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Hot water washing of rice husk for ash removal: The effect of washing temperature, washing time and particle size

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ABSTRACT

Rice husk is a carbon neutral and renewable source of energy and a biomaterial precursor, abundantly available at no cost. High ash content of rice husk is problematic for both applications and thus, reduction of ash constituents can be seen as a viable and sustainable solution. The effect of temperature, particle size and washing time on total ash removal percentage as well as elemental removal was evaluated. The ash leaching behavior was analyzed by fitting to a pseudo-second order kinetic model. 88–95% of K_2O, 60–83% of P_2O_5, 38–54% of Fe_2O_3, 34–62% of Al_2O_3, 30–46% TiO_2 and 19–24% of SiO_2 has removed through water washing. According to the (K + Na)/(Ca + Mg) and (Si + K + P)/(Ca + Mg) molar ratios, ash melting tendency is expected to reduce significantly during combustion of washed rice husk. Increase of Ca and Mg in washed rice husk due to use of city water is beneficial for the ash chemistry. Washing temperature of 50 °C and 15 min of washing time with 1–1.4 mm particle size is recommended for washing pretreatment of rice husk.

1. Introduction

With an increasing demand for sustainability, biomass have become a major carbon neutral energy source as well as a source of biomaterials. It has been used for decades as an energy source in domestic purposes and some industrial purposes. Recently, usage of agricultural wastes as a fuel source and a precursor for biomaterials have increased significantly.

Rice is one of the world's most grown crops. Followed by India and Indonesia, China is the world leader in rice production. Rice is presently the world's third most commonly produced crop after sugar cane and maize and the production is just under 700 million tons per year. Rice husk is the outer shell of the paddy grain, which is not edible. Rice husk is normally 20–22% of the total weight of milled paddy by weight [1]. Thus, every year significant amount of rice husk is produced in rice mills.

Combustion, pyrolysis, gasification, anaerobic digestion and fermentation are some energy applications of rice husk where combustion is the most common application [2,3]. However, a major operational problem of using agricultural wastes as fuel in boilers and furnaces are slagging, fouling, corrosion and agglomeration [4]. This is due to ash content and other inorganic constituents contain in agricultural residuals [5]. Potassium, calcium, silicon, sulfur and magnesium contain in agricultural residuals can cause depositions in combustion chambers. This will reduce the heat transfer efficiencies requiring frequent maintenance and therefore reduce the overall operational efficiency. The ash deposited on surface is hard to remove and those alkali metals account for corrosion of metal surfaces. Further, biomass, which contains high amount of ash susceptible for incomplete combustion and lead to many emissions which are harmful for health and environment. During the combustion of biomass, which contains elements such as sulfur, nitrogen, chlorine and heavy metals can create hazardous pollutants [6]. Thus, it shows the importance of pretreatment of biomass before combustion to maintain the quality and lifetime of boilers and furnaces.

Other possibilities to utilize rice husks include production of bio composites as building materials or biochar as a soil amendment [7]. Rice husk is also used as an adsorbent either in the raw form or by producing activated carbon [8,9]. In addition, new applications such as catalytic supports, battery electrodes, capacitors, and gas storage are also gaining interest in the recent past [10]. However, due to the high ash content in rice husk (around 20%) pretreatment is required for effective utilization of rice husk. For example, high ash content results in a low specific surface area in the produced activated carbon, which is a limitation of its effective application.

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Many researchers have experimented washing techniques as a pretreatment method for many different types of biomass. These include biomass such as different types of wood biomass, rice husk, rice straw, corn stalk, corn husk, etc. [1,4–6,11–21]. These results have shown that the washing techniques play a considerable role as a pre-treatment technique in removing ash. Agricultural waste consists with the soluble ash-forming matter dominated by potassium, chlorine, and phosphorus, even though it contains some silicon as well [22].

Hot Water leaching is a simple method that removes most of the water-soluble ions of potassium, sodium, calcium, magnesium, chlorine, and sulfur. This could yield significant improvement in ash fusion temperatures, thus reducing the fouling and slagging possibilities [23]. The impact of washing is minimal on Higher Heating Value [24]. Washing with various washing techniques including spraying and soaking of tap water and distilled water as well as the effect of natural rain-washing have been studied for ash fusibility of rice straw and wheat straw. Rain washing can effectively remove potassium and chlorine and subsequently, ash fusion temperature is increased [25]. However, the commercial implementation of rain washing is difficult since it depends on rainfall intensity and distribution, agronomic practices as well as field-specific factors [26]. Further, high loss of organic material due to bio deterioration is a major drawback in this system [6].

The efficiency of washing basically depends on the biomass type, particle size, water-to-biomass ratio, washing time and temperature. A positive effect on ash removal is reported with increasing temperature for wheat straw, rice straw, corn stalk, cotton stalk, candlenut wood and rice hull [5], greenhouse crop residue [13], Empty fruit bunch and Palm kernel shell [20], *Arundo donax* [14]. However, temperature is not very critical within the range 20 °C and 40 °C [15] and no substantial improvement from 50 °C to 70 °C also [27]. The positive effect of high temperatures is mostly due to removal of silica, and highly soluble inorganics, K and Cl are removed in high percentages at all water temperatures [5]. Studies on Eucalyptus [18], Empty fruit bunch [20] and Cassava stems [15] have suggested that washing time less than 5 min is enough to reach equilibrium. However, washing time around 30 min is required when only mild shaking is used [27]. Small particle size has reportedly removed more ash [14], however, very small particle size tends to remove organics as well [15]. According to a study on roadside grass, Miscanthus, wheat straw and spruce bark, the washing efficiency improves fast with the increase of the water-to-biomass ratio, although above a ratio of 10 the improvement is rather slower [27].

The gap between the findings and industrial issues are the high cost for the washing, high requirement of water, space, washing time and the energy requirement for drying the washed samples. Thus, it is important to determine how the washing technique can be used for the purpose of ash removal and how the removal efficiency vary with the process parameters. Hot water washing of rice husk is limited to a few studies [5,28] and the removal of Potassium is highlighted, though only the effect of washing temperature has been studied [5]. Through this research, the ash leaching behavior will be analyzed by fitting to a pseudo-second order kinetic model. Further, the effect of temperature, particle size and washing time on ash removal percentage as well as elemental removal will be evaluated for rice husk. In addition, several molar ratios will be applied to evaluate the ash melting tendency of washed rice husk. The study allows to determine the options and economic conditions that can be used in the industry in order to remove ash from rice husk before application.

2. Methodology

2.1. Sample preparation and parameter identification

Rice husk, which was not encountered rain was selected in order to conduct the experiments. This was then sieved into three discrete size ranges, 1–1.4 mm, 1.4–2.8 mm, 2.8–5.6 mm 5.6 mm was the largest particle size that was encountered during sieving. Then, the range is determined by selecting half of the upper limit value until 1 mm. Four samples of 10 g from each size range were weighed to prepare a total of 12 samples.

In this study, a high water to biomass ratio of 80 was selected to minimize the influence of the parameter. City water supply was used as the water source to leach out the mineral constituents. 40 °C, 50 °C, 65 °C and 75 °C selected as the washing temperature for Rice husk. 40 °C and 50 °C selected as the lower temperature and 65 °C and 75 °C has been selected as the higher-level temperature.

2.2. Washing experiments

Four beakers were filled with 800 ml of city water and the temperature of the water bath was fixed. After the water in the beakers achieve the desired temperature, 10 g samples from three sizes were added to three separate beakers while keeping the remaining beaker as a reference sample. The conductivity change in water was measured with reference to reference sample to understand the behavior of leaching of constituents. At every 5 min the suspension was well stirred, and conductivity was measured for 2 h.

At the end of the 2 h the leachate was drained from the beaker and the washed rice husk samples were left to dry in ambient air for one day. Subsequently the samples were dried at 105 °C in the oven for 3 h to remove the remaining free moisture.

2.3. Analytical methods

Determination of ash quantity in washed rice husk samples was carried out according to the ASTM standard test method for ash in biomass (E1755 − 01). To improve the sensitivity in the determination of ash, the sample size was selected as 3 g. The ash composition Fe, Ca, Mg, Na, K, Al, Ti and P of raw and washed rice husk were analyzed by using ICP-OES (Inductively coupled plasma - optical emission spectrometry). The true silicon content in rice husk ash was not reflected in ICP-OES results and calculated by taking the balance and confirmed by XRF (X-ray Florescent) analysis with the exposure time of 300 ms.

2.4. Kinetic model

The experimental data were fitted to the pseudo-second order kinetic model as given in the Equation (1) in derivative form. Here k is the second order leaching rate constant and C_s is the saturated leaching capacity. Equation (2) is obtained by integrating Equation (1) and by rearranging Equation (2), the linear form of Equation (1) is obtained as shown in Equation (3). By fitting the data into t/C_t vs t, k is calculated from the intercept and C_t is obtained from the slope. The initial leaching rate h is expressed by Equation (4) [29].

$$\frac{dC_t}{dt} = k(C_s - C_t)^2 \qquad \text{Equation 1}$$

$$C_t = \frac{kC_s^2 t}{1 + kC_s t} \qquad \text{Equation 2}$$

$$\frac{t}{C_t} = \frac{t}{C_s} + \frac{1}{kC_s^2} \qquad \text{Equation 3}$$

$$h = kC_s^2 \qquad \text{Equation 4}$$

2.5. Molar ratios

The traditionally used indices to predict the deposits of coal ash display mixed results when applied to biomass fuels and hence not suitable for analyzing the deposition tendency of biomass combustion [30]. The molar ratios (K + Na)/(Ca + Mg) and (K + Na + Ca + Mg)/(Si + P) along with Si/P has previously been introduced [31] and used to predict the probability of ash slagging and formation of fine particulate emissions during biomass combustion [13,15]. Further, prediction of the ash-melting temperature with the molar (Si + K + P)/(Ca + Mg) ratio has been previously proposed [32]. Even though these molar ratios cannot use solely to predict the ash chemical behavior of biomass fuels, they were applied to understand the tendency.

3. Results and discussion

In this study, four areas of ash removal were studied. Firstly, the ash leaching behavior of rice husk with time and particle size with respect to conductivity was analyzed by fitting to a pseudo-second order kinetic model. Secondly, the ash removal percentage was calculated by determining the ash content of raw and washed rice husk. Next, the removal of different inorganic compounds were quantified and finally, ash melting tendency was discussed by using several molar ratios.

3.1. Leaching behavior

The leaching behavior of inorganic matter from rice husk can be studied in terms of the conductivity of the leachate. According to the data obtained during the experiments, initially there is a rapid increment in conductivity followed by a slow saturation period. This is reflected in Fig. 1 with a sharp increase of the conductivity within the first 15 min of the 2 h leaching period. The initial rapid leaching can be attributed to the driving force of fresh water and the surface of the solid following gradual uptake [17].

The results were analyzed using a second-order leaching model. The values of the fitted parameters of the pseudo-second order kinetic model are summarized in Table 1. It can be seen that the R^2 of the data fitting is all above 0.99. Therefore, the pseudo-second order kinetic model is well suitable to describe the leaching kinetics of inorganic species from rice husk.

The data in Table 1 shows that small particle size generally increases the leaching rate constant and the initial leaching rate whereas saturated leaching capacity is increased marginally. However, the effect of smallest particle size on the leaching rate constant and the initial leaching rate is substantial, even though only a marginal difference can be seen between large and medium particle sizes. A higher surface area increases the exposure of the inorganic species to the leaching medium and promotes the leaching. Large particle size increases the time required for wetting process as well as the diffusion path length [29].

Higher temperature increases the leaching rate constant and the initial leaching rate up to 50 °C and drops thereafter. However, the increase of the leaching rate constant is not very significant compared to the increase of the initial leaching rate. The saturated leaching capacity continuously increases with the temperature. At higher temperatures (65 °C and 75 °C), the initial leaching rate is slower and a longer leaching time is needed to reach equilibrium.

Although both temperature and particle size shows almost similar effect on the initial leaching rate, the particle size shows a substantial impact on the leaching rate constant and the temperature is more influential on the saturated leaching capacity.

Washing temperature of 50 °C and 15 min of washing time with 1–1.4 mm particle size can be recommended for washing pretreatment of rice husk. Further, 88% of the saturated leaching capacity can be achieved within first 15 min and therefore longer washing time is not necessary. Even though the saturated leaching capacity is higher with high temperatures, the benefit is offset by the longer leaching time, cost of water heating, water evaporation loss as well as the susceptibility of thermal decomposition of organic compounds in rice husk. When it comes to industrial applications, hot water washing can be accomplished for example by using cooling water, leaving the condenser at a temperature of 40–50 °C. As the washed biomass may contain high content of moisture, it is recommended to air dry in the field, or dry using hot stack gas.

3.2. Ash removal

The percentage removal of the ash content of washed rice husk samples were evaluated compared to the ash content of the raw rice husk which is 19.95%. As shown in Fig. 2, both particle size and the temperature increase the percentage ash removal. As expected, the particle size of rice husk has higher impact compared to the temperature effect. This is reflected in the kinetic data (Table 1) where small particle size is significantly affecting the initial leaching rate as well as the leaching rate constant compared to the temperature effect. Even at the lowest temperature, high removal of ash can be achieved by reducing the particle size. This value is higher than that of the largest particle size washed at 75 °C.

An optimum value cannot be found for removal of ash, however, an increasing trend was observed with increasing temperature and decreasing particle size. Within the studied temperature range, maximum up to 25% of ash was removed using hot water washing for 2 h of washing time. Even though the best pretreatment efficiency was obtained with smaller particle size and high temperature washing combination, for industrial purposes this would not be an economical solution. Since the effect of temperature on small particle sizes is less compared to large particle sizes, the medium temperature and small particle size recommended based on the leaching behavior is acceptable. For the recommended washing temperature of 50 °C and 1–1.4 mm particle size, 23.5% ash was removed within 2 h of washing time. If 15 min washing time is applied, around 20.6% ash removal can be expected.

3.3. Removal of inorganic compounds

The percentage removal of the inorganic compounds of washed rice husk samples were evaluated compared to that of the raw rice husk. Considering the reported chemical compositions of rice husk ash [5], it was assumed that all compounds in the ash sample are in their stable oxide form.

According to Fig. 3 and Fig. 4, the main inorganic constituents in rice husk are SiO_2, K_2O, P_2O_5 and CaO. Among them, K_2O and P_2O_5 content is significantly reduced by washing treatment. While 60–83% of P_2O_5 has removed through water washing, K_2O shows the highest removal of 88–95%. Previous studies have also reported

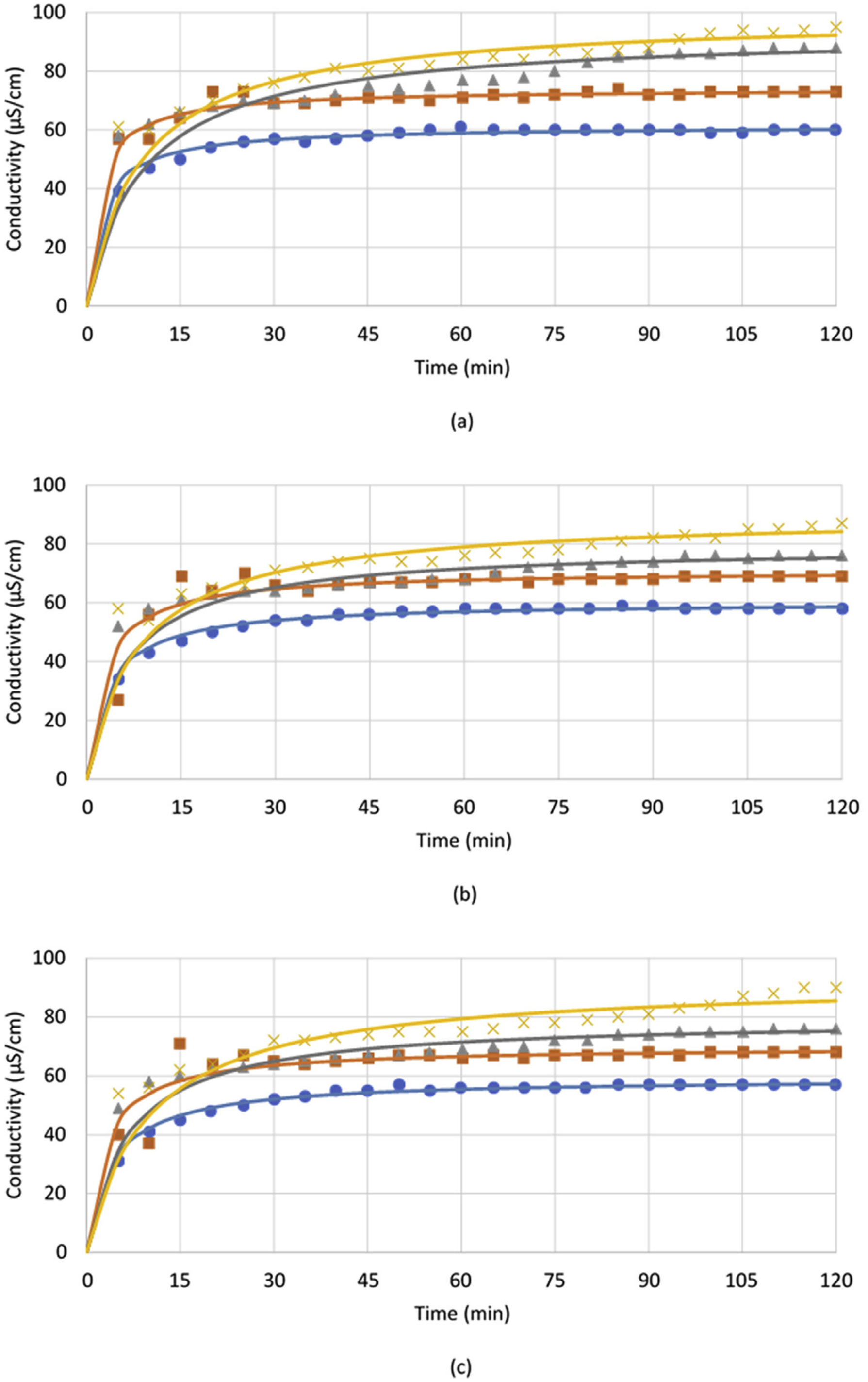

Fig. 1. Variation of conductivity with time for different particle sizes and washing temperatures (a) 1–1.4 mm (b) 1.4–2.8 mm (c) 2.8–5.6 mm Kinetic model: solid lines Experimental data ● 40 °C ■ 50 °C ▲ 65 °C × 75 °C.

similar results with 80–95% of the potassium and 53% of the phosphorus removal [5,28]. A considerable removal of Fe_2O_3 and Al_2O_3 can be observed where 38–54% Fe_2O_3 and 34–62% Al_2O_3 has removed. Even though the presence if TiO_2 is small, water washing could be able to remove a significant fraction of 30–46%.

The SiO_2 content in Rice hush ash is high as 96% of the total weight. Around 19–24% of SiO_2 is removed by water washing. This is due to the silicon structure of rice husk does not readily dissolve in water, but, an acid or base treatment can weaken the silicon structure, which helps to dissolve more [1,33]. However, SiO_2 alone is not a problem because it has a melting point above 1400 °C [1]. The presence of alkali metals is the key factor for fouling during combustion [25]. Reduction of K_2O by 95% is a great achievement because during combustion, K is more susceptible to form KCl, KOH and K_2CO_3. These compounds are subjected to secondary reactions with SiO_2 and melt at lower temperature (about 800 °C). This results indirect effect on fouling and corrosion properties of fly ash [22].

Table 1
Kinetic parameters obtained with different particle sizes and washing temperatures.

Particle size: 1–1.4 mm				
Parameter	40 °C	50 °C	65 °C	75 °C
k (cm μS^{-1} s^{-1})	10.93×10^{-5}	11.13×10^{-5}	1.94×10^{-5}	1.9×10^{-5}
h (μS cm^{-1} s^{-1})	0.411	0.611	0.169	0.186
C_s (μS cm^{-1})	61.35	74.07	93.46	99.01
R^2	0.9994	0.9994	0.9929	0.995
Particle size: 1.4–2.8 mm				
Parameter	40 °C	50 °C	65 °C	75 °C
k (cm μS^{-1} s^{-1})	7.95×10^{-5}	8.15×10^{-5}	3.25×10^{-5}	2.19×10^{-5}
h (μS cm^{-1} s^{-1})	0.289	0.41	0.205	0.178
C_s (μS cm^{-1})	60.24	70.92	79.37	90.09
R^2	0.9995	0.9984	0.9971	0.9953
Particle size: 2.8–5.6 mm				
Parameter	40 °C	50 °C	65 °C	75 °C
k (cm μS^{-1} s^{-1})	6.89×10^{-5}	7.97×10^{-5}	3.16×10^{-5}	1.81×10^{-5}
h (μS cm^{-1} s^{-1})	0.241	0.39	0.199	0.155
C_s (μS cm^{-1})	59.17	69.93	79.37	92.59
R^2	0.9997	0.998	0.9974	0.9901

k - second order leaching rate constant, *h* - initial leaching rate, C_s - saturated leaching capacity.

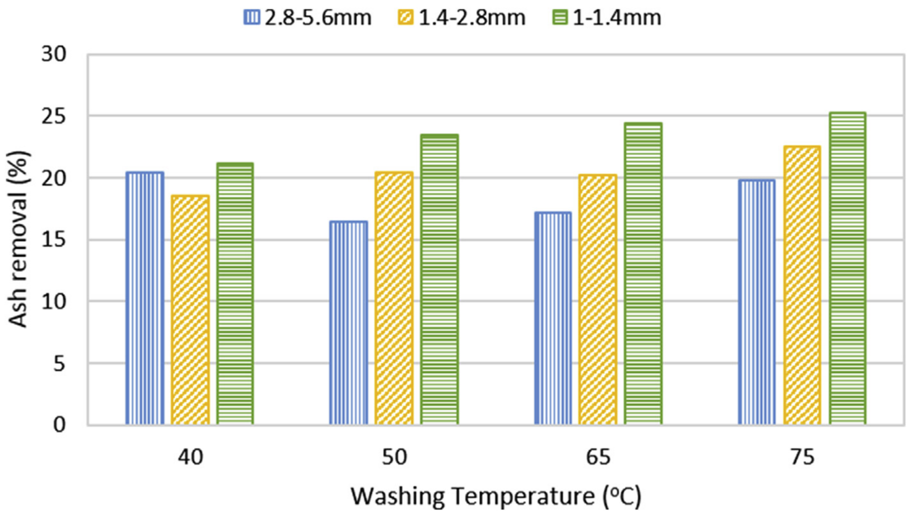

Fig. 2. The effect of particle size and washing temperature on percentage removal of ash.

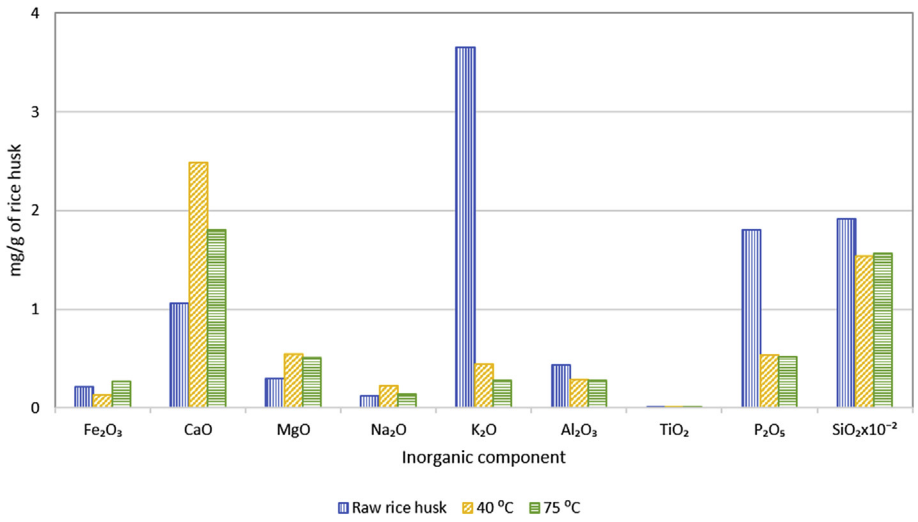

Fig. 3. Effect of temperature on the removal of inorganic components for 2.8–5.6 mm particle size.

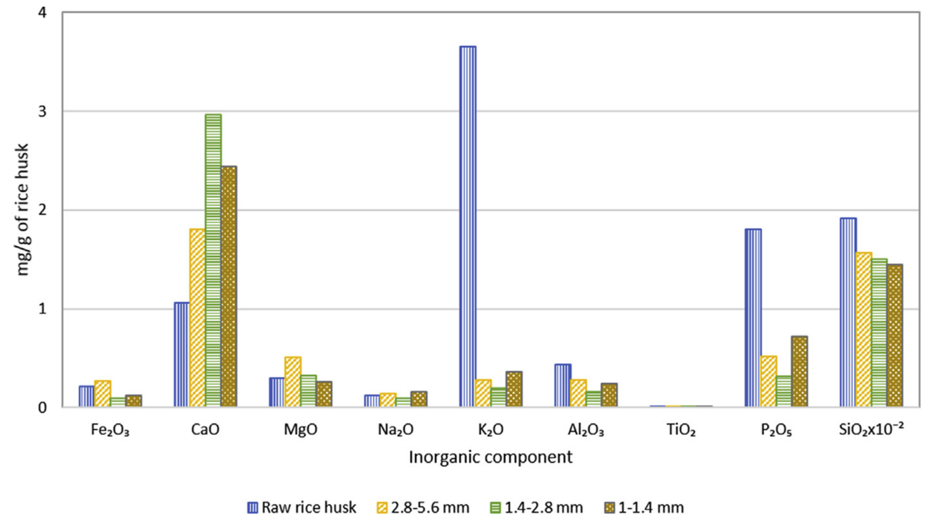

Fig. 4. Effect of particle size on the removal of inorganic components at 75 °C.

CaO, MgO and Na_2O concentrations have increased in washed samples. A previous study on washing sorghum biomass using city water supply has reported a similar behavior [21]. This is likely due to use of normal city water instead of distilled or demineralized water. In industrial scale pretreatment, use of city water is expected because demineralized water is expensive. Thus, it is more susceptible to increase in CaO, MgO and Na_2O concentrations in rice husk based on the source of water. However Ca is an inert compound during combustion and do not influence the flue gas chemistry. This also acts as a base to neutralize the acid compounds produced due to combustion of sulfur materials [22].

A significant reduction of components can be achieved by washing, however, changing the temperature and particle size has little effect, but the removal of components trend continues with increasing temperature and reducing particle size.

3.4. Ash melting tendency

All the raw and washed rice husk samples had high Si/P molar ratio due to the high Silicon content of rice husk and the ratio was further increased due to a high loss of Phosphorus compared to Silicon during washing. This suggests that the ash transformation reactions are mainly dominated by silicate formation.

When the ratio of $(K + Na + Ca + Mg)/(Si + P)$ is considered, having a surplus of basic components compared to the acidic components play a crucial role in the formation of fine particulate emissions and deposits [31]. In case of rice husk, acidic components are in excess and hence no such issue is expected, even if raw form of rice husk is used. Further reduced tendency was observed due to washing pretreatment.

According to Fig. 5 and Fig. 6, interesting covariation is observed for molar ratios $(K + Na)/(Ca + Mg)$ and $(Si + K + P)/(Ca + Mg)$, the ratios proposed for predicting the slagging tendency. The latter has introduced the Si and P to the numerator considering that they also have a contribution to decrease the sintering temperature. K_2O, P_2O_5 and SiO_2 reportedly show an increasing trend of ash fusion temperature, whereas CaO shows the opposite trend [34]. Adding Na to the numerator has no any significant change in the molar ratio due to low Na content of rice husk compared to other constituents in the numerator.

According to Figs. 5 and 6, in the raw rice husk, $(K + Na)$ is in surplus compared to $(Ca + Mg)$, with a molar ratio around 3. There is a risk of molten K-silicate particles to aggregate, forming larger droplets that may initiate slag formation. After washing pretreatment, irrespective of the washing temperature and particle size, the molar ratio becomes less than 0.3 where $(Ca + Mg)$ is in excess. This is due to effective removal of alkali metals; K and Na as well as the increase of alkaline earth metals; specially Ca due to use of city water for washing. According to the literature, CaO and MgO will dissolve into the K-silicate melt and some K be driven off, increasing the melting temperatures reducing the tendency for slag formation [35]. Therefore, combustion of washed rice husk may

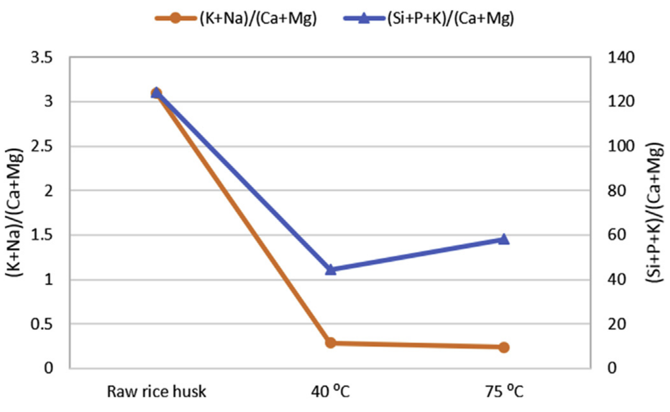

Fig. 5. Effect of temperature on the molar ratios for 2.8–5.6 mm particle size.

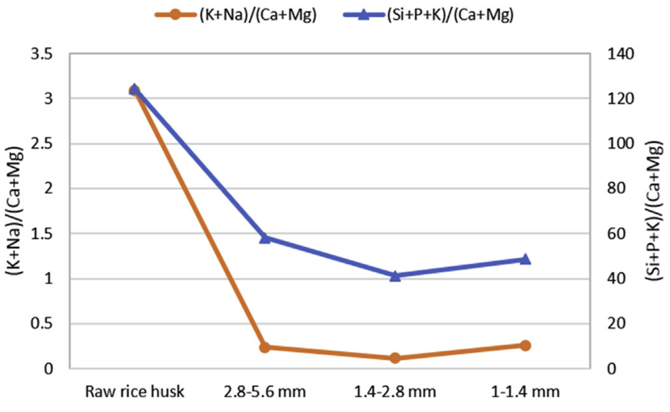

Fig. 6. Effect of particle size on the molar ratios at 75 °C.

produce bottom ash consisting of silicates rich in Ca and Mg. Use of city water for washing is somewhat advantageous in this regard. A similar trend was observed for $(Si + K + P)/(Ca + Mg)$ ratio as well. A linear trend of decreasing melting temperatures with an increasing value of $(Si + K + P)/(Ca + Mg)$ molar ratio has been reported for fuels including Phosphorus rich fuels [32]. Obviously, high ash melting temperature can be expected during washed rice husk combustion.

4. Conclusion

Hot water washing of rice husk as a pretreatment method is an effective method to remove inorganic ash forming compounds. According to the study, the pseudo-second order kinetic model is suitable to describe the leaching kinetics of inorganic species from rice husk. The effect of particle size is more influential on the leaching rate and the temperature shows a substantial impact on the leaching capacity. This means that small particles can leach faster while high washing temperatures can leach more. Therefore, grinding larger particles can reduce the size of the washing tank while heating the washing water can improve the quality of washed rice husk.

Washing temperature of 50 °C and 15 min of washing time with 1–1.4 mm particle size can be recommended for washing pretreatment of rice husk. For the recommended washing temperature and particle size, around 20.6% ash removal can be expected within 15 min of washing time. 88–95% of K_2O, 60–83% of P_2O_5, 38–54% of Fe_2O_3, 34–62% of Al_2O_3, 30–46% TiO_2 and 19–24% of SiO_2 has removed through water washing. Therefore, hot water washing is effective for removing Potassium and Phosphorus mainly.

Considering the molar ratios $(K + Na)/(Ca + Mg)$ and $(Si + K + P)/(Ca + Mg)$, ash melting tendency could be expected to reduce significantly during combustion of washed rice husk. Further, increase of Calcium and Magnesium in washed rice husk due to use of city water is beneficial in this regard.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Yasas Wishvajith Bandara: Conceptualization, Methodology, Investigation, Writing - original draft. **Pringa Gamage:** Conceptualization, Methodology, Investigation, Writing - original draft. **Duleeka Sandamali Gunarathne:** Supervision, Writing - review & editing.

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