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Optimum yield parameters of MWCNT/MnO_2 nanocomposite

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Abstract

The objective of this study is to illustrate the optimum parameters adopted to evaluate maximum yield of MWCNT/MnO_2 nanocomposite. In this paper, the effect of stirring time, Concentration of H_2SO_4 (aq) solution, solution temperature and drying time on yield of MWCNT/MnO_2 nanocomposite was investigated. The orthogonal array, signal-to-noise ratio, and percentage contribution of all parameters are employed to study the yield of MWCNT/MnO_2 nanocomposite. After conducting the experiments the yield was measured and Signal to noise ratio was calculated. With the help of graphs, optimum parameter values were obtained at parameter level $A_2B_2C_2D_1$.

The percentage contributions of parameters and regression analysis confirmed the concentration of H_2SO_4 has significant effect on yield of MWCNT/MnO_2 powder.

1. Introduction

Taguchi method optimizes the variation occurs in a process with the help of design of experiments. The objective of Taguchi method is to produce high quality product at low cost and the product or process is most robust with respect to noise factors. Taguchi's approach to parameter design provides the design engineer with an efficient method for determining near optimum design parameters for performance and cost [1, 2]. The Taguchi method use orthogonal arrays from design of experiments theory to study many variables with a small number of experiments. Using orthogonal arrays significantly reduces the number of experimental configurations to be studied. Gh. Tahmasebi et al.[3] presented optimization of Nano calcium carbonate production parameters to achieve the optimum production rate by using Taguchi method. It was found that the optimum level of process parameters were solution flow rate of 9 lit/min, gas flow rate of 20 lit/min, and solution concentration of 70 gr/lit and the production rate was enhanced by 168% in comparison to the mean value of the experimental results. I. Lozić et al. used Taguchi method of experimental design to evaluation the spray drier process parameters influence on particle size diameter of alumina powder [4]. The analysis of the results showed that in order to achieve the smallest particle size the best combination of parameters for is lower concentration and pump performance as well as higher value of inlet temperature and spray flow rate. Sheng-Meng Wang et al. [5]applied Taguchi's quality engineering to the in-situ supercritical fluid extraction and chemical derivatization of amphetamines from water to afford the optimal experimental conditions and hence achieved the highest recoveries of the analytes and the best robustness of the quantitation out of the least number of experimental runs. Achyut K. Pandaa used Taguchi method to identify the factors and their interactions that may affect the thermo-catalytic degradation of waste polypropylene to liquid fuel in a batch reactor [6]. The yield of liquid fuel in this process was greatly influenced by factors such as temperature, catalyst concentration and acidity of catalyst. From regression model, It was found that yield of liquid fuel in this process were highly dependent on temperature followed by acidity of catalyst and catalyst concentration.

S Kamaruddin et al. attempted to improve the quality characteristic of an injection molding product by optimizing the injection molding parameters using the Taguchi method [7]. The analysis of the results shows that the optimal combination for low shrinkage are low melting temperature, high injection pressure, low holding pressure, long holding time and long cooling time. Arshad Noor Siddiquee et al. focused on optimizing deep drilling parameters based on Taguchi method for minimizing surface roughness and experiments were conducted on CNC lathe machine using solid carbide cutting tool on material AISI 321 austenitic stainless steel [8]. Four cutting parameters such as cutting fluid, speed, feed and hole depth, each at three levels except the cutting fluid at two levels were considered. The cutting parameters such as speed, feed, and cutting fluid mainly influenced the surface roughness. Zahid A. Khan et al. investigated the effect of the wire electrical discharge machining process parameters on the surface roughness average and the kerf width of the stainless steel (SS 304) [9]. The pulse ON time is found to be the most influential factor for both the surface roughness and the kerf width. The analysis of variance showed that the pulse ON time was the most significant controlled factor for affecting the multiple responses in the electrical discharge machining. P. Jayaraman et al. presented a novel approach for the optimization of machining parameters on turning of AA 6063 T6 aluminium alloy with multiple responses based on orthogonal array with grey relational analysis [10]. They found that the feed rate, depth of cut were prominent factors which affect the turning of aluminium alloy. Md. Israr Equbal et al. studied hot closed die forging of spring saddle and DEFORM TM 3D FEM based simulation software was used to examine the effect of design and process parameters on forging load and billet temperature loss [11]. Grey relational analysis, along with Taguchi method were employed to optimize the forging load and billet temperature loss simultaneously.

In the present research, optimized yield parameters of MWCNT/MnO_2 nanocomposite has been investigated without consideration of interaction effect based on the Taguchi's method which is a powerful tool used in design of experiments.

2. Process optimization by using Taguchi method

2.1 Selection of yield parameters with their levels

Total nine experiments were conducted between 2 hr.-4hr. stirring time, 1.9M-2.1 M concentration of H_2SO_4(aq), 75 ^{0}C- 95 ^{0}C solution temperature, and 12 hr.-48 hr. drying time. The yield parameter and their levels is shown in table 1. Taguchi's L9 orthogonal array was used to design the experiments with four factors such as stirring time, Concentration of H_2SO_4 (aq) solution, solution temperature and drying time at three levels. Taguchi's L9 Orthogonal Array is shown in table2.

Table1: Yield parameters and their levels

Parameter	Level-1	Level-2	Level-3
Stirring time (A)	2	3	4
Concentration of H_2SO_4(aq) (B)	1.9	2.0	2.10
Solution temperature (C)	75	85	95
Drying Time (D)	12	24	48

Table 2: L9 Orthogonal Array

Parameter/Level			
1	1	1	1
1	2	2	2
1	3	3	3
2	1	2	3
2	2	3	1
2	3	1	2
3	1	3	2
3	2	1	3
3	3	2	1

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2.1.1 Materials

Multiwall Carbon nanotubes (purity > 95%) used as filler material and purchased from Redex Nano Laboratory, Uttar Pradesh, India.

Fig. 1: MWCNT

Fig.2: KmnO4

2.1.2 Synthesis of powder of MWCNT/MnO$_2$ nanocomposite

A novel technique [12, 13] has been used to synthesize MWCNT/MnO$_2$ nanocomposite. In short, the experimental synthesis process of the nanocomposite is described below.

A 500-ml beaker is charged with 0.1 gram multiwall carbon nanotube immersed in 250 ml at 1.9 M- 2.1M concentration level of boiling sulphuric acid (H_2SO_4) aqueous solution at stirring time 2 hr.- 4 hr. After dispersion, 4 gram $KMnO_4$ powder were added at different temperature parameter level ie. 75^{0}C-95^{0}C along with stirring. The yield was observed and the solution color changed from purple to brown. The system was then cooled down to room temperature. The obtained yield was washed with de-ionized water and acetone to remove the remaining ions, and then dried at 65 ^{0}C in a laboratory oven for 12hr.-48 hr. The experimental setup is shown in figure 3.

Fig.3: Experimental setup

3.Chemical characterization of MWCNT/ MnO$_2$ nanocomposite powder

3.1 XRD pattern of MWCNT/ MnO$_2$ nanocomposite

X-ray diffraction is non-destructive technique which gives the information about the crystallographic structure. The crystallographic structure of the nanocomposite was investigated MiniFlex, with Cu Kα radiation operated at 30kV and15mA, Start-10^0 Stop-70^0 and Step increment 0.02^0.

Figure 4(a) shows the xrd pattern of MWCNT. The XRD pattern of the MWCNT shows diffraction peaks at 26.6^0 indexed at (002) reflection of graphite [12-14].

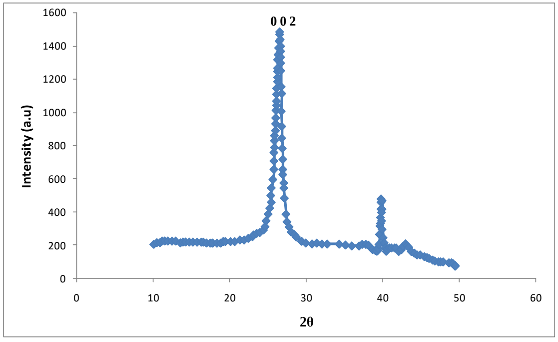

Fig. 4(a): XRD Pattern of MWCNT

The XRD data of MWCNT/MnO2 nanocomposite is shown in figure: 4(b). In addition to (002) reflection, the other diffraction peak can be indexed to the tetragonal α-MnO2 single-crystal structure, space group: I4/m (87) with lattice constants of a = 0.982 nm and c = 0.2863 nm and it also confirmed the formation of MWCNT/MnO2 nanocomposite [12, 13].

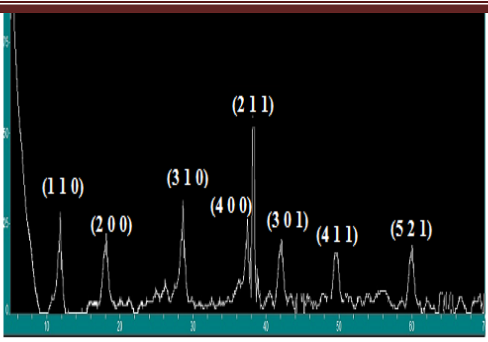

Fig.4 XRD pattern of 5 wt% MWCNT/MnO$_2$ nanocomposite

4. Experimental results analysis and discussion

4.1 Signal to noise ratio

The signal to noise ratio (S/N ratio) was used to measure the sensitivity of the quality characteristic being investigated in a controlled manner. The S/N ratio can be defined as S/N ratio [14].

$$\eta = -10\log(MSD) \qquad (1)$$

Where, *MSD: mean*-square deviation for the response.

To obtain optimum yield, the-higher-the-better response has been used. Table 3 shows the experimental response yield data and the corresponding S/N ratio.

Table3: Experimental yield and S/N ratio results for L9 orthogonal array

	Parameter/Level					
Experiment No.	Stiring time (A)	Concentration (B)	Solution temperature (C)	Drying Time (D)	Yield (gm)	S/N ratio
1	1(2)	1(1.9)	1(75)	1(12)	1.80	5.249022
2	1(2)	2(2.0)	2(85)	2(24)	1.94	5.756035
3	1(2)	3(2.1)	3(95)	3(48)	1.88	5.483157
4	2(3)	1(1.9)	2(85)	3(48)	1.85	5.343435
5	2(3)	2(2.0)	3(95)	1(12)	1.96	5.845121
6	2(3)	3(2.10)	1(75)	2(24)	1.89	5.529236
7	3(4)	1(1.9)	3(95)	2(24)	1.81	5.153571
8	3(4)	2(2.0)	1(75)	3(48)	1.89	5.529236
9	3(4)	3(2.10)	2(85)	1(12)	1.87	5.436832

Figure 5 shows the S/N response graph for total yield. The initial optimum combination of parameter level is $A_2B_2C_2D_1$ as shown in table 4. Fig.6 to Fig.9 shows the variation of yield with different parameters at different levels.

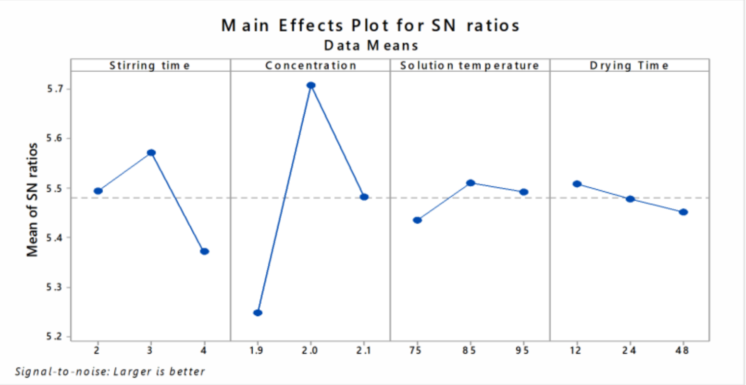

Fig. 5: S/N response graph

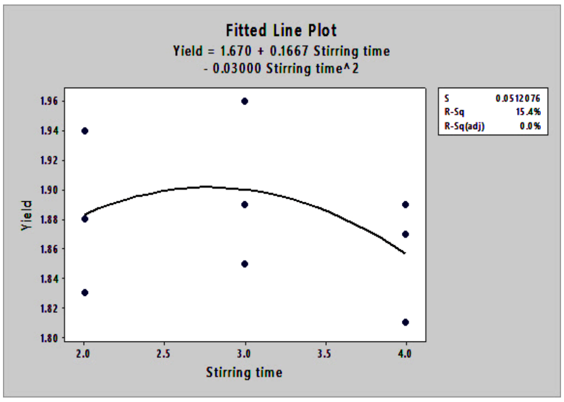

Fig.6: Fitted line plot yield with stirring time

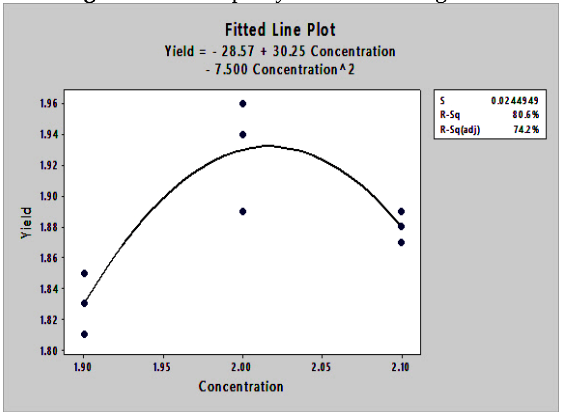

Fig.7: Fitted line plot yield with concentration

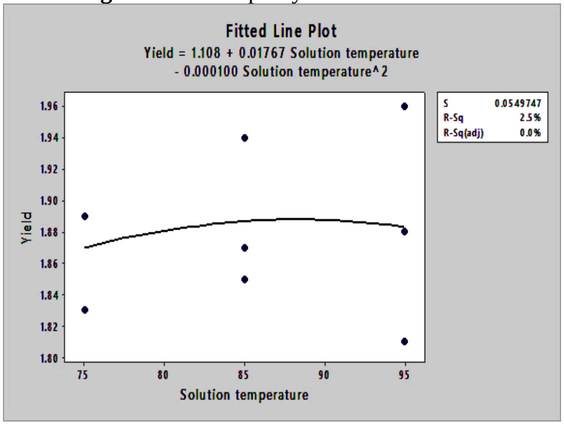

Fig.8: Fitted line plot yield with solution temperature

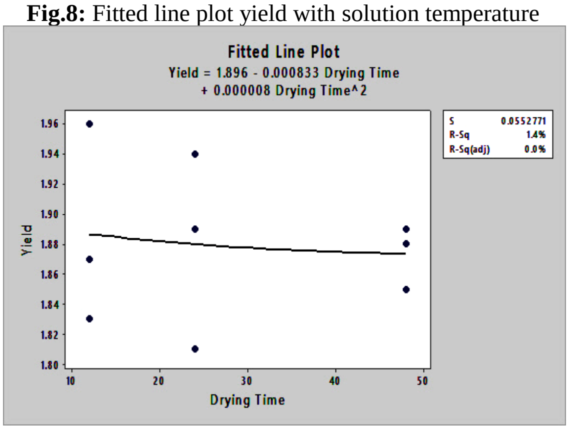

Fig.9: Fitted line plot yield with drying time

Table 4: Initial optimum level of parameters

Sl. No	Parameter	Level
1	Stirring time	3 hr.
2	Concentration of H_2SO_4(aq)	2.0M
3	Solution temperature	85^0C
4	Drying Time	24 hr.

4.2 Analysis of Variance (ANOVA)

The analysis of variance is used to find the relative importance of parameters on yield of MWCNT/ MnO_2 nanocomposite. The contributions of parameter concentration of H_2SO_4 (aq) was 80.9146%. Similarly, the contribution of other parameters is shown in table 5. The concentration of H_2SO_4 has significantly effect on yield and the yield was maximum at 2.0 M.

Table 5: Percentage contributions of parameters

Source	DF	Seq SS	% contribution
Stirring time	2	0.060704	1.53765
Concentration	2	0.319438	80.9146
Solution temperature	2	0.009524	2.4125
Drying Time	2	0.005117	1.2962
Residual Error	0	-	-
	8	0.394784	-

4.3. Regression Analysis

The regression analysis gives the relationship between yields with different parameters. The relationship is given below.

Yield=1.373-
0.0133 Stirring time+ 0.250 Concentration+ 0.00067 Solution temperature - 0.00036 Drying Time

The Regression Analysis confirmed the effect of concentration of H_2SO_4 yield was significant in comparison to another factors consideration.

5.Conclusions

The yield of MWCNT/MnO_2 was measured under different experimental conditions. The S/N response graph shows initial optimum combination of parameter level $A_2B_2C_2D_1$. The percentage contributions of parameters confirmed the parameter concentration of H_2SO_4 has significant effect on response and optimum concentration level of H2SO4 is 2M solution. Regression analysis also confirmed process parameter concentration of H_2SO_4 has significant effect on response.

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