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Optimising the Yield of Silicon Carbide Synthesised from Indigenous Biomass Husk using Different Catalysts

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Abstract

Biomass husk is a cost effective source to produce silicon based ceramics. Present work deals with the catalytic synthesis of silicon carbide from biomass husk of indigenous rice as raw materials. Rice husk samples were treated with different concentrations of sodium silicate, 40% hydrochloric acid and a mixture of 40% HCl and 2 gl^{-1} Na_2SiO_3. Treated husk were pyrolysed at 1350°C in argon atmosphere and then oxidised to eliminate unreacted carbon. Products were characterised by XRD, SEM and EDS techniques in order to confirm the carbide formation, to identify carbide morphology and to conduct elemental analysis respectively.Maximum yield was obtained in case of catalytic treatment with 2 gl^{-1} Na_2SiO_3. Acid treatment, although showed no considerable increase in yield, fostered whiskers formation instead of particles.

Keywords: Silicon carbide; Whiskers; Sodium silicate; Pre-treatment; Pyrolysis; Catalysis

Introduction

Silicon carbide offers a wide spectrum of electrical, chemical and mechanical properties, for which it is used in a variety of modern applications [1]. Silicon Carbide has high density, good thermal conductivity, extreme hardness, excellent corrosion and thermal shock resistance.These versatile properties enable it to beused in high temperature and structural applications. Due to high energy band gap and saturated drift velocity, it is also used for high temperature semiconductor material as compared to silicon based materials [2]. High purity silicon carbide can be synthesised from rice husk (RH) which generally contains 71 – 87 wt% organic components such as cellulose, lignin and sugar [3], and 13-29 wt% inorganic components. Oxidation of RH vaporizes the organic part and residual inorganic component is termed as rice husk ash (RHA) [4,5] more than 95% of which comprises ultrafine silica [6]. RHA is being used for some specific purposes such asceramic glaze, insulator, roofing shingles, waterproofing chemicals, oil spill absorbent specialty paints, flame retardants, carrier for pesticides and insecticides, fertilizer conditioner and zeolites synthesis [7]. The formation of SiC from RH pyrolysis is accomplished in a single or two stages. In single stage method, RH is pyrolysed at 1300-1500°C [8]. The amorphous silica in the RH can produce silicon carbide, silicon nitride, silicon oxynitride, pure silicon and other silicon based compounds depending upon the composition, atmosphere, reaction temperature and pre-pyrolysis treatment. However, the following chemical reactions occur during the carbothermal reduction of RH in the inert or reducing atmosphere at elevated temperatures [9]:

$$SiO_2\,(s) + 3\,C\,(s) \rightarrow SiC\,(s) + 2\,CO\,(g) \quad \text{(i)}$$

$$SiO_2\,(s) + 2\,C\,(s) \rightarrow Si\,(s) + 2\,CO\,(g) \quad \text{(ii)}$$

$$SiO_2\,(s) + C\,(s) \rightarrow SiO\,(g) + CO\,(g) \quad \text{(iii)}$$

And overall reaction is:

$$SiO_{2(amorphous)} + 3C_{(amorphous)} \rightarrow SiC_{(crystalline)} + 2CO_{(gas)}$$

Prior to pyrolysis, RH can be subjected to various catalytic treatments. Pyrolysis of HCl treated RH led to improved yield of SiC as compared that obtained from untreated raw RH [10]. RH can be treated with alkaline solution such as that of sodium hydroxide.Sodium silicatecan also be used as catalyst and has been proved beneficial in order to get enhanced yield of the products [11]. Using hydrated cobalt chloride and hydrated iron chloridealong with ammonium hydroxideas catalysts produce silicon carbide at comparatively low temperatures [12]. A study on pyrolysis of RHin the plasma arc reactor under argon atmosphere inferred that considerable product yield can be obtained in very short time [13]. Some other useful catalytic treatments include the use of iron chloride, cobalt chloride and nickel chloride [9].

Experimental Work

RH was pre-treated with three different reagents, viz. Na_2SiO_3, HCl and HCl + Na_2SiO_3. Firstly, samples were prepared by soaking RH for 24 h in solutions of different concentrations of Na_2SiO_3 in distilled water ranging from 1-6 g/l(designated as A1-A6). Secondly, RH was boiled in 5 N HCl solution for 1 h. Acid treated rice husk (B1) was thoroughly rinsed with distilled water. For the third category (C1), RH was boiled in 5 N HCl solution for 1 h, thoroughly washed with distilled water, dried at 90°C for sufficient time and then soaked in 2 g/l solution of Na_2SiO_3 in distilled. All the samples were ground to-mesh and stored in a drying oven. Finely ground and dried RHwere subjected to different processes including direct pyrolysis, oxidation of pyrolysed powder to remove residual carbon and finally HF treatment to remove SiO_2 and get pure SiC powder.

Pre-treated samples were pyrolysed one by one in SentroTech (STT-1650) tube furnace that was heated from ambient to 1350°C at a constant rate of 5°Cmin^{-1}, soaked at maximum temperature for 30 min

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and then allowed to cool at the same rate. Argon gas was purgedduring the whole course of heating and cooling at a rate of 0.1 lmin^{-1}. Oxidation of pyrolysed products was carried out at 750°C for 3 h in SentroTech (ST-1800) box furnace. The pyrolysed products after removal of unreacted carbon were identified by means of X-Ray diffraction analysis (using PANalytical Diffractometer) using Cu-K_α radiation. Following the XRD analysis, oxidised powder wastreated with 40% hydrofluoric acid in order to obtain pure SiC powder. Morphology of the desired product (SiC powder) was examined by means of scanning electron microscope (SEM SU-3500). Energy dispersive spectrometry (EDS) coupled with SEM provided elemental analysis of the products.

Results and Discussion

Two peaks corresponding to SiO_2 and SiC appeared in each diffractogram. The average value of SiC peaks was 2θ = 35.977, belong to (111) planes of β-SiC. All the peaks of SiO_2 appeared approximately at same angles with an average value 2θ = 21.771. During continuous heating, RH powder undergoes various changes such as volatilisation of organic matter, graphitisation of carbon, crystallisation of amorphous silica, formation of silicon carbide whiskers and conversation of SiC whiskers into particles. Presence of silica was in the form of cristobalite. The Figure1 shows a comparison of XRD patterns of the pyrolysed

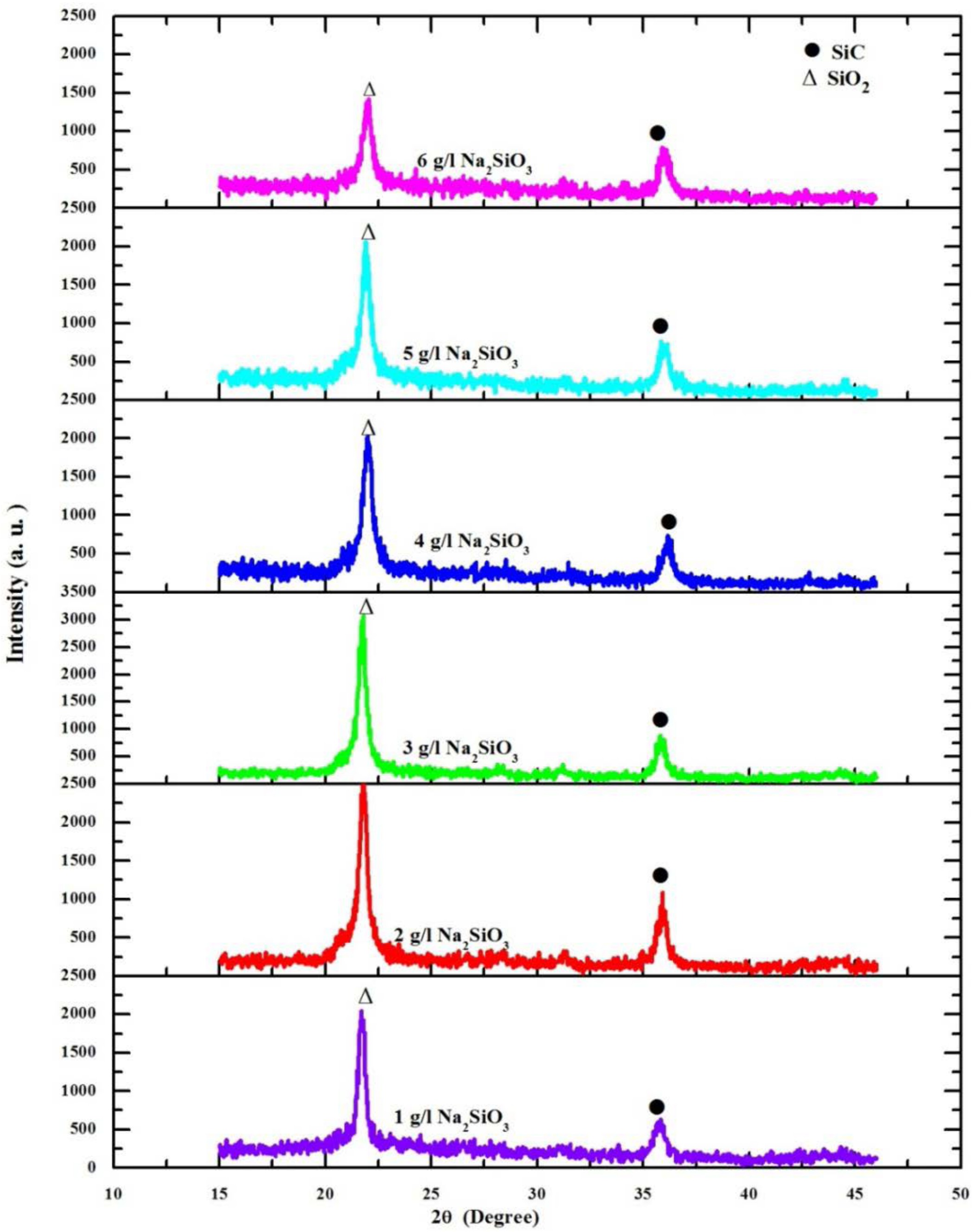

Figure 1: Comparison of XRD patterns of samples A1-A6.

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products from sodium silicate treated RH. The highest peak of SiC was observed from pyrolysis of sample A2.

Figure 2a and 2b shows micrographs of pure SiC powder obtained from samples A2 and B1. It is evident that silicate treated sample produced micron size carbide particles of irregular geometry whereas acid treatment fostered whiskers formation.

The EDS spectrum of A2 is shown in Figure 3 and corresponding elemental analysis given in Table 1 illustrate that the product contained Si, C, O and some other impurities present in the form of metallic oxides in the powder.

The effect of concentration of sodium silicate with respect to weight percent of SiC and SiO_2 is shown in Figure 4. As the concentration of Na_2SiO_3 increased from 2-6 g/l, the high concentration caused a thick coating of silicate on the surface of RHs which promotes the formation of SiO_2, because it actsas a barrier between the SiO gas in the media and the amorphous C in RH thereby delaying the pyrolysis reactions. Moreover,as the temperature of pyrolysis reactions increases from 1350 to 1550°C the formation of whiskers also decreases.Presence of impurities such as Mn, K, Ca and other complex silicates facilitate the nucleation of whiskers of SiC.

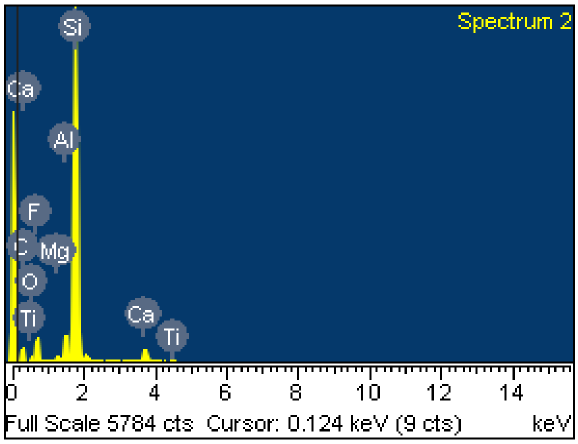

Figure 3: EDS Pattern of SiC powder.

Elements	C	O	F	Mg	Al	Si	Ca	Ti	Total
Weight (%)	31.56	6.78	11.58	0.42	2.40	43.75	2.98	0.53	100
Atomic (%)	48.56	7.84	11.26	0.32	1.65	28.79	1.37	0.20	

Table 1: Elemental Analysis of SiC powder.

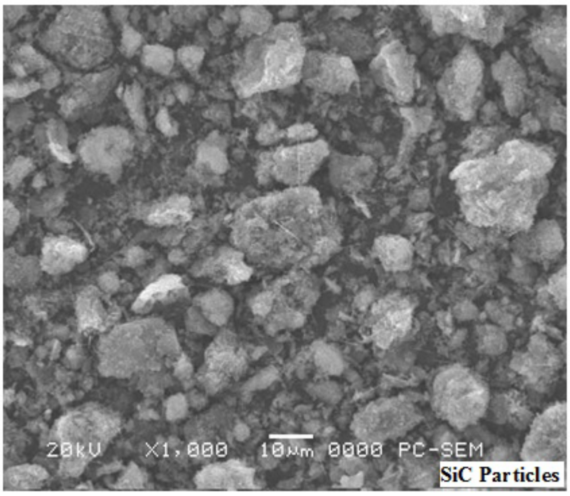

Figure 2a: SEM micrographs of SiC powder obtained from A2.

Figure 2b: SEM micrographs of SiC powder obtained from B1.

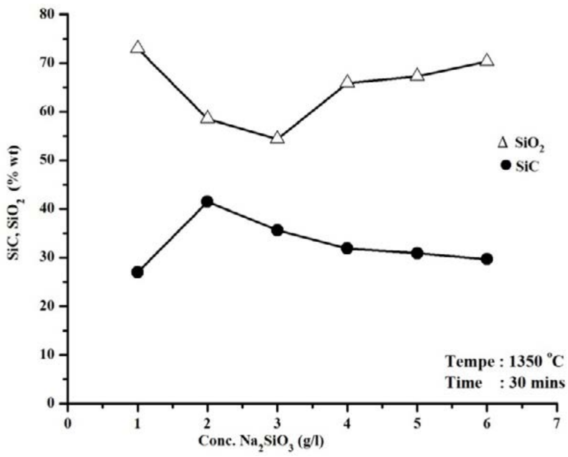

Figure 4: Effect of concentration of Na2SiO3 on the yield of SiC.

Calculated theoretical yield of SiC from RH is 58.3 wt% and practically the yield obtained was 51 wt% [11]. Maximum yield obtained in this study was 41.46 wt% in case of sample A2.

Conclusions

Pre-treatments with sodium silicate proved effective to improve the yield of SiC from rice husks. Pyrolysis of pre-treated rice husks with 2 g/l (Na_2SiO_3) resulted in maximum yield i.e. 41.46 wt%. Pre-treatment with hydrochloric acid had slight effect on increasing the yield of the product.

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