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Fabrication of PMMA/$CaCO_3$ Nanocomposites: Optimization of Tensile Strength through Taguchi's Analysis

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Abstract- PMMA/$CaCO_3$ nanocomposites have been fabricated by in-situ polymerization. The influence of concentration and stirring speed on tensile strength have been studied. The experimental results were optimized using Taguchi's approach. L9 Orthogonal arrays of Taguchi, the signal-to-noise (S/N) ratio and the analysis of variance (ANOVA) are employed to find the optimal input parameter levels and to analyze the effect of these parameters on tensile strength. Experimental results showed that tensile strength PMMA/$CaCO_3$ nanocomposites increased up to 53 % at 0.6 wt. % $CaCO_3$ and 1000 rpm in comparison to corresponding pure PMMA. The experimental results and various statistical approaches namely, Taguchi methods, Conceptual S/N ratio and drew similar conclusions. The ANOVA analysis showed that speed is more dominant in comparison to concentration.

Index Terms- ANOVA analysis, In-situ polymerization, Nanocomposites, Tensile strength, Taguchi method.

I. INTRODUCTION

In the past decade, polymer nanocomposites are the materials which have attracted a great deal of research interest. Polymer nanocomposites have improved mechanical properties, such as high strength, toughness and hardness [1]. Final properties of polymer nanocomposites depend upon the methods of mixing of filler material and matrix material. The interaction, distribution and concentration of constituents depends upon compounding technique.

Generally in-situ polymerization method is used for synthesis of various Poly (methyl methacrylate) (PMMA) / inorganic nanocomposites, because the type of nanoparticles and the nature of polymer can vary a wide range to meet the requirements [1].

Poly (methyl methacrylate) (PMMA) is transparent, hard, stiff, low water absorption and outstanding outdoor weathering properties [2]. In plastic industries calcium carbonate ($CaCO_3$) is widely used mineral fillers because it is easily available around the world, easy to grind or reduce to a specific particle size, compatible with a wide range of polymer resins and economical. In addition, when the particle size is carefully controlled, $CaCO_3$ helps increase impact strength and flexural modulus (stiffness) and tensile strength [3].

A substantial amount of work has focused on PMMA as the matrix material. PMMA/$CaCO_3$ nanocomposites were synthesized by several researchers for the thermal and mechanical properties. The work done by Xiaoyu L et al.[4] which was with a PMMA/ modified nano-$CaCO_3$ nanocomposites, that results reported in this paper shows 35 % increased tensile strength with respect to pure PMMA by adding up to 1. wt. % fillers. However the work done by Nivin et al.[5] which was with a PMMA/TiO_2 nanocomposites, that results reported in this paper shows 40.47% increased tensile strength with respect to pure PMMA by adding 1.25 wt. % fillers. In the same way the similar type of result obtained by Yang et al [6] which was with a PMMA/silica nanocomposites, that results reported in this paper shows 48.97% increased tensile strength with respect to pure PMMA by adding 5 to 10 % wt. fillers. But the Kim et al [7] achieves the highest increment in tensile strength to 50 % by adding multiwall nanotube of 1% in PMMA matrix.

In conventional polymer composites, micron-size $CaCO_3$ is used as a filler material that is difficult to disperse in the polymer matrix, which leads to coarsely blended composites and hence agglomeration, poor physical and mechanical properties [8].

To overcome these problems, a new method has been reported for the synthesis of PMMA/ $CaCO_3$ nanocomposites by in-situ polymerization reaction. In present study, nanoparticles were added at different stirring speeds during reaction. It was observed that nano-$CaCO_3$ is comparatively well dispersed into the matrix at higher speed, due to which tensile strength shown improvement that may be useful for commercial applications. The settings of polymerization process parameters were determined by using Taguchi's experimental design method. Orthogonal arrays of Taguchi, the signal-to-noise (S/N) ratio and the analysis of variance (ANOVA) are employed to find the optimal levels and to analyze the effect of stirring speed and filler concentration on tensile strength.

II. MATERIALS AND FABRICATION TECHNIQUE

The methyl methacrylate (MMA) monomer with a purity ≥ 99% was purchased from Merk, the free radical initiator, benzoyl peroxide (BPO) with a purity > 98%, was provided by Loba Chemie. For the preparation of nanocomposites, nano-$CaCO_3$ provided by Sisco research laboratories Pvt. Ltd. was used. All other chemical were used without further purification.

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In this work nanocomposites have synthesised at three different speeds and at every level of speed the concentration of nano-$CaCO_3$ varied from 0.2% to 0.6 wt. % of pure MMA. So nine different composites have been synthesised along with one pure PMMA for the comparisons of properties.

Fig. 1 shows experimental setup, firstly 650 ml of MMA is taken into 1000 ml beaker. When the temperature of the water bath reached to 80℃ then beaker is clamped. Then a fixed quantity of free radical initiator BPO is added and mixed thoroughly by glass rod to initiate the reaction. Then speed of the stirrer is adjusted to 600 rpm by speed regulator. Now 0.2 wt. % of $CaCO_3$ nano powder is added. After that the solution was left for 90 min. under continuous stirring and a constant heat of 80 ℃ is provided to complete the reaction. After completion of reaction add content of beaker approx. 250 ml methanol (Non solvent) taken in a 1000ml beaker with mild agitation with glass rod. In the second and third series of experiments, speed of the stirrer changed to 800 rpm and 1000 rpm respectively and same procedure have been repeated to get the all composites as reported by Sajjad et al [8].

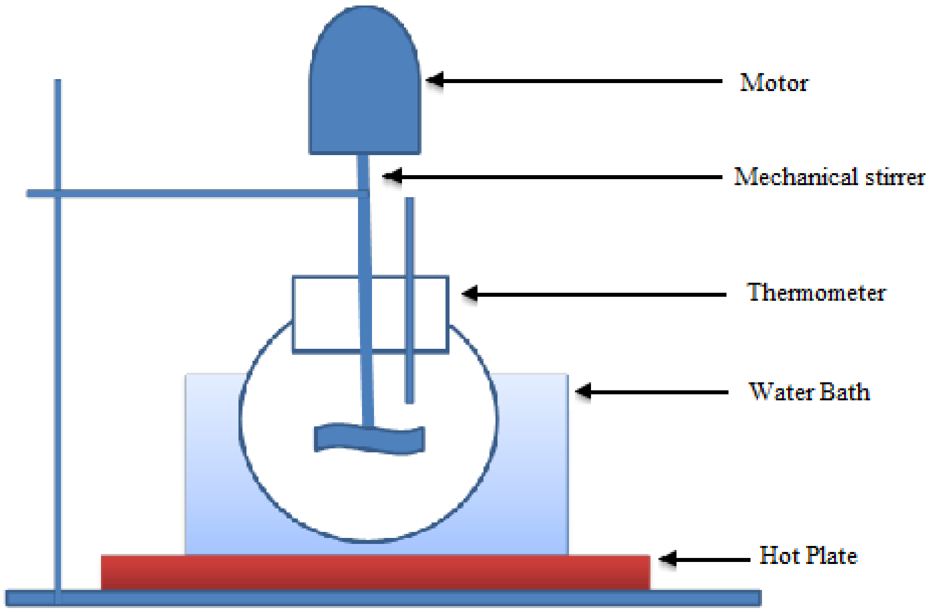

Figure 1: Experimental setup

III. RESULTS AND DISCUSSION

TENSILE STRENGTH

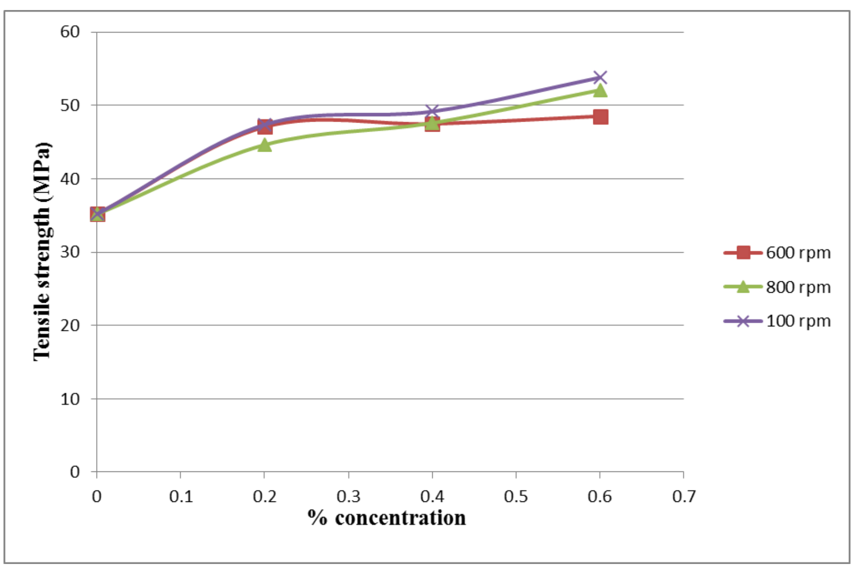

Figure 2: Variation of tensile strength versus concentration at different speeds

The fig. 2 shows that pure PMMA exhibits tensile strength of 35.17 N/mm^2. When the filler concentration is increased to a level of 0.2% the strength increased to 47.08 N/mm^2, 44.62 N/mm^2 and 47.38 N/mm^2 at 600 rpm, 800 rpm and 1000 rpm respectively.

Again, when further increment in filler concentration up to 0.4% the tensile strength improves to 47.48, 47.60 and 49.17 N/mm^2 at 600, 800 and 1000 rpm respectively.

Similarly, at 0.6% filler concentration level again increments in the tensile strength have been recorded to 48.53 N/mm^2, 52.09 N/mm^2 and 53.80 N/mm^2 at 600 rpm, 800 rpm and 1000 rpm respectively.

The figure also exhibits that when filler concentration is increased keeping stirring speed constant tensile strength increases. The tensile strength changes from 47.08 N/mm^2 to 48.53 N/mm^2 by changing filler concentration form 0.2 to 0.6 % at 600 rpm. Again, tensile strength increases to 44.62 N/mm^2 to 52.09 N/mm^2 and 47.38 N/mm^2 to 53.80 N/mm^2 at 800 rpm and 1000 rpm respectively.

The tensile strength of the all nanocomposites synthesised at different speed and concentration via in-situ polymerization shows a large increase over PMMA. This improvement can be attributed to uniform dispersion of nano-$CaCO_3$ in polymer matrix as reported by Bharat et al [9]. So, it also indicates that, with an increase of nano-$CaCO_3$ content in the nanocomposites, the tensile strength also increases as reported by Cooke et al, Koo et al and Huang et al [10] [11] [12].

The work done by Xiaoyu L et al.[4] which was with a PMMA/ modified nano-$CaCO_3$ nanocomposites, that results reported in this paper shows 35 % increased tensile strength with respect to pure PMMA by adding up to 1. wt. % fillers. However the work done by Nivin et al.[5] which was with a PMMA/TiO_2 nanocomposites, that results reported in this paper shows 40.47% increased tensile strength with respect to pure PMMA by adding 1.25 wt. % fillers. In the same way the similar type of result obtained by Yang et al [6] which was with a PMMA/silica nanocomposites, that results reported in this paper shows 48.97% increased tensile strength with respect to pure PMMA by adding 5 to 10 % wt. fillers. But the Kim et al [7] achieves the highest increment in tensile strength to 50 % by adding multiwall nanotube of 1% in PMMA matrix. In the current work the increase in tensile strength is 52.97% at 1000 rpm and 0.6% concentration level which is more in comparison to the previous work.

STATISTICAL ANALYSIS OF RESULTS

TAGUCHI ANALYSIS

Taguchi methods used widely in engineering analysis and to study the effect of multiple variables simultaneously involving a series of steps which must follow a certain sequence for the experiment to yield an improved understanding of process performance [13].

In this work nine experiments were conducted at different concentrations and speeds. Taguchi L9 orthogonal array was used, which has nine rows corresponding to the number of tests, with two columns at three levels. For the purpose of observing the degree of influence of the process parameters on tensile strength, two factors, each at three levels, are taken into account, as shown in Table 1. Table 2 Taguchi L9, showing the tensile strength values corresponding to each experiment.

Table 1: Process parameters

Variables	Code	Level 1	Level 2	Level 3
Concentration	A	0.2	0.4	0.6
Speed (rpm)	B	600	800	1000

Table 2: Taguchi L9

Experiment No.	A	B	Tensile Strength (N/mm^2)
1	0.2	600	47.08
2	0.2	800	47.48
3	0.2	1000	48.53
4	0.4	600	44.62
5	0.4	800	47.60
6	0.4	1000	52.09
7	0.6	600	47.38
8	0.6	800	49.17
9	0.6	1000	53.80
Pure PMMA			35.17

Table 3: Taguchi Analysis: Tensile Strength versus Concentration, Speed

Experiment	Concentration	Speed	Tensile Strength (N/mm^2)	S/N ratio
1	0.2	600	47.08	33.4567
2	0.2	800	47.48	33.5302
3	0.2	1000	48.53	33.7202
4	0.4	600	44.62	32.9906
5	0.4	800	47.60	33.5521
6	0.4	1000	52.09	34.3351

7	0.6	600	47.38	33.5119
8	0.6	800	49.17	33.8340
9	0.6	1000	53.80	34.6156

Table 4: Response Table for Signal to Noise Ratios

Larger is better

Level	Concentration	Speed
1	33.57	33.32
2	33.63	33.64
3[a]	33.99	34.22
Delta	0.42	0.90
Rank	2	1

Optimum level [a]

The results for various combinations of parameters were obtained by conducting the experiment as per the orthogonal array. The tensile strength value were analyzed using software MINITAB 15. Table 2. Taguchi L9, shows tensile strength values which is average of three replications. The effect of input parameters such as concentration and stirring speed on tensile strength has been analyzed using signal to noise ratio response shown in table 3. Taguchi Analysis: tensile strength versus Concentration, Speed. Table 4. Response Table for Signal to Noise Ratios, shows the ranking of process parameters using signal to noise ratios obtained for different levels for tensile strength.

Fig. 3 Effect of process parameters on Tensile strength, shows the effect of the process parameters on tensile strength values. A greater S/N value corresponds to a better performance. Therefore, the optimal level for the tensile strength is the level with the greatest S/N value. Based on the analysis of the S/N ratio, the optimal input factors for the tensile strength obtained at 0.6 % concentration (level 3) and 1000 (level 3).

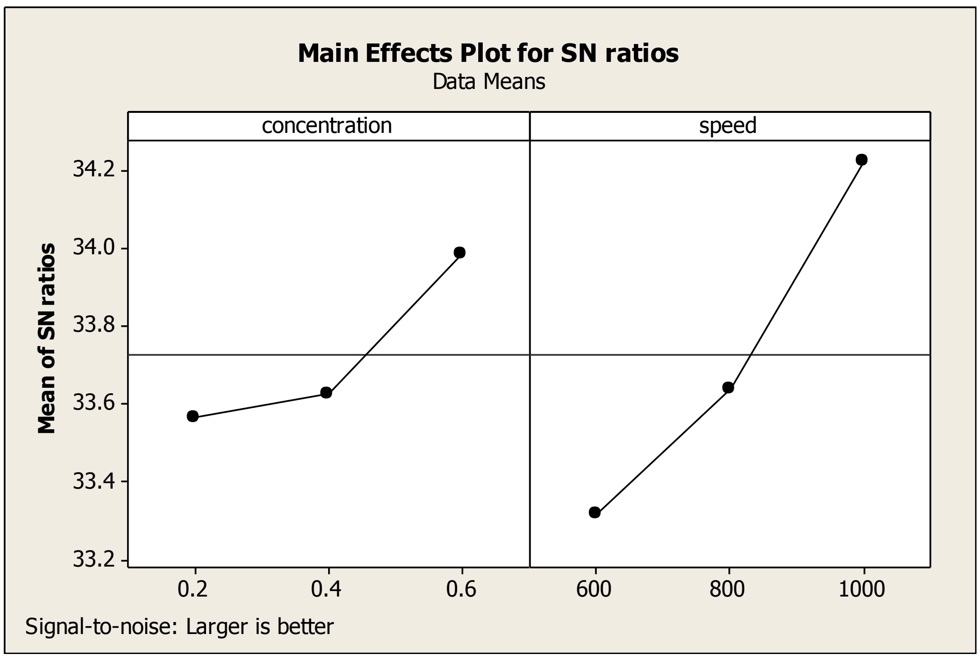

Figure 3: Effect of process parameters on Tensile strength

ANOVA ANALYSIS

ANOVA analysis of tensile strength

Table 5: Two-way ANOVA: Tensile strength versus speed, concentration

Source	DF	SS	MS	F	P	% contribution
Speed	2	10.0753	5.0376	1.88	0.266	16.40
Concentration	2	40.6082	20.3041	7.56	0.044	66.10
Error	4	10.7464	2.6866			17.49
Total	8	61.4299				

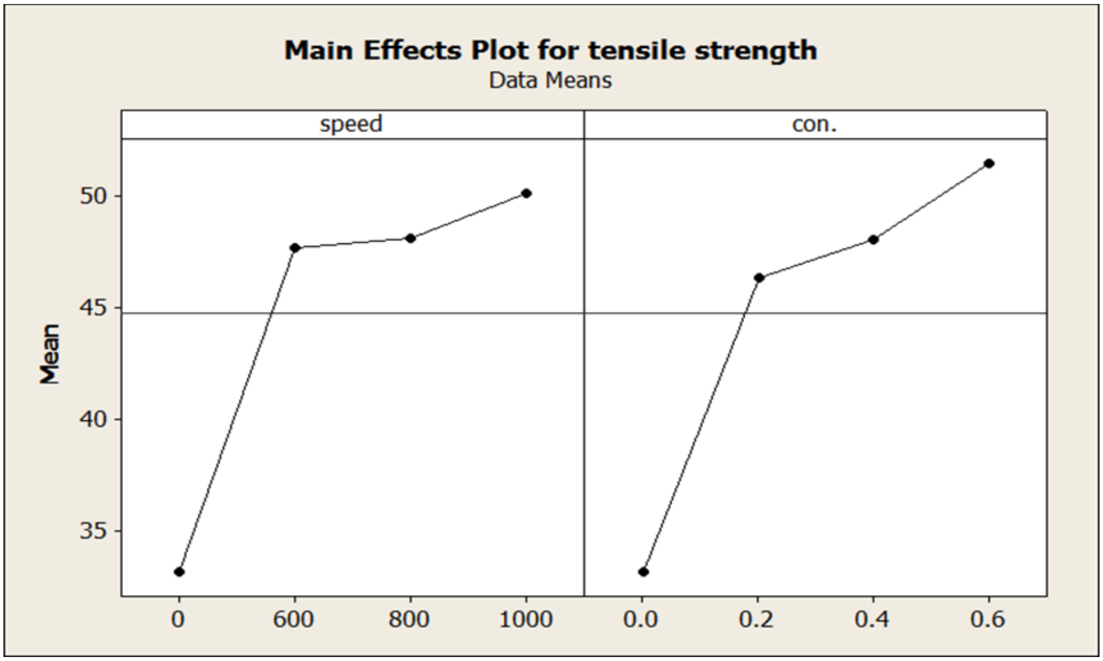

Figure 4: Main effect plot for tensile strength

IV. CONCLUSION

In the present experimental work we have successfully synthesized PMMA/$CaCO_3$ nanocomposites at different concentration and different stirring speeds by in-situ polymerization. In addition to this following conclusions are drawn from the analysis.

1. The tensile strength of composites have increased by the incorporation of nano-$CaCO_3$ powder.
2. The experimental results show that (i) concentration and (ii) speed are the important parameters for the enhancement of tensile strength.
3. Taguchi methods, Conceptual S/N ratio and ANOVA approaches for data analysis drew similar conclusions
4. For tensile strength, the significant parameters are concentration followed by stirring speed. The maximum increase in tensile strength is 53 % at 1000 rpm and 0.6% concentration level.
5. In case of tensile strength, the role of concentration and stirring speed are 66.10% and 16.40% respectively as analysed by ANOVA.
6. The optimum conditions for maximum tensile strength is calculated at 0.6 % concentration and 1000 rpm by Taguchi's optimization method..

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