

Characterization of Silica from Sodium Hydroxide Treated Rice Husk

Syed H. Javed, Tajwar S., Shafaq M., M. Zafar , M. Kazmi

Abstract

Silica derived from rice husk under controlled combustion produces reactive pozzolana also known as amorphous silica. Rice husk contains approximately 20 percent silica which is extractable under control burning conditions. Reducing the impurities by using organic acids from rice husk before thermal treatment does not improve the amorphicity of the silica. Treating the rice husk with sodium hydroxide solution also reduce the impurities present on the surface. In present paper the effect of various dilute solutions of sodium hydroxide on amorphous silica and on bond strengths of O-Si-O have been studied. The rice husk was treated with 0.05N and 0.1N solutions of NaOH for three hours at 600C and then dried in oven at 1050C for 24 hours. The dried rice husks were thermally degraded in tube furnace for three hours at 600 0C and 700 0C and then analyzed by FTIR. It has been observed that NaOH reduces the O-Si-O bond strength at 600 0C and improves the O-Si-O bond strength at 700 0C.

Key Words: Rice Husk, Sodium Hydroxide, Amorphicity, Bond Strength, FTIR, XRD.

Introduction

Rice Husk (RH) contains unusual amount of silica compared to all other biomasses. Approximately 20 % silica has been found in hydrated form in rice husk which can be recovered as amorphous silica under controlled burning conditions [1]. The treatment of rice husk as a source of energy is departure from the perception that husks present disposal problems. Generating energy from rice husk has great potential particularly in rice producing areas. Rice husk upon burning produces rice husk ash (RHA). The silica in the ash undergoes structural transformations depending on the conditions of combustion. For RHA as a marketable product, it should have more amorphous silica than crystal silica or should have high quality crystal silica that can be used as a filler in polymeric and ceramic composites. Two forms of silica dominate

in combustion process, which are lechatelerite, an amorphous form, and cristobalite, a crystalline form. Amorphous silica of high purity, small particle size and high surface area has tremendous potential as an adsorbent and catalyst support in different chemical synthesis. In order to prepare amorphous silica of high purity, the treatment of rice husk with various chemicals before and after combustion was attempted by many authors [2, 3]. At 400 °C to 700 °C mostly amorphous ash is formed where as above 700 °C mostly crystalline silica is formed [4]. These types of silica have different properties and it is important to produce ash of the correct specification for particular end use. There is a wide variety in the physical and chemical properties of RHA. The chemical and physical properties of the ash may be influenced by the soil chemistry, paddy variety, climatic conditions and fertilizer used. Rate of burning the

rice husk also influences the quality of RHA. Fast rate of burning produces RHA containing unburnt rice husk and carbon residues where as slow burning improves the quality of RHA upto 600 °C. The unburnt component is predominantly carbon which can be measured by reheating a sample of the ash in the furnace. The difference in the mass of the sample before and after heating is referred as the "loss on ignition" (LOI). The LOI value is normally the same as that of the carbon content of the ash. The percentage of the carbon in RHA varies according to the combustion process.

For RHA as a marketable product, it should have more amorphous silica than crystal silica or should have high quality crystal silica that can be used as a filler in polymeric and ceramic composites. Amorphous silica of high purity, small particle size and high surface area has tremendous potential as an adsorbent and catalyst support in different chemical synthesis. In order to prepare amorphous silica of high purity, the treatment of rice husk with various chemicals before and after combustion was attempted by many authors [5, 6].

Mineral acid leaching of rice husk has been reported to be more effective than untreated rice husk for extraction of silica. Acids like HCl, HNO₃, and H₂SO₄ of varying concentrations have been used to remove metallic ingredients and found that the HCl is the most effective. Researchers also tried organic acids on RHA but it has been found that pretreatment of RH is better than post treatment [7]. The purpose of pretreatment is to reduce carbon contents in RHA. The pretreatment of rice husk with any organic acid does not improve the amorphicity of the RHA produced. The bond strength of SiO₂ is another important factor influencing the quality of the amorphous silica. Higher bond strength indicates tendency toward formation of crystalline silica [8].

The bond strengths can be identified by using the FTIR machine. Infrared spectrum is a characteristic of chemical bonding in the sample; different chemical compounds will have their own characteristic absorption spectrum. Infrared Spectroscopy is a useful technique in the

identification and comparison of various materials. Each material has a unique infrared spectrum which can be used to identify and compare unknown materials.

Treating the rice husk with dilute solutions of NaOH removes the impurities and also purify the cellulose present in the rice husk.

In present study the rice husk was treated with dilute solutions of NaOH at 60 °C for three hours and then thermally degraded at 600 °C in tube furnace for producing amorphous silica. The objective of the study is to study the effect of pretreatment on SiO₂ bond strength and on amorphicity of the silica.

Experimental Procedure

Rice husk was collected from local rice mill during milling season. The rice husk was first washed with deionized water for five minutes followed by drying in oven at 105 °C for 24 hours. Two samples each of 100 grams were selected and treated with 0.05N and 0.1N solutions of NaOH at 60°C each for three hours. The two samples were then dried in oven for 24 hours at 105 °C and designated as NH-1 and NH-2. The untreated sample was designated as S. The two samples were then placed in porcelain crucible and calcined in electric tube furnace at 600 °C and 700 °C respectively each for two hours. The tube furnace was programmed at the rate of 10 °C /min up to the required temperatures and cooled down to room temperature.

The samples obtained from electric furnace were ground and passed from 200 mesh sieve and stored in air tight plastic container for further experiments.

FTIR

FTIR spectra were taken using KBr pellet technique. Approximately 1 mg of NH-1, NH-2 and S each were mixed with 100 mg of KBr and ground, pressed, dried and pellets were prepared. The FTIR spectra were scanned in the range of 400 cm⁻¹ – 4000 cm⁻¹. FTIR spectra were taken on FT/IR spectroscopy (JASCO model 4100).

The results obtained are shown in Figs. 1 and 2.

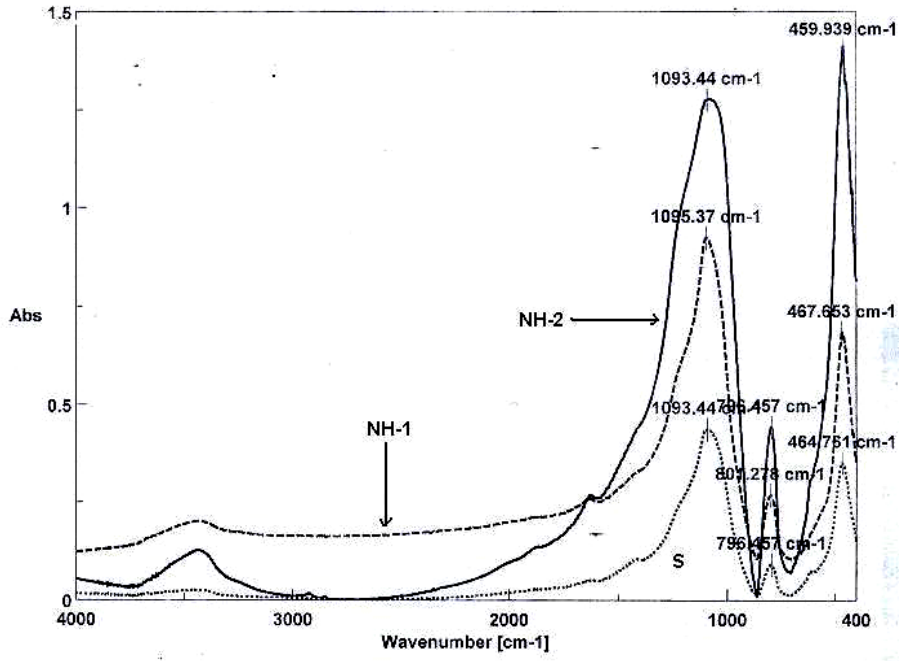


Fig.1: FTIR spectra of NH-1, NH-2 and S at 600 °C.

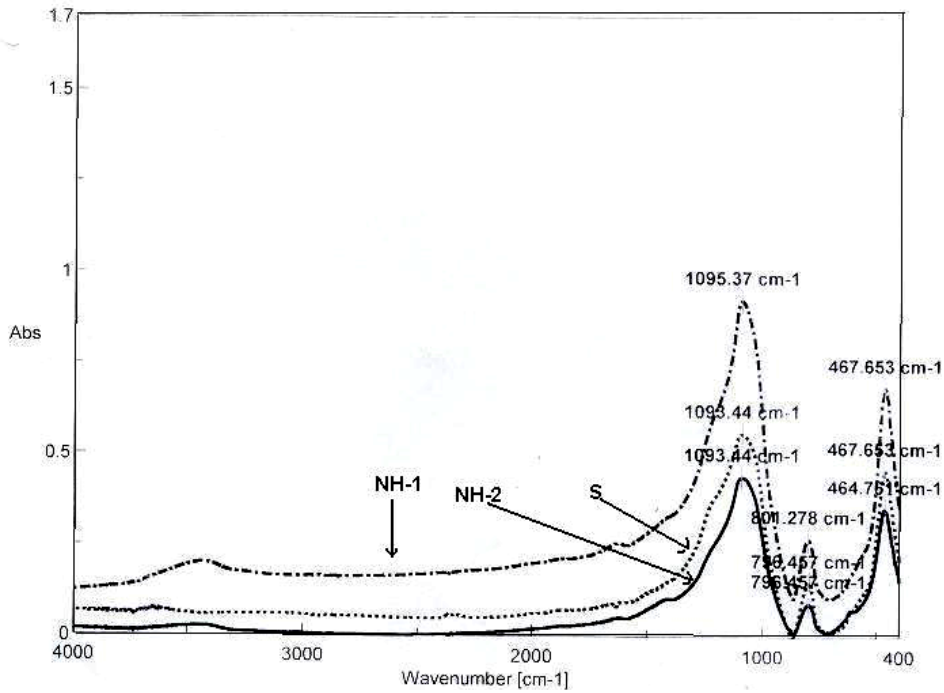


Fig.2: FTIR spectra of NH-1, NH-2 and S at 700 °C.

Results and Discussion

FTIR Spectra

FTIR spectra show four bands as shown in Figures 1, 2. The bands 461 cm^{-1} to 476 cm^{-1} belongs to O-Si-O bending vibration. Bands 1091 cm^{-1} to 1097 cm^{-1} and 797 cm^{-1} to 806 cm^{-1} belongs to O-Si-O stretching vibration. Bands at 3427 cm^{-1} to 3636 cm^{-1} is due to the chemisorbed water and surface hydroxyl groups. The intensity of band NH-2 at 3636 cm^{-1} in Figure 1 is highest indicating more chemisorbed water.

The bands from 1632 cm^{-1} to 1647 cm^{-1} belongs to C-O group. The bands of C-O for NH-1, NH-2, and S have been observed at 1643 cm^{-1} , 1627.63 cm^{-1} and 1632.45 cm^{-1} respectively.

In Figure 1 the bands are of high intensity indicating weak bond strength where as in Figure 2 the intensity of the bands is low indicating high bond strength. The bands of high stretching and bending vibrations are due to the weak bonds in O-Si-O where as low stretching and bending vibrations are due to strong bonds. Low vibration indicates the crystal formation in the ashes obtained at 700 $^{\circ}\text{C}$. Noticeable crystal formation takes place after 650 $^{\circ}\text{C}$.

At higher temperatures the formation of crystal silica increases rapidly. Increase in bond strengths indicates the increase in crystal silica formation. To confirm FTIR results the analytical method was used to determine amorphous silica as given below.

Determination of Amorphous Silica

To find out the percentage of amorphous silica, analytical technique has been used [9]. This technique involves the titration of $\text{Ba}(\text{OH})_2$ solution against the paste of glycerol and RHA using phenolphthalein as an indicator. The results are summarized in Table 1.

Table 1: Amorphous silica at different temperatures

Temperature $^{\circ}\text{C}$	% Amorphous Silica		
	S	NH-1	NH-2
600	75.2	82.2	80.3
700	73.5	75.6	70.5
800	55.5	58.2	53.8

Amorphous silica in case of NH-1 has been found higher than S and NH-2 for each calcinations temperature shown in table 1. More amorphous formation in case of S has been found beyond 600 $^{\circ}\text{C}$. The results revealed that 0.05N solution of NaOH is more favorable for the formation amorphous silica from 600 $^{\circ}\text{C}$ to 800 $^{\circ}\text{C}$.

Conclusions:

Treating the rice husk with dilute solution of NaOH (0.1N) improves the bond strength of O-Si-O of the silica obtained after burning at 700 $^{\circ}\text{C}$ and reduces the quantity of amorphous silica. Silica obtained from pretreated (0.1N NaOH) rice husk at 600 $^{\circ}\text{C}$ reduces the bond strength and improves the amorphicity of the ash. It has been observed that treating the rice husk with 0.1N NaOH is more favorable than treating with 0.05N NaOH.

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