

## Magnesium Silicate Synthesis from Rice Hull Ash

Sir:

Rice hulls are a co-product of the rice industry and are composed of about 20% silica. The hulls are combusted commercially to produce a rice hull ash (RHA) that contains more than 60% silica. This silica has been used as the raw material for the production of various silica-based products, such as silica gel (1), silica xerogel, silicate films, and sodium silicate (2). Rice hull silica could be used to produce magnesium silicate but this has not been reported in the literature or done commercially. Synthetic magnesium silicates, known as Florisil (3), Magnesol XL (4), or Mizukaife F1 (5), are common adsorbents used commercially for chemical analysis and chemical processing, such as regeneration of used frying oils.

A sample of RHA obtained from Turkey (Thrace) that contained 77% silica and had a surface area of 5.9 m<sup>2</sup>/g (6) was subjected to alkali silica extraction using the method of Kalapathy and Proctor (7). Six hundred milliliters of 1 M sodium hydroxide was added to 100 g of RHA in a 1-L flask, covered with a watch-glass, and boiled for 1 h with constant stirring using a magnetic stirring bar and a hot plate equipped with magnetic stirring capacity. The resulting sodium silicate solution was filtered through Whatman #41 ashless filter paper to separate the carbon residue, which was subsequently washed with 50 mL of boiling water. The filtrate and washing were combined and allowed to cool to room temperature; the silica content was determined as described by Kalapathy and Proctor (7) and found to be 68.2 g/L.

Magnesium silicate production was achieved by mixing 250 mL of the sodium silicate solution with a magnesium sulfate solution prepared by dissolving 18.7 g of MgSO<sub>4</sub>·7H<sub>2</sub>O in 150 mL of water at room temperature. A white precipitate immediately formed, which was separated by vacuum filtering, washed with distilled water, and then washed with acetone. The precipitate was dried at 110°C for 3 h. A portion of the precipitate was activated in a muffle furnace at 600°C for 7 h. A separate precipitate sample and Florisil (Merck, Darmstadt, Germany; 100–200 mesh) were heated at 1100°C for 2 h to compare heat-induced structural changes.

X-ray diffraction (XRD) patterns of the activated product and Florisil were obtained by a dual goniometer X'pert XRD system (Philips Electronic Instruments, The Hague, The Netherlands) using an acceleration voltage of 40 kV and a current of 45 mA. Figure 1 shows the XRD patterns of the precipitate and Florisil, respectively, heated to 600°C. Both panels indicate an amorphous silica structure, as previously reported (8). The XRD patterns of the precipitate and Florisil heated to 1100°C, shown in Figure 2, are almost identical.

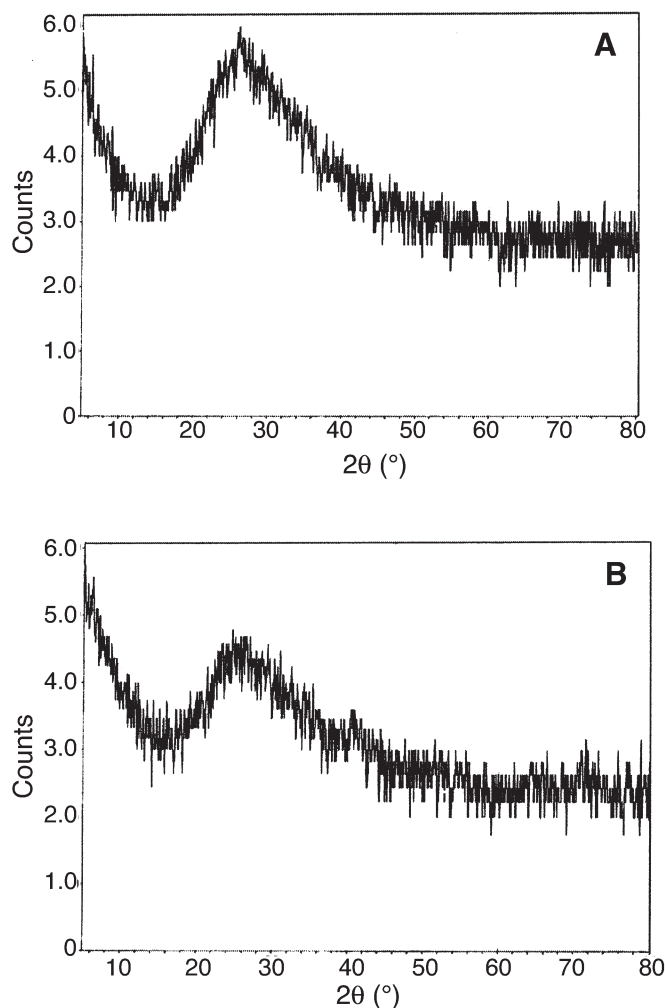


FIG. 1. X-ray diffraction patterns of (A) magnesium silicate and of (B) Florisil activated at 600°C.

Features of the XRD patterns confirm that both materials have the structure of cristobalite SiO<sub>2</sub> [Joint Committee for Powder Diffraction Standards (JCPDS) card no. 27-605] and MgSiO<sub>3</sub> (JCPDS, card no. 19-768).

The chemical compositions of magnesium silicates were determined by the soda ash fusion method (9) and atomic absorption spectrophotometry (PerkinElmer 110 B) and are shown in Table 1. Precipitated magnesium silicates had almost the same magnesium oxide content as Florisil, but the lower silica content indicated a less pure sample.

The surface areas of Florisil and of activated and nonactivated precipitate were determined by the BET method (model Autosorb 1; Quantachrome Instruments, Boynton Beach, FL). The adsorption capacities of the activated and nonactivated precipitates were compared with that of Florisil by adding 0.5

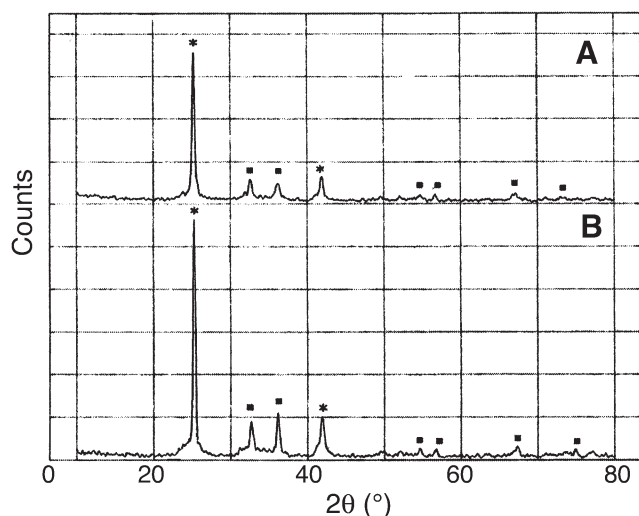


FIG. 2. X-ray diffraction patterns of Florisil (A) and of magnesium silicate (B) heated to 1100°C. (\*) Cristobalite  $\text{SiO}_2$ , (■) cristobalite  $\text{MgSiO}_3$ .

TABLE 1  
Chemical Compositions of Magnesium Silicates Produced from Rice Hull Ash and Florisil

	MgO (%)	$\text{SiO}_2$ (%)
Nonactivated	12.9	62.8
Activated at 600°C	13.8	73.7
Florisil	15.5	84.0

g of each sample to 50 mL of 0.02 N oleic acid in hexane and mixing in a shaker bath for 30 min. A control experiment was also conducted using oleic acid solution without adsorbent. The residual oleic acid in solution was determined by alkali titration. Table 2 shows the surface areas and adsorption capacities of Florisil and of the activated and nonactivated precipitates. The activated precipitate had a smaller surface area and adsorption capacity than Florisil but the nonactivated precipitate had a larger surface area and adsorption capacity. FTIR studies showed that magnesium silicate physically adsorbed oleic acid through hydrogen bonding of the carbonyl group to surface silanol groups. Oleic acid also is chemically adsorbed to magnesium silicate through formation of a carboxylate ion, which forms an ionic bond with metal oxide on the surface (10). Thus, the differences in the chemical compositions of the precipitates and Florisil may cause the differences in oleic acid adsorption capacities. On the other hand, both the adsorption capacities and the surface areas of the activated and nonactivated precipitates were very different, meaning that activation at 600°C caused a great change in the magnesium silicate structure, which has not yet been explained.

TABLE 2  
Adsorption Capacities and Surface Areas of Magnesium Silicates and Florisil

	Adsorbed oleic acid (mg OA/g adsorbent)	Surface area ( $\text{m}^2/\text{g}$ )
Florisil	87	181
Activated magnesium silicate	28	123
Nonactivated magnesium silicate	100	245

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