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Efficient production of nanoporous silicon carbide from rice husk at relatively lower temperature

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Synthesis of nanoporous SiC (np-SiC)

np-SiC was synthesized through the following four steps:

The first step: 100 grams of RH were weighed and transferred into 1L round-bottom flask, then added about 500mL 20 wt% hydrochloric acid and heated and refluxed for 12 hours. The reaction products were filtered with Buchner funnel and washed with deionized water. The filter cakes and filter liquors were collected for further processing. The collected filter cakes were dried at 120 °C for 6 hours in the blast air oven. The filter liquors were distilled to recover hydrochloric acid and further evaporated to dryness and calcined at 700 °C to recover useful potassium and phosphorus elements in rice husks. The prepared dried filter cakes in this step were named as the intermediate product 1 (IP1), and the calcined residues of the filter liquors were named as the final product 1 (FP1).

The second step: The prepared IP1 in the first step was calcined at 700 °C in argon atmosphere for 6 hours and the calcined products were named as the intermediate product 2 (IP2). In this step the IP1 is pyrolyzed and the volatile elements including oxygen and hydrogen are volatilized and the prepared black powder products mainly consist of carbon and silica.

The third step: The IP2 was mixed with magnesium powder in weight ratio of 3:1, and transferred into a sealed stainless steel autoclave, then heated to react for 12 hours at specified temperature (650 °C, 800 °C, 950 °C and 1000 °C). The reaction products were treated with boiling 20 wt% sulfuric acid and 40 wt% hydrofluoric acid plus 63 wt% nitric acid successively to removed magnesium oxide, silicon, silica and other by-products and purities. The treated products were filtered and the filter cakes were dried at 120 °C for 6 hours. The prepared products in this step were named as the intermediate product 3 (IP3). The IP3 mainly consists of un-reacted carbon and porous SiC.

The fourth step: The IP3 was calcined at 600 °C in air atmosphere for 6 hours and the carbon in the products was burned and removed and the reseda np-SiC powder products were prepared. In this paper the reseda np-SiC powder products can also be named as the final product 2 (FP2).

Characterization methods

The morphologies of the samples were observed using a scanning electron microscopy (SEM, JEOL-JSM-6700F). Transmission electron microscopy (TEM), high resolution electron microscope (HRTEM), energy dispersive spectroscopy (EDS) and selected area electron diffraction (SAED) were performed on a JEM-ARM200F with acceleration voltage of 200 kV. The X-ray diffraction (XRD) tests were carried out on a Philips X'Pert Pro Super X-ray diffractometer. The X-ray source was graphite monochromated Cu-Kα (wavelength λ = 1.5418 Å) and the scanning speed was 0.05 deg/s. The range of scanning angle was 2θ=10-80°, and the voltage and current were 40kV and 40mA, respectively. The thermogravimetric analysis tests were carried out on a simultaneous thermal analyzer (STA) 8000 (PerkinElmer) at a heating rate of 10 °C/min from 30.00 °C to 1000.00 °C in an air atmosphere and an inert atmosphere and the gas flow is 50.0 ml/min. The specific surface area (BET) and pore size distribution were characterized by nitrogen adsorption desorption isotherm. The instrument is an ASAP 2460 Surface Area and Porosity Analyzer (Micromeritics Instrument Corp.). The concentrations of elements of carbon (C), hydrogen (H) and oxygen (O) in the samples were determined using a VarioEL III analyzer (elementar). The concentrations of trace metal elements in the RHs and IP1 were determined using an inductively coupled plasma atomic emission spectrometer (ICP-AES) (Optima 5300DV, Perkinelmer) after the samples were treated with a microwave digestion system (ETHOS1, Milestone).

The concentrations of carbon (C) element were determined using an infrared carbon and sulfur determination analyzer (TS-HWC10, Nanjing Teruisi Analusis Instrument Manufacturing Co., Ltd.). The trace metal elements and SiC in the final product np-SiC were determined according to International Standard ISO 9286:1997 (E). Phosphorus and potassium in the FP1 were determined according to International Standards ISO 6598:1985 (E) and ISO 17319:2015 (E) respectively. The concentration of silica in the IP2 was determined by Gravimetric method and by referring to International Standard ISO 4158:1978 (E).

The samples obtained in different steps were photographed and shown in Figure S1.

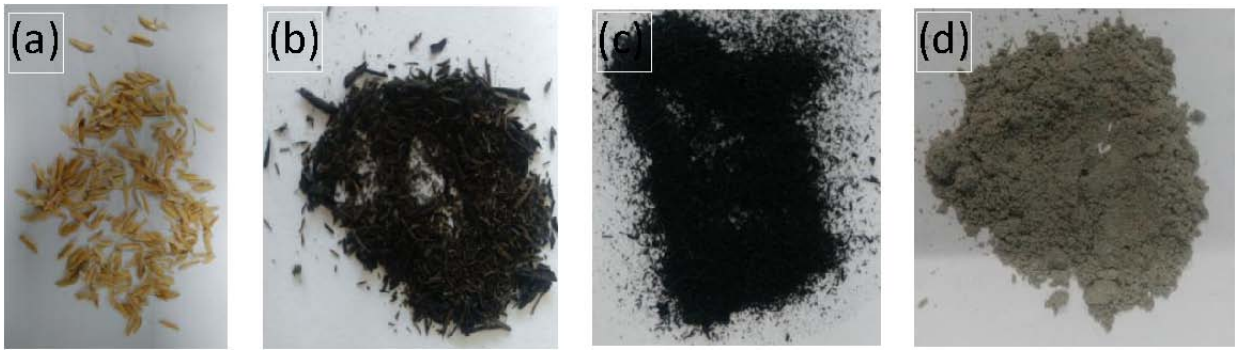

Figure S1 The appearance of the samples (*a*) RH, (*b*) IP1, (*c*) IP2 and (*d*) np-SiC-950.

Table S1 Chemical elements determined in RH and IP1.

Samples	Compositions (mg/g)																		
	C	O	H	SiO_2	P	Cl	S	N	K	Ca	Na	Mg	Al	Fe	Mn	Zn	Cr	Ni	Cu
RH	337.1	398.2	52.0	135.1	0.6	1.2	0.8	0.7	2.5	1.0	0.1	0.4	0.02	0.015	0.1	0.03	0.001	0.0007	0.0008
IP1	496.2	147.3	21.0	290.8	0.04	0.02	0.1	0.08	0.06	0.04	0.06	0.02	0.01	0.007	0.04	0.01	0.0007	0.0004	0.0003

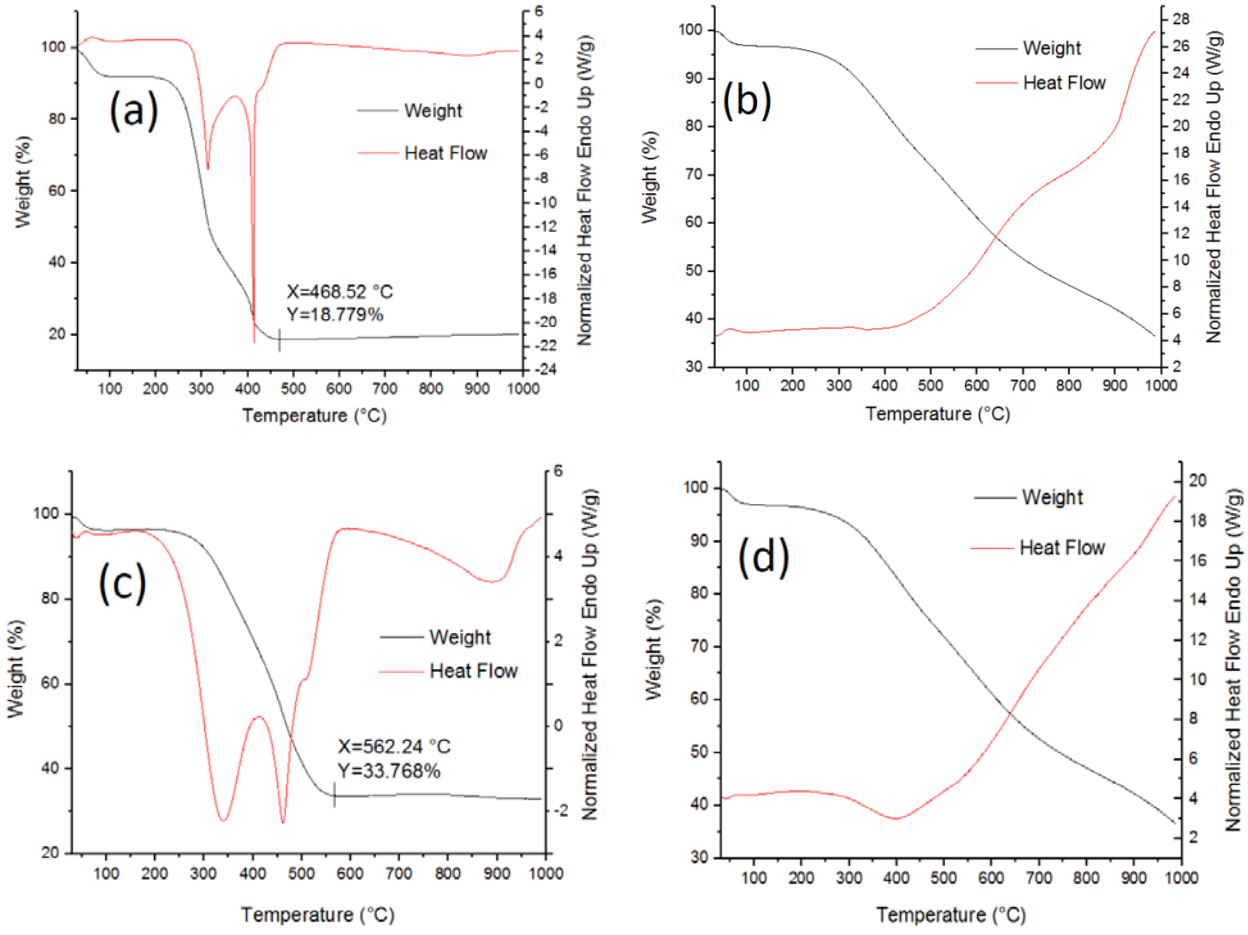

Figure S2 TGA-DSC curves of RHsand IP1 in nitrogen and air atmosphere: (a) RH, air atmosphere; (b) RH, N_2 atmosphere; (c) IP1, air atmosphere; (d) IP1, N_2 atmosphere.

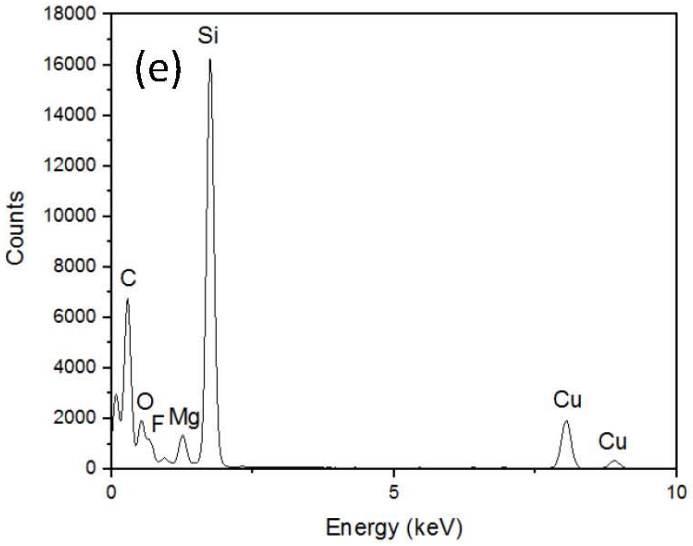

Figure S3 EDS analysis results for np-SiC-950.