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Optimization of Iron Removal by Rice Husk Using Response Surface Methodology Approach

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Abstrak. Penelitian dilakukan untuk mengamati penyisihan besi (II) dari larutan cair dengan teknik adsorpsi - batch menggunakan dua varian adsorben sekam padi (sekam padi murni - RRH) dan sekam padi teraktivasi natrium (ARH). Hasil penelitian menunjukkan bahwa kedua adsorben mampu digunakan sebagai adsorben untuk mereduksi besi (II) dari larutan berair. Pada penelitian ini, pengaruh 4 variabel bebas pada dua level rendah dan tinggi meliputi pH (5 dan 9), dosis adsorben (20 dan 80 gr/100 mL), waktu kontak (60 dan 240 menit) dan konsentrasi awal larutan uji (20 dan 60 mg/l) terhadap respon (persentase penghapusan besi (II) menggunakan RRH dan ARH) dirancang dan dianalisis secara statistik menggunakan response surface methodology. Signifikansi faktor-faktor independen dan interaksinya diuji dengan menggunakan analisis varians (ANOVA) dengan batas kepercayaan 95%; (α = 0,05). Nilai optimum dari variabel terpilih diperoleh dengan mensubstitusi model regresi polinomial yang sesuai dan dengan menganalisis plot kontur permukaan respons serta dengan menyelidiki plot efek utama dari empat faktor. Proses adsorpsi sangat bergantung pada pH. Persentase hingga 100% dicapai pada pH 7 untuk kedua adsorben.

Kata Kunci: besi (II), Sekam padi murni, Sekam padi teraktivasi, Response surface methodology.

Abstract. The removal of iron (II) from aqueous solution by batch adsorption technique using two rice husk adsorbent variants (raw rice husk - RRH) and sodium activated rice husk (ARH)) was investigated. The result shown that both of adsorbent has been successfully used as adsorbent for reducing iron (II) from aqueous solution. In recent study, the effects of 4 factors at two level low and high including pH (5 and 9), adsorbent dosage (20 and 80 gr/100 mL), contact time (60 and 240 minutes) and initial concentration (20 and 60 mg/l) to response (percentage of iron (II) removal using RRH and ARH) was designed and analyzed statistically using response surface methodology. The significance of independent factors and their interactions were tested by means of the analysis of variance (ANOVA) with 95% confidence limits (α_c = 0.05). The optimum values of the selected variables were obtained by substituting the fit polynomial regression model and by analyzing the response surface contour plots as well as by investigating main effect plot of four factors. Adsorption process was found to be highly pH dependent. The percentage up to 100% was achieved in pH 7 for both adsorbent.

Keywords: Iron(II), Raw Rice Husk, Activated Rice Husk, Response surface methodology

INTRODUCTION

A major concern worldwide in the recent years is environmental pollution due to the increase of industrialization and of technology. The presence of heavy metals in the water and wastewater through industrial processes and domestic activities often represents a risk to environment. Iron is found to be a metal widely used in industries such as car, coating, steel industries and etc. There are numerous techniques used for reducing heavy metals from industrial effluent. These includes ion-exchange, electrochemical reduction, evaporation, solvent extraction, reverse osmosis, flocculation and coagulation, chemical precipitation, membrane filtration, filtration and adsorption (Chuah et al., 2005; Kasman et al., 2012; Kasman & Ibrahim, 2016). However, adsorption is one of the most efficient methods of removing heavy metals pollutants from water and wastewater. Moreover, the adsorption provides an attractive alternative treatment, especially if the adsorbent is inexpensive and readily available.

Various cheaper materials including industrial, biomass and agricultural waste, have been used in this adsorption methods by some researchers. (Kasman & Ibrahim, 2016; Saha et al., 2023; Triwiswara et al., 2020; Zafar et al., 2020) reported that rice husk either modified or unmodified were effective to reduce different heavy metals in batch system and column system. Previous researches employed unmodified and modified rice husk. Mostly, rice husk was modified or activated with different chemical concentration such as sodium carbonate, sulfide acid, nitric acid and phosphate for reduce different heavy metals and other pollutants. On the other hand, some of researches also used rice husk ash in their research. But only a few reports discussing about the use of rice husk for iron removal, and evaluate their result including the influences of all factors in adsorption process statistically (Chuah et al., 2005; Gun et al., 2022).

For knowing the influences of individual factors as well as their interactive influences, Response surface methodology (RSM) has been suggested to be one of statistic software to analyze these problems. RSM which is a combination of mathematical and statistical techniques that are useful for modeling and analysis in application where a response of interest is influenced by several variables and the objective is to optimize this response in addition to reduce the number of experiment (Gun et al., 2022; Kasman & Ibrahim, 2010). The aims of this present study was to explore the capability of rice husk in modified and unmodified as an adsorbent for reducing iron in aqueous solution and to know the optimum condition for its adsorption process. Response surface methodology (RSM) was used to design experiment and to analyze the experiment results as well as to determine optimum conditions.

METHOD

Preparation of adsorbent

There were two adsorbents used in this study, raw rice husk (RRH) and activated rice husk (ARH). Rice husk obtained from a nearby rice mill was washed several times with tap water continued with distilled water to remove all the dirt particles and impurities. The washing process was stopped until no color in the wash water. For preparing RRH, the washed rice husk was dried at 100ºC to get rid of moisture and impurities to be constant weight. Then ground and sieved to a particle size of ≤ 600 µm. After completing washing with tap water and distilled water, for preparing ARH, then the washed rice husk was dried at 60ºC for some hours, soaked in 0.1 M NaOH for 4 hours. The treated rice husk was filtered and rinsed with tap water continued with distilled water to remove the excess sodium hydroxide from rice husk. Then dried again at 100ºC in oven to be constant weight and finally ground to particle size of ≤ 600 µm. All of adsorbents were kept in air tight container at room temperature until used.

Chemicals

Stock solution of iron(II) (1000 mg L^{-1}) was prepared by dissolving ferrous sulfate ($FeSO_4.7H_2O$) in distilled water, according the procedures stipulated in the APHA The pH of each last solution was adjusted to the required value with diluted and concentrated H_2SO_4 and NaOH solution. All the chemicals used were of analytical grade.

Experimental design and data analysis

Batch adsorption studies were conducted to determine the adsorption of iron (II). The four factors including pH, initial concentration of iron (II) aqueous solution, Adsorbent dosage and contact time were varied in this experiments as shown in table 1. For each run, at room temperature, a desired amount of adsorbent was added to 100 ml of an adjusted pH and a desired Fe(II) concentration ($FeSO_4.7H_2O$) in Erlenmeyer flasks. Then the flasks were agitated on a shaker at constant speed 160 rpm in a desired contact time for the adsorption equilibrium. After the equilibrium period, samples were filtered and placed in small container to be analyzed by the Inductively Couple Plasma (ICP). The 27 runs with all combinations of factors for each adsorbent were randomly performed according to a box-behnken statistical design (BBD) for linear, quadratic and interaction effects of four factors. All the experiments conducted in duplicate and mean values were presented. The removal efficiency of the iron(II) solution was defined as:

$$Y = \left(\frac{C_i - C_e}{C_i}\right) x\ 100$$

where, C_i = initial concentration (mg/l), C_f = final concentration (mg/l) and m = mass of adsorbent (g/l).

Table 1
Levels Of Factors for Each Adsorbent

Factor	Symbol	Low	High
pH	X1	5	9
Dosage (g/100 ml)	X2	2	8
Contact time (minutes)	X3	60	240
Initial Concentration (mg/l)	X4	20	60

Source: processed data

In RSM, the form of the relationship between the response, in here the percentage of iron(II) removal, *Y* (dependent variables) and factors, *X* (the independent variables) is normally done by applying a low order polynomial and if the response is well modeled by a linear function, then the function of *Y* is the first-order model as equation below:

$$Y = \beta_0 + \beta_i X_i + \beta_{ii} X_{ii} + \cdots \beta_k X_k + \epsilon$$

But, if a curvature appears in the system, then a polynomial of higher degree must be used such as quadratic models as shown is equation. In other words, when the experimenter is relatively close to optimum, a second-order model is usually required to approximate the response because of curvature in the true response surface (Abdmunaf Atta et al., 2022; Aslani et al., 2023)

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_{ii}^2 + \sum\sum_{i<j=2}^{k} \beta_{ij} X_i X_j + \epsilon$$

where, *Y* in equation is the dependent variable (response variable) to be modeled, β, is constant regression coefficients of the model, *X is* the independent variables (factors), k is the number of factors studied and ϵ is the error.

RESULTS

The result were analyzed using MINITAB® 14 software. In this study, the respective contour plots were obtained for RRH and ARH based on effects of the four factors (pH, adsorbent dosage, contact time and initial concentration). The main effects and interaction between the factors and the responses were determined using Analysis of variance (ANOVA). The quality of the fit polynomial model was expressed by the coefficient of determination R^2, and model terms were evaluated by the P-value (probability) with 95% confidence level. Average percentage of iron removal using both of raw rice husk and activated rice husk in all combination of factors were almost up to 100% with the least percentage 50.88%. It indicates rice husk has good adsorption capacity of heavy metals iron(II) as investigated by previous researchers, but in this recent study, the condition of process was optimized to get optimum removal.

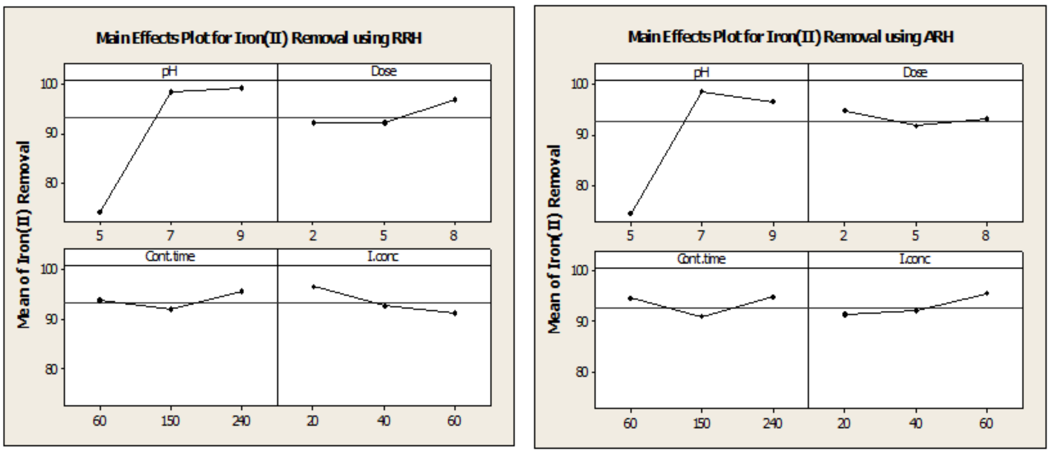
However, the optimum condition of iron(II) adsorption obtained based on the main factors which represent deviations of the average between high and low levels for each one of them (Abdmunaf Atta et al., 2022; Aslani et al., 2023) From Figure 1, for both RRH and ARH above, it was noticed that pH, adsorbent dosage, contact time and initial concentration of iron (II) solution effected to iron (II) adsorption. However, it was observed pH and adsorbent dosage have very significant effect during adsorption processing for both RRH and ARH. The similar condition was shown for pH, dosage and contact time in adsorption using RRH and ARH but not for initial concentration effect. Different with ARH, Using RRH, the effect of initial concentration is significant and showed negative or in other words, iron (II) removal increase with decrease of initial concentration.

Regression model and statistical analysis

The result of effect, regression, standard error, T and P value are shown in Table 3. The effects are statistically significant when P-value, defined as the smallest level of significance leading to ejection of null hypothesis, is less than 0.05. The application of the response surface methodology based on the estimation of the factors indicated an empirical relationship between the response and input factors (Table 3) for both RRH and ARH. It was expressed by the following quadratic equation below which substituted from significant regression coefficient was determined by P-value (Table 3).

% removal of iron (II) using RRH, Y_{RRH}
$= (-97.1675) + 48.0054X1 + 9.4215X2 - 1.5708X4 - 3.0598X1^2 - 1.0303\ X1X2 - 0.0245X1X3 + 0.2481X1X4.$
% removal of iron (II) using ARH, Y_{ARH}
$= (-74.6316) + 46.7213X1 - 7.1632X2 - 3.3519X1^2 - 1.1963X1X2$

The quality of the fit equation was expressed by the coefficient of determination R^2 that determined whether a factor contributes significantly to the variance of the data. The recent result shows there is high dependence and correlation between the observed values and predicted values of responses due to value of R^2 and adjusted R^2 both adsorbent (RRH and ARH) is more than 80% or in other word regression model provides an excellent explanation of the relationship between the independent variables (pH, adsorbent dosage, contact time, initial concentration) and the response (% adsorption).



Source: processed data

Figure 1.
Main effect plot for iron(II) removal using raw rice husk (RRH) and using activated rice huks (ARH)

Table 2
Response surface regression for RRH and ARH

Term	RRH				ARH			
	Coef	SE Coef	T	P	Coef	SE Coef	T	P
Constant	-97.1675	35.8396	-2.711	0.01	-74.6316	20.3399	-3.669	0.001
pH	48.0054	6.6749	7.192	0.000	46.721	3.7882	12.333	0.000
Dose	9.4215	3.4291	2.748	0.009	-7.1632	1.9461	-3.681	0.001
Cont.time	0.1896	0.1143	1.659	0.105	-0.0336	0.0649	-0.518	0.608
I.conc	-1.5708	0.5366	-2.928	0.006	0.5053	0.3045	1.659	0.105
pH*pH	-3.0598	0.4232	-7.23	0.000	-3.3519	0.2402	-13.955	0.000
Dose*Dose	-0.0352	0.1881	-0.187	0.853	-0.0747	0.1068	-0.7	0.488
Cont.time*Cont.time	0.000	0.0002	0.026	0.979	0.0001	0.0001	0.721	0.475
I.conc*I.conc	-0.0028	0.0042	-0.662	0.512	-0.0031	0.0024	-1.287	0.206
pH*Dose	-1.0303	0.3258	-3.162	0.003	1.1963	0.1849	6.47	0.000
pH*Cont.time	-0.0245	0.0109	-2.252	0.03	-0.0001	0.0062	-0.011	0.991
pH*I.conc	0.2481	0.0489	5.076	0.000	-0.0069	0.0277	-0.249	0.804
Dose*Cont.time	-0.0026	0.0072	-0.356	0.724	0.0014	0.0041	0.335	0.74
Dose*I.conc	-0.0175	0.0326	-0.537	0.595	-0.0238	0.0185	-1.285	0.206
Cont.time*I.conc	0.0001	0.0011	0.0097	0.923	0.0001	0.0006	0.122	0.904

RRH : S = 5.529 R-Sq = 85.9% R-Sq(adj) = 80.9%
ARH : S = 3.138 R-Sq = 93.8% R-Sq(adj) = 91.5%
Source: processed data

Furthermore, to ensure a good model, the test for significance of regression model was performed and applying the analysis of variance (ANOVA) for iron (II) removal in aqueous solution using RRH and ARH, the result of which are performed in Table 4. Values of Prob > F less than 0.050 indicate model terms are significant for iron (II) adsorption using RRH and ARH. The lack of fit (LOF) describes the variation of the data around the fitted model. The P values for lack of fit for both RRH and ARH (< 0.05) presented in Table 3 showing that the LOF statistically is significant, implying significant model correlation between the variables and process responses.

High value of parameter estimate for factors X1, X2, X3, X4 and interaction between of factors showing a high level of significance indicates the importance of these variables in adsorption process. Iron(II) adsorption using RRH, the factor X1 (pH) and X2 (adsorbent dosage) have positive effect, X4 (initial concentration of solution) has negative effect and X3 (contact time) has no effect, whereas, in case of iron(II) adsorption using ARH, factor X1(pH) has positive effect, and X2 (adsorbent dosage) has negative effect, but X3 (contact time) and X4 (initial concentration of solution) have no effect.

Table 3
Analysis of variance for RRH and ARH

	RRH						ARH					
Source	DF	Seq SS	Adj SS	Adj MS	F	P	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	14	7274.4	7274.4	519.597	17	0.000	14	5785.2	5785.2	413.223	41.97	0.000
Linear	4	4090	2744.7	686.165	22.45	0.000	4	3009.7	2330.3	582.571	59.17	0.000
Square	4	1923	1923	480.759	15.73	0.000	4	2345.3	2345.3	586.321	59.55	0.000
Interaction	6	1261.4	1261.4	210.226	6.88	0.000	6	430.2	430.2	71.707	7.28	0.000
Residual Error	39	1192.2	1192.2	30.57			39	384	384	9.846		
Lack-of-Fit	10	1075.1	1075.1	107.513	26.63	0.000	10	179.6	179.6	17.96	2.55	0.024
Pure Error	29	117.1	117.1	4.038			29	204.4	204.4	7.048		
Total	53	8466.6					53	6169.2				

Source: processed data

In order to detect and explain the systematic departures from the assumptions that errors are normally distributed and are independent of each other and that the error variances are homogeneous, normal probability plot of residual is carried out. As seen in fig.2 and fig. 3, residual plot of percentage of iron(II) removal using RRH and ARH showed in two differences graphical graph. Normal probability of residuals for iron(II) adsorption using RRH and ARH showed similar trend that each samples tested of adsorbent is randomly distributed between -0.2 to 0.2 except 2 points outliers. This result is in satisfactory level and almost no serious violation of the assumptions underlying the analyses. Shortly, the normal probability plot supports the residual versus fitted plot due to P value less than 0.05.

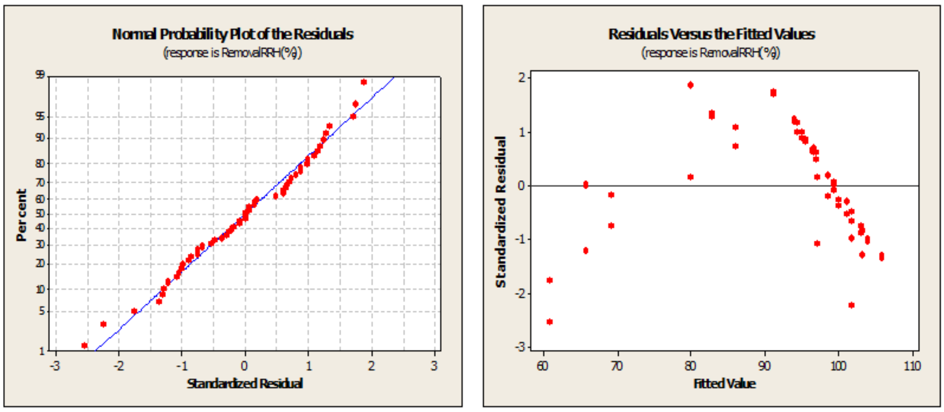


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Figure 2
normal probability plot of residuals and percentage iron (II) removal vs residuals for iron (II) adsorption using RRH

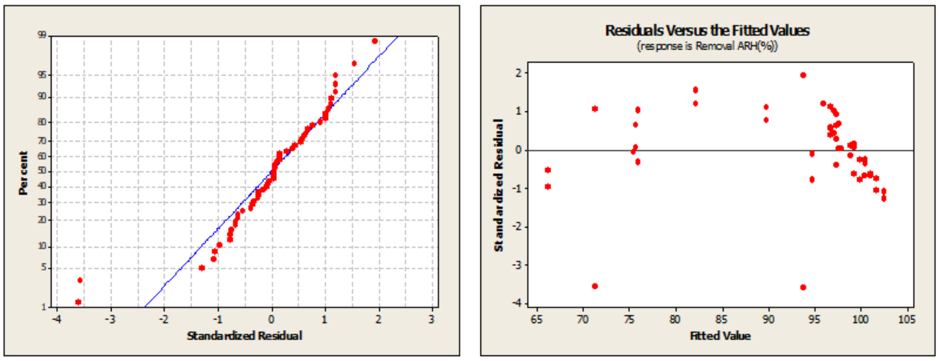

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Figure 3
Normal probability plot of residuals and percentage iron(II) removal vs residuals for iron(II) adsorption using ARH

Effect of pH

As shown in Table 2 and Table 3, pH has a significant effect for iron(II) adsorption for both of adsorbents. The value of P either RRH or ARH is 0.000 or P < 5%. Interaction between pH and dose; pH and contact time; pH and initial concentration was shown in contour (fig. 4 and fig. 5) notice that the adsorption of iron(II) was favored in normal pH or alkali pH(>7), the percentage of iron(II) removal for RRH and ARH up to 100%. (Saha et al., 2023; Triwiswara et al., 2020; Zafar et al., 2020) also reported similar trends that removal percentage increase with increase of pH because protons compete with metal ion for adsorption site on the adsorbent surface and the concomitant decrease of negative charge of the same surface as well.

Effect of adsorbent dosage

Adsorbent dosage also has main role in adsorption processing for both of adsorbent (RRH and ARH), from Table 2, Figure 1, 4 and 5, as expected that adsorption increase with increasing adsorbent. However, the deviation of graph in main effect plot for adsorbent dosage using ARH (fig. 3) showing little different with deviation of graph using RRH, but it still noticed the adsorption capacity was higher in higher adsorbent dosage. As explained by previous researches that the phenomenon of increase in adsorption capacity with increase in adsorbent dosage was due to the availability of more and more adsorbent surfaces for the solutes to adsorb (Kasman et al., 2012; Nwabanne et al., 2022).

Effect of Initial iron concentration and contact time

Effect of contact time to adsorption of iron (II) using ARH and RRH was not as significant as other factors (pH and adsorbent dosage). As a result of iron(II) removal using RRH and ARH was noticed that percentage of iron(II) removal from low level to high level of contact time was not too different. Base on phenomenon shown in fig.1, 4, and 5, contact time 60 minutes was supposed that enough time for adsorption processing. As expected, using RRH, the percentage of iron(II) removal increased as decreased initial concentration of iron (II) solution. In contrast, the result obtained for iron(II) removal using ARH was shown that initial concentration had small effect or no significant. Although, these conditions (effect of contact time and initial concentration) were not significant, but it is important to determine these factors value to optimize adsorption process.

Apart from the effect of all factors, type of adsorbent is not as significant as other factors, either raw rice husk (unmodified rice husk) or activat. d rice husk (modified rice husk) has shown excellent performance as in adsorption iron(II) process as reported by some researches (Abdmunaf Atta et al., 2022; Aslani et al., 2023). The interaction between these factors gives better explanation of the process, as shown in regression model and anova analysis. The pH value, adsorbent dosage, initial concentration and contact time as well as interaction between of them was observed significant for adsorption process. Clearly, the optimum condition for both adsorbent (RRH and ARH) was expressed in quadratic model equation. pH 5 (acid) was extremely not conducive for iron(II) adsorption, the percentage of iron(II) adsorption increased with increase pH (5 to 9), but pH 7 is enough to achieve good removal percentage. The highest percentage of iron(II) removal for RRH and ARH was achieve higher adsorbent dosage, in case 5 gr/100 mL of adsorbent dosage was supposedly conducive for adsorption process. Percentage of iron (II) removal using RRH and ARH is in range 50.88% - 99.70% and 64.16% - 99.67% respectively. The effect of pH to adsorbent dosage is linear, whereas pH is high when the dosage is high.. As a mentioned above, range of adsorption percentage was stabile for any conditions and interaction between factors. Initial concentration is only significant for RRH, the lower initial solution concentration, the higher iron(II) removal. Contact time was not too significant in this process. The adsorption was shown almost constant after 60 minutes contact time for both adsorbents.

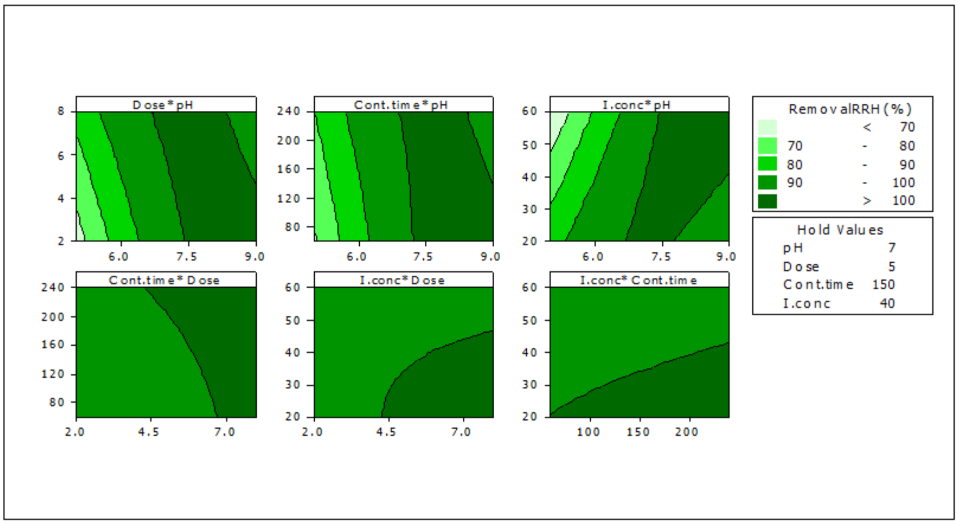

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Figure 4

Response surface contour for Iron removal using raw rice husk (RRH); dose vs pH; cont.time vs pH; I.conc vs pH; Cont. time vs dose; I.conc vs dose; I.conc vs cont time.

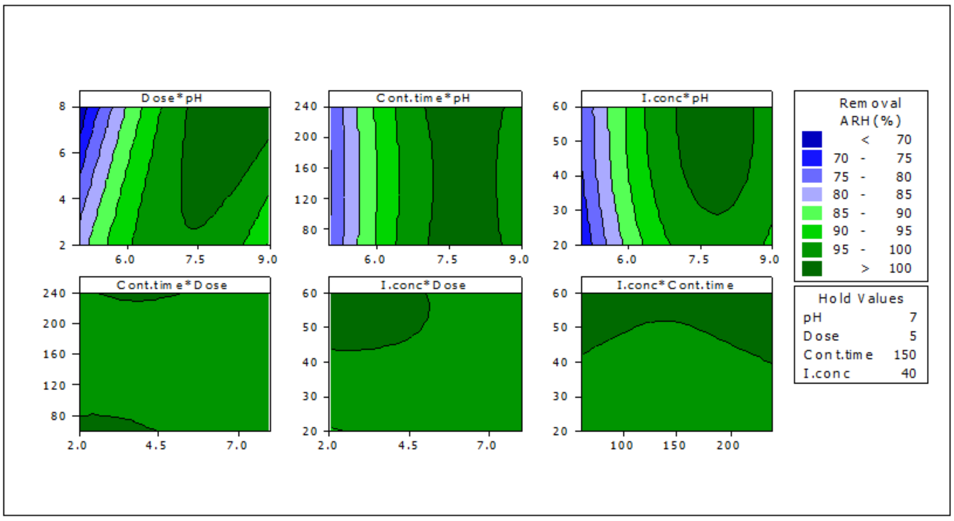

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Figure 5

Response surface contour for Iron removal using activated rice husk (RRH); dose vs pH; cont.time vs pH; I.conc vs pH; Cont. time vs dose; I.conc vs dose; I.conc vs cont time.

CONCLUSION

The aims of this present study was to explore the capability of rice husk in modified (activated rice husk) and unmodified (raw rice husk) as an adsorbent for reducing iron in aqueous solution and to know the optimum condition for its adsorption process. Response surface methodology (RSM) was used to design experiment and to analyze the experiment results as well as to determine optimum conditions. RSM is undoubtedly a good technique for studying the effect of major process factors to response by significantly reducing the number of experiments and henceforth, facilitating the optimum conditions. The result show that raw rice husk and activated rice husk has been successfully used as adsorbent for reducing iron(II) in aqueous solution. The percentage of iron(II) removal both of adsorbent (RRH and ARH) was not too different. Hence, economically raw rice husk (unmodified rice husk) is better choice for industrial application choice. In adsorption processing using RRH and ARH, pH and dosage were very significant. On the other hand, initial concentration of iron(II) solution was significant for RRH only and contact time was not too significant for both adsorbent. The highest percentage of iron(II) removal for RRH and ARH was 99.79% and 99.56% respectively. It was achieved at pH 7 and adsorbent dosage 5 gr/100 ml solutions. To optimize condition, the quadratic model equation can be applied.

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