JOURNAL OF MATERIALS SCIENCE 23 (1988) 21–24

Investigation of combustion of raw and acid-leached rice husk for production of pure amorphous white silica

A. CHAKRAVERTY, P. MISHRA, H. D. BANERJEE*
*Post Harvest Technology Centre and *Material Science Centre, Indian Institute of Technology, Kharagpur 721 302, India*

The effects of various acid treatments of rice husk on removal of its metallic ingredients and different combustion temperatures on production of amorphous silica (white ash) were studied. Leaching of husk in dilute HCl (1 N) was proved to be effective in substantially removing most of the metallic ingredients and producing ash completely white in colour. Irrespective of the treatments given, the minimum temperature required for complete combustion within a reasonable period was found to be 500° C. The combustion time varied from 5 h at 500° C to 1.5 h at 700° C. The ash residues obtained from complete combustion of acid-treated husk samples were completely white in colour. On the other hand, under similar conditions, the ash residues obtained from untreated husk remained light brown. The acid treatment of husk did not affect the amorphicity of the silica.

1. Introduction

Amorphous silica forms a primary source of pure silicon, which is widely used in manufacturing solar cells for photo-voltaic power generation [1]. The conventional process of extracting this silicon in pure form from the natural deposits of quartzite rock or quartz sand involves a high cost of production [2]. On the other hand, the carbon-free ash (white ash) obtained from rice husk is rich in high grade silica [3]. It contains metallic impurities at very low concentrations [4] which can be further purified by a simple acid-leaching process [5, 6]. Many investigators studied the structure of this ash-silica [7, 8]. Recent investigations show that reasonably pure polycrystalline silicon can be prepared from rice-husk white ash by a metallothermic reduction process [5]. However, except for some studies on the thermal degradation of rice husk [9, 10], very little systematic work has been done to determine suitable conditions for production of pure white ash. In view of this, the present work was undertaken with the following objectives: (1) to determine the effects of various acid treatments on removal of metallic impurities from rice husk, and (2) to determine the effects of different acid treatments and furnace temperatures on the time required for complete combustion and production of amorphous white ash from rice husk.

2. Experimental details

2.1. Material preparation

Rice husk obtained from an Indian high-yielding variety of paddy, *Mashuri*, was thoroughly wet cleaned, dried and then ground to 40 mesh. The ground husk was leached in different concentrations of HCl (1 N, 3 N, 5 N and 11.3 N), H_2SO_4 (4.5 N, 9 N and 18 N) and HNO_3 (4.5 N, 9 N and 18 N) at 50 ± 2° C for 2 h. After leaching, the husk was thoroughly washed with distilled water and then dried.

2.2. Analyses for metallic impurities in husk ash

Analyses for some common metallic impurities present in the ash obtained from untreated as well as acid-treated husk samples were performed using a Flame Photometer (Model, C150, AIMIL, India) for sodium, potassium and calcium and an Atomic Absorption Spectrophotometer (Varian Techtron, Model 1100, Australia) for iron, magnesium, manganese, zinc and copper. For the above chemical analyses, the ash samples were prepared by placing about 5 g husk in silica crucibles inside an electric muffle furnace and slowly raising its temperature to 500 ± 10° C and maintaining it for 8 h.

Ash solutions were prepared for chemical analyses in the following manner. Exactly 0.2 g ash was taken in a platinum crucible of 50 ml capacity. About 2 ml conc. H_2SO_4 and 5 ml HF were added to it in sequence and the crucible was heated slowly on a hot plate to dryness. This process was repeated three times to remove the whole silica content of the ash, leaving only the metallic constituents. The metallic residues thus left in the crucible were dissolved in 5 ml conc. HCl and the volume of the solution was made up to exactly 25 ml by repeatedly washing the crucible with distilled water. The solutions thus prepared were kept in tightly closed polyethylene bottles. Before analysis the instruments were calibrated with standard solutions of the respective elements.

0022–2461/88 $03.00 + .12

TABLE I Effect of acid leaching of husk on removal of metallic ingredients from rice husk

Treatment	Metallic ingredients as oxides (p.p.m.)								
	Na	K	Ca	Fe	Mg	Mn	Zn	Cu	Total
Untreated	5125	11 750	7042	946	2562	1192	179	31	28 827
1 N HCl treated	46	50	58	362	187	19	19	6	747
3 N HCl treated	33	33	46	262	146	7	19	6	552
5 N HCl treated	25	HD	25	146	125	3	19	6	349
11.3 N HCl treated	12	HD	8	129	125	3	17	6	300
1 N H_2SO_4 treated	87	25	2500	233	729	25	102	12	3 713
4.5 N H_2SO_4 treated	92	25	2417	225	646	25	98	6	3 534
9 N H_2SO_4 treated	75	25	2500	229	625	19	85	6	3 564
18 N H_2SO_4 treated	75	50	2667	217	625	24	83	6	3 747
36 N H_2SO_4 treated	117	100	3333	221	437	171	98	6	4 483
4.5 N HNO_3 treated	17	25	19	500	83	19	17	8	688
9 N HNO_3 treated	8	25	11	442	83	12	12	6	599
18 N HNO_3 treated	4	25	7	392	62	6	12	HD	508
1 N HCl + 4.5N HNO_3 (1:3)	4	25	3	375	52	12	19	6	496

2.3. Controlled combustion of husk using muffle furnace

About 5 g untreated as well as acid-treated husk samples were taken in silica crucibles and placed in an electric muffle furnace which was already set at the desired combustion temperatures. The different temperatures considered for combustion of untreated husk were 300, 350, 400, 450, 500, 600 and 700° C. The samples were thus kept in the furnace until complete combustion took place. The treated samples were kept in the furnace at 300, 400, 500, 600 and 700° C for 24 h or until complete combustion had occurred. Complete combustion was ascertained by visually observing the samples at frequent intervals.

2.4. X-ray analysis

The X-ray diffraction spectra of the various ash samples were taken at a chart speed of $2° \text{min}^{-1}$ with the help of an X-ray diffractometer (Model, CCON-1500, USSR) using Mo*Kα* radiation.

3. Results and discussion

The amount of metallic ingredients present in untreated and various acid-treated husk samples are given in Table I. The results indicated that in untreated husk the concentrations of sodium, potassium and calcium are greater compared to iron, magnesium, manganese, zinc and copper. Treatment of husk with either HCl, H_2SO_4 or HNO_3 at lower concentrations proved to be effective in removing the above metals to a substantially lower level. Further removal of metals from husk occurred in small amounts with increasing concentration of the acid. The amounts of the above metals in H_2SO_4-treated husk were more than those of the HCl and HNO_3-treated husk samples. This confirmed that the treatment of husk with H_2SO_4 is not suitable for removal of some metallic ingredients present in it. This was possibly because of the formation of metallic sulphates, some of which are not easily soluble in water. The amounts of metals present in HCl- and HNO_3-treated husk samples were found to be comparable for all metals except iron, for which HCl treatment was better than HNO_3 treatment.

Table II shows the effect of combustion temperature on total time required for production of white ash from rice husk. It was observed that the total time decreased from 48 h to 1.5 h with increase in combustion temperature from 300 to 700° C. Thus at a low combustion temperature, a prolonged heating was required for complete combustion of husk to white ash, This was because the burning of fixed carbon in the husk took place at a slow rate at low temperatures.

The combined effect of acid treatment and combustion temperature on the total time required for production of white ash is shown in Table III. It was observed that irrespective of the treatments given the husk samples did not turn to white even after 24 h combustion at 300 and 400° C. At 500° C and above, the ash residues obtained from treated samples only were completely white in colour. On the other hand, under similar conditions, light brown ash was obtained from untreated husk samples. This indicated that certain metallic ingredients present in husk (responsible for the light brown colour of its ash) are removed due to leaching in HCl. Irrespective of the treatment given, the time required for complete combustion and production of white ash remained the same at any particular combustion temperature. For a sample size of 5 g it varied from 5 h at 500° C to 1.5 h at 700° C. This indicated that acid treatment of husk did not have a significant effect on the time required

TABLE II Effect of combustion temperature on total time required for production of white ash from rice husk. Sample condition: ground (40 mesh) with no acid treatment. Sample size: 5 g (approx.)

Combustion temperature (° C)	Approx. time (h)	Colour of ash
300	24	Black
	36	Brown
	48	Light brown (whitish)
350	24	Brown
	36	Light brown (whitish)
400	24	Brown
	30	Light brown (whitish)
450	16	Light brown (whitish)
500	5	Light brown (whitish)
600	3.5	Light brown (whitish)
700	1.5	Light brown (whitish)

TABLE III Effect of acid leaching of rice husk and combustion temperature on time required for production of amorphous white ash. Sample condition = ground (40 mesh). Sample size = 5 g (approx.)

Treatment	Combustion temperature (°C)									
	300		400		500		600		700	
	Approx. time (h)	Colour of ash	Approx. time (h)	Colour of ash	Approx. time (h)	Colour of ash	Approx. time (h)	Colour of ash	Approx. time (h)	Colour of ash
Untreated	24	Black	24	Brown	5	Light brown (whitish)	3.5	Light brown (whitish)	1.5	Light brown (whitish)
1 N HCl treated	24	Black	24	Brown	5	White	3.5	White	1.5	White
3 N HCl treated	24	Black	24	Brown	5	White	3.5	White	1.5	White
5 N HCl treated	24	Black	24	Brown	5	White	3.5	White	1.5	White

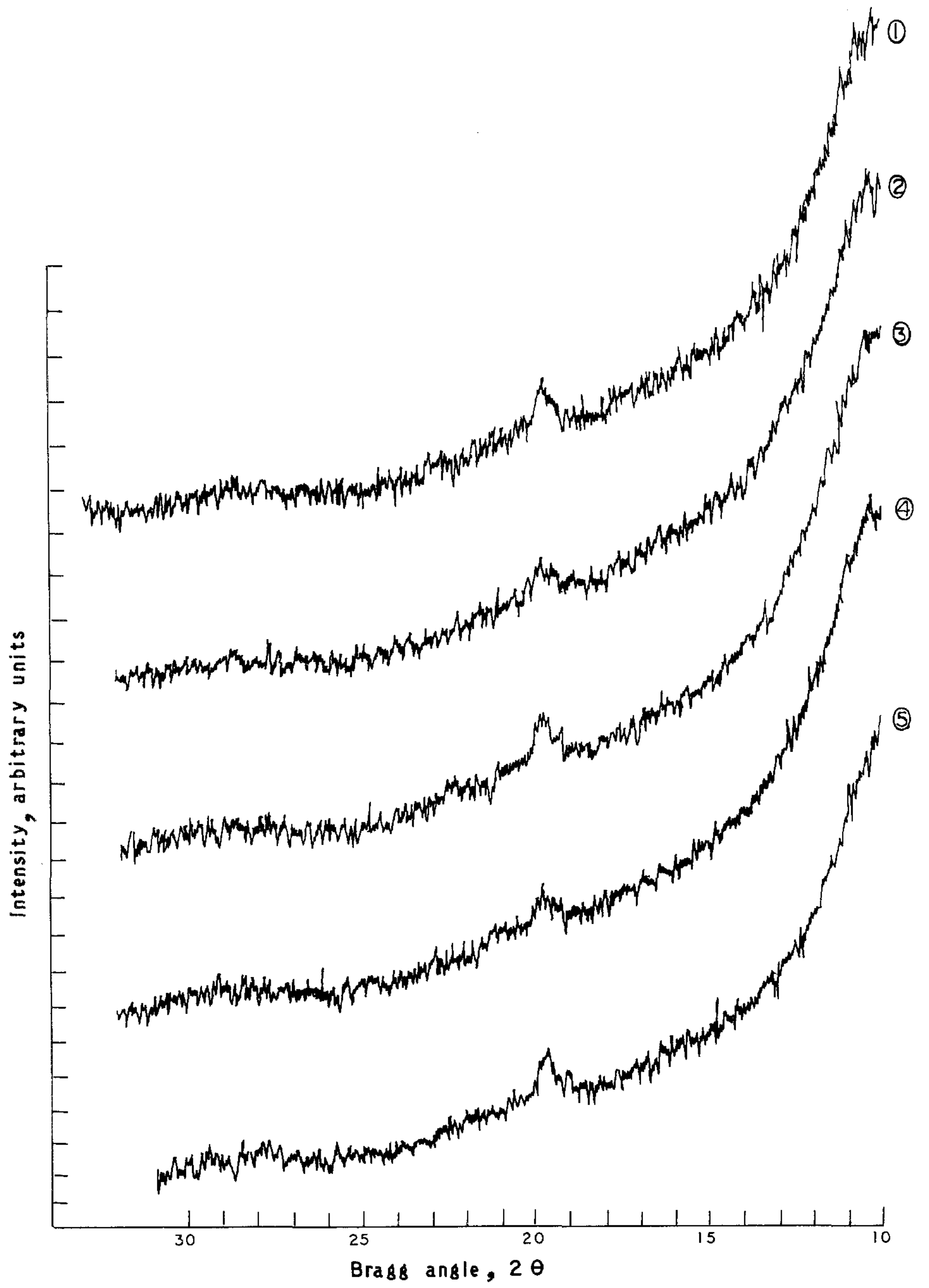

Figure 1 X-ray diffractogram of white ash obtained from HCl-leached husk samples, fired at 700° C for 1.5 h. White ash obtained from (1) 1N HCl-treated husk at 50° C, 2 h, (2) 3N HCl-treated husk at 50° C, 2 h, (3) 5N HCl-treated husk at 50° C, 2 h, (4) 11.3N HCl-treated husk at 50° C, 2 h, (5) untreated husk.

for complete combustion and production of white ash.

The X-ray diffractogram of the ash residues obtained from combustion of untreated and various HCl-treated husk samples of 700° C is shown in Fig. 1. The diffraction pattern in all cases indicated the amorphous nature of the silica in ash produced at a temperature of 700° C. It also confirms that acid treatment of husk did not affect the silica structure.

4. Conclusions

Within the limits of the present studies the following conclusions can be drawn.

1. HCl leaching of husk is superior to H_2SO_4 and HNO_3 for removing the metallic ingredients.
2. Acid leaching of husk in dilute HCl (1 N) helps in producing ash completely white in colour.
3. A combustion temperature of 500° C and above is required to produce white ash within a reasonable time.
4. The time required for complete combustion of husk and production of amorphous white ash (silica) varies from 5 h at 500° C to 1.5 h at 700° C.
5. Acid leaching of husk does not affect the structure of its ash-silica.

References

1. T. L. CHU, S. S. CHU and R. W. KALM, *J. Electro-Chem. Soc.* **125** (1978) 595.
2. J. DIELT, D. HELMREICH AND E. SIRTL, "Crystal: Growth, Properties and Applications 5, Silicon" (Springer-Verlag, Berlin, Heidelberg, 1981) p. 55.
3. D. F. HOUSTON, "Rice Chemistry and Technology" (American Association of Cereals Chemists, Inc., St Paul, Minnesota, 1972) p. 301.
4. R. SINGH, B. K. DHINDAW and R. C. MAHESWARI, *Agril. Mechanisation in Asia, Africa and Latin America* (1981) 57.
5. H. D. BANERJEE, S. SEN and H. N. ACHARYA, *Mater. Sci. Engng* **52** (1982) 173.
6. J. A. AMIC, *J. Electro-Chem. Soc.* **129** (1982) 864.
7. D. M. IBRAHIM and M. HELMY, *Thermochim. Acta* **45** (1981) 79.
8. M. A. HAMAD and I. A. KHATAB, *ibid.* **48** (1981) 343.
9. M. A. HAMAD, *J. Chem. Tech. Biotechnol.* **31** (1981) 624.
10. A. CHAKRAVERTY, P. MISHRA and H. D. BANERJEE, *Thermochim. Acta* **94** (1985) 267.

*Received 24 March
and accepted 8 June 1987*