Experimental Investigation on Properties of DGEBA Based Epoxy Resin

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Abstract - *The Mechanical, Chemical, Thermal properties of Diglycidyl ether of bisphenol-A (DGEBA) based epoxy resin material with varying content of SiO_2 are studied in order to find out best composition. The ultimate aim is to modify the epoxy resin to improve mechanical and thermal properties. Tensile test and fracture toughness KIC of analyte as per ASTM E399 of SEN-B specimen performed on Instron 5980 universal testing system. Thermogravimetric Analysis (TGA) is performed on TGA 5500 by TA Instruments. Differential Scanning Calorimetry Analysis is done on DSC 4000 system by PerkinElmer.*

Key Words: Diglycidyl ether of bisphenol-A (DGEBA), SiO_2, SEN-B, Tensile Test, Fracture test, Thermogravimetric Analysis (TGA), Differential Scanning Calorimetry Analysis.

1. INTRODUCTION

There has been a denotation of epoxy resin to the engineering field from decades. It has played a vital role in terms of their material properties that is chemical, adhesive, thermal and electrical. It has been widely used in fibre reinforcement inside the composite materials and adhesive.

Many approaches have been done in order to increase the characteristics, performance of epoxy resin which includes adding of nano composites, some rubber additives/agents, thermoplastic stuffings, inorganic fillers and upgrading its chemical composition are in common practice. [1]

In the last decade it has been proved that improvement in mechanical and thermal characteristics of unadulterated polymer composition can be achieved by reinforcing composites in a very minute percentage of strong stuff/fillers. These modifications can be achieved by traditional processing techniques which results in negligible harmful effects on processability, aesthetics, thickness, capacity of structure. These compounds are considered for multiple uses which include electronics, automotive, packaging, marine and aviation industries etc. [2-3]

According to the results it has been observed flourishing combination with epoxy resin. Based on the results nano-scale fillers has been considered as filler material to manufacture high outcome composite material with upgraded properties [4-7].

As compare to microscale fillers, nano-clays have larger aspect ratio, plate morphology combined with less cost and low content requirement which make them a perfect filler reinforcement upgraded in structural properties have significant interest in nano composite material in numerous applications [8,9].

The integration of element is crucial in nano composites to produce a nano material with upgraded properties [10-12]. To obtain the best results from nano particles a homogeneous mixing is required the various mixing techniques of nano composites fabrication discussed in reference [13-14].

Epoxy resin have numerous applications in the field of electrical insulation, it has property to withstand high voltage system. Due to their low cost, light weight, ability to be moulded in any shape and electrical insulation of polymer matrix, there is a need to develop numerous types of resin to overcome generating requirement of high-performance thermoset material. In the above-mentioned field various applications are circuit board, bushing board and structural component [18,19]. The required result is composite with good mechanical integrity and high insulation properties [20,21].

In this study special attention is given to Mechanical, Chemical, thermal properties of epoxy resin in order to develop a material with best properties.

2. MATERIALS

The material used for preparation of analyte specimen are commercially available Diglycidyl ether of Bisphenol-A (DGEBA) based epoxy resin Epofine -5900 purchased from Changzou Chemical factory, Jiangsu province, China. Pathilic Anthydride (commercial name-HY905) and Dimethyl benzylamine (commercial name-DY061) as accelerator, both materials are purchased from Huntsman Advanced material (material)Pvt.Ltd. Silica powder 300 mesh is purchased from a local vendor from Aurangabad.

3. PREPARATION

To remove the moisture content in SiO_2 powder and preheating the DGEBA epoxy resin Epofine 5900 and silica powder are placed in a furnace for period of 2hrs at constant temperature of 150 °C. After preheating materials are taken in ratio as follows,

Resin : Hardener : Accelerator = 1 : 1 : 0.004

The quantity of SiO_2 is varied to make four different type of materials as 0 wt.%, 30 wt.%, 63 wt.%, 70 wt.%. As per above mentioned composition materials are mixed in a mixing chamber at 130 °C and 600 Mbar vacuum pressure for a time period of 50 min after that mixture is poured in a preheated mold of required specimen.

4. Experimental

4.1 Tensile strength

The tensile test is performed using Instron 5980 Universal Testing System, a machine with flexibility to test tensile, compression, shear, peel, cyclic and bend test for metals composites and advanced plastics as per ASTM standard.

According to ASTM D638 standard the specimen size is selected. ASTM D638 specifies method for testing the tensile strength of epoxy resin material for calculating their mechanical properties and outline accuracy requirement for test frames accessories used. The specimens for ASTM D638 are dumbbell shaped with either a 25 mm or 50 mm gauge length, a 50 mm gauge length is recommended.

As per ASTM D638, specimen type 1 is selected whose dimension are as follows. (Refer the figure-1)

Full length (l_3) = 165 mm
Parallel length (l_2) = 57 mm
Gauge length (l_1) = 50 mm
Parallel section width (strong) = 13 mm
Thickness (h) = 3.4 mm
Grip section width (strong) = 19 mm
Distance between grips = 115 mm

The test is performed on four specimens at crosshead speed of 1 mm/min with increasing weight percentage of SiO_2 in each specimen.
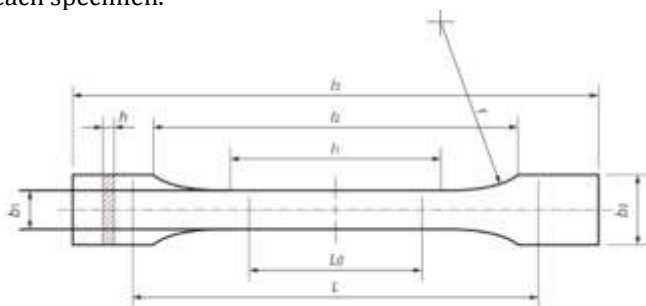

Fig -1: Dumbbell shaped specimen

4.2 Fracture toughness

Fracture toughness is an indication of the amount of stress required to propagate a preexisting flaw. Flaws may appear as cracks, voids, metallurgical inclusions, weld defects, design discontinuities, or some combination thereof. also denoted by K_{IC} [22,23].

Fracture toughness is measured of a three-point single edge notch bend (SEN-B) specimen as shown in figure-2 with the help of Instron 5980 universal testing system as per ASTM E399 standard

K_{IC} determined by ASTM E399 standard method characterizes the resistance of a material to fracture in a neutral environment in the presence of a sharp crack under essentially linear-elastic stress and severe tensile constraint, such that

The state of stress near the crack front approaches plane strain. The crack-tip plastic zone is small compared to the crack size, specimen thickness, and ligament ahead of the crack.

A V-notch at center of specimen is created. To make a crack of depth 3.2±0.2 mm, a razor blade is pressed at the bottom most point of the notch. The sample is of 10 mm thickness and the fracture toughness K_{IC} is calculated by following equation.

$$K_{IC} = (F\,S) / (B\,W^{(3/2)})\,f(x)$$

Where,
F = load required to start propagation of pre-crack
B = thickness of sample
S = distance between support reactions
W = width of the sample
A = total depth of notch/crack
f(x) = shape factor
f(x) depend on specimen design and loading and it is a dimensionless quantity.
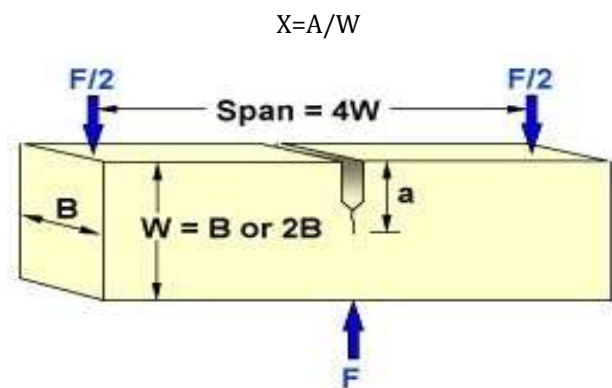
Where,

$$X = A/W$$

Fig -2: SENB specimen

4.3 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) is a method of thermal analysis in which changes in physical and chemical properties of materials are measured as a function of increasing temperature (with constant heating rate) or as a function of time (with constant temperature and/or constant mass loss). In simple words the properties of analytes are studied as they change with temperature [24-26].

If some property, which changes at some particular temperature range is known then the type of reaction that is taking place can be found out.

In this experimental technique the mass of an analyte is monitored as a function of its temperature or time and for the result two types of curve can be obtained, one is mass wt.% versus temperature and if the temperature is available in terms of ^{0}C/min the mass versus time curve can also be potted.

The TGA 5500 from TA Instruments Inc. is used to carry out Thermogravimetric analysis which possess ambient to 1200 ^{0}C temperature range and linear controlled heating rate of 0.1 to 500 ^{0}C/min.

A sample of 1 kg mass is placed in the machine in the presence of air as environment. The temperature is increased from room temperature to 1000 ^{0}C at a rate of 10 ^{0}C/min. The plot of mass change in percentage versus temperature or time (known as TGA curve) is typical result of TGA analysis.

4.4 Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry, or DSC, is a thermal analysis technique that looks at how a material's heat capacity (Cp) is changed by temperature. A sample of known mass is heated or cooled and the changes in its heat capacity are tracked as changes in the heat flow. This allows the detection of transitions such as melts, glass transitions, phase changes, and curing. Since we work here with polymeric material, the glass transition is important to understanding the material [27-30ss].

At first the sample is prepared with the composition of 63 wt.% of SiO_2 only because this composition is found best in all other tests of mechanical properties. The sample size is selected as 10 mg. To neutralize any antecedent effect of temperature sample is preheated to 200 ^{0}C at a constant heating rate of 25 ^{0}C/min subsequently it is quenched to -25 ^{0}C. Differential Scanning Calorimeter (DSC 4000 System, 100-240V/50-60Hz by PerkinElmer) is used to measure heat flow from -25 ^{0}C to 225 ^{0}C at 25 ^{0}C/min.

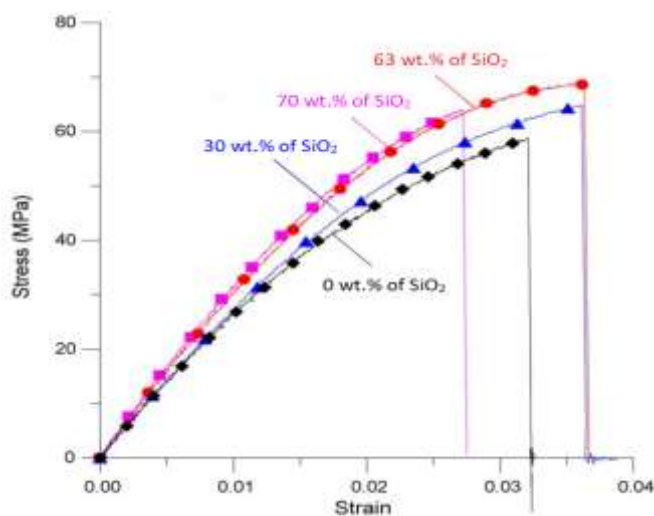## 5. RESULTS AND DISCUSSION

5.1 Tensile strength

Chart -1: Effect of SiO_2 on tensile stress-strain curve

Four specimens of DGEBA were prepared with different percentage of SiO_2 according to ASTM D638 standard and the test is performed. The values obtained from the test were plotted on stress strain curve. The tensile properties were tabulated as shown in table-1s. In all of the four cases after the maximum stress value is reached the specimens took very subordinate amount of time to fail. Though no apparent yield point can be decided from the results the curve deviate from straight line to some extent prior to reaching the maximum stress.

Based on the table-1 and chart-1 it can be said that as we go on increasing the SiO_2 percentage in DGEBA its strength increases. Out of all the compositions the one with 2 wt.% of SiO_2 is by far the best with 18.32% gain in strength. If the percentage is further increased it does not show that much improvement, But the modulus increases almost steadily with weight percentage of SiO_2.

Table -1: Tensile properties of SiO_2 modified epoxy

Materials	Strength (MPa)	Gain in strength (%)	Modulus (GPa)	Gain in Modulus (%)
Neat epoxy	59.43±2.32	-	2.73±0.12	-
30 wt.% SiO_2	65.34±0.43	09.94	2.85±0.32	4.39
63 wt.% SiO_2	70.09±2.52	17.93	3.12±0.18	14.28
70 wt.% SiO_2	64.43±3.08	08.41	3.42±0.22	25.27

5.2 Fracture toughness

The four sample which are tested for fracture toughness contains unadulterated, 30 wt.%, 63 wt.% and 70 wt.% SiO_2 nanoparticles and the calculated K_{IC} values for those materials are delineated in the graph.

The unadulterated DGEBA sample have the lowest fracture toughness value out of all the four samples and the third sample which contains 63 wt.% of SiO_2 nanoparticles have the highest K_{IC} value that is 17.82. It is observed that as we go on increasing the weight percentage the beyond that the K_{IC} value start to reduce.

Since lower density of cross links leads to increase in ductility of material it can be a cause for increment in critical stress intensity factor (K_{IC}) in other sample, but it is not the sole effect of density of cross link there may be other factors such as the microstructure of the epoxy changes with addition of SiO_2 and becomes more heterogeneous in nature this may automatically leads to lower density of cross links or it may have separate effect in the form of increasing toughness.
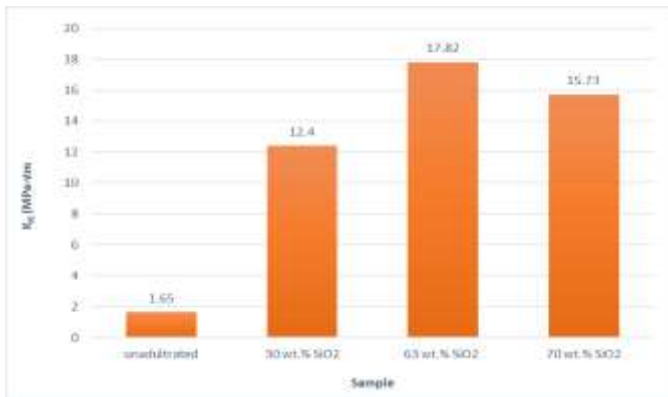
Chart -2: Fracture toughness (K_{IC}) values off samples

5.3 Thermogravimetric Analysis (TGA)

The chart-3 shows the mass change in percentage versus temperature TGA curve. From the graph the nature of curve can be considered as multistage decomposition curve. This curve reveals the multistage decomposition processes as a result of various reactions but the stages are not clearly defined in the curve due to fast heating rate or no intermediates.

The general causes of weight loss may be desorption, drying, dehydration, desolvation, sublimition, vaporization, and solid-state reaction in some cases, but in our specimen dehydration or drying is not possible because the silica nano-particles as well as the epoxy resin were heated to 150 ^{0}C and kept there for 2hrs to eliminate moisture so there is no moisture present in the analyte. The major causes of weight loss might be sublimation and vaporization and desorption may have contributed to some extent.

Four samples of different composition percentage of SiO_2 in the epoxy resin were placed in machine and Thermogravimetric analysis is performed. The machine automatically produces the curves for respective sample as shown in chart-3. First, we defined temperature for start of decomposition as 5% reduction in mass. The start of decomposition for the analyte samples that is for 0 wt.% (unadulterated), 30 wt.%,63 wt.% and 70 wt.% is at 221 ^{0}C, 246 ^{0}C, 265 ^{0}C, 254 ^{0}C respectively.
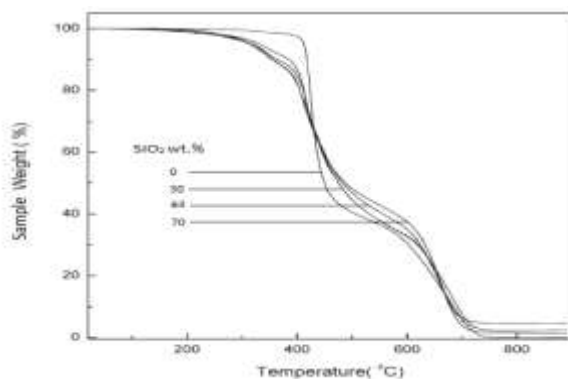

Chart -3: TGA curves

5.4 Differential Scanning Calorimetry (DSC)

The inflection point temperature from the chart-4 gives the glass transition temperature of Diglycidyl ether of bisphenol A (DGEBA). The glass transition (T_g) has been called the "melting of amorphous material" and as unscientific as that is, it's an adequate description. In non-crystalline and semi-crystalline polymer of any type – synthetic high polymers such as polypropylene and polystyrene, natural polymers like rubber, or biological polymers such as proteins – the glass transition is the best indicator of material properties. As the glass transition changes due to either different degrees of polymerization or modification by additives, the physical properties of the material change. Similarly, material properties also change dramatically above the T_g. The hard and solid nature of sample turns to soft and mobile like jelly. The value of T_g for DGEBA sample is 56.23 ^{0}C.
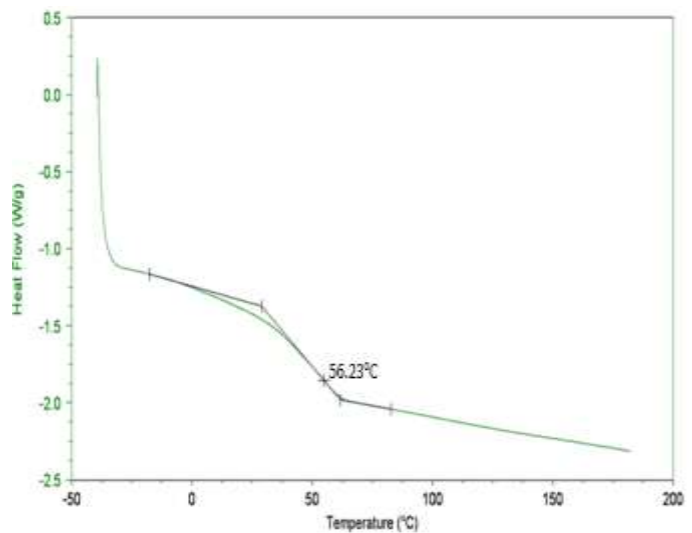

Chart -4: DSC curves

6. CONCLUSIONS

Mechanical, chemical and thermal properties were experimentally investigated on Diglycidyl ether of bisphenol-A based SiO_2 modified epoxy resin composite as per the experimental results following conclusions were reached.

   1. Highest gain in strength of 17.93% is obtained from 63 wt.% of SiO_2 and for the same gain in modulus is 14.28%.

   2. Highest fracture toughness of 7.82 among all the materials obtained from 63 wt.% of SiO_2.

   3. Starting temperature of decomposition of sample with 63 wt.% of SiO_2 is 265 ^{0}C which differ by 44 ^{0}C from unadulterated sample.

   4. TGA result shows that DGEBA was fully degraded above 600 ^{0}C in both air and nitrogen atmosphere and transition temperature T_g=56.23 ^{0}C.

The analysis of samples revealed that when 63 wt.% of SiO_2 is mixed with the composition mentioned in material gives modified properties.

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