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Production of High Purity Amorphous Silica from Rice Husk

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Abstract

Combustion of the rice husk produces rice husk ash, which consists of mainly silica. High purity silica can be produced by controlled combustion after acid treatment. In this study, leaching of rice husk with hydrochloric acid and sulfuric acid were carried out prior to combustion to obtain purer silica. It was found that pre-treatment of the rice husk with sulfuric acid had accelerated the hydrolysis and decomposition of organic components as revealed by thermogravimetry (TG) and Scanning Electron Microscopy (SEM) analyses. In a systematic study, the combustion of un-leached, hydrochloric acid-leached and sulfuric acid-leached rice husks were performed in a muffle furnace at 500, 600, 700, 800 and 900°C for 2 h. Results demonstrated that all the samples produced amorphous silica (SiO₂) and the average particle size were in the range of 0.50 to 0.70µm. The effect of combustion at different temperatures between 500°C and 900°C on the silica production is very small, particularly at temperature above 600°C. Thus, amorphous silica with purity above 99% as confirmed by X-Ray Fluorescence (XRF) analysis can be produced by hydrochloric and sulfuric acids leaching of the rice husk, followed by controlled combustion at 600°C for 2 h. The BET surface area of the silica produced after leaching the rice husk with hydrochloric acid was higher (218 m²/g) than with sulfuric acid (209 m²/g). The silica obtained has potential application as filler in plastics and rubber compounding.

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1. Introduction

Rice husk is the outer covering of the rice grain, which is a by-product of the rice milling process. It is an agricultural waste material in all rice-producing countries. Most of the rice husk usually ends up either being dumped or burned in open spaces, thus causing damage to the land and environmental pollution. Much efforts have been made to utilize the rice husk including as an alternative fuel for energy production^{1, 2}, production of activated carbon³ and as a raw material for manufacture of industrial chemicals based on silica and silicon compounds⁴.

The major components of rice husk are organic materials such as hemicellulose, cellulose and lignin totaling about 75 - 90% and the remaining ash content of $17 - 20\%^5$. The ash mainly consists of >90% silica and some metallic impurities. Combustion of rice husk under controlled conditions leads to the productions of rice husk ash containing almost pure silica. The metallic impurities such as iron (Fe), manganese (Mn), calcium (Ca), sodium (Na), potassium (K) and magnesium (Mg) that influence the purity and color of the silica could be eliminated by pre-treatments with hydrochloric acid, sulfuric acid or nitric acid prior to combustion⁶.

It has been reported that at 600 to 1000°C and depending on the time of combustion⁷, amorphous silica is formed, but at higher temperature, crystalline silica is obtained. In this systematic study, the combustion of un-leached, hydrochloric acid-leached and sulfuric acid-leached rice husks were performed in a muffle furnace at 500, 600, 700, 800 and 900°C for 2 h. The aim of the present study is to investigate the optimum conditions for obtaining high purity silica. The properties studied include functional groups determination, structure properties, SiO₂ content, Brunauer-Emmett-Teller (BET) surface area and particle size.

2. Materials and Methods

2.1. Production of silica from rice husk

Rice husk was obtained from BERNAS rice mill, Tg.Karang, Selangor, Malaysia. The rice husk was washed with sodium dodecyl sulfate solution at constant stirring for 10 min to remove dirt and water soluble impurities. Then, the rice husk was further rinsed with distilled water to remove surfactant. It was first dried at room temperature and later dried in an air-oven at 110°C for 24 h. The washed rice husk obtained is designated as un-leached rice husk. Then, the washed rice husk was separately treated with hot acid at ~60°C with hydrochloric acid or sulfuric acid at concentration of 0.5 M for 30 min with constant stirring. After the acidic solution was drained off, the rice husk was rinsed with distilled water until free from acids, filtered and air-dried. The acid-leached rice husk was then dried in an air-oven at 110°C for 24 h. The un-leached rice husk and acid-leached rice husk was placed in a muffle furnace and heated at 500, 600, 700, 800 and 900°C for 2 h to obtain un-leached rice husk ash and acid-leached rice husk ash, respectively.

2.2. Characterizations of rice husk

The thermogravimetric (TG) analysis was performed on a Perkin Elmer TGA6 instrument. Sample of 5 - 7 mg was heated at heating rates 20°C/min from 50°C to 900°C under nitrogen atmosphere with flow rate of 20 mL/min.

The Scanning Electron Microscopy (SEM) analyses of the un-leached and acid-leached rice husk were conducted on a FESEM JSM 6701F (JOEL). The sample was placed onto the specimen stub and coated with platinum evaporative coating under high vacuum. It was operated at 15kV with 15mm working distance.

2.3. Characterizations of silica

The functional groups in the sample were determined using a Thermo Scientific FTIR Nicolet 6700 equipped with attenuated total reflectance (ATR) accessory. The spectra were recorded with 32 scans at a resolution of 4 cm⁻¹ in the range of 4000-400 cm⁻¹.

The X-ray diffraction (XRD) patterns were obtained using Bruker D8 Discovery X-ray Diffractometer using CuKα operated at 40 kV and 40 mA and 2Theta between 5° to 50°. The EVATM Software was used to record and analyze the structural pattern of sample.

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