



Studies on Mechanical and Thermomechanical Properties of Haematite Particle Reinforced Polyester Composites

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Abstract—In the present study, the purpose was to investigate the effect of haematite filler particles, having less than 100 μ m reinforcing into polyester resin. The behaviour of reinforcing fillers investigated under SEM and EDAX test. To find out mechanical and thermomechanical characteristics compression test and DMA tests have been carried out respectively. The fillers were varied in their weight percentage i.e. 0%, 2%, 4%, 6%, 8% and 10% by keeping matrix polyester resin constant for all cases. The SEM test shows that haematite particles do not have regular shape hence there maybe adhesion between matrix phases. The EDAX test shows that iron content is more compared to carbon and oxygen hence particles gather together due to strong interparticle magnetic dipole attractions. Maximum compressive strength is obtained for 8% w/w is 169 N/mm². The glass transition region T_g obtained for 8% and 10% w/w are 91.49 °C and 92.61°C respectively. Cole-Cole plot have been shown homogeneity of particle matrix adhesion.

Keywords— Polyester composites, haematite particles, compressive strength, DMA and SEM-EDAX test.

I. INTRODUCTION

In composites the foundation of classification, as matrix (polymer, metal and ceramic composites) and in foundation of reinforcement, as (optical fiber, particle and laminated composite). In a material of two stage, a stage as the material of matrix is a polymer and second stage as a scattered stage as the particle of reinforcement. A scope wide spread various forms of behaviour can obtain the matrix and reinforcement material that and combine [1]. Particle polymer matrix composites will possibly present different attributes, such as influence, forced induction, electricity and hot conductivities and many more [4] with [5]. The particle that the interaction between matrices and strengthens through their stages is not a simple concept, in area that the polymerization composes, imperfect cementation, because contracts the mechanical stress, hole and micro crack or even as a result of containing of geometric solid. It assigns matrix - particle interphase has had important influence the transfer matrix and reinforcement of between mechanical complete compound achievement its unique attribute-controlled loadings. However, its behaviour brittle, compares the metal and has the low heat and limited physical characteristics. The pellet of reinforcement has the low beam to depth ratio to improve its some mechanical attributes [12]. In order to realize the compound materials of optimum performance, this is actual load transmission entire matrix a connection of particle is necessary [14]. Among space the particle in each stage was supposed to be full of the matrix, blank that is also impossible to have. In this case, matrix volume at least is a system and behaviour of assignment is some unique segment markets, to support to bring the compression load [16]. The dynamic mechanical analysis is a very useful method, research the composite construction of behaviour. It had been shown is an effective method, investigates, the polymer that relaxes thus studies the behaviour under the pressure in the composites of particle and temperature of material dissimilar condition and composition. All above confirmation its functions in definite machine capability. Recent DMA, brings to many attention, because after it has provided the storage modulus, the loss modulus and tan Delta, the flexibility of main chain (T_g) carries on the survey, analyses is carried out. Similarly, the definition of T_g is temperature, a biggest mechanical antivibration parameter (tan Delta) or a loss

coefficient (E ") (18-20). In our research, Tg carries on the foundation of appraisal, the tan Delta curve of maximum peak value.

II. EXPERIMENTAL DETAILS

A. Experimental details

The matrix was an Orthophthalic acid based general purpose polyester resin with catalyst as MEKP (methyl ethyl ketone peroxide) and accelerator (cobalt naphthanate). Polyester resin is low cost standard economic resin being viscous low density and has good mechanical properties. Filler material is haematite particles(Fe_2O_3) less than $100\mu m$. Haematite were harder than iron but it is brittle. It has good mechanical properties. Materials were brought from Zenith stores, SP road, Bengaluru. The following figures shows polyester resin with hardeners and haematite particles.



Fig. 1 Polyester resin and haematite (Fe_2O_3 particles)

B. Fabrication of specimens.

The samples were manufactured by adding different amounts of particles (i.e. from 0% to 10% in weight fractions of total mixture) to the matrix. For both tests samples were manufactured using simple moulding (open moulding process). The mould is composed of wooden strips on the flat surface of granite for laminates of dimension $100X70X5$ mm and PVC pipes for cylindrical surface dimension $\varnothing 20X40L$ mm it is as shown in figure. The boundary surfaces were coated with wax for easy removal of material. The wooden strips were mounted on granite surface by pasting glue tape for laminates and PVC pipes were mounted using clay on outer surface. Mixing was done manually stirred for 4-5 minutes and then poured into mould as shown in figure 2-3.

After removal of material specimens were kept for post-curing process ($80-120^\circ C$) with an interval of 1 hour each. For good bonding between particles and matrix and to improve properties post-curing process was done. For DMA test samples were prepared by water jet cutting process of dimension $50X5X4$ mm.



Fig. 2 Mould prepared for laminates and cylindrical surface specimens.



Fig. 3 Specimens fabricated in mould.

C. Compression test

Under a behaviour difference of compression test material serious load. Carries on the attention of compression and distortion value in the different variable load specimens. The pressure load and strain have described, tabulated as a stress-strain diagram, is used to determine the elastic limit, the proportional limit and yield point and compressive strength. The axial compression test is a useful procedure, by determining the plastic mobile behaviour, needs the isomorphism the compression test condition. When the surface tube-surface of this centre has the friction compression carries on, but the nodular graphite casts the defeat to limit using the tube forms and under the pressure and tense condition of control. The elasticity and resistant to compression breakdown behaviour attribute brittle material or low ductility the material for survey, the different strains in the compression test axial compression test are very useful. In all circumstances, the specimen of use should prevent the L/D ratio (i.e. large L L/D = 2), to prevent the distortion that the cutting pattern and threads up. The compression test, was conducted related has tested the loading rate of machine at 2 mm/min.

D. Dynamic Mechanical Analysis. (DMA).

The polymer composite of dynamic assessment property a trend dynamic mechanical analyzer (DMA 8000Perkin elimer) are employed. The rectangular specimen has the size is 50 X 5 X 4 mm, for test. The dynamic storage modulus, the loss modulus and dissipation factor survey as a temperature range of function the heating rate of 30 to 110°C expressing is 2 °C/min, the frequency range from 0.1 hz to 1hz. This experiment in a pressure amplitude of three-point bending tests is 0.05%. Saved the modulus to describe viscoelastic behaviour of material of degree hardness and has become certain proportion store energy cycle of in the loading process. Loses the reason of energy the loss modulus expressed is the dissipative flow. Proportionality coefficient of loss, to save modulus giving tangent value phase angle Delta, tan Delta, this is the so-called damping factor, and may be regarded as a material of energy consumption.

III.RESULTS AND DISCUSSIONS

A. DMA Test Results

Storage Modulus

Dynamic modulus of change as a temperature of function, has the polyester composite materials by Fe₂O₃ that the neat polyester and strengthens. In all cases, matrix is to maintain constant, reinforcement 2%, 4%, 6%,8% and 10% w/w. The material of dynamic machine capability composites can have the obvious change, even if a small change in the particle of chemistry and physical property. Saves the modulus to express the material of degree of hardness (5). The chart may be divided into three different regions; leathery, glassy and rubbery region. As increasing of temperature saves module to reduce, all composites, this may put the blame on the polymer chain of molecular mobility will increases [17]. Storage modulus, will discover the surrounding glass transition region (40-100°C) Regarding neat polyester (45-100°C). The influence, strengthens the Fe₂O₃ loads the dynamical properties of composites, as illustrated below expressed in 1 hz. What is obvious, the influence micro reinforcement of storage modulus. Compared with neat for 2%, 4%and 6% have been dropped, neat polyester (in 40-90 ° C). For 8% and 10%had enhancement, from (in 45-100 ° C). In addition, was 8% and results of 10% Fe₂O₃ particles increases the storage modulus is 9- 7.477.0e e9 Pa from the 6.56e9 Pa pure polyester. 8% and 10%, bringing the pellet that w/w loads to display the highest physical characteristics. When Contrariwise, saves the modulus 2%, 4% and 6% still

maintained low compared with pure polyester. The reason of drop possibly is the interaction between low particles, but does not intend to precipitates to the interaction of matrix; Interaction between these members has caused the accumulation of granule, therefore quality bad physical characteristics.

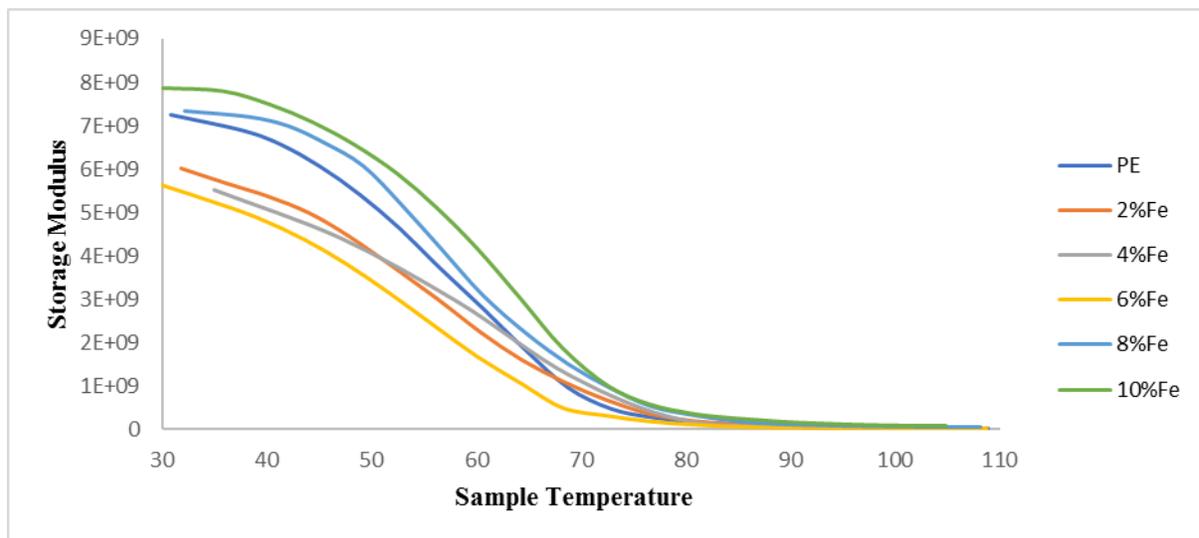


Fig. 4 Storage modulus vs sample temperature

Loss Modulus.

The loss coefficient is to weigh radiation as the weight energy, namely material of coherent response. The effect in the chart demonstrating, the Fe_2O_3 strengthening polyester matrix composites of temperature modulus value to loss. Has given wide spread peak value temperature range 45-60 ° C in the neat polyester. Loss coefficient curve display the trend and in peak region that all the situations of reinforcement made one be interested, temperature range 45-65 ° C. Compared with the peak value, various types of ingenious polyesters 2% and 6% w/ws, as shown, compared with is low, this possibly as a result of bad cementation. Can note, the Tg value of obtaining from the loss modulus of maximum peak value 8% and 10%w/w, brings to increase (approximately 2-4 °C) compared with, neat polyester (~ 56.51 °C). The final effect is the reinforcement enhances the density and face area. The effect of reinforcement will depend on cementation and matrix, but bad cementation effect, material of energy consumption. The Tg change value is the tabulation in Table 1.

TABLE 1 LOSS MODULUS Tg VALUES FOR REINFORCEMENTS

Sl. No.	Reinforcements	Loss modulus, Tg in °C
1	Neat polyester	56.51
2	Polyester+2%	56.25
3	Polyester+4%	58.12
4	Polyester+6%	55.51
5	Polyester+8%	58.91
6	Polyester+10%	60.09

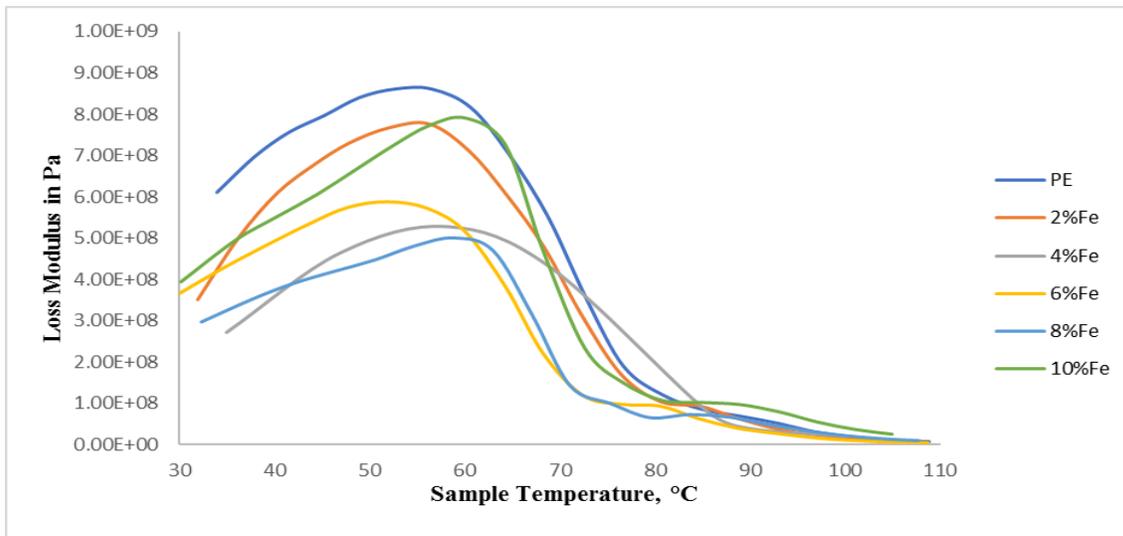


Fig. 5 Loss modulus vs sample temperature

Tan Delta

The antivibration is a dynamic behaviour precipitation strength composite construction of important parameter relevant research. Antivibration changed temperature. Tan Delta peak value discovered high, w/w % the reinforcement. Table 2 had demonstrated the Tg value is the reinforcement. In this case, the only peak value appears in high temperature range has been appraising. Was discovered as 8% w/w compared with high biggest Tg with 10% w/w, compared with neat polyester (increase of approximately 4°C). Compared with the low value Tg, the neat polyester 2%, 4% and 6% w/w. The Tg low value, the reason is the agglomeration or the invalid content on the material. This expressed the tan Delta curve.

TABLE 2 TAN DELTA, Tg VALUES FOR REINFORCEMENT

Reinforcement	Glass transition value, Tg °C
Neat polyester	88.92
Polyester+2%	88.37
Polyester+4%	85.69
Polyester+6%	88.01
Polyester+8%	91.49
Polyester+10%	92.61

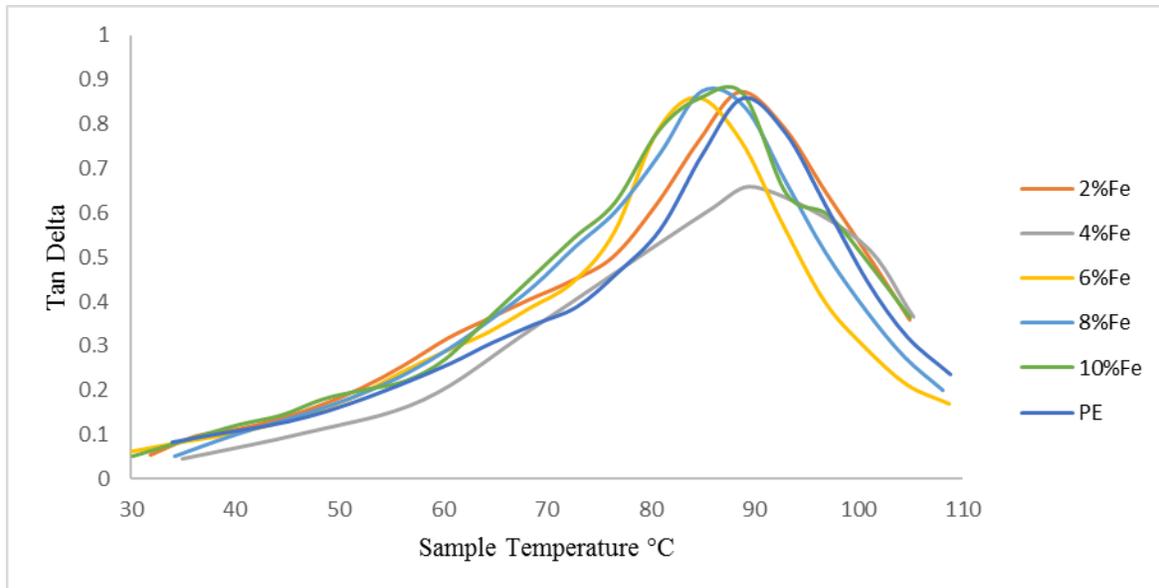


Fig. 6 Tan delta vs sample temperature

1. Cole-Cole plots.

A material of serious polarization for Debye of representative, dielectric Onsager equality. A mountain peak of relaxation is not full, enumeration polymer of viscoelastic response. Plot that Cole-Cole is dielectric relaxes the result of obtains E'' to E' , frequency of each point correspondence. After cross-linked polymer pellet of structural change, except for the polymer matrix can determine that uses the Cole-Cole method. In the chart had demonstrated Cole-Cole plot, loss coefficient data $\log E''$ the value was indicated as the function memory module diary E' . The nature of this land, represents the nature the system. The polymer system of homogeneity it is reported that is representing -and-a-half ring charts (10-11). The adhesion of semicircle shape matching good micro matrix in the figure.

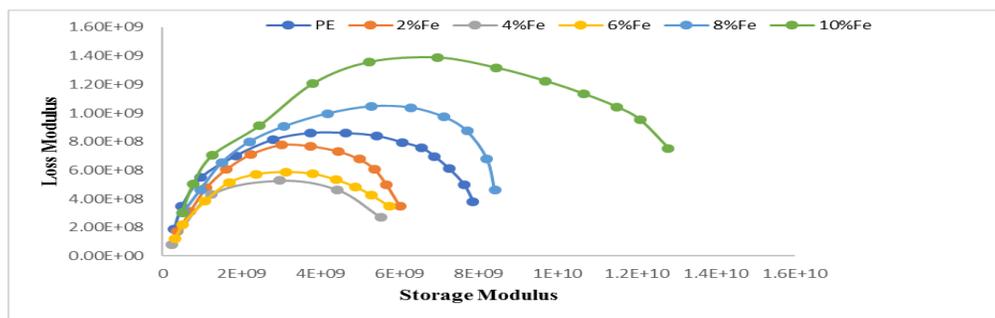


Fig. 7 Cole-Cole plot

B. Compression Test Results

Compressive Strength.

The pressure that the performance of this material, withstands is by the strength test, such as compression test. The test showed that the ability of material, underwent the behaviours of other application compression loads and machineries. Preparations of these experiments, according to D695 ASTM standard, and encounter the compression test in UTM. The specimen level, the goal is the $\varnothing 20 \times 40$ L mm, weight treatment the particle of at different times 2%, 4%, 6%, 8% and 10%. The following table 3 and graph has provided the end value of experiment. In this study, compressive strength is increased for increasing w/w reinforcements up to 8% w/w and decreased for 10% w/w reinforcement. In all cases compressive strength is more compared to neat polyester.

TABLE 3 COMPRESSIVE STRENGTH FOR REINFORCEMENT

Sl. No.	Weight fraction %	Compressive strength in N/mm ²
1	Polyester+ 0%	124.68
2	Polyester + 2%	135.28
3	Polyester + 4%	147.75
4	Polyester + 6%	159.09
5	Polyester + 8%	169.15
6	Polyester + 10%	157.18

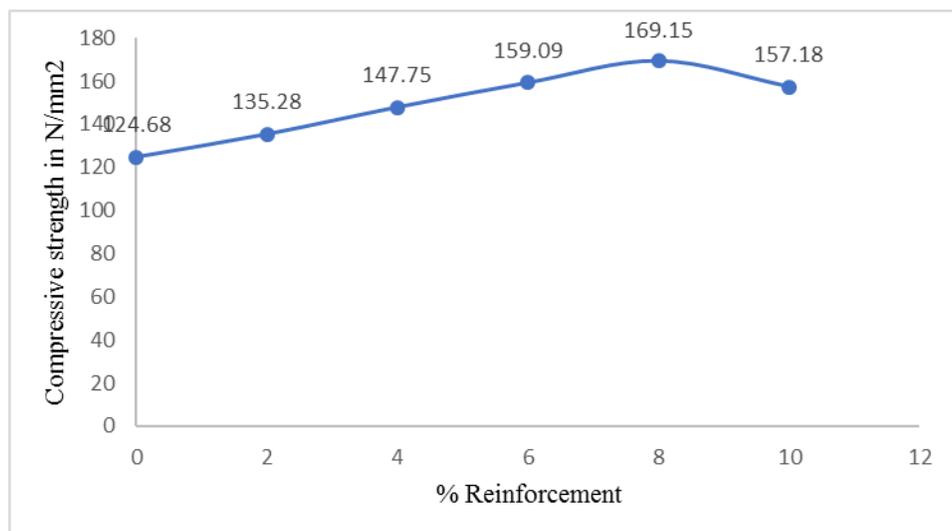


Fig. 8 compression strength vs % reinforcement

C. SEM-EDAX Tests.

Scanning Electron Microscope (SEM) and Energy Dispersive Analysis of X-ray (EDAX) was obtained using TESCAN Vega 3 LMU with Secondary Electron Detector. The SEM test revealed the microscopic structure of the specimen at surface level for 8% and 10%. It has shown the presence of materials with bonding of matrix and reinforcement material. The EDAX test revealed that they consist of iron content is more that of carbon and oxygen in 10% haematite reinforcement they tend to form large agglomerations. Figure 9 and 10 shows images and table 5 shows EDAX result.

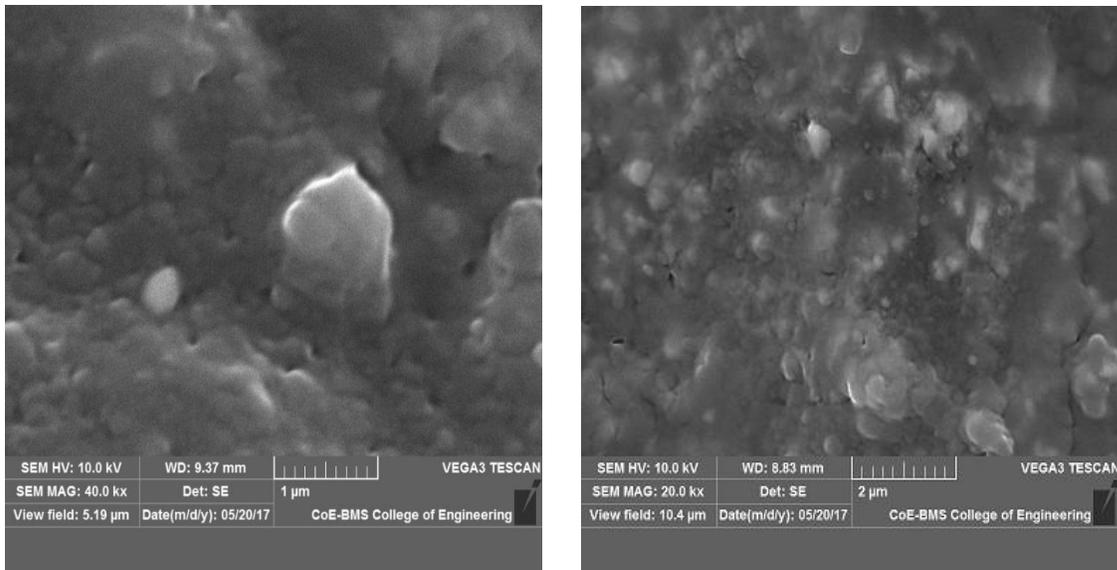


Fig. 9 SEM images of 8% and 10% reinforcement.

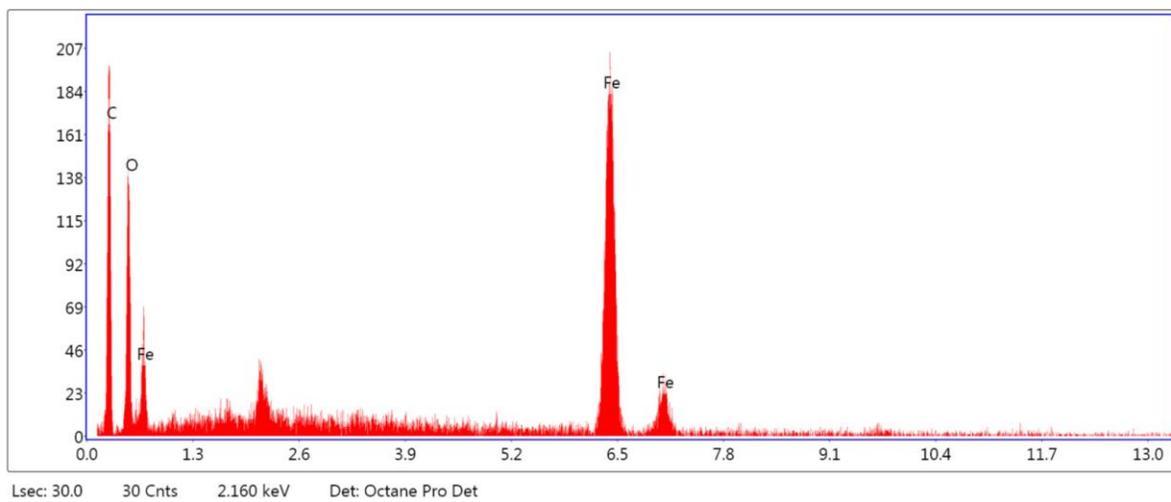
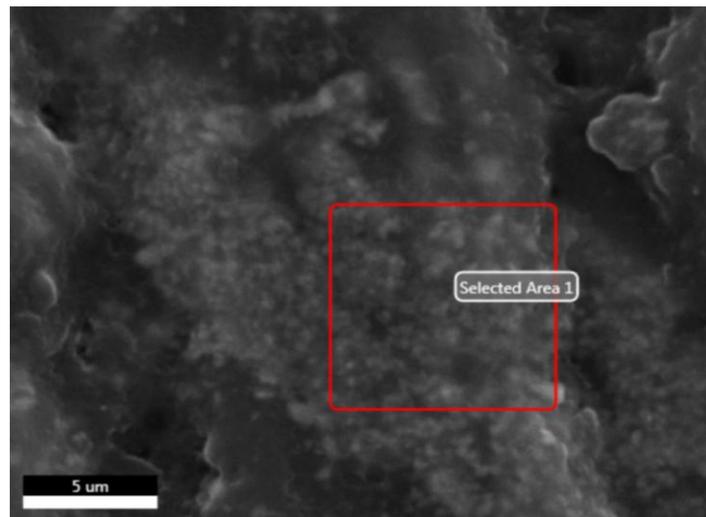


Fig. 10 EDAX image for 10% reinforcement

TABLE 4: EDAX RESULTS OF 10% HAEMATITE REINFORCEMENT

Element	Weight %	Atomic %	Net Int.	Error %	Kratio
C K	38.04	61.93	62.17	10.02	0.1461
O K	18.78	22.95	47.01	11.67	0.0468
Fe K	43.18	15.12	175.36	3.70	0.3762

IV. CONCLUSIONS

The fabrication and determination of mechanical and thermo-mechanical properties of polyester based Fe₂O₃ reinforced composite leads to following conclusions:

The successful fabrications of a new class of polyesters based composites reinforced with Fe₂O₃ particles have been done. The present investigation represented that reinforcements significantly influence different properties of composites. The maximum compressive strength 169.15 N/mm² is obtained for 8% particle reinforcement.

The maximum glass transition region T_g 91.49 °C and 92.61 °C is obtained for 8% and 10% particle reinforcements respectively. Cole-Cole plots represent homogeneity of composites that it shows polyester composites has good viscoelastic behaviour.

The SEM and EDAX test has shown the behaviour of particles and matrix adhesion at surface level for 8% and 10% SEM images shown that Fe₂O₃ particles are mixed with matrix phase. EDAX test revealed that iron content is more compared to carbon and oxygen content so this concludes that particles tend to agglomerate due to strong interparticle magnetic dipole-dipole attractions.

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