Experimental Optimization In Polymer BLEND Composite Preparation Based On Mix Level of Taguchi Robust Design

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ABSTRACT

L_{18} orthogonal array in mix level of Taguchi robust design method was carried out to optimize experimental conditions for the preparation of polymer blend composite. Tensile strength and neutron absorption of the composite were the properties of interest. Filler size, filler loading, ball mixing time and dispersion agent concentration were selected as parameters or factors which are expected to affect the composite properties. As a result of Taguchi analysis, filler loading was the most influencing parameter on the tensile strength and neutron absorption. The least influencing was ball-mixing time. The optimal conditions were determined by using mix-level Taguchi robust design method and a polymer composite with tensile strength of 6.33 MPa was successfully prepared. The composite was found to fully absorb thermal neutron flux of 1.04 x 10^5 n/cm^2/s with only 2 mm in thickness. In addition, the filler was also characterized by scanning electron microscopy (SEM) and elemental analysis (EDX).

ABSTRAK

Tatasusunan ortogon L_{18} dalam tahap campuran kaedah rekabentuk teguh Taguchi telah dilakukan untuk mengoptimumkan keadaan-keadaan eksperimen bagi persediaan rencam campuran polimer. Kekuatan tegangan dan penyerapan neutron rencam merupakan ciri-ciri yang penting pencerapan. Saiz pengisi, pemuatan pengisi, masa adunan kaedah bebola dan agen penyebaran dipilih kerana parameter atau faktor-faktor yang mana dijangka menjejaskan ciri-ciri komposit. Hasil dari analisis Taguchi, pemuatan pengisi adalah paling mempengaruhi parameter kekuatan tegangan dan penyerapan neutron. Masa adunan kaedah bebola dilihat memberi pengaruh yang tersedikit. Keadaan-keadaan optimum telah ditentukan dengan menggunakan campuran tahap kaedah rekabentuk teguh Taguchi dan satu polimer komposit dengan kekuatan tegangan 6.33 MPa telah berjaya disediakan. Komposit didapati menyerap sepenuhnya fluks neutron terma 1.04 x 10^5 n/cm^2/s dengan hanya ketebalan 2 mm. Sebagai tambahan, pengisi juga dicirika dengan kaedah mikroskop elektron pengimbasa (SEM) dan penganalisis unsur (EDX).

Keywords: *Taguchi robust design, Polymer blend composite, Tensile strength, Neutron absorption*

INTRODUCTION

Many research works have been carried out to study the effect of processing parameters on the resulted polymer composites (Ying Wan *et al*., 2005; Postawa and Koszkul, 2005; Sudarisman and Davies, 2008; Senol Sahin and Pasa Yayla, 2005; Mourad *et al*., 2005; Das *et al*., 2002). This is due to the fact that only at certain processing condition the composite achieves the expected properties. This condition is regarded as the optimal processing condition for that

particular system. However, to arrive at this optimal condition, many experimental trials are needed as the number of parameters involved increased. A complete set of parameter combination is established by using full factorial design. The purpose is to understand the relationship between the different processing parameters and finally arrive at the optimal processing condition. It is crucial to identify contribution of each parameter to the system as during processing the materials are subjected to mechanical and thermal stresses, radiation, and other types of influences simultaneously (Das *et al*., 2002). Full factorial design suffers a drawback in term of time and cost which lead to another technique, fractional factorial design. The technique investigates only a fraction of all the possible combinations. This technique reduces time and cost but still requires rigorous mathematical treatment, both in the design of the experiment and in the analysis of the results. This later leads to simplification and standardization of the fractional factorial design or known as Taguchi method (Roy, 1990).

Taguchi method is a robust design method which uses a combination of mathematical and statistical technique in a very systematic manner. The purpose of the Taguchi method is to develop an understanding of the individual and combined effects of various design parameters from a minimum number of experiments. This facilitates the establishment of the optimal combination of design parameters with the capability of reducing variations in the product quality by rendering the parameter design robust to the effects of noise. Noise is an uncontrollable factor which present in the experiment. Thus, the experimental condition having the least variability is the optimum condition. Variability is quantified by Taguchi method using signal to noise (S/N) ratio (Bendell *et al*., 1989; Taguchi, 1990). A high value of S/N implies that the signal is much higher than the random effects of the noise factors (Roy, 1990; Taguchi, 1993; Taguchi and Chowdhury, 2000) which is desirable in any given problem. Three type of (S/N) ratio is available (Montgomery, 1997) and it is selected based on the quality characteristic under study.

The current study involves the preparation of thermoplastic-natural rubber (TPNR) composites for use as neutron shielding material in TRIGA Mark II nuclear research reactor. High thermal neutron cross-section material such as B_4C is used as filler in order to provide shielding effect against thermal neutrons (Chilton *et al*., 1984; Shultis and Faw, 1996; Price *et al*., 1957). The properties of interest are the strength of this material and the ability to fully absorb thermal neutron flux of 1.04×10^5 n/cm^2/s. The ability is measured in terms of the material thickness required to achieve full absorption. The Taguchi method is used to optimize the experimental conditions in the preparation of this composite. Previous work successfully used this method in their optimization work. However, most of them used the same level in their parameters or factors (George *et al*., 2004; Kamyabi-Gol *et al*., 2009; Sudhir Kumar *et al*., 2008; Dyi-Cheng Chen and Cheng-Fu Chen, 2006). The current work illustrates the use of mix level Taguchi robust design which is often required in the experimental work.

EIGHT STEPS OF TAGUCHI METHOD

Basically Taguchi method involves 8 steps. The steps are illustrated below based on the current work.

i. Identification of the main function – to obtain high tensile strength and thermal neutron absorption properties.

ii. Identification of the noise factors, testing conditions and quality characteristics - variation of viscosity of liquid natural rubber and the filler humidity as noise and quality characteristic are tensile strength and thermal neutron absorption properties.

iii. Identification of the objective function to be optimized - both properties are in the "higher-the-better" type. S/N ratio is as defined as (Montgomery, 1997):

iv. Identification of the control factors and their levels - control factor are filler size, filler loading, ball mixing time and dispersion agent concentration. All factors have 3 levels except for dispersion agent concentration, which have 6 levels. This is called mix level approach. Control factor and their level are shown in Table 1. Noise factor and their level are shown in Table 2.

Table 1: Control factor and their level

Control Factors		Levels					
		1	2	3	4	5	6
A	Dispersant amount (%)	0	0.2	0.4	0.6	0.8	1.0
B	Filler loading (%)	10	40	60	-	-	-
C	Filler size (μ)	5	13.5	30	-	-	-
D	Mixing time (hrs)	12	18	24	-	-	-

Table 2: Noise factor and their level

Noise Factors		Levels	
		1	2
E	Filler humidity	High	Low
F	Viscosity of liquid natural rubber	High	Low

v. Selection of orthogonal array matrix experiment - L_{18} orthogonal array with 2 noise factor. The array is shown in Table 3 below.

Table 3: L_{18} orthogonal array for 1 control factor with 6 levels and 3 control factors with 3 levels.

Trial Number	Control Factors				Response		S/N
	A	B	C	D	E1F2	E2F1	
1	1	1	1	1	$Y_{1,1}$	$Y_{1,2}$	SN_1
2	1	2	2	2	$Y_{2,1}$	$Y_{2,2}$	SN_2
3	1	3	3	3	$Y_{3,1}$	$Y_{3,2}$	SN_3
4	2	1	1	2	$Y_{4,1}$	$Y_{4,2}$	SN_4
5	2	2	2	3	$Y_{5,1}$	$Y_{5,2}$	SN_5
6	2	3	3	1	$Y_{6,1}$	$Y_{6,2}$	SN_6
7	3	1	2	1	$Y_{7,1}$	$Y_{7,2}$	SN_7
8	3	2	3	2	$Y_{8,1}$	$Y_{8,2}$	SN_8
9	3	3	1	3	$Y_{9,1}$	$Y_{9,2}$	SN_9
10	4	1	3	3	$Y_{10,1}$	$Y_{10,2}$	SN_{10}
11	4	2	1	1	$Y_{11,1}$	$Y_{11,2}$	SN_{11}
12	4	3	2	2	$Y_{12,1}$	$Y_{12,2}$	SN_{12}
13	5	1	2	3	$Y_{13,1}$	$Y_{13,2}$	SN_{13}
14	5	2	3	1	$Y_{14,1}$	$Y_{14,2}$	SN_{14}
15	5	3	1	2	$Y_{15,1}$	$Y_{15,2}$	SN_{15}
16	6	1	3	2	$Y_{16,1}$	$Y_{16,2}$	SN_{16}
17	6	2	1	3	$Y_{17,1}$	$Y_{17,2}$	SN_{17}
18	6	3	2	1	$Y_{18,1}$	$Y_{18,2}$	SN_{18}

A1 represents dispersant amount at 0 %. Other notation follows accordingly.

vi. Conduct the matrix experiment.

vii. Data analysis and estimation of optimum performance characteristics

viii. Verification of experiment

EXPERIMENTAL PROCEDURE

Filler treatment

Boron carbide is added with neopentyl(dially)oxy tri(dioctyl)pyrophosphate titanate (Lica 38) as dispersant agent in various concentration ranging from 0.2-1.2% of filler weight. For each concentration, the filler and dispersant is ball

mixed using horizontal mixer at 60 r.p.m. The mixing time is varied according to 12 hours, 18 hours and 24 hours. The treated boron carbide is then washed with the solvent by stirring for 1 minute in order to remove excessive dispersant. The boron carbide.particles were separated from the solvent by centrifugal technique at 4000 r.p.m for 5 minutes. Finally the boron carbide is dried in the oven at 80°C for at least 24 hours.

Composite preparation

Thermoplastic-natural rubber used in this study was incorporated with various percentage of boron carbide (B_4C). Treated boron carbide with natural rubber (NR), thermoplastic (HDPE) and liquid natural rubber (LNR), were compounded in Haake twin blade internal mixer for a predetermined optimum time and mixing scheme. Finally the composite compound was heated press to form a slab sample with 1mm in thickness.

Filler and composite characterization

The treated filler are subjected to chemical analysis (EDX) to ensure the present of dispersant agent. This is achieved by monitoring the element of P and Ti in the sample which comes from the dispersant agent. Typical EDX spectra for treated filler with 0.2% LICA is shown in Fig. 1.

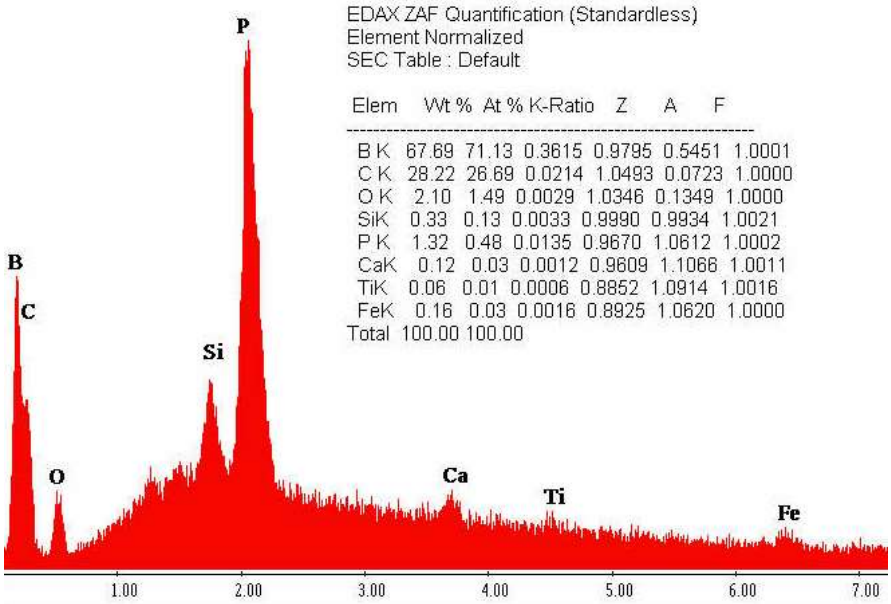
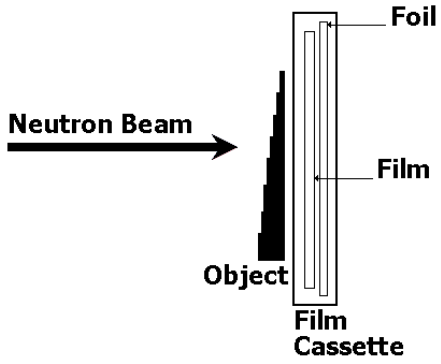


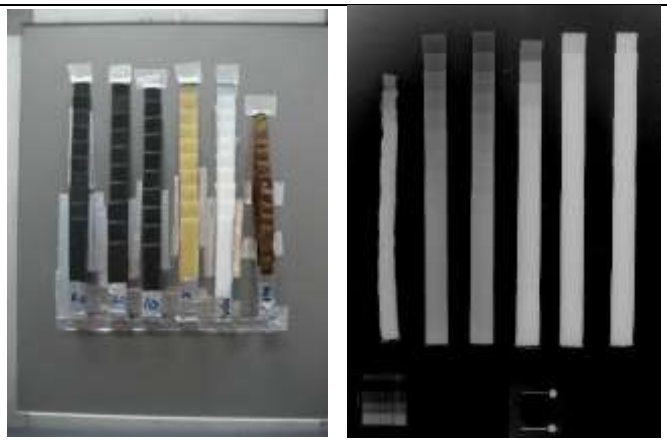
Fig. 1: EDX spectra for treated filler with 0.2% LICA.

The particle morphology is also observed by using SEM- FEI Quanta 400 model. The composite is subjected to tensile test according to ASTM 412. The reading is from the average of 6 tests. Neutron absorption test is carried out at TRIGA Mark II research reactor. The thermal neutron flux at the sample stage is 1.04×10^5 n/cm^2/s. The technique used to evaluate the neutron absorption in the sample is using neutron radiography technique (P. Von Der Hardt and H. Rottger, 1981). The technique utilizes Gadolinium converter foil and SR45 KODAK film placed in a film cassette. The sample is in the form of step-wedge ranging from 1-10mm in thickness. The arrangement is shown in Fig. 2. The sample is exposed to neutron beam for 15 minutes at the TRIGA Mark II beam port. The sample is characterized by analyzing the resulted radiography film. The film is developed and subjected to density measurement. Full neutron absorption is identified by measuring the image density formed in the exposed film. Figure 3(a) and 3(b) shows typical front view radiographic arrangement and exposed film. Then the thickness samples that are able to fully absorb neutron is determined.



(a)

Fig. 2: Radiograph arrange.- side view



(b)

Fig.3: (a) Sample arrangement on the film cassette, (b) Image formed on the exposed film.

RESULTS AND DISCUSSIONS

Table 4: S/N results for tensile strength and neutron absorption test

Trial Number	Control Factors				Response for Tensile Strength(MPa)		S/N ratio (dB)	Response for Neutron Absorption Test (mm^{-1})		S/N ratio (dB)
	Dispersant Amount (%)	Filler Loading (%)	Filler Size (μ)	Mixing Time (Hours)	E1F2	E2F1		E1F2	E2F1	
1	0	10	5	12	7.18	7.35	17.22	0.13	0.12	-18.08
2	0	40	13.5	18	5.28	5.11	14.31	0.48	0.48	-6.37
3	0	60	30	24	2.55	2.67	8.32	0.48	0.49	-6.32
4	0.2	10	5	18	7.27	7.39	17.30	0.12	0.12	-18.10
5	0.2	40	30	24	5.28	5.11	14.31	0.50	0.49	-6.12
6	0.2	60	30	12	2.49	2.59	8.10	0.49	0.50	-6.11
7	0.4	10	13.5	12	6.50	6.23	16.07	0.12	0.12	-18.09
8	0.4	40	30	18	5.11	4.83	13.92	0.50	0.50	-6.03
9	0.4	60	5	24	3.10	3.29	10.08	0.48	0.48	-6.32
10	0.6	10	30	24	5.42	5.42	14.68	0.12	0.12	-18.19
11	0.6	40	5	12	5.78	5.56	15.06	0.50	0.50	-6.04
12	0.6	60	13.5	18	2.91	2.93	9.31	0.49	0.49	-6.24
13	0.8	10	13.5	24	6.42	6.51	16.21	0.12	0.12	-18.09
14	0.8	40	30	12	5.21	4.87	14.04	0.46	0.45	-6.80
15	0.8	60	5	18	3.78	3.85	11.63	0.49	0.49	-6.22
16	1.0	10	30	18	5.30	5.21	14.41	0.12	0.12	-18.23
17	1.0	40	5	24	5.70	5.51	14.97	0.45	0.46	-6.83
18	1.0	60	13.5	12	2.83	2.88	9.10	0.49	0.49	-6.21

Table 5(a): Average performances of all the factors at different levels for tensile strength.

Factors	S/N					
A	S/N_{A1}	S/N_{A2}	S/N_{A3}	S/N_{A4}	S/N_{A5}	S/N_{A6}
	13.28	13.23	13.36	13.02	13.96	12.83
B	S/N_{B1}	S/N_{B2}	S/N_{B3}	-	-	-
	15.98	14.43	9.42	-	-	-
C	S/N_{C1}	S/N_{C2}	S/N_{C3}	-	-	-
	14.38	13.22	12.24	-	-	-
D	S/N_{D1}	S/N_{D2}	S/N_{D3}	-	-	-
	13.26	13.48	13.10	-	-	-

Table 5(b): Average performances of all the factors at different levels for neutron absorption test.

Factors	S/N					
A	S/N_{A1}	S/N_{A2}	S/N_{A3}	S/N_{A4}	S/N_{A5}	S/N_{A6}
	-10.26	-10.11	-10.15	-10.16	-10.37	-10.43
B	S/N_{B1}	S/N_{B2}	S/N_{B3}	-	-	-
	-18.13	-6.36	-6.24	-	-	-
C	S/N_{C1}	S/N_{C2}	S/N_{C3}	-	-	-
	-10.26	-10.19	-10.28	-	-	-
D	S/N_{D1}	S/N_{D2}	S/N_{D3}	-	-	-
	-10.22	-10.20	-10.31	-	-	-

Table 6: Pooled ANOVA of S/N for tensile strength

Source	SS	DOF	V	F-ratio	SS’	P(%)
A	2.23	5	0.45	Pooled	-	-
B	141.08	2	70.54	639.86	140.57	88.92
C	13.69	2	6.84	62.08	13.18	8.33
D	0.44	2	0.22	Pooled	-	-
Error (pooled)	0.66	6	0.11	-	4.35	2.75
Total (T)	158.10	17	-	-	158.10	100

Table 7: Pooled ANOVA of S/N for neutron absorption

Source	SS	DOF	V	F-ratio	SS’	P(%)
A	0.25	5	0.05	Pooled	-	-
B	559.79	2	279.89	4035.2	559.69	99.85
C	0.03	2	0.02	Pooled	-	-
D	0.04	2	0.02	Pooled	-	-
Error (pooled)	0.42	6	0.07	-	0.84	0.15
Total (T)	560.53	17	-		560.53	100

The effect of processing parameters against tensile strength and neutron absorption is studied based on Figure 4(a) and Figure 4(b). It was found that mean S/N ratio for tensile strength is 13.279 dB and for neutron absorption is -10.244 dB. A high value of S/N is selected and for any factor which shows similar trend of S/N, that factor is considered could be set at any level. Table 9 summarizes the selection for each tensile strength and neutron absorption properties.

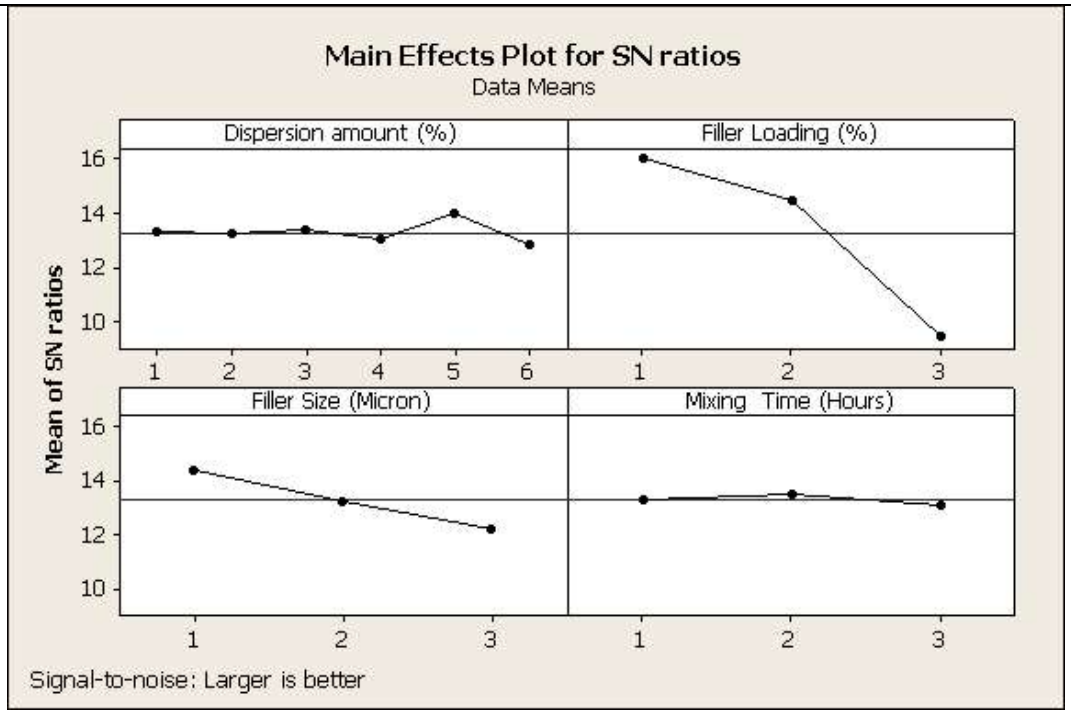


Figure 4(a): Response graph of all factors for tensile strength

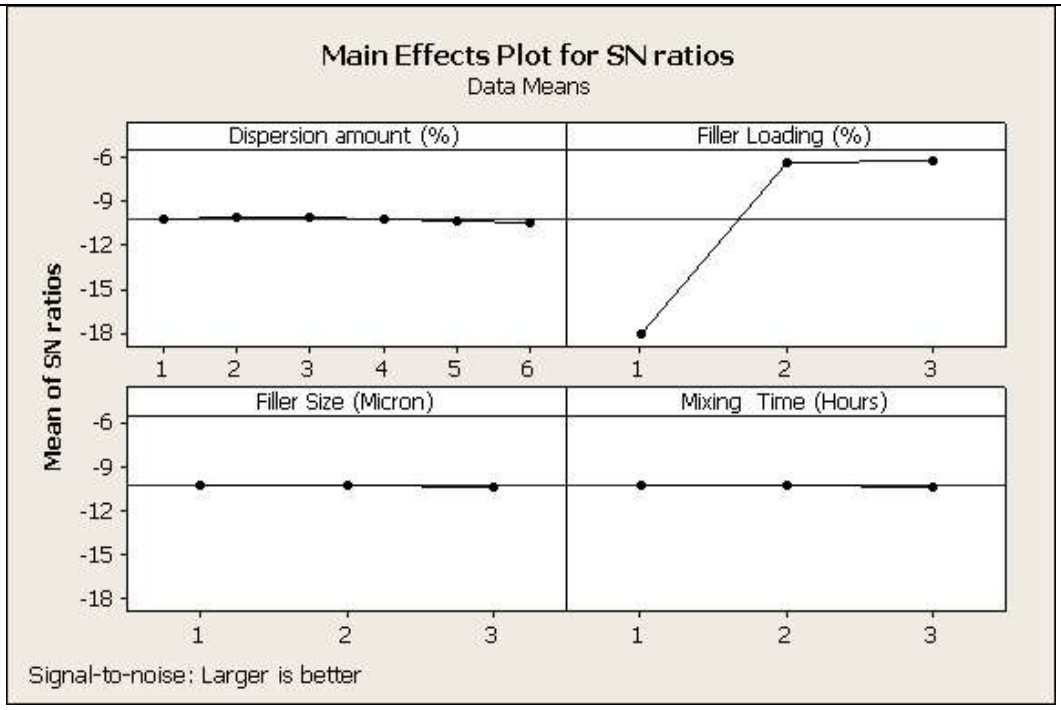


Figure 4(b): Response graph of all factors for neutron absorption

Table 8: Results of the prediction and confirmation experiment

Source	Levels	Tensile Strength (MPa)	S/N ratio	Neutron Absorption (mm^{-1})	S/N ratio
Prediction	A5,B2,C1,D1	6.454	16.196	0.474	-6.486
Confirmation	A5,B2,C1,D1	6.332	16.030	0.474	-6.477

Table 9: Results of S/N selection

	A	B	C	D
Tensile strength	5	1	1	Any level
Neutron absorption	Any level	2 or 3	Any level	Any level
Optimized selection	5	2	1	1

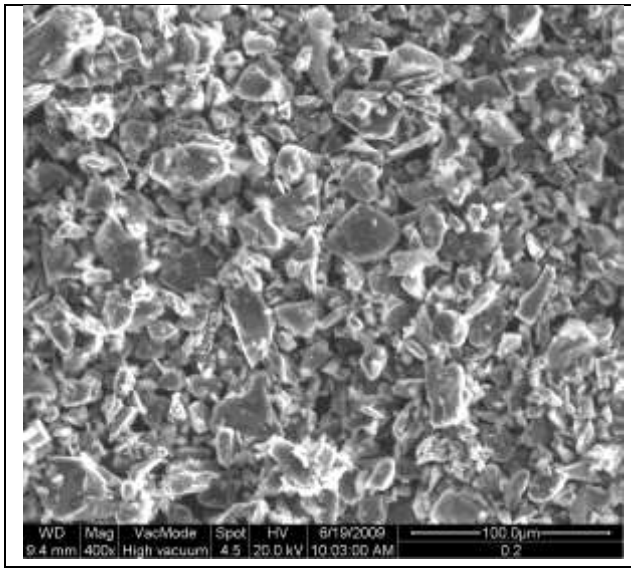


Fig. 7: Treated particle with 0.2% dispersant. (400 X)

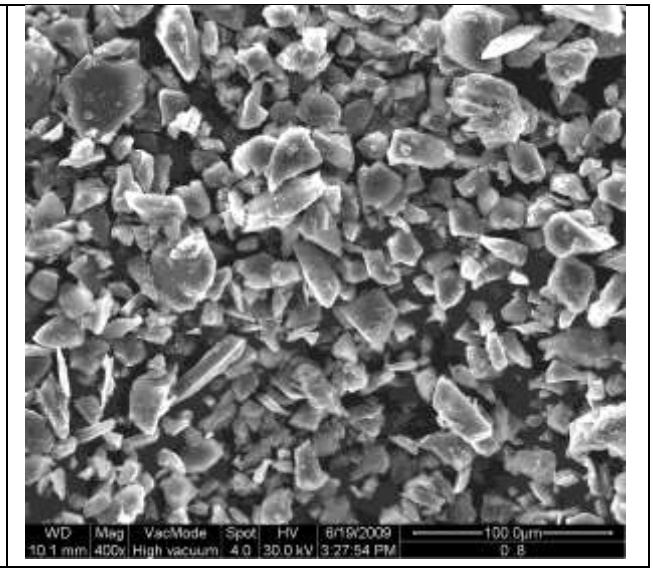


Fig. 8: Treated particle with 0.8% dispersant. (400 X)

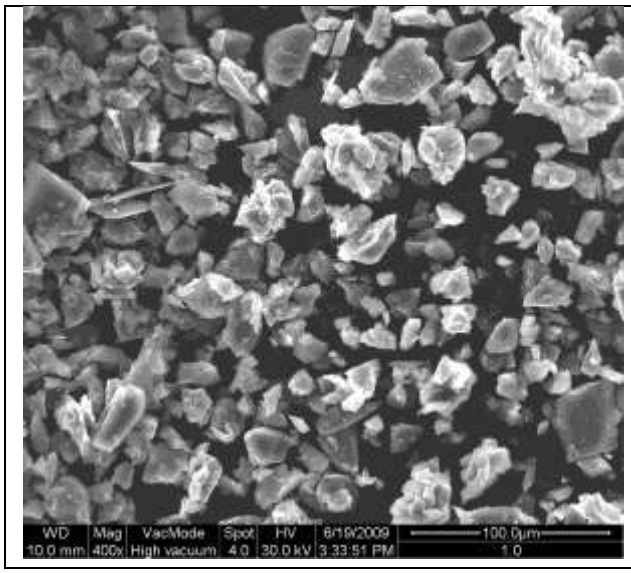


Fig. 9: Treated particle with 1.0% dispersant. (400 X)

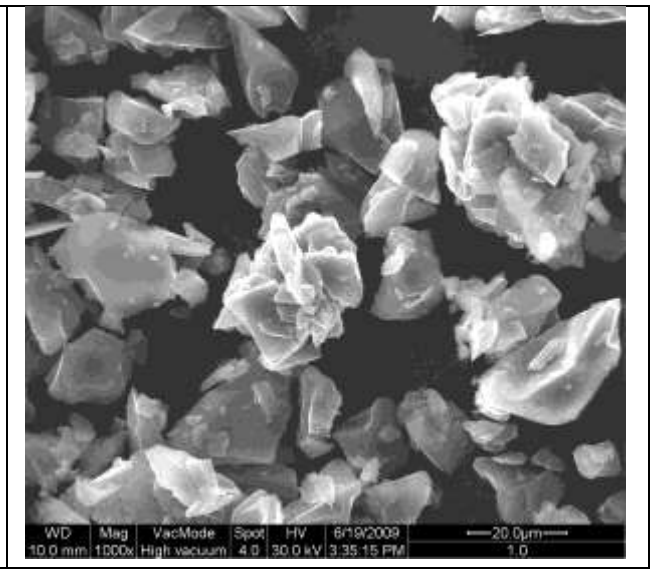


Fig. 10: As in Fig. 9 but at higher magnification (1000 X)

It is clear that for each quality characteristic under study exhibit its own factor combination. However, in this work only one factor combination is possible to be applied for both quality characteristics in order to achieve optimization. The said composite should have high neutron absorption and high tensile strength properties. It is found that factor B or filler loading is crucial for both quality characteristics. In this work, 10% filler loading is preferable to achieve high tensile strength but for neutron absorption, 40% filler loading is the minimum to fully absorb the neutron flux of 1.04×10^5 $n/cm^2/s$. Therefore the best option is to select 40% filler loading with a compromise in a slight reduction of tensile strength property. This is to ensure the composite could function as thermal neutron radiation shielding material with an acceptable tensile strength. Level 1 is selected for factor D as this would reduce the experimental time. By considering both of the quality characteristics, the optimal level of design parameters is A5, B2, C1 and D1.

A5 is selected as one of the optimized parameter even there is only a slight increase as compared to other levels. This leads to further analysis to support the selection by observing the particle morphology. It was found from Fig. 7 and 8 that particles treated with 0.2% dispersant are close together as compared to 0.8% dispersant. Particles at 0.8% dispersant are loosely detached from each other. As the amount of dispersant is increased to 1%, the particles start to form aggregates as shown in Fig. 9. Fig. 10 shows the particle aggregation at higher magnification. The aggregation consists of smaller particles that are attracted together due to stronger van der Waals force as compared to electrostatic repulsive force (Kimiyasu Sato et al., 2009). Thus A5 is the best option as supported by morphology analysis.

In order to find out statistical significance of various processing factors, ANOVA is performed on S/N data for both quality characteristics. The result of ANOVA for the quality characteristic tensile strength is given in Table 6. Here, the percent contribution indicates that filler loading contributes the most toward the variation observed on tensile strength followed by filler size. Dispersant amount and mixing time is insignificant parameter based on 95% confidence level. This agrees to the plots in Figure 4(a). Since the error is only 2.75%, all the major parameters are included and the error measurement is not excessive (Ross, 1989).

The result of ANOVA for the quality characteristic neutron absorption is given in Table 7. Filler loading was found the only to contribute toward the variation observed on neutron absorption. Other factors are insignificant parameter based on 95% confidence level. This agrees to the plots in Figure 4(b). Since the error is only 0.15%, all the major parameters are included and the error measurement is also not excessive (Ross, 1989).

The last step is to predict and verify the improvement of the quality characteristic using the optimal level of the design parameters. It is calculated based on Montgomery, in the case of tensile strength, the value of $[S/N]_m$ calculated from Table 4 is 13.279. Also, $[S/N]_i$ for A5, B2, C1 and D1 can be obtained from Table 5(a) and the values are 13.96, 14.43, 14.38 and 13.26, respectively. By using these values, $[S/N]_{predicted}$ can be written as;

$$[S/N]_{predicted} = (13.279) + [\,(13.96-13.279) + (14.43-13.279) + (14.38-13.279) + (13.26-13.279)\,]$$
$$= 16.196$$

Finally the value of predicted tensile strength is 6.454 MPa. The predicted neutron absorption can also be computed by the same procedure. Table 8 shows the comparison of the predicted tensile strength and predicted neutron absorption against the actual data. Avarage of 5 experiments is conducted to represent the actual data. It was found that the predicted values are consistent with the actual results. Consequently, optimization of polymer composite preparation which has high neutron absorption and tensile strength is successfully implemented based on mix level of Taguchi robust design.

CONCLUSIONS

The work could be considered as an evidence of the applicability of mix level Taguchi robust design method in determining the optimal condition with a minimum number of experiments. It was found in the present work that filler loading is the most influencing parameter for both neutron absorption and tensile strength. The least influencing parameter for both is ball mixing time. Optimization of polymer composite preparation which has high neutron absorption and tensile strength is achieved through the factor combination of 0.8% dispersant amount, 40% filler loading, 5μ filler size and 12 hours ball mixing time. This polymer composite is analyzed based on application for use as shielding against thermal neutron flux of 1.04×10^5 $n/cm^2/s$. Mix level design is very useful as it can provide a factor with more levels which could lead to a right level selection. This is shown in the present work for determination of dispersant amount which later confirmed by particle morphology observation.

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