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DESIGN OF EXPERIMENT TO OPTIMIZE THE YIELD OF SILICON CARBIDE WHISKERS FROM BIO MASS MATERIAL

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Abstract— **Design of experiment is a powerful Quality Engineering tool that can help researchers and engineers to identify main variables which affects the performance. Silicon carbide particles and whiskers were produced by pyrolysis of stoichiometerically balanced rice husk. The aim of this paper is to increase the yield of silicon carbide whiskers by optimizing process conditions. Rice milling generates a by-product known as husk. This husk contains about 75 % organic volatile matter and the balance 25 % is inorganic matter. Inorganic matter is rich in silica and organic matter is rich in carbon. To balance stoichiometric proportion, rice husk was treated with different silica sources. Different process conditions that affect the quality of whisker formation were identified and experiments were conducted using Taguchi's design of experiment. Four factors at three different levels were selected and L9 array design of experiments was conducted. To determine the effect each variable on the output, the signal-to-noise ratio was calculated and factor effect diagram was drawn for each experiment conducted. The process parameters optimized using this design include silica source, pyrolysis temperature, silica to carbon ratio and heating rate. Silicon carbide produced from rice husk was characterized using scanning electron microscope to identify the microstructure. From Taguchi factor effect diagram it has been found that carbon to silica ratio and pyrolysis temperature plays a major role in whisker formation among the selected levels.**

Index terms- **Taguchi, Design of experiment, Silicon carbide, Whiskers.**

I. INTRODUCTION

The term experiment is defined as the systematic procedure carried out under controlled conditions in order to discover an unknown effect, to test or establish a hypothesis, or to illustrate a known effect. When analyzing a process, experiments are often used to evaluate which process inputs have a significant impact on the process output, and what the target level of those inputs should be used to achieve a desired result (output). Experiments can be designed in many different ways to collect this information. Design of Experiments (DOE) is also referred to as Designed Experiments or Experimental Design. The Taguchi method involves reducing the variation in a process through robust design of experiments. The overall objective of the method is to produce high quality product at low cost to the manufacturer [1].

The Taguchi method was developed by Dr. Genichi Taguchi of Japan. Poor quality in a process affects not only the manufacturer but also society [2]. He developed a method for designing experiments to investigate how different parameters affect the mean and variance of a process performance characteristic that defines how well the process is functioning. The experimental design proposed by Taguchi involves the usage of orthogonal arrays to organize the parameters affecting the process and the levels at which they should be varied; it allows for the collection of the necessary data to determine which factors most affect product quality with a minimum amount of experimentation, thus saving time and resources[3-6]. Analysis of variance on the collected data from the Taguchi design of experiments can be used to select new parameter values to optimize the performance characteristic.

Biomass materials like wood, energy crops, agricultural residues and food waste after pre-processing into suitable forms for various conversion technologies, provide feedstock for a variety of bioenergy end products and end uses. In this study rice husk is used to synthesize silicon carbide. Rice milling generates a by-product known as husk. This surrounds the rice grain. Chemical composition of rice husk is found to vary from sample to sample. The differences in the type of paddy, crop year, climatic and geographical conditions, sample preparation and method of analysis, is the reason for this variation. Pyrolysis of rice husk leads to the formation of silicon carbide particles and whiskers [7]. Whiskers are elongated single crystals, typically having cross-sectional diameters of 0.1–10 micrometers and lengths of 10–1000 micrometers. Because they are single crystals, which ideally contain very few dislocations and the same crystallographic orientation throughout, their strength and Young's modulus (stiffness) is expected to be very high; their values do, in fact, approach those predicted from bond strength calculations. Due to their hardness and strength, these whiskers are highly interesting as reinforcing materials in ceramics [8, 9].

The main aim of this study is to identify the influence of process parameters like pyrolysis temperature, carbon to silica ratio, silica source and heating rate and to optimize the process condition using design of experiment to maximize the yield of silicon carbide whiskers.

II. EXPERIMENTAL PROCEDURE

Rice husk samples were collected from local rice milling plant. The rice husk samples were sieved to remove residual rice and clay particles and washed in de-ionized water to remove any residue. They were then dried in an electric oven at 110°C for 24 h to avoid deterioration due to moisture content. Fifty grams of rice husk was taken in a graphite crucible of 12 mm wall thickness and 85 mm inner diameter, and closed with graphite lid. Rice husk quantity was chosen such that it occupies two third of the available space. The open space allows the gaseous reaction to take place and facilitates the vapor to escape. To avoid contamination, the inner surface of the container is covered with graphite foil before loading the samples. High temperature vacuum/inert atmosphere furnace (Therlek) was used for pyrolysis. A graphite crucible was used for carrying rice husk. The loaded graphite crucible was placed on the reaction chamber of the furnace and vacuum pump started. Initially vacuum was applied to remove oxygen. Once the required vacuum is created, the system automatically starts argon gas purging and temperature rise. High purity argon gas was used for purging and purged at a rate of 0.3 liters/min. Pyrolysis was carried out at 1200, 1300, 1400, 1500, 1600, 1700 and 1800°C with a heating rate of 10°C/min and dwelling time of 30 min. Cooling rate was 20°C/min till 300°C and after that natural cooling till room temperature.

In the present reaction system four significant operating parameters, each at three levels, are selected based on literature survey and experience. Significant factors that affect the silicon carbide yields are selected based on literature survey and experience. Here the objective is to maximize the yield of silicon carbide whiskers. The variables are heating rate, pyrolysis temperature, silica source and carbon/silica ratio (Table 1.1). Heating rate is varied from 5, 10 and 20°C/min and the selected pyrolysis temperatures are 1400, 1600 and 1800°C. Sodium silicate (SS), colloidal silica (CS) and quartz (QZ) are selected as different silica sources and the C/SiO_2 ratio is varied from 1.2, 0.9 and 0.7. Therefore an L9 orthogonal array is chosen and the experimental conditions are given in Table 1.2. Each set of experiment is repeated thrice and the average value is taken for analysis.

Table 1.1 Design of experiment – Factors and levels

Factors	Level 1	Level 2	Level 3
Temperature [°C]	1400	1600	1800
Silica source	colloidal silica	sodium silicate	quartz
Heating rate [°C/min]	5	10	20
C/SiO_2 ratio	1.2	0.9	0.7

To determine the effect each variable on the output, the signal-to-noise ratio, or the SN number, needs to be calculated for each experiment conducted. There are three types of signal-to-noise ratio based on the objective are smaller the better (LB), larger the better (HB) and nominal the better (NB). Here the objective is to maximize the yield of whiskers so larger the better formula is used.

Larger the Better:

$$\left(\frac{S}{N}\right) = -10\log(MSD)$$

Where,

$$MSD = \frac{1}{R}\sum_{j=1}^{R}\left(\frac{1}{{y_j}^2}\right)$$

S/N – Signal to Noise ratio
R = Number of repetitions
y_j = Target value
$MSD = Mean\ square\ deviation$

Table 1.2 Design of experiment L9 array

Design	L9 array				
	Experiment No.	Temperature (°C)	Heating rate - °C/min	Silica source	Carbon/silica ratio
A1B1C1D1	1	1400	5	CS	1.2
A1B2C2D2	2	1400	10	SS	0.9
A1B3C3D3	3	1400	20	QZ	0.7
A2B1C2D3	4	1600	5	SS	0.7
A2B2C3D1	5	1600	10	QZ	1.2
A2B3C1D2	6	1600	20	CS	0.9
A3B1C3D2	7	1800	5	QZ	0.9
A3B2C1D3	8	1800	10	CS	0.7
A3B3C2D1	9	1800	20	SS	1.2

III. RESULTS AND DISCUSSION

The experiments were conducted with the aim of relating the influence of silica source, pyrolysis temperature, carbon to silica ratio and heating rate on silicon carbide whiskers yield. On conducting the experiments as per the orthogonal array, results were obtained and factor effect diagram is drawn for each factor (Fig. 1). It has been observed that yield of silicon carbide whisker increases with increase in temperature from 1400 to 1600°C and drop is observed from 1600 to 1800°C. Conducting a reaction at higher temperature delivers more energy into the system and increases the reaction rate by causing more collisions between particles. At temperatures between 1400 and 1600°C, the rate of release of SiO and the rate of formation of SiC might be sufficiently low for the growth of SiC whiskers whereas at temperatures above 1400°C, the rate of release of SiO and the rate of SiC formation might be high enough for the growth of SiC. At temperature above 1600°C, the structure changed from whiskers to agglomerated powder. In summary, temperature upto 1600°C is favorable for SiC whiskers.

It is important that silica and carbon react completely and form SiC. A higher C/SiO_2 ratio was used against the stoichiometric ratio of 0.6. More silica was reduced to SiO, and a significantly higher conversion to SiC was achieved for slightly higher C/SiO_2 ratio than stoichiometric proportion. Carbon/silica ratio is critical for the reaction to form silicon carbide. By adding extra silica, the formation of SiC is increased by better utilization of carbon in rice husk [10]. Slightly higher percentage of carbon is needed against the stoichiometric ratio, since some of carbon is released as CO along with argon gas during pyrolysis.

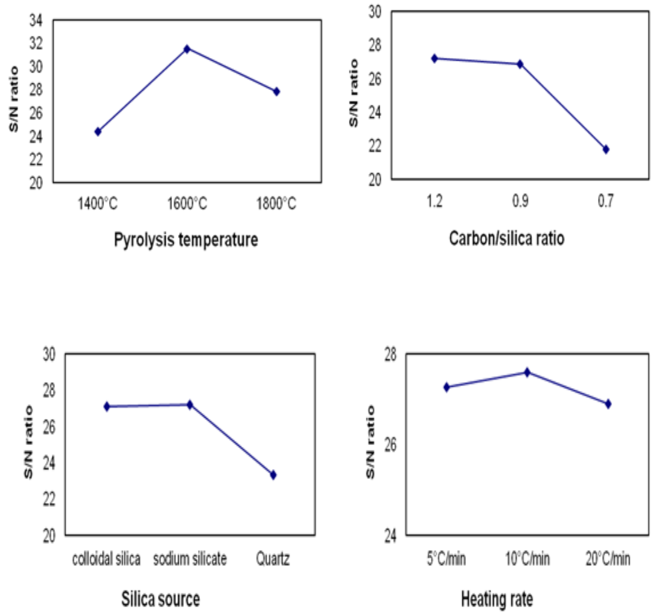

Figure 1. Factor effect plot a) Effect of pyrolysis temperature b) Carbon/silica ratio c) Silica source and d) Heating rate

It can be inferred that colloidal silica is found to be a good silica source for higher SiC whisker yield, when compared to sodium silica and quartz. The SEM microstructure of silicon carbide derived from raw rice husk and silicon carbide derived from colloidal silica treated rice husk is shown (Fig. 2). It is observed that the colloidal silica treated rice husk yielded more whisker formation than raw rice husk. Among the selected levels of heating rate, there is very weak interaction between the process parameters in affecting the yield. The responses at different levels of process parameters for a given level of parameter value are almost parallel. Among the selected heating rate, 10°C/min is found to be better.

Carbon to silica ratio and Pyrolysis temperature play significant role in silicon carbide whisker formation The SiC whisker yield is better at a pyrolysis temperature of 1600°C, with the heating rate of 10°C/min, using colloidal silica as silica source to maintain the carbon to silica ratio of 0.9.

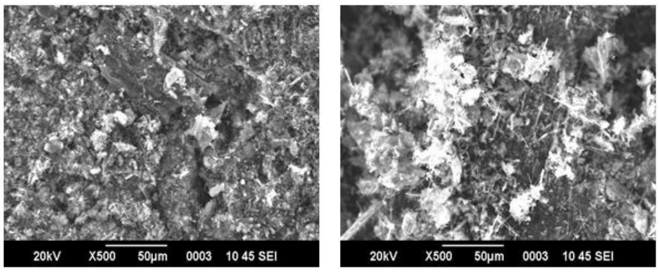

Figure 2. SEM micrographs of pyrolyzed SiC derived from (a) raw rice husk and (b) silica enriched rice husk.

IV. Conclusions

Process optimization is carried out using Taguchi's design of experiment to increase the yield of silicon carbide whiskers from rice husk. From the factor effect diagram, it is found that the silica source, carbon to silica ratio and pyrolysis temperature has the major impact among the selected factors in whisker yield improvement. Heating rate plays minor role among the selected factors. Formation of whisker is found to be high in case of colloidal silica treated rice husk. Optimum condition found to be 1600°C as pyrolysis temperature with the heating rate of 10°C/min with colloidal silica as silica source with the carbon to silica ratio of 1.2. By controlling the reaction conditions, it is possible to control the morphology of the SiC synthesized.

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