Research article

Parametric and kinetic study of washing pretreatment for K and Cl removal from rice husk

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ABSTRACT

Rice husk is an abundant agricultural waste that can be used in combustion applications. The use of agricultural waste in combustion applications is hindered by slagging, fouling, and corrosion due to elements such as K, Cl, and Si. With the aim of removing those problematic elements using water washing, lab-scale experiments were conducted to study the effect of washing parameters; temperature, time, and liquid to solid ratio and to recommend suitable washing conditions. The washing temperature of 65 °C and liquid to solid ratio of 100 was selected as the best process conditions. Within 10 min of washing time, 75% of Cl and 50% of K can be removed. Pseudo-second-order kinetic model is suitable to describe the leaching behavior, and the kinetic parameters were obtained for the leaching of K and Cl. In industrial installations, hot condensate from process use of steam can be recommended as a good source of washing medium.

1. Introduction

Biomass is one of the renewable energy sources which can be categorized into four main types; agricultural crops and waste materials, wood and wood processing waste, animal manure and human sewage, food waste. Rice husk is one of the abundant agricultural waste which is currently underutilized.

Rice is grown in more than a hundred countries worldwide. Annual production in 2019 is around 504 million tons of milled rice and around 452 million tons are produced in Asia, representing 90% of global production [1]. Global rice consumption is increasing with population growth and rice production is also increasing accordingly at a rate of 5 million tons per year within 2009–2019 [1]. It is expected to grow rice production further in the near future and even with the same annual production rate, it is expected to produce 519 million tons of milled rice in 2022. Rice husk is normally 20–22% of the total weight of milled rice by weight [2] which results in a generation of significant waste of around 109 million tons in 2022.

There are three main thermochemical processes used for the conversion of biomass to energy such as combustion, gasification, and pyrolysis. The most common application of agricultural waste is combustion in boilers and furnaces [3]. Ash forming elements of biomass are K, Na, Ca, Mg, Si, Al, Fe, and Ti. High maintenance cost derived from severe sintering, slagging, fouling and corrosion risk should be expected due to the presence of alkali and alkaline earth metals such as Na, K, Ca, Mg, and chlorides in biomass, especially in agricultural waste [4–8].

During the combustion process, K is released and easily combines with other elements [9]. There are three types of primary vapors, K (g), KOH (g), and KCl (g). If the Cl is present, the dominant vapor is KCl (g). Condensed KCl is melting at the furnace temperature at 770 °C. KCl is highly corrosive and the presence in superheater metal surfaces is problematic. Alkali chlorides remain in flue gases that may form particles and make fly ash more corrosive than chloride-free fly ash. Alkali vapors such as KOH, KCl, and K_2CO_3 react with SiO_2 and form alkali silicates [9]. Silicates cause major sintering and fouling problems. Therefore, K, Cl, and Si are the most problematic elements to be avoided.

Water washing is an effective pretreatment method to remove troublesome elements from biomass [10]. There are different water washing techniques such as spraying, flushing, and soaking [11]. Washing biomass in multiple steps has also been studied recently [12]. The water washing technology of biomass can leach a substantial amount of K, Cl, and S [11]. Around 90% of alkali content is in the water-soluble form [13] and water washing pretreatment is an effective method to remove alkali from herbaceous biomass [14].

Some inorganic constituents are insoluble in water, but some are highly soluble in dilute acids such as nitric acid, phosphoric acid,

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hydrochloric acid, and acetic acid. Acid leaching treatment can remove more inorganic constituents compared to water washing [15–17]. Silica is the major constituent of agricultural waste ash and silica content varies 87–97 wt% of ash in rice husk [6]. There are some studies on the removal of silica from rice husk by using sodium hydroxide [2]. However, the cost associated with the reagents and additional neutralization steps as well as the high loss of organic matter limit the use of acid or alkali pretreatment processes. Therefore, water washing is considered as a low-cost, simple, and effective pretreatment technology. Further, washing pretreatment combined with torrefaction is also of interest nowadays [18–22].

The effectiveness of water washing depends on process parameters such as washing temperature, washing time, liquid to solid ratio, and particle size [10,23–25]. When considering the industrial applications, mainly water, energy, space, and duration for the washing pretreatment need to be optimized in order to minimize the pretreatment cost. However, the effect of washing parameters on the washing performance has been studied very rarely for rice husk [11]. We recently studied the effect of washing temperature, washing time, and particle size on rice husk washing performance using tap water [26]. Deionized water on the other hand will result in a higher driving force due to no ions in the washing medium. In industrial installations, hot condensate returned after the process use of steam can be a good source of such washing medium. Further, the leaching kinetics of K and Cl from rice husk has not been studied yet. Therefore, the present study aims to study the effect of washing parameters; temperature, time, and liquid to solid ratio and recommend suitable washing parameters for rice husk washing while obtaining the kinetic parameters for the leaching of K and Cl.

2. Methodology

2.1. Sample preparation and parameter identification

Rice husk that has not encountered any rain washing was selected for the experiments. It has been reported that the effectiveness of water washing depends on process parameters such as washing temperature, washing time, liquid to solid ratio, and particle size [10,23–25]. Only the large size particles (2.8–5.6 mm) were selected for the tests partly due to the majority of rice husk particles being within this range and to ensure reasonable pretreatment conditions for the entire particle size range of rice husk. De-ionized water with electrical conductivity of 1 μS/cm was taken as the washing medium to maintain a better driving force for leaching compared to tap water which contains ions. The effect of three washing parameters; washing temperature in the range of 30–75 °C, liquid to solid (L/S) ratio of 20–100, and washing time up to 90 min were analyzed. Room temperature was kept as the lowest washing temperature and washing temperature above 75 °C was not considered due to high water evaporation losses, cost of water heating, and susceptibility of thermal decomposition of organic matter at higher temperatures. Considering the cost of water and the reactor capacity as well as previous literature study range of 70–80 [11, 22], the maximum L/S ratio of the study was limited to 100. Even though previous studies have used long washing durations spanning from 3-6 h [11, 22], the maximum study washing time was limited to 90 min considering the practicality of production rates in industrial applications and considering limited effect beyond 90 min.

2.2. Washing experiments

To find the best temperature, the L/S ratio of 100 was kept constant at four different temperatures 30 °C, 50 °C, 65 °C, and 75 °C. Except at the 30 °C which was the ambient temperature, the water bath temperature setting was fixed at the desired temperature and a beaker with 600 ml of de-ionized water was kept inside the water bath until the desired temperature is attained. Then 6 g of sample was added to the beaker. At every

five minutes interval, the suspension was stirred with a glass rod and conductivity was measured for 90 min. After 90 min, the beaker was taken out from the water bath and the leachate was filtered out for the composition analysis.

To find the best L/S ratio, five beakers were prepared with the same liquid volume of 600 ml and the solid mass was varied to achieve different L/S ratios 20, 40, 60, 80, and 100. Then the above procedure was followed at the best washing temperature.

To find the leaching kinetics of K and Cl at the best washing condition, the concentration values of K and Cl were obtained at 5, 10, 15, 30, 60, and 90 min by following the above procedure.

2.3. Analytical methods

After each test procedure, filtered leachate samples were analyzed elementwise using Inductively Coupled Plasma Mass Spectroscopy (ICP-MS). A raw rice husk sample was also analyzed to quantify the initial elemental concentration using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). The raw rice husk composition is shown in Table 1.

2.4. Kinetic model

The first-order kinetic model is not suitable for processes having two distinct leaching steps; a relatively fast initial leaching process and a slower residual leaching process [27]. Then the pseudo-second-order kinetic model was selected to describe the leaching behavior of elements by measuring electrical conductivity and elemental compositions.

The experimental data were fitted to the pseudo-second-order kinetic model given in Eq. (1) [25,26,28–30].

$$\frac{dC_{i,t}}{dt} = k(C_{i,s} - C_{i,t})^2 \tag{1}$$

k - Second-order leaching rate constant (g mg^{-1} min^{-1})
$C_{i,s}$ - Equilibrium concentration (mg/g) of species i
$C_{i,t}$ - Concentration (mg/g) of species i at leaching time t

When the above equation is integrated by applying boundary conditions of t = 0 to t and $C_{i,t}$ = 0 to $C_{i,t}$ the Eq. (2) is obtained and it can be arranged to form the linear form as in Eq. (3). The initial leaching rate can be expressed as in Eq. (4).

$$C_{i,t} = \frac{C_{i,s}^2 kt}{1 + C_{i,s}kt} \tag{2}$$

$$\frac{t}{C_{i,t}} = \frac{t}{C_{i,s}} + \frac{1}{kC_{i,s}^2} \tag{3}$$

$$h = kC_{i,s}^2 \tag{4}$$

3. Results and discussion

This study is based on the electrical conductivity values of washed water at fixed time intervals. The electrical conductivity of de-ionized water was measured before adding the sample into the beaker and it was constant (1 μS/cm) irrespective of the temperature. Electrical conductivity is directly proportional to the ionic content of a solution [29]. Further, the removal of elements such as K, Cl, and Si was quantified and the leaching behavior of elements was studied by applying three parameters; washing temperature, L/S ratio, and washing time. Finally, the kinetic parameters of K and Cl removal were determined.

Table 1. Composition of rice husk.

Element	Fe	Ca	Mg	Na	K	Al	Ti	P	Si	Cl
Composition mg/g of biomass	0.149	0.760	0.179	0.094	3.032	0.231	0.002	0.395	89.6	1.075
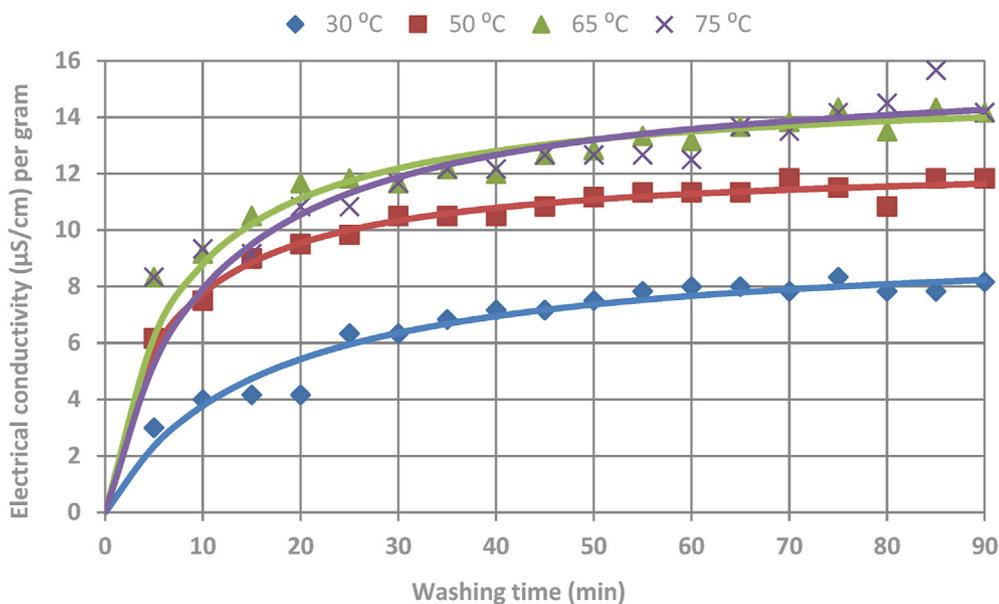
Figure 1. Variation of electrical conductivity with washing time at different temperatures.

3.1. Effect of washing temperature on the leaching behavior

Electrical conductivity was measured at four different temperatures keeping an L/S ratio of 100 and its graphical representation is shown in Figure 1.

Electrical conductivity is increasing with washing time. Within the first five minutes of washing, all the tests achieved similar conductivity values except the experiment at 30 °C. The fraction which is dissolved very fast is not much influenced by the temperature. However, beyond five minutes 65 °C and 75 °C washing temperatures show higher conductivity values compared to that of 50 °C. It has been reported that the secondary slow release is diffusion-limited [31] and therefore, it can be influenced by particle size, water temperature, and other washing parameters. Within the test duration of 90 min, the conductivity values are much similar in the 65 °C and 75 °C cases. Apparently, water-soluble fractions have already been released at a temperature of 65 °C.

The second-order kinetic linearized equation was applied at four different temperatures. Calculated kinetic parameters and corresponding R^2 values are tabulated in Table 2 where k is the overall leaching rate constant, h is the initial leaching rate at time equals zero (at the time starts to leach), C_s is the saturated leaching capacity and R^2 is the goodness of fit. All the R^2 values are above 0.98 and it suggests that the proposed pseudo-second-order kinetic model successfully represents the leaching behavior of elements with respect to different temperatures.

The overall leaching rate constant shows an increasing trend up to 50 °C followed by a drop. The secondary release of inorganic species is

affected by the concentration of the inorganic species in the leachate [31] and at higher temperatures, this could be the reason to observe less overall leaching rate even though the initial leaching rate is high. In our previous studies of washing rice husk with tap water gave the same trend with a maximum at 50 °C [26]. When using tap water, the initial leaching rate was maximum at 50 °C and with deionized water, the initial leaching rate is increasing up to 65 °C and decreasing thereafter. The difference could be due to the higher driving force when no ions in the washing medium in the present case. Therefore, 65 °C was selected as the best washing temperature basically due to the high initial leaching rate which is more important than the overall leaching rate due to the short washing cycles expected. Further, saturated leaching capacity has no significant increase after 65 °C. It reveals that all the water-soluble fractions have been released at 65 °C and further increase of temperature is not useful. A similar study conducted using conductivity measurements has reported increasing initial leaching rate and the overall leaching rate constant within temperature range 40–60 °C [30].

When it comes to industrial applications, hot water washing can be accomplished for example by using a part of condensate returned after the process use of steam. In addition to the heat content, condensate is basically distilled water with no ions in it. Therefore, the use of deionized water (or condensate returns) increases the driving force to remove more alkali metals. The use of tap water (or cooling water returns) on the other hand may reduce the ash melting tendency due to the deposition of stable alkaline earth metals such as Ca in tap water in addition to the removal of alkali metals like K [26]. Finally, the source of water depends on the availability at a low cost. It has been proposed that increased washing temperature can likely serve as a substitute for reducing particle size which offers potential cost saving in milling and reduce the loss of material during washing [32].

Filtered effluents were analyzed elemental wise and significant removal was observed only with K, Cl, and Si. Elemental removal and removal percentages are shown in Figure 2(a) and (b) respectively. Being a refractory, Si removal was always below 1% and was not considered in Figure 2(b).

Table 2. Calculated kinetic parameters at different temperatures.

	30 °C	50 °C	65 °C	75 °C
k (cm μS^{-1}s^{-1})	11.1×10^{-5}	22.2×10^{-5}	15.4×10^{-5}	10.5×10^{-5}
h (μS cm^{-1} s^{-1})	0.010	0.034	0.035	0.026
C_s (μS cm^{-1})	9.66	12.42	15.11	15.85
R^2	0.9813	0.9959	0.9952	0.9836
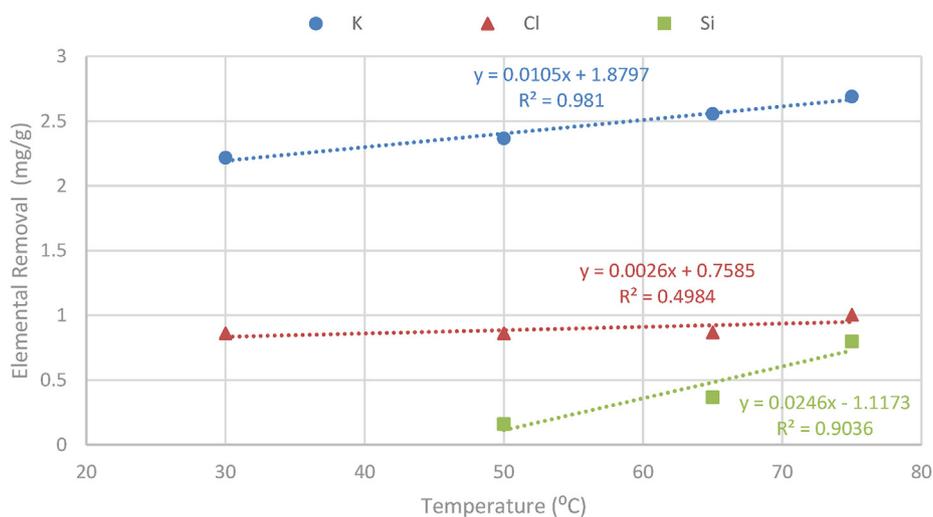
(a)
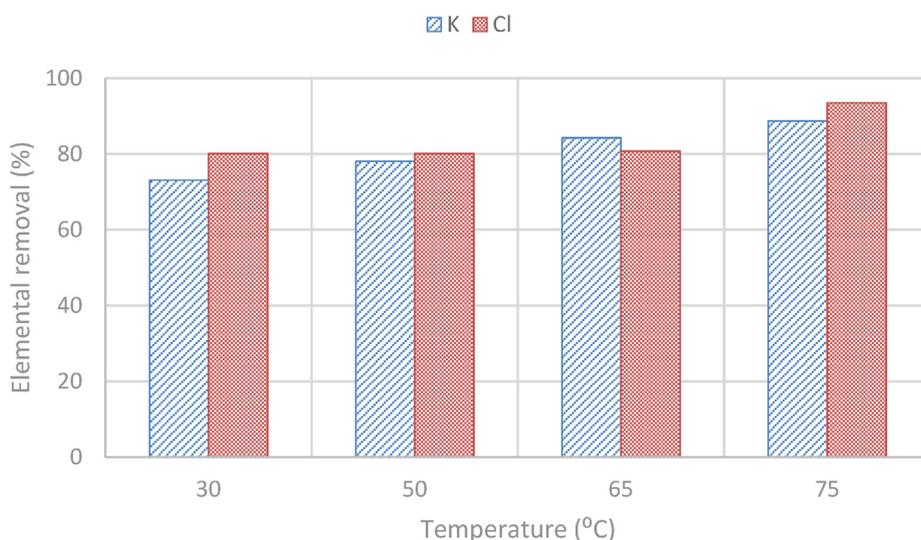
(b)

Figure 2. Variation of elemental removal with washing temperature (a) removal in mg/g (b) removal in %.

According to Figure 2(a), leaching concentrations of elements linearly increase with temperature. Si shows the highest leaching dependency on the temperature and does not leach at 30 °C at all. The water solubility of Si appreciably increases with temperature possibly due to the combined effect of the acidic nature of the leachate as a result of Cl removal and the temperature effect which enhances the removal of refractories. The leaching of K also depends on the temperature, whereas very weak dependency can be observed for Cl. A similar trend has been observed by previous researchers with empty fruit bunches [31]. Cl being extensively water-soluble [33] results in maximum removal despite the temperature. Due to its diffusion-limited secondary release [31], the temperature dependency of K is significant. Temperature dependency of K leaching has been observed by a previous study on rice husk washing conducted at

three temperature levels and an L/S ratio of 80 for a 3 h duration [11]. They have proposed that more K$^+$ ions that form complexes with organic acid anions break away from the biomass matrix when the water temperature is increased. According to Figure 2(b), more than 80% of both K and Cl can be removed by washing at 65 °C. In a previous study of rice husk washing at the temperature range of 30–90 °C for 3 h duration with deionized water; 85% to >90% of K and 68%–85% of Cl have been removed [11]. At 60 °C washing temperature, both K and Cl removal was around 85% which is very similar to the results of the present study. In another study, 95% of K and 54% of Cl has removed after washing with deionized water (L/S of 70) at 60 °C for 6 h [22]. Comparatively less Cl removal has been observed by them which could be due to the different forms of Cl present in the rice husk structure, differences in analytical
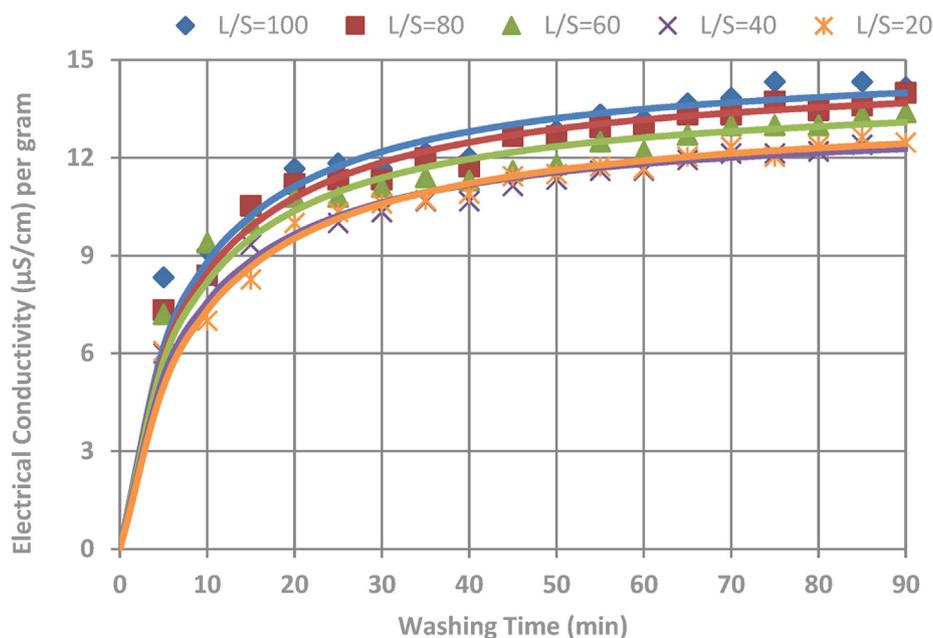
Figure 3. Variation of electrical conductivity with washing time at different L/S ratios.

methods, differences in washing conditions, and so on. However, if the primary requirement of washing treatment is to remove Cl, washing at 30 °C is recommended because 80% Cl removal can be achieved at 30 °C.

3.2. Effect of the L/S ratio on the leaching behavior

Electrical conductivity was measured for five different L/S ratios at 65 °C as shown in Figure 3.

Electrical conductivity is increasing with washing time. Similar to the experiments with varying washing temperatures, within the first five minutes of washing, all the tests achieved similar conductivity values. However, beyond five minutes high L/S ratios show higher conductivity values and the L/S ratio of 100 shows the highest conductivity profile. This is again due to the secondary slow release is the stage that is strongly influenced by washing parameters.

To analyze the leaching behavior in more detail, the pseudo-second-order kinetic model was applied to all the L/S ratio cases. Table 3 summarizes the calculated kinetic parameters and the corresponding R^2 values. All the R^2 values are above 0.99 and it suggests that the proposed second-order kinetic model successfully represents the leaching behavior of elements with respect to different L/S ratios.

The overall leaching rate constant shows an increasing trend up to L/S of 40 followed by a drop. As discussed before, the secondary release of inorganic species is affected by the concentration of the inorganic species in the leachate [31] and this could be the reason to observe less overall leaching rate at higher L/S ratios. The initial leaching rate is continuously increasing with the increase of L/S. This could be a result of less competition between inorganic species leaching into the water due to higher water quantity [31, 34]. Therefore, L/S of 100 was selected as the

best L/S ratio basically due to the high initial leaching rate which is more important than the overall leaching rate due to the short washing cycles expected. Further, the increase of saturated leaching capacity is diminishing towards L/S of 100 because competition between inorganic species leaching into the water becomes weak due to higher water quantity available and further increase of L/S becomes less meaningful.

When increasing the L/S ratio, it needs more water per unit mass of rice husk. In such a case, the cost of water is high and the reactor capacity is also limited. Previous studies have used de-ionized or distilled water to the sample ratio in the range of 70–80 [11, 22]. Considering the economic and scale factors as well as the previous studies, the maximum L/S ratio of 100 was chosen as the best L/S ratio considering the feasibility in industrial applications.

Filtered effluents were analyzed elemental wise and significant removal was observed only with K, Cl, and Si. Elemental removal and removal percentages are shown in Figures 4(a) and (b) respectively. Si removal was always below 1% and was not considered in Figure 4(b).

Figure 4(a) shows that the leaching concentrations of three elements linearly increase with increment of L/S ratio. Virtually Si has no dependency and Cl shows a very weak dependency. It is known that Si being a refractory is far less soluble in water [24] and therefore the amount of water has no role in the Si removal. On the other hand, Cl being extensively water-soluble [33] again results in maximum removal despite the amount of water. Only K shows appreciable dependency due to its diffusion-limited secondary release [31] and hence increase of L/S ratio is desired only if the primary requirement of the washing treatment is to remove K. However, compared to Figure 2(a), the leaching dependency of K on the L/S ratio is far less than on temperature. This trend has been observed by the other researchers as well with empty fruit bunches [31] and wheat straw [34]. According to Figure 4(b), more than 80% of both K and Cl can be removed at an L/S ratio of 100.

3.3. Leaching kinetics of K and Cl at best washing conditions

Using best process conditions, at 65 °C washing temperature and L/S ratio of 100, elemental removal of K and Cl were obtained at different time intervals as shown in Figure 5.

According to Figure 5, Cl attains equilibrium essentially at 10 min where no considerable removal thereafter. K takes more time to reach equilibrium gradually increasing from 39% at 5 min to 84% at 90 min. Si

Table 3. Calculated kinetic parameters for different L/S ratios.

L/S ratio	20	40	60	80	100
k (cm $\mu S^{-1} s^{-1}$)	14.2×10^{-5}	16.5×10^{-5}	16.2×10^{-5}	15×10^{-5}	15.4×10^{-5}
h ($\mu S\ cm^{-1} s^{-1}$)	0.026	0.029	0.032	0.033	0.035
C_s ($\mu S\ cm^{-1}$)	13.62	13.32	14.14	14.81	15.11
R^2	0.998	0.997	0.995	0.997	0.995
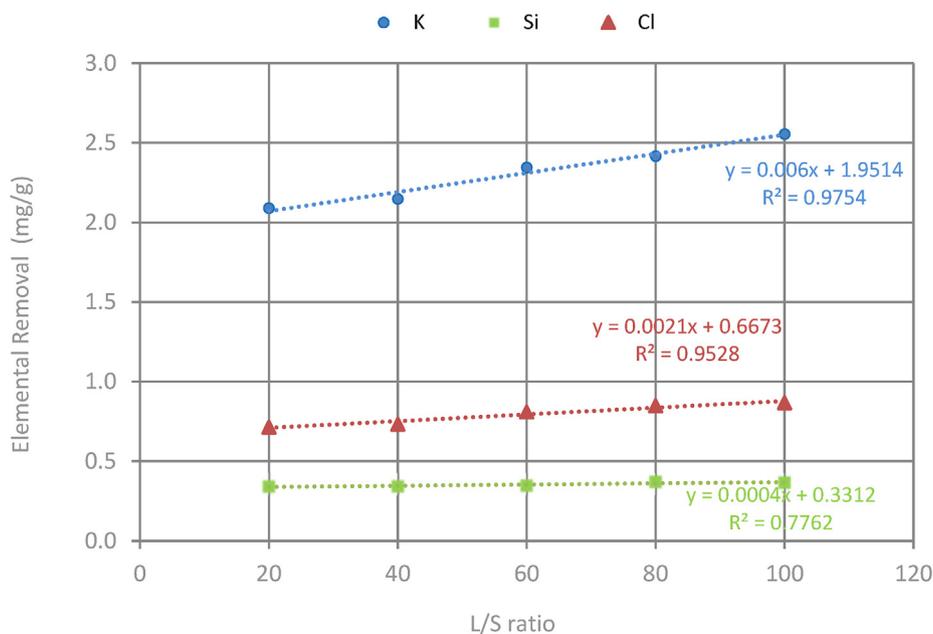
(a)
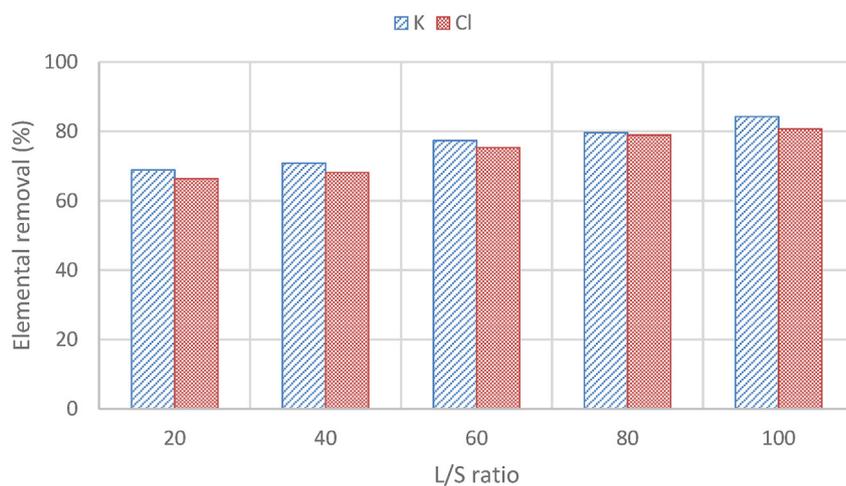
(b)

Figure 4. Variation of elemental removal with L/S ratio (a) removal in mg/g (b) removal in %.

started to leach only after 60 min of washing time still in minor quantities (not shown in the figure). This indicates less solubility of K compared to Cl and the least solubility of Si. This is due to the difference in water-soluble fractions of different components. In agricultural waste, about 60–90% of K and almost all of Cl exist as water-soluble forms [33]. Si is known to be a refractory with little or no solubility in water [24]. The reason for starting Si removal after 60 min could be due to the acidic nature of the leachate as a result of Cl removal which enhances the removal of refractories. Acid leaching is known to be effective in removing Si [15]. At 90 min duration, more than 80% of K and Cl have been removed. It is interesting to note that even within 10 min of washing time, 75% of Cl and 50% of K is removed which is significant. Aligning with these results it has been reported for the wheat straw that

up to 34–53% removal of K and 61–81% removal of Cl can be achieved within 5–10 min [35]. Further, five minutes of washing has been reported to achieve 80–90% Cl removal 60–90% K removal, and 10–30% of Si removal in coastal wood samples [36]. Previous studies on rice husk washing have used long washing durations spanning from 3-6 h [11, 22]. However, long washing durations are impractical when it comes to industrial applications due to the lower production rate and the requirement of a large reactor volume to cater the required demand. A recent study also has revealed the favorable nature of short washing durations and large feedstock sizes for practical and industrial-scale implementations [34].

It has been reported that high-temperature chlorine corrosion is effectively reduced when chlorine concentrations are reduced such that
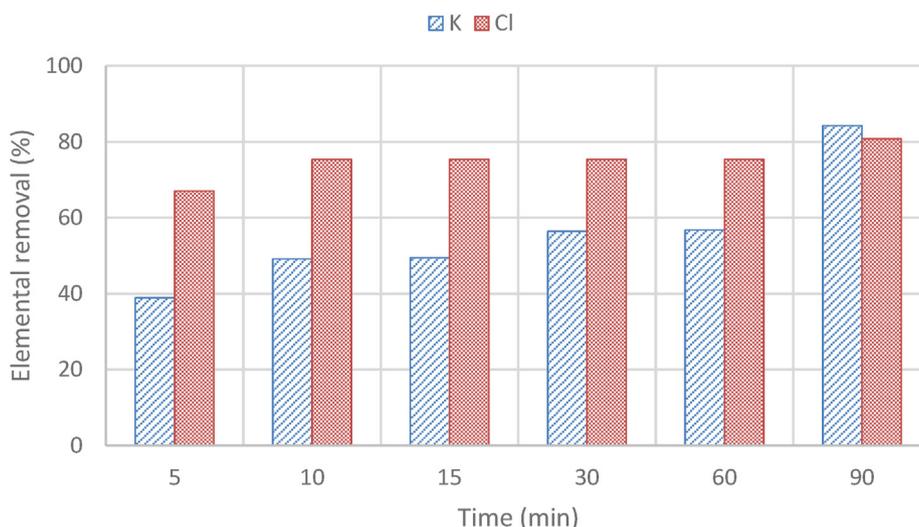
Figure 5. Variation of elemental removal with washing time.

Table 4. Calculated kinetic parameters for K and Cl.

	K	Cl
k (g mg^{-1}s^{-1})	0.0006	0.0141
C_s (mg/g)	2.514	0.864
h (mg g^{-1} s^{-1})	0.004	0.011
R^2	0.92	0.998

Fuel-Cl < 30 mg/MJ [37]. Taking HHV of rice husk as 15 MJ/kg which is a typical value [3], Fuel-Cl after 10 min of washing is around 18 mg/MJ which is far less than anticipated for safe operation. The raw rice husk contains Fuel-Cl around 72 mg/MJ which is beyond the safe margin. The ratio Cl/(K + Na) quantifies the proportion of alkali metals that could be vaporized as volatile chloride. There is a risk of the formation of corrosive deposits when the ratio is higher than 0.3 [38]. Raw rice husk has a value around 0.4 indicating the risk of formation of corrosive deposits and after 10 min of washing, it reduces to 0.19 resulting a lowered risk.

The pseudo-second-order kinetic model was applied to all the time intervals and Table 4 summarizes the calculated kinetic parameters and corresponding R^2 values. R^2 values always being higher than 0.92, it can be observed that the proposed second-order kinetic model can successfully represent the leaching behavior of K and Cl with respect to different time intervals.

According to Table 4, both the overall leaching rate constant and the initial leaching rate values are higher with Cl. Similarly, it has been reported that the overall rate of leaching is much faster for Cl compared to K in empty fruit bunches [31]. The model predicts 83% K removal and 80% Cl removal at saturation whereas actual removal at 90 min was 84% of K and 81% of Cl respectively. The pseudo-second-order kinetic model seems to predict the K and Cl removal with substantial accuracy. At saturation, comparatively higher leaching capacity has been reported for K than Cl in empty fruit bunches which is similar to the observations of the present study [31]. In previous studies, leaching kinetic parameters have been derived for Na, K, Mg and Ca in mallee biomass [28] and K, Cl, S, and N in empty fruit bunches [31]. Direct comparison of kinetic parameters is difficult due to the differences in biomass types and process conditions.

3.4. Recommendations for industrial applications

A washing temperature of 65 °C and an L/S ratio of 100 with 10 min of washing time can be recommended for significant removal of K and Cl to mitigate combustion-related issues. The obtained best washing parameters can be used to design a rice husk washing pretreatment facility for industrial applications. The use of hot condensate returned after the process use of steam can be recommended as a good source of washing medium. The moisture content of washed rice husk is high. Therefore, several methods such as mechanical dewatering, air drying, and drying using hot flue gas can be recommended to remove moisture before the combustion applications. Further, the leachate contains a high amount of potassium and other minerals with a good fertilizer value [39]. Therefore, leachate can be reused for irrigation purposes as an organic liquid fertilizer.

4. Conclusion

This study aimed at how the rice husk can be upgraded by washing pretreatment in a technically and economically feasible way. Electrical conductivity measurements were used to evaluate the effect of washing temperature, L/S ratio, and washing time on the washing performance. The washing temperature of 65 °C was chosen as the best temperature within the 30–75 °C temperature range. The L/S ratio of 100 was chosen as the best L/S ratio within 20–100. Washing temperature has an appreciable effect on Si removal, a considerable effect on K removal, and a very weak effect on Cl removal. The L/S ratio has an appreciable effect on K removal, a very weak effect on Cl removal whereas almost no effect of Si removal. Further, the pseudo-second-order kinetic model successfully represents the overall leaching behavior as well as the leaching of K and Cl. At the selected temperature and L/S ratio, within 10 min of washing time, 75% of Cl and 50% of K are removed from rice husk, and therefore water washing can be considered as an effective method for the removal of K and Cl. In industrial installations, hot condensate returned after the process use of steam can be recommended as a good source of washing medium.

Declarations

Author contribution statement

Melani Anuradha Peiris: Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Duleeka Sandamali Gunarathne: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Data availability statement

Data will be made available on request.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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