

# Nano Nickel Oxide/Vinyl Ester Composites with Improved Mechanical Strength



K. Rajeswari, G. Ramesh, S. Guhanathan

**Abstract:** Nickel(II)oxide is a promising material which suits for many applications due to its speciality characters such as electrochromic, photocatalytic activity, semiconductor nature, etc. But use nickel oxide as filler in polymer composites is not well studied. In this work, nano nickel oxide filled vinyl ester matrix composites were fabricated by reinforcing various weight fractions of filler. The composites so prepared were characterized by mechanical strength analysis, Fourier Transform Infrared Spectroscopy and Scanning Electron Microscopic analysis. Outcome of the analysis showed that addition of nano NiO caused significant improvement in mechanical strength under tensile and bending stress and still further enhancement after the vinyl functionalization of nano nickel oxide.

**Keywords:** Nano composites, nickel oxide, vinyl ester, surface functionalization, tensile strength, flexural strength

## I. INTRODUCTION

Presently, with the fast improvement of science and innovation, materials assume an essential part in the international economy and safety. New materials are the premise of new technologies, and materials science, vitality innovation and data science have turned into the three mainstays of present day science and innovation. As the human population is booming every year, a demand for materials of specialty characters is also arising and hence innovative development of material science and technology has become unavoidable in order to cater the needs of the rapidly growing population of the world. Polymer matrix composites are such kind of emerging materials which are being used in various fields of engineering like aerospace, mechanical, optical, electrical, etc. [1]. Thermosets and thermoplastics are the two main sorts of polymers used in polymer matrix composites. Thermosets have qualities such as a well-bonded three-dimensional molecular structure after curing [2] - [3]. They decompose instead of melting on heating. Merely changing the basic composition of the resin is enough to alter the conditions suitable for curing and

determine its other characteristics [4]. Vinyl ester is an economically viable thermoset polymer possessing adorable chemical resistance, thermal stability and flame retardancy [5]. But polymers show poor abrasion resistance, low mechanical strength and stiffness compared to other structural materials, for example, metals and compounds and consequently their usage for structural applications has been limited to some degree [6]. To combat these issues, strategies such as process modification [7], reinforcement of functional fillers and fibres [8] – [9], optimal material selection are being followed by researchers. Nano sized fillers were found to be effective in improving the properties of polymer composites over macroscopic fillers. Nano clay [10], silica [11], metal oxides [12] – [14] are the major functional nano fillers of interest in particulate filled polymer composites. Further, their surface modification is another way by which the end use properties of particulate filled composites are strengthened [15] – [17]. In this work, an attempt was made to use nano nickel oxide as filler, due to its unique characteristics [18] – [19], in vinyl ester matrix and the tensile and flexural properties of so prepared composites were studied.

## II. MATERIALS AND METHODS

### A. Materials

Vinyl ester dissolved in 30% v/v styrene monomer, methyl ethyl ketone peroxide (MEKP) catalyst and cobalt naphthenate additives were procured from Vasavibala resins private limited, Chennai, Tamil Nadu, India and used as such. Nano Nickel oxide (average particle size <50nm), coupling agent vinyltrimethoxysilane, were purchased from M/s. Sigma Aldrich India (Pvt) Ltd and.

### B. Surface modification of Nano nickel oxide

Vinyltrimethoxysilane (VMS) of concentrations 1%, 2% and 3% v/v in ethanol were prepared and their pH was adjusted to 4.5 - 5.5 by adding dilute acetic acid. Surface modification of nano nickel oxide was carried out by stirring the nano powder with the silane solution for about 5 minutes and air dried at room temperature for 48 hours.

### C. Casting of Composites

Nano nickel oxide of weight fractions ranging from 0.1 – 1.0% were mixed with vinyl ester resin, and fabricated by vertical open lay-up Resin Transfer Moulding using a glass mould with 3mm separator. The content of the mould was air cured for about 25 minutes at room temperature and post cured under microwave irradiation (2.4 GHz) at 240W for 30 minutes.



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The fabricated composite plates were cut by water jet cutting machine.

## D. Paper Submission Criteria

The tensile, flexural strength and modulus of the composites were determined as per standard ASTM D-638 type – I ASTM D790 respectively using Instron 3382 UT machine of capacity 250 KN with moving crosshead of 50 mm/min. KBr pelletized samples of composites were characterized by Fourier Transform Infrared Spectroscopy (FTIR) between the range 4000 cm<sup>-1</sup> to 400 cm<sup>-1</sup> by using Alpha Brucker spectrometer with resolution 4 cm<sup>-1</sup> at room temperature. The effect of nano particle loading on mechanical strength of the composites was studied by examining the failure pattern of the tensile fractured surface using SEM JEOL JSM 6390 LV scanning electron microscope with resolution 3nm.

## III. RESULTS AND DISCUSSION

Nano Nickel Oxide is another important material which is characterised by its photo catalytic activity, photochromic nature and good surface characteristics. But no much work have been reported on nano nickel oxide reinforced polymer matrix composites. So in this investigation, an attempt was made to fabricate a set of vinyl ester composites reinforced with nano nickel oxide. When compared to the microscopic particulates, influence of reinforcement of nano sized particulates in polymer matrix composites cannot be neglected as it results in significant change in the properties to a large extent even on smaller loading [Singh et al, 2012]. With this consideration, nano nickel oxide filled vinyl ester composites were prepared containing 0.1 – 1.0 % by weight of NiO and their tensile and flexural properties of shown in Table1:

Table 1. Tensile and flexural properties of Nickel oxide/ Vinyl Ester Composites

Sample Code	% by weight of Vinyl ester resin	% by weight of Nano nickel oxide	Ultimate Tensile Strength (MPa)	Tensile Modulus (GPa)	% Elongation at tensile break	Flexural Strength (MPa)	Flexural Modulus (GPa)
VEP	100	-	46.35	1.66	2.80	74.63	3.47
VE/N1	99	0.1	51.84	1.72	3.02	81.96	4.44
VE/N2	98	0.2	54.54	1.79	3.04	83.12	4.79
VE/N3	97	0.3	55.47	1.84	3.02	86.42	4.98
VE/N4	96	0.4	<b>55.76</b>	1.87	2.98	88.34	5.12
VE/N5	95	0.5	54.92	<b>1.96</b>	2.80	<b>90.42</b>	5.18
VE/N6	94	0.6	54.77	1.89	2.90	90.31	5.20
VE/N7	93	0.7	51.17	1.91	2.68	89.55	5.18
VE/N8	92	0.8	50.50	1.82	2.78	88.03	5.13
VE/N9	91	0.9	50.95	1.85	2.76	83.39	4.99
VE/N10	90	1.0	49.65	1.88	2.64	81.66	4.71

The average particle size of nano nickel oxide used in this work was < 50 nm and hence possess large surface area to volume ratio and high surface energy. This phenomenon of the filler could cause a distinctive enhancement in mechanical strength even on incorporation of 0.1% w/w of nano particles. It is evident from the results shown in Table 1 that 0.1 % weight fraction of nano nickel oxide addition was found to aggrandize the tensile strength by 11.8 % and tensile modulus by 3.6 %. On increasing the weight fraction of nano NiO, it was observed an anisotropic improvement in tensile strength and modulus. The maxima of tensile strength and modulus were seen at 0.4 % and 0.5 % loading respectively beyond which loading of nano particles was found to deteriorate the

strength and modulus. The nano size effect of the filler leads to development of complexity in the interface of polymer matrix and nano particles. With increasing weight fraction of nano NiO, the extent of agglomeration of particles also increases and in turn the average particle size also get hiked. This leads to formation of more interfacial stress points in polymer matrix which cause decline in strength. However, nano NiO reinforced composites were found to have better strength than that of neat vinyl ester composite. Tensile strength represents rigidity of the composite whereas the tensile modulus is a measure of stiffness. From the experimental outcome, it was noted that, both tensile strength and tensile modulus increase with addition of nano filler and thus the NiO/vinyl ester composites are inflexible and stiffer than the neat vinyl ester composite. Continuous drop in percent elongation at tensile break also further support the above said fact.

The flexural properties also were measured as a function of nano NiO loading and the results show that an identical pattern of variation as seen in tensile properties i.e. 0.1% loading of nano NiO has resulted about 10% hike in flexural strength and about 28% enhancement in flexural modulus. Both the flexural strength and flexural modulus were found to be at 0.5% and 0.6% weight fraction respectively. At this stage the flexural strength was 21% higher than that of neat vinyl ester composite and in case of flexural modulus it was about 50% higher. Further addition of nano NiO ended up with linear decline in flexural properties as noted from fore shown figures. When a material is subjected to 3-point bending, the material experience both compressive stress and tensile stress. So higher the flexural strength, higher the ability of the material to resist bending deformation. As a whole, both flexural strength and modulus are improved with nano NiO loading and hence stiffer the material. But the nano particle surface characteristics could be altered by liquid phase adsorption using alkoxy silane coupling agents. Nano particle aggregation might be reduced by such phenomenon which interconnects the polymer matrix with nano particles either by physical or chemical forces of attraction as shown in Figure 1.

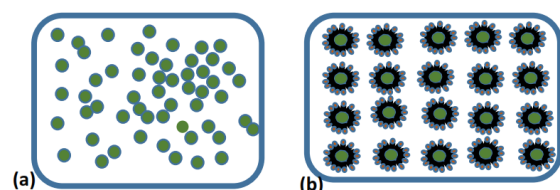


Fig. 1. Schematic representation of particle distribution (a) before and (b) after surface modification of filler particles

The nano particles are encapsulated by silane and thus resistant to agglomeration. As shown in figure, the coupling agent encapsulate the nano particles either by physical or chemical adsorption thereby reduce the aggregation. Since the surface treatment of nano NiO by silane is an adsorption process, its extent greatly influenced by the concentration of both adsorbent and adsorbate as per Langmuir adsorption hypothesis.

So in the present work, for a fixed amount of filler, the maximum concentration of silane coupling agent required for complete coverage of nanoparticle surface was obtained based on a trial and error method.

The surface functionalized nano nickel oxide with 1%, 2% and 3% solution of silane in ethanol was filled with vinyl ester to prepare the composites. The mechanical properties of vinyl functionalized nano NiO reinforced vinyl ester composites are presented in Table 2 and their relative comparison is given in Figures 2 and 3.

Table 2. Mechanical properties of surface treated NiO/vinyl ester composites

Sample Code	% v/v concentration of silane	Ultimate Tensile Strength (MPa)	Tensile Modulus (GPa)	Flexural Strength (MPa)	Flexural Modulus (GPa)
VE/NV1	1	58.24	1.98	94.51	5.44
VE/NV2	2	60.42	2.08	94.87	5.46
VE/NV3	3	60.97	2.03	95.95	5.72

It is essential to note, from the following bar diagrams, that the silane coupling of nano nickel oxide was found to bring about a considerable positive effect on mechanical properties of NiO/vinyl ester composites. 3 % concentration of silane in ethanol was observed to be the optimum concentration.

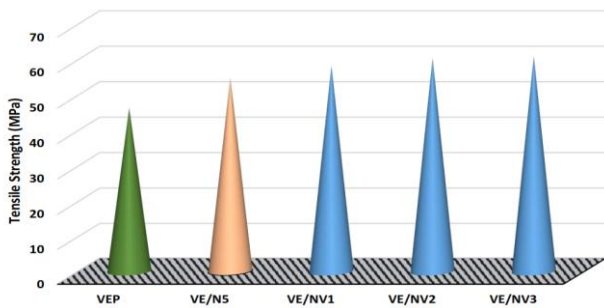


Fig. 2 Influence of silane coupling on tensile strength of NiO/Vinyl Ester Composite

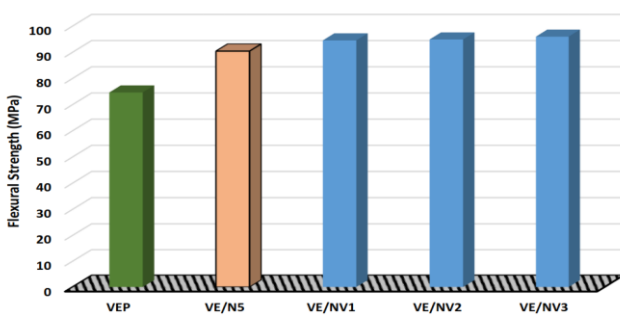
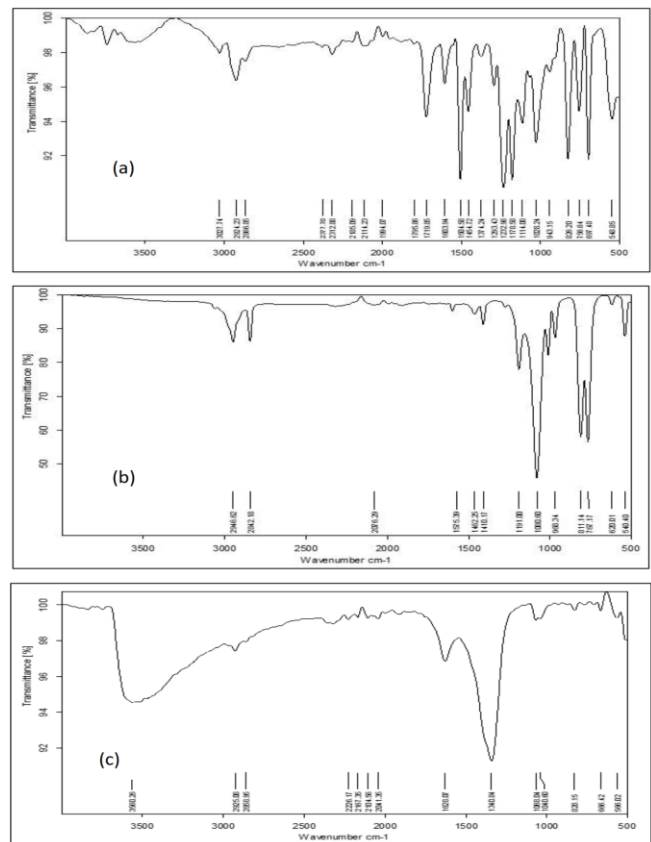


Fig. 3 Influence of silane coupling on flexural strength of nano NiO/Vinyl Ester Composite

It is evident, from Figure 2, that the tensile strength of vinyl functionalized nickel oxide reinforced vinyl ester composite has increased about 6 MPa from untreated NiO/vinyl ester composite containing the same weight fraction of NiO and an adorable 31.5 % enhancement from neat vinyl ester composite. On comparing the modulus of untreated composites, surface treated composite shows a slight hike but significant improvement with respect to neat composite i.e. about 28 % increase in tensile modulus. From the foresaid outcomes, it is inferred that surface treated NiO/vinyl ester composites are more rigid than the surface

untreated or neat composites. Flexural properties of the composites also were seen to be overhauled to a considerable level by silane treatment process. As showed up in Figure 3, the flexural strength is about 21 % excess when contrasted with untreated NiO composite however extended by 29 % from vinyl ester neat composite. Flexural modulus was extended by 0.5 units with reference to untreated NiO and 69 % when all is said in done from the neat composite. Tensile and flexural strength are relative to each other since both involve measure of resistance of tensile stress. But in addition to tensile stress, flexural strength is added with the resistance of compressive stress also and thus flexural strength is always greater than tensile strength for a composite.

Fourier Transform Infrared Spectroscopy was used to characterize the physico-chemical interaction between the components of the composites. Figure 4 shows the results of FTIR characterization of composites and additives used. On comparing the FTIR spectra of untreated NiO (Figure 4(c)) and that of vinyl silane treated NiO (Figure 4(d)), a new and strong absorption peak is observed at 1039 cm<sup>-1</sup> which is assigned to Si-O-Si bond and peak at 1126 cm<sup>-1</sup> due to incorporation of Si-O-C bond. This is a supporting confirmation for the chemical interaction between nano particles and silane coupling operator and how the coupling procedure enhances the interfacial attachment between nano particle and polymer matrix. Because of arrangement of bonding between nano particles and silane, the physical agglomeration of nano particles is diminished and accordingly the strength is made stride.





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