

Wear and Hardness Characterization of Synthesized Polystyrene Composites

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ABSTRACT: In the present work, four fillers calcium carbonate, graphite, mica and talcum powder of micrometer size in the form of powder with three level of concentration 1%, 2%, 3% by weight were synthesized with polystyrene. The composites were produced by in-situ polymerization method and the samples were prepared by hand operated injection moulding machine. The hardness and wear tests were performed to study the effects of fillers and their concentrations on the composites. The maximum improvements 102.08% in synthesized PS-CaCO₃ composite and 62.83% in case of synthesized PS-talcum powder composite for hardness and wear resistance respectively, were depicted. Two-way ANOVA using “Minitab 15” software was used to see the contributions and significance of the input parameters on the output parameters. The addition of fillers are found to be significant while concentrations of fillers insignificant at 5% level of significance.

KEYWORDS: Composites, Polystyrene, Wear, Hardness and ANOVA.

I. INTRODUCTION

Today most of the composites are polymer based and have versatile properties offered by polymeric materials. The matrix in a polymer composite serves both to maintain the position and orientation of the reinforcement and protects them from adverse environmental effects. In addition, it helps to distribute the applied load by acting as a stress transfer medium [1]. The main functions of the reinforcing material in a composite are to carry the load and provide structural properties like stiffness, strength and thermal stability [2]. Several procedures are known to synthesize polymer composites. These include melt intercalation method, solution method and in-situ polymerization. In in-situ polymerization method, the layered silicate is swollen within the liquid monomer. Polymerization of the monomer occurs in the interlayer of the clay mineral, resulting in an expanded interlayer distance. Polystyrene belongs to the group of standard thermoplastics and it can be used in an extremely wide range of applications [3]. Polystyrene is chemically nonreactive and used to make containers for other chemicals, solvents and even food items. Polystyrene is soluble in solvents that contain acetone, such as most aerosol paint sprays and cyanoacrylate glues. It has density 1.05 g/cc and melting point 240 °C.

II. LITERATURE REVIEW

Zheng et al.[4] in 2004 studied the characterizations of expanded graphite/polymer composites prepared by in situ polymerization. The expanded graphite was made by oxidation of natural graphite flakes, followed by thermal expansion at 600 degree C. Microscopic results disclosed that the expanded graphite has a legume-like structure, and each “legume” has a honeycomb sub-structure with many diamond-shaped pores. Chatterjee A& Mishral S [5] in 2013 explored about the Nano-Calcium carbonate (CaCO₃)/Polystyrene (PS) core-shell nanoparticle: All composites were prepared with 0.10–5.0 wt% loading on Brabender Plastograph. It was shown that rheological, thermal, mechanical and morphological properties were improved as hybrid nano-CaCO₃/PS particles reinforced matrix. Sreekanth et al.[6] in 2009, studied the effects of mica with varying concentration on mechanical, thermal, electrical, rheological and

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morphological properties of polyester thermoplastic elastomer. The specimens of the composites with concentrations 5 to 40 weight % of mica were prepared by twin screw extrusion. They found that flexural strength and modulus were increased with mica concentration, whereas tensile strength was decreased at higher concentrations. Lapcik et al.[7] in 2008, studied about the effect of the talcum(talc) filler content on the mechanical properties of Polypropylene composites.They found that the increase in filler content lead to an increase in the strength of the composite material with a simultaneous decrease in the fracture toughness. The increase in mechanical strength simultaneously led to a higher brittleness, which was reflected in a decrease in the mean impact strength from the initial 18 kJ/m² to 14 kJ/m². Bharat A et al. [8] synthesized water based nanocomposites consisting of nano-CaCO₃ by in-situ polymerization of MMA. Quantity of nano-CaCO₃ was varied as 0, 2 and 4% of monomer quantity. They concluded that the synthesized nanocomposites showed improved ultra violet (UV) absorption and improved mechanical properties as compared to neat PMMA.

OBJECTIVE OF THE STUDY

In this work, the effects of different additives and their concentrations on mechanical properties of synthesized polystyrene polymer matrix composites produced through in situ polymerization method is explored and the properties of the output parameters of the composites are analyzed by a software “Minitab 15” using two-way analysis of variance (ANOVA).

III. EXPERIMENTATION

1. Input and Output Variables

Table 1 Input and Output Variables

Input Variables		Output Variable
Filler Materials	Graphite Powder, Mica Powder, Talcum Powder, CaCO ₃	Hardness Analysis Wear Characteristics
Filler Concentration	1%, 2%, 3% By Weight	
Stirrer Speed	800±50 rpm	

Table 2 Quantity of Additives used

Mass of Styrene (in grams)	% of Additives By Weight	Mass of Additives (in grams)
590.85	1	5.9085
590.85	2	11.817
590.85	3	17.7255

Table 3 Materials and Equipments

Materials and Equipments	Description
Styrene monomer	Styrene, chemical formula C ₈ H ₈ , the monomer used in polymerization was purchased from Otto Company
Methanol	Methanol was purchased from CDH Pvt. Ltd in 500ml bottles
Acetone	Acetone was purchased from CDH Pvt. Ltd in 500ml bottles
Benzoyl peroxide	Benzoyl peroxide was purchased from LobaChemie Pvt. Ltd in 500g bottles
Glassware	For smooth completion of experiments glassware and equipment were used

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2. Properties of Additives

Calcium carbonate (CaCO_3) is a white powder and colorless crystal. Its melting point is 825°C and specific gravity 2.83. It is stable and has negligible water solubility. Graphite is good conductor of electricity having specific gravity 2.2. Structure of graphite has hexagonal rings and stable forms of carbon. Mica has density 2.9 g/cm^3 and is transparent, flexible, elastic, and chemically inert and can easily resist actions of heat, light, water, oil, solvents, and alkalies. It is strongly absorbent to ultraviolet radiations, prevents penetration of destructive Sun-rays. Pulverized talcum powder has wide industrial applications as filler in rubber, textile, plastic, linoleum, asbestos products, polishes and soaps; as a loading agent for all kind of papers; as a carrier of insecticidal and for coating calcium ammonium fertiliser.

3. Experimental Procedure

(a) Washing of styrene: In washing process, the styrene monomer is taken into separating funnel and shaken with 10% solution of NaOH in water. This Method is suitable for water insoluble monomer. The inhibitor reacts with alkali and the reaction product being water soluble, comes out of the monomer phases and can be removed along with the aqueous phase. On allowing standing after through shaking, the monomer and alkali solution separate. The alkali solution is removed from the separating funnel and washing with alkali is done several times to obtain pure styrene.

(b) Synthesis of polystyrene composites (by in-situ polymerization) Calculation of initiator: Through literature it has found that 0.011g benzoyl peroxide (BPO) is required for 5.5 ml of monomer. Hence in this case 1.3g of BPO was taken for 650ml of monomer.

(c) Calculation of amount of styrene: Since in this work, four different additives are added at three different concentrations, so, their respective amounts are given below:

Density of styrene = 0.909 g/cm^3

Volume of styrene = 650ml

Hence mass of styrene = $0.909 \times 650 = 590.85 \text{ g}$

Before preparation of the composites, pure styrene was synthesized at 90°C with the aid of BPO as the initiator. The polymerization time was approximately 3 hours and the initiator concentration was according to the quantity of styrene monomer taken i.e.; 1.3g of BPO for 650 ml of styrene in 1000 ml of beaker. Figure 1 shows experimental setup. Synthesis of polymer composites by in-situ polymerization basically involved the dispersion of additives in styrene followed by free-radical polymerization initiated by the addition of benzoyl peroxide. Styrene monomer containing the desired amount of additives was mechanically mixed by stirrer for 30 minutes at room temperature in order to obtain better dispersion. The BPO was then added to the mixture to initiate the polymerization which took place at 90°C at speed $800 \pm 50 \text{ rpm}$ of stirrer in each case for approximately three hours till the monomer solution becomes viscous i.e.; completion of the reaction. After completion of the reaction add to the contents of beaker approximately 250 ml of methanol (non solvent) and doing mild agitation with glass rod in beaker.

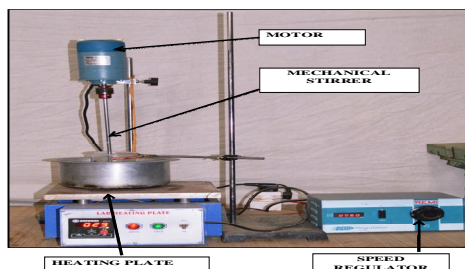


Figure 1 Experimental setup

4. Collecting and Drying of Composites

The methanol along with composites are collected into a plate and left the sample to dry for 48 hours. Fig 2 shows different synthesized composites.

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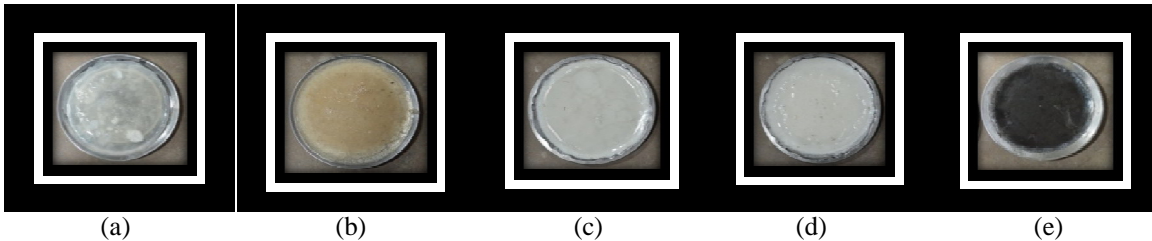


Figure 2 (a) Pure polystyrene, Polystyrene composites with (b) Mica (c) Calcium Carbonate (d) Talcum powder (e) Graphite

5. Sample Preparation

The composites obtained at the end of polymerization were kept in an oven at 120°C for 2-3 hours to complete polymerization and remove the remaining moisture and liquid styrene. Then the products were ground and subsequently injection molded in a full ounce hand operated injection moulding machine using the fabricated die. The specimens for mechanical characterization were prepared by hand operated injection molding machine of one ounce capacity of OrientCompany. During moulding; barrel temperature 210°C, mold temperature 30°C were taken for the preparation of each sample.



Figure 3 Fabricated adjustable die



Figure 4 Prepared samples

IV. RESULTS

1. Hardness Test

Rockwell hardness tester was used for analysis of hardness test with M- scale in Rockwell hardness testing having 1/4 inch ball indenter. The specimen size was prepared according to the ASTM D 785. The total load of 100 kg was used. The test was carried out in NSIC Aligarh (India) at atmospheric conditions (25°C). Mean of three readings for each sample were taken.

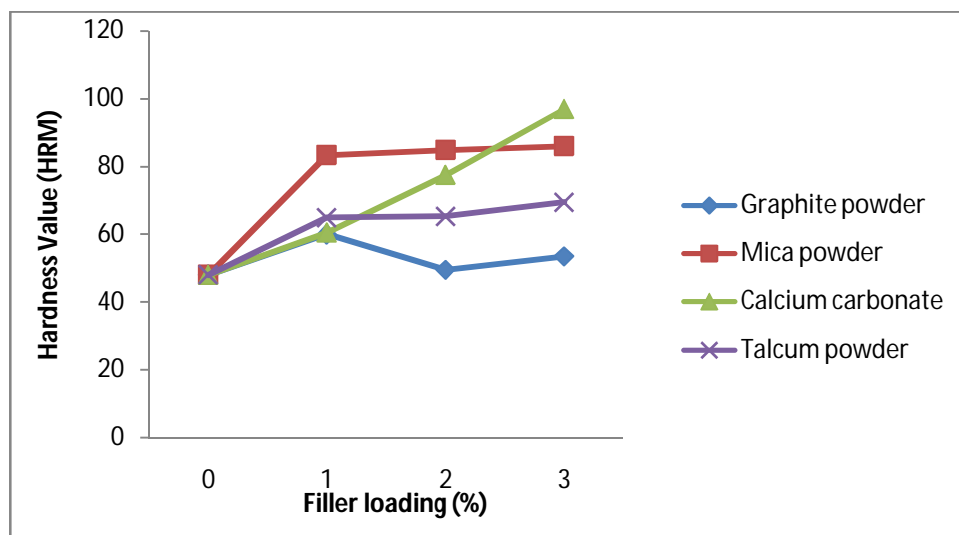


Figure 5 Effect of additives on Hardness value of synthesized polystyrene composites

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Table 4 ANOVA Table for Hardness Test

Source	DF	SS	MS	F-value	P-value	% Contribution
Filler loading	3	1777.32	592.44	5.59	0.019	45.04
Fillers	3	1213.92	404.64	3.82	0.051	30.76
Error	9	954.00	106.00			24.20
Total	15	3945.24				100

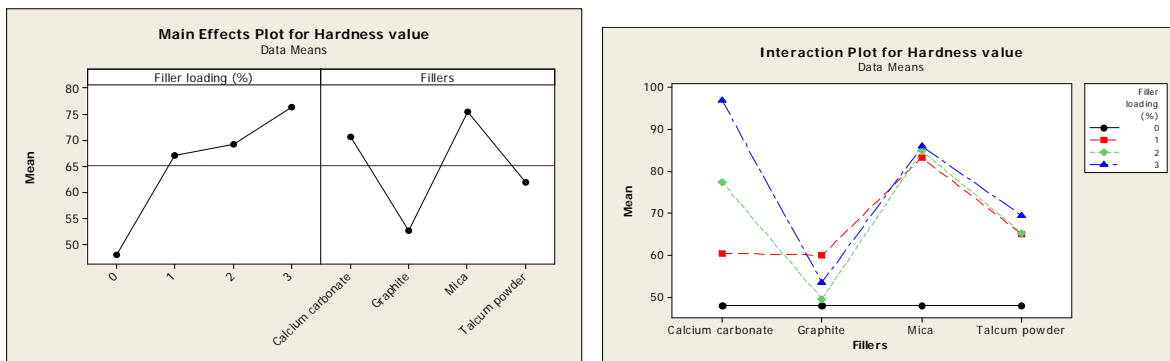


Figure 6 Response graphs for Hardness Test

2. Wear Test

Pin-on-disc test apparatus, of Ducom Company Bangalore (India), was used to investigate the dry sliding wear characteristics of the synthesized polystyrene composites. Composites were pressed against a rotating EN32 steel disc (hardness 65 HRC) of diameter 70mm and thickness 8mm by applying the constant encounter normal load of 2kg ($\approx 20N$) at a room temperature about 25°C. The speed of the rotating disc was controlled at a constant speed of 500±2 rpm. The time for each sample was set for 5 minutes. The wear testing machine was microprocessor controlled, in which height loss and frictional force can be monitored simultaneously by a PC-based data logging system. The wear was calculated by using the following formula:-

$$\text{Wear / min (\%)} = \frac{\text{change in height}}{\text{original height}} \times 100$$

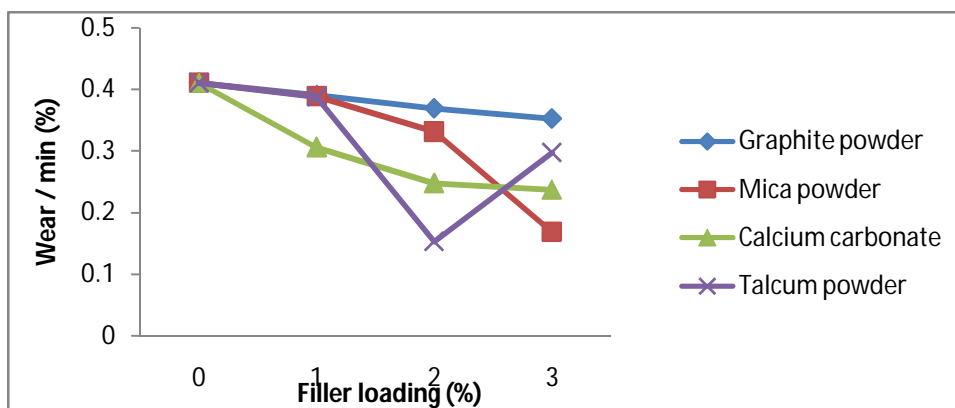


Figure 7 Effect of additives on Wear of synthesized polystyrene composites

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Table 5 ANOVA Table for Wear

Source	DF	SS	MS	F-value	P-value	% Contribution
Filler loading	3	0.061494	0.0204981	5.06	0.025	54.34
Fillers	3	0.015204	0.0050679	1.25	0.348	13.43
Error	9	0.036479	0.0040528			32.23
Total	15	0.113173				100

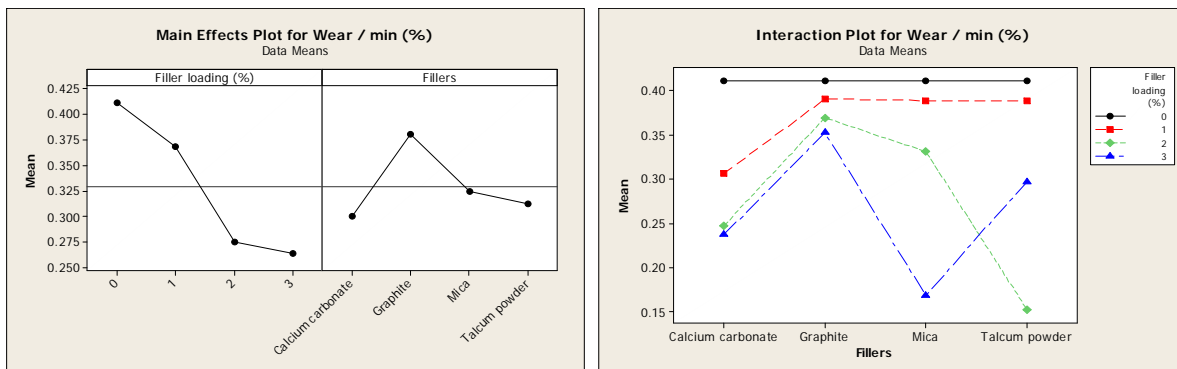


Figure 8 Response graphs for Wear

V. DISCUSSION

1. Hardness Test

Figure 5 shows the effect of additives on hardness value of synthesized polystyrene composites. It was found that the value of Rockwell hardness for pure PS 48.0 in M-scale. In case of PS-graphite composites, as the concentration of graphite increases, hardness value increases at 1% graphite and then decreases at 2% and then further increases. The maximum increase was 25% in hardness value in case of PS-graphite composite. For PS-mica composites, as the percentage of mica increases to 1%, there is very high increase in hardness and then at 2% and 3% concentrations, it increases at small rate. It was seen that if percentage of mica further increased, then hardness value likely to increase. The maximum increase is 79.16% in hardness value in case of PS-mica composite. For PS-CaCO₃ composite, as the concentration of CaCO₃ changes from 1% to 3%, there was an increment in hardness value with high rate to a maximum value of 97.0, which was 102.08% increase in hardness value. It was due to good and accurate dispersion of CaCO₃ in the PS matrix. This improvement can be attributed to uniform dispersion of CaCO₃ in polymer matrix as reported by Bharat et. al [8]. The same pattern was found for PS-talcum powder composite, but rate of increment was less as compared to the calcium carbonate. This small change after 1% concentration was revealed due to the agglomeration of talcum powder in the PS matrix. So the maximum increase was 44.79% in hardness value in case of PS-talcum powder composite. Table 4 shows two way ANOVA table for hardness test. From the table, it can be depicted that the percentage contributions of filler loading and fillers are 45.04% and 30.76% respectively while percentage contribution of errors is 24.20%. The filler loading was significant because p-value was less than 5% while filler was insignificant because p-value was equal to 0.051 which is greater than 5% at 5% level of significance. The 24.20% error is the combination of various errors such as random error, experimental error, machining error, human error, and error during analysis, etcetera. Figure 6 shows the response graphs for hardness test in which main effects plot shows the mean value of the hardness while interaction plot shows the interaction among the different additives with the composites. Non parallel lines show the interaction effects.

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2. Wear Test

Figure 7 shows the effect of additives on wear rate of synthesized polystyrene composites. For pure PS the wear in percent was found to be 0.4106. In case of PS-graphite composites, as the concentration of graphite increases, the wear % decreases to 0.3906, 0.3690 and 0.3525 at 1%, 2% and 3% of graphite respectively. The maximum improvement of 14.15% was predicted at 3% loading of graphite in the PS matrix. For PS-mica composites, as the concentration of mica increases, the wear % decreases drastically. The maximum improvement of 58.96% was observed at 3% loading of mica. This improvement in wear resistance was due to the better mixing of the mica in the matrix. The improvement of 42.30% for PS-CaCO₃ composite, was found. For PS-talcum powder composites, the wear rate drastically reduced at 2% talcum powder to a minimum as compared to the above composites and the wear resistance was improved 62.83%. The flow characterization as reported by Shahabaz et.al [9] also shown the better improvement in composite. Figure 8 shows the response graphs for wear test in which main effects plot shows the mean value of the wear rate and variation of wear rate with respect to mean value while interaction plot shows the interaction among the different additives with the composites. Non parallel lines show the interaction effects i.e. more deviation from parallel lines mean high interaction effects and vice-versa. Table 5 shows two way ANOVA table for wear test and it was predicted that the percentage contributions of filler loading and fillers are 54.34% and 13.43% while 32.23% for errors. The filler loading was significant while insignificant for fillers at 5% level of significance.

VI. CONCLUSIONS

In the present work, polystyrene with four additives namely graphite powder, mica powder, calcium carbonate powder and talcum powder by in-situ polymerization was successfully synthesized and the following conclusions are drawn:

1. The better results are found in synthesized PS-talcum powder and PS-CaCO₃ composites because of good dispersion of talcum powder and CaCO₃ in PS matrix.
2. The hardness of synthesized PS was increased with all the additives with a maximum increase of 102.08% in synthesized PS-CaCO₃ composite and minimum increase of 25% in case of PS-graphite composite. The order of maximum improvement in hardness strength of PS is found to be Graphite powder < Talcum powder < Mica powder < CaCO₃ powder.
3. The contribution of filler loading is 45.04% and the contribution of fillers is 24.20% in case of hardness test was analysed by ANOVA.
4. The wear resistance of synthesized PS was improved with all the additives with a maximum improvement of 62.83% in case of synthesized PS-talcum powder composite and minimum improvement of 14.15% in case of synthesized PS-graphite composite. The order of maximum improvement in wear resistance of PS is found to be Graphite powder < CaCO₃ powder < Mica powder < Talcum powder.
5. The contribution of filler loading is 54.34% and the contribution of fillers is 13.43% in case of wear test was analysed by ANOVA.
6. The addition of fillers are found to be significant while concentrations of fillers insignificant at 5% level of significance.

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