Journal of Material Science and Mechanical Engineering (JMSME)
p-ISSN: 2393-9095; e-ISSN: 2393-9109; Volume 3, Issue 1; January-March, 2016 pp. 27-30

http://www.krishisanskriti.org/Publication.html

Development and Characterization of NBR/Silica Nanocomposites

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Abstract—*Stable silica nanofluid was prepared with and without surfactants. The prepared nanofluid was blended properly with Acrylonitrile Butadiene rubber (NBR) latex and was allowed to dry to form NBR/Silica nanocomposites. Studies were conducted on the dispersion of nanoparticles, mechanical properties such as tensile and tear properties, morphology using scanning electron microscopy (SEM) etc. It is observed that unlike conventional composites the nanocomposites exhibit superior properties at very lower level of filler loading. This is explained in terms of the large interfacial interaction between the matrix and filler. Nanofluid route is a good option to disperse highly aggregating nanoparticles in rubber matrices.*

1. INTRODUCTION

Nanocomposites consisting of polymer matrix and filler materials have inspired scientist to develop newer composites with superior properties. However dispersion of nano particles in solid matrices has always been difficult to achieve task [1]. As high surface energy of the nanoparticles increases their tendency to form aggregates, it is difficult to properly disperse them in matrices using the conventional mixing process. Several conventional methods such as melt mixing method, sol-gel process, solvent casting and two roll milling. [2, 3, 4] Ahmed K.et.al., detailed about reinforcement of natural rubber hybrid composites based on marble sludge/Silica and marble sludge/rice husk derived silica to scrutinize the cure characteristics, mechanical and swelling properties of such hybrid composite.[5] Shamsiah Awang Ngah.et.al., prepared rubber based composites with an anhydride-cured epoxy matrix modified using core–shell rubber (CSR) particles and silica nanoparticles for toughness measurement [6] Cao Jianli,et.al., Series of Gd_2O_3/X-NBR composites were prepared by in-situ reaction at different temperatures (100°C and 160°C) and different processing time. The influence of processing time and processing temperature on dispersion of Gd_2O_3 was investigated, and the relationship between magnetic property and temperature was also discussed.[7] Vojislav Jovanović,et,al., investigated the effect of carbon black filler (CB) (loading 60–100 phr) on the cure kinetics, mechanical properties, morphology and thermal stability of acrylonitrile-butadiene/ethylene-propylene-diene (NBR/EPDM) rubber blends.[8] Nazli G. Ozdemir,et,al., present the effects of nano carboxylic acrylonitrile butadiene (CNBR-NP) and nano acrylonitrile butadiene (NBR-NP) rubbers on the interlaminar shear strength, fracture toughness and Charpy impact strength of glass fibre/dicyandiamide-cured epoxy matrix composites (GFRP).[9]

The increase of filler loading leads to improper dispersion due to agglomeration of filler particles. The main aspect in preparing nanocomposites is to achieve a very high degree of dispersion nanoparticles in the matrix. This indeed significantly improved the overall properties of the nanocomposites. Similar works reveals that NBR matrix with different filler materials like nanosilicate, nanoFe_3O_4, Na-MMT emulsion improved Mechanical properties and optimum loading of filler material [11-15].Conventional methods for the addition of filler particles agglomerating with increase of filler loading

Two-step method is the most widely used method for preparing nanofluids. Nanoparticles, nanofibers, nanotubes, or other nanomaterials used in this method are first produced as dry powders by chemical or physical methods. Then, the nanosized powder will be dispersed into a fluid in the second processing step with the help of intensive magnetic force agitation, ultrasonic agitation, high-shear mixing, homogenizing, and ball milling. Two-step method is the most economic method to produce nanofluids in large scale, because nanopowder synthesis techniques have already been scaled up to industrial production levels. Due to the high surface area and surface activity, nanoparticles have the tendency to aggregate. The important technique to enhance the stability of nanoparticles in fluids is the use of surfactants. However, the functionality of the surfactants under high temperature is also a big concern, especially for high-temperature applications.

Wei et al. developed a continuous-flow microfluidic microreactor to synthesize copper nanofluids. By this method, copper nanofluids can be continuously synthesized, and their microstructure and properties can be varied by adjusting parameters such as reactant concentration, flow rate, and

additive. CuO nanofluids with high solid volume fraction (up to 10 vol%) can be synthesized through a novel precursor transformation method with the help of ultrasonic and microwave irradiation[10]

In this study incorporation of nanosilica in NBR was done with nano fluid, in order to obtain uniform dispersion of the nanosilica in NBR. The obtained nanofluid is blended with NBR latex and later crosslinked with curing Dicumyl-Peroxide. The morphology, curing characteristics, mechanical properties have been analyzed.

2. EXPERIMENTAL

Materials:

Acrylonitrile Butadiene Rubber (NBR) latex with 50% dry rubber content was supplied from Elikom Ltd., Gujarath. Nanosilica (SiO_2) with particle size 15nm, 99.5% purified was supplied from Sigma Aldrich, Bengalure. Other materials like Cetrimide; DCP was supplied from Associate chemicals Calicut, Kerala.

Preparation:

Preparation of nanocomposites The nanosilica was taken for 1wt% to the matrix material to 100ml of distilled water was taken and mixed in crucible using magnetic stirrer for 60min. Then the solution was kept in an Ultrasonic sonicator for 60min for the proper dispersion of nanoparticles in the distilled water. On the other hand with 1wt% Cetrimide (Anionic Surfactant) was taken and mixed thoroughly with 100ml distilled water using magnetic stirrer. The two solutions mixed together using magnetic stirrer. Other sample was taken with 1wt% cationic surfactant with same quantity of distilled water.

The prepared solution was mixed with NBR latex by mechanical stirrer for sufficient time. Composite was allowed to dry in hot air oven at 100^0C to remove water content. Dicumyl-Peroxide was then added as a vulcanizing agent using two roll mixing mill. Rheograph analysis of the developed sample was done using Elastograph REV2.2. The optimum cure temperature and time were obtained. Composite was kept in compression moulding at 150^0C at 100Kgf load at optimum curing time for complete vulcanization

3. CHARACTERIZATION TECHNIQUES

Tensile and Tear:

The specimens were mounted according to ASTM 624D from samples using hydraulic press for both tensile and tear. The samples were tested using INSTRON 3365.

Morphology:

The morphology of fractured surface obtained from the tensile measurement was examined through Scanning Electron Microscope (SEM) HITACHI SU6600.The sample are coated with a thin gold layer under vacuum condition, in order to prevent electrostatic charge while examining.

FTIR:

Fourier Transmission Infrared Spectroscopy were studied with JASCO 4000 Spectrometer, taking 32 scans for each sample with a resolution of 2Cm^{-1}, ranging bandwidths from 400 to 4000 Cm^{-1}

Cure Characteristics: The cure characteristics of compounds were studied on the Elastograph REV2.2 testing instrument operated at 150^0C with torque range 2 N-m and deformation at a period of 30min.

4. RESULTS AND DISCUSSION

FTIR:

FTIR spectroscopy values were calibrated at lower wavelength side i.e. at asymmetric behavior side was 3521 $Cm^{-1}$for pure NBR latex due to OH groups present in it. When silica was added to the NBR, peaks shifted to higher bandwidth side with a value of 3500 Cm^{-1}. When silica is added with cationic surfactant the bonding between the silica and NBR was

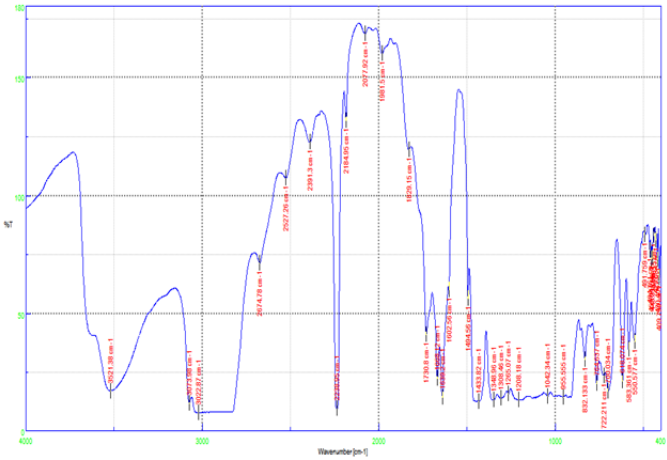

Fig. 1: FTIR for Pure NBR

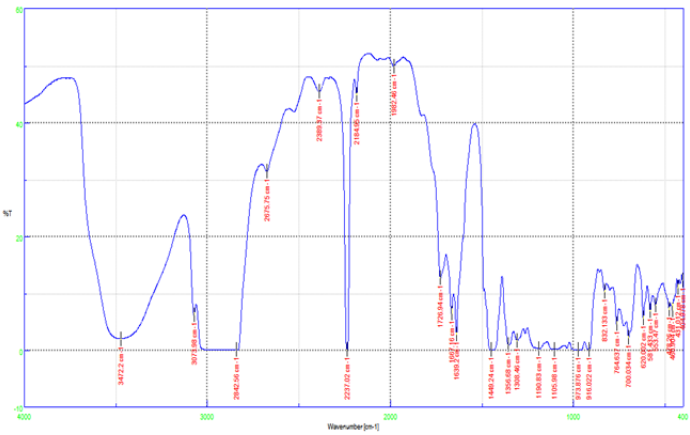

Fig. 2: FTIR for NBR+SiO_2+Cetrimide

improved due to that the peak value shifted from 3521 $Cm^{-1}$to 3472 Cm^{-1}.In case of "–C≡N" bond the peak values shifted from 2238 Cm^{-1} to 2242 Cm^{-1} for silica composition due to strong interactions between "–C≡N" and SiO_2. further movement to higher bandwidth level "–C=C–" peak values shifted from 1630 $Cm^{-1}$to 1640 Cm^{-1}due to the influence of surfactant on the SiO2 and NBR. The results reveal that adding of silica into NBR latex creates interaction between the molecules. [16-17]

Tensile and Tear Properties

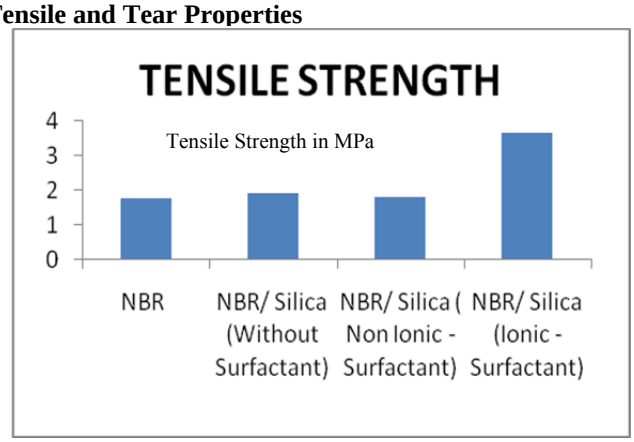

Fig. 3: Tensile Strength in MPa

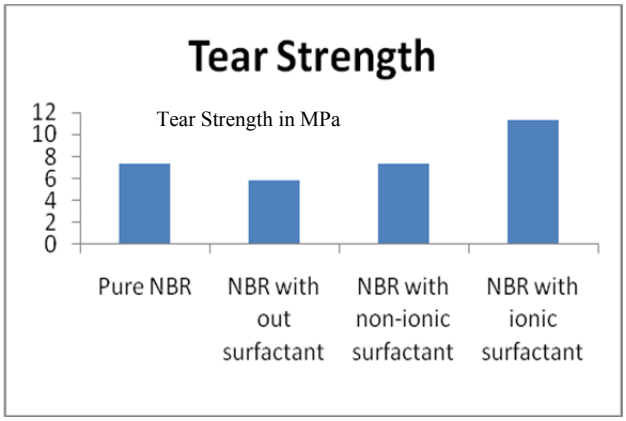

Fig. 4: Tear strength in MPa

Tensile testing was done according to ASTM D624 reveals that addition of filler material silica influences the Properties to greater extent. For 1wt% of silica with cationic surfactant increases the value upto 95% to the pure NBR.

Fig3 shows comparative tensile properties of NBR with filler material nanosilica along with the addition of different types of surfactants for 1wt% of silica. Addition of surfactants improves the bonding between filler particle and matrix. Non-Ionic surfactant failed to create proper bonding between NBR Latex and silica, where as Ionic surfactant succeeded due to the presence of OH groups in NBR latex were interacted well with amide groups of Cetrimide.

Tensile testing was done according to ASTM D624 reveals that addition of filler material silica influences the Properties compared with Pure NBR.

Fig. 4 shows the comparative tear properties of NBR latex with filler material silica with addition of surfactant.

Morphology

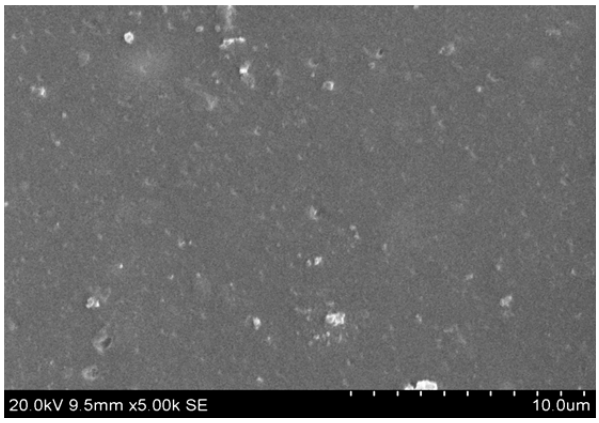

Fig. 5: SEM for Pure NBR

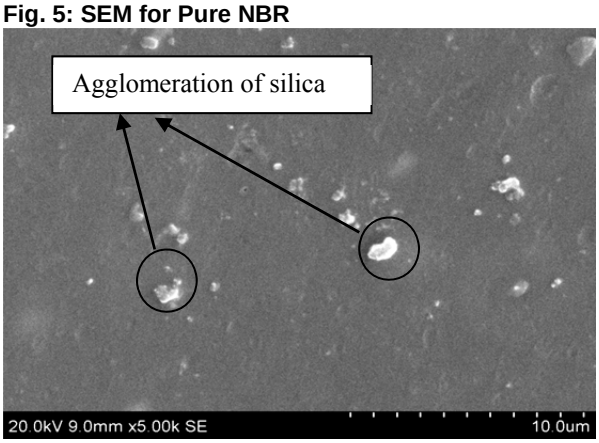

Fig. 6: SEM for NBR+ 1wt% SiO_2 (without surfactant)

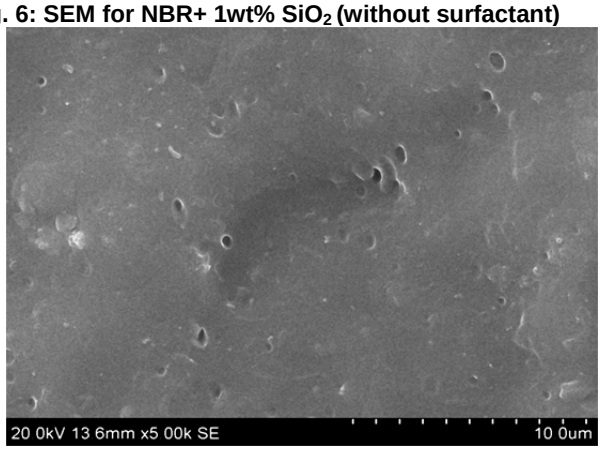

Fig. 7: SEM for NBR+ 1wt% SiO_2 (with Non-Ionic surfactant)

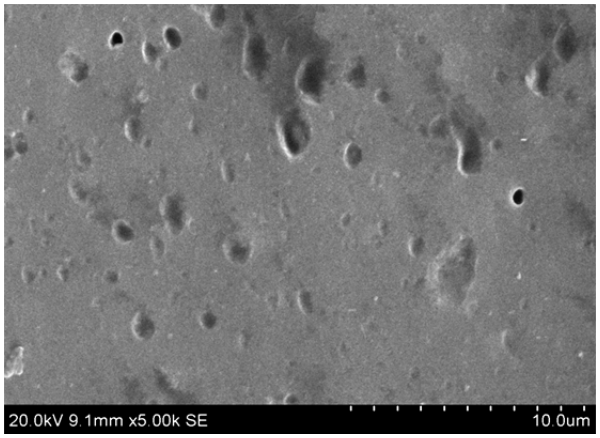

Fig. 8: SEM for NBR+ 1wt% SiO_2 (with Ionic surfactant)

Scanning Electron Microscope images reveals that dispersion of filler particles in NBR matrix. In Figure7 shows white particles which represent the nanosilica with some agglomeration where as in Fig8 agglomeration problem minimized. Proper dispersion of filler particles shows that the interfacing between nanoparticles and Matrix were improved when compared with conventional fillers (Fig. 8).

5. CONCLUSION

Reinforcing of rubber materials with addition of filler material improved mechanical properties upto 95% compared with Pure Rubber. The conventional mixing methods causes agglomeration problem which was minimized with nanofluid preparation. Mechanical properties were improved to greater extent even at low filler loading due to proper dispersion of nanoparticles which was observed in morphological studies. FTIR Spectroscopy studies reveal that interfacial interaction between matrix and reinforcement were improved with addition anionic surfactant. Further developments have to be done in finding up of optimum filler loading.

6. ACKNOWLEDGEMENTS

This work was supported in part of granting my M.Tech thesis work at National Institute of Technology Calicut, Kerala.

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