Preparation of Polyphenylenesulfide (PPS)/Clay Nano Composite and Its Characterisation

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ABSTRACT

Clay is an inorganic material which is freely available in the nature. It is layered structure and high aspect ratio. It is widely used in the plastic industries to improve the thermal, mechanical and barrier properties of polymers. The clay is hydrophilic in nature and not having good compatibility with polymers. Therefore, necessary modification is required to overcome this difficulty to make polymer/clay nanocomposites. In present study, the modified clay (C-15A) was incorporated in high performance polymer, Polyphenylene sulfide (PPS), using hot pressing technique. Nanocomposites were characterized by scanning electron microscopy (SEM), Vickers hardness tester, pin on disk wear test machine. The experimental density was very close to that of theoretical density. The Vickers micro hardness was improved by 13.5 % at 2 weight% clay. The specific wear rate of PPS/Clay Nanocomposite is low at 1 wt% clay content, compared to pure PPS. There after it increases with increasing clay content due to agglomeration of clay.

Keywords: PPS, Clay, Nano composite, wear rate, micro hardness.

1. INTRODUCTION

Polymer Matrix Composite (PMC) is the material consisting of a polymer (resin) matrix combined with a fibers or particles reinforcing dispersed phase. PMC are very popular due to their low cost and simple fabrication methods. Nanoparticles reinforced polymers attract more and more attentions due to their unique properties resulting from the nano-scale structures. The extremely high surface area is one of the most attractive characteristics of nanoparticles because it facilitates creating a great amount of inter phase in a composite and thereby, a strong interaction between the fillers and the matrix at a rather low nano-filler loading. Incorporation of nano filler in polymer leads to increasing properties of the composite such as mechanical, electrical, tribological etc.

Polyphenylene sulfide (PPS) is an organic polymer consisting of aromatic rings linked with sulfides. PPS is one of the most important high temperature polymers because it exhibits a number of desirable properties. These properties include resistance to heat, acids and alkalies, and to mildew, to bleaches, aging, sunlight, and abrasion. It absorbs only small amounts of solvents and resists dyeing. The polyphenylenesulfide has attracted considerable interest as engineering polymer due to its both high modulusand tensile strength and its good dimensional stability. Its high deflection temperature (around 227 °C), flame resistance, and excellent stability in organic liquids determine many of its applications. These properties make the PPS a polymer particularly useful in automotive and electronic industries. Nanocomposites prepared with montmorillonite show improved strength, modulus, heat distortion temperature and barrier properties. In spite of much attractive improvement in physical and mechanical properties of the polymer/ (intercalated or exfoliated) clay nanocomposites, significant drawback low fracture toughness has greatly limited their engineering applications [2].

2. LITERATURE REVIEW

1. Jinghui Yanget al. studied that Polyphenylene sulfidehaving Outstanding retention of mechanical properties under continuous use up to 338°F (170°C). It has excellent chemical resistance and It is very good electrical insulator. It is also having high mechanical strength, high strength-to-weight ratio and good corrosion resistant. PPS has the wide applications in aircrafts, automotives and electronics.
2. Xiaojun Wang et al. studiedNanocomposites prepared with montmorillonite show improved strength, modulus, heat distortion temperature and barrier properties. In spite of many attractive improvements in physical and mechanical properties of the polymer/ (intercalated or exfoliated) clay nanocomposites, a significant drawback low fracture toughness has greatly limited their engineering applications

3. Liliana B et al foundthatthe PPS is largely used in the manufacture of ball valves, electrical sockets, battery and telephone components, optical-fiber cables, electronic component encapsulation, and as a thermoplastic matrix for advanced composites.
4. Ziang et al. studies the tribological properties of PPS, SCF/micro TiO_2 composites. In recent years a tremendous interest was raised in scientific and industrial communities to apply polymer composites in sliding components, where their self lubricating properties can be exploited to avoid the need for oil or grease lubrication accompanied with the problems of contamination
5. HasmukhA Patel et al. studiedThe most commonly used clay in the synthesis of polymer nanocomposites is Carbon Fibres which is the major constituent of bentonite. It is well known that filler anisotropy, i.e. large length to diameter ratio (aspect ratio), is especially favourable in matrix reinforcement.
6. RajaniSrinivasanwas studied Montmorillonite, kaolinite, and illite,which widely used because of their high specific surface area, unique structure, chemical and mechanical stability, a variety of surface and structural properties, and low cost.
7. R. K. Goyaletal.found out that. Worn out surface of pure PPS shows sign of adhesive wear Which may be due to removal of flaky debris from the pure PPS surface. This indicates that adhesion is dominant wear mechanism for pure PPS.

3. EXPERIMENTAL WORK

3.1 Materials

PPS (Polyphenylene sulfide) was obtained from SIGMA-ALDRICH, Co, St. Louis, USA. Reported particle size from company is 100 µm and density is 1.36 g/cc. The modified clay (C-15A) with density of 1.66 g/cc was obtained from Southern Clay Co. Ltd. The solvent Ethanol was of SQ grade, and Conductive silver paste was purchased from local supplier.

3.2 Experimental Procedure

Calculation for wt. % Pure and PPS/Clay Nanocomposites

For solvent treated PC, the required weight for sample fabrication is calculated by normal formula given as:

$$m = V \times \rho$$

Theoretical density and weight of polymer and filler required for fabrication of PPS/Clay nanocomposites can be calculated by "Rule of Mixture". It is stated as

$$\rho_{th} = \rho_m . V_m + \rho_f . V_f$$

$$V_f = \frac{W_f}{\left[W_f + (1 - W_f) \times {\rho_f}/{\rho_m}\right]}$$

4. CHARACTERIZATION METHODS

Characterization of materials widely used to investigate thermal, mechanical, chemical, electrical, and metallurgical properties of materials. In present study samples were characterized for densityScanning electron microscopy (SEM), Micro hardness, and Wear rate with the help of different techniques.

4.1. Calculation of theoretical density

Density is a physical property of matter, as each element and compound has a unique density associated with it. Density defined in a qualitative manner as the measure of the relative "heaviness" of objects with a constant volume also gives an idea about soundness of the samples. Theoretical densities of the PPS/Clay nanocomposites were calculated by the rule of mixture using the density of PPS and Clay are1.35 g/cc. and 1.66 g/cc respectively. For the theoretical density it was assumed that no voids were present in the samples and no loss of material during the sample preparation. With the help of following equation theoretical densities of the composites can be determined.

$$\rho_{th} = \rho_m . V_m + \rho_f . V_f$$

4.2. Calculation of experimental density

The experimental density of the pure and nanocomposites can be determined with the help of Archimedes principle. The media of testing was ethanol having specific gravity 0.79 g/cc. The test was carried by using lab made density setup. The experimental density was calculated withthe help of following Equation

$$\rho_{exp} = \frac{W}{W - W_l} \times \rho_{liq}$$

$$Porosity \% = \frac{Theoritical\ density - Experimental\ density}{Thereotical\ density} \times 100$$

4.3. Vickers Microhardness

Vickers hardness test is non-destructive technique to check the hardness. This was used for determining the mechanical properties of materials. Hardness is defined as resistance to plastic deformation. Micro hardness was measured for the pure PPS and its nanocomposites with help of Vickers micro hardness tester (FM 700 futuretech) with a Vickers diamond pyramidal indenter. A constant load of 100 g was applied for dual time of 15 sec. Average value of seven readings was reported as the Vickers hardness of samples. The micro hardness can be calculated by Equation.

$$Hv = 1.8544 P/d^2$$

Where,

P = Applied load (in kg) and d = Average indentation diameter in mm.

4.4. Wear properties

The wear test was carried out using pin on disc machine manufactured by Magnum Engineering, Bangalore in dry condition. Before starting, 1000 size emery paper was used to create surface roughness on counter surface. Wear test was carried out for 1km travel distance under load of 10 kg. The specific wear rate can be calculated with the help of following equation.

$$\text{Specific wear rate} = \frac{\Delta m}{\rho FL} \times 10^3 \ \ (mm^3/N - m)$$

Where,

Δm = m_1-m_2

m_1= Initial mass of the sample

m_2= Final mass of sample after wear test

ρ= Density of sample

F=Load applied

L=Sliding distance during wear

5. RESULTS AND DISCUSSION

5.1 Density OfPps/Clay Nanocomposites:

The table 1 and Fig 1 reveal theoretical and experimental density of the pure and PPS/Clay nanocomposites. The densities of samples were measured by the Archimedes principle. Theoretical density of the PPS and clay is 1.36 g/cc and 1.66 g/cc which wereconsidered for calculation of theoretical density of nanocomposites. When the nanoclay wasadded in the PPS matrix, the density of the nanocomposites was increased by increasing claycontent in the matrix. The experimental density of the PPS and its nanocomposites having 1to 10 wt % nanoclay almost match the theoretical density value which indicates that the samples were porosity free.

Table 1 Theoretical and Experimental density PPS/Clay nanocomposites

Sr. No.	Sample code	% Clay in PPS		Thereotical Density (g/cc)	Experimental Density (g/cc)
		By Weight	By volume		
1	NC-0	0	0	1.360	1.360
2	NC-1	1	0.008	1.363	1.366
3	NC-2	2	0.016	1.366	1.368
4	NC-3	3	0.025	1.368	1.370
5	NC-5	5	0.041	1.374	1.380
6	NC-8	8	0.067	1.383	1.388
7	NC-10	10	0.083	1.388	1.384

Note- NC-X, where X indicates wt% of clay in PPS matrix. NC means nanocomposites.

For calculation of composite density theoretical density of clay was 1.66g/cc

However in the case of 10 wt% nanocomposites, the experimental density was found slightly less than the theoretical density which indicates that there may be some porosity present in the sample. At higher clay contents agglomeration of clay layers takes place and produces porosity which in turn reduces the experimental density. Similar trend was reported for others

polymer/ clay nanocomposites [8]. The porosity free and dense samples improve all the properties of the material.
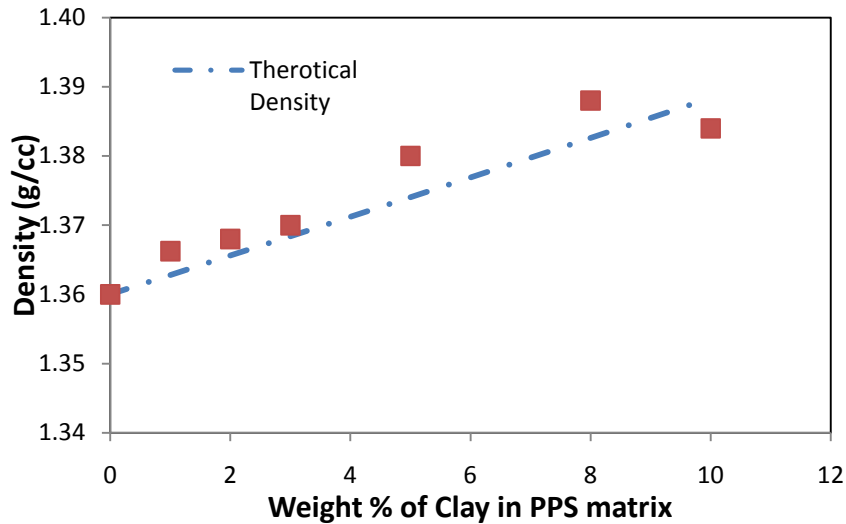

Fig 1 Theoretical and experimental density Vs volume % of clay in the matrix.

5.2 Vickers Microhardness

Fig 2 shows the micro hardness of PPS/clay nanocomposites with 0 to 10 wt% nanoclay content. An average hardness of 7 indentations was measured (Table 2) and a graph was plotted in Fig 2. At the beginning, it was noticed that the hardness increased with increasing clay content. In this study the maximum hardness measured when nanoclay reached 2 wt%. A decline of the hardness also observed on further increasing the .nanoclay content.

At higher clay content micro hardness of Nanocomposite is decreased due to formation of more numbers of clusters and increasing the size of clusters. When it reaches to critical limit, reinforcing function of nanoclay decreases.

Table 2 Vickers micro hardness of PPS/Clay nanocomposites

| Sample Code	Micro hardness of Sample at 100 gm and 15 sec (kg/mm^2)							Average Micro hardness
	Reading 1	Reading 2	Reading 3	Reading 4	Reading 5	Reading 6	Reading 7	
NC 0	24.2	25.9	22.5	23.9	24.5	23.8	24.7	24.214
NC 1	28.7	25.2	27.4	26.1	26.6	27.2	27.5	26.957
NC 2	27.2	29.7	25.4	26.8	26.3	28.5	28.4	27.471
NC 3	22.3	23.6	22.3	28.1	23.7	22.9	21.5	23.486
NC 5	22.2	19.1	21.9	19.1	18.7	19.1	22	20.300
NC 8	18.5	18.4	17.7	20.8	16.7	20	16.7	18.400
NC 10	16.4	17.5	17.8	14.4	16.3	18.7	16.9	16.857
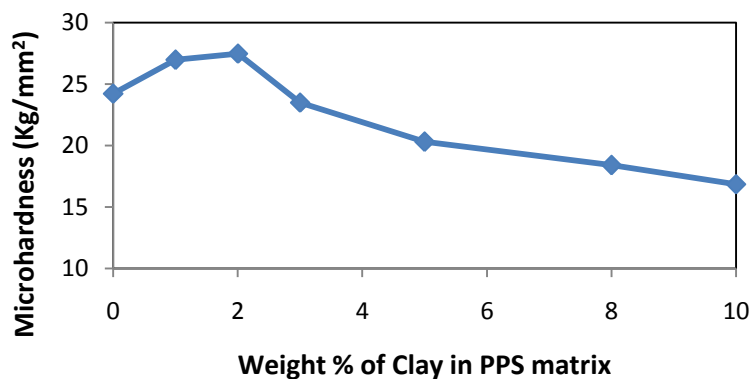
Fig 2. Hardness Vs weight % of clay in the PPS matrix.

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5.3 Wear Rate

Specific wear rate of pure and PPS/Clay nanocomposites are shown in the table 3 and Fig 3. The specific wear rate was calculated by recoding weight loss from the wear test at the constant load (10 kg) and traveling distance (1Km). Velocity of disc is constant (1 m/s) during the test. Fig. 3 shows that specific wear rate of PPS/Clay nanocomposites decreases at 1 wt% clay. However, it increases with increasing clay content up to 3 wt% and thereafter there is no signifiacant change but it is lower than pure PPS. In brief, PPS/clay nanocomposites at 1 wt% clay decreases wear rate by one order and are suitable for wear purpose. Similar results were also found out in case of other polymer/clay nanocomposites [14].

As wear rate decreases only up to 1wt% and thereafter is increases as increasing clay content. The increasing trend might be due to agglomeration of clay layers.

High wear resistance is due to size of nano additives are very small as of surrounding polymer chains which increases the bonding to polymer matrix. Also nano sized filler tends to produce a tenacious transfer layer on the counter surface, which protects the composite surface thereby reducing friction and wear of nanocomposites.

SEM micrographs of worn surfaces of pure PPS and its nano composites shown in fig. 4. Worn out surface of pure PPS shows sign of adhesive wear as shown in fig 4 (a, c). Which may be due to removal of flaky debris from the pure PPS surface. This indicates that adhesion is dominant wear mechanism for pure PPS. Fig 4 (b, d) shows mild abrasive wear for NC-1 nanocomposite [15].

Table 3 Specific wear rate of PPS/Clay nanocomposites

Sample Code	Initial Weight (gm)	Final Weight (gm)	Weight loss	Velocity (m/s)	Distance (m)	Force (N)	Wear Rate (mm^3/n-m)
NC-0	0.6334	0.5694	0.064	1	1000	100	4.74E-04
NC-1	0.6672	0.6652	0.002	1	1000	100	1.48E-05
NC-2	0.4529	0.4381	0.0148	1	1000	100	1.10E-04
NC-3	0.5882	0.5466	0.0416	1	1000	100	3.08E-04
NC-5	0.6608	0.6115	0.0493	1	1000	100	3.65E-04

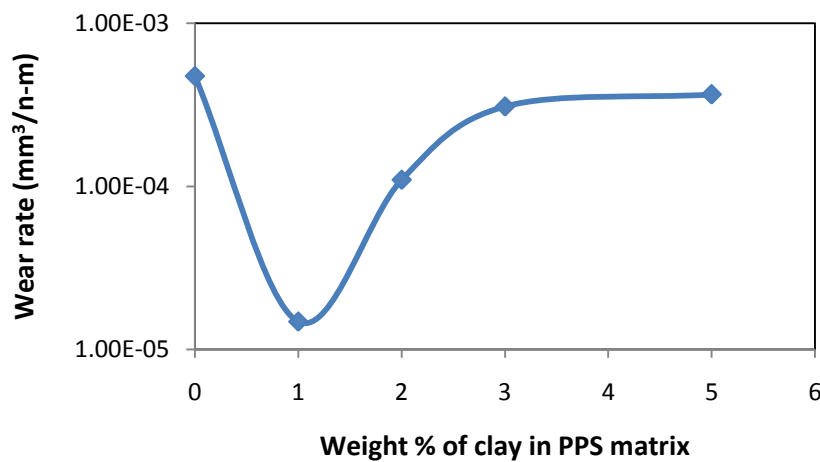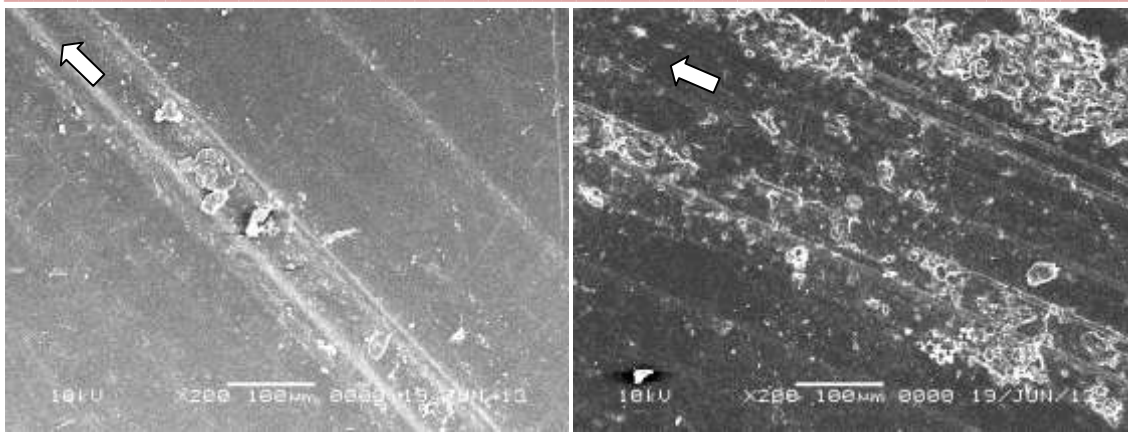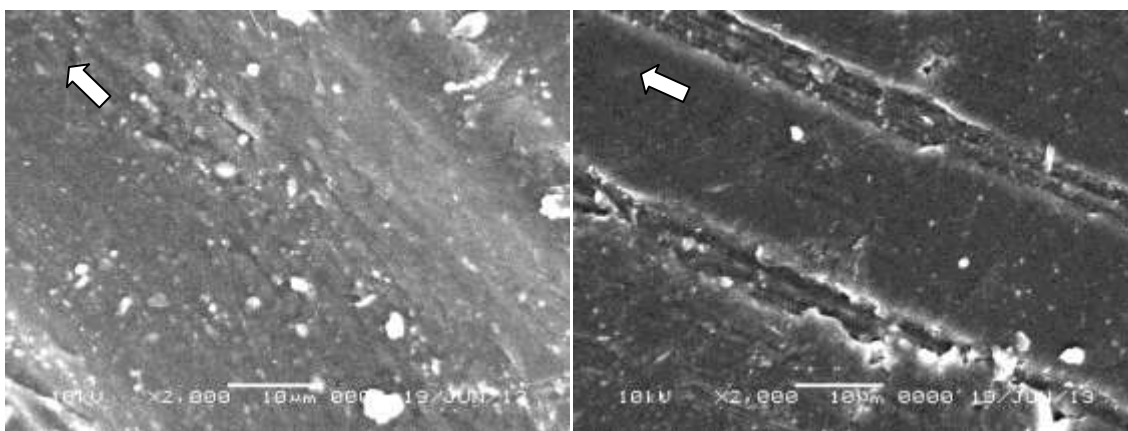Fig 3 Specific wear rate of PPS/Clay nanocomposites

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(a) (b)

(c)   (d)

Fig 4. SEM of worn surfaces of (a, c) NC-0, (b, d) NC-1 at 200X and 2000X. Arrow indicates the sliding direction.

6. CONCLUSION

1. High performance PPS/Clay nanocomposites were successfully fabricated by mechanical alloying method followed by hot compaction. The clay content was varied from 0 to 10 wt%. Experimental density was very close to that of theoretical density.
2. Micro hardness of nanocomposite was improved by 13.50 % at 2 wt% clay. Thereafter it decreases due to agglomeration of clay.
3. Wear rate of PPS/Clay Nanocomposite is low at 1 wt% clay content, compared to pure PPS. There after it increases with increasing clay content due to agglomeration of clay.
4. SEM study of warn out surfaces of Pure PPS shows adhesive wear mechanism, whereasnanocomposites shows abrasive wear.

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