



Observation of Physio-Chemical Differences of Rice Husk Silica under Different Calcination Temperatures

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Authors' contributions

This was a collaboration work of all authors. The main author RS designed, performed, analyzed, interpreted and prepared the first draft of the manuscript. Authors VCL and BCL provided technical support and revised the manuscript. Author MT supervised the research. All authors read and approved the final manuscript.

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ABSTRACT

Aims: Amorphous silica in rice husk ash is a reliable and sustainable resource, although rice husks are currently an issue for farmers. Amorphous silica is a resource in various industrial applications and can easily be obtained from rice husks if they are treated correctly. If the treatment is not conducted properly, amorphous silica becomes crystalline, which is carcinogenic. In this study, physio-chemical observations of the transformation of amorphous to crystalline silica were conducted under “no-fixed carbon” conditions.

Study Design: Rice husks were burned at different temperatures in an electric furnace and their ash was analyzed for solubility, fixed carbon, volatile matters, ash, and moisture. XRD, WDX, and SEM were used for physical state analysis.

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Location and Duration of Study: The study was conducted in a laboratory at Toyama Prefectural University from April 2015 to March 2017.

Methodology: Rice husks from Koshihikari (*Oryza sativa L.*) were used in this study. Rice husks were calcinated in a laboratory-scale electric furnace (KBF794N1, Koyo for 500-1,100°C, Lindberg, Koyo for 1,500°C) for two hours at 500-1,500°C after being washed with acetic acid (5%). The physical state of rice husk silica was determined by X-ray diffraction analysis (XRD: MultiFlex 40 kV, 30 mA, CuK α , 2 θ : 5-80°, Rigaku). The rice husks were ground into a powder and analyzed by XRD.

Results: It was found that amorphous silica in rice husks transformed into crystalline silica at burning temperatures of over 1,000°C. Total silica (T-silica) in the ash burned at 500°C—800°C, almost equal to the burning temperature of soluble silica (S-silica), and beyond 900°C, the non-S-silica portion of T-silica increased. From the morphological observations, at low temperatures the rice husk ash was fragile and did not hold its original shape; in contrast, the rice husk ash held its original shape at high temperatures. At extremely high temperatures, such as 1,000 and 1,500°C, the original shapes seemed to shrink by melting on the ash's surface of the ash.

Conclusions: Under no influence of fixed carbon, the amorphous silica in rice husks transformed to crystalline silica at temperatures over a 1,000°C burning temperature. The T-silica in the ash that burned at 500°C—800°C were almost entirely S-silica, and then over 900°C, the non-S-silica portion increased in T-silica.

Keywords: Rice husk; ash; silica; calcination; solubility; surface observation; SEM.

1. INTRODUCTION

Rice husks are fundamental biomass resources because they are produced annually, making them sustainable and reliable. In contrast, wood biomass takes a long time to grow, so people have to wait to use it, which is not sustainable or reliable. Approximately 150 million tons of rice husks are generated globally per year, with two million tons from Japan [1]. Although rice husks have been proposed for many uses, there are few examples of sustainable applications using them, which could be due to farmers considering them to be a problem and paying a disposal fee for their management.

Rice husks have been used as adsorbents in wastewater treatment [2] and fuel for energy generation [3]. Rice husk “ash” has more options for application than the rice husks themselves. Rice husk ash has been widely proposed for various industrial uses including coatings, pigments, cement, insulators, rubber, and electronics [1]. Tateda [4] proposed a method for recycling rice husks, using them as fuels in a proposed sustainable local energy system and suggested that rice husk ash could be used as a fertilizer for growing healthy rice plants.

To recycle rice husk ash, the ash must have a certain, essential condition - the silica in the ash must be amorphous. The silica in rice husks is

also known as “plant opal,” and is amorphous [5,6]. Soluble silica is assimilated by rice plants, and they use it to form their body structure for preventing damage from wind, evaporation of water from rice, and invasion by pests. As soluble silica is amorphous, plant opal is also amorphous.

Amorphous silica is transformed by exposure to temperature. Under high temperatures, amorphous silica turns to quartz, tridymite, or cristobalite. Rice husk silica must be carefully handled, because crystalized silica is a carcinogen, according to a WHO report [7]. The transformation, however, is not always the same, and the existence of alkali metals and conditions of calcination would produce different materials.

For recycling rice husks, the production of rice husk ash has more advantages than the rice husks alone, as mentioned earlier. A burning process is required to remove unwanted materials for recycling, such as carbon, alkali metals, and volatile materials. Silica in the ash must remain amorphous, which is highly influenced by the burning temperatures.

There are many examples of rice husks being burned for recycling. Experiments have been performed at the following temperature ranges; 300—860°C [8], 600—700°C [9], 500—900°C [10], 300—900°C [11], 400—900°C [12], 700—800°C [13], 20—1,000°C [14], 400—900°C [15],

250–600°C [16], 900–1,050°C [17], 400–900°C [18], 0–600°C [19], 600–850°C [20], and 700–900°C [21]. The amorphous silica crystallizes at around 1,000°C, but this depends on the conditions of the rice husks. None of the studies mentioned above provided comprehensive observations under changes in temperature, such as the transformation of amorphous to crystalline silica, which, to the authors knowledge, has had no studies.

To recycle silica in rice husk ash, the production of white rice husk ash is important. The lower the amount of fixed carbon in the ash, the better the quality of silica. In this study, physical and chemical changes in rice husk ash, especially the silica in the ash, were observed under minimal fixed carbon conditions. Two studies have used temperatures higher than 1,000°C to observe the transformation from an amorphous to a crystalline state. Nakata et al. [22] conducted calcination from 400–1,500°C but once pyrolyzed rice husks were produced, they were calcined. Sarangi et al. [23] chose burning temperatures of 400–1,200°C, and the rice husks were calcined under N₂ gas to promote pyrolysis to produce crystalline silica, which is a completely different process. The silica ash turns to crystalline silica if alkali metals and fixed carbons remain in the ash. It is very important to understand physical and chemical changes as functions of calcination temperatures.

2. MATERIALS AND METHODS

All data were triplicated and their mean values were used to produce figures.

2.1 Rice Husks and Ash Preparation

Rice husks from Koshihikari (*Oryza sativa* L.) were used in this study. Rice husks were calcinated in a laboratory-scale electric furnace (KBF794N1, Koyo for 500-1,100°C, Lindberg, Koyo for 1,500°C) for two hours at 500-1,500°C after being washed with acetic acid (5%). The 500°C minimum was selected from authors' previous study [4].

2.2 Qualitative Analysis of Amorphous and Crystallized State of the Rice Husk Silica

The physical state of rice husk silica was determined by X-ray diffraction analysis (XRD:

MultiFlex 40 kV, 30 mA, CuK α , 2 θ : 5-80°, Rigaku). The rice husks were ground into a powder and analyzed by XRD.

2.3 Measurement of Fixed Carbon, Ash, Volatile Matters, and Moisture

Fixed carbon, ash, volatile matter, and moisture content in rice husk ash were determined following the Japan Industrial Standards (JIS) M 8812-8, -6.4.1, -7.2.4, and 5.2.4a, respectively [24].

2.4 Measurement of Element Contents in Rice Husk Ash and Content Estimation of Oxides

A length dispersive X-ray fluorescence spectrometer (WDX: PW2440, Spectris) was used to measure element content in rice husk ash, and, based on the results, estimate oxides.

2.5 Solubility Measurement of Rice Husk Silica (S-silica)

The solubility of rice husk silica (S-silica) was measured following the Japan Standard Methods for solubility of silica in fertilizers (4.4.1.c) [25].

2.6 Measurement of Total Silica in Rice Husk Ash (T-silica)

The total silica in rice husk ash (T-silica) was measured following modified Japan Standard Methods for total silica content in fertilizers (4.4.1.d) [25]. The acid extraction process in the method was skipped.

2.7 Surface Observation of Rice Husk Ash

The surfaces of rice husk ash particles were observed at macro and micro scales by a hand zoom recorder (Digital Micro Mobile Z, Biomedical Science) and a field emission scanning electron microscope (FE-SEM, JSM-7001FTTLS, JEOL), respectively.

3. RESULTS

3.1 Solubility of Rice Husk Silica as a Function of Calcination Temperature

Solubility is presented as the percentage of soluble silica in the total silica. The degree of

“amorphous state” silica in rice husks can be evaluated by “solubility.” The higher the solubility, the better the amorphous state of silica in the rice husk ash [26]. Fig. 1 shows the solubility at each calcination temperature.

Solubility was high through 500—900°C, which means that more silica in rice husk ash was in an amorphous state. Solubility rapidly decreased at 1,000°C, however. The same trend was observed by Tateda et al. [27], but it was gentler and decreased to 83% at 1,000°C, compared to below 40%, observed here. Solubility was 3.6% at 1,000°C, and decreased to 0.6% at 1,500°C. Deshmukh et al. [18] proposed a silica activity index that was based on the soluble fraction dissolving in boiling 0.5 M NaOH within 3 minutes. In this paper, solubility was discussed as the soluble fraction dissolved in HCl and NaOH, described in detail by Tateda et al. [27], which

could have similar values to the silica activity index.

3.2 Analysis of Four Contents of Rice Husk Ash

Four major components of rice husk ash; water, volatile matter, ash, and fixed carbon, were analyzed. The results are presented in Fig. 2. As the figure shows, fixed carbon was almost lost from the surface of ash at all temperatures, so the color of the ash under all temperatures was white. Tanaka et al. [22] reported that the ash of pyrolyzed rice husks contains a fixed carbon content of 0.46% at 500°C, and it decreases to 0.02% at 800°C. This implies that more carbon remains on the surface of the rice husks after pyrolysis treatment. Volatile matter was eliminated at 800°C, and water content was lost after 1,100°C. After 1,100°C, the ash component dominated rice husk ash.

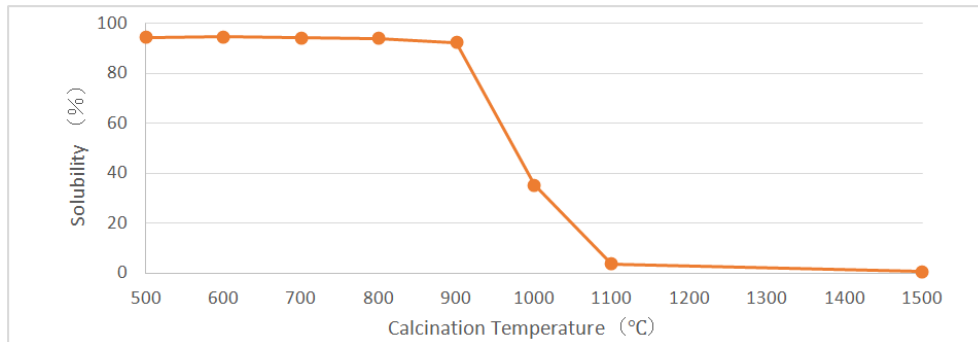


Fig. 1. Solubility trend as a function of calcination temperature

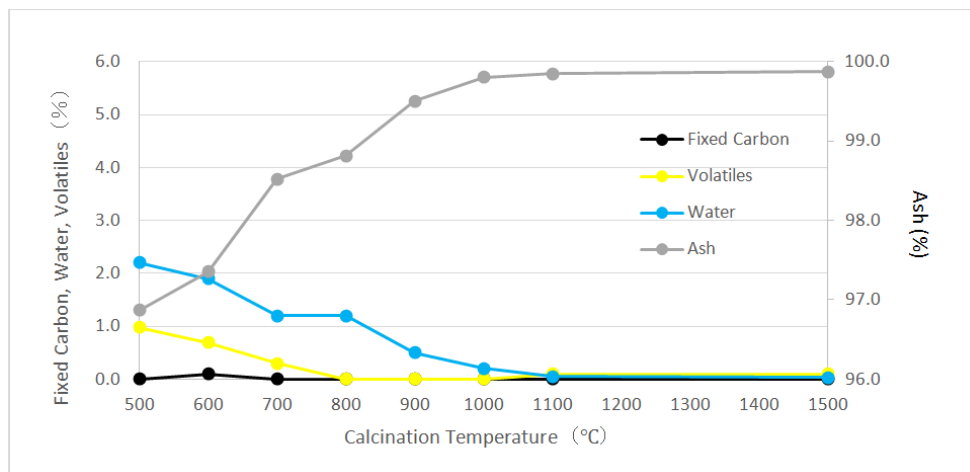


Fig. 2. Four components of rice husk ash

3.3 Analysis of Components of Rice Husk Ash after Removing Fixed Carbon

Based on the results of section 3.2, the ash did not contain fixed carbon, therefore it has three portions: ash, volatile matter, and water. The ash portion should consist of silica and non-silica components. By measuring the total silica in the ash, more details, especially in the ash portion, were estimated (Fig. 3).

In rice husk ash, the ash portion included T-silica and ash without T-silica. In the figure, T-silica includes both S- and non-S-silica. The ash without T-silica portion could have been decreasing as a function of temperature. The types of oxides which comprised the ash-except-T-silica portion were analyzed. Oxides such as P_2O_5 , CaO , Fe_2O_3 , and K_2O were dominant, which was also stated by Fernandes et al. [14] and Nakata et al. [22]. The ash portion of rice husk ash mainly consists of T-silica and the oxides mentioned above. According to Bie et al. [9], K_2O decomposes at high temperatures, and holds more fixed carbon in ash. Fig. 2 shows that no carbon remained at high temperatures, which differs from the results of Bie et al. [9], because rice husks were burned after acetic acid washing, and the K_2O content was below 0.3%. In contrast, rice husks burned without washing had a K content of 6.1–8.1% (2.3–2.8% as K_2O). The high percentage of K_2O influenced the residual fixed carbon in the ash. One type of the *Oryza sativa L.* (Koshihikari) rice plant was used in this study and the ash content (oxidized elements in the

portion of ash excluding T-silica) was dependent on where the rice plants grew [19]. This could be the consequence of differences in fertilizers. Due to differences in paddy field soil properties, farmers use their own fertilizers.

3.4 XRD Analysis

XRD analysis for each temperature was conducted to verify whether the silica in rice husk ash was amorphous or crystallized. Fig. 4 a)-c) shows the XRD curves for the ash samples, with calcination temperatures. Fig. 4a represented XRD curves from 500–1,000°C because they were almost the same.

According to the figure, the silica in rice husk ash burned at 500–1,000°C had an amorphous nature, while the silica burned at 1,100°C had more of a crystalline nature, and at 1,500°C it had a cristobalite nature. Deshmukh et al. [18] conducted similar experiments and obtained XRD curves similar to those seen in Fig. 4. They washed rice husks with HCl followed by water, and then burned them at 400 to 900°C. They concluded that amorphous silica turned crystalline at 900°C. However, this phenomenon was not observed in the current study. It is highly unlikely that washing by HCl caused the difference because, in general, amorphous silica without impurities (such as alkali metals and fixed carbon) does not turn into crystalline form below 1,000°C, as seen in this study. Notably, Deshmukh et al. [18] do not specify the medium used to burn the rice husks in their study.

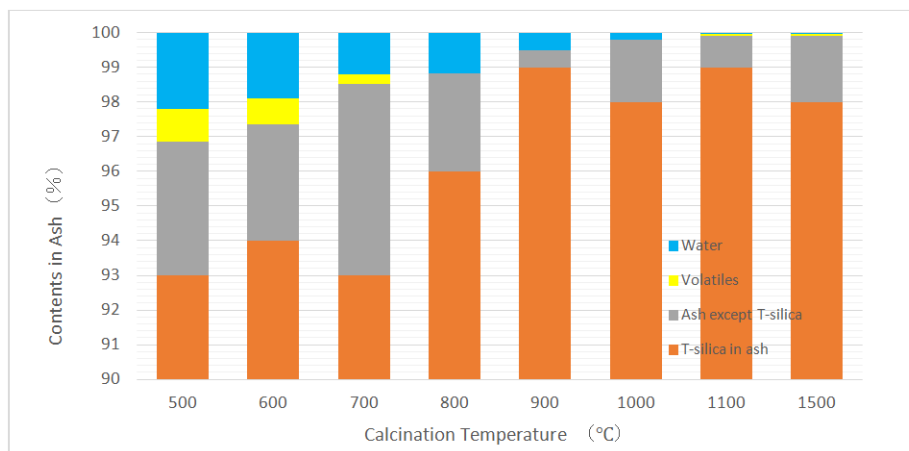


Fig. 3. Detailed content of the ash portion

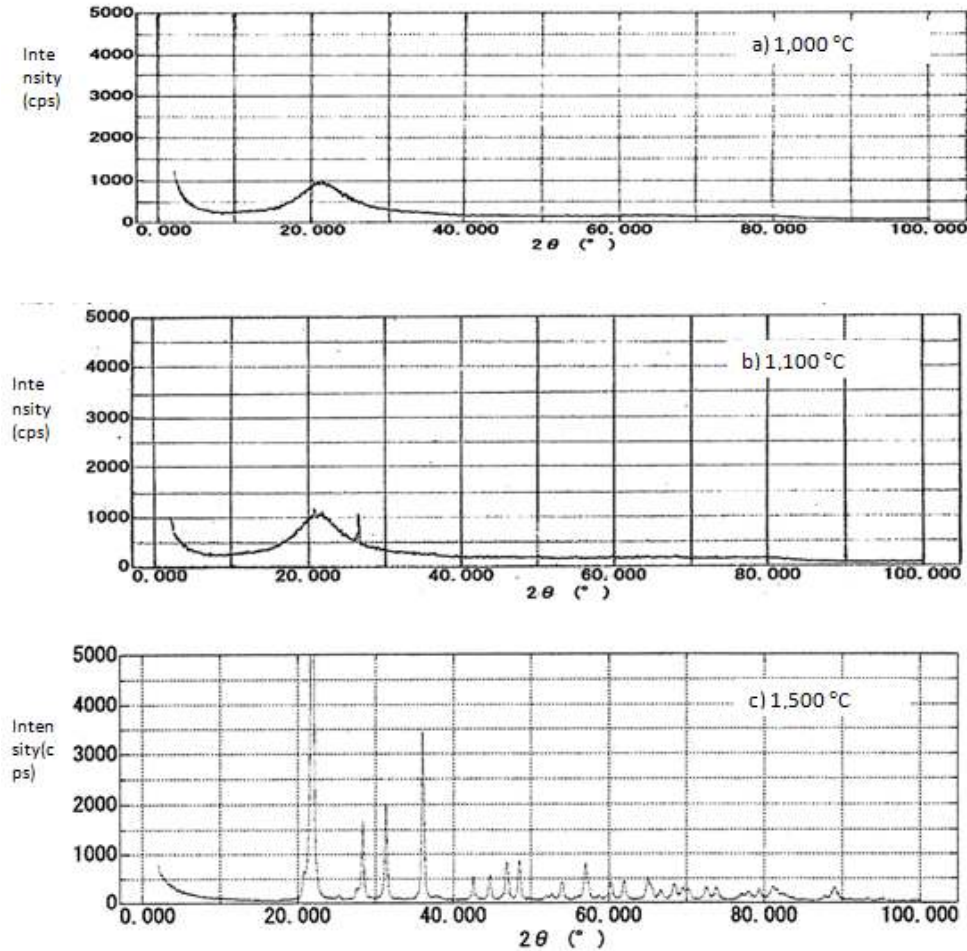


Fig. 4. XRD curves at different calcination temperatures. a) 1,000°C, b) 1,100°C, c) 1,500°C

3.5 Surface Observation of Rice Husk Ash

Surface observations were performed at macro and micro scales using a handy zoom camera and a FE-SEM, respectively.

According to Fig. 5, rice husk ash burned at the lower temperatures, i.e., 500 and 600°C, was broken into small pieces, indicating that the ash burned at those temperatures was fragile and easily broken. Ash burned at 700–1,500°C held the rice husk shape, however, the shape seemed to shrink for samples burned at over 1,000°C, i.e., 1,100 and 1,500°C, because of the extremely high temperatures. This indicates that ash at 1,500°C calcination became rounded and narrower in width in comparison to the samples burned at 700–1,000°C. The close observation, but SEM, of rice husk ash has not

been reported anywhere according to authors' knowledge.

Fig. 6 shows surface observations at a micro scale for samples burned at 600°C, 1,000°C, 1,100°C, and 1,500°C, which were observation of the inner surface. As the samples at 500°C and 600°C and the samples at 700°C through 1,000°C had similar appearances, the 600°C and 1,000°C samples were selected as representative samples, respectively, for micro observation. There was no great morphological difference between the samples at 1,000°C and 1,100°C. The wall thickness of the pores seemed much thicker at 600°C than at the higher temperatures of 1,000°C and 1,100°C. The surface of the sample burned at 1,500°C had completely different morphology. It became very smooth, which could be the result of melting due to the extremely high temperature

(1,500°C). Nakata et al. [22] have presented SEM photos in their paper. However, the rice husks used in their study were processed, that is, compressed and crushed before burning. Thus, a comparison between their work and the current study cannot be made because

the ash contained pieces of broken rice husks. According to their results, the SEM photo at 500°C is similar to the one at 600°C in this study, and the SEM photo at 900°C matches the one at 1500°C in our study very closely.

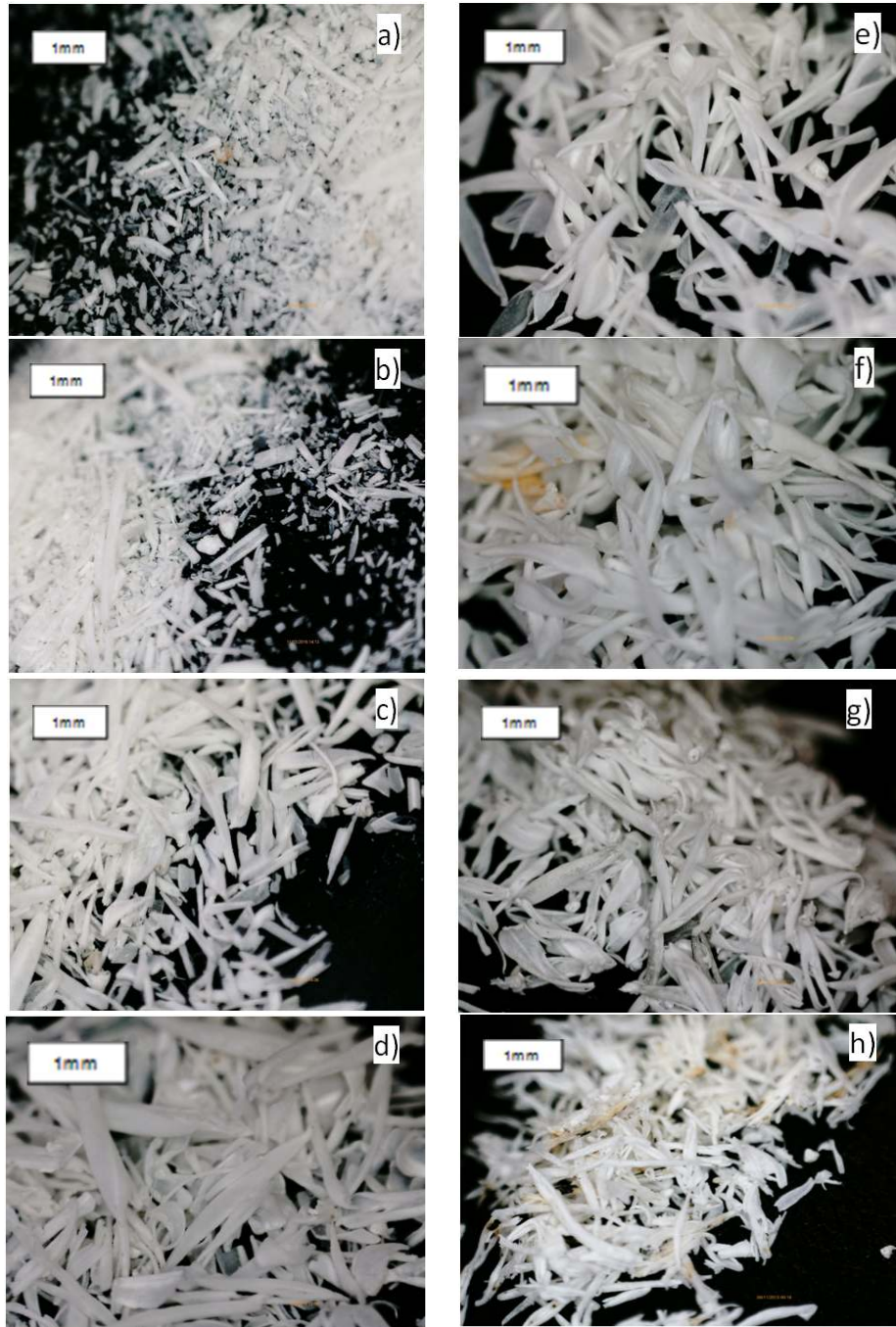


Fig. 5. Close observations at macro scale of rice husk ash at each calcination temperature
a) 500°C, b) 600°C, c) 700°C, d) 800°C, e) 900°C, f) 1,000°C, g) 1,100°C, h) 1,500°C

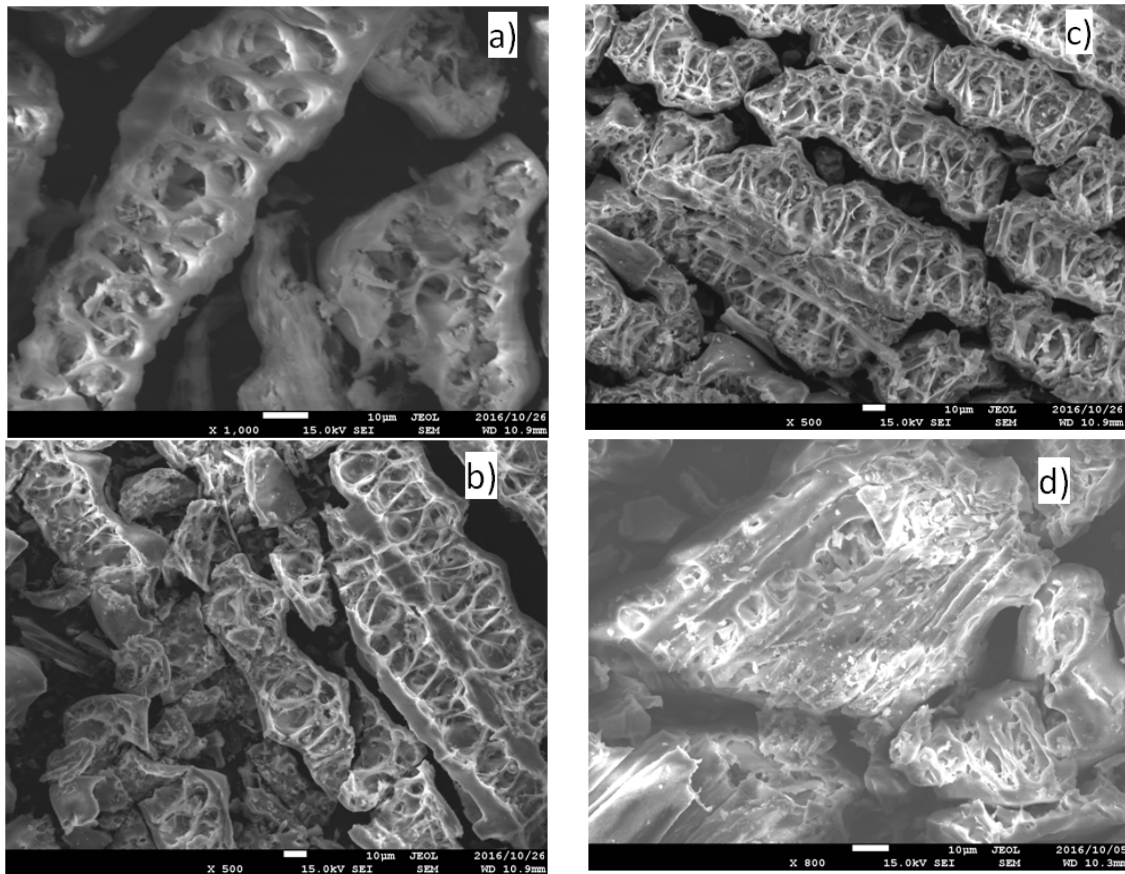


Fig. 6. FE-SEM results for the samples; a) 600°C, b) 1,000°C, c) 1,100°C, and d) 1,500°C

4. DISCUSSION

Regarding Fig. 1, the solubility of the sample washed by citric acid decreased to only 83.1% according to Tateda et al. [27]; in contrast, the solubility of the sample washed acetic acid in this study decreased to 35.3%. Although it was not considered in this study, the effects of different washing solvents on the solubility of rice husk silica are interesting and should be studied. In addition to rice husks, the solubility of crystal, tridymite, and opal was measured, and their values were 0.4%, 0.4%, and 49.3%, respectively. According to Fig. 1, silica under the 1,000°C treatment could have a similar nature to opal, which is amorphous and not crystallized, according to Fig. 4, and showed low solubility. The silica at 1,500°C, which had a solubility of 0.6%, had the same solubility as crystallized crystal and tridymite, corresponding with the results of Fig. 4.

As solubility was calculated by (measured soluble silica (g)/ sample taken at the beginning

(g)) x 100, the solubility values can be directly applied to Fig. 3 due to the loss of carbon from the ash (Fig. 2). Considering measurement errors, S-silica constituted all the T-silica at 500°C–800°C. The differences in the occupational percentage of T-silica and S-silica at burning temperatures above 900°C were approximately 5 points, 63 points, 95 points, and 97 points at 900°C, 1,000°C, 1,100°C, and 1,500°C, respectively. The silica in rice husk ash is usually expressed as T-silica, and the soluble portion is S-silica and amorphous. The remainder is non-S-silica, some of which are amorphous and crystallized.

From 500°C to 800°C, all of the T-silica was S-silica. At 900°C, approximately 5% of 99% was non-S-silica, and the other 94% was S-silica. At 1,000°C, approximately 63% of 98% was non-S-silica, and 35% was S-silica. At 1,100°C, 95% of 99% was non-S-silica, and 4% was S-silica. At 1,500°C, 97% of 98% was non-S-silica, and 1% was S-silica. As silica in rice husks is initially amorphous and soluble, T-silica is S-

silica, and the silica burned at 500°C to 800°C shows this nature. The silica in rice husks changes its nature under high heat and begins to lose solubility, but it is still amorphous in nature, so, amorphous nature does not mean soluble. Under higher heat, the amorphous nature changes to crystalline; the change point was 1,100°C, according to Fig. 1.

Impurities such as alkali metals, particularly potassium, strongly influenced the transformation of amorphous silica to crystalline in rice husks [28]. The rice husks burned above 800°C produced cristobalite [15], because the rice husks were burned without being washed so impurities remained on the surface of the rice husks. In contrast, rice husk silica in this study crystallized at temperatures over 1,000°C, because impurities on these rice husks were low and did not affect the acceleration of crystallization.

Volatile matter in rice husks consisted of cellulose, lignin, pentosans, and other organic matter, in the order of high to low content [29]. The calories of volatile matter in rice husks are usually low, and volatile matters that burn more easily or less easily have low and high calorie content, respectively [19]. As the analysis of volatile matter in Fig. 2 was not for rice husks, but for rice husk ash, the volatile portion observed in this figure would be those that were difficult to burn. The percentage of volatile matter decreased as calcination temperatures increased, and was non-existent in samples burned at temperatures over 800°C. The volatile material trend was similar to that of water content. The surface of the ash melted, became shiny, and entered glass-like states as calcination temperature increased, which is why water and volatile material could not remain on the surface of the ash, which diminished.

Ash morphology can be observed in Fig. 5. Rice husk ash was fragile when the rice husks were burned at 500°C and 600°C. Ash was easily broken down and became powder under those temperatures. Ash treated at temperatures over 700°C became firm and kept the rice husk shape. The shape shrank as the temperatures increased, and it appeared to become thinner and firmer. According to Fig. 2, water content decreased as temperatures increased and was almost nonexistent at 1,100°C and 1,500°C. Bakar et al. [10] stated that silica produced at 600°C burning was the best of silica burned between 500–900°C,

according to its surface area and particle size, which is consistent with the results from this study; it is implied that the fragile nature of ash burned at 500°C and 600°C is attributed to the largest and smallest values of surface area and particle size, respectively. Xiong et al. [12] stated that the densification of rice husk ash decreased with increasing calcination temperatures. Their results are consistent with the results of this study, according to Fig. 2.

More close observations at a micro scale were conducted by FE-SEM of the reverse surface of rice husks. The structures of rice husks are more noticeable as temperatures increased, however, at the highest temperatures the structures could not be clearly recognized due to the surface melting.

5. CONCLUSIONS

Under no influence of fixed carbon, the amorphous silica in rice husks transformed to crystalline silica at temperatures over a 1,000°C burning temperature. The T-silica in the ash that burned at 500°C–800°C were almost entirely S-silica, and then over 900°C, the non-S-silica portion increased in T-silica. Volatile matter was nonexistent after burning at 800°C. The ash portion excluding T-silica was mainly composed of P, Ca, Fe, and K oxides. For morphological observations, at low temperatures the rice husk ash was fragile and did not hold its original shape; in contrast, rice husk ash held its original shape at higher temperatures. At extremely high temperatures, such as 1,000°C and 1,500°C, the original shapes seemed to shrink by melting on the surface of the ash; this observation was supported by SEM analysis.

COMPETING INTERESTS

Authors have declared that no competing interests exist

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