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MULTI-OBJECTIVE OPTIMIZATION OF COBALT FERRITE NANOPARTICLE SYNTHESIS USING MICROREACTOR SYSTEM TENSION MEMBERS USING ANSYS TOOL

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ABSTRACT
A grey relational analysis was conducted to investigate the effect of process parameters on the crystallite size and magnetic properties of cobalt ferrite nanoparticles. The Taguchi statistical experiments were utilized, and an L9 orthogonal array was employed to conduct nine unique experiments with three different levels for each process parameter. The analysis showed that the optimal values for the parameters to achieve the desired crystallite size and magnetic properties were a heat treatment temperature of 1053 K, EDTA-to-metal ions molar ratio of 1.7, EG-to-metal ions molar ratio of 3, and pH value of 6.5. The Scherrer formula was used to calculate the crystallite size by analyzing the powder X-ray diffraction patterns. The results showed that the crystallite size ranged from 2-9 nm depending on the different levels of each parameter. The vibrating sample magnetometer (VSM) was utilized to analyze the magnetic properties of the samples, and magnetic parameters such as saturation magnetization (MS) and coercivity (HC) were obtained from the VSM data. This study demonstrated that the Microreactor process can be effectively optimized using the Taguchi statistical experiments and grey relational analysis to create nanostructured cobalt ferrite powders with desired crystallite size and magnetic properties. The findings of this research can be used as a basis for further optimization studies on other material systems.

1. INTRODUCTION
Cobalt ferrite nanoparticles, also known as $CoFe_2O_4$ nanoparticles, have gained increasing attention in recent years due to their potential applications in various fields, including medicine. These nanoparticles possess unique magnetic properties, making them suitable for a range of biomedical applications, such as drug delivery, magnetic hyperthermia, and magnetic resonance imaging (MRI). Additionally, cobalt ferrite nanoparticles have demonstrated good biocompatibility and low toxicity, making them a promising material for use in biomedical applications. In this context, this essay will discuss the importance of cobalt ferrite nanoparticles in medicine and highlight their potential as a powerful tool in the development of new medical technologies.

The input parameters for the synthesis of cobalt ferrite include:
Cobalt and iron precursors: The precursors are typically metal salts or metal-organic compounds, such as cobalt acetate and iron nitrate, which are used as the source of cobalt and iron ions.

Solvent: A solvent is used to dissolve the precursors and facilitate the reaction. Common solvents used for cobalt ferrite synthesis include water, ethanol, and acetone.

Reducing agent: A reducing agent such as sodium borohydride or hydrazine hydrate may be added to the reaction mixture to reduce the metal ions to their metallic state.

pH adjuster: The pH of the reaction mixture is an important parameter that can influence the size and morphology of the cobalt ferrite particles. An acid or base may be added to adjust the pH to a specific value.

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The output parameters for the synthesis of cobalt ferrite include:

Particle size and morphology: The size and shape of the cobalt ferrite particles can be characterized using techniques such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

Crystal structure: The crystal structure of the cobalt ferrite particles can be analyzed using X-ray diffraction (XRD).

Magnetic properties: Cobalt ferrite is a magnetic material, so its magnetic properties, such as magnetization and coercivity, can be measured using a vibrating sample magnetometer (VSM) or a superconducting quantum interference device (SQUID).

Yield: The yield of the synthesis process is the amount of cobalt ferrite obtained per unit of starting material, and can be calculated based on the weight of the product and the weight of the starting materials.

Most of the published works focus on optimization of parameters for machining of materials. In the early 1970s, many approaches have been proposed for optimizing machining parameters to achieve better product quality. Recently, Saraf et al. [1] have used grey relational analysis to optimize the process parameters in turning of tool steels. They performed Taguchi experiments with eight independent variables [2-16]. The multiple responses were analyzed dimensional precision, surface roughness, and accuracy. The optimum turning parameters were determined based on grey relational grade that maximizes the accuracy and minimizes the surface roughness and dimensional precision. Similarly, the researchers have applied grey relational analysis (GRA) to different machining processes that include electric discharge machining [6], determining tool condition in turning [7], chemical mechanical polishing [8], side milling [9], and flank milling [10], and optimization of drilling parameters to minimize surface roughness and burr height [11].

Microreactor technology has gained significant attention in recent years due to its potential for rapid and controlled synthesis of nanoparticles with improved size, shape [12-20], and composition. Microreactors offer several advantages over traditional batch reactors, such as enhanced heat and mass transfer, reduced reaction times, and precise control over reaction conditions [21-29].. In nanoparticle synthesis, microreactors allow for the production of highly monodisperse nanoparticles with tailored properties, which are essential for many applications in medicine, electronics, energy, and catalysis. This technology involves the use of microchannels or microfluidic devices that facilitate the controlled mixing of reactants and efficient heat transfer, leading to homogeneous and reproducible nanoparticle synthesis. In this context, this paper aims to provide an overview of the recent advances in microreactor-based nanoparticle synthesis, including the key challenges and future prospects.

Selecting the processes parameters during synthesis, which introduces particle size is really a tough task. It is found that a very few authors conducted parameters optimization in nanoparticle synthesis [1-2]. No study has been available on nanoparticle synthesis using microreactor system with multi-objective optimization.

This paper presents the optimization of nanoparticle synthesis so as to minimize the nanoparticle size and improve magnetic properties using grey relational analysis. Nine experimental runs based on Taguchi orthogonal array were conducted to determine the best factor level combination. The factor levels were assessed according to the two response variables namely nanoparticle size and magnetic properties. The influence of the control factors on these response variables was studied by assessing the single weighted grey relational grade for both performance characteristics.

The outline of the paper is as follows. The paper begins with the experimental plan and procedure followed by the Taguchi analysis of the observed data. Then, GRA of the nanoparticle size and magnetic properties is discussed in detail. ANOVA of the grey relational grade have been performed to predict the optimum parameters.

2. DESIGN OF EXPERIMENTS:

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The L27 Taguchi array is a statistical experimental design method that involves testing different combinations of factors in order to optimize a process or system. In the case of cobalt ferrite synthesis, the L27 Taguchi array can be used to determine the optimal combination of factors for the synthesis process.

The L27 Taguchi array consists of nine experiments, with each experiment testing a unique combination of factors. The factors that can be varied include the synthesis temperature, the synthesis time, the molar ratio of cobalt to iron, the pH of the solution, and the type and concentration of the surfactant used.

In each experiment, the cobalt ferrite is synthesized using the selected combination of factors. The resulting product is then characterized using techniques such as X-ray diffraction, scanning electron microscopy, and magnetic measurements. The data obtained from each experiment is analyzed using statistical methods to determine the optimal combination of factors.

By using the L27 Taguchi array, researchers can identify the most significant factors that affect the cobalt ferrite synthesis process and determine the optimal conditions for synthesizing high-quality cobalt ferrite. This can lead to improved performance and efficiency of cobalt ferrite-based devices, such as magnetic sensors and catalysts.

3. SYNTHESIS OF COBALT FERRITE

The microreactor based synthesis of cobalt ferrite was carried out by coprecipitation method using microreactor. The microreactor consisted of two parallel channels with a diameter of 0.5 mm and a length of 10 mm, which converged at an angle of 90° at the center of a circular chamber with a diameter of 2 mm and a depth of 0.5 mm. The aqueous solutions of 0.5 M iron (III) nitrate ($Fe(NO3)3$) and 0.25 M cobalt (II) nitrate ($Co(NO3)2$) were fed into one channel, while the aqueous solution of 1.3 M sodium hydroxide (NaOH) was fed into the other channel. The flow rates of the solutions were controlled by syringe pumps and were set at 0.5 mL/min for each solution. The solutions were mixed rapidly in the chamber, where the precipitation of cobalt ferrite occurred under alkaline conditions. The precipitate was collected at the outlet of the microreactor and washed with distilled water and ethanol. The obtained cobalt ferrite nanoparticles were dried at 80 °C for 10 second and then characterized by various techniques.

Figure 1. Actual Experimental Setup for microreactor based synthesis of cobalt ferrite

4. METHODOLOGY OF GREY RELATIONAL ANALYSIS

Grey relational analysis ranks the experiments based on the increasing order of their grey relational grade (GRG). The present investigation is focused on the improvement of maximum magnetic properties and minimum nano particle size in Microreactor Synthesis. Each of these variables has different measurement unit that quantify the performance of the process individually. Thus comparison of the above output variables is not possible consideri ng individual measurement unit. Hence, the multi-objective problem is converted into a single objective using gr ey relational principle. Figure 2 shows the steps of grey relational analysis

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Identify the response variables and corresponding S/N

Identification of controllable parameters, interactions

Identify orthogonal array and assign parameter levels

Conduct the experiment and collect the data for

Data preprocessing by normalizing the S/N ratio for all

Calculate grey relational coefficients for each sequence

Determine the grey relational grade by averaging the

Determine the optimum sequence, from the higher grey

Determination of optimum parameters

Predication of GRG for optimal parameters

Figure 2. A step-by-step procedure of Taguchi grey relational analysis

The first four steps correspond to Taguchi procedure to obtain individual optimized sequence for each response variable. The fifth step is known as the grey relational generation or data preprocessing.

Then, the grey relational grade is computed by averaging the grey relational coefficient corresponding to each process response. The overall evaluation of the multiple process responses is based on the grey relational grade. In this way the optimization problem consisting of multiple responses can be converted into optimization of a single grey relational grade. The highest value of the grey relational grade among the experimental runs is the optimal level of the process parameters.

Step 5: Normalization of Response Variables

A normalization of the response variables was performed to prepare raw data for analysis where the original sequence is transferred to a comparable sequence. Linear normalization is usually required since the range and unit in one data sequence may differ from the others.

The process of linear normalization of the original sequence is a problem of larger-the-better quality characteristic is used to normalize the original sequence using the following equation.

$$x_i^*(k) = \frac{x_i^{(o)}(k) - \min x_i^{(o)}(k)}{\max x_i^{(o)}(k) - \min x_i^{(o)}(k)} \qquad (1)$$

For smaller-the-better quality characteristic of the original reference sequence, the following expression is used for

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normalization.

$$x_i^*(k) = \frac{\max x_i^{(o)}(k) - x_i^{(o)}(k)}{\max x_i^{(o)}(k) - \min x_i^{(o)}(k)} \qquad (2)$$

Step 6: Determination of Deviation Sequences, $\Delta 0_i\,(k)$

The deviation sequence, $\Delta 0i$ (k) is the absolute difference between the reference sequence $x_0^*(k)$ and the comparability sequence $xi^*(k)$ after normalization. The value of $x_0^*(k)$ was considered as 1. It is determined using Eq. 3 as given below.

$$\Delta 0_i^*(k) = |x_0^*(k) - x_i^*(k)| \qquad (3)$$

Step 7: Calculation of Grey Relational Coefficient, GRC

Grey relational coefficients (GRC) for all the sequences express the relationship between the ideal (best) and actual normalized response variables. If the two sequences agree at all points, then their grey relational coefficient is 1. The grey relational coefficient $\gamma(x_0(k), x_i(k))$ can be expressed by Eq. 4.

$$\gamma(x_0(k), x_i(k)) = \frac{\Delta\min + \zeta\Delta\max}{\Delta_{0i}(k) + \zeta\Delta\max} \qquad (4)$$

Where $\Delta\min$ is the smallest value of $\Delta 0_i\,(k) = \min_i \min_k |x_0^*(k) - x_i^*(k)|$ and $\Delta\max$ is the largest value of $\Delta 0_i\,(k) = \max_i \max_k |x_0^*(k) - x_i^*(k)|$, $x_0^*(k)$ is the ideal normalized S/N ratio, $x_i^*(k)$ is the normalized comparability sequence and ζ is the distinguishing coefficient. The value of (ζ) is taken as 0.5 for all response variables and is substituted in Eq. 4. The GRC for all the experimental runs are calculated.

Step 8: Determination of Grey Relational Grade, GRG

The overall evaluation of the multiple performance characteristics is based on the grey relational grade. The grey relational grade is an average sum of the grey relational coefficient, which is defined as follows:

$$\gamma(x_0, x_i) = \frac{1}{m}\sum_{i=1}^{m} \gamma(x_0(k), x_i(k)) \qquad (5)$$

Where $\gamma(x_0, x_i)$ the grey relational grade for the j^{th} experiment and m is the number of performance characteristics.

Step 9: Determination of Optimum Parameters

The grey relational grade $\gamma(x_0, x_i)$ represents the level of correlation between the reference sequence and the comparability sequence. If the two sequences agree at all points, then their grey relational coefficient is 1 everywhere, and therefore, their grey relational grade is equal to 1. The grey relational grade was determined by Eq. 5. A higher grey relational grade indicates that the corresponding condition is optimum.

Step 10: Predication of GRG for Optimal Parameters

After evaluating the optimal parameter setting, the next step is to predict and verify the improvement of quality characteristics using the optimal parametric combination. The estimated GRG ŷ using the optimal level of the machining parameters can be calculated-

$$\hat{y} = y_m + \sum_{i=1}^{o}\left(\bar{y}_i - y_m\right) \qquad (6)$$

Where **y** is the total mean grey relational grade, **y** is the mean grey relational grade at optimum level, and *o* is the number of main design parameters that affect quality characteristics. **y** is the predicated GRG value that calculated by Eq. 6.

5. RESULT AND DISCUSSION

5.1 Calculation of Grey Relational Coefficient

Using Eq. 1 $x_i^*(k)$ of Size is to be calculated. Also, using Eq. 2 $x_i^*(k)$ of MP is to be calculated. Then from Eq. 3 $\Delta 0_i$ for all two responses is to be calculated. Finally, from Eq. 4 grey relational coefficient (GRC) of MP and Size are to be calculated.

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5.2 Calculation of Grey Relational Grade

Using Eq. 5 and GRC values of MP and Size, grey relational grade (GRG) is obtained. The GRG values for all eight experiments are show in Table 1.

Table 1: GRG calculation

Sr. No	GRC of Size	GRC of MP	GRG
1	1.0000	0.3377	0.6689
2	1.0000	0.3333	0.6667
3	0.5021	0.5868	0.5444
4	0.9835	0.3900	0.6867
5	0.9835	0.3620	0.6727
6	0.4296	0.5894	0.5095
7	0.9675	0.5069	**0.7372**
8	0.9675	0.4312	0.6993
9	0.3754	0.5949	0.4852
10	0.9835	0.3620	0.6727
11	0.9675	0.3620	0.6647
12	0.4630	0.5894	0.5262
13	0.5021	0.5150	0.5086
14	0.4630	0.4356	0.4493
15	0.4007	0.5922	0.4964
16	0.4630	0.6539	0.5585
17	0.4007	0.4581	0.4294
18	0.3531	0.5977	0.4754
19	0.5484	0.3768	0.4626
20	0.4630	0.3924	0.4277
21	0.4296	0.5908	0.5102
22	0.4630	0.7003	0.5817
23	0.4296	0.5256	0.4776
24	0.3754	0.5949	0.4852
25	0.4007	1.0000	0.7003
26	0.3754	0.5894	0.4824
27	0.3333	0.5991	0.4662

According to the experiment design, it is clearly observed from Table 1 that the process parameters setting of experiment no. 7 has the highest grey relational grade. Therefore, experiment no.7 is the optimal process parameters setting for maximum magnetic properties and minimum particle size simultaneously (i.e. the best multi-performance characteristics) among the twenty-seven experiments. The analysis showed that the optimal values for the parameters to achieve the desired crystallite size and magnetic properties were a heat treatment temperature of 1053 K, EDTA-to-metal ions molar ratio of 1.7, EG-to-metal ions molar ratio of 3, and pH value of 6.5.

The procedure followed is: I) group the grey relational grades by factor level for each column in the orthogonal array, II) take the average of coefficients and grade values for each experiment of the L_{27} full factorial design were calculated by applying the following equations.

γ_{A1}=1/4(0.699 + 0.690+ 0.663 + 0.536) = 0.647

γ_{A}=1/4(0.567 + 0.574 + 0.528 + 0.554) =0.556

The grey relational grade values for each level of the machining parameters were calculated using the same method. Figure 3 shows Variation of the grey relational grade (GRG) in the experiments for the multi-performance characteristics.

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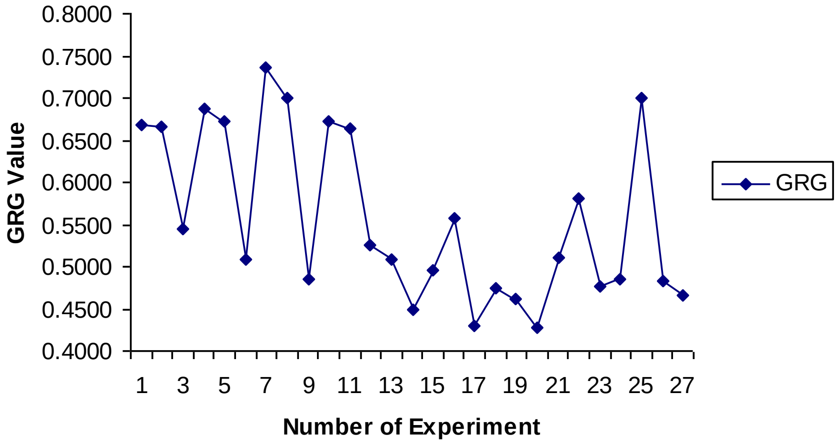

Figure 3. Variation of GRG in the experiments

6. CONCLUSIONS

The gray-based Taguchi method is used to determine the optimal input factor of the nanoparticle synthesis with the multi-performance characteristics has been given in this paper. A gray relational analysis of the MP and Particle Size gives the single performance characteristic called the gray relational grade. It shows that the performance characteristics of Microreactor synthesis such temperature and time is the most effecting parameters.

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