RICE HUSK ASH SILICATE COMPOSITE AS FLAME RETARDING AGENT IN DEHYDRATED CASTOR SEED OIL ALKYD RESIN PAINT COATING

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Abstract: Flame retardant gloss paint coatings were prepared using medium oil length alkyd resin from dehydrated castor seed oil while superfine micro silica particles were used as flame retardant agent. The silica particles of mean size 13.43 µm were extracted from rice husk agricultural waste using the sol-gel method and were added to the paint coating at the rate of 2.5, 5.0, 7.5 and 10.0% of the total mass. All the flame retardant paints exhibited excellent paint properties such as drying time, specific gravity and colour. They all have flash point above 100^0C and retard flame spread. The vertical burning test (modified UL94-V test) which gives the response of the samples in normal room conditions on application of flame at 20 seconds revealed that the silicate composite paint coating exhibited flame retardant property. The limiting oxygen index (LOI) of the silicate composite dehydrated castor oil alkyd resin paints (SCDCOAP) all have values that are above 21.0 and the vertical burning time of the paint coatings also confirmed the flame retardancy of the silicate composite paint coatings. It was generally observed that the flame retardancy of the samples increased with an increase in the superfine silica compositions.

Keywords: Physico-chemical properties, Alkyd resins, flame retardant, limiting oxygen index

Introduction

Fire incidents have been widely reported to have caused great economic lost to man. In some cases irreparable injuries were inflicted on the victims and in some severe accidents, lives were lost. Man inquest for safety from this life threatening incident devised several measures for preventing and mitigating the effects of fire disasters. One of such measures is the use of flame retardant materials in buildings, household utensils and coating metal surfaces with flame retardant paint coatings (Mouritz and Gibson, 2006).

Intumescent coating and reactive flame retardant agents for coating are well known to many building superintendent, contractors, electronic manufacturers, rubber and plastic products producers and even firefighters. However, the cost of intumescent coatings and the environmental damage caused by halogenated flame retardant agents made researchers to commence findings on other less expensive and environmentally friendly flame retardant additives that can be incorporated into polymer products including coatings (Gerard *et al.*, 2010).

Flame retardant coatings are special coatings designed to protect the substrate in the event of fire incident. The ideal flame retardant paint or coating should be incombustible, maintain its integrity upon exposure to fire. Also, the coating must be suitably decorative and easily applied, maintain its fire retardancy and decorative appeal during exposure to weather elements or repeated washing and scrubbing, it also should be easily recoated with conventional or fire-retardant coatings and preferably be no costlier than conventional coatings.

Increase in the growth of rice in Nigeria invariably generated greater problem of managing the rice husk agricultural waste which account for 20% of the rice paddy milled. Rice husk has been widely reported to be a great environmental threat to land and the surrounding area where it is dumped (Ajay *et al.*, 2012). But rice husk ash is rich in silica, account for about 80 to 97% of the ash and has proven to be good source of superfine silica which could be an effective flame retardant agent based on its thermal stability.

In this research, the oil from castor plant which is a renewable source and that can be grown in commercial quantity in any parts of Nigeria and beyond was used to synthesize alkyd resin which was later used to prepare flame retardant paint coating using superfine micro silica particles as flame retarding agent.

Materials and Methods

Materials

All chemical reagents including cetyltrimethylammonium bromide (CTAB), NaOH, H_2SO_4, KOH, xylene and benzene used for this work were of analytical grade and were used without further purification except otherwise stated. The rice husk and castor seeds were both obtained from Basawa area in Zaria, Kaduna State, Nigeria.

Extraction of castor seed oil

The castor seed oil was extracted using solvent extraction method with the aid of soxhlet apparatus and n-hexane was used as the extraction solvent as reported by Omowanle *et al.* (2018a). The washed, oven dried castor seeds were ground and filled into the thimble of the soxhlet apparatus and the solvent (n-hexane) was heated to temperature of 60^0C for five hours and thereafter allowed to cool. The collected extract was heated and evaporated using rotary evaporator to obtain solvent-free oil.

Determination of the physico-chemical properties of castor oil

The physico-chemical properties which are the specific gravity, colour, acid value, saponification value and the iodine value of the oils were determined using AOAC (2000) standard methods.

Preparation of dehydrated castor seed oil (DCO)

The extracted castor seed oil was dehydrated in a round bottom flask with 2% (wt%) of $NaHSO_4$ catalyst as reported by Nway and Mya (2008). The system was heated to temperature of 140^0C under vacuum and the dehydration time was 75 min. The physico-chemical properties especially the iodine value, saponification value and the acid value of the dehydrated castor oil were carried out using AOAC (2000) standard method.

Preparation of the dehydrated castor seed oil alkyd resin

The alkyd resin was prepared using alcoholysis method as explained by Omowanle *et al.* (2018b), using the dehydrated castor seed oil, glycerol and phthalic anhydride while Calcium (II) oxide was used as catalyst.

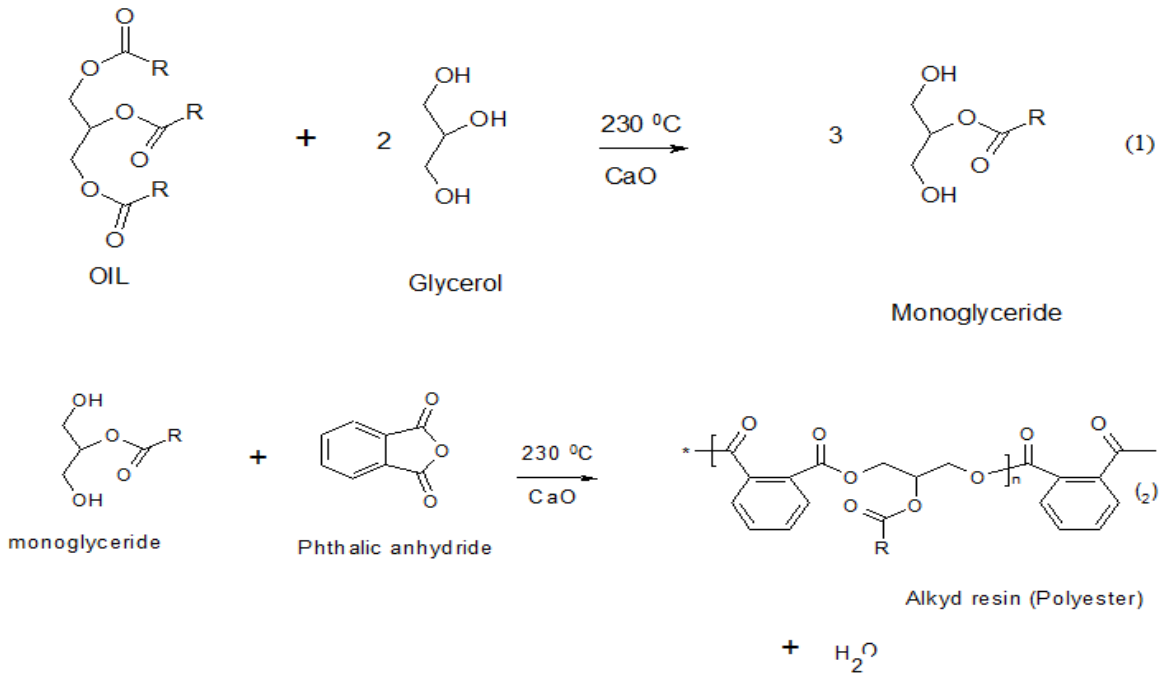

Table 1: Recipe for the preparation of dehydrated castor seed oil alkyd resin

S/N	Reagents	MDCOA
1.	Dehydrated castor Seed Oil	214.62
2.	Phthalic Anhydride	143.41
3.	Glycerol	91.97
	Total	**450.00**
4.	Duration of Monoglyceride	3 h 55 min
5.	Duration of Polyesterification	6 h 10 min

MDCOA= Medium oil length dehydrated castor oil alkyd resin

Preparation of rice husk silica

The silica particles were extracted from the rice husk using the method outlined by Van *et al.* (2013). The rice husk was ashed in a muffle furnace at a temperature of 550^{0}C for 5 h to remove all the hydrocarbons associated with the rice husk. 50 g of the rice husk ash was refluxed 1.2 Molar solution of HCl and later with 5 Molar solution of H_2SO_4 for two hours each at temperature of about 100^{0}C to remove all the incorporated metal oxides besides silica oxide in the rice husk ash. The slurry was filtered and washed thoroughly with distilled water until the pH is 7.

The silica slurry was refluxed with 3.5 Molar solution of NaOH for 5 h to obtain sodium silicate solution. The solution was later filtered and washed with distilled water and solid sample cooled and kept separately.

The cetyltrimethylammonium bromide (CTAB) which was the surfactant used was dissolved in an equal amount of butanol and water at 2.0% weight. The sodium silicate solution was gradually added to the CTAB, water and butanol mixture while stirring at the temperature of about 60^{0}C and the stirring continued for a while after addition of the sodium silicate solution. Thereafter, 0.5 Molar solution of H_2SO_4 was gradually added to the mixture until the pH is about 4 to commence the hydrolysis-condensation reaction. The mixture was then allowed to settle for about 6 h at temperature of about 60^{0}C. The silicate particles were found at the boundary between the butanol and the aqueous phase.

$$NaOH + SiO_2 \rightarrow Na_2SiO_3 + H_2O \quad (3)$$

$$Na_2SiO_3 + H_2SO_4 \rightarrow SiO_2 + Na_2SiO_3 + H_2O \quad (4)$$

The silica particles were filtered out of the boundary phase between the butanaol and the aqueous phase and washed with distilled water to remove all the associated solvent and surfactant. The superfine silica was dried in an oven at moderate temperature before further characterization. The final product was stored in desiccator and later characterized by Fourier Transform Infra Red Spectroscopy (FTIR), Scan Electron Microscopic (SEM) and Imagej analytical software tools as outlined by GK-12 Program of Drexel University (2010) and Vippola *et al.* (2016).

Preparation of silica composite flame retardant paints

Silica composite paints were prepared using standard formulation as reported by Karthikeyan *et al.* (2014) and Oladipo *et al.* (2013) but with little modification. Silica particles were used in the formulation at concentration of 2.5, 5.0, 7.5 and 10%, respectively (of the total reaction mass) as flame retardant agent while the dehydrated castor oil (DCO) alkyd resin was used as binder in the paints. In all the paint samples only medium oil length alkyd resin was used. Control medium oil length alkyd resins (DCO) paint was also prepared and its properties were also determined.

Fig. 1: DCO Alkyd resin flame retardant silicate composite paint

Determination of the properties of DCO alkyd resins paints

Specific gravity test

The specific gravity of the paint sample was carried out using standard method as explained by Omowanle *et al*. (2018b).

Opacity test

The opacity test was carried out as reported by Uthman (2011) and Omowanle *et al.* (2018b) this test was done by coating 20 cm^2 surfaces with the paint samples.

Drying time

The drying time of the silicate composite paints was determined using ASTM D1640 standard methods of Drying, Curing, or Film Formation of Organic Coatings at room temperature. The following tests were carried out; Set-to-touch Time, Dust-free Time and Dry-through (Dry-to-handle) Time as reported by Omowanle *et al.* (2018b).

Determination of the properties of the silicate composite flame retardant paints

The general properties of the silicate composite paints such as colour, drying time, specific gravity and opacity were determined using standard methods as reported by Omowanle *et al*, 2018 b.

Test of flammability of the silicate composite flame retardant paints

The following flame retardant tests were carried out on the samples; Flash point, UL 94 Vertical flame Spread (flame application time; 20 seconds) and Limiting Oxygen Index.

Determination of flash point of paint and silicate composite paints

The flash points of the samples paint and silicate composite paints were also determined using Cleverland open cup method according to ASTM D9266 (IP Standards) as reported by Njoroge (2013).

Vertical burning test (modified UL 94-V test)

Flame retardancy of the control sample and the silicate composite alkyd resin paint coatings were determined using the vertical (ASTM D 568 – 77) method. Coated *mansonia* wood specimens of 100×120 mm were used for all the samples. In the vertical tests, a vertical fixed sample was ignited and burnt for 10 s with a 1 inch methane flame, and the burning time was measured after the flame was removed. The test is repeated for all the samples (Basak *et al.*, 2015). The Bunsen burner flame was applied to the specimen twice (10 s each). After each application the time of self-sustained combustion was recorded. A second application of the flame follows immediately after self-extinguishment of the specimen in the first application. A V-0 classification is given to material that is extinguished in less than 10 s after any flame application and the mean combustion time for the five specimens tested (10 flame applications) did not exceed 5 s, and no combustible drips was observed. For V-1 rating or classification was received by a sample with maximum combustion time < 50 s and mean combustion time for five specimens

< 25 s and no combustible drips were observed. Sample is classified V-2 if it satisfied the combustion time criteria of V-1, but flammable drips ignite the cotton under the sample (Alexander and Charles, 2007).

Limiting oxygen index (LOI)

The value of limiting oxygen index (LOI) is defined as the minimal oxygen concentration [O_2] in the oxygen/nitrogen mixture [O_2/N_2] that either maintains flame combustion of the material for 3 min. or consumes a length of 5 cm of the sample, with the sample placed in a vertical position (the top of the test sample is inflamed with a burner) (Laoutid *et al.*, 2008).

The LOI is expressed as;

$$LOI = 100 \quad \frac{[O_2]}{[O_2]+[N_2]} \qquad (5)$$

The higher the LOI, the better the flame retardancy of the test sample. The LOI was carried out according to ASTM D 2863 standard as reported by Abba *et al.* (2012). Specimen sample of 5.0 × 1.0 cm coated with the silicate composite flame retardant alkyd resin paint was mounted vertically at the centre of the combustion tube of the limiting index equipment. The mixture of nitrogen and oxygen gases flows upstream the tube and after about 30 second purge of the column, the coated sample was ignited at the top of the tube with the aid of an igniter. The minimum amount of oxygen concentration that supports combustion for each case was noted. This process was repeated three times for each sample and the average value was taken as LOI for that particular sample. Control test was also carried out and the value was used to determine extent of flame retardancy of the sample. The limiting oxygen index equipment for the test was supplied by Wuhan Guoliang Instrument Company Limited, China and of the JF-3 Digital Temperature Oxygen Index Tester.

Results and Discussion

Physico-chemical properties of dehydrated castor oil (DCO)

The physicochemical properties of the castor seed oil and the dehydrated castor seed oil are similar as shown in Table 3 there was significant increase in the iodine value which made the oil suitable for the preparation of paint coating being a drying oil after dehydration. It should be noted that Drying oil has iodine value greater than 130.

Table 3: Physico-chemical properties of dehydrated castor oil (DCO)

S/N	Physico-chemical Properties	Castor Seed Oil	Dehydrated Castor Oil
1.	Colour	Colourless	Light
2.	Specific gravity	0.960	Brown
3.	Acid value (mg KOH/g)	12.69	0.934
4.	Saponification value (mg	184.30	18.75
5.	KOH/g)	97.61	189.40
6.	Iodine value (gI_2/100g)	304.89	131.00
	Average Molecular Weight (Mw)		296.20

Table 4: Physico-chemical properties of the medium oil length DCO Alkyd resin

S/N	Physico-chemical properties	MDCOA
1.	Colour	Dark Brown
2.	Specific gravity	0.94
3.	Acid value (mg KOH/g)	5.61
4.	Saponification value (mg KOH/g)	277.80
5.	Iodine value (gI_2/100g)	80.37
6.	Average Molecular Weight (Mw)	201.94

Physico-chemical properties of the medium oil length DCO alkyd resin

The physicochemical properties of the medium oil, dehydrated castor seed oil was as given in Table 4. The properties of the alkyd resin were similar to alkyd resins prepared from other plant seed oils such as methyl ester of rubber seed oil (Ikhuoria *et al.*, 2004) and *ximenia Americana* (wild olive) seed oil (Oladipupo *et al.*, 2013). The medium oil length alkyd resin of the dehydrated castor oil has acid value of 5.61 (mg KOH/g) which revealed that the resin was formed at high percentage extent of reaction (Aigbodion and Okiemien, 1996). The iodine value of 80.37 (gI_2/100g) shows that the resin will dry at normal weather conditions (when used for both indoor and outdoor coatings) without difficulty.

FTIR spectra of medium DCO alkyd resin

The FTIR spectra of the medium oil length DCO alkyd reveal clearly polyester molecular structure of the resin. The absorption band at the frequency wavelength 3444.1 cm^{-1} is that of -OH this was from Hydroxyl excess of □, ß-Hydroxyl groups of the glycerol in the formulation.

The peaks at 2926.0, 2855.1 cm^{-1} are assigned to -CH_2, CH_3 of the aliphatic chain of the free fatty acids in the dehydrated castor oil used in the preparation of the resin. The peak at 1722.0 cm^{-1} was for -COO-, the ester group of the alkyd resin. Other peaks in the fingerprint region also confirmed polyester structure of the alkyd resin, though the absorption peaks that are of interest are those beyond the finger print region (1500 – 1000 cm^{-1}) as every organic molecule has absorption in the finger print region and the absorptions are due to the vibration of the entire molecular skeleton (Anderson *et al.*, 2004).

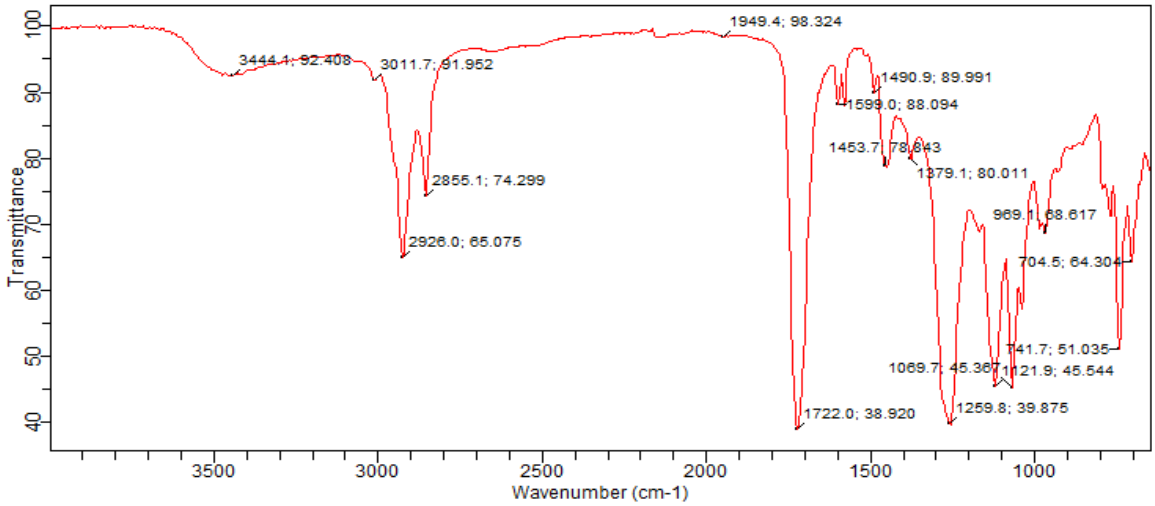

Fig. 2: FTIR Spectra of medium DCO alkyd resin

SEM analysis of silica particles using 2.0% CTAB

The SEM micrograph of the silica particles was analyzed using imageJ software SEM analytical tool (Vippola *et al.*, 2016). The fer et distance (the long distance between two points on the circumference of the particle) of 2,971 particles analyzed has a mean value of 13.428 µm and the SEM micrograph (of the 2.0% CTAB derived silica particles) is given in Fig. 3.

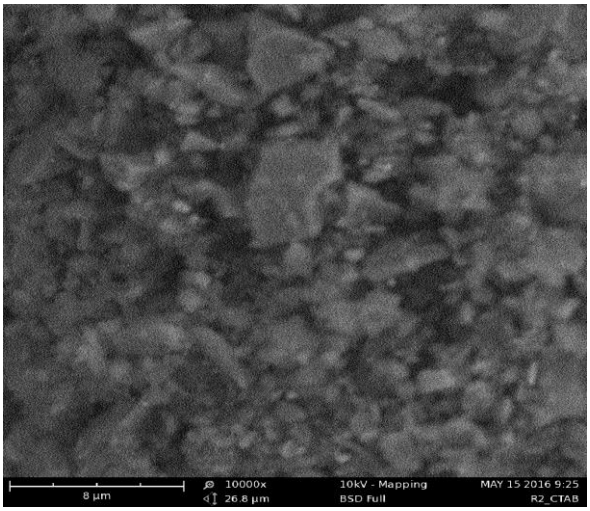

Fig. 3: SEM Micrograph of silica particles using 2.0% CTAB

Characterization of the rice husk silica

The FTIR spectra of rice husk ash silica particles obtained from sol-gel methods using 2.0% Cetyltrimethylammonium bromide (CTAB) as surfactant, have absorption peaks in the regions of about 1013.8 and 775.3 cm^{-1} (Fig. 4) which are due to asymmetric and symmetric stretching modes of SiO_2 (Ugwekar and Abhishek, 2015). The mean values of the particle sizes of the 2.0% CTAB derived silica particles was 13.428 µm which revealed that 2.0% CTAB is a good surfactant in extracting silica by sol-gel method (Van *et al.*, 2013).

Properties of the silicate composite paints

The physical properties of the dehydrated castor oil alkyd resin paint and silicate composite paints were as given in Table 5. All the paint samples are of flame red colour derived from yellow and red colour pigments during formulation as given in Table 2. The specific gravity of the paint samples is similar to the values reported for gloss paint by other authors (Oladipupo et *al.*, 2013).

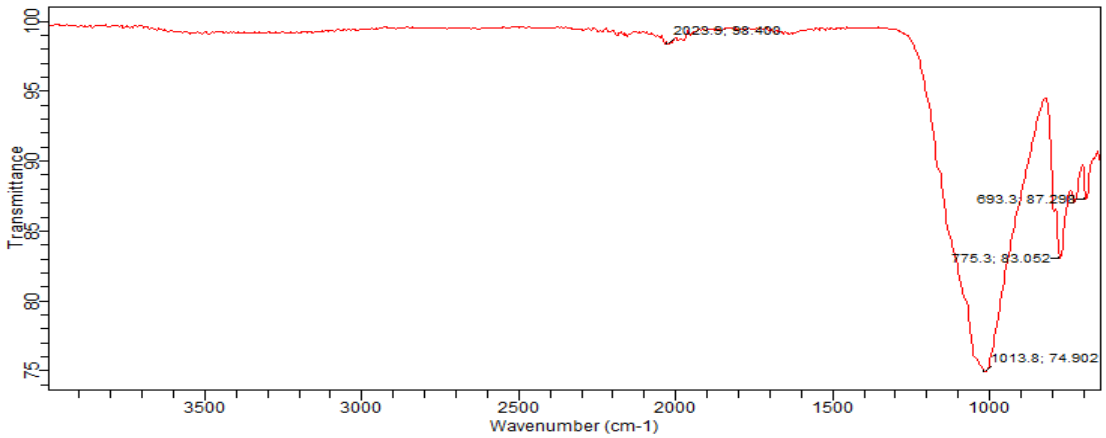

Fig. 4: FTIR Spectra of Silica using 2.0% CTAB

Table 5: Properties of the silicate composite paints

S/N	Paint samples	Colour	Spec. gravity	Set-to-touch (Min)	Dust-free (Min)	Dry-through (Hr)
1.	DCOAP	Flame red	1.14	30	120	Overnight
2.	2.5% DCOAP	Flame red	1.14	30	120	Overnight
3.	5.0% DCOAP	Flame red	1.17	30	120	Overnight
4.	7.5% DCOAP	Flame red	1.17	25	90	Overnight
5.	10.0% DCOAP	Flame red	1.18	25	90	Overnight

DCOAP: Dehydrated castor alkyd resin paint

All samples of the DCO and silicate composite paints has excellent properties compared with that of wild olive oil alkyd paint as reported by Oladipupo *et al.* (2013). The drying properties of the paints make them good for both indoor and outdoor applications (Omowanle *et al.*, 2018b).

Flame retardant tests of silicate composite paints

Flash points of silicate composite paints

All the silicate composite paints have flash points above 100ºC and this shows that they are non-flammable and can therefore be stored and transported under normal weather conditions.

Table 6: Flash points of the silicate composite paints

S/N	Silicate composite paints samples	Flash points (ºC)
1	SCDCOAP	140.0
2	2.5%SCDCOAP	148.0
3	5.0%SCDCOAP	160.0
4	7.5%SCDCOAP	168.0
5	10.0%SCDCOAP	180.0

SCDCOAP: Silicate composite dehydrated castor alkyd resin paint

Flash point is a very important safety and flammability parameter. The flash points of dehydrated castor oil alkyd silicate composite paints were as given in Table 7. The flash points of silicate composite paints were higher than that of non- silicate composite paint and the flash points were observed to increase with the amount of the micro size silica particles content of the paints. The 2.5% (148.0^{0}C), 5.0% (160.0^{0}C), 7.5% (168.0^{0}C) and 10.0% (180.0^{0}C) for silicate composite dehydrated castor oil paints (SCDCOP). This shows that silica conferred some degree of thermal stability on the silicate composite paints. The kerosene solvent used in the preparation of the gloss paint has flash point of about 60^{0}C, and the flash point of the silicate composite paint samples was between 125 to 180^{0}C which revealed that the addition of silica in the paint formulation has greatly increased their thermal stability. Silica particles have been reported to be among most thermally stable inorganic substances with bond energy of 798 KJ/mol (Kashiwagi *et al.*, 2003).

Vertical burning test (modified UL94-V test)

The vertical burning test (modified UL 94) results shown in Table 7 revealed positive V_0 performance for all the paint samples except for the dehydrated castor seed oil paint (DCOAP) which is V_1. The flame application period was 20 seconds instead of 10 seconds to enable the coated samples to ignite. The DCOAP extinguished the applied flame at about 15 seconds and the 2.5, 5.0, 7.5 and 10.0% silicate composite dehydrated castor oil paint (SCDCOAP) extinguished their flame in less than 10 seconds showing that the addition of silicate has imparted flame retardancy to the paint coatings.

The results confirmed that low loading of superfine micro size silica particles can bring about flame retardancy in polymer materials (Laoutid *et al.*, 2009; Abba *et al.*, 2012). Previous research had attributed the flame retardant effect of silicate to char formation on top of burning polymer. The primary reason behind the effectiveness of the silicate as flame retardant is their platelet geometry, high aspect ratio and very high specific surface area and studies have also proven that flame retardancy of silicate was due to reduction in heat releasing rate (Gilman, 2000).

Table 7: Vertical burning test (modified UL 94-V test)

S/N	Silicate Composite Paints	Mean period of self-extinguishment (S)	Dripping of flaming materials	UL94-V Rating
1.	SCDCOP	15	No dripping	V_1
2.	2.5%SCDCOP	<10	No dripping	V_0
3.	5.0%SCDCOP	<10	No dripping	V_0
4	7.5%SCDCOP	<10	No dripping	V_0
5.	10.0%SCDCOP	<10	No dripping	V_0

Table 8: Limiting oxygen index (LOI) and vertical burning time of silicate composite paint coatings

S/N	Samples	Limiting oxygen index (LOI) values	Vertical burning time (seconds)
1	Uncoated Sample	18.7	80
2	DCOAP	21.5	148
3	2.5% SCDCOAP	23.0	168
4	5.0% SCDCOAP	25.5	179
5	7.5 % SCDCOAP	26.6	170
6	10.0% SCDCOAP	28.9	176

Limiting oxygen index (LOI) and the vertical burning time of silicate composite paint coatings

The limiting oxygen index values of dehydrated castor oil alkyd paint and the silicate composite dehydrated castor oil paints are as given in Table 8. It is obvious from the results of the limiting oxygen index values of the uncoated *mansonia* wood, coated *mansonia* wood without silica and coated *mansonia* wood samples with silica, that the micro size silica particles conferred flame retardancy on the substrate. The LOI value of uncoated *mansonia* wood was 18.7 which means that it is totally combustible as it combustion can be self-sustained at normal environmental conditions with 21% oxygen. And, the dehydrated castor oil alkyd paint (DCOAP) has LOI values of 21.5 which show that it is almost "combustible" because their LOI value is fairly greater than 21 and, the fact that the LOI value of the dehydrated castor oil alkyd paint (DCOAP) is significantly greater than the uncoated sample shows that they displayed little degree of flame retardancy which could be attributed to the titanium dioxide in the paint formulation. Zhenyu *et al.* (2007) had earlier reported that titanium dioxide can improve flame retardancy of coating and that nano-TiO_2 is more efficient than general TiO_2 in improving thermal stability of polymer because the general TiO_2 usually require high loading.

The silicate composite dehydrated castor oil alkyd paint (SCDCOAP) samples have higher values than 21 which shows that they are "self-extinguishing" and are "non-combustible" because their combustion cannot be self-sustained in air of oxygen value of 21 at ambient temperature without external energy contribution, hence, they are flame retardant paint coatings (Lauotid *et al.*, 2009).

The vertical flame spread time or the vertical burning time (in seconds) of the samples in the combustion chamber of the limiting oxygen index (LOI) equipment and the values of the LOI of the sample at that particular time is given in Table 8. The uncoated *mansonia* wood sample has the flame spread completely through its 100×12 mm length in 80 seconds while its LOI value was 18.7. The coated wood sample with dehydrated castor oil alkyd paint (DCOAP) spread flame through its length in 148 seconds at LOI value of 21.5 and the silicate composite dehydrated castor oil alkyd paints (SCDCOAP) of 2.5, 5.0, 7.5 and 10.0% silicate compositions have the vertical flame spread values of 168 seconds (LOI value of 23.0), 179 seconds (LOI value of 25.5), 170 seconds (LOI value of 26.6) and 176 seconds which has LOI value of 28.9, respectively.

From the results of vertical flame spread, it is clear that the addition of micro size silica particles in the coating delay the spread of the flame and the severity of the combustion (higher LOI). This shows that the silica particles acted as flame retardant since one of the primary action of flame retardant coating is to reduce flame spread and prolong combustion period (PDRA, 2012).

The silica particles acting as flame retardant in the above mentioned paint coatings will undoubtedly be as a result of its thermal stability. Silica is one of the more stable mineral flame retardant ever known with melting point of $1{,}713^0$C and Si-O bond energy of 798 KJ/mole (Haynes, 2011). During combustion energy is absorbed to melt the SiO_2 to form a protective layer over the surface and thereby reducing the available energy in the system to generate volatile gases required to sustain the combustion (Masaki, 2010).

Conclusion

Flame retardant silicate composite gloss paint coatings were prepared using medium oil length alkyd resin from dehydrated castor seed oil while the superfine micro size silica particles extracted from rice husk were used as flame retardant agent. The micro size silica particles extracted from rice husk agricultural waste using the sol-gel method has mean size 13.43 µm. All the flame retardant paints exhibited excellent paint properties such as drying time, specific gravity and colour. They all have flash point above 100^0C and retard flame spread. The vertical burning test (modified UL94-V test) which gives the response of the samples in normal room conditions on application of flame at 20 seconds revealed that the silicate composite paint coatings exhibited flame retardant property. The limiting oxygen index (LOI) and the vertical burning time of silicate composite paint coatings confirmed the flame retardancy of the silicate composite paint coatings in numerical values. It was generally observed that the flame retardancy of the samples increased with an increase in the micro size silica particles compositions.

Conflict of Interest

The authors declare that there is no conflict of interest related to this study.

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