the other formulations and was proved in subsequent Fire Test, XRD and TGA analysis.

5.2 Recommendations

Among the recommendations to improve this study are to carry out the experiment using different inorganic fillers. Improvisation on increasing the heat shielding effect so that it could be used not just onto steel products but also other metal products that degrades at different temperature. Besides that, scanning electron microscope (SEM) can be done to study the charring layer and morphological structure in order to obtain more precise and accurate results.

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APPENDIX A

Samples Preparation and Fire Test:

Coatings are applied to steels as per the formulations. Each formulations coating are applied for 4 small steel (5X5) and 1 large steel (10X10). It is then let for natural drying under room temperature for 1 month.

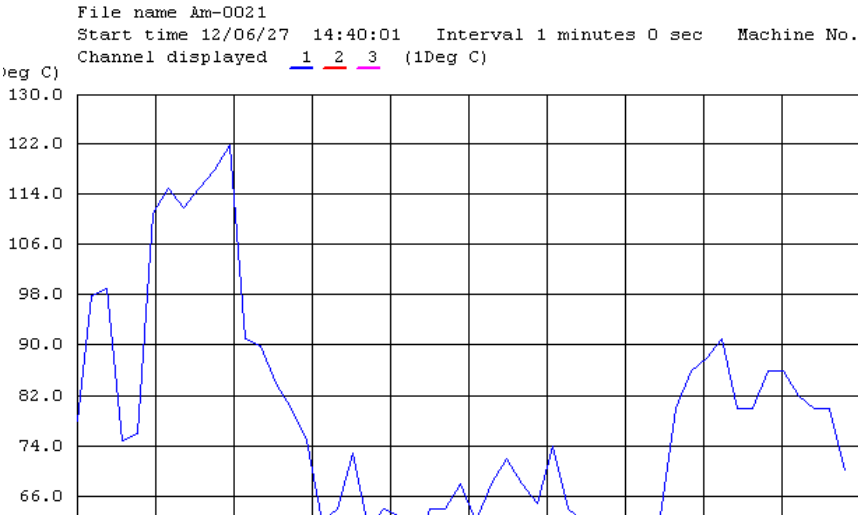
Samples arranged in tray and will be placed under furnace for 7 hours for burning and dwelling. The air suction is ensured switched on to remove the flame produced from the burning.

Samples obtained from furnace test. As can see, all the samples forms char expansion. From each formulation, 1 small sample is chosen and is tested under furnace.

APPENDIX B

Heat shielding effect results:

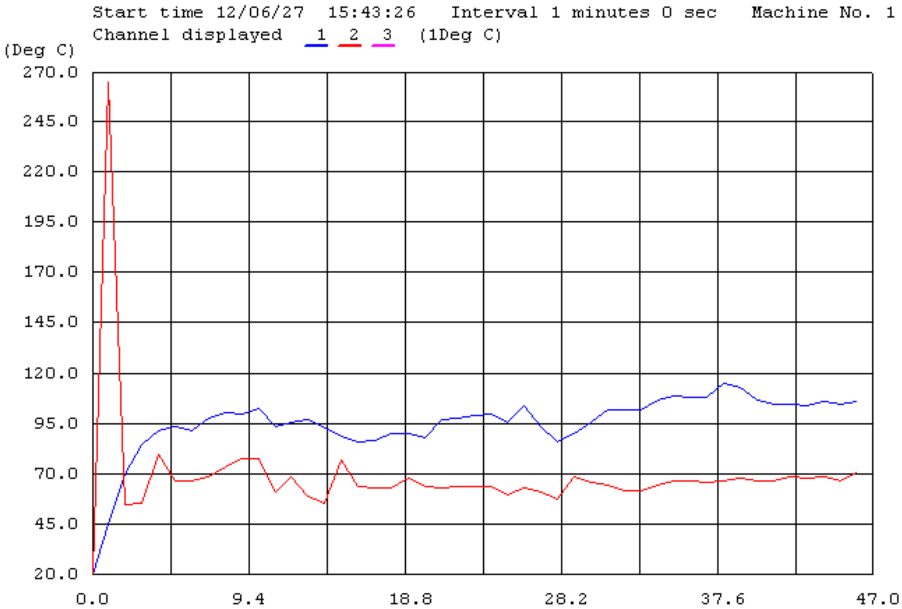
Results for Formulation 1 (0% ATH & Fumed Silica Grounded)



Start time 12/06/27 14:40:01 Interval .

SampleNo.	H: M: S	1ch.[C]	2ch.[C]	3ch.[C]
000001	00:00:00	78	BOUT	BOUT
000002	00:01:00	98	BOUT	BOUT
000003	00:02:00	99	BOUT	BOUT
000004	00:03:00	75	BOUT	BOUT
000005	00:04:00	76	BOUT	BOUT
000006	00:05:00	111	BOUT	BOUT
000007	00:06:00	115	BOUT	BOUT
000008	00:07:00	112	BOUT	BOUT
000009	00:08:00	115	BOUT	BOUT
000010	00:09:00	118	BOUT	BOUT
000011	00:10:00	122	BOUT	BOUT
000012	00:11:00	91	BOUT	BOUT
000013	00:12:00	90	BOUT	BOUT
000014	00:13:00	84	BOUT	BOUT
000015	00:14:00	80	BOUT	BOUT
000016	00:15:00	75	BOUT	BOUT
000017	00:16:00	62	BOUT	BOUT
000018	00:17:00	64	BOUT	BOUT
000019	00:18:00	73	BOUT	BOUT
000020	00:19:00	61	BOUT	BOUT
000021	00:20:00	64	BOUT	BOUT
000022	00:21:00	63	BOUT	BOUT
000023	00:22:00	57	BOUT	BOUT
000024	00:23:00	64	BOUT	BOUT
000025	00:24:00	64	BOUT	BOUT
000026	00:25:00	68	BOUT	BOUT
000027	00:26:00	62	BOUT	BOUT
000028	00:27:00	68	BOUT	BOUT
000029	00:28:00	72	BOUT	BOUT
000030	00:29:00	68	BOUT	BOUT
000031	00:30:00	65	BOUT	BOUT
000032	00:31:00	74	BOUT	BOUT
000033	00:32:00	64	BOUT	BOUT
000034	00:33:00	62	BOUT	BOUT
000035	00:34:00	59	BOUT	BOUT
000036	00:35:00	60	BOUT	BOUT
000037	00:36:00	62	BOUT	BOUT
000038	00:37:00	60	BOUT	BOUT
000039	00:38:00	63	BOUT	BOUT
000040	00:39:00	80	BOUT	BOUT
000041	00:40:00	86	BOUT	BOUT
000042	00:41:00	88	BOUT	BOUT
000043	00:42:00	91	BOUT	BOUT
000044	00:43:00	80	BOUT	BOUT
000045	00:44:00	80	BOUT	BOUT
000046	00:45:00	86	BOUT	BOUT
000047	00:46:00	86	BOUT	BOUT
000048	00:47:00	82	BOUT	BOUT
000049	00:48:00	80	BOUT	BOUT
000050	00:49:00	80	BOUT	BOUT
000051	00:50:00	70	BOUT	BOUT

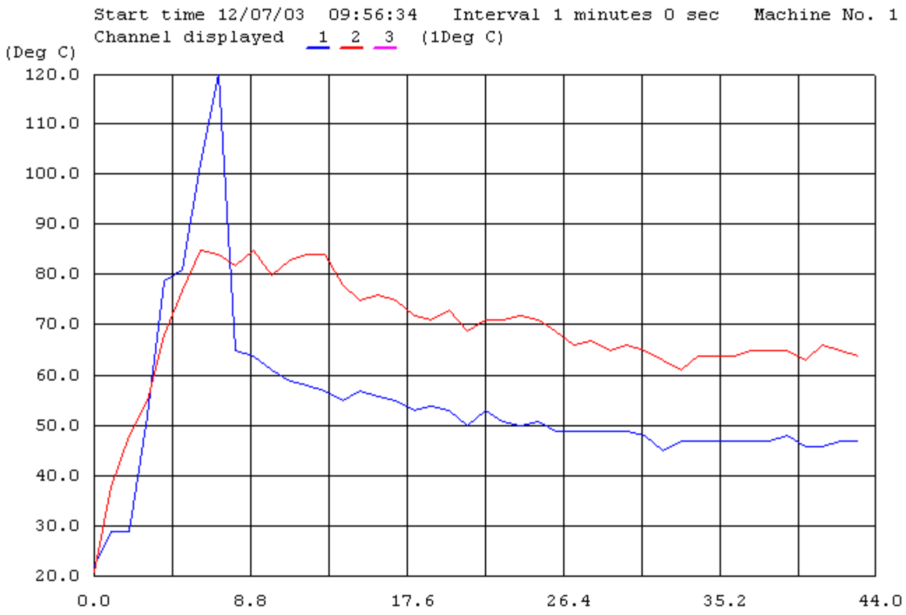
Results for Formulation 4 (1.5% ATH & Fumed Silica Grounded)



Start time 12/06/27 15:43:26 Interval

SampleNo.	H: M: S	1ch.[C]	2ch.[C]	3ch.[C]
000001	00:00:00	20	20	BOUT
000002	00:01:00	45	265	BOUT
000003	00:02:00	70	55	BOUT
000004	00:03:00	85	56	BOUT
000005	00:04:00	92	80	BOUT
000006	00:05:00	94	67	BOUT
000007	00:06:00	92	67	BOUT
000008	00:07:00	98	69	BOUT
000009	00:08:00	101	74	BOUT
000010	00:09:00	100	78	BOUT
000011	00:10:00	103	78	BOUT
000012	00:11:00	94	61	BOUT
000013	00:12:00	96	69	BOUT
000014	00:13:00	97	59	BOUT
000015	00:14:00	93	56	BOUT
000016	00:15:00	89	77	BOUT
000017	00:16:00	86	64	BOUT
000018	00:17:00	87	63	BOUT
000019	00:18:00	90	63	BOUT
000020	00:19:00	90	68	BOUT
000021	00:20:00	88	64	BOUT
000022	00:21:00	97	63	BOUT
000023	00:22:00	98	64	BOUT
000024	00:23:00	99	64	BOUT
000025	00:24:00	100	64	BOUT
000026	00:25:00	96	60	BOUT
000027	00:26:00	104	63	BOUT
000028	00:27:00	94	61	BOUT
000029	00:28:00	86	58	BOUT
000030	00:29:00	90	69	BOUT
000031	00:30:00	96	66	BOUT
000032	00:31:00	102	65	BOUT
000033	00:32:00	102	62	BOUT
000034	00:33:00	102	62	BOUT
000035	00:34:00	107	65	BOUT
000036	00:35:00	109	67	BOUT
000037	00:36:00	108	67	BOUT
000038	00:37:00	108	66	BOUT
000039	00:38:00	115	67	BOUT
000040	00:39:00	113	68	BOUT
000041	00:40:00	107	67	BOUT
000042	00:41:00	105	67	BOUT
000043	00:42:00	105	69	BOUT
000044	00:43:00	104	68	BOUT
000045	00:44:00	106	69	BOUT
000046	00:45:00	105	67	BOUT
000047	00:46:00	106	71	BOUT

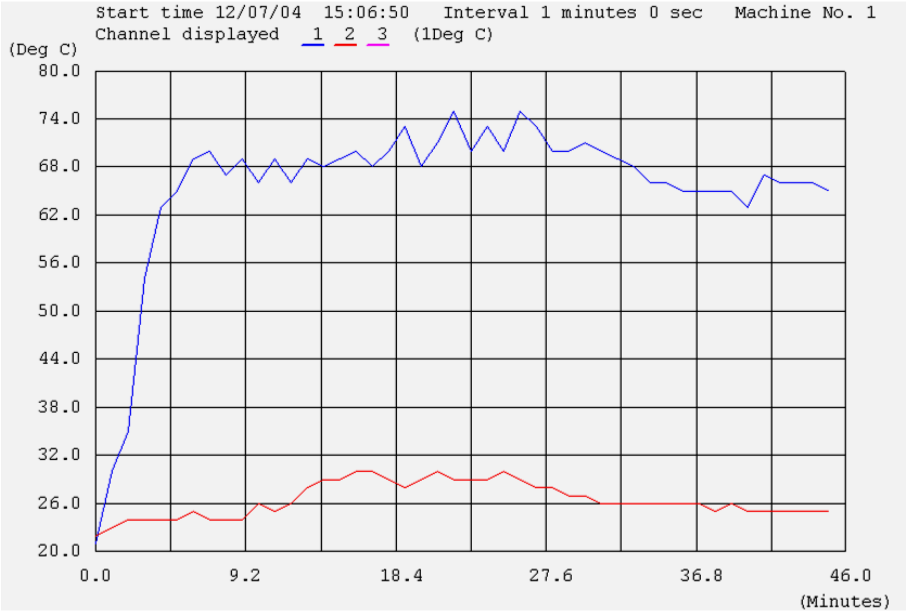
Results for Formulation 5 (2.0% ATH & Fumed Silica Grounded)



Start time 12/07/03 09:56:34 Interval 1 m

SampleNo.	H: M: S	1ch.[C]	2ch.[C]	3ch.[C]
000001	00:00:00	22	21	BOUT
000002	00:01:00	29	38	BOUT
000003	00:02:00	29	48	BOUT
000004	00:03:00	52	55	BOUT
000005	00:04:00	79	68	BOUT
000006	00:05:00	81	77	BOUT
000007	00:06:00	102	85	BOUT
000008	00:07:00	120	84	BOUT
000009	00:08:00	65	82	BOUT
000010	00:09:00	64	85	BOUT
000011	00:10:00	61	80	BOUT
000012	00:11:00	59	83	BOUT
000013	00:12:00	58	84	BOUT
000014	00:13:00	57	84	BOUT
000015	00:14:00	55	78	BOUT
000016	00:15:00	57	75	BOUT
000017	00:16:00	56	76	BOUT
000018	00:17:00	55	75	BOUT
000019	00:18:00	53	72	BOUT
000020	00:19:00	54	71	BOUT
000021	00:20:00	53	73	BOUT
000022	00:21:00	50	69	BOUT
000023	00:22:00	53	71	BOUT
000024	00:23:00	51	71	BOUT
000025	00:24:00	50	72	BOUT
000026	00:25:00	51	71	BOUT
000027	00:26:00	49	69	BOUT
000028	00:27:00	49	66	BOUT
000029	00:28:00	49	67	BOUT
000030	00:29:00	49	65	BOUT
000031	00:30:00	49	66	BOUT
000032	00:31:00	48	65	BOUT
000033	00:32:00	45	63	BOUT
000034	00:33:00	47	61	BOUT
000035	00:34:00	47	64	BOUT
000036	00:35:00	47	64	BOUT
000037	00:36:00	47	64	BOUT
000038	00:37:00	47	65	BOUT
000039	00:38:00	47	65	BOUT
000040	00:39:00	48	65	BOUT
000041	00:40:00	46	63	BOUT
000042	00:41:00	46	66	BOUT
000043	00:42:00	47	65	BOUT
000044	00:43:00	47	64	BOUT

Results for Formulation 8 (1.0% ATH & Fumed Silica Non Grounded)



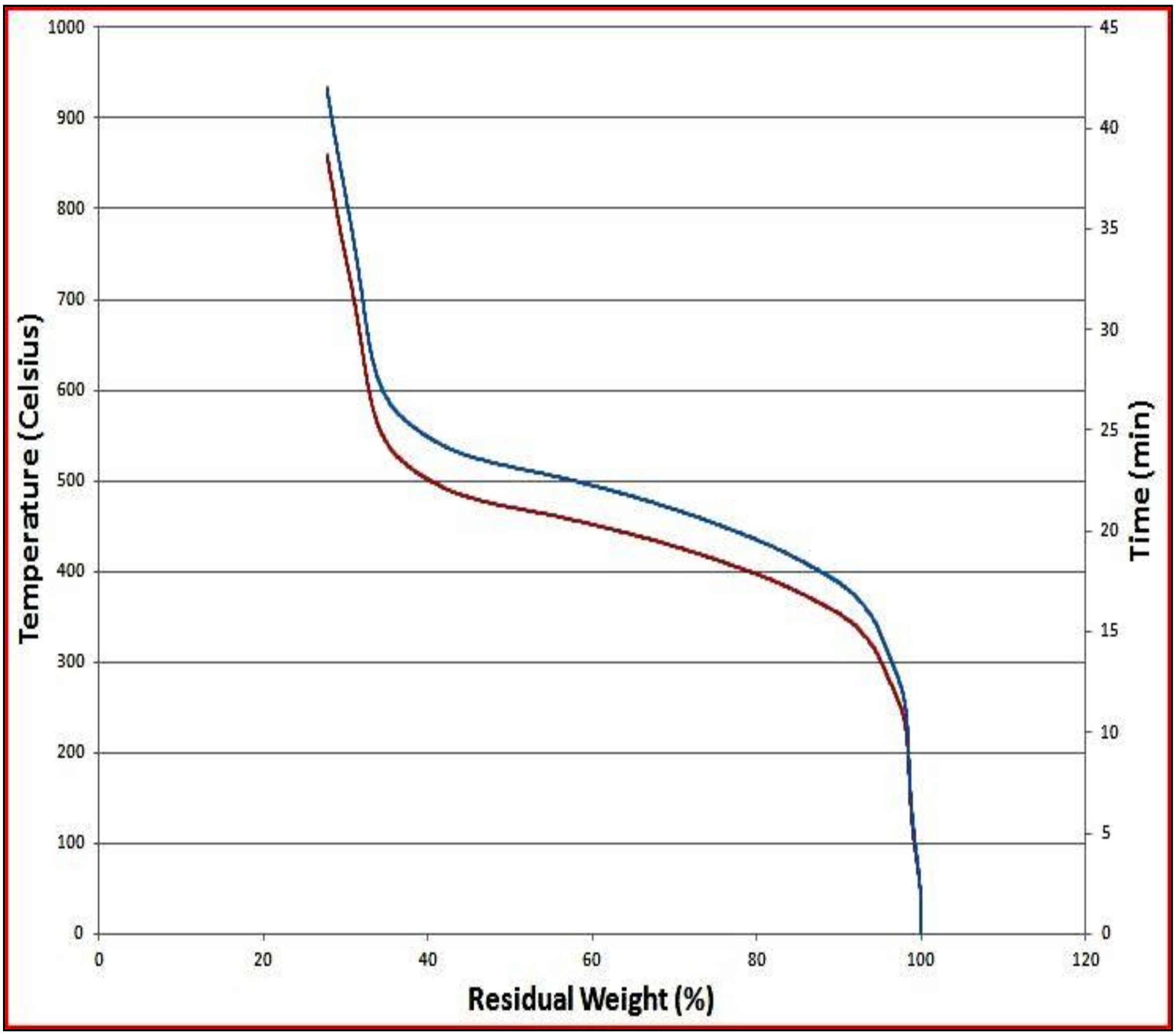
Start time 12/07/04 15:06:50 Interval 1

SampleNo.	H: M: S	1ch.[C]	2ch.[C]	3ch.[C]
000001	00:00:00	21	22	BOUT
000002	00:01:00	30	23	BOUT
000003	00:02:00	35	24	BOUT
000004	00:03:00	54	24	BOUT
000005	00:04:00	63	24	BOUT
000006	00:05:00	65	24	BOUT
000007	00:06:00	69	25	BOUT
000008	00:07:00	70	24	BOUT
000009	00:08:00	67	24	BOUT
000010	00:09:00	69	24	BOUT
000011	00:10:00	66	26	BOUT
000012	00:11:00	69	25	BOUT
000013	00:12:00	66	26	BOUT
000014	00:13:00	69	28	BOUT
000015	00:14:00	68	29	BOUT
000016	00:15:00	69	29	BOUT
000017	00:16:00	70	30	BOUT
000018	00:17:00	68	30	BOUT
000019	00:18:00	70	29	BOUT
000020	00:19:00	73	28	BOUT
000021	00:20:00	68	29	BOUT
000022	00:21:00	71	30	BOUT
000023	00:22:00	75	29	BOUT
000024	00:23:00	70	29	BOUT
000025	00:24:00	73	29	BOUT
000026	00:25:00	70	30	BOUT
000027	00:26:00	75	29	BOUT
000028	00:27:00	73	28	BOUT
000029	00:28:00	70	28	BOUT
000030	00:29:00	70	27	BOUT
000031	00:30:00	71	27	BOUT
000032	00:31:00	70	26	BOUT
000033	00:32:00	69	26	BOUT
000034	00:33:00	68	26	BOUT
000035	00:34:00	66	26	BOUT
000036	00:35:00	66	26	BOUT
000037	00:36:00	65	26	BOUT
000038	00:37:00	65	26	BOUT
000039	00:38:00	65	25	BOUT
000040	00:39:00	65	26	BOUT
000041	00:40:00	63	25	BOUT
000042	00:41:00	67	25	BOUT
000043	00:42:00	66	25	BOUT
000044	00:43:00	66	25	BOUT
000045	00:44:00	66	25	BOUT
000046	00:45:00	65	25	BOUT

APPENDIX C

TGA Results:

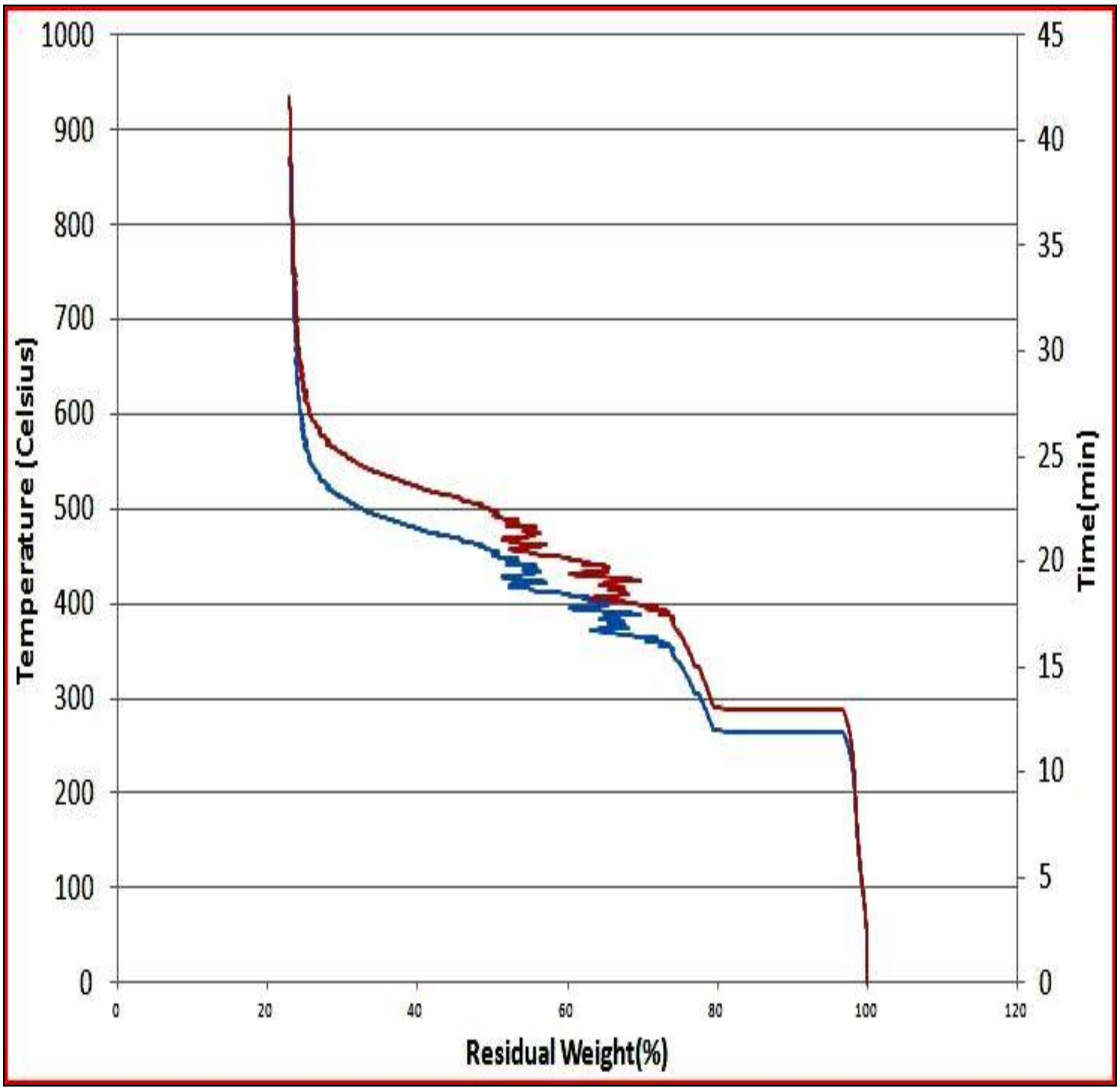
TGA result for Formulation 1 – 0% ATH and Fumed Silica at 800˚C



Graph of Temperature (Celsius) vs. Residual Weight (%)

Graph of Time (min) vs. Residual Weight (%)

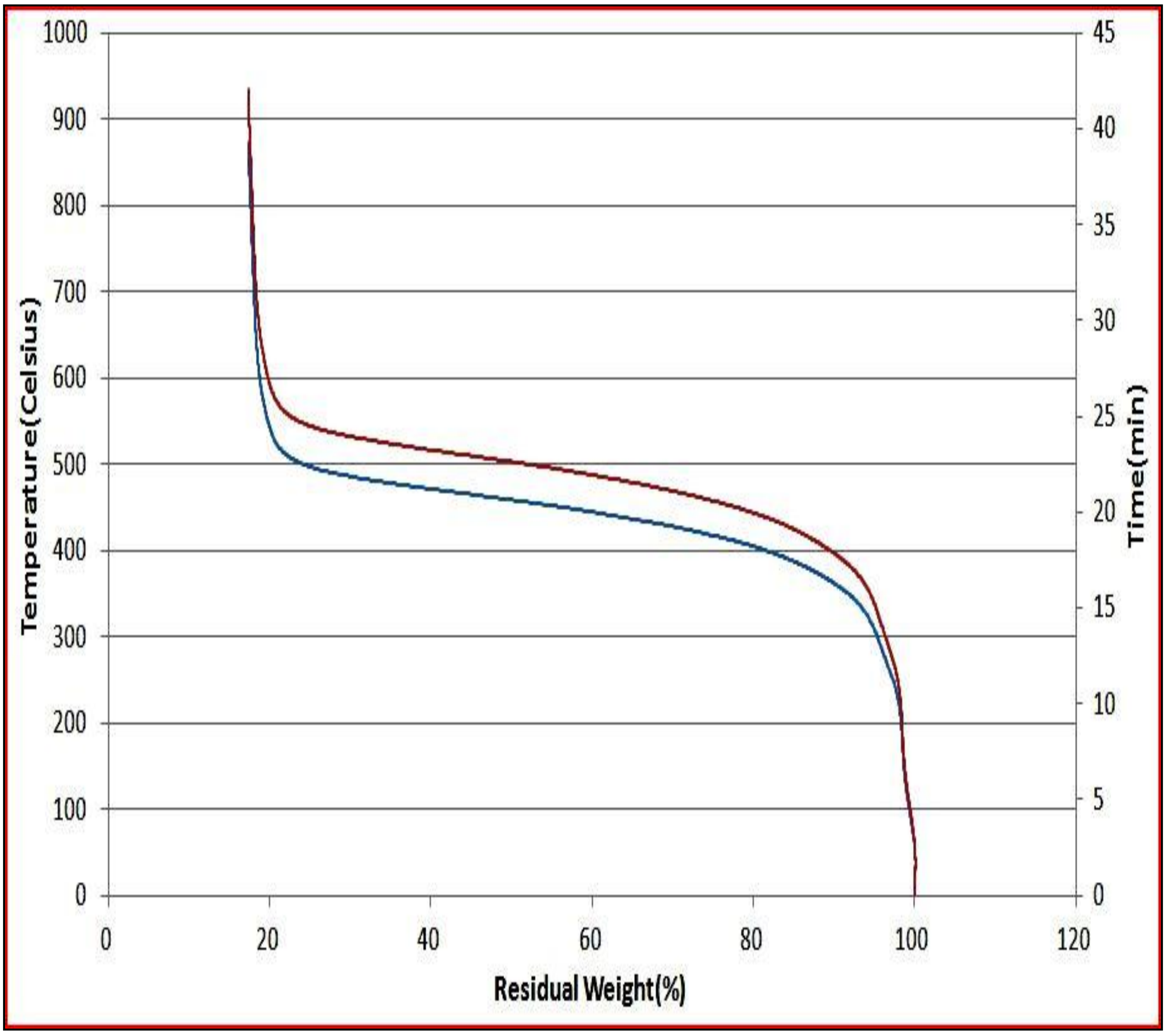
TGA result for Formulation 4 – 1.5% ATH and Fumed Silica grounded at 800˚C



Graph of Temperature (Celsius) vs. Residual Weight (%)

Graph of Time (min) vs. Residual Weight (%)

TGA result for Formulation 8 – 1.0% ATH and Fumed Silica non grounded at 800˚C



Graph of Temperature (Celsius) vs. Residual Weight (%)

Graph of Time (min) vs. Residual Weight (%)