



Effects of calcination parameters on the silica phase of original and leached rice husk ash

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ABSTRACT

This paper presents a contribution to the study of the effects of calcination parameters, including temperature and time, on the silica phase of original and leached rice husk ash. X-ray diffraction (XRD) analysis reveals the presence of totally crystalline silica in the original rice husk ash prepared at 800 °C. Fortunately, no indication of the presence in crystalline phase is found in the leached rice husk ash due to leaching. However, specific surface area analysis indicates that the high calcination temperature or the long calcination time renders the amorphous silica in the leached rice husk ash less activity. Therefore, the leaching procedure, the low calcination temperature and the short calcination time are proposed in order to prepare more active amorphous silica from the rice husk.

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1. Introduction

The annual production of rice leaves behind about 40 million tons of husk as waste products in China. When the rice husk (RH) is calcined at moderate temperature, the ash contains about 92–97% silica which exists in amorphous form and exhibits high activity [1–3]. Therefore, during the last few decades, the rice husk ash (RHA) has been used as an important raw material for producing silicon-based materials (e.g. solar grade silicon, silicon carbide and silicon nitride ceramics [4,5]) and concrete additives [6]. However, impurities (e.g. sands and alkali metals) adhering to the RH debase the quality of products significantly. Leaching is considered as a promising technique for removing these impurities from the RH. Although an acid solution is more effective to remove alkali metals [7–9], leaching with water is applied more widely due to its acceptable efficiency and simple process [10–13].

The calcination temperature and time are two important factors to define whether the silica in the RHA remains amorphous or becomes crystalline [14]. Some studies have been performed to confirm an appropriate temperature for preparing the amorphous silica from the RH. Rozainee et al. [2] found that the temperature of 600–800 °C is appropriate by reviewing many literatures. However, few studies have been carried out considering the effects of calcination time.

The aim of this paper is to study the effects of calcination parameters, including the temperature and time, on the phase of silica in the original and leached rice husk ash (ORHA and LRHA).

Based on the research results, an appropriate process is proposed to prepare the active amorphous silica from the RH.

2. Materials and methods

2.1. Preparation of the RHA

A specified RH, i.e. Wuyunjing 7# (in China), is used as the raw material. It is divided into two parts. One is original and the other is suffered from water leaching. The leaching process is performed in 25 ± 1 °C deionized water with a water application rate 80 ml/g for 4 h. Subsequently, each sample is held at a maximum temperature (600, 700 and 800 °C) for 0.5, 1.5, 2.5 and 3.5 h. Each RHA sample is named for clarification, e.g. ORHA600-0.5 which implies the ORHA calcined at 600 °C for 0.5 h.

2.2. Characterization of the RHA

The surface morphologies of RH and RHA are examined using SEM (JSM-6380LV, JEOL, Japan). The mineralogical analysis is performed using XRD (D8 ADVANCE, BLUKER, Germany). Moreover, the specific surface area of LRHA is determined according to the nitrogen adsorption method (JW-04, JWGB, China).

3. Results and discussion

Because a small quantity of exterior impurities adheres to the internal surface of RH, only the external surface morphologies of RH and RHA are observed in this study. As shown in Fig. 1a, a large

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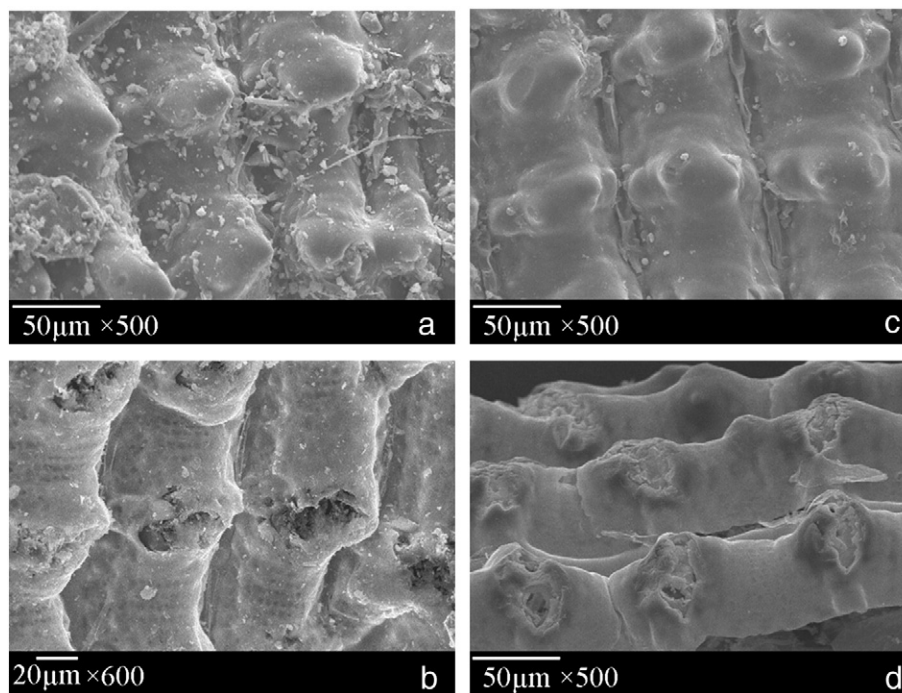


Fig. 1. The external surface morphologies of RH and RHA. (a) ORH; (b) ORHA; (c) LRH; (d) LRHA.

quantity of exterior impurities adheres to the external surface of original rice husk (ORH). Two factors mainly account for their origin, i.e. sands [15] and RH powders. They tend to react with potassium to form alkali rich melts (Fig. 1b). Once carbon is entrapped in these melts, it cannot be easily oxidized as it is not direct contact with air for further conversion even at higher calcination temperature or under higher oxygen gage pressure [1,2,8]. Fortunately, the external surface of leached rice husk (LRH) is almost free from the exterior impurities after being leached in the deionized water (Fig. 1c). In addition, no melts can be found on the external surface of LRHA (Fig. 1d).

Fig. 2 shows the changes of the contents of ORHA and LRHA with the calcination temperature and time. Because of higher volatility of some metallic elements at higher calcination temperature [16], a reduction of the contents of ORHA and LRHA is observed with the calcination temperature increase. Moreover, the contents of LRHA are less than those of ORHA due to the removal of impurities (cf. Fig. 2a and b). As shown in Fig. 2a, the contents of ORHA dramatically decrease with the calcination time increase, which attributes to the slow oxidation of carbon residue in ORHA. However, the contents of LRHA have no obvious change when the calcination time is longer than 1.5 h (Fig. 2b), which implies no black or gray particles and almost pure silica existing in LRHA.

Fig. 3 shows the XRD patterns of ORHA and LRHA. The phase transformation of the silica with the calcination temperature and time increase can be obviously found from the patterns. As shown in Fig. 3a, there is no indication of silica presence in crystalline form in ORHA600, which implies that the exterior impurities adhering to the ORH are mainly the RH powders. However, the silica in ORHA700 presents in both amorphous and crystalline form. The narrow peaks about $2\theta = 22^\circ$ are noticed in Fig. 3b, which indicates an incipient crystallization process. In addition, the silica in ORHA700 shows the ever severe crystallization with the calcination time increase. Worse still, the silica in ORHA800 totally presents in crystalline form (Fig. 3c). The results indicate that the silica phase of the ORHA is sensitive to both the calcination temperature and time. Although the LRH is suffered from the same calcination temperature and time, a broad peaks about $2\theta = 22^\circ$ are noticed in Fig. 3d–f, which indicates an

essentially amorphous form of silica in the LRHA. It can be reasonably inferred that the sensitivity of the silica phase of ORHA to the calcination temperature and time may be relevant to the alkali metals which facilitate the initiation of the formation of cristobalite [17]. Moreover, the RHA which remains amorphous after being calcined at 700°C for 6 h in literature [2] may refer to the LRHA.

Unfortunately, not all amorphous silica is considered as an active raw material as the presence of SiOH groups and $\text{Si}(\text{OSi})_4$ tetrahedra makes it less active [18,19]. It is well known that the specific surface area is one of the most important properties for the active materials. Therefore, the specific surface area of LRHA is determined according to the nitrogen adsorption method. As shown in Fig. 4, the specific surface area of LRHA decreases when the calcination temperature and time increase. The specific surface area of LRHA600-0.5 is $104\text{ m}^2/\text{g}$. However, it rapidly decreases to $10\text{ m}^2/\text{g}$ for LRHA800-3.5. The dramatic decrease in the specific surface area is proportional to the calcination temperature and time, which is caused by agglomeration effect and diminishing porosity [3].

4. Conclusions

The silica phase of ORHA is sensitive to the calcination temperature and time by the facilitation of alkali metals. High calcination temperature or long calcination time leads to the formation of crystalline silica. However, the silica in LRHA remains amorphous in a wide range of calcination temperature and time, which confirms the benefit of leaching on the preparation of amorphous silica from the RH. Taken activity into account, leaching procedure, as well as the low calcination temperature and short calcination time is proposed in order to prepare more active amorphous silica from the RH.

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