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Studies in effect of alumina nano-particles on the mechanical, thermal, morphological and electrical properties of poly (vinyl chloride)

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ABSTRACT

Polyvinylchloride (PVC)/nano-alumina composites were prepared using high speed mixer, two-roll mill and compression moulding. In the composite the concentration of nano-alumina was varied from 0 to 7 phr. The prepared composites were characterized for mechanical, thermal, electrical and morphological properties. Optimal improvement in properties was obtained at 5 phr concentration of nano-alumina in the PVC matrix. Tensile strength, tensile modulus and shore D hardness were found to have increased by about 101.9, 47.7 and 25.0 % respectively, at 5 phr loading of nano-alumina in the PVC. Thermal conductivity and electrical resistivity also increased with increase in concentration of nano-alumina in PVC.

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KEYWORDS

Polyvinylchloride;
Nano-alumina;
Compression molding;
Shore D;
Conductivity;
Resistivity.

INTRODUCTION

Polymer nanocomposites are prepared by dispersing nanoscopic inorganic particles, typically 10-100 Å in at least one dimension, an organic polymer matrix to dramatically improve the performance properties of the polymer. Polymer nanocomposites represent a new alternative to conventionally filled polymers. Because of their nanometer sizes, filler dispersion nanocomposites exhibit markedly improved properties when compared to the pure polymers or their traditional composites. These include increased modulus and strength and outstanding barrier properties^[1].

Poly (vinyl chloride) (abbreviated as PVC) nanocomposites are prepared from a very long time. Zhu et al. studied the effect of poly (methyl methacry-

late)-grafted-nanosilica and a copolymer of styrene, *n*-butyl acrylate and acrylic acid - grafted-nanosilica on the properties of PVC. Poly (methyl methacrylate) and a copolymer of styrene, *n*-butyl acrylate and acrylic acid formed an adhesive interface between nano-silica and PVC significantly improving its tensile strength and elongation at break^[2]. Xie et al. investigated the polymerization of vinyl chloride in the presence of calcium chloride nanoparticles to prepare PVC nanocomposites. Optimal improvement in tensile strength, tensile modulus, impact strength and toughness were obtained at 5 phr loading of calcium carbobate nanoparticles^[3]. Wan et al. used organically treated clay nanoparticles as reinforcing material in PVC matrix. There was found an appreciable improvement in mechanical properties and processability of PVC on addition of the organically

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modified clay below 5 wt% concentration^[4]. Wang et al. investigated the thermal degradation and charring characteristics of PVC/clay nanocomposite using X-ray photoelectron spectroscopy. They found decreased chain stripping of the PVC macromolecules due to the presence of clay nanoparticles^[5]. Sanz et al. prepared magnetopolymeric nanocomposite using nearly spherical $\text{Co}_{80}\text{Ni}_{20}$ of around 65 nm diameter, adding it in concentrations from 0.5 to 50 wt% having combined magnetic and polymeric properties^[6]. Awad et al. investigated the properties of nanoclay PVC composites prepared using tallow-triethanol-ammonium ion (a less basic amine) modified nanoclay, producing intercalated and exfoliated systems^[7].

It was found, to our best knowledge, that no study is reported of using nano-alumina as a reinforcing material in PVC matrix. So, the present study deals with the use of nano-alumina as a reinforcing material in PVC. The prepared nanocomposites were investigated for mechanical (tensile), thermal (thermal conductivity), morphological (scanning electron microscopy) and electrical (surface and volume resistivity) properties.

EXPERIMENTAL

Materials

PVC (K57 suspension resin, bulk density: $\sim 0.53 \text{ gm/cm}^2$, particle size: ~ 200 mesh, inherent viscosity: ~ 0.72) was procured from Vansh Polyvinyl India Pvt. Ltd., Mumbai, India. Lead based one pack stabilizer was obtained from Ala Chemicals, Mumbai, India. Dioctyl phthalate was procured from Fine Organics Pvt. Ltd., Mumbai, India. Nano-alumina (99.8% purity, specific surface area $\sim 70 \text{ m}^2/\text{g}$, α phase, white colour, rod shaped, 120-200 nm diameter and 650-850 nm length) was procured from Reinste Nano Ventures, New Delhi, India. All chemicals were used as obtained without any purification or modification.

Composite preparation

PVC, one pack stabilizer (5 phr of PVC), dioctyl phthalate (30 phr of PVC) and nano-alumina (as per formulation) was dry blended in a high speed mixer for 10 min. Melt blending was performed using a two-roll mill at 190°C for 20-30 min, until all the mix gets converted into a uniform molten mass. To prepare samples

for testing, the molten mass was compression moulded using an upward stroke compression-moulding machine having upper and lower platen temperature as 185°C and 190°C , and 15 MPa pressure was applied. Before compression moulding, the material obtained from two-roll mill was pre-dried in oven for 3-4 hrs at 70°C , to remove any adsorbed or absorbed moisture. Pressure and heat was applied for 15-20 min during the compression stage, followed by cooling to room temperature which took about 30-45 min. Sheet (20 cm x 20 cm, 2 sheets) obtained was then cut to get samples for mechanical, thermal, morphological and electrical testing. Cut samples were conditioned for 48 hours before testing. Quantity of nano-alumina in each batch was calculated on the weight basis of PVC. Formulations prepared are shown in TABLE 1. Flow Chart of the study is shown in Figure 1.

TABLE 1 : Prepared PVC/nano-alumina composites

Sr. No.	Sample Name	Polyvinyl chloride (PVC)		Dioctyl Phthalate		Nano-Alumina		One Pack Stabilizer	
		%	gm	phr	gm	phr	gm	phr	gm
1.	PVCC	100	500	30	150	-	-	5	25.0
2.	PVC1	100	500	30	150	1	5	5	25.0
3.	PVC3	100	500	30	150	3	15	5	25.0
4.	PVC5	100	500	30	150	5	25	5	25.0
5.	PVC7	100	500	30	150	7	35	0	25.0

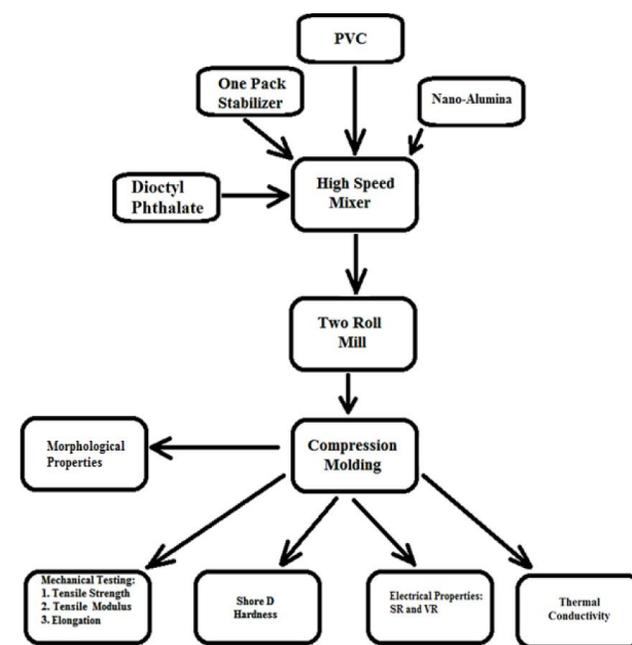


Figure 1 : Flow chart of the study for PVC/nano-alumina composite

Characterizations

(a) Mechanical properties

Tensile properties; tensile strength, tensile modulus and elongation at break were measured at ambient condition using a universal testing machine (LR-50K, Lloyds Instrument, UK), according to ASTM procedure D638 at a crosshead speed of 50 mm/min.

Shore D hardness was determined in accordance with ASTM standard D2240 (test sample dimension: 2 cm x 2 cm x 0.2 cm). Samples were tested at ten different locations.

(b) Thermal properties

The thermal conductivity (W/mK) was calculated by the product of the thermal diffusivity (mm^2/s), specific heat (J/gK) and density (g/cm^3), using Unitherm 2022 (Anter Corpo, USA) thermal conductivity tester according to ASTM E 1530.

(c) Morphological properties

Scanning electron microscopy (SEM) analysis was performed with JEOL 6380 LA (Japan). Samples were fractured under liquid nitrogen to avoid any disturbance to the molecular structure and then sputtered with platinum before imaging.

(d) Electrical properties

Electrical properties like Surface Resistivity and Volume Resistivity were measured using Million Meghometer (LS-3B, Siva Instruments, India), according to ASTM standard D257. 10 samples were tested.

RESULTS AND DISCUSSION

Mechanical properties

Tensile Strength, Tensile Modulus, Elongation at Break (mm), and Shore D Hardness values obtained for the PVC/nano-alumina composites are plotted in Figures 2-5 respectively. It was observed that tensile strength, tensile modulus, and shore D hardness increased with increase in concentration of nano-alumina in the PVC matrix, but upto the concentration of 5 phr, above which they remained nearly constant or decreased. Contrasting trend was observed for elongation at break (mm). 5 phr is the optimized concentration for addition of nano-alumina in the PVC matrix.

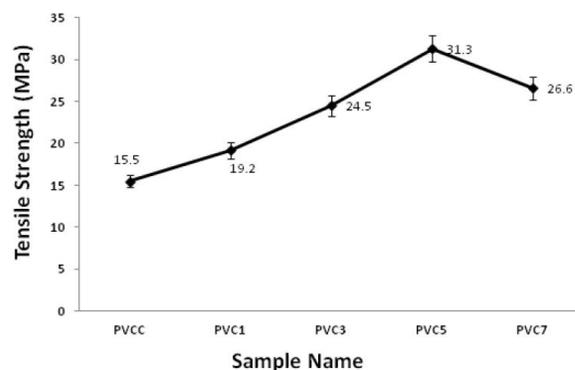


Figure 2 : Tensile strength obtained for PVC/nano-alumina composites

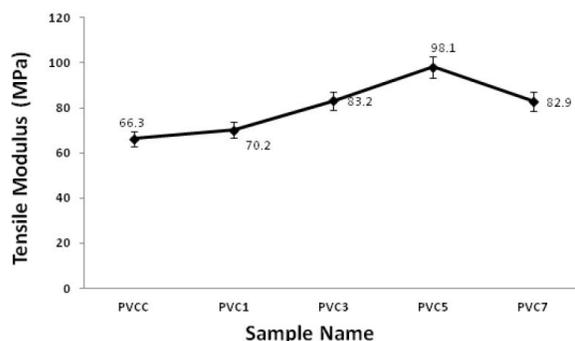


Figure 3 : Tensile modulus obtained for PVC/nano-alumina composites

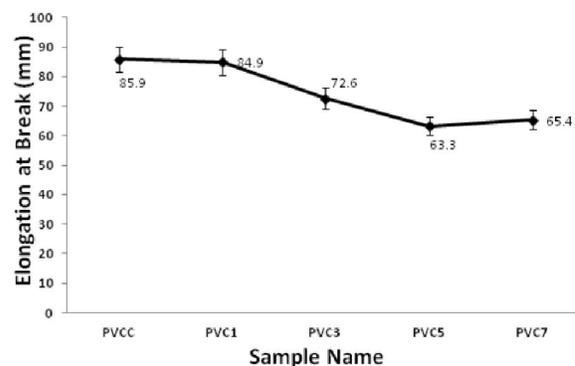


Figure 4 : Elongation at break (mm) obtained for PVC/nano-alumina composites

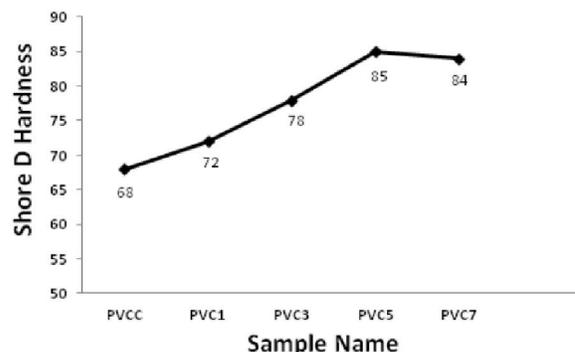


Figure 5 : Shore D Hardness obtained for PVC/nano-alumina composites

Full Paper

There are two reasons for the improvement of the properties, first is the better compatibility between the PVC matrix and nano-alumina; second is the uniform dispersion of nano-alumina in the PVC matrix. Alumina has good compatibility with the PVC matrix. Thus, the addition of nano-alumina acted as the reinforcing points in the PVC. Uniform dispersion helped in better interaction between PVC and nano-alumina, acting as a point of physical cross-linking of PVC. This helped in holding the PVC molecules at their fusion position even on application of the load. This also helps in better orientation of the PVC molecules about the alumina nanoparticles, increasing the crystallinity of the matrix PVC, thus improving the above mentioned properties. Nano-alumina concentration upto 5 phr concentration remained uniformly distributed in the PVC matrix, above which (i.e. at 7 phr) nano-alumina might have formed aggregates, decreasing the total surface area available for bonding with PVC. These aggregates might also give rise to the stress concentrate points in the PVC, giving easy initiation of the cracks, decreasing the load handling capacity and thus the above mentioned properties. Dioctyl phthalate has a very important role in dispersion of nano-alumina in PVC matrix. Dioctyl phthalate helps takes nano-alumina with it during the dispersion making nano-alumina get uniformly spread in the PVC.

As the interaction between PVC and nano-alumina increased upto 5 phr concentration, it became more difficult for the PVC molecules to move about each other decreasing the elongational property. This decrease in molecular movement decreases the flexibility of the matrix, not allowing the load to get transferred through it, decreasing the impact strength.

Tensile strength, tensile modulus and shore D hardness were found to have increased by about 101.9, 47.9, 25 % respectively; which are appreciable for 5 phr loading of nano-alumina in PVC matrix.

The above mentioned supposition can be confirmed using x-ray diffraction analysis and scanning electron microscopy.

Morphological property

Scanning electron micrographs obtained for PCV5 and PVC7 are shown in Figure 6 (a) and (b) respectively. Uniformly and individually distributed alumina

nano-particles can be properly seen in the SEM image of PVC5, justifying the explanation given above. While in PVC7 aggregates of nano-alumina were seen, with also the points of stress concentrate. Nano-alumina has very good interaction with the PVC matrix as seen from the images.

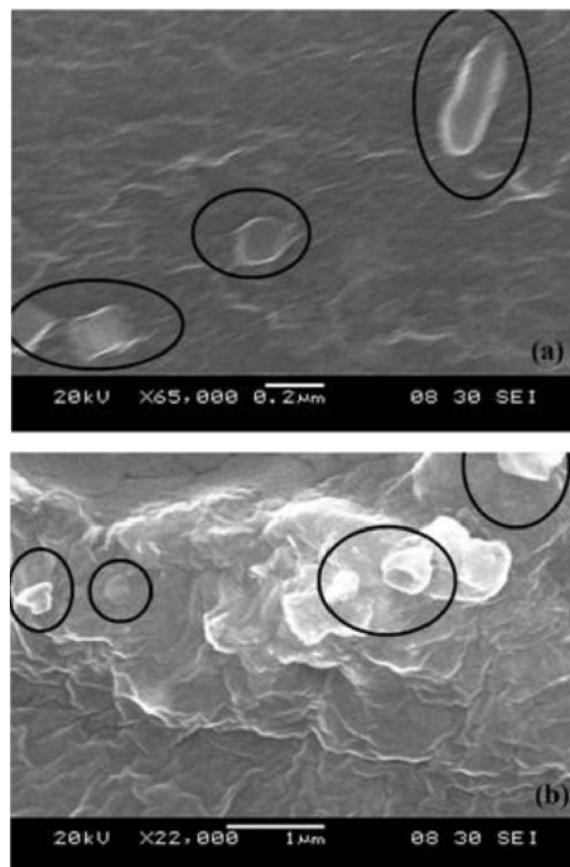


Figure 6 : Scanning electron micrographs for (a) PVC5 and (b) PVC7

Electrical properties

Surface and Volume Resistivity values are very important to understand the electrical properties for a composite material. Surface and Volume Resistivity values are in the form of graph in Figures 7 and 8. It was found that both surface and volume resistivity increased with increase in concentration of nano-alumina in PVC matrix.

It can be seen that the rate of increase in volume resistivity is more than that the rate of increase in surface resistivity for the prepared nanocomposites. This proves the uniform and proper distribution of nano-alumina in the bulk of the matrix and comparatively very less gets dispersed or leached out at the surface.

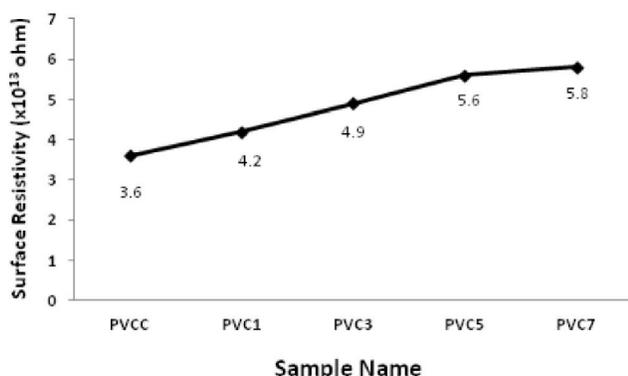


Figure 7 : Surface resistivity values obtained for PVC/nano-alumina composites

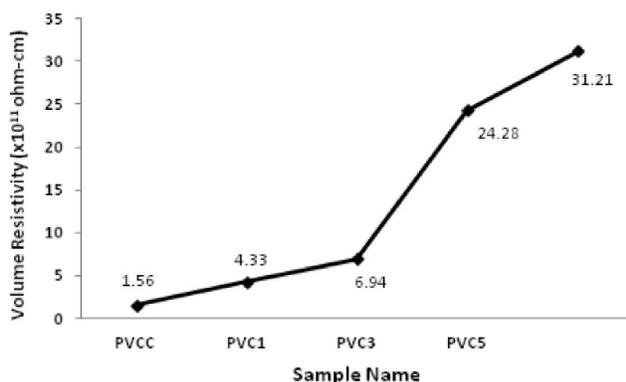


Figure 8 : Volume resistivity values obtained for PVC/nano-alumina composites

Thermal property

Thermal Conductivity values obtained for the prepared PVC/nano-alumina composites are plotted in Figure 9. Thermal Conductivity was found to have increased with increase in concentration of nano-alumina in PVC matrix. Alumina is a thermally conducting material, inducing this property into the composite. Increase in thermal conductivity will help in increasing the thermal stability of the composite, making the composite to be processed at higher temperature range.

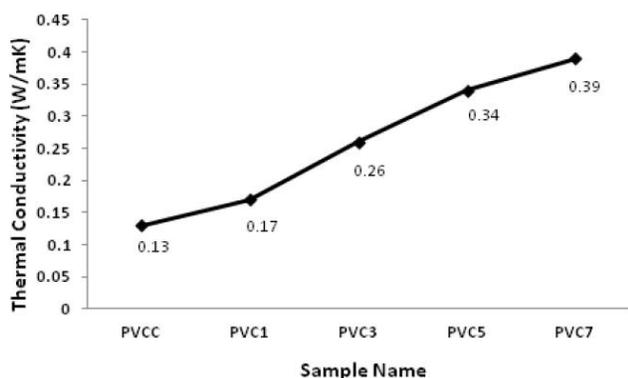


Figure 9 : Thermal conductivity values obtained for PVC/nano-alumina composites

CONCLUSION

PVC/nano-alumina composites were successfully prepared and characterized for mechanical, thermal, electrical, morphological and crystallinity properties. Nano-alumina was found to have better interaction and uniform dispersion in the PVC matrix as seen through scanning electron micrographs. Tensile strength, tensile modulus, flexural strength, flexural modulus and shore D hardness were found to have increased by about 46.8, 54.9, 25.1, 37.8 and 11.3 % respectively. Appreciable improvements were also observed in thermal conductivity and electrical resistivity values on addition of nano-alumina. Optimal improvements in the properties were observed at 5 phr concentration of nano-alumina.

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