

Taguchi Optimization of Fracture Toughness of Silicon Carbide Extracted From Agricultural Wastes

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Abstract

In India, agricultural wastes such as palm ash and rice husk, which are abundant which have a high potential for usage as usable renewable energy and silica. Silicon carbide (SiC) is utilized for various applications due to its high hardness, compressive strength, and good wear resistance. In this work, a cleaner and green methodology was adopted to produce SiC from various agricultural wastes like peanut shells, rice husks, *sugar cane* extracts, and corn cob. Pyrolysis experiments were carried out by varying parameters such as heating temperature (600 to 800 °C), heating time (160 to 180 min), and quantity of waste (450 to 550 g) to convert agricultural wastes into powder SiC. X-ray diffraction, Raman, Fourier transform infrared spectroscopy and Scanning electron microscope confirms the formation of SiC phase in SiC. The sintering process parameters such as heating rate (5 to 15⁰C/min), cooling rate (5 to 15⁰C/min), and pressure (60 to 80 MPa) were selected for finding fracture toughness of sintered SiC. The process parameters for the pyrolysis and sintering process were optimized by the *Taguchi optimization technique*. Confirmations tests were conducted with optimum process parameters and the results indicated that confirmations results are correlated with predicted results.

1. Introduction

Chemical processing of feedstock from biomass would improve consumption and give economical value to farm waste. The various research on the processing of biomass into usable fuels and chemicals are carried out using different processes such as thermo-chemical processing, gasification, and pyrolysis. Water, bio char (or a carbonaceous solid), tars, and natural gases such as methane, hydrogen, carbon monoxide, and dioxide are available biomass pyrolysis materials [1–5]. Especially, biochar is used in various fields such as mechanical, electronics, aviation, and machinery industries [6–8]. Further, the heating of bio char produced a more effective product known as Silicon Carbide (SiC). Salema et al. [9] explored the pyrolysis process using corn stalk biomass briquettes through a 2.45 GHz frequency microwave (MW) reactor with 3kW power generation. This research opens a new dimension to the creation of pyrolysis technology for large scale. Liu has been analyzed microwave-assisted pyrolysis for the processing of bio based oil. The optimum bed temperature for pyrolysis and catalyst are 550 and 425°C respectively [10]. The microwave power levels had a significant influence on high temperatures and heating speeds. Microwave-assisted pyrolysis (MWAP) would need less input energy and processing time, so there is a greater thermo-chemical decomposition of the biomass feedstock [11]. Dai et al. [12] developed soap content bio-oil and biochar through microwave-assisted fast catalytic pyrolysis process. The addition of Bentonite increases the production of bio-oil. The chemical and physical properties of biochar extracted from MWAP Biosolids were studied at pyrolysis temperatures from 300 to 800°C [13]. The biochar developed from biosolids was carried out with preliminary phosphorus adsorption experiments. This work has shown that the temperature of pyrolysis influences a biochar-specific surface area, ash, and reactive material but has no significant effect on the resultant biochar's pH, chemical structure, and crystalline phases. Biochar rates are decreasing as the pyrolysis temperature rises. [14]. Hydrothermal Carbonization Method for making Silicon Carbide liquid fertilizer from Agricultural Waste

was developed by Chang et al. [15]. The low quality of the material extracted hampered its abrasive application. Sol-gel has been an excellent way of synthesizing nanopowder, with many excellent properties such as high-quality, stable chemical activity, and enhanced material sintering [16]. The correct method and pretreatment to enhance the degree of silica extraction from sugar cane bagasse were investigated by Embong et al. Pretreatment with an acid solution to sugarcane bagasse is useful which removes alkali metals [17]. On thermal processing, silica transforms into cristobalite (silica) which is crystalline. This micro silica may be a form of advanced materials such as SiC. [18]. The laser-induced periodic surface structures (LIPSS) of a single SiC crystal were analyzed by Binbin Meng et al. The results indicated that surface adjustment using a second Femto laser reduces the pressing and extrusion of the substrate and enables substrate removal. With the present manufacturing methods, various forms of material defects are generated in SiC substrates [19]. Hossain et al. [20] used the central composite design to evaluate possible experimental conditions for optimal yields of H₂ and biochar from OPF microwave pyrolysis. The input parameters (temperature, power of the microwave, and flow rate of N₂) demonstrated a complete overview of the laboratory method for both H₂ and biochar reactions to an application package. This approach decreases the number of tests considerably. Actual vs. expected graphs clearly illustrate that the actual H₂ and yield values for the biochar are well balanced with the projected values.

Our research group [21] already investigated the utilization of municipal solid wastes for making composites. The results indicated that it can be used for partition wall and ceiling panels. In this work, agricultural waste such as sugarcane extracts, peanut shells, rice husks, and corn cob are used for producing silicon carbide (SiC) using the pyrolysis and sintering process. Pyrolysis and sintering process parameters are optimized by the Taguchi optimization method.

2. Experimental Methodology

2.1 Material selection

Agricultural wastes such as sugarcane extracts, peanuts shells, corn cob, and rice husks (Fig. 1) are used for producing silicon carbide (SiC) by pyrolysis process due to their rich silica content. An industrial microwave oven is used to perform the pyrolysis process, for which argon gas is used to create an inert condition that makes the industrial microwave oven chamber free from oxygen. Table 1 provides the chemical content of the rice husk, sugar cane extracts, peanut shells, and corn cob used in this work. Rice husk contains about 75% organic volatile matter (Fig. 1a). Rice husk ash (RHA) contains silica about 85 to 90%. Sugarcane bagasse ash (SCBA) is having a silica content of 96.93% (Fig. 1b). Peanuts are widespread in the tropics and subtropics region and are essential for both small and large commercial producers. The inedible outer covering which is called Peanut shells (Fig. 1c) containing 56.4% of Silica content. Corn cob (Fig. 1d) is composed of cellulose and lignin and also having significant elements such as silicon, calcium, and aluminum. Relatively high silicon content was contained in corn cobs. Corn cob ash (CCA) contains a silica content of 96.6%.

2.2 Sample preparation

Silicon carbide (SiC) powder of particle size 1 μm was produced by pyrolysis process using agricultural wastes such as sugarcane extracts, peanut shells, corn cob, and rice husks which were combined to make powder mixture by grinding machine. This mixture was kept in an industrial microwave oven at a different temp of 160 $^{\circ}\text{C}$, 170 $^{\circ}\text{C}$, and 180 $^{\circ}\text{C}$ for a different time duration of 160 min, 170 min, and 180 min respectively. Powdered SiC obtained was kept in a vacuum heat furnace for the sintering process to convert into a solid form. A total of nine samples of each mass 15 g and size of 30 x 5x 3 mm are selected for fracture toughness testing.

2.3 Characterization Techniques

X-ray diffraction (XRD) tests were used to analyze the XRD spectra of powdered SiC by Rigaku 200 B X-ray diffractometer (40 kV and 100 mA). It was recorded from 5 to 90 $^{\circ}$ with a speed of 5 $^{\circ}$ /min. Raman spectroscopy was performed from 200-1300 cm^{-1} using Raman spectrometer Model- Enspectr R532 to analyze the raman spectra of SiC. Further, the Fourier transform-infra red (FTIR) spectroscopy JASCO 6300 instrument was used to analyze FTIR spectra of the SiC. powdered SiC were mixed with KBr in a ratio of (1:100) by mass for making compressed pellets. It was performed from 450-4000 cm^{-1} range with 4 cm^{-1} resolutions and a total scan of 16 for each sample. The morphological structure of powdered SiC was observed by scanning electron microscope (SEM) using MIRA3 LMH microscope operating at 10kV, 10 mm WD, and 30 Pa chamber pressure.

2.4 Indentations fracture (IF) Technique

In this research, the indentations fracture (IF) method is used for calculating the fracture toughness of sintered SiC. Fractured halves of the specimen used for this method are shown in Fig. 2 The diagonal length (2a) and crack length (2c) were recorded for each impression. This fracture toughness test was carried out by INNOVA TEST NEXUS 4303 micro Vickers hardness tester at a load of 290 N for 10s. Crack length (c) and indentation diagonal length (a) on each sample of sintered SiC was measured. The fracture toughness values were calculated by following equation 2 [19].

$$\text{Fracture toughness, } K_c = 0.0264 E^{0.5} P^{0.5} c^{-1.5} a \quad (2)$$

Where

K_c -fracture toughness ($\text{MPam}^{1/2}$)

E -Young's modulus of a sample (MPa)

P -load given (N)

c -half crack length (mm)

a -indentation diagonal length (mm)

2.5 Design of experiment (DOE)

In general, traditional experimental design techniques require a large no of experiments which increases the cost. To avoid this, Taguchi experimental design technique is used which has orthogonal arrays to analyze the process parameter with fewer experimental runs [22]. For the design of the experiment, MINITAB software is used. Taguchi technique is used to investigate the impact of the variables and their interactions on process performance [23]. Taguchi's method of the experiment is one of the most popular and useful statistical methods which is used to analyze the influence of more than one variable and its interactions on the process performance. For each parameter, three levels were chosen and an orthogonal array of L9 (3^3). For each element, the S/N ratios and average responses were plotted against each of its levels in the graphical method. The S/N ratio is the key quantity that has to be determined to achieve an optimal solution like the experiment [24]. Signal to noise ratio is used to determine the mean response for each experiment. The "larger the better" was selected to find the ratio S/N is given in equation 1.

$$\frac{S}{N} = -10 \log \frac{1}{n} \sum \frac{1}{y^2} \quad (1)$$

Where

y - experiential data

n - total number of experiments

Three process parameters such as heating temperature ($^{\circ}\text{C}$), time (min), and quantity of agro-waste (g) were taken to perform the pyrolysis process. The sintering process was conducted in a hot press furnace by varying parameters such as heating rate ($^{\circ}\text{C}/\text{min}$), cooling rate ($^{\circ}\text{C}/\text{min}$), and inside chamber pressure (MPa) (Table 2).

3. Results And Discussion

3.1 Characterization of synthesized SiC

X-ray diffraction of SiC shows main peaks at (111), (200), (220), (311) planes of the SiC phase. which shows the formation of SiC [25] (Fig. 3). In the Raman spectrum, only two peaks are observed at 776 and 930 cm^{-1} . It is evidence of the transverse mode (TO) and longitudinal mode (LO) of 3C-SiC [26] (Fig. 4). FTIR spectra are shown absorption peak at 820 cm^{-1} is attributed to SiC stretching mode and a wider peak at 3700 cm^{-1} indicated to adsorbed water (Fig. 5). The SEM image of SiC is shown in Fig. 6. It is composed of sphere-shaped particles.

3.2 Pyrolysis process analysis

In the Pyrolysis process, the three factors are heating temperature, heating time and quantity of wastes and their levels are (600 to 800 $^{\circ}\text{C}$), (160min to 180 min), and (450 to 550 g) respectively. Different experimental combinations are obtained by Taguchi orthogonal design. The experimental results obtained by performing the process with these parameters are given in Table 3. The linear model analysis of the S/N ratio for three input parameters heating temperature ($^{\circ}\text{C}$), time (min), and quantity of agro wastes (g) was analyzed using Mini Tab software [27]. The average effect response and S/N ratio are given in Table 4 and its main effects plot for means and S/N ratio are shown in Fig. 7. It is understood that the quantity of wastes has more impact on the production process of powder SiC whereas heating temperature has less impact on the process (Table 4). The higher value of the S/N ratio shows the optimum level [28]. The optimal parameter level for the pyrolysis process for conversion of agro-waste into powdered SiC is the heating temperature at level 1, heating time at level 3, and quantity of wastes at level 2. It is observed that as the heating temperature and quantity of wastes increases, the powdered SiC also increases (Fig. 7). The optimal parameters for powdered SiC were obtained at heating temperature (600 $^{\circ}\text{C}$), heating time (180 min), and the quantity of wastes (500g) as per S/N ratio results.

3.3 Sintering process analysis

The sintering process was carried out in a hot press furnace by varying three input parameters i.e heating rate, cooling rate, and pressure inside the chamber. The value of fracture toughness is calculated by using Equation 2 [29]. The value of fracture toughness in terms of stress concentration factor is given in Table 5 which is determined by Equation 2. Experimental results for fracture toughness of sintered SiC results are given in Table 6. The linear model analysis of the S/N ratio for three input parameters, heating rate, cooling rate, and pressure was analyzed in Mini Tab software. The average effect response and S/N response ratio for fracture toughness of sintered SiC are given in Table 6 and Fig. 8 shown its main effects plot for means and S/N ratio. It is understood that the cooling rate has more impact on the fracture toughness of sintered SiC whereas the heating rate has less impact on the process (Table 7) [30]. The optimal level of parameter for the sintering process is the heating rate (level 3), cooling rate (level 2), and pressure (level 2). It is observed that as the heating rate increases, the fracture toughness value decreases and then increases however when cooling rate and pressure increase it increases then decreases (Fig. 8). The optimal cutting parameters for fracture toughness of sintered SiC were obtained at a 15 $^{\circ}\text{C}/\text{min}$ heating rate, 10 $^{\circ}\text{C}/\text{min}$ cooling rates, and 60 MPa of pressure as per the S/N ratio results.

3.4 Validation test

The Optimal values of parameters for the conversion of agricultural wastes into powder SiC by the Pyrolysis process, conversion of powder SiC into solid form by a Sintering process, are shown in Table 8. The errors predicted for the production of powdered SiC and fracture toughness of sintered solid SiC are 0.26 and 8.4% respectively. The confirmation test results obtained using optimal process parameters for production of powdered SiC and fracture toughness of sintered SiC are 88.4322 g and 4.32 MPa m^{1/2}. It is concluded that the Taguchi optimization method effectively predicts the optimum process parameter levels.

4. Conclusion

The powdered SiC was prepared from various agro wastes such as peanut shells, rice husks, sugar cane extracts, and corn cob. The heating temperature at 600°C, heating time (180 min), and quantity of wastes 9500g were shown optimal range for the pyrolysis process. XRD and Raman's spectroscopy confirm the formation of the SiC phase in SiC. The cooling rate at heating rate 15⁰C/min, cooling rates (10⁰C/min), and pressure (60 MPa) were shown optimum values for fracture toughness of sintered SiC. Taguchi results indicated that the obtained optimal values can be used in making automotive water pump seals and bearing by the manufactures. This cleaner and eco-friendly approach was another potential method for use of agricultural wastes for synthesizing high-quality advanced materials. Also, SiC which is extracted from agricultural residues can reduce the manufacturing cost.

Declarations

The material and methodology used in this study / research was in accordance with the ethical standards of the Institute and University.

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Author Contribution Amit Kumar Thakur and Mohd Tariq designed and performed the experiment. Ajay Kumar Kaviti and J Ronald Aseer wrote the manuscript. Anita Gehlot analyzed data statistically. Rajesh Singh and Ajay Kumar Kaviti critical review of the manuscript.

Data Availability The relevant data will be provided as and when necessary.

Code Availability Not applicable.

Conflict of Interest Author claims no interest of conflict.

Consent to Participate Informed consent was obtained from all the participants involved in this study.

Consent for Publication The authors consented for submission and publication of this research work / article in Silicon Journal.

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Research involving Human Participants and/or Animals Not Applicable

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Tables

Due to technical limitations, table 1-8 is only available as a download in the Supplemental Files section.

Figures

Figure 1

Preparation of Silicon Carbide using a) rice husk b) Sugar cane extracts c) Peanut shell d) Corn cob

Figure 2

See image above for figure legend.

Figure 3

XRD patterns of synthesized SiC

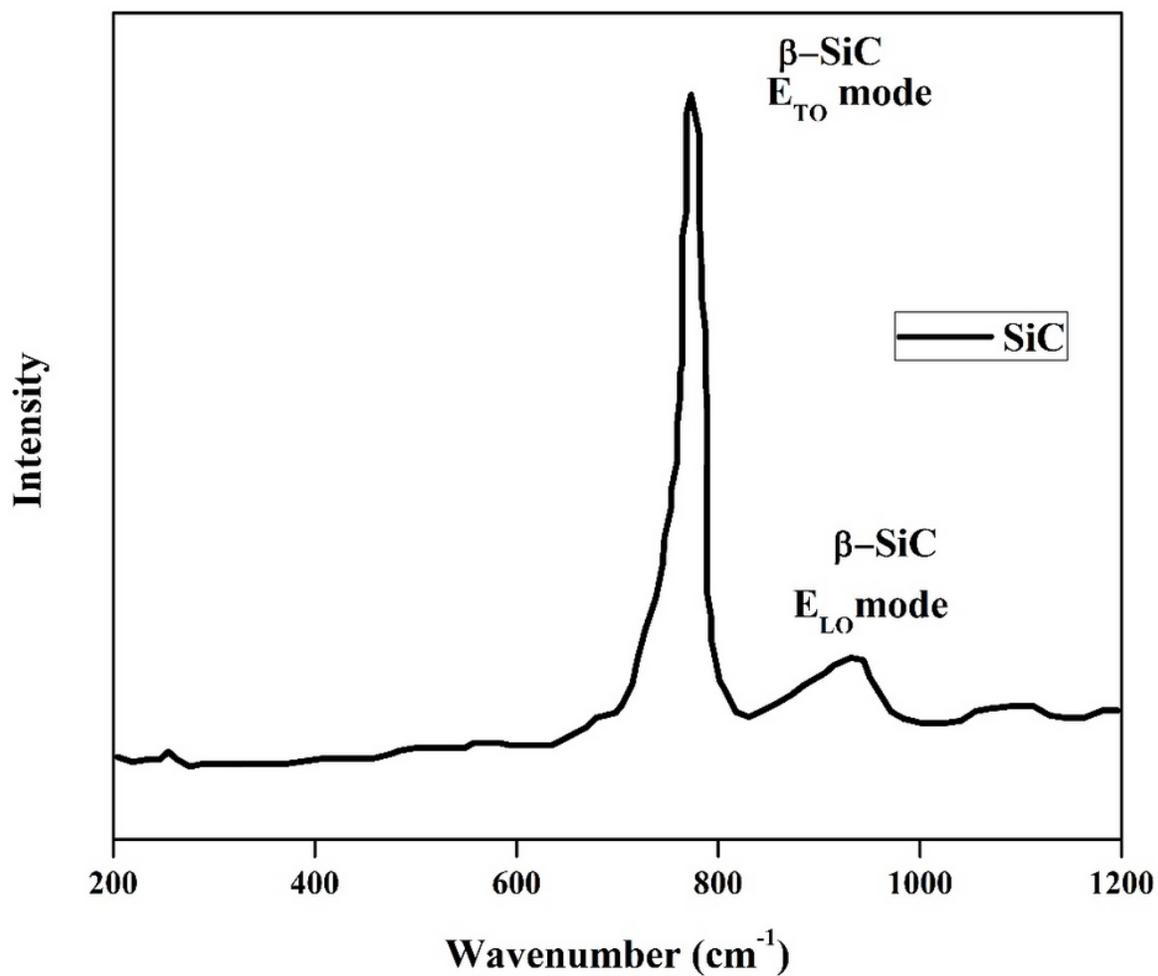


Figure 4

Raman spectra of synthesized SiC

Figure 5

FTIR spectra of synthesized SiC

Figure 6

SEM image synthesized SiC

Figure 7

See image above for figure legend.

Figure 8

See image above for figure legend.

Supplementary Files

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