Processing and Characterization of Active Silica Obtained from Rice Husk Ash

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Abstract. The rice husk is a residue of the rice production that has been used more and more as fuel in heat generation for drying rice, due its high calorific value and availability. This combustion produces rice husk ash constituted of amorphous silica as a major component. Rice husk ash is an alternative source for high specific area silica. In this paper processing and characterization of active silica obtained from rice husk ash is presented. The relative amount of silica was increased after burning out the carbonaceous material at different times and temperatures. A 95% silica powder could be produced after heat-treating at 700ºC for 6 h. The specific surface area of particles was increased after wet milling from 54 to 81 m²/g.

Introduction

The beneficiation of rice generates as by-product the rice husk that corresponds to about 23% of its initial weight. This husk can be used as a fertilizer in agriculture [1] or as an additive for cement and concrete fabrication [2,3]. Due to its high silicon content rice husk has become a source for preparation of elementary silicon [4,5] and a number of silicon compounds [6], specially silica [7,8], silicon carbide [9,10] and silicon nitride [10].

An increasing application of rice husk is as fuel in heat generation for drying rice, due its high calorific power (approximately 16720 kJ/kg). In this combustion rice husk ash (RHA) is produced. The burning of rice husk in air always leads to the formation of silica ash, which varies from gray to black depending on inorganic impurities and unburned carbon amounts [11].

In this paper processing and characterization of high specific surface area silica from RHA is presented. The relative amount of silica was increased by heat treatment and the specific surface area was improved by milling.
Experimental procedure

The raw material used for the experiments was a RHA obtained in a local Industry (Fumacense Ltda., Morro da Fumaça, SC, Brazil) after burning rice husk during the process of rice manufacture.

The first step for producing a high specific surface area silica or active silica (AS) from RHA consists of a thermal treatment in several temperatures. The aim of this step is to increase the relative amount of silicon oxide by reduction of carbonaceous materials present in the samples, as well as to burn out other undesirable components detected by chemical analysis. The ash samples were submitted to heat treatment in ceramic crucibles of 24.5 cm diameter (Oxford S/A, São Bento do Sul, SC). Heating cycles were carried out in air in an electric oven (Shaly, model Lab 18-1300CR) with a heating rate of 10°C/min. Each sample was held to a maximum temperature (400, 500, 600 or 700°C) for 1, 3 or 6 h. The samples were cooled down inside the oven.

The grinding step to decrease mean particles size and increase specific surface area was carried out in a high impact mill (Gabrielli, model Mill2) with a porcelain jar and microspheres of high alumina as grinding medium. Wet milling cycles of 10, 40 and 80 min were performed.

Quantitative chemical analyses of RHA were accomplished by X-ray fluorescence (XRF, Philips, model PW 2400). Mineralogical analysis was performed by X-ray diffractometry (XRD, Philips, model Xpert) with radiation CuKα. The of particles size distribution was determined by laser diffraction (Cilas, model 1064L). Specific surface area of RHA particles was determined by the nitrogen adsorption according to B.E.T. (Micrometrics, model ASAP 2000).

Results and discussion

The RHA sample after burning out at 700°C for 6 h presented the highest amount of silica, Table 1, compared to the other samples. This sample was chosen to be submitted the grinding step. The relative contents of other elements increased in general. Small divergences of this rule are probably caused by inherent uncertainty of measurements. The percentage of loss on fire decreased after heat treatment. The most common trace elements in RHA are sodium, potassium, calcium, magnesium, iron, copper, manganese and zinc [12]. Differences in composition are due to geographical factors, year of harvest, sample preparation and analysis methods.

<table>
<thead>
<tr>
<th>Components expressed as oxides</th>
<th>RHA as received</th>
<th>RHA after burning out</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>72.1</td>
<td>94.95</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>0.30</td>
<td>0.39</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>0.15</td>
<td>0.26</td>
</tr>
<tr>
<td>CaO</td>
<td>0.43</td>
<td>0.54</td>
</tr>
<tr>
<td>Na₂O</td>
<td>0.5</td>
<td>0.25</td>
</tr>
<tr>
<td>K₂O</td>
<td>0.72</td>
<td>0.94</td>
</tr>
<tr>
<td>MnO</td>
<td>0.15</td>
<td>0.16</td>
</tr>
<tr>
<td>TiO₂</td>
<td>0.05</td>
<td>0.02</td>
</tr>
<tr>
<td>MgO</td>
<td>0.70</td>
<td>0.90</td>
</tr>
<tr>
<td>P₂O₅</td>
<td>0.06</td>
<td>0.74</td>
</tr>
<tr>
<td>Loss on fire</td>
<td>24.3</td>
<td>0.85</td>
</tr>
</tbody>
</table>

Table 1 Chemical composition of RHA before and after burning out at 700°C for 6 h.
Burning out temperature and time are important factors to define whether silica remains amorphous, as in RHA, or become crystalline. In spite of higher temperatures and times of heat-treating, the structure of silica present in the ash remained essentially amorphous, Fig. 1.

![XRD of RHA: (a) as received, (b) after burning out at 700°C for 6 h.](image)

**Fig. 1** XRD of RHA: (a) as received, (b) after burning out at 700°C for 6 h.

The particles mean size of a RHA sample after burning out at 700°C for 6 h was around 33 µm, Fig. 2a, and all particles were lower than 112 µm. After the milling for 80 min the mean size was reduced for 0.68 µm, being 100% particles lower than 6 µm, Fig. 2b.

![Particle size distribution of RHA: (a) as received, (b) after wet grinding for 80 min in a jar mill.](image)

**Fig. 2** Particle size distribution of RHA: (a) as received, (b) after wet grinding for 80 min in a jar mill.
As raw material, RHA presented a specific surface area around 177 m$^2$/g. After burning out at 700ºC for 6 h, this value was reduced to 54 m$^2$/g. This reduction in specific area is proportional to heating temperature and time, which causes a sintering effect, decreasing porosity and causing agglomeration of particles. After wet grinding for 80 min, the particles specific area increased to 81 m$^2$/g.

The as received RHA samples are black with some gray particles, resulting from different stages of the carbon combustion during burning of rice husk, Fig. 3a. The active silica obtained after the heating and grinding presents reduced size of particles and gray coloration due to the lower content carbonaceous material, Fig. 3b.

**Fig. 3** Macrosopies of RHA: (a) as received; (b) after burning out at 700ºC for 6 h, and wet grinding for 80 min in a jar mill.

**Conclusion**

It was possible to obtain high specific area silica from the rice husk ash after heat-treating and milling processing. Applying simple techniques, it is possible to transform industrial residues in useful raw materials avoiding damages to the environment.

**Acknowledgements**

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**References**


