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Studies on Dynamic Mechanical Properties (DMA) of Nickel Nanoparticles in Polyester Matrix Composites.

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Abstract: This paper describes the study of Dynamic Mechanical Properties of Nickel Nanoparticles reinforced in Polyester Matrix in different proportions. The study is performed by fabrication of specimens and preparing by water jet cutting and tested for DMA properties. The samples are weighed before and after conducting and weight loss of the samples are also recorded. The properties of Storage Modulus, Loss modulus and Tan Delta are noted and analysed. Elemental analysis was done by EDAX and SEM studies were done. This work describes the properties of composite which behaves as a result of bonding between the Nanoparticles reinforcement in the polymer matrix. The properties vary as per the filler reinforcement providing varying properties.

Keywords: **Dynamic Mechanical Analysis, Nickel Nanoparticles, Polyester, Glass Transition Temperature.**

I. INTRODUCTION

Polyester resin is in use for many researchers for applications in many fields. Its properties have increased after reinforcing fibres and fillers into it. Natural fibres additions have improved its properties in many ways. Bagasse[1], pineapple leaf fibre[2], cotton/kapok fibre[3], jute[4], coconut[5], glass fibre[6] have been reinforced in polyester and many properties have been studied. New techniques and developments are also discussed in terms of natural fibres studying their mechanical and interfacing properties.[7-11]. Polyester resin is also tested for various properties using filler particles like fly ash, natural particles, macadamia nutshell particle,Al2O3. [12-15].

Though the variety of materials has been added in polyester resin the research works are limited with Nanoparticles reinforcements. The strong mechanical properties of metal Nanoparticles have to be studied with all conditions and therefore we are using metal particles as reinforcements. Silver Nanoparticles [16]and Iron oxide Nanoparticles [17] has been used in polyester for determining its properties. Nickel Nanoparticles has been used in polyester fabric and tested for magnetic properties [18-19]. The magnetic properties of nickel Nanoparticles can be determined in many ways using SQUID, VSM magnetometers. [20-23]. the limited use of Nickel Nanoparticles in the above literature unique properties has to be determined. Other than simple mechanical properties the material properties with temperature provides more insight to material behaviour in elevated temperatures and its mechanical efficiencies. With DMA, the Glass Transition Temperature can be found. The Dynamic Mechanical Analysis (DMA) performed on a material provides important information of material behaviour with respect to the Modulus variation with the temperature. DMA test provides more insight on mechanical modulus and temperature dependant behaviour. The deformation applied on the sample in a cyclic manner and the DMA results are recorded in form of graph. When an oscillatory force is applied on the sample, the changes in stiffness and Damping is recorded. The storage modulus, loss modulus and tan delta values provide details of modulus information, mechanical properties in frequency range, sensitive glass transition temperature, Curing reactions, impact of fillers, and interaction of the fillers and matrix in various conditions. [24-32]

II. MATERIALS AND METHODS

A. Materials.

Nanoparticles (Nano-labs, India, <50nm) was used as reinforcement. Table 1 shows the prominent properties of Nickel Nanoparticles, and Table.2 shows properties of Polyester and Hardener(As provided by supplier).

TABLE 1.
PROPERTIES OF NICKEL NANOPARTICLES.

Material	Average Size	Purity	SSA	Morphology	Supplier	Bulk Density	True Density	Molecular Wt
Nickel Nano particles	40-50nm	99.5%	12-16m2/g	Spherical	Nano labs, India	0.08-0.20 g/cm3	8.902 g/cm3	58.69

TABLE 2.
PROPERTIES OF POLYESTER AND HARDNER.

Material	Type	Viscosity at 25oC □(cP)	Density at 25oC	Supplier	Hardner/Accelerator	Specific gravity
Polyester	Orthophtalic	250-350	1.10 g/cm3	Zenith, Bangalore	MEKP/Cobalt	1.13

The catalyst is used as Hardener. The catalyst initiates a chemical reaction which allows the resin to transform into a solid state. It is also called as "curing" or "polymerization." Methyl Ethyl Ketone Peroxide (MEKP) is the chemical hardener used with polyester resin. The catalyst is added only less amount only, (approx2ml of catalyst per 100gm of resin). All precautionary care must be taken while fabricating composite and using the resin and chemicals.

B. Preparation

Polyester based Nickel Nanoparticles reinforced composite material was prepared by reinforcing Nickel (Ni) particles (<50nm) by 0%, 0.3%, 0.6%, 0.9%, and1.2%, (P0,P1,P2,P3,P4) weight fraction that of Polyester was prepared by casting in a mold cavity by open mould process. Polyester and Nanoparticles were weighed properly for the above said percentages and using rule of mixtures, samples were prepared. A wooden mould (cost effective) was prepared for size 70mmx100mmx6mm. The wooden strips were then placed on a granite plate maintaining horizontal position of mould. Wax was coated on the inside and boundaries of the mold prepared. This wax provides coating for the casting to be removed easily after curing. Nickel Nanoparticles for different percentages were mixed thoroughly in Polyester before adding catalyst and accelerator and was stirred mechanically. This stirring continued till uniform distribution was observed. After thinning of Polyester and uniform distribution of Nanoparticles, Catalyst (MEKP) for 2% and Cobalt accelerator for 1% was added for resin. Again stirring continued for uniform distribution of hardener and the mixture was poured into the mold cavity (Figure 1.a) concentrating the pouring to a point. The mixture occupies the mould dimensions. The mould was allowed to cure at room temperature for 24 hours. The prepared mould were investigated for any damages or complexity and were removed carefully form the mould (Figure 1b). As wax was coated the samples were removed easily (Figure 1c). The samples were then post cured for 80oC for 3 hours in a conventional oven. Post curing was done to ensure the completion of cross linking of polymer chains with the Nanoparticles. The composite material prepared was carefully removed from oven after cooling down naturally for 12 hrs. The material was then cut by water jet cutting (Figure 1d) for required dimensions according to ASTM standards for DMA.

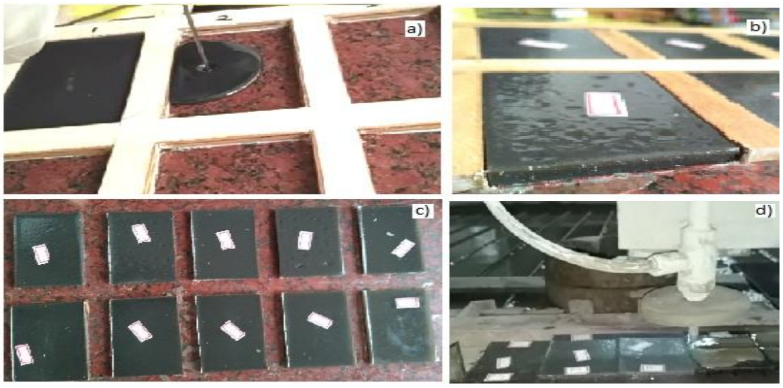

Figure 1. a) Pouring Mixture into cavity b)removing prepared samples c)Samples d)Waterjet cutting for required dimensions.

III. CHARACTERISATION.

A. DMA-Dynamic Mechanical Analysis

The apparatus used is Dynamic Mechanical Analyser, Perkin Elmer 8000 Figure 2a). DMA is a technique where small deflection is applied to a sample in cyclic manner. This allows the material response to stress, temperature frequency & other values to be studied. The DMA plots the Loss Modulus, Storage Modulus and Tan Delta with respect to change in temperature. This test method is designed to determine the glass transition temperature of continuous fiber reinforced polymer composites by identifying peak values of Loss Modulus. The DMA Tg value is frequently used to indicate the upper use temperature of composite materials, as well as for quality control of composite materials.

Parameters	Values
ASTM Standards: DMA test specimen	50×6×(3-5) (mm) ASTM D 7028.
Type of test	3 point Bending test. (Figure 2b)
Load	Static Load-1N, Dynamic Load-2N.
Frequency	1Hz
Temperature range	30-150oC
Rate of Temperature	5oC/min

TABLE III.MACHINE SETTINGS FOR DMA

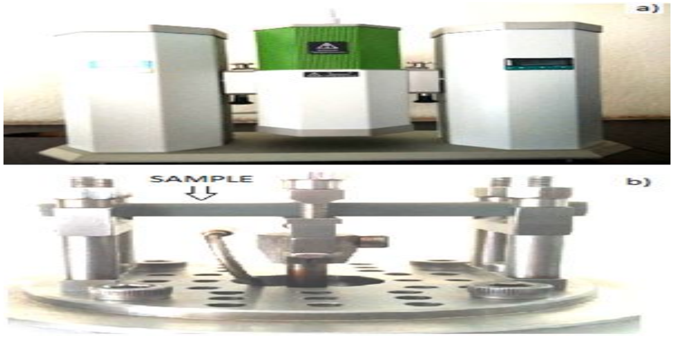

Figure 2.a) DMA apparatus PERKIN ELMER8000.b)Sample fixture for 3point bending test.

B. EDAX & SEM Analysis

The constituent elemental analysis by EDAX and SEM analysis is performed on the sample and powders and is studied. The Table4 shows the parameters considered in the TESCAN Vega 3 LMU machine.

TABLE 4.
PARAMETERS OF SEM APPARATUS

SEM Make	Magnification	SEM HV Range	Working Distance	Make
TESCAN VEGA 3 LMU	10,00,000X	5-30kV	1-40mm	Czech Republic

IV. RESULTS AND DISCUSSION:

A. *Weight Analysis*

According to ASTM standards the samples were weighed before and after the DMA test. The weight loss for a particular combination is represented as average of two samples. The results are represented in the Figure3 for all combinations.

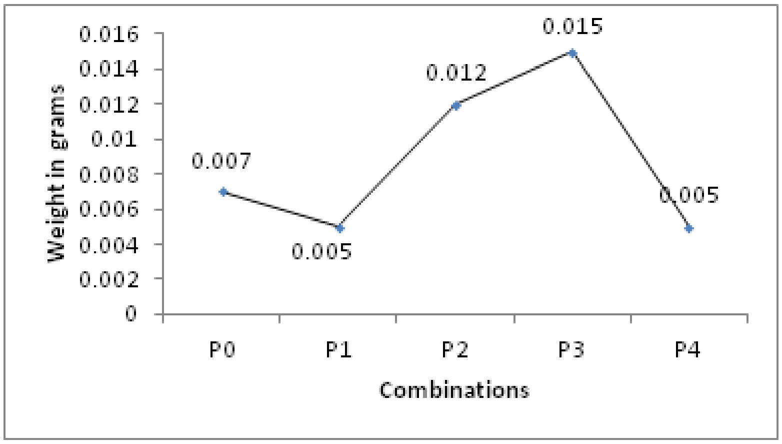

Figure 3. Average Weight Loss difference in samples (DMA test)

The prepared sample is weighed before and after DMA had some differences. It was clearly noted that the sample weight was significantly reduced after DMA testing. This is due to the thermal stability of polymer. The plain P0 and P1 combination itself has major difference in the particle addition. With the addition of nanoparticles for P1 combination the weight loss is less. With the further addition of particles, with P2, P3, and P4 combinations majot weight loss differences are noted. Highest weight loss is noted in P3 combination. This is due to the bonding between the fillers and matrix. The graph also shows that the nanoparticles reinforced in a polymer matrix, affects the performance with respect to temperature and loading. The weight loss is signficant in understanding the particle reinforcement in polyester matrix and its bonding behaviour.

B. *Storage Modulus*

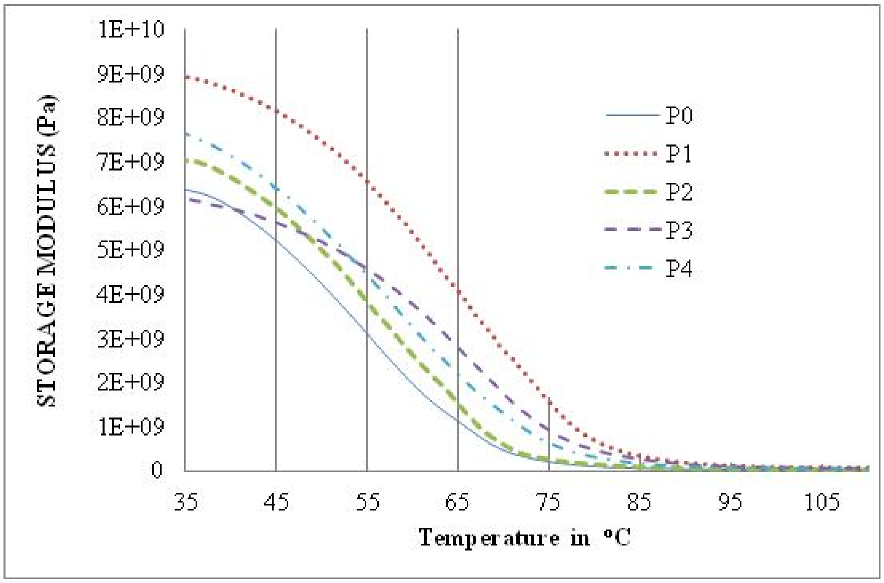

Figure 4. Storage Modulus vs Temperature

The above Figure 4 represents Storage modulus vs. Temperature of all combinations. Storage Modulus (E’) is the stiffness of a sample and it is the energy stored in a sample during the loading cycle. The drop in the storage modulus curve describes the change in state of the material from glassy state to rubbery state. [27]. It also indicates the physical softening of the material [27].Also it indicates that the material heated beyond this point will be poor in performance [27]. This sigmoidal change has to be considered for the analysis. The drop in Storage modulus is around 40^{o}C for P0 and around 45^{o}C for P1 and reduces to 45^{o}C for P2 again. For P3 it is 55^{o}C and P4 is45^{o}C. During deformation, the stored energy reflects materials elasticity, because of elastic deformation. It provides the ability of resistance to dynamic deformation [28]. The storage modulus of P1 combination was three orders higher than P0. This is also accounted by D.Hu et al [28]. The storage modulus is around 600MPa for P0 at 30^{o}C and for first reinforcement combination it has increased to around 900MPa 30^{o}C. By further addition of filler, the storage modulus decreases [28]. Behaviour of storage modulus variation is also accounted by Margem et.al. [29,31]. As the temperature increases the storage modulus decreases showing the mobility of components are loosely packed in rubbery region. High storage modulus is accounted when the temperature is less and the components are packed together and provide high storage strength. [33]

C. Loss Modulus

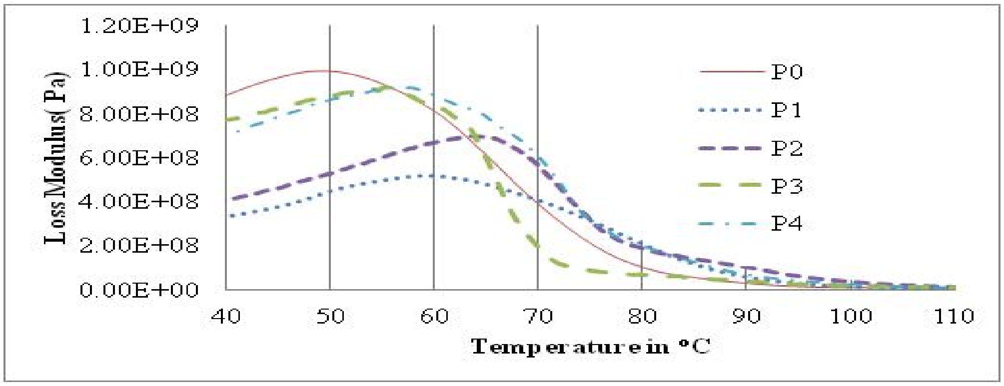

Figure.5. Loss Modulus vs Temperature

The above Figure5 shows Loss modulus represented with function of temperature for all combinations. Loss modulus E’’ peaks determines the physical property changes occurred in a specimen which relates to glass transition Temperature (Tg). [26]. Loss modulus is the measure of energy dissipated in the form of heat per cycle for every deformation. This glass transition occurs while heating the sample from glassy state to rubbery state and material becomes more flexible and soft. This is the temperature at which the molecular chains become loose while heating. The same will be vice versa while cooling. [27]. In this region there is a peak value of temperature. It is observed that all other combination provides different peaks with respect to plain polyester P0. Due to chain mobility of particles the peaks suggest the structural relaxation behaviour of composites [29]. It is observed from the graph that for pure polyester, P0 peak value is 50^{o}C, and for first reinforcement, P1 combination peak value is62^{o}C, which is around 20% increase in temperature. This gradual increase may be due to the increase in resistance in filler material [33]. For P2 it is 68^{o}C and P3 63^{o}C and finally P4 combination it is reduced to 60^{o}C. The Tg values obtained from the peaks have to be studied along with Tan Delta peaks that determine damping of the material. The peak values are represented in a form of graph for easier identification. Figure 6(a) shows the peak values of Loss modulus.

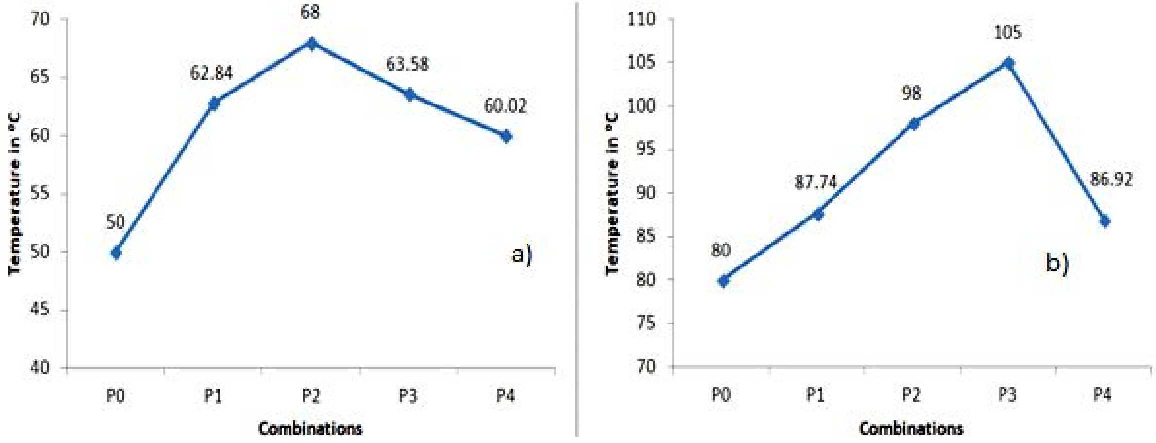

Figure 6.a). Loss modulus Peak b) Tan Delta Peak

D. Tan Delta (Damping factor.

Figure7 represents Tan Delta of all combinations. Tan Delta is the ratio of Loss Modulus (flow) to Storage Modulus (stiffness).The peak in tan delta region occurs at higher temperature and is important. The Tan delta peak is accounted for determining changes in sample that depends on filler reinforcement [26,27]. It also provides the damping behaviour of the material. The higher the delta value, the material is more viscous. If tan delta is approaching 0, then elastic behaviour is observed [24-27]. The peak values are represented in the Figure 6.b). It should be noted that the Tan Delta values differ for P0 and it decreases gradually for all other combinations. The peak Tan Delta region increase from 80^{o}C to 87^{o}C for first filler reinforcement. And increases gradually and peak value of 105^{o}C is observed in P3 combination and reduces for next reinforcement. This increase in Tan Delta is noted by Margem.et.al [29]. The increase in tan delta determines the increase in the material property. With the addition of more Nanoparticles, it has reduced the movement of molecular chains and therefore, tan delta peaks reduced [28]. The initial increase may be due to good adhesion of Nanoparticles with the matrix.

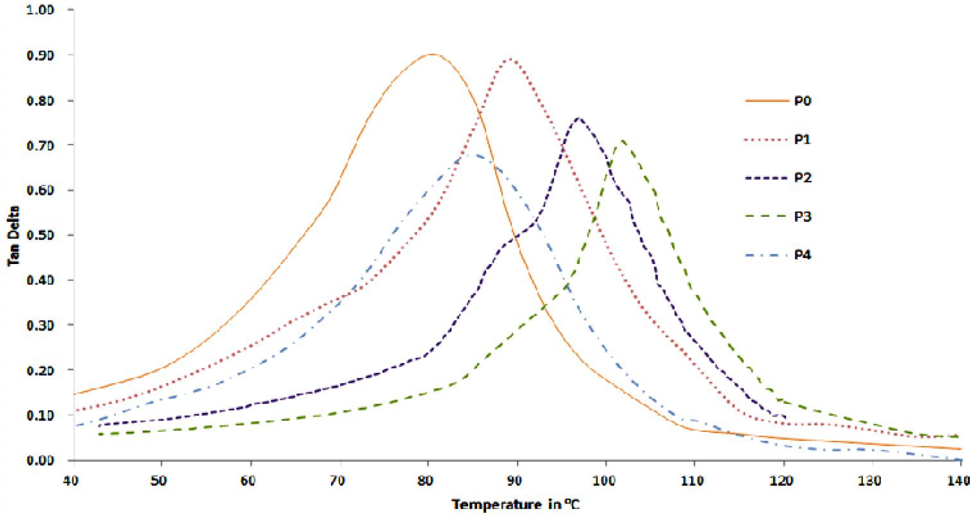

Figure7. Tan Delta vs Temperature.

E. Characterisation by SEM and EDAX:

SEM and EDAX analysis was performed for the specimens and the clear images were selected and saved. The instrument provided the details of nano materials inside the polyester matrix were identified and displayed in Figure 8. The SEM images show the nickel nanoparticles in 500nm range and can be identified. The SEM image also describes the uniform distribution of filler in the matrix.

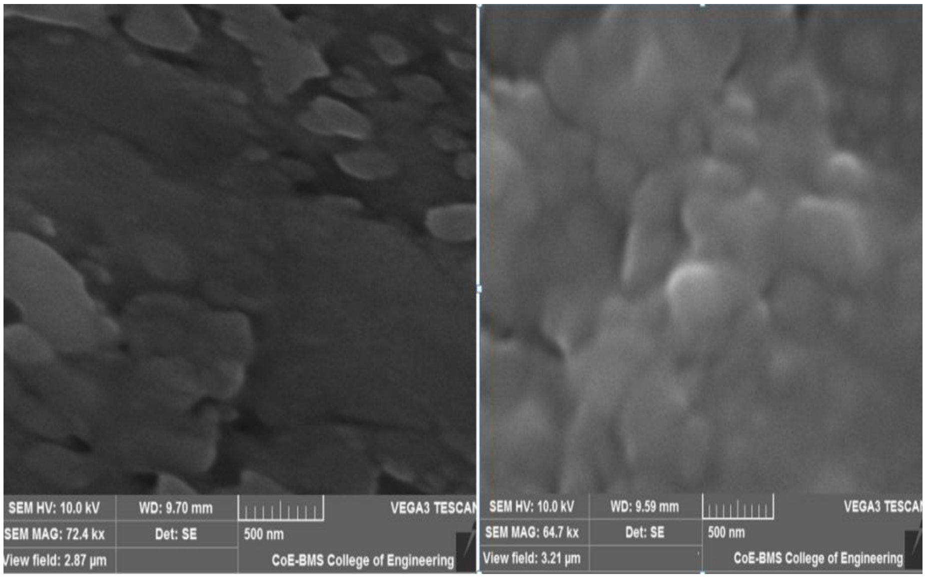

Figure 8. SEM images of 500nm scale for the composite.

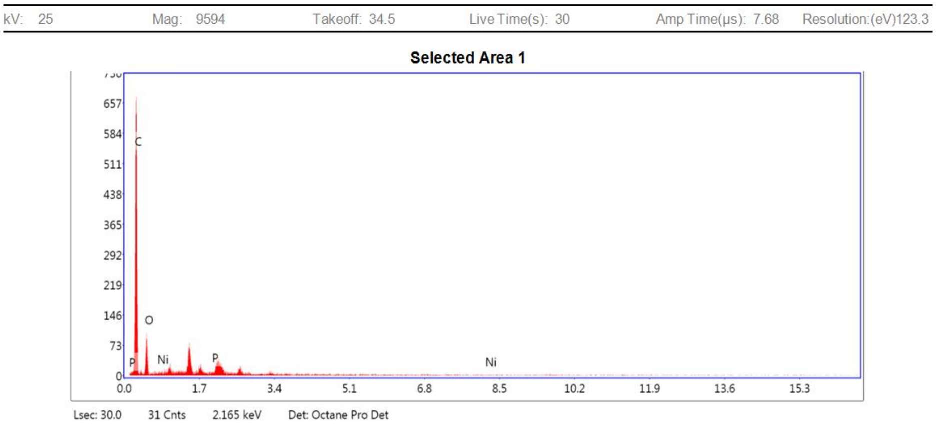

Figure 9.EDAX analysis graph.

The above Figure9 shows EDAX report of the composite. The Nickel reinfoced is shown in small quantity in the range between 6.5 to 8.5 keV in the graph. As per the standard values of EDAX apparatus, to identify the nickel reinforcement, it is known that Nickel range is 7.477keV(Standard of apparatus).

V. CONCLUSIONS

The polymer composite fabricated using nickel nanoparticles and polyester was tested for DMA and the results provide strorage modulus of the composite varies with the addition of the nanoparticles. This provides more insight on bonding between the polymer and matrix. The varied results prooves nanoparticles can alter the stiffness of the material with very little percentages. The glass transition temperature determines the maximum usage of the sample with P2 combination at a temperature of 68^{o}C. The storage modulus increases for P1 combination. The applications could be used with the thermal stability, damping behaviour and stiffness proeprties that are analysed properly. These properties could lead to the potential application of nanoparticles in a limited range of temperature and mechnaical property. These properties also reveal the bondng between the filler and matrix that could lead to failure of the material with incerasing percentages of reinforcement.

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