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Optimization of rice husk pretreatment for energy production

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A B S T R A C T

One of the most widely cultivated crops in the world is rice, leading to millions of tons of rice husks (also known as rice hulls or chaffs). The large amount of this lignocellulosic biowaste has resulted in an extensive search for its utilization. One such usage of this waste is for the production of electricity, such as in combined heat and power or gasification units. However, one of the disadvantages of using rice husks is their high silica content which produces large amounts of undesirable ash upon combustion leading to operation problems such as slagging and clogging. Here, alkali pretreatment for the extraction of silica in the form of sodium silicate has been studied using response surface methodology (RSM) and Analysis of Variance (ANOVA). Three independent variables namely reaction temperature, duration, and alkali concentration were considered using a Box–Behnken design (BBD). The operating conditions were optimized under different scenarios. The first optimization focused on the two goals of high ash removal and high solid yield while the next optimization rounds added the criteria of low NaOH usage and robust design (using propagation of error (POE)). The final treated rice husks can therefore be more suitably used as feed for thermal and/or electric units. The developed empirical predictive models were successfully validated through additional experimentation.

1. Introduction

With the increase in public awareness regarding sustainable development, the utilization of renewable resources is on the rise. In the wake of problems associated with fossil fuels, biomass is commonly recognized as one the most important alternatives [1]. One of the most widely cultivated plants in the world is rice. China is the world leader in rice production followed by India and Indonesia. At just under 700 million tons per year, rice is currently the third most widely produced crop in the world after sugar cane and maize [2]. The grain is covered and protected by an outer layer known as the husk or hull. The husk is not edible, is removed in the first stage of the milling process, and composes approximately 20–22% of the total weight of the milled paddy [3,4]. Rice husks are particularly known among agricultural biowastes for their low percentage of protein and available carbohydrates, and high percentage of ash containing mostly silica. The exact composition of rice husk which influences its proximate and ultimate analysis depends on many factors such as the weather and cultivation conditions [5–8]. The silica content in the dry season (mean 18%) has been reported to be higher than in the wet season (mean 15%) [3].

Various applications have been proposed for rice husk utilization [9–11] from which silica production [12–16] and energy generation [17–23] have been among the most scrutinized. However, one of the main drawbacks to energy (electricity) generation from rice husks is their high ash content [24]. Aside from the fact that the higher heating value of biomass is inversely proportional to its ash content [25], the silica can form undesired deposits and slags during combustion, which can lead to operational problems [26]. The melting of the rice husk ashes [27] has been known to cause agglomeration, fouling, and corrosion of heat transfer surfaces [28,29].

The removal of silica from rice husks by using sodium hydroxide has been documented from as early as nearly a century ago [30]. Recent years have also seen numerous sporadic studies regarding treatment of husks with sodium hydroxide [1,31–40]. Nonetheless, a parametric study using Response Surface Methodology (RSM) has never been provided on the alkali leaching of silica from raw rice husk. For example Singh et al. have previously used RSM to evaluate

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the saccharification of rice straw and hull by microwave–alkali pretreatment, but have unfortunately not considered the silica content as a response [36].

RSM is a statistical technique for modeling and analysis of the effects of designated independent variables on selected response variables. Various RSM designs help obtain statistically valid results with running limited experimental trials. The powerful and popular tool of RSM has been applied widely across fields [41–43]. In this manuscript, RSM is used for the first time to optimize operating conditions which allow for maximum silica removal while retaining most organics in the solid phase. If this target is achieved, the rice husks will become more suitable for heat and energy generation purposes. The extracted silicate can in turn be used for the production of silica products with further treatment [44–46] such as in the production of zeolites through hydrothermal reactions. Elsewhere, Bazargan et al. have used RSM to investigate the effect of alkali treatment on the moisture content and drying kinetics of rice husks which is another important factor effecting their usage in energy production [47]. In the literature, the word “ash” is usually used to refer to solid residues of combustion. Although no combustion is employed herein, the word ash is used to refer to the mineral content of the husk removed with alkali treatment.

2. Materials and methods

2.1. Alkali extraction of silica

The rice husks were harvested in China and donated by Peako Biomass Energy Ltd. The husks were kept in an oven at 90 °C and used as-is without washing or any other pretreatment. In each experimental run, the husks were treated with a sodium hydroxide solution at the desired temperature under reflux. The solid loading was fixed at 10 g/L of solution. A magnetic stirrer was used in every experiment to ensure adequate stirring. After the reaction duration was completed, the solids were washed with 4 L of deionized water per gram of husk to ensure all soluble fractions were removed. The as-prepared husk was dried and collected.

2.2. Ash removal measurements

In the literature, the major constituent of rice husk ash is shown to be silica (SiO_2), although other element oxides such as Al, K, P, Ca, Mg, Na, and Fe are also present is small amounts [28,48]. Since the overwhelming majority of the ash is composed of silica, it is safe to use the ash content as a good estimate of silica content. The composition of the rice husk as measured by X-ray fluorescence (XRF) spectroscopy is provided in the Supplementary Material. The XRF data shows that the majority of the ash is composed of silicon (84.1% wt), followed by potassium (9.4% wt), and calcium (3.2% wt). Since potassium oxides, nitrate, carbonate, sulfate, and silicate are all soluble in water, the aqueous sodium hydroxide solution will be capable of removing at least 93.5% of the minerals. Predicting the solubility of the various other mineral components which may exist is less straightforward.

The ash content is measured using thermogravimetric analysis (TGA). The samples are heated to 650 °C for a prolonged period under air atmosphere until constant weight is reached. The ash removal efficiency is defined as:

$$\text{Ash removal}\,\% = \left(1 - \frac{W_f \times \text{Ash}_f}{W_i \times \text{Ash}_i}\right) \times 100 \tag{1}$$

where W_i and W_f are the weight of the rice husks before and after the treatment, and Ash_i and Ash_f are the initial and final ash content (weight %) of the husk respectively.

2.3. Design of experiment

A 3-factor, 3-level Box–Behnken design (BBD) was used for the Design of Experiments (DOE). The three parameters to evaluate were selected as temperature (X_1), treatment duration (X_2), and NaOH concentration (X_3). The NaOH concentration is defined as the weight ratio of NaOH in the solution to dry rice husk. For example, a 25% concentration represents a 1:4 ratio (i.e. the weight of NaOH used is solution is one fourth of the dry weight of the rice husk). Ash removal percentage and solid yield were used as response variables. The low, center, and high levels of each factor were denoted as −1, 0 and +1. The corresponding values for these factors are shown in Table 1.

A total of 17 experiments were planned by the Design Expert v.7 statistical software in randomized order. A polynomial equation was used to model the mathematical relationship between the variables and responses as follows:

$$Y = \beta_0 + \sum_{i=1}^{N} \beta_i X_i + \sum_{i=1}^{N} \beta_{ii} X_i^2 + \sum_{i=1}^{N-1} \sum_{j=i+1}^{N} \beta_{ij} X_i X_j \tag{2}$$

where Y is the predicted response and N is the number of variables, fixed at 3 in this study. X_i and X_j are the independent variables, and β_0, β_i, β_{ii} and β_{ij} are the intercept term, the linear effects, the squared effects and the interaction effects, respectively.

3. Results and discussion

In this study, three factors with three levels were used in a BBD to evaluate alkali leaching of silica from rice husks. The results of the BBD experiments are displayed in Table 2. A “logit” transformation, defined as follows, is applied to the ash removal response (y):

$$y_{\text{trans}} = \ln\left(\frac{y - y_l}{y_u - y}\right) \tag{3}$$

where y_{trans} is the response after the transformation has been applied, y_l is the lower limit of the response (here set at 0%) and y_u is the upper limit of the response (here set at 100%). The logit transformation is best suited for responses bound between lower and upper limits.

3.1. Fit summary

The fit summary, including the sequential model sum of squares, lack of fit tests, and model summary statistics for ash removal and solid yield are presented in Tables 3 and 4 respectively. By considering the Sequential Model Sum of Squares, the highest order polynomial where additional terms are significant and the model is

Table 1
Experimental levels of the independent variables.

Factor	Name	Units	Low (−1) Actual Value	Center (0) Actual Value	High (+1) Actual Value
X_1	Temperature	°C	25	57.5	90
X_2	Time	h	4	14	24
X_3	NaOH Concentration (wt alkali/wt husk)	%	1	13	25