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Evaluation of Fire-Retardant Properties of Emulsion, Text-Coat and Gloss Paints Modified with Bio-Based Extract of Acalypha Wilkesiana

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Abstract. The impact of fire on concrete buildings has been found to tend towards strength reduction of the concrete building. Fire-retardant paints help to reduce the impact of fire on buildings and many researches are on to improve on fire-retardant paints. To this end, this paper investigated the fire-retardant properties of the bio-based extract of Acalypha wilkesiana in emulsion, text-coat and gloss paints. Acalypha leaves were subject to extraction in water and kerosene for three days. Emulsion, text-coat and gloss paints were produced. The fire point of the paint samples with a varying mass of extract was obtained to compare the flammability and obtain the optimum mass of extract needed for the best performance. The cube coated with 2 layers of text-coat paint of 0.45 g yielded the highest compressive strength. The best performing type of paint was text-coat paint. The coarse sand added during the production of text-coat paint assists in protecting the substrate material. Emulsion paint was the next best performing paint type while gloss paint was not advisable to be used for this purpose. The introduction of Acalypha wilkesiana as an additive had the fire point of the sample increased alongside the ignition time and the optimum mass of Acalypha wilkesiana extract that yielded the best fire-retardation of the paint was found to be 0.45g.

1. Introduction

As indicated in authentic historical sources, the Chinese and the Egyptians used vinegar and alum as their fire-retardant agents which were initially known as combustion limiting agents for wood and natural polymers [1]. The Chinese applied a mixture of the alum and vinegar to wood and afterwards, an upper layer of clay would be added [2]. This reduced and delayed the rate of fire spread. About 3000 years ago, in Egypt, reed and grass which have previously been soaked in seawater would be used for roofing purposes [3]. Dried mineral salts which have crystallized are also used as fire-retardants [4]. Generally, the description of fire-retardants can be backdated to ancient Roman times by Aulus Gellius [5]. Wood was observed by Sulla in 86 BC to withstand fire during the siege in Piraeus. This was because the wood had previously been soaked in alum [6]. In 1821, the first attempt to highlight the scientific principles of fire retardation was made by Gay-Lussac, who proposed several composition recipes for making fire-retardants for cellulose materials from ammonium phosphate and borax. These recipes are still in use to date [6].

In general, fire-retardants are applied as follows: 65% for plastics, 25% for rubber (including aluminium trihydrate in carpet underlay), 5% for textiles, 3% for coatings/adhesives and only 2% for wood and paper [7]. However, over the years' fire-retardants have been used majorly on buildings and construction. Fire-retardant materials are important due to the increase in fire losses over the years [8].

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Fire-retardants have become commonly used in buildings to reduce the risks associated with fire outbreaks [9]. The suitability of these materials can be assessed by carrying out standard tests on each sample by exposing it to a single fire scenario which helps decide the end-use of the material [10]. These tests have been useful in the past but new methods are being developed to improve the quality of the materials. The new methods require the use of mathematical models of ignition and fire spread to fully understand how a particular material will perform under several fire scenarios [11], [12].

Due to the consequent increase in operating pressure and temperature of several processes, more efficient fire retardation properties are required. Environmentalists have pressurized the European market into looking for diphenyl oxide-free fire-retardant systems. Thus, a new generation of multipurpose environmentally-friendly (bio-based) brominated fire-retardants has been recently introduced which offers additional benefits. The purpose of the new methods developed is to create a new generation of fire-retardant materials based on a deeper understanding of the combustion process to produce materials to meet increasingly inflexible requirements that will demand improved fire safety in the future [13].

In the last decade, technology has drifted from halogen-based fire-retardants and towards those based on phosphorus, silicon, metal hydrates, other metal salts and especially bio-friendly materials. This trend is likely to continue as long as halogenated fire-retardants still pose a hazard to the environment. This explains why a lot of research is being carried out on alternatives to halogenated fire-retardants.

Based on the authors' exhaustive search, plant extract Acalypha wilkesiana has not been used as fire-retardants in paints. The aim of this study was to investigate the fire-retardant properties of the bio-based extract of Acalypha wilkesiana in emulsion, text-coat and gloss paints.

2. Methodology

2.1. Raw Materials and Reagents

The raw materials used in this research were leaves of Acalypha wilkenasia (shown in Fig. 1), cement, gravel, cube moulds, water, kerosene and the reagents employed are emulsion (biocide, titanium dioxide, calcium carbonate, polyvinyl acrylic), text-coat (titanium dioxide, Polyvinyl Acrylic (PVA), nitrosol, biocide, pigment, calcium carbonate), and gloss (alkyd resins, pigment and drier).



Fig. 1. Acalypha wilkesiana leaves

2.2. Equipment and production

The equipment used to carry out different tests is cup flash-point tester, drying oven and mass balance. The general production process of paints is as represented in Fig. 2.

2.2.1. Extraction with Water

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The steps used for the extraction with water are discussed in this section. The Acalypha plant samples were collected and air-dried for seven days. The dried plants were grounded into powdered form with a mortar and pestle. 100 g of the powdered leaf was weighed into four 250 ml conical flasks containing 250 ml of distilled water. The mixture was then covered and stirred every 24 h using a glass sterile glass rod for 3 days. The mixture was then filtered through filter paper. The resulting yellowish-green filtrates were then concentrated and refrigerated at 40°C before testing.

2.2.2. Extraction with Kerosene

The steps used for the extraction with water are discussed in this section. The Acalypha plant samples were collected and air-dried for seven days. The dried plants were grounded into powdered form with a mortar and pestle. 100 g of the powdered leaf was weighed into four 250 ml conical flasks containing 250 ml of distilled water. The mixture was then covered and stirred every 24 h using a glass sterile glass rod for 3 days. The mixture was then filtered through filter paper. The resulting yellowish-green filtrates were then concentrated and refrigerated at 40°C before testing.

2.2.3. Procedure for the paint production

i. For the production of the emulsion paint, 2L of water, titanium dioxide was added with calgon and mixed for 15 minutes in a large container. Calcium carbonate was added and stirred properly for about 15 minutes to prevent the formation of lumps in the pigments. The mix properties for the run one are shown in Table 1.

RUN ONE: EMULSION PAINT PRODUCTION

- i. The chemicals were measured accurately on a weighing scale (for powdery chemicals) and graduated containers (for liquids).
- ii. 2L water was poured into an empty container. Titanium dioxide was added and tinge with calgon.
- iii. The pigment powders were added into the container and mixed for about 10-15mins.
- iv. The required amount of Acrylic was added to the mixture and stirred well. Nitrosol that had been mixed with water was added and stirred rigorously and uniformly for 5-10mins.
- v. Formalin was then be added and the solution was completely stirred
- vi. The plant extract of the Acalypha species was then added in varying masses between 0.0-`3.0g.
- vii. The solution of emulsion paint produced was then applied on the various substrate surfaces.

Table 1. Mix properties of paint for run one

INGREDIENTS	1	2	3	4	5	6	7	8
Water (L)	2L							
Calgon (Kg)	0.01kg							
Calcium carbonate (Kg)	17kg							
Polyvinyl Acrylic (P.V.A) (Kg)	1.20kg							
Nitrosol (Kg)	0.13kg							
Formalin (cL)	5cL							
Pigment (Red Oxide) (Kg)	0.25kg							
Acalypha Extract (g)	0.00g	0.30g	0.45g	0.60g	1.00g	1.25g	1.50g	3.00g

The mix properties of paint for the run two is related in the table 2.

RUN TWO: TEXTCOAT PAINT PRODUCTION

- i. The chemicals were measured accurately on a weighing scale (for powdery chemicals) and graduated containers (for liquids).
- ii. 2L water was poured into an empty container. Titanium dioxide was added and tinge with calgon.
- iii. The pigment powders were added into the container and mixed for about 10-15mins.
- iv. Calcium carbonate was added and stirred properly for about 10-15 mins to prevent the formation of chumps in the pigments.
- v. The required amount of Acrylic was added to the mixture and stirred well. Nitrosol which had been mixed with water was added and stirred rigorously and uniformly for 5-10mins.
- vi. Formalin was then be added and the solution was completely stirred
- vii. Marble dust was then added to the mixture and stirred thoroughly.
- viii. The plant extract of the Acalypha species was then added in varying masses between 0.0-3.0g.
- ix. The solution of textcoat paint was then applied on the various substrate surfaces **Table 2.** Mix properties of paint for run two

INGREDIENTS	1	2	3	4	5	6	7	8
Water Calgon	2L 0.01kg							
Calcium carbonate	17kg							
Polyvinyl Acrylic (P.V.A)	1.20kg							
Nitrosol	0.13kg							
Formalin Pigment (Red Oxide)	5cL 0.25kg							
Acalypha Extract	0.00g	0.30g	0.45g	0.60g	1.00g	1.25g	1.50g	3.00g

The mix properties of paint for the run three is related in the table 3 and the mix properties of Paint for the run four is related in the table4..

EXPERIMENT RUN THREE: GLOSS PAINT

i. The bucket was filled with kerosene and mixed with alkyd resin.

- ii. The titanium dioxide was then mixed with the solution.
- iii. The dispersant (soya lecithin) was added. The solution was then properly mixed.
- iv. Pigment was then added to the perfectly mixed mixture.
- v. The Acalypha plant extracted with kerosene was added in varying masses between 0.0-3.0g and the solution is completely stirred
- vi. The solution of gloss paint was then applied on the various substrate surfaces
- viii. Samples are allowed to dry completely for several days prior to analysis.

Table 3. Mix properties of paint for run three

INGREDIENTS	1	2	3	4	5	6	7	8
Water Calgon	2L 0.01kg							
Calcium carbonate	17kg							
Polyvinyl Acrylic (P.V.A)	1.20kg							
Nitrosol	0.13kg							
Formalin Pigment (Red Oxide)	5cL 0.25kg							
Acalypha Extract	0.00g	0.30g	0.45g	0.60g	1.00g	1.25g	1.50g	3.00g

The mix properties of paint for the run three is related in the table 4.

Table 4. Mix properties of paint for run four

INGREDIENTS	1	2	3	4	5	6	7	8
Alkyd Resin	400g							
Titanium Dioxide	160g							
Dispersant (Soya Licithin)	1.1kg							
Kerosene	70g							
Pigment (Yellow Oxide)	0.25kg							
Acalypha Extract (with kerosene)	0.0g	0.3g	0.45g	0.60g	1.00g	1.25g	1.50g	3.00g

2.3. Casting of Concrete Cubes

In the casting of concrete cubes, 1:2:4 proportioning was employed as mix-ration. Water was gradually added to the mortar while the mixture was properly mixed before it was transferred into mould dimension of $100 \text{mm} \times 100 \text{mm} \times 100 \text{mm}$ for the casting. Slump test was carried out on the fresh concrete as shown in Fig. 2. The concretes were de-moulded after 24 h, cured in water for 3 days and then sun-dried for 4 h as shown in Fig. 3 and Fig. 4.





Fig. 2. Slump test

Fig. 3. Cubes curing in water

Fig. 4. Sun-drying of cubes

2.4. Application of Paint on Concrete Cubes

The application of paint on the concrete cubes was carried out in two phases. The first phase was the application of a layer of the emulsion, text-coat and gloss paint on the concrete cubes while the second phase was the application of two layers of all the three produced paint on the concrete cubes as shown in Fig. 5.





Fig. 5. Coated concrete cubes

2.5. Analysis and Testing

2.5.1. Fire Point Test

The paint sample of equal mass was placed in the cup flash point tester as shown in Fig. 6. The flashpoint tester was turned and the temperature of the sample flashed was noted for comparative analysis. The varying masses of gloss paint samples extract were filled to the mark in the cup flash point tester. The samples were analysed to test for the effectiveness of the fire-retardant properties of Acalypha wilkesiana.



Fig. 6. Paint sample at fire point

2.5.2. Mass Loss Analysis

The cube samples were weighed before and after heating. The initial and the final masses were recorded. This was done for all the samples.

3. Results and Discussion

3.1. Raw Materials and Reagents

Fire Point The fire point of the samples as shown in Fig. 7 increased from 480°C to 650°C and reaches its peak at 980°C for a mass of extract of 0.00 g, 0.30 g, and 0.45 g respectively. At a higher mass of extract, the fire point begins to decrease steadily from 980°C to 820°C and it decreased till it reached 500°C. This indicates that the optimum mass of extract needed for effective fire retardation is 0.45 g to achieve a fire point of 980°C. This indicates Acalypha wilkesiana possesses fire-retardant properties by increasing the fire point of the paint thus delaying ignition time. However, an increasing amount of extract in the sample beyond the optimum mass required would cause the paint to fail and reduce fire point. This may be due to the high chloride concentration in Acalypha wilkesiana leaves. From research, a threshold chloride content of atmospheric service is 46 ppm Cl (0.046 g/L of Cl) [14]. The optimum mass of plant extract in the paint that yielded the best measure of performance (fire retardation) was 0.45 g (150 mL of extract) which contains 532.5 mg of Cl.

Research has shown that there is a high surface concentration of chlorides resulting in the permeation into porous coatings [15]. Chloride in extremely high concentration penetrates concrete by diffusion and absorption of the fluid containing the chloride. Chlorides absorb moisture from the air, through the coating through osmosis and thus reduces the corrosion cell resistance of the coating. This elevates the chloride levels at the coating-substrate interface. Hence, resulting in premature coating failure and corrosion. Salts in high concentration would be uniformly distributed on the substrate surface and therefore result in uniform visual corrosion. It is known that high levels of contaminants like chloride or sodium affect the quality or duration of coating performance because they induce stress corrosion cracking [16-19]. This explains why the effectiveness of the fire-retardant coatings would reduce when the extract is added beyond the optimum required mass (0.45 g). Since the threshold limit of chloride depends on the desired protection life of the coating system and the coating thickness, it is advisable to maintain the mass of extract in the paint to 0.45 g and the coating thickness to 2 layers.

Table 5. Summarised result of fire point analysis on the gloss paint sample

Mass of extract (g)	Fire point (°C)
0.00	48
0.30	65
0.45	98
0.60	82
0.80	70
1.00	68
1.25	58
1.50	54
3.00	50

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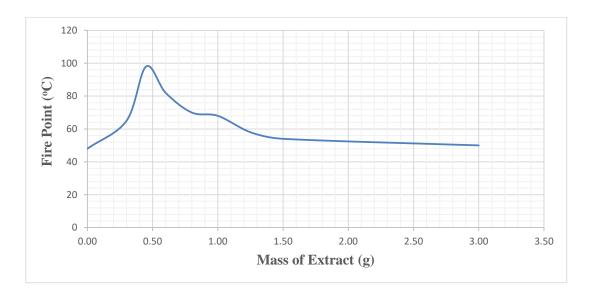


Fig 7. Fire point against the mass of extract

3.2. Mass Loss Test

The results of the mass loss test carried out on the coated concrete are shown in Table 6. The plots for the samples coated with emulsion paint, text-coat paint and gloss paint are related in Fig. 8, 9, and 10 respectively, while Fig. 11 relates the summary of the three different paints.

Table 6. Summary of residual mass analysis

Mass of	Residual Mass (g)											
Extract	Text-co	at			Emulsi	on			Gloss			
(g)	1 Layer		2 Layer	rs	1 Layer	r	2 Layer	rs	1 Layer	ŗ	2 Layers	
· C	250°C	350°C	250°C	350°C	250°C	350°C	250°C	350°C	250°C	350°C	250°C	350°C
Control	9.0											
0.00	8.6	8.4	8.5	8.5	8.5	8.4	8.6	8.5	8.5	8.3	8.4	8.2
0.30	8.7	8.8	8.8	8.8	8.6	8.7	8.8	8.8	8.7	8.4	8.5	8.3
0.45	8.9	8.9	8.9	8.9	8.9	8.8	8.9	8.9	8.8	8.5	8.6	8.4
0.60	8.6	8.8	8.7	8.8	8.8	8.6	8.8	8.6	8.7	8.3	8.5	8.3

A comparative analysis of the residual mass of the emulsion paints with the varying mass of extract after being subjected to 250°C and 350°C for 1 h is shown in Fig. 8. It was observed that the cubes heated at 350°C had lower residual masses than cubes heated at 250°C. From Fig. 8, it was observed that the least mass loss was obtained by the concrete cubes coated with 2 layers of emulsion coating at 250°C. The next best performing concrete cubes were those coated with 2 layers of emulsion coating at 350°C followed by the cubes coated with 1 layer at 250°C and lastly by the cubes coated with 1 layer of emulsion coating and subjected to 350°C. However, for each sample, the residual mass decreased from an initial mass of 9.0 g to the least mass obtained at 0.3 g of extract and increased to its peak at 0.45 g of extract and decreased at 0.60 g of extract. The higher the mass of extract added to the coating, the

higher the residual mass of concrete cubes after heating. This shows that the emulsion paint protects the substrate from heat and mass loss and the measure of performance is dependent on the number of layers of paint used, the mass of extract in the paint and the temperature to which they are subjected.

The various residual mass of concrete cubes coated with text-coat paint containing a varying mass of extract before and after being subjected to heat is related in Fig. 9. It was observed that the best performing cubes with the least mass loss were those coated with 2 layers of text-coat paint at 250°C, followed by those coated with 1 layer of text-coat paint at 250°C, followed by those coated with 2 layers of text-coat paint and heated at 350°C. However, for each sample, the residual mass decreased from an initial mass of 9.0 g to the least mass obtained at 0.3 g of extract and increased to its peak at 0.45 g of extract and decreased at 0.60 g of extract. The higher the mass of extract added to the coating, the higher the residual mass of concrete cubes after heating. This shows that text-coat paint protects the substrate from heat and mass loss and the measure of performance is dependent on the number of layers of paint used, the mass of extract in the paint and the temperature to which they are subjected.

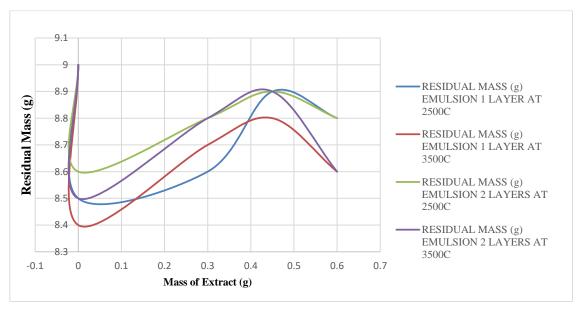


Fig. 8. Residual mass against the mass of extract in emulsion paint

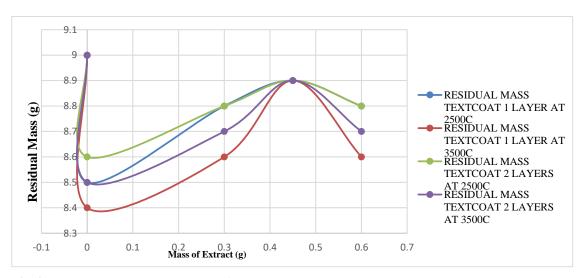


Fig. 9. Residual mass against the mass of extract in text-coat paint

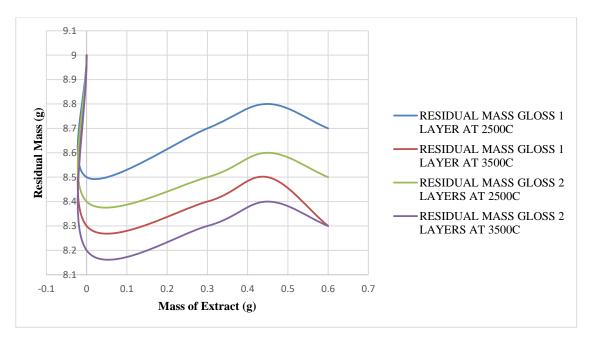


Fig. 10. Residual mass against the mass of extract in gloss paint

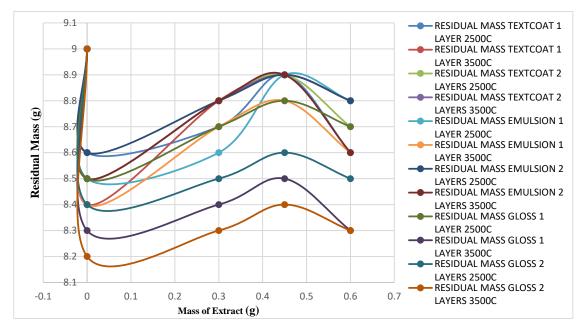


Fig. 11. Residual mass (g) against the mass of extract (g)

Fig. 11 shows the various residual mass of concrete cubes coated with gloss paint containing a varying mass of extract before and after being subjected to heat. It was observed that the best performing cubes with the least mass loss were those coated with 1 layer of gloss paint at 250°C, followed by those coated with 2 layers of gloss paint and heated at 250°C and the least residual mass was obtained by cubes coated with 2 layers of gloss paint and heated at 350°C. However, for each sample, the residual mass decreased from an initial mass of 9.0 g to the least mass obtained at 0.3 g of extract and increased to its peak at 0.45 g of extract and decreased at 0.60 g of extract. The higher the mass of extract added to the coating, the higher the residual mass of concrete cubes after heating. This shows that gloss paint protects the substrate from heat and mass loss and the measure of performance is dependent on the number of layers of paint used, the mass of extract in the paint and the temperature to which they are subjected to.

The residual mass of all the cubes coated with the various paint types with a varying mass of extract before and after subjecting it to 250°C or 350°C is related in Fig. 6. This analysis is done to compare the behaviour of the coating types and select the coating type with the best performance. The cubes coated with 2 layers of text-coat paint showed the least mass loss before and after heating both at 250°C and 350°C and the cubes coated with gloss paint showed the most mass loss before and after heating especially at a higher temperature of 350°C.

3.3. Compressive Strength Analysis

The compressive strength test results carried out for the concrete cubes coated both in one layer and in two layers are related in Table 7, 8, and 9 respectively.

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Table 7. Comparison of compressive strengths of cubes coated with one layer of the various paint samples

Mass of	Compressive Strengths (kN)										
Extract (g)	Text-coat	1 layer	Emulsion	l layer	Gloss 1 layer						
	250°C	350°C	250°C	350°C	250°C	350°C					
Control	450										
0.00	230	210	240	180	170	130					
0.30	270	230	300	230	230	200					
0.45	330	320	350	310	290	280					
0.60	300	280	290	220	200	190					

Table 8. Comparison of compressive strengths of cubes coated with two layers of various paint samples

Mass of Extract (g)	of	Compressive Strengths (kN)									
		Text-coat 2	2 layers	Emulsion 2	2 layers	Gloss 2 lay	Gloss 2 layers				
		250°C	350°C	250°C	350°C	250°C	350°C				
Control		450									
0.00		450	450	450	450	450	450				
0.30		240	240	240	240	240	240				
0.45		250	250	250	250	250	250				
0.60		350	350	350	350	350	350				

Table 9. Summary of compressive strength analysis

Mass of	Compressive Strength (KN)												
Extract	Text-co	oat			Emulsi	Emulsion				Gloss			
(g)	1 Layer	Layer 2 Layers		1 Layer	1 Layer		2 Layers		1 Layer		`S		
	250°C	350°C	250°C	350°C	250°C	350°C	250°C	350°C	250°C	350°C	250°C	350°C	
Control	450												
0.00	230	210	240	250	240	180	280	220	170	130	180	150	
0.30	270	230	350	300	300	230	330	280	230	200	210	180	
0.45	330	320	400	370	350	310	370	300	290	280	230	200	
0.60	300	280	340	290	290	220	320	270	200	190	190	160	

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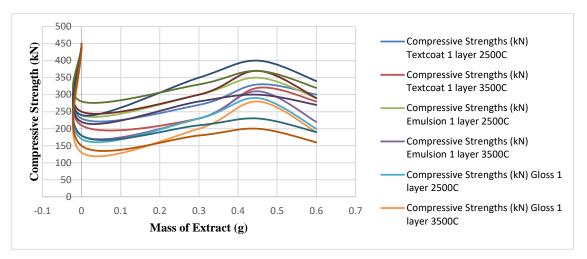


Fig. 12. Compressive Strength against Mass of Extract for all samples

In Fig. 12 a comparative analysis of the compressive strength value obtained from the different cubes coated with different layers of the different types of paint containing a varied mass of extract after being subjected to 250°C and 350°C for 1 h. From Fig. 12, it was observed that the best compressive strength was obtained by the concrete cubes coated with 2 layers of text-coat coating at 250°C. The next best performing concrete cubes were those coated with 2 layers of emulsion coating at 250°C followed by the cubes coated with 1 layer of emulsion heated at 250°C, followed by cubes coated with 1 layer of emulsion heated at 350°C, followed by cubes coated with 2 layers of emulsion heated at 350°C, followed by the cube coated with 1 layer of gloss and heated at 250°C, followed by the cube coated with 1 layer of gloss and heated at 350°C, followed by the cube coated with 2 layers of gloss and heated at 250°C and lastly by the cubes coated with 2 layers of gloss coating and subjected to 350°C. However, for each sample, the compressive strengths decreased from an initial compressive strength of 450 kN to a lower compressive strength value attained at 0.3 g of extract and increased to its peak at 0.45 g of extract and decreased at 0.60 g of extract. This shows that the emulsion paint, as well as the text-coat paint, effectively protect the substrate from heat and the gloss paints are not effective fire-retardant paint.

It was observed from Table 9, that the least compressive values are obtained from the cubes coated with gloss paint. The higher the number of layers, the lower the compressive strength. It was also observed that the compressive strengths of the cubes coated with emulsion were better than those coated with gloss paint. This shows that emulsion paint is a better performing fire-retardant paint than gloss paint. It was also observed that the compressive strengths of the cubes coated with 2 layers were better performing except those coated with gloss paint. It can also be deduced from Table 9 that the cubes with the best compressive strength after exposure to heat were those coated with text-coat paint. It can also be deduced that those coated with 2 layers were better and those heated at 250°C than those heated at 350°C.

This shows that the emulsion paint, as well as the text-coat paint, effectively protect the substrate from direct contact with the heat thus reducing mass loss as well as from compressive strength. The measure of performance is dependent on the number of layers of paint used, the mass of extract in the paint and the temperature to which they are subjected to. However, the gloss paints are not effective fire-retardant paint. It is suspected that this is due to the kerosene contained in the paint which may also add as a fuel for the heating process. Thus leading to a high amount of concrete degradation.

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4. Conclusion

A study on the effectiveness of fire-retardant paint on building materials with a varied mass of extract, number of layers of coating applied on the substrate, and the temperature of heating was carried out in this research work. The findings made are related as follows:

- 1. The introduction of Acalypha wilkesiana as an additive in paint increases the fire point of the sample; thus increasing the ignition time.
- 2. The optimum mass of Acalypha wilkesiana extract required to yield the best fire-retardation of the paint is 0.45g.
- 3. The best performing type of paint is text-coat paint. The coarse sand added during the production of text-coat paint assists in protecting the substrate material. Emulsion paint is the next best performing paint type. Gloss paint is not advisable to be used for this purpose.
- 4. The substrate material is effectively protected by increasing the number of layers of coating added. The best performing analysis was coated with 2 layers of text-coat paint. This does not apply to gloss paint because of the kerosene contained in the paint which contributes to the fire load of the material.
- 5. Concrete performs better when exposed to low temperature. The mass loss was higher and the compressive strengths of the samples heated at 350°C were lower than those heated at 250°C. Hence, the higher the temperature, the more damage is done to the substrate material.

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