



Application of Taguchi method for theoretical prediction of optimum parameters in rubber nano-composites

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ABSTRACT

We know that Taguchi method is popular for predicting various optimum parameters by conducting only a small number of simulation experiments. Orthogonal experimental design and analysis technique are used in this method to predict optimum parameters theoretically. In this research we used Taguchi method for optimization of mechanical properties of nitrile rubber based nanocomposites. Tensile strength and Mooney viscosity being the most important properties of rubber products, were selected for optimization (condition kept was higher the better). Variable for experiments were Nano clay, precipitated silica and ionic liquid in varied combination. Analysis of variance (ANOVA) was performed to identify the significant factors affecting the response and the best possible factor level combination was determined.

Key words: Compounding, Ionic liquid, Minitab 18, Mooney viscosity, Nano clay, NBR, Precipitated silica, Taguchi method, Tensile strength

INTRODUCTION

Ionic liquids (ILs), a group of salts existing as liquids at relatively low temperatures, which are considered as desirable green solvents, has been developed as a new class of solvent for natural polymers. ILs has many attractive properties, such as negligible vapour pressures, enhanced reaction rates, improvement of selectivity and yields, and easy reuse of catalysts. The use of ionic liquids in rubber compounding enhances the cure characteristics and improves the ionic conductivities of rubber [1, 2]. It facilitates the dispersion of fillers and hence reduces the

curing time [3, 4]. Many investigations have been done on the nitrile rubber compounds using the liquids and the results were found to be quite promising

Anna et al. studied the influence of imidazolium based ionic liquids on the acrylonitrile-butadiene rubber composites. T_g was found to be decreased as the length of alkyl side chains was varied from ethyl to hexyl. It was also found that there was decrement in the mechanical properties of rubber compounds [5]. Anna et. al. also studied the influence of alkyl methyl imidazolium bis (trifluoromethylsulfonyl) imide [AMIM TFSI] on the carboxylated nitrile-butadiene(XNBR)/layered double hydroxide (MgAl-LDH) composites. The alkyl chain length was varied from methyl to hexyl and it was found that the hexyl side chain gave the maximum plasticizing effect in the material and most significantly increased the ionic conductivity of the material [6].

Nitrile butadiene rubber is a polar rubber, has good resistance to a wide variety of oils and solvents and hence it is widely used in products like oil seals, pipe protectors, automotive and aeronautical industries [7]. NBR in the unfilled form has very low tensile strength and is not electrically conductive. Silica and other fillers are used for reinforcement. It is found that nitriles exhibited the highest interaction with silica probably through the hydrogen bond interaction between the -CN group and silanol groups. Incorporating silica to NBR enhances its electrical insulating properties. Huang et al. consider that inorganic particles may reduce the carrier mobility in the composite materials, thereby increasing their electrical insulating properties [8, 9].

Taguchi method is a statistical method developed by Taguchi and Konishi [10]. Initially it was developed for improving the quality of goods manufactured (manufacturing process development), later its application was expanded to many other fields in Engineering, such as Biotechnology [11] etc. Professional statisticians have acknowledged Taguchi's efforts especially in the development of designs for studying variation. Success in achieving the desired results involves a careful selection of process parameters and bifurcating them into control and noise factors. Selection of control factors must be made such that it nullifies the effect of noise factors. Taguchi Method involves identification of proper control factors to obtain the optimum results of the process.

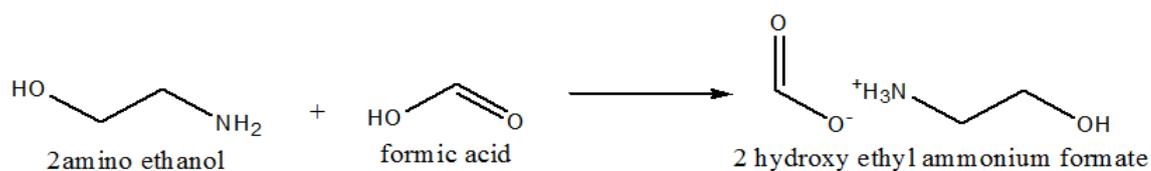
In the present studies different level of parameters such as precipitated silica, nano clay and ionic liquid have been chosen which are important ingredients and play important role in compounding and also affect the tensile strength. Taguchi method was used to optimize the levels of compounding ingredients to achieve optimum tensile strength and Mooney viscosity which are important properties of finished rubber products.

MATERIALS AND METHODS

Synthesis of IL

This liquid was synthesized by mixing of formic acid into 2-amino ethanol (Rankem, India) in equimolar ratios without further purification. The mixing was carried out in ice bath for keeping

temperature constant at 0°C to 5°C. The addition of acid the reaction mixture was stirred vigorously with the help of overhead stirrer. The addition took place in 1 hr time and after that the reaction mixture was stirred continuously for 24 hrs at room temperature, and this process resulted into viscous clear liquid [6].



Scheme 1. Synthesis of ionic liquid.

Rubber compound preparation:

The compounding of rubber was carried out on a two-roll mill available in the laboratory. Total 27 batches of rubber compounds were prepared with different loadings of the synthesized IL, precipitated silica and nano clay. The prepared compounds were vulcanised using the conventional sulphur based cross linking system. For vulcanisation compression moulding machine was used at 160°C. The compounding recipe of the 27 batches prepared are tabulated below and all ingredients are taken on phr (parts per hundred of rubber) basis.

Table 1. Formulations of rubber compounds

Ingredients (phr)	1	2	3
NBR	100	100	100
ZnO	5	5	5
MBTS	2	2	2
Stearic acid	1	1	1
Sulphur	2	2	2
silica	10	15	20
Nano clay	2.5	5	7.5
Ionic liquid	2	4	6

CHARACTERISATION:

Tensile strength:

Stress–strain tests were performed with a universal material testing machine (Future Foundation, Delhi, India) with a crosshead speed of 500 mm min⁻¹. The test specimens were prepared according to ISO 283 standard [12] instructions. To measure mechanical properties, different dumbbell-shaped specimens were punched from each rubber sample [13].

Mooney viscosity:

The Mooney viscosity of compounds was determined with a moving die (Future Foundation, Delhi, India) Mooney viscosity is a measure of the viscosity of a rubber. As per ISO 1796 the test piece was prepared. It is indicated by the torque required to rotate the disk embedded in a rubber compound specimen and enclosed in the die cavity under specified conditions [14].

DESIGN OF EXPERIMENTS:

In this paper taguchi method of experimental design is used to optimize compounding recipe to achieve highest Tensile Strength and Mooney viscosity of cured NBR compounds. This method uses orthogonal arrays (OA), which can stipulate the way of conducting the minimal number of experiments, which could give the full information of all the factors that affect the performance parameters [15]. The orthogonal array experiments are used here since they allow the simultaneous variation of many parameters and also the investigation of interactions between various parameters are possible. Statistical analysis, such as analysis of variance (ANOVA), is then employed to determine the relationship between the processing conditions and the response value, for example, tensile strength, Mooney viscosity. The main advantage of taguchi is that the number of experiments conducted in most of the cases is lesser than that of any other experimental design method using a statistical approach. Later regression analysis has been done to form the optimal solution of the problem. The taguchi method uses a statically measure of performance called signal to noise ratio. Noise is referred to as any factor that is too expensive to control. While “signal” represents the desired target for the final good product or process. It takes both the mean and variability into account. This is a performance measure to choose control levels that best cope with noise [16].

In the Taguchi method, the investigation parameters are called design variables or control factors which affect the output variable. Three factors are used in this study with three level for each factor. Table 1 shows the process control variables and their levels. The array chosen was L27 orthogonal array, to investigate the impact of three variables: Precipitated silica (variable A), nano clay (variable B) and ionic liquid (variable C) during the test. The mean of means characteristics was classified into three groups, smaller is the better, nominal is the better, and larger is the better. In this study, the mean of means large is the better was chosen as a larger tensile strength represents a better quality characteristic.

According to the taguchi design concept L27 orthogonal array is chosen for the experiments as shown in Table 2. In this study, the observed values of precipitated silica, nano clay and ionic liquid are varied at three levels. Each experimental trail is performed as per the conditions mentioned in L27 and optimization of the observed values in determined by comparing the standard method and analysis of variance (ANOVA) which is based on the taguchi method.

Table 2 Orthogonal array for L 27 design

L27	Level 1	Level 2	Level 3
1	1	1	1
2	1	1	2
3	1	1	3
4	1	2	1
5	1	2	2
6	1	2	3
7	1	3	1
8	1	3	2
9	1	3	3
10	2	1	1
11	2	1	2
12	2	1	3
13	2	2	1
14	2	2	2
15	2	2	3
16	2	3	1
17	2	3	2
18	2	3	3
19	3	1	1
20	3	1	2
21	3	1	3
22	3	2	1
23	3	2	2
24	3	2	3
25	3	3	1
26	3	3	2
27	3	3	3

We have used Minitab 18 software throughout the paper. Minitab calculates a separate mean for each combination of control factor levels in the design. Delta measures the size of the effect by taking the difference between the highest and lowest characteristic average for a factor [16]. The ranks in a response table help you quickly identify which factors have the largest effect. The factor with the largest delta value is given rank 1, the factor with the second largest delta is given rank 2, and so on.

Data analysis:

In this study the objective is to determine the effects of the parameters, to perform the analysis of variance (ANOVA) and to establish the optimum conditions based on the taguchi method. The performances calculated for each experiments of L27 by using the observed values. For tensile we considered that the higher is better and for Mooney nominal is better. Table 3 lists the taguchi analysis test results for Tensile strength and Mooney viscosity for signal to noise ration and Table 4 show results for means.

Table 3: Response table for signal to noise ratio considering higher is better

Level	Silica	Nano clay	IL
1	4.625	5.857	-2.599
2	3.656	2.872	6.674
3	3.790	3.342	7.995
Delta	0.969	2.985	10.594
Rank	3	2	1

Table 4: Response table for Means considering higher is better

Level	Silica	Nano clay	IL
1	40.51	44.84	37.04
2	46.80	49.39	52.00
3	62.56	55.64	60.83
Delta	22.05	10.8	23.79
Rank	2	3	1

RESULT AND DISCUSSION:

As per the earlier studies reported [17], IL was found to increase 19% tensile strength at 5 phr level. However, in our case it was found to improve tensile strength even at 6 phr level possibly due to favourable reinforcing effect created by IL in conjugation with nano clay. The best combination of the

An increase in the mechanical properties with the use of IL was attributed to the increased cross link density and enhanced interaction of inorganic filler with polymer matrix [18].

IL also precedes a plasticizing effect in rubber compounds, however, at higher level of usage may lead to significant decrease in mechanical properties. IL was found in general to reduce the optimum cure time of compounds.

The effects of efficiency of each ingredient for various level conditions in tensile strength and Mooney is shown by Figure 1 and 2, Mean plot of tensile and Mooney respectively. From the figure 2, it is clear that, Ionic liquid and Silica plays significant role. High content of Ionic liquid in compounding produces high tensile strength and Mooney. Similarly higher content of

precipitated silica produces higher tensile strength. Improved tensile strength was result of enhanced crosslink density due to presence of silica [19]. But in Mooney graph it shows less effect of silica than ionic liquid. The compounds have less effect of nano clay on both properties. But minor variation is due to enhanced rubber-clay interaction and effective dispersion of small sized clay molecules [20].

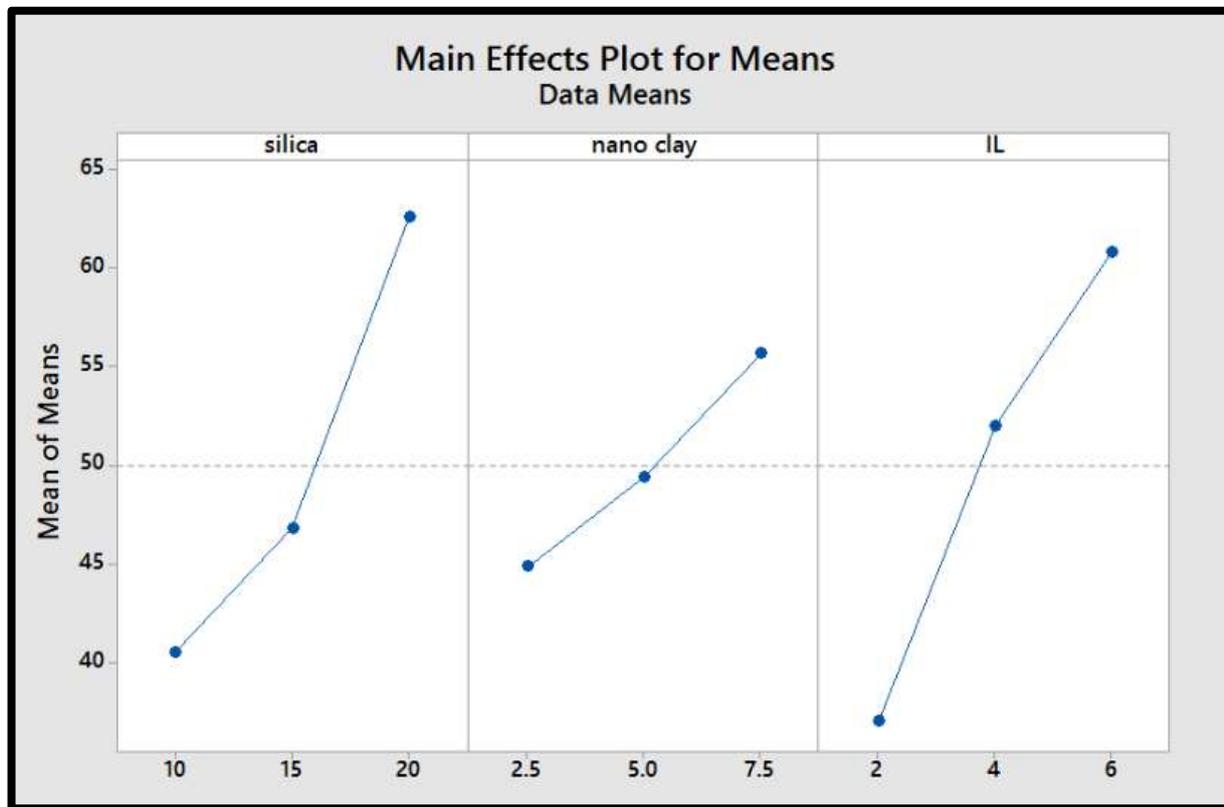


Figure 1: Means plot for tensile test

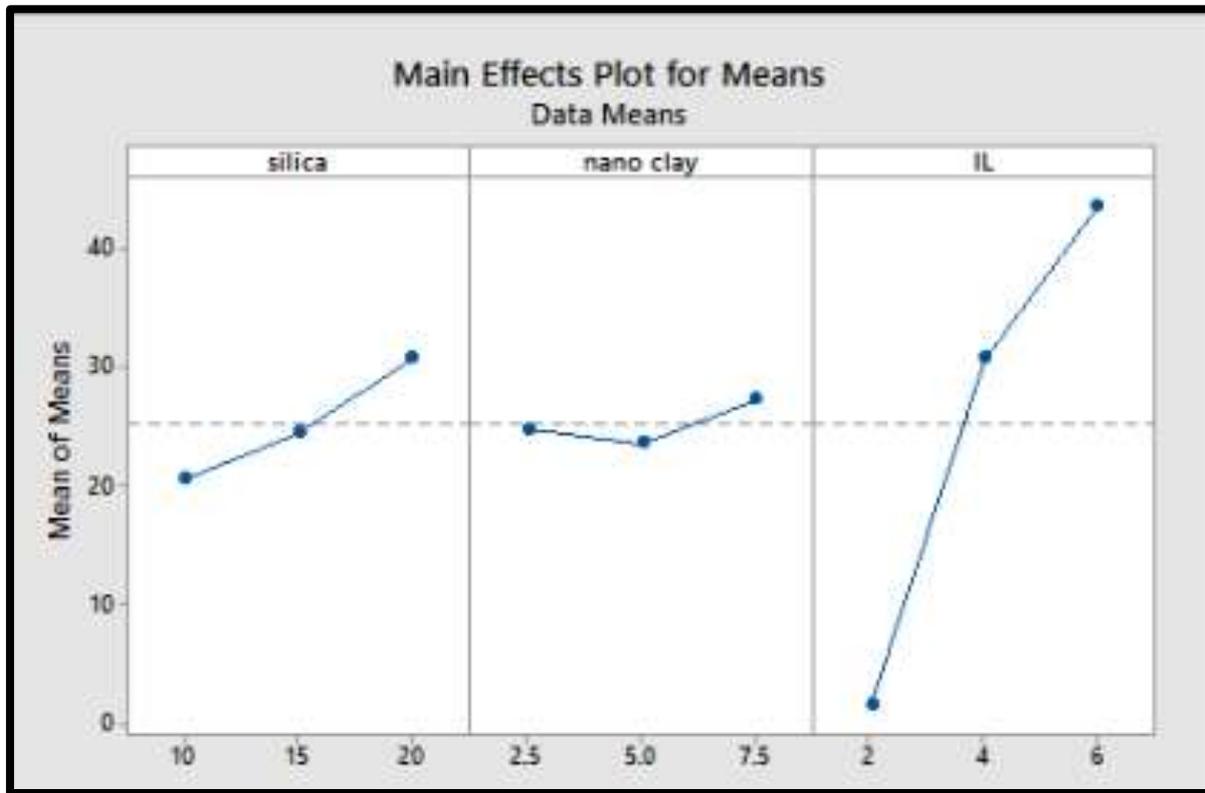


Figure 2: Means plot for Mooney test

CONCLUSION:

The experimental results show that contribution of different ingredients such as precipitated silica, nano clay, and ionic liquid. For this experiment our main focus is to generate higher tensile strength and Mooney viscosity. According to the proposed levels it was concluded that the maximum tensile strength could be achieved at 20 phr silica, 7.5 phr nano clay and 6 phr IL. Similarly for Mooney viscosity best combination is 20 phr silica, 7.5 phr nano clay and 6 phr IL. Also by using this taguchi technique we can find that higher content of Ionic liquid and Silica create high tensile strength and Mooney viscosity. Taguchi optimal solutions give the better results for compounding of NBR and it also reduces the number of experiments that are required for finding its performance metrics.

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