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ALKALI RESISTANCE OF POLYESTER-CLAY NANOCOMPOSITES

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ABSTRACT

Polyester clay nanocomposite was prepared by using organic modified clay (Dodecylamine treated montmorillonite) as the reinforcement. Conventional clay filled composite was processed using inorganic clay (Bentonite) as filler at different weight percentages. Then the composite samples were kept under base environment (1 mol NaOH solution) for 21 days and tested for impact and flexural properties. Barrier properties were studied for samples kept under base environment. The percentage retention in impact and flexural properties for nanocomposites is extremely good compared to conventionally clay filled composites. Nanocomposites exhibit better barrier properties.

Key words: Isopthalic polyester, Clay, Nanocomposites.

1. INTRODUCTION

Polymer can be tailored for better mechanical properties by reinforcing it with Inorganic material. Inorganic additives are introduced into polymer systems as fine solids to act either as fillers or as reinforcing agents. The montmorillonite clay receives considerable attention among the inorganic constituents[1,2]. The montmorillonite clay being hydrophilic in nature hinders the intercalation of polymers inside the intergallery region of clay silicates and affects the homogeneous dispersion. By replacing the hydrophilic Na^+ cations with a hydrophobic onium ion with long alkyl chains, the compatibility between silicate layers and polymer matrix is established and also the intergallery distance of the clay is increased. This onium ion exchanged clay is helpful in formation of intercalated or exfoliated nanocomposites[3,4]. In the present work polyester clay nanocomposites were prepared by using dodecylamine treated montmorillonite as the reinforcing agent and isopthalic polyester resin as the matrix system. Alkali resistance and mechanical properties of these nanocomposites were determined and compared with the conventionally clay filled composites.

2. EXPERIMENTAL PROCEDURE

The resin used for matrix system is isopthalic polyester resin (PE), procured from Vasavibala resins (P) Ltd, Chennai. The inorganic clay (IOC) used is Bentonite (Emerck (India) Pvt Ltd, Mumbai). This inorganic clay was modified using dodecylamine (OMC) as reported elsewhere[5]. 300 ml of thermoset isopthalic polyester resin was taken in a container. Inorganic clay and organic clay of definite proportion (1, 2, 3, 4 and 5 wt %) was added to produce conventionally filled composites and nanocomposites respectively. Then it was mixed well for 1h using a mechanical stirrer and then catalyst (MEKP) and accelerator (Cobalt Naphthanate) were added to initiate the curing reaction. Sheets of size 300 X 300 X 3 mm^3 was cast in a glass mould and allowed to cure for 24 h at room temperature and post cured at 70°C for 3 h.

X-ray diffraction (XRD) study was carried out to confirm the formation of nanocomposites. It was carried out with a scanning rate of 2°/min with Cu-Kα radiation.

Flexural properties were evaluated using Instron 4301 UTM as per ASTM D-790 standard. Izod impact strength was determined by using Frank impact testing machine following ASTM D-256. These properties were evaluated for the nanocomposites and compared with the conventionally filled composite

Composite samples of size 50 X 50 X 3 mm^3 were kept in 1% NaOH (alkali medium) for 21 days to study the alkali absorption resistance of the nanocomposite material. The flexural and impact samples were also kept in alkali environment to study the ability of the composite material to retain its mechanical properties.

3. RESULTS AND DISCUSSION

X-ray diffraction patterns (Fig.1) show that, a peak appears at the 2θ value of 7° for inorganic clay and 5.1° for organoclay. Addition of inorganic clay to the polymer matrix does not show any significant change in peak position. But organic clay addition to the polymer matrix shows the disappearance of peak and confirms the amorphous nature of the material and the formation of nanocomposites. This also confirms that the clay silicate layer is well dispersed in the polymer matrix, which is required for property enhancement through effective reinforcement.

The flexural strength and flexural modulus of nanocomposites is better compared to conventional clay filled composites (Figs. 2 & 3). Flexural strength and modulus of the unfilled polyester is 101 MPa and 2.8 GPa respectively. Flexural strength increases up to a maximum of 114 MPa for the organoclay content of 2 wt% and flexural modulus is 4.3 GPa for the clay content of 4 wt%. The improvement in flexural properties is due to the uniform dispersion and interaction of nanosize clay layers with the polymer matrix, as well as on the rigid nature of the clay layers. At higher clay content (4 & 5 wt %) flexural strength decreases slightly. The uniform dispersion of clay platelets hinders the cross linking of the polymer chains. As organoclay content increases, more and more the clay platelets will be uniformly dispersed and that hinders the cross linking in large scale. Hence the flexural strength decreases slightly in spite of the reinforcing effect. Inorganic clay addition does not show any significant change in flexural strength and flexural modulus.

The impact strength of the unfilled polyester sample is 20 J/m. Addition of organoclay significantly improves the impact strength (Fig.4). The nanosize clay platelets play a vital role for the improvement in impact strength, since the nanofiller acts as crack stoppers. With increase in organoclay content crack propagation follows a tortuous pathway resulting in higher impact energy.

Alkali resistance for both nanocomposites and conventionally filled composites is shown in Figs 5 & 6. The absorption resistance for nanocomposites is good compared to the conventionally filled composites. Alkali resistance increases with the addition of organoclay. It is extremely well for organoclay content of 3 wt%. It decreases on further clay addition, but it is well above than that of unfilled polyester. The nanosize clay platelets act as barrier and increase the mean effective path for the molecules to travel[6]. The reason for the decrease in barrier resistance at high clay content may be due to the presence of pores. As clay content increases, more clay platelets will be dispersed throughout the matrix, the matrix becomes highly viscous and the entrapped air during shear mixing finds it difficult to escape out of the matrix system and remains as pore after curing. These pores are responsible for the reduction in barrier resistance. For the conventional composites, alkali resistance decreases with increase in clay content. The

inorganic clay is hydrophilic in nature, easily absorbs alkali solution in addition to the polymer and hence the alkali resistance decreases with increase in clay content.
The percentage retention in flexural strength, flexural modulus and impact strength of nanocomposites is quite high compared to conventional clay filled composites (Figs. 7, 8 & 9). The percentage retention in flexural modulus is 93, 88, 85, 84 and 88% and flexural strength is 94,92,94,92 and 91% and impact strength is 92, 89, 89, 92 and 91% for the clay content of 1, 2, 3, 4 & 5 wt% respectively. In the nanocomposites, the silicate layers are fully dispersed in the polymer matrix and act as barrier by increasing the mean effective path of the alkali medium to enter inside the matrix. So, this avoids the alkali absorption and also hinders the internal corrosive attack of the polymer system.

Where as conventional clay filled composites exhibits comparatively poor capability to retain its properties. The conventional composite absorbs alkali solution when kept under corrosive alkali environment. The corrosive alkali medium may also chemically attack the polymer structure internally and deteriorates the composite properties.

4. CONCLUSION

The nanocomposite is produced by using organoclay as reinforcing agent and tested for alkali resistance. The nanocomposites possess good barrier resistance for alkali environment and retain its property under corrosive environment. The conventional clay filled composites deteriorates and loses its capability to retain its property under corrosive alkali environment.

5. REFERENCES

1. Pinnavaia T J and Beall G W, *Polymer – clay nanocomposites,* Wiley series in polymer science; 2000.
2. Alexandre M and Dubois P, *Mater. Sci. Eng*. 28 (2000) 1.
3. Lagaly G, *Appl. Clay. Sci.* 15 (1999) 1.
4. Le Baron P C, Wang Z and Pinnavaia T J, *Appl. Clay. Sci.* 15 (1999) 11.
5. Maharaphan R, Lilayuthalert W, Sirivat A and Schwank J W, *Compos. Sci. Technol.* 61 (2001) 1253.
6. Lan T, Padmananda D K and Pinnavaia T J, *Chem. Mater*. 6 (1994) 573.\

FIGURES

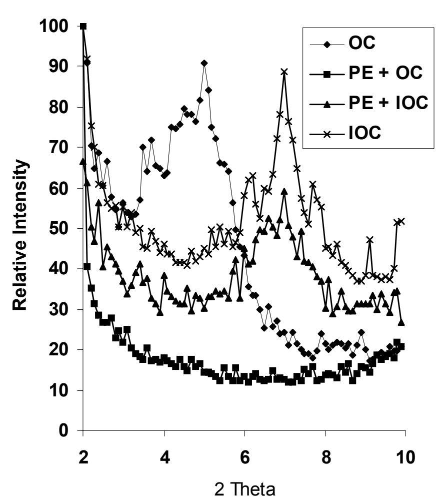

Fig.1. X-ray diffraction patterns of organic clay (OC), inorganic clay (IOC) and Composites.

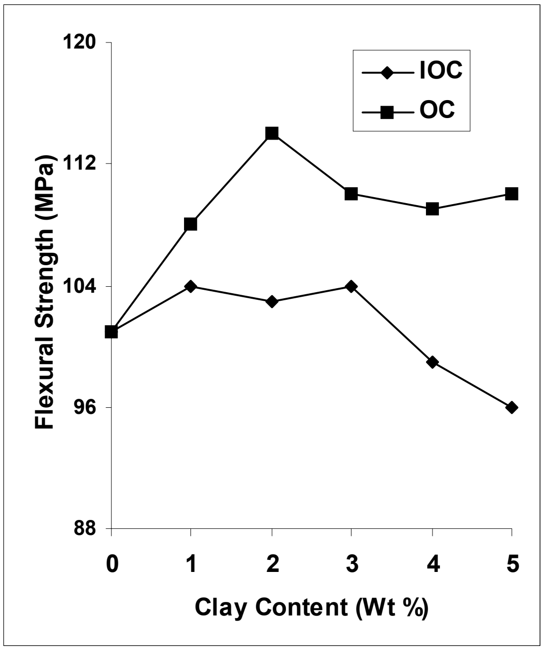

Fig.2. Flexural Strength of Composites

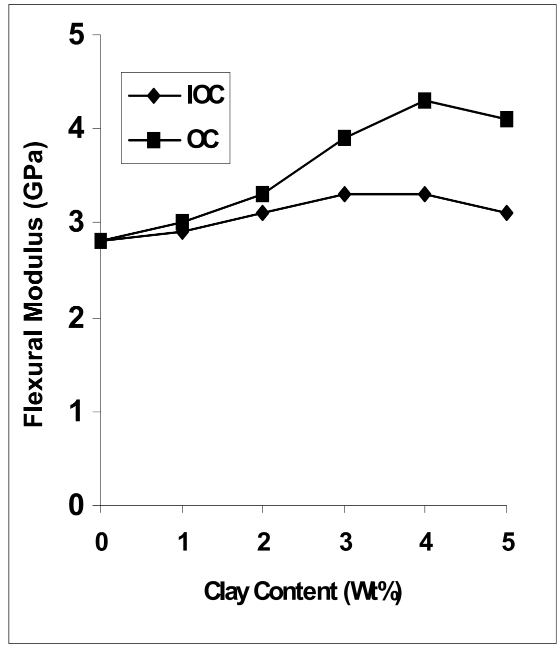

Fig.3. Flexural Modulus of Composites

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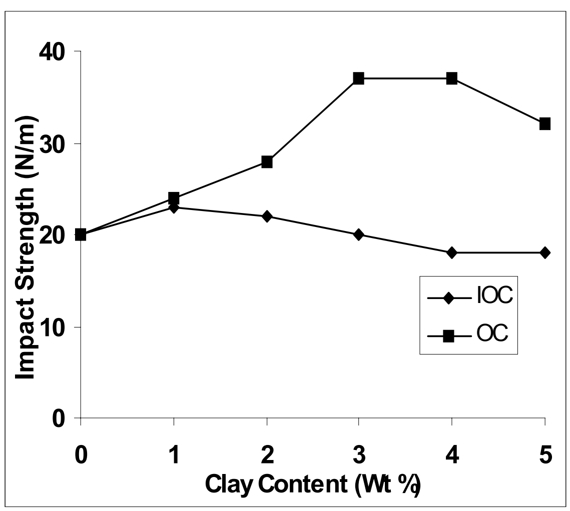

Fig.4. Impact Strength of Composites

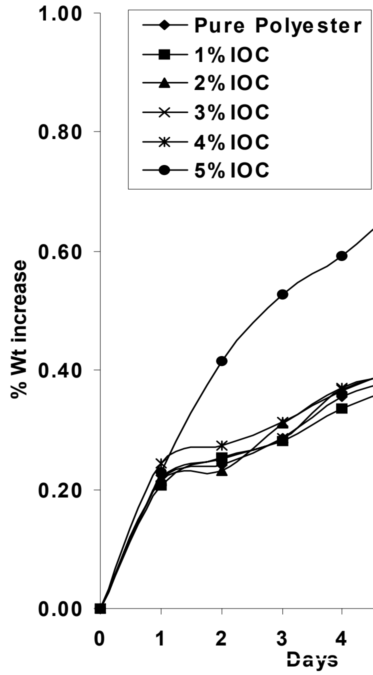

Fig.5. Absorption of Alkali in Conventional Clay Composites

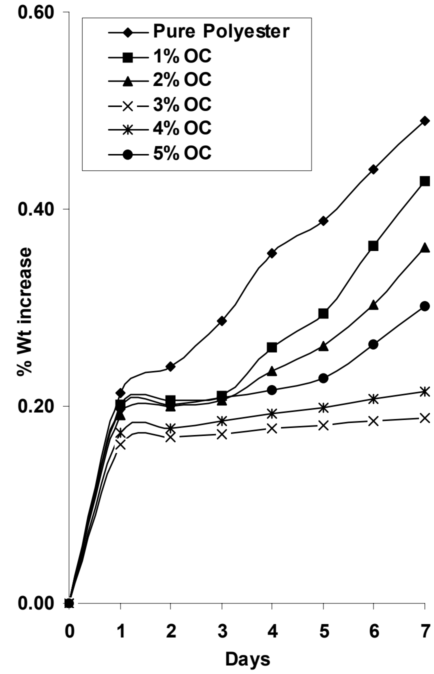

Fig.6. Absorption of Alkali in Nanocomposites.

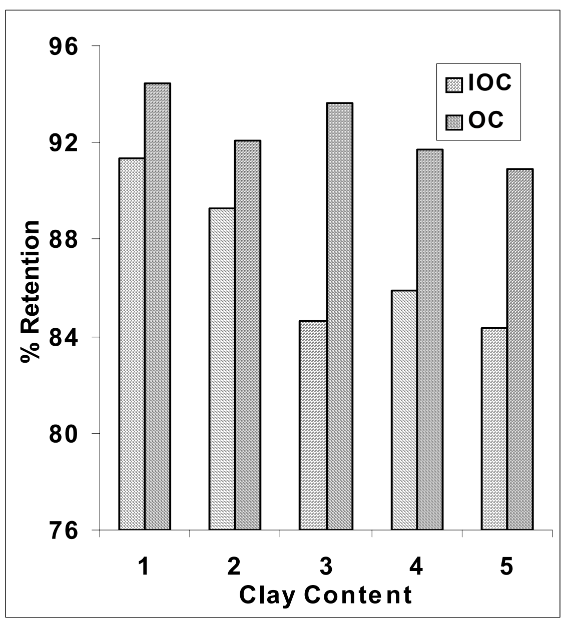

Fig.7. Percentage retention in Flexural Strength of Composites (Alkali medium)

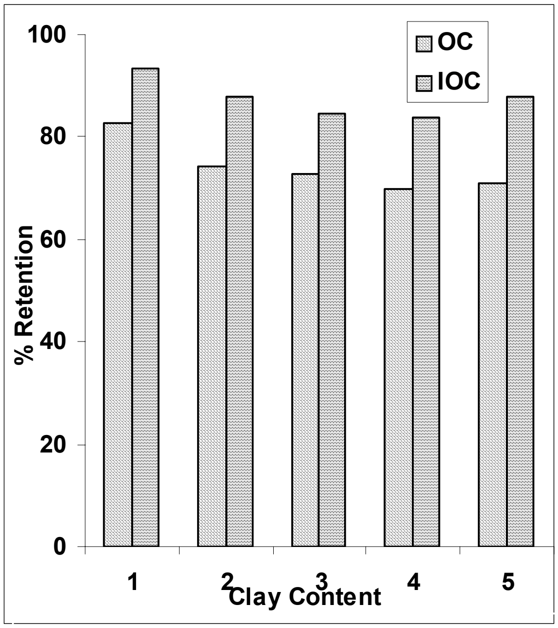

Fig.8. Percentage retention in Flexural Modulus of Composites (Alkali medium)

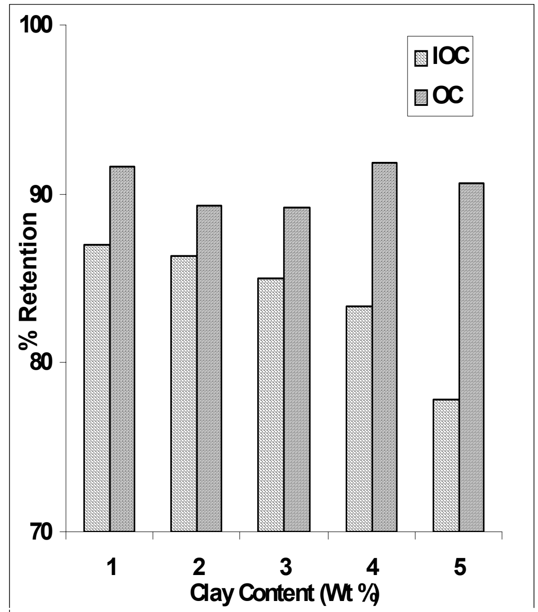

Fig.9. Percentage retention in Impact Strength of Composites (Alkali medium)