Effects of Compaction on Silica Solubility and Physical Properties of Rice Husk Ash for Fertilizer Use

RYOKO SEKIFUJI1, LE VAN CHIEU2, MASAFUMI TATEDA∗, and HIROSHI TAKIMOTO3

1School of Environmental Engineering, Toyama Prefectural University/Toyama 939-0398, Japan
2Project Management Department, Vietnam National University/Hanoi, Vietnam
3Faculty of Bioresources and Environmental Sciences, Ishikawa Prefectural University
/ Ishikawa 921-8836, Japan

Abstract

Extremely lightweight of rice husks due to low specific gravity is disadvantageous for accelerating its usage. To overcome this disadvantage, rice husks are sometimes compacted, that is, pelletized or solidified to assist easy and cheap collection and hauling. However, the compaction process may negatively affect the properties of silica in the husk. In this study, the influence of the compaction process was studied through the solubility and physical properties of silica. Two types of fuels, pellet and stick fuels, were synthesized and their influence on the properties of the fuel ash was evaluated. Consequently, stick fuels were better than pellet fuels in terms of fertilizer use of the ash during their use in residential stoves and small commercial heaters. Contrastingly, rice husks in the existing form were better than stick fuels under washing conditions; however, rice husks had disadvantages in terms of cost and handling during collection and hauling. Selection of rice husks in their existing form or compacted rice husks must be carefully considered based on the business approaches of the rice production stakeholders.

Key Words: rice husk; fuel; pelletization; solubility; fertilizer; silica

INTRODUCTION

The development and subsequent applications of recycling technologies are important for waste recycling; however, collection and hauling are important aspects because their cost may impose financial burdens on the recycling scheme1). Rice husks are problematic for rice production stakeholders owing to their bulky nature, which is disadvantageous during hauling for long distances as the specific gravity of rice husks is 0.1 ton/m3). Therefore, when rice husks have to be hauled for long distances, they should be compacted as pellets or sticks 10 times more than the native form. Additionally, compact rice husk forms, such as pellets and sticks, may promote the use of rice husks as fuels because of their low collection and hauling costs. The heating value of rice husks possess is approximately 12 MJ/kg (dry base) of energy; hence, they serve as potential fuel sources. Sheng and Azevedo (2005)2) proposed that the biomass heating value should be estimated for determining various biomass component properties and chemical compositions. Studies have assessed the use of rice husks for the following purposes: fuels such as biochar3), hot water production 4), mixed fuel synthesis for electricity generation5), solid fuel through torrefaction6), fuel by fluidized-bed combustion7), power generation by gasification8), rice husk-to-fuel conversion by three conversion technologies (hydrothermal carbonization with pelletization, pyrolysis, and anaerobic co-digestion processes9), and thermal bio-
conversion including ethanol production technologies\textsuperscript{10}. Quispe et al. (2017)\textsuperscript{11} studied various agricultural residues and transformed rice husks into fuel through pyrolysis. The complete recycling of rice husk incineration ash is required to use rice husks as a fuel source\textsuperscript{11}. The biggest challenge in using rice husks as a fuel source is that they leave a large amount of silica ash residue, which is approximately 20\% in weight, after incineration. Thus, the different rice production stakeholders face difficulties in managing the ash in the absence of proper ash management. Therefore, the application of silica fertilizers in paddy fields has been proposed for the complete use of rice husk ash\textsuperscript{12}. Rice plants require intensive silica application for healthy growth. As rice is a staple food in Japan, the demand for silica is high to facilitate the large-scale production of rice. At present, silica is imported for application in Japanese paddy fields. However, silica originating from rice husks of Japanese rice fields (called indigenous silica) can serve as a good substitute for imported silica. This basic concept is illustrated in Fig. 1.

In Japan, after paddy harvest in autumn, the paddy devoid of straw is milled, resulting in the production of rice along with rice husks. The rice husks are burned in a boiler, and then subjected to heat recovery and \( \text{CO}_2 \) recycling. Hot water or electricity is produced from the heat recovery process, and high value-added agricultural products are harvested from \( \text{CO}_2 \) recycling\textsuperscript{13}. For example, strawberries are known to become sweeter when grown in high \( \text{CO}_2 \) environments. Incineration of rice husk results in the production of ash rich in silica, which is subsequently used as a silica fertilizer. The use of exogenous silica fertilizers originating from other industrial sectors, such as the iron industry in Japan, could be substituted with rice husk ash containing indigenous silica. Multiple applications of silica, such as in the production of concrete, insulators, food, cosmetics, catalysts, detergents, and solar panels, have been reported\textsuperscript{14}. However, we believe that the use of rice husk ash as a silica fertilizer is the best alternative for the complete recycling of rice husk ash after heat recovery, especially in Japan, where the silica content in soil is very low\textsuperscript{12}. Silica in rice husk currently is disposed as a waste although rice plants need silica for healthy growth. The quality of ash as a silica fertilizer for paddy fields has been evaluated using the solubility index of silica in rice husks\textsuperscript{12}. The solubility of silica in the incineration ash of rice husks has been evaluated; however, the solubility of silica in the incineration ash of rice husks in the form of compact pellets and sticks has not yet been studied.

Ríos-Badrán et al. (2020)\textsuperscript{16} synthesized pellet fuel and compared a fuel made of only rice husk with a mixture of rice husk and wheat. Additionally, knowledge of how the compaction process affects the solubility of silica in rice husks is important when considering the use of rice husks as fuels. Several studies have reported extremely low solubility of silica and low availability of biogenic silica in water\textsuperscript{17-20}. Silica forms with long availability periods are expected to have slow effects on rice plants, unlike silica gels and calcium silicate, and they can be applied every year. The use of short materials as silica fertilizer in rice production, converting waste into resources, and heat recovery are

![Ideal Agriculture Loop](image_url)
important for rice production stakeholders globally. The objective of this study was to evaluate the solubility and physical properties of silica in incineration ash of compact, pelletized, and solidified rice husks.

**MATERIALS AND METHODS**

Sample preparation of rice husk and rice husk fuel Rice husk from Koshihikari (*Oryza sativa* L.) was used in this study. The rice husk was pelletized and solidified after washing with 5% citric acid for removing alkali metals such as potassium and sodium. The rice husk was placed in a laundry net and soaked in 5% citric acid solution at a 1:10 (rice husk: citric acid solution in volume) ratio for 24 h, rinsed with water, and dried for compaction. Pellet and stick fuels were produced by pelletization and solidification (Fig. 2) using KNP-701 and FHM-120, respectively (Fig. 3). The machine specifications are presented in Table 1. The stick fuel was approximately 30 cm in length. The specific density of pellet and stick fuels was 1.4 and 1.2 ton/m$^3$ (wet base, unpublished data), respectively, whereas that of the native form of rice husks (as-are) was 0.11 ton/m$^3$. The rice husk was washed with 5% acetic acid. Acetic acid was selected for washing the as-are samples because it is cheaper (0.8 ×) than citric acid; additionally, according to previous studies, citric acid and acetic acid demonstrated similar washing efficiencies.

**Solubility measurement** The solubility of silica in rice husk ash was measured following a method described by Tateda *et al.* (2016)$^{15}$, with some modifications. This method was based on the standard method 4.4.1.c$^{21}$, but the first step of hydrochloric acid treatment was skipped to save time. However, the results were similar to those obtained using the standard method. Currently, there is no standardized method for measuring the solubility of rice-husk-derived silica; method 4.4.1.c was not designed for silica fertilizer derived from rice husks, but for fertilizers containing silica gel.

**Measurement of the physical properties of rice-husk ash** The physical properties of rice-husk ash are good indicators for determining the quality of ash$^{22}$. The properties were estimated according to the relative content of fixed carbon, ash, volatile solids, and moisture, which were determined using JIS M 8812; the subsequent values for the content of moisture, ash, volatile solids, and fixed carbon were JIS M 8812-5.2.4a, 8812-6.4.1, 8812-7.2.4, and 8812-8, respectively$^{23}$. X-Ray diffraction (XRD) was performed using MultiFlex (40 kV, 30 mA, CuKa, 2θ: 5–80°, Rigaku).

**Experimental procedure** Approximately 60 g of the as-are washed [Fig. 4(A)], pellet fuel [Fig. 4(B)], and stick fuel [2.5 cm in length, Fig. 4(C)] samples were placed in a stainless steel vat and calcinated in a
laboratory-scale electric furnace (KBF794N1; Koyo) at 400–900 °C for 15–60 min. The ash of the pellet and stick fuels was powdered in a mixer, and the solubility of silica and the physical composition of rice-husk ash were measured. The solubility of ash in the as-are samples was measured without powdering. Experiments were performed in triplicates, and the corresponding mean values ($p<0.05$) were used for the statistical analyses.

RESULTS AND DISCUSSION

Incineration of pellet and stick fuels

Figure 5 shows the ash content of the two fuels after incineration at 400–900 °C for 15–60 min. Long durations of incineration and high temperature exposure resulted in whiter ash. At 900 °C, the ash appeared whiter than at other temperatures with an increase in the duration of incineration. For pellet fuels, a mixture of white and black ash was obtained. White ash was located on the outer side, whereas black ash was located on the inner side of the pellet when the pellet fuel was burned. Additionally, the ash on the outer and inner sides appeared merged in the photographs. The photographs of the stick fuels clearly indicated that the outer side was white, whereas the inner side was black; however, they appeared black at 900 °C. Figure 6 shows the powdered state of each fuel. Some stick fuels appeared extremely white at high temperatures and long durations of incineration (Fig. 5), but powdered stick fuel was comparatively less white and appeared blacker than the powdered pellet fuels (Fig. 6), implying that only the surface of the stick fuels was white, whereas the ash underneath was black. After 15 min, all samples turned black, and the ash appeared white with an increase in temperature. The color difference was evident in the pellet samples compared with the stick samples.

Effects of incineration temperatures and durations on silica solubility

The trends in
solubility at different compaction temperatures are shown in Fig. 7. The solubility of the pellet fuels varied at 15, 30, and 60 min for Fig. 7(A)–(C), respectively. Furthermore, the solubility of ash at 60 min of incineration showed a radical fluctuation among the incineration temperatures. The solubility of ash was the highest at 60 min among the three durations of incineration, and it decreased to 70% at 800 °C, and finally, to approximately 20% at 900 °C. The sample incinerated for 30 min consistently exhibited higher solubility than that incinerated for 15 min, with almost a similar solubility trend at 900 °C. The sample incinerated for 15 min was very stable in terms of solubility at all temperatures, and remained at nearly 50%. In contrast, in the case of stick fuels, a narrow range of solubility was observed among the samples. Radical fluctuations were not observed in any of the samples. The solubility of the sample after 60 min remained the highest (Fig. 7(C)), but it was almost similar to that of samples incinerated for the other two durations at 900 °C. The most stable sample in terms of solubility was the sample incinerated for 30 min, with its solubility fluctuating between 40% and 50%. The solubility of the pellet and stick fuel samples substantially varied after 60 min. The solubility of the pellet fuel after 60 min of incineration exceeded 80%, which was the highest value at 500 °C, whereas that of the stick fuel reached only 60%, which was the highest value for the stick fuel. A decreasing trend in solubility was observed for stick fuel samples than for pellet fuel samples because the pellet fuel had a larger surface area than stick fuels. The formation of fuel might have led to these results.

Pellet fuel can be easily exposed to air because of its small size; conversely, stick fuel cannot be easily exposed to air because of its comparatively larger size; moreover, it is prepared by compacting a larger mass of rice husks. As shown in Fig. 6, the color of the powdered stick fuel was blacker than that of the powdered pellet fuel, implying that the stick fuel exhibited poor burning performance compared with pellet fuel, leading to increased fixed carbon in the ash, which consequently decreased the solubility. Additionally, the solubility of the as-are unwashed and washed is shown in Fig. 7.
After 15 min of incineration (Fig. 7(A)), the solubility of the stick fuel was the lowest at 400 °C and fluctuated in the range of 40% –45% after 500 °C. In contrast, the solubility of the pellet fuel remained stable throughout the incineration temperature range of 400 –900 °C. However, after 30 min of incineration, an opposite solubility trend was observed for the pellet and stick fuels (Fig. 7(B)). The solubility of the pellet fuel showed a decreasing trend with increasing calcination temperature, whereas that of the stick fuel demonstrated an increasing trend as the temperature increased. However, the solubility of the two fuels was the same at 900 °C. After 60 min of incineration, high solubility was observed at 500 °C for both fuels (Fig. 7(C)). The solubility of the stick fuel fluctuated between 50% and 60%, whereas the pellet fuel showed very high solubility and a drastic decrease at 900 °C. The lowest solubility was observed at 900 °C for both fuels; the solubility of pellet fuel was 24.0%, and silica in the ash was already crystallized, which could be deduced based on the cristobalite peaks in the X-ray diffraction analysis results (Fig. 8(D)).

The solubility trends of the as-are unwashed and washed are shown in Fig. 7. Regardless of the calcination duration, the as-are washed samples showed an increase in solubility at low temperatures and almost stable solubility trends up to 900 °C, whereas the as-are unwashed samples showed a decrease in solubility as the calcination temperature increased. The silica solubility of unwashed samples incinerated at 900 °C for 30 and 60 min was very low, approximately 10% and less than 10%, respectively, and silica of samples incinerated for 30 and 60 min was crystallized (Fig. 8(B) and (C)), whereas that of samples incinerated at 900 °C for 15 min was not crystallized (Fig. 8(A)). The significant differences in solubility trends between the as-are unwashed and washed samples as a function of calcination temperatures can be attributed to the presence of alkali metals, such as potassium, which originates from the fertilizer used during rice production. Potassium decreases the crystallization temperature of silica24)–26). Among the three calcination durations, regardless of the washed and unwashed conditions, the 15-min incinerated samples showed the lowest but most stable solubility. Moreover, the difference in the appearance of
ash between the as-are unwashed and washed samples was evidently distinct in the color of appearance (Fig. 9). Therefore, as-are unwashed samples or raw rice husks are not suitable as fuels, and pellet fuel is not suitable, especially when it is used in a stove, where the generated ash stays for longer durations because the temperature reaches nearly 1,000 °C. Additionally, the ash generated at the bottom of the stove is not removed within an hour. Compact stick fuel may be better than compact pellet fuel; however, the solubility performance of the stick fuel is uncertain if the duration of incineration exceeds 1 hour because the solubility of stick fuel decreases after 800 °C.

As mentioned earlier, the two fuels used in this study were washed with acid before the compaction process. The solubility trends of the two fuels and the as-are washed samples were observed. Pellet fuel showed a similar solubility at 60 min of calcination at 500–800 °C to that of the as-are washed samples (Fig. 7), but the trends between the fuels were not completely similar; conversely, the solubility trends between the stick fuel and as-are washed samples were very similar, although the solubility values were different.

**Physical properties of the fuels** Figure 10 shows the physical properties of rice husk ash after calcination at different calcination temperatures and durations, and the corresponding solubility under each condition. A comparison of the physical properties of the ash of compact rice husks (pellet and stick fuels) and uncompact as-are unwashed and washed samples revealed that fixed carbon mostly remained in the ash of compact rice husks. Additionally, a large proportion of fixed carbon remained in the 60-min calcinated stick fuel sample of the ash. This indicates that highly dense compact fuel might not receive enough heat, especially the interior of the fuel sample, to burn the fixed carbon.

The observations in Figs. 5 and 6 support this discussion because, as mentioned earlier, the superficially white appearance of the stick fuel samples at 600–800 °C for 60 min turned black when the samples were powdered. Conversely, the pellet fuel samples remained white under the same conditions. This implies that the stick fuels beneath the outer layer did not receive enough heat and retained a high proportion of fixed carbon inside the fuel. Compact pellet fuel performed better in terms of heat penetration than stick fuel, and its fixed carbon content was significantly lower than that of the stick fuel after 60 min of calcination. The difference in the surface area supports this discussion. The surface areas of 60 g each of pellet and stick fuels (Fig. 4) were approximately 342 and 100 cm², respectively. Because the surface area of pellet fuel was three times greater than that of stick fuels, pellet fuels burned more effectively than stick fuels.

Figure 10 shows the solubility of the silica in ash. By comparing the content of ash to the solubility value, the status of the silica in the ash can be estimated. The solubility indicates the proportion of soluble silica in the ash. It should be noted that “the ash portion” and “the ash” are different. After calcination, rice husks become ash. The ash is composed of ash, water, volatile solids, and fixed carbon portions. Among these, the ash portion majorly consists of silica and less...
than 2% of oxides, such as Al$_2$O$_3$, CaO, Na$_2$O, K$_2$O, P$_2$O$_5$, and Fe$_2$O$_3$; therefore, the ash portion is approximately equal to silica content (data not shown). The physical state of silica changes with the changes in the temperature and incineration duration. Silica in rice husks is originally amorphous and turns to a crystalline form at high temperatures and long durations of incinerations. The silica in the three samples, as-are unwashed at 900 °C and 30 min, as-are unwashed at 900 °C and 60 min,
Effects of Compaction on Silica Solubility and Physical Properties of Rice Husk Ash for Fertilizer Use

and pellet fuel at 900 °C and 60 min, was crystallized (Fig. 10). However, the silica in other samples was not crystalline (Fig. 10), but the silica in some instances was in the intermediate or insoluble state (Fig. 11). When the percentage of the ash portion and that of silica solubility is the same, we can assume that the entire silica content in the ash was soluble. The 15- and 30-min as-are washed samples followed this trend at approximately all the calcination temperatures. When the percentage of the ash portion was higher than that of silica solubility, the excess ash percentage was considered as “insoluble silica.” For example, in case of 30-min samples of pellet fuel, the ash portion percentage and the solubility percentage were the same at 400 and 500 °C, indicating that all silica in the ash was soluble. Later, the solubility percentages decreased, whereas the percentage of the ash portion remained constant. Finally, the two percentages started differing at ≥ 600 °C. The percentage of the ash portion was higher than that of soluble silica and the corresponding excess percentage of the ash portion was considered “insoluble silica.” At 900 °C, the ash portion was 68.6% and the solubility was approximately 45.2%; the excess percentage of approximately 23.4% was “insoluble silica.” In the case of 60-min pellet fuel sample at 900 °C, the ash portion was approximately 95.2% and the solubility was 24.2%; the excess percentage of the ash portion was 71.0%. In this case, silica was crystallized, as determined using the XRD analysis; 71.0% of ash consisted of crystalline and insoluble silica although their percentages were not distinct based on the analysis conducted in this study. As crystalline silica and insoluble silica are not suitable for silica fertilizer, the percentage of ash equal to that of solubility is ideal.

The use of the two types of fuels discussed in this study mainly targeted residential stoves or small commercial heaters. The ash generated from burning rice husks in stoves and heaters is usually exposed to high temperatures and remains for long durations in stoves and heaters. Therefore, stick fuel is better than pellet fuel because the solubility trends of stick fuel as a function of calcination temperatures were more stable than those of pellet fuel, and the gap between the ash portion and solubility was stable with minimum fluctuations in the solubility trend (Fig. 10). In the case where pellet fuels were used, the silica in pellet fuels was crystallized and turned into a carcinogenic substance because pellet fuels were more influenced by temperature and incineration duration than stick fuels. Thus, the ash of these fuels will remain for a long duration, for at least a few hours, and will be exposed to high temperatures (more than 900 °C) in stoves and heaters. According to the results, stick fuels are better than pellet fuels in terms of safety to human health and the use of ash as a fertilizer. Furthermore, on comparing the compact fuels with the native form (as-are) of fuel, the presence or absence of washing step strongly influenced the solubility. Rice husks should be washed for use as a fuel in residential stoves and small commercial heaters. The two types of rice husk fuels used in this study were washed. The solubility would substantially decrease, unlike the solubility data in Fig. 10, if the fuels were synthesized from as-are unwashed samples. Under washed conditions, the use of as-are (the native form of rice husks) as a fuel is better than that of compact fuels in terms of ash use as a fertilizer; however, the selection of rice husks as a fuel must be carefully determined because the native form of rice husks has a high disadvantage during collection and hauling1).

Pellet fuels were made from rice husks alone and with wheat. The mixed pellet fuel was superior in terms of higher heat values and less residue according to Ríos-Badrán et al. (2020)16. The reduction in residue (i.e. ash) reached 11–13%, which represents a very attractive advantage for rice husk heat recovery. However, even the 10% reduced
residue is higher than the residue from burning wood (less than 1% of the pre-burned weight). Therefore, finding a way to recycle this residue is critical for rice husk recycling. Among many alternative uses for rice husk silica\(^2\)-\(^11\), its use as silica fertilizer is a promising recycling option that has been proposed by local rice producers in Japan.

**CONCLUSIONS**

Stick fuel was better than pellet fuel because the solubility trends of stick fuel as a function of calcination temperatures were more stable than those of pellet fuel. Stick fuels are better than pellet fuels in terms of safety and the use of its ash as a fertilizer. Rice husks should be washed for use as fuel in residential stoves and small commercial heaters. Stick fuels are the preferred type of rice husk ash for use as fertilizers and for use in residential stoves and small commercial heaters. However, the native form of rice husks was better than the compact stick fuels under washed conditions. Therefore, the selection of the type of rice husk fuel should be based on the application of the fuels.

**REFERENCES**

1) Sekifuji R., Le Van C., Tateda M., and Takimoto H.: Sustainability of a rice husk recycling scheme. Int. J. Recycl. Org. Waste Agric., 9, 411-421 (2020) https://doi.org/10.30486/IJROWA.2020.1900077.1074.

2) Sheng C. and Azevedo J. L.: Estimating the higher heating value of biomass fuels from basic analysis data. Biomass Bioenergy, 28, 499-507 (2005). https://doi.org/10.1016/j.biombioe.2004.11.008.

3) Yadav K., Tyagi M., Kumari S., and Jagadevan S.: Influence of process parameters on optimization of biochar fuel characteristics derived from rice husk: a promising alternative solid fuel. BioEnergy Res., 12, 1052–1065 (2019). https://doi.org/10.1007/s12155-019-10027-4.

4) Tateda M.: Production and effectiveness of amorphous silica fertilizer from rice husks under a sustainable local energy system. J. Sci. Res. Rep., 9, 3 (2016) https://doi.org/10.9734/JSRR/2016/21825.

5) Zinla D., Gbah P., Kofi P. M., and Koua B.K.: Characterization of rice, coffee and cocoa crops residues as fuel of thermal power plant in Côte d'Ivoire. Fuel, 283, 119250 (2021). https://doi.org/10.1016/j.fuel.2020.119250.

6) Aslam U., Ramzan N., Aslam Z., Iqbal T., Sharif S., Hasan S.W., and Malik A.: Enhancement of fuel characteristics of rice husk via torrefaction process. Waste Manag. Res., 37, 737–745 (2019). https://doi.org/10.1177/0734242X19836820.

7) Singh R. I., Mohapatra S. K., and Gangacharyulu D.: Studies in an atmospheric bubbling fluidized-bed combustor of 10 MW power plant based on rice husk. Energy Convers. Manag., 49, 3086–3103 (2008). https://doi.org/10.1016/j.enconman.2008.06.011.

8) Yoon S. J., Son Y. I., Kim Y. K., and Lee J. G.: Gasification and power generation characteristics of rice husk and rice husk pellet using a downdraft fixed-bed gasifier. Renew. Energy, 42, 163–167 (2011). https://doi.org/10.1016/j.renene.2011.08.028.

9) Unrean P., Fui B. C., Rianawati E., and Acda M.: Comparative techno-economic assessment and environmental impacts of rice husk-to-fuel conversion technologies. Energy, 151, 581–593 (2018). https://doi.org/10.1016/j.energy.2018.03.112.

10) Lim J. S., Manan Z. A., Alwi S. R., and Hashim H.: A review on utilisation of biomass from rice industry as a source of renewable energy. Renew. Sustain. Energy Rev., 16, 3084–3094 (2012). https://doi.org/10.1016/j.rser.2012.02.051.

11) Quispe I., Navia R., and Kahhat R.: Energy potential from rice husk through direct combustion and fast pyrolysis: a review. Waste Manag., 59, 200–210 (2017). https://doi.org/10.1016/j.wasman.2016.10.001.

12) Sekifuji R. and Tateda M.: Taste evaluation of rice grown in soil treated with commercial silica and recycled rice husk silica. J. Sci. Res. Rep., 17, 1–9 (2017). https://doi.org/10.9734/JSRR/2017/38828.

13) Sekifuji R. and Tateda M.: Study of the feasibility of a rice husk recycling scheme in Japan to produce silica fertilizer for rice plants. Sustain. Environ. Res., 29, 1–9 (2019). https://doi.org/10.1186/s42834-019-0011-x.
14) **Pode R.**: Potential applications of rice husk ash waste from rice husk biomass power plant. Renew. Sustain. Energy Rev., 53, 1468–1485 (2016). https://doi.org/10.1016/j.rser.2015.09.051.

15) **Tateda M., Sekifuji R., Yasui M., and Yamazaki A.**: A proposal for measuring solubility of the silica in rice husk ash. J. Sci. Res. Rep., 11, 1 (2016).

16) **Ríos-Badrán I. M., Lázaro-Ocampo I. García-Trejo J. F., Santos-Cruz J., Gutiérrez-Antonio C.**: Production and characterization of fuel pellets from rice husk and wheat straw. Renew. Energy, 145, 500–507 (2020). https://doi.org/10.1016/j.renene.2019.06.048.

17) **Holt P. F. and King D. T.**: Solubility of silica. Nature, 175, 514–515 (1955).

18) **Lanning F. C., Ponnaiya B. W., and Crumpton C. F.**: The chemical nature of silica in plants. Plant Physiol., 33, 339–343 (1958). https://doi.org/10.1104/pp.33.5.339.

19) **Lanning F. C.**: Plant constituents, silicon in rice. J. Agric. Food Chem., 11, 435–437 (1963).

20) **Lenher V. an, Merril H. B.**: The solubility of silica. J. Am. Chem. Soc., 39, 2630–2638 (1917). https://doi.org/10.1021/ja02257a013.

21) **FAMIC (Food and Agricultural Materials Inspection Center)**: Standard Method for Fertilizer Analysis (2015) (in Japanese). http://www.famic.go.jp/ffis/fert/obj/shikenho_2016_3.pdf#page=1. (Accessed 30 January 2021).

22) **Sekifuji R., Le V. C., Tateda M., and Takimoto H.**: Solubility and physical composition of rice husk ash silica as a function of calcination temperature and duration. Int J. Recycl. Org. Waste Agric., 10, 19–27 (2021). https://doi.org/10.30486/IJROWA.2021.1899156.1069.

23) **JIS (Japan Industrial Standard)**: Coal and coke – Methods for proximate analysis (2004) (in Japanese). https://kikakurui.com/m/M8812-2006-01.html. Accessed 29 March 2020.

24) **Nakata Y., Suzuki M., Okutani T., and Kikuchi M., and Akiyama T.**: Preparation and properties of SiO₂ from rice hulls. J. Ceram. Soc. Jpn., 97, 842–849 (1989). https://doi.org/10.2109/jcersj.97.842.

25) **Real C., Alcala M. D., and Criado J. M.**: Preparation of silica from rice husks. J. Am. Ceram. Soc., 79, 2012–2016 (1996). https://doi.org/10.1111/j.1151-2916.1996.tb08931.x.

26) **Shinohara Y. and Kohyama N.**: Quantitative analysis of tridymite and cristobalite crystallized in rice husk ash by heating. Ind. Health, 42, 277–285 (2004). https://doi.org/10.2486/indhealth.42.277.

27) **Tateda M., Sekifuji R., Yasui M., and Yamazaki A.**: Case study: Technical consideration to optimize rice husk burning in a boiler to retain a high solubility of the silica in rice husk ash. J. Sci. Res. Rep., 11, 1–11 (2016). https://doi.org/10.9734/JJSRR/2016/27902. (Submitted 2021. 6. 14) (Accepted 2021. 7. 16)
