Study on Flame Retardancy, Mechanical, and Thermal Property on Epoxy Based Aluminium Hydroxide and Aerogel Composite Material

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Abstract: Composite materials are very versatile and are utilized in wide variety of applications. A composite is a combined material created by synthetic assembly of two or more components selected filler or a reinforcing agent and a compatible binder (i.e., a resin) in order to obtain specific characteristics and properties.

In this study, horizontal burning, vertical burning and limiting oxygen index (LOI) were conducted to evaluate the effects of mixed of silica aerogel and aluminium hydroxide on flame retardancy for epoxy resin. The influence of mechanical and thermal properties of two kinds composites are aluminium hydroxide and silica aerogel for epoxy resin composites ($Al(OH)_3$ –epoxy, silica aerogel – epoxy) were studied. The mechanical properties of the composites are investigated and compared to those two composites. The enhancement effect of the epoxy resin on the Tensile Strength, Stiffness and Compressive Strength of the silica aerogel composite is greater than that of the aluminium hydroxide composite. The Thermal Conductivity is also evaluated experimentally. As per ASTM standards, the test specimens preparation and testing is carried out, and results are presented.

Keywords:- **Silica Aerogel, Aluminium Hydroxide, Thermal Conductivity, Mechanical Strength, flame retardancy, Flammability LOI,**

I. INTRODUCTION

Aerogel, which is light and nano-porous, is a very attractive material to be used as candidate for super insulating material. It has very low density and thermal conductivity due to its high porosity and specific surface. In 1932, Kistler first measured the thermal conductivity of silica aerogel which is about 0.02 W/m K^0 at normal atmospheric condition, and about 0.01 W/m K^0 under vacuum condition [1]. Many researchers also have investigated heat transfer behaviors inside aerogel materials[2,3]. Heat transfer in silica aerogel is closely associated with its complex nano-porous structure. Previous analytical studies on silica aerogel's thermal performance mostly based on some kinds of regular typical structure. Zeng et al. [4].For improving the poor strength of silica aerogel, the most common method is adding reinforcing fibers into aerogel and this could also influence the heat conduction and thermal radiation. Many researchers have studied fibrous material's thermal performance experimentally and analytically. Tong and Tein [5] studied the radiative heat transfer in a grey medium made up by fibers distributed randomly in space by using the two-flux approximation model. The main aim of this paper is to synthesize Aluminium Hydroxide / Epoxy Composite Filled with Silica Aerogel Material as per ASTM standards and to investigate the effect of Aluminium Hydroxide and Silica Aerogel on mechanical and thermal behavior of composites with different proportions. The Aluminium Hydroxide $Al(OH)_3$ enhances the fire resistance of the composite material and Silica Aerogel is expected to reduce the thermal conductivity of the composite material.

II. EXPERIMENTATION AND METHOD

2.1. Raw Materials

The composites are prepared from commercially available Epoxy Resin (L-12) along with the Hardener K-6, manufactured by M/s.Atul Industries Ltd, and are used as the matrix material and the curing agent. Silica Aerogel particles (Lumira LA1000) supplied from Cabot Corporation are used. These particles are hydrophobic in nature having a average pore diameter of 20 nm,

particle size range of 0.7- 4.0 mm, density of 120 kg/m^3 and thermal conductivity of 0.01 W/m K^0 as suggested by the manufacturer. These particles are shown in Fig.1. Aluminium Hydroxide a white amorphous powder with a density of 2420 kg/m^3, which is used as common fire resistant material.

Fig. 1: Silica Aerogel

2.2. Specimen Preparation

The specimens of Aluminium Hydroxide / Epoxy composites filled with Silica Aerogel material of different proportions were prepared. The Resin to Hardener volume proportion is kept up at 100:9 as suggested by the manufacturer. Silica Aerogel will be used as filler material in Aluminium Hydroxide / Epoxy composite. Stir-mixing is the most generally utilized technique for manufacturing of the particulate composites. Fabrication of the composites was done at the room temperature by Hand Lay-up technique. Initially, wooden Moulds were prepared for specimen preparation as per ASTM standards. Firstly during preparation a glass sheet is placed then the mould is placed on the glass sheet. Now the mixture is poured into the mould and a glass sheet is placed on the mould filled with the mixture then a load of 10kg is applied and is allowed for curing for 24 hr. Following compositions are prepared as depicted in Table 1.

Table 1: Compositions used for specimen preparation

Sl. no	Sample Designation	Epoxy %	$Al(OH)_3$ %	Silica Aerogel %
1	A1	80	20	0
2	A2	85	15	0
3	A3	90	10	0
4	A4	80	15	5
5	A5	85	10	5
6	A6	90	5	5

2.3. Tests and Measurements

2.3.1. Limiting Oxygen Index

LOI are widely used to characterize the flame retardancy of materials and to investigate the effectiveness of flame retardants. In this study, the LOI values were measured on a oxygen index instrument as shown in Fig 2 (Konkan Speciality Polyproductspvt Ltd. Scientific Service Group - Mangalore) according to ASTM D2863. The samples were melded to the proper size (120 mm _10 mm _ 10 mm). The test procedures were as following: the sample was fixed vertically in the combustion cylinder and was flowed by the mixture of oxygen and nitrogen from the bottom, the top of the sample was ignited by a butane gas flame, recorded the time and length of the combustion, then the minimum oxygen concentration just to maintain a stable combustion can be determined.

Fig 2. Experimental Set Up Of LOI Test

2.3.2. Horizontal Burning Test

The horizontal burning test was used to determine the relative rate of burning of composite specimen according to ASTM D-2863 with an instrument show in fig 3. (Konkan Speciality Polyproducts Pvt Ltd. Scientific Service Group - Managalore). The samples were made to a size of 120 mm _ 10 mm _ 10 mm. In the horizontal burning test, the sample was oriented horizontally and placed in a test chamber, then ignited the end of the sample applied a flame from a Bunsen Burner for 30 s, the time until the flame extinguished itself and the distance the burn propagated must be measured, then figured out the linear burning rate in mm per minute.

Fig 3. Horizontal and vertical test chamber gas torch

2.3.3. UL-94 Vertical Burning Test

The Vertical burning test was used to determine the relative rate of burning of composite specimen according to UL-94 with an instrument show in fig 3. (Konkan Speciality Poly products Pvt Ltd. Scientific Service Group - Managalore). The samples were made to a size of 120 mm _ 10 mm _ 10 mm. In the Vertical burning test, the sample was oriented Vertical and placed in a test chamber, then ignited the end of the sample applied a flame from a Bunsen Burner for 30 s, the time until the flame extinguished itself and the distance the burn propagated must be measured, then figured out along with long axis vertical rate in mm per minute and also three important requirement has been achieved.

They are V0, V1, V2.

2.3.2.1 Requirements for V-0

- The specimens may not burn with flaming combustion for more than 10 seconds after either application of the test flame.
- The total flaming combustion time may not exceed 50 seconds for the 10flame applications for each set of 5 specimens.
- The specimens may not burn with flaming or glowing combustion up to the holding clamp.
- The specimens may not drip flaming particles that ignite the dry absorbent surgical cotton located 300 mm below the test specimen.
- The specimens may not have glowing combustion that persists for more than 30 seconds after the second removal of the test flame.

2.3.2. 2 Requirements for V-1

- The specimens may not burn with flaming combustion for more than 30 seconds after either application of the test flame.
- The total flaming combustion time may not exceed 250 seconds for the 10 flame applications for each set of 5 specimens.
- The specimens may not burn with flaming or glowing combustion up to the holding clamp.
- The specimens may not drip flaming particles that ignite the dry absorbent surgical cotton located 300 mm below the test specimen.
- The specimens may not have glowing combustion that persists for more than 60 seconds after the second removal of the test flame.

2.3.2.3 Requirements for V-2

- The specimens may not burn with flaming combustion for more than 30 seconds after either application of the test flame.
- The total flaming combustion time may not exceed 250 seconds for the 10 flame applications for each set of 5 specimens.
- The specimens may not burn with flaming or glowing combustion up tothe holding clamp.
- The specimens can drip flaming particles that ignite the dry absorbent surgical cotton located 300 mm below the test specimen.
- The specimens may not have glowing combustion that persists for more than 60 seconds after the second removal of the test flame.

2.4. Mechanical Properties

2.4.1 Compression Test

Following the ASTM D695-95 method, the UTM was used to measure the strength of the material under Compression. By using thin films, about 20 mm, of the previous conditioned samples, the measurements were conducted with a 500 mm/min crosshead speed at room temperature. Six measurements were taken for each sample and the data were averaged to obtain a mean value.

2.4.2 Tensile Test

The Tensile test of Al (OH)3 / Epoxy Composites filled with or without Silica aerogel material (A1,A2…,A6) has been performed using UTM. The experiment is done according to the American Society of Testing and Materials (ASTM). As per standard, ASTM D3039 the specimens with dimensions 250mm length 25mm width and 10mm thickness were prepared. The specimen was loaded under tensile load until the failure occurs. This test is carried out to determine tensile strength and modulus of elasticity of the specimen.

2.5. Thermal Conductivity Test

The Thermal conductivity of the composite is determined according to ASTM E1530 guarded heat flow meter under steady state condition. In this experimental setup, the composite specimen to be tested is held under a compressive load between two metal slabs so that there is a good contact resistance between the specimen and slab surfaces. A heat flux transducer is provided at the lower surface as the heat flows through the slabs an axial temperature gradient is established between the slabs. To measure the temperature the thermocouples are placed between the slabs. By measuring the temperature change over the composite specimen along with the output from heat flux transducer the thermal conductivity of the specimen is determined for the known specimen thickness. As per ASTM standards the specimen dimensions are of 100 mm diameter and 6mm thickness. The Thermal Conductivity is determined by employing one-dimensional Fourier's law of conduction. The Experimental setup as shown in Fig. 4.

$$Q = -K A\frac{dt}{dx} \quad (1)$$

Where,

Q= Heat transfer rate (W)

K= Thermal conductivity (W/mºC)

A=Area (m2)

dt/dx = Temperature gradient

Fig 4. Thermal conductivity setup shown in detail.

III. RESULTS AND DISCUSSION

3. Flammability

3.1. Limiting oxygen index

The experimental results of LOI test for different compositions are shown in the Table 2.

Table 2: Flammability test results of with and without aerogel contents.

Composite Specimen	Minimum Oxygen Concentration Required For The Burning
0% Aerogel (epoxy 85%,$Al(OH)_3$ 15%)	23.06%
5% Aerogel (epoxy 85%,$Al(OH)_3$ 10%)	23.75%

The LOI values of the with aerogel and without aerogel are shown in Table 2. The LOI of the without aerogel is 23.06%. As the aerogel content is increased from 0 wt% to 5 wt%, the LOI increases from 23.06% to 23.75%. These results indicate that the mixed flame retardant modified with aerogel exhibits better flame retardancy than that of the Without aerogel. The below fig 5 shows that adding aergel material is decreasing flame retardant. It reveals that aerogel plays an important role of flame retardant when the epoxy burn and only a small content of the mixed flame retardants can upgrade the horizontal burning of epoxy from without aerogel to with aerogel,

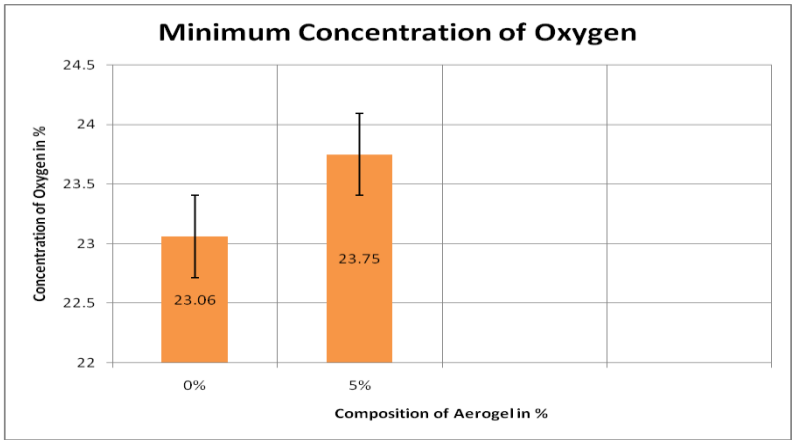

Fig. 5 : Comparison of with and without aerogel composite.

3.2. Horizontal Burning

The experimental results of horizontal Burning Test for different specimen compositions are shown in the Table 3.

Table 3: Comparison of thermal conductivity for different configurations

SAMPLE	THICKNESS(mm)	DAMAGED LENGTH(mm)	REMARKS
A1	10	25	HB PASS
A2	10	28	
A3	10	26	

A4	10	23	**HB PASS**
A5	10	26	
A6	10	21	

The horizontal burning test is widely applied to evaluate the extent and the linear burning rate of the combustion. From Table 1, it can be seen that the horizontal burning of epoxy resin (ER) is A1,A2,A3, which is higher than that the of A4,A5,A6.This indicates that the addition of Aerogel improve the flame retardancy of epoxy resin. The horizontal burning substantially decreased from without aerogel to with aerogel by the addition of only 5wt% aerogel. It reveals that aerogel plays an important role of flame retardant when the epoxy burn and only a small content of the mixed flame retardants can upgrade the horizontal burning of epoxy from without aerogel to with aerogel,

Fig. 6 shows the horizontal burning test graph plot is obtained by using Horizontal test chamber gas torch for the compositions A1, A2, A3 without the addition of filler material Silica Aerogel. The flame for compositions A1, A2, A3 are 23, 26, 21. It is observed that the composition A2 is having slightly high flame value than A1 and A3 compositions. Similarly for the compositions A4, A5, A6 with the addition of 5% of Silica Aerogel filler material. The Flamability for compositions A4, A5, A6 are 23, 26 and 21. It is observed that with the addition of filler material, the flammability have decreased.

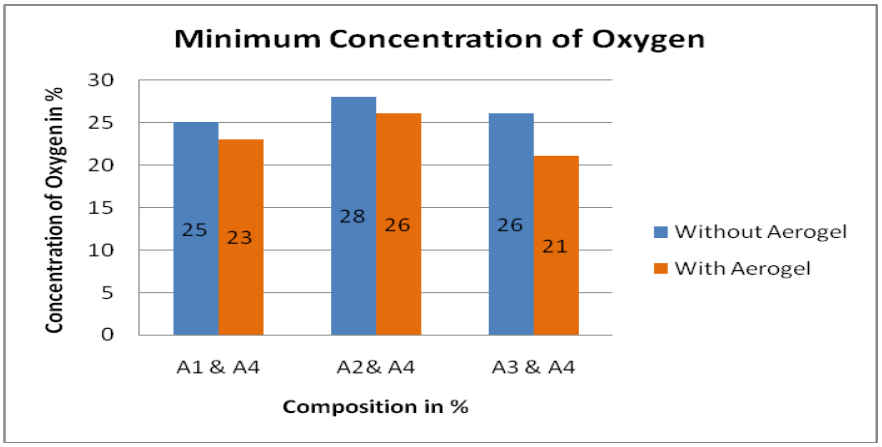

Fig. 6 : Comparison of with and without aerogel composite.

3.3. Vertical Burning

The experimental results of vertical Burning Test for with aerogel and without aerogel specimen compositions are shown in the Table 4.

Table 4: Comparison of with and without aerogel specimen

		Time of combustion		Total flaming for set of five specimen (seconds)	UL 94 flammability classification
Specimen		After first flame application	After second flame application		

	Thickness (mm)	Flaming (seconds)	Flaming (seconds)	Flaming plus glowing (seconds)		
Without Aerogel	10	30>	30>	-	Above 250sec	V0,V1,V2 pass
With Aerogel	10	30>	30>	-	Above 250sec	V0,V1,V2 fail

From the tabular column we can observe that V0, V1, V2 requirements are obtained for excluding aerogel material but after adding 5% wt of aerogel composite material it fails V0, V1, V2 requirements. It shows that the time taken to burn the specimens with Aerogel materialis more than the specimens without Aerogel. Thus we can say that Aerogel behaves as INSULATING MATERIAL.

3.4. Mechanical properties

3.4.1 Compression Test

The experimental results of compression test for different compositions are shown in the Table 5.

Table 5: Compressive Properties of the Composites

Sample No	Composition	Compressive Strength (MPa)	Young's modulus (MPa)
1	A1	26.085	169.71
2	A2	27.458	95.53
3	A3	25.889	42.59
4	A4	37.853	164.007
5	A5	29.714	156.55
6	A6	36.574	280.72

Silica Aerogel is a Mesoporous material. It has a greater specific surface area, larger pore diameters [10, 17] , therefore the $Al(OH)_3$ particles tend to accumulate [17] in the pores of Silica Aerogel resulting in a good reinforcing effect. This may be the reason for compressive strength and modulus enhancement. Thus from all the six compositions it is observed that the compositions A4, A5 and A6 with the addition of filler material Silica Aerogel showed the increased Compressive strength when compared with the compositions A1, A2 and A3. It is compared in the Fig. 7

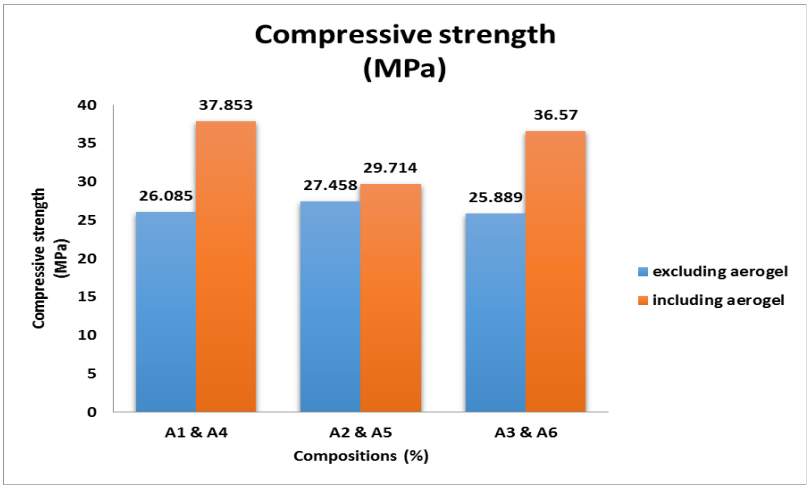

Fig. 7: Comparison of compressive strength with different compositions

3.4.2 Tensile Test

The experimental results of tensile test for different specimen compositions of the composite are reported in Table 6.

Table 6: Comparison of Ultimate Tensile Strength and Young's Modulus for Different Compositions

Sample no	Composition	Ultimate Tensile Strength (MPa)	Young's Modulus (MPa)
1	A1	2.94	57.54
2	A2	3.20	30.96
3	A3	4.30	530.864
4	A4	3.39	26.82
5	A5	3.98	34.57
6	A6	5.50	393.07

Fig. 8 shows the compared with the compositions A1, A2, A3 tensile strength for the compositions A4, A5, A6 are high due to the addition of filler material Silica Aerogel. The reason may be that since Silica Aerogel is a mesoporous material with high specific surface area and large pore diameter, the $Al(OH)_3$ particles tend to accumulate in the pores of Aerogel resulting in a good interfacial bonding. The incorporation of Silica Aerogel increased the Tensile Strength but Modulus of the material has decreased, this behaviour between the mesoporous Silica Aerogel and $Al(OH)_3$ / Matrix polymer cannot be understood at this stage which calls for further experiments to be conducted.

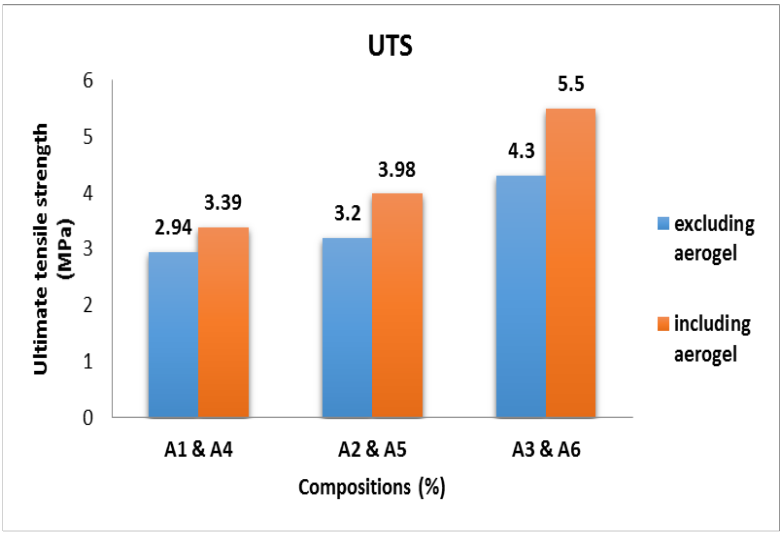

Fig. 8: Comparison of Ultimate Tensile Strength for different compositions

3.5. Thermal Conductivity Test

The experimental results of Thermal Conductivity Test for different specimen compositions are shown in the Table 7.

Table 7: Comparison of thermal conductivity for different configurations

Composition	Thermal Conductivity (K) W/mºC
A1	0.8187
A2	0.9865
A3	0.9854
A4	0.6618
A5	0.4044
A6	0.8371

The Table.7 show that the thermal conductivity for the compositions A1, A2, A3 are 0.8187, 0.9865, 0.9854 W/mºC. It is observed that the thermal conductivity values are very close to each other and the thermal conductivity of the composite material is influenced by its compositions [15]. The composition A1 is having slightly less thermal conductivity, this may be because of some chemical decomposition of Al $(OH)_3$ and release of small amount of water [12].This water is expected to reduce the heat flow.

As the filler material Silica Aerogel is added to the composite the thermal conductivity of the compositions A4, A5, A6 composite filled with Silica Aerogel material reduces to 0.6618, 0.4044 and 0.8371 W/mºC. It is observed that the composition A5 is having very less thermal conductivity than A4 and A6 this may be due to the good interfacial bonding between Silica Aerogel and $Al(OH)_3$ / Epoxy matrix polymer resulting in a good reinforcing effect when compared to other two compositions. The incorporation of silica aerogel leads to decrease in Conductivity since Aerogels are mesoporous with large diameters of 20

nm. Aerogels are good conductive insulators because they are composed entirely with air, and air is a very poor heat conductor. Fig. 9 shows the comparison of thermal conductivities of all the six compositions.

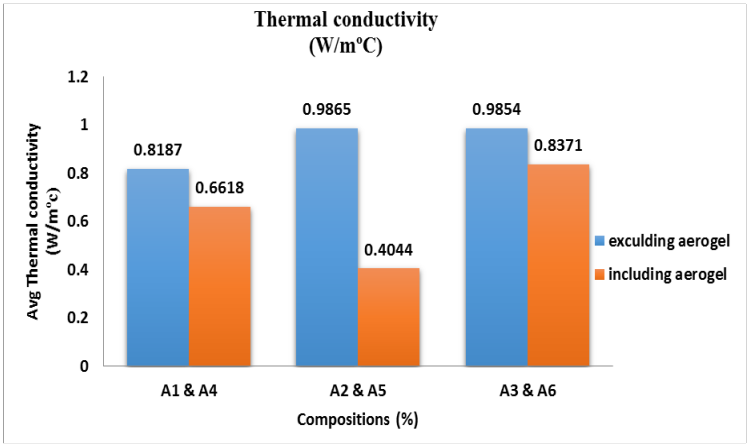

Fig. 9: Comparison of Thermal Conductivities of all the six compositions

IV. CONCLUSION

On the basis of the results of the effects of flame retardants on with the silica aerogel composite specimens and without the addition of Silica Aerogel, mechanical properties and thermal property of epoxy based aluminium hydroxide and aerogel Composite Material, the following conclusions can be drawn.

(a) The flammability tests on the specimens of different compositions as per ASTM standards (like HBT and VBT), with and without aerogel and we found that the usage of aerogel decreases flammability behaviour and LOI of aerogel composite specimen also increases significantly i.e Oxygen concentration required for the materials with aerogel increases by 0.69%.

(b) Mechanical tests such as tensile test and compression tests were conducted. Thus from result of compositions it is witnessed that with the addition of filler material Silica Aerogel, the composite material showed slightly increased Ultimate Tensile Strength and Compressive strength. This is due to the mesoporous structure of silica aerogel filler material.

(c) From the Thermal conductivity test it is perceived that with the addition of only 5% of the filler material SilicaAerogel, the thermal conductivity of the composite specimen reduced to greater extent when compared with the composite specimens without the addition of Silica Aerogel.

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