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Statistical Optimization of Zinc Removal Using Activated Carbon and Magnetic Biochar

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ABSTRACT

Due to the increasing of industrial wastage, content of metal in waste water has increased drastically. The effectiveness of Zn2+ removal from aqueous solution by different adsorbents was experimentally studied. The effect of the process parameters such as pH, adsorbent dosage, agitation speed and contact time were examined whereby the experimental design of the investigation was conducted using Design Expert® Version 6.08. The statistical analysis revealed that that optimum condition for magnetic bochar and activated cabon was at pH 10, dosage 0.09 g, agitation speed and time of 120 rpm and 60 min respectively. It is found that the removal efficiency of Zn2+ for an initial concentration of 10 mg/l using magnetic biochar was 95% and using activated carbon was 65%. Based on the results, it was proven that magnetic biochar emerges as the most promising adsorbent in the removal of Zn2+ compared to activated carbon

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INTRODUCTION

The advance development of industrial technology has contributed a serious damage to the environment by increasing the pollution rate. Various chemical industries such as metal, textile, fertilizers [1] and many other industries releases various type of heavy metals such as zinc [2] Heavy metals can easily enter the food chain and causes various deceases to human such as kidney damage and eventually could lead to cancer [3]. Current methods used to treat heavy metals are ion exchange, chemical precipitation [2] and filtration [3]. Currently adsorption process has been widely used due its simplicity, cheap and easy to scale-up nature. Various type of adsorbent is used in adsorption process such as silicon, manganese and aluminum [4] but exhibit insufficient result. Activated carbon (AC) is widely used due to its high surface area [3] and sufficient removal percentage compared to other adsorbent but it’s slightly expensive. Activated carbon has very short range pg crystallinity due to its graphitic domains which are much smaller in comparison with the nanocarbon materials [5]. Recently, an alternative technology was developed to use engineered/modified biochars to remove various pollutants, including phosphate, heavy metals, and organic compounds, from aqueous solutions [6,7] known as the magnetic biochar. Magnetic biochar (MB) is produced by agriculture biomass waste [8,9] such as cotton stalks, rice straw and empty fruit brunch (EFB). Since commercial activated carbon is expensive, production of cheaper activated carbon from EFB and agricultural waste which is also known as magnetic biochar is also widely used and exhibit better removal than commercial activated carbon.

MATERIAL AND METHOD

Raw Material:

Magnetic biochar obtained from university of Malaya produced from empty fruit brunch. stock solution containing 1000 mg/L of Zn^{2+} metal ions analytical grade were purchased from Merck and used as received.

Batch Adsorption:

Adsorption studies were conducted using stock Zn^{2+} solution of 10. mg/L treated using various variables

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which are dosages of the adsorbent from 0.03 to 0.09 gram, pH of the adsorbent from 3 to 10, agitation speed from 60 rpm to 120 rpm and agitation time from 60 minutes to 120 minutes at a constant temperature of 50°C. Each selected dosage and pH of adsorbent (magnetic biochar and AC) are added to 100 ml of 10 mg/L of stock Zn^{2+} solution with different selected agitation time and speed based on the Design of Expert 6.0 (DOE). The initial and final concentrations of Zn^{2+} stock were determined using the Atomic Spectrometer (PerkinElmer AAnalyst 400). The differences between the initial and equilibrium metal ions concentrations determined the amount of metal ions being absorbed by the CNTs and novel magnetic biochar.

The adsorption capacities of magnetic biochar and AC at time t as well as Zn^{2+} adsorption equilibrium, qe (mg/g) were computed based on the following equation:

$$qt = (C0-Ct)V/m \qquad qe = (C0-Ct)V/m \qquad (1)$$

Where C0, Ct and Ce are the initial concentrations of Zn^{2+} solution, concentration of Zn^{2+} at time t and equilibrium concentration of Zn^{2+} respectively; V volume of Zn^{2+} stock solution (L) and m the weight of the adsorbent used.

RESULTS AND DISCUSSION

(Statistical optimization of removal of Zinc using MB and AC):

Optimization condition of these two adsorbents, MB and AC biochar are crucial to investigate in order to determine the best adsorbent in the removal of Zn^{2+} from aqueous solution. Experiment for optimization studies was conducted based on the Design of Expert (DOE) program where all possible combinations of all the input factors with the minimal number of experimental runs that could possibly be optimized are located. The results from the experiments were analyzed using the analysis of variance (ANOVA) as shown in Table 1 and 2 using AC and MB respectively. Adsorbent dosage, agitation time, agitation speed and pH of the adsorbent are the variables that are employed for the analysis in the design to obtain the removal percentage of Zn^{2+} from aqueous solution. The mean square of the regressed model compares to the mean square of the residuals (errors) is indicated by the fisher F-test value. The F-test which is 39.57 and 96.91 for AC and MB respectively indicates that models are significant. The developed model equation is as follows:

For AC,

$$Removal\ \%\ of\ Zinc = 52 + 10.625\,A - 2\,B + 3.125\,C - 4.25\,D - 4.875\,AB + 4\,AC + 6.375\,AD \qquad (2)$$

For MB,

$$Removal\ \%\ Zinc = 87.5 + 1.25B + 0.625C + 2.625D - 1.25AB + 0.125AC - 2.125AD \qquad (3)$$

Where, the response was the removal of Zinc from aqueous solution, A is the coded value of pH of the adsorbent, B is the coded value of dosage of adsorbent, C is agitation time and D is the coded value of agitation speed. The coefficients with one factor represent the effect on the particular factor, while the coefficients with two factors represent the interaction between the two factors. The positive sign in the equation indicates synergistic effect whereas negative sign indicates antagonistic effect.

Table 1: Analysis of variance (ANOVA) for the removal of Zn^{2+} (%) with magnetic biochar.

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	239	7	34.14286	19.82488	< 0.0001	significant
A	0	1	0	0	1.0000	
B	25	1	25	14.51613	0.0042	
C	6.25	1	6.25	3.629032	0.0892	
D	110.25	1	110.25	64.01613	< 0.0001	
AB	25	1	25	14.51613	0.0042	
AC	0.25	1	0.25	0.145161	0.7120	
AD	72.25	1	72.25	41.95161	0.0001	
Pure Error	15.5	9	1.722222			
Cor Total	432.2778	17				
Curvature	434.6	1	434.6	34.6	0.0003	significant
R-Squared 0.989 Adj R-Squared 0.88 Pred R-Squared 0.85 Adeq Precision 19.7 Std. Div. 1.21						

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The value of the correlation coefficient (R^2 = 0.98) and the adjusted determination coefficient (R^2_{Adj}=0.97) for MB as an adsorbent while R^2 = 0.9883 and R^2_{Adj} = 0.9781 for AC as adsorbent implied that both models are very close to one, indicating a high significance of the model

Table 2: Analysis of variance (ANOVA) for theremoval of Zn^{2+} (%) with Activated carbon.

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	239	7	34.14286	19.82488	< 0.0001	significant
A	0	1	0	0	1.0000	
B	25	1	25	14.51613	0.0042	
C	6.25	1	6.25	3.629032	0.0892	
D	110.25	1	110.25	64.01613	< 0.0001	
AB	25	1	25	14.51613	0.0042	
AC	0.25	1	0.25	0.145161	0.7120	
AD	72.25	1	72.25	41.95161	0.0001	
Pure Error	15.5	9	1.722222			
Cor Total	432.2778	17				
Curvature	243.4	1	243.4	24.3	0.0006	significant
R-Squared 0.939						
Adj R-Squared 0.891						
Pred R-Squared 0.856						
Adeq Precision 14.78						
Std. Div. 1.41						

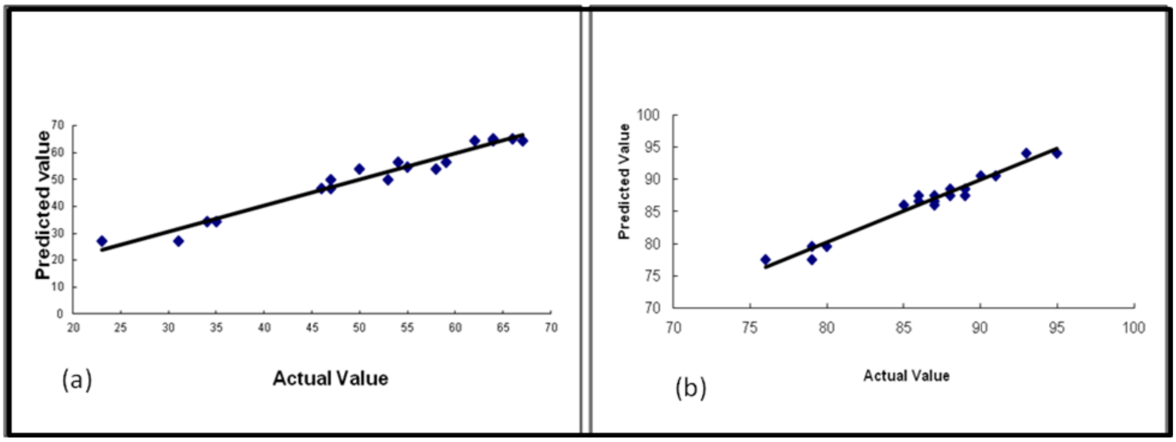

Fig. 1: Relation between Predicted and Actual Removal percentage of Zn^{2+} from aqueous solution using a) AC and b) magnetic biohar.

Figures 1(a) (b) demonstrate relation between predicted and actual value for removal of Zn^{2+} removal from aqueous solution using AC and MB respectively. The graph clearly shows that the actual and predicted values are close, indicating that the models developed are successful in bridging the correlation between the adsorbent used for the removal of Zn^{2+} from aqueous solution. Comparing the MB and AC as an adsorbent, it is proven based on experiments that MB emerges as a better adsorbent in the removal of Zn^{2+} as the removal percentage is more than.

Figures 2(a)-(c) and 3(a)-(c) represent the 3-dimensional plot for the removal of Zn^{2+} using functionalized and magnetic biochar respectively. Figure 2(a) and Figure 3 (a) denote that as the adsorbent dosage increases, the pH of the adsorbent (MB and AC) decreases, and the removal of Zn^{2+} (%) decreases as well. It can be concluded that the removal of Zn^{2+} using both MB and AC can be maximized by capitalizing on the adsorbent dosage and pH. As for figure 2 (b) and figure 3 [b), they demonstrate that as the agitation time increases, the pH of the adsorbent reduces while as shown in figure 2 (c) and figure 3 (c), as the agitation speed increases, the pH of the adsorbent decreases. The surface plots indicate that the optimal conditions for the removal of Zn^{2+} from aqueous .Solution involved interactions between the low level of pH of the adsorbent with the high agitation time, speed and adsorbent dosage. Comparing the MB and AC as an adsorbent, it is proven based on experiments that functionalized MB emerges as a better adsorbent in the removal of Zn^{2+} as the removal percentage is more than 74% while the highest removal using AC is only 55%. Based on the statistical results, the optimum production conditions for the removal of Zn^{2+} from aqueous solution for both MB and AC are when the adsorbent dosage is 0.06 gram, pH of adsorbent at 3 and agitation time and speed at 90 minutes and 90 rpm respectively. Thus, MB is a better adsorbent than the AC.

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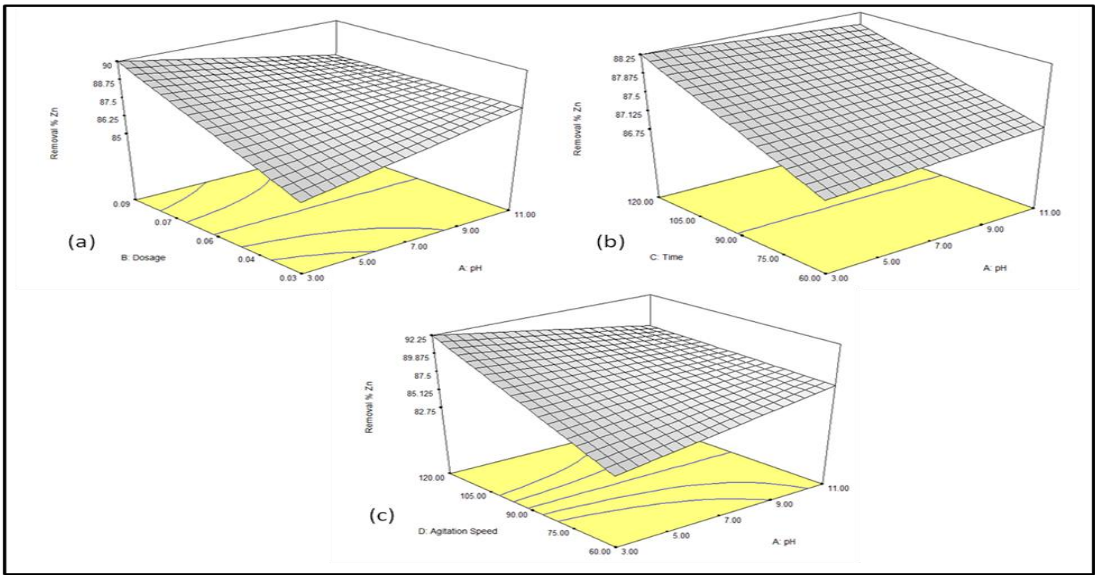

Fig. 2: (a) A 3-D interaction plot of the removal of Zn^{2+} using MB, interaction of MB dosage and pH, (b) A 3-D interaction plot of the removal of Zn^{2+} using MB, interaction of agitation time and MB pH, (c) A 3-D interaction plot of the removal of Zn^{2+} using MB, interaction of agitation speed and magnetic biochar pH.

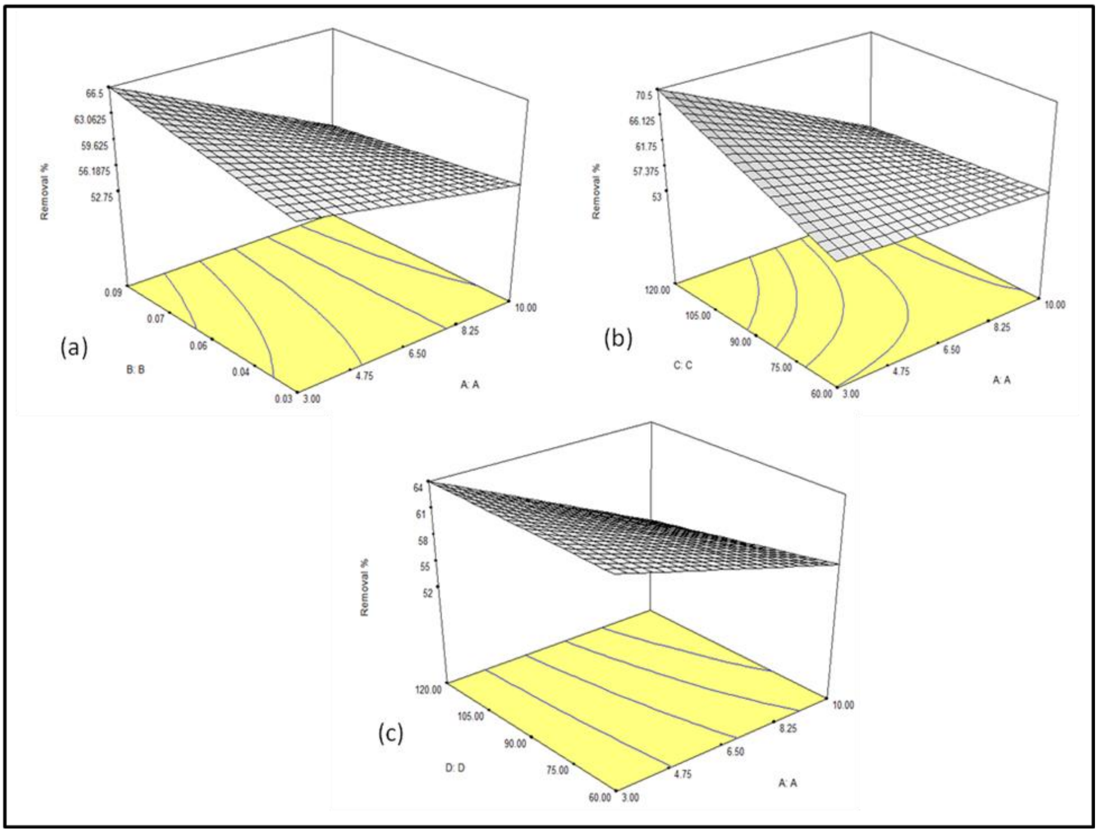

Fig. 3: (a) A 3-D interaction plot of the removal of Zn^{2+} using AC, interaction of AC dosage and pH, (b) A 3-D interaction plot of the removal of Zn^{2+} using AC, interaction of agitation time and AC pH, (c) A 3-D interaction plot of the removal of Zn^{2+} using AC, interaction of agitation speed and pH of AC.

Characterization of magnetic biochar:

The FESEM technique was employed to observe the surface physical morphology of the prepared MB and commercial AC samples. Figure 4 (a-b) AC and (c-d) magnetic biochar at different magnifiation scale. Figure 4(a-b), there was very little pores available on the surface of the precussor.Figure 4(c-d) produced magnetic biochar at optimum condition,pores of different size and different shape wee developed as observed. The microporosity is opened and widened with a shift to meso- and macroporosity as the removal of the exterior of the particle is significant at high burn-offs. This reflects that N_2 is effective in creating well developed pores on the surfaces of the precursor, hence leading to novel magnetic biochar with large surface area and porous structure, and the compactness of the shell structure is seen to have high adsorption capacity on the removal of Zn^{2+} from aqueous solution.

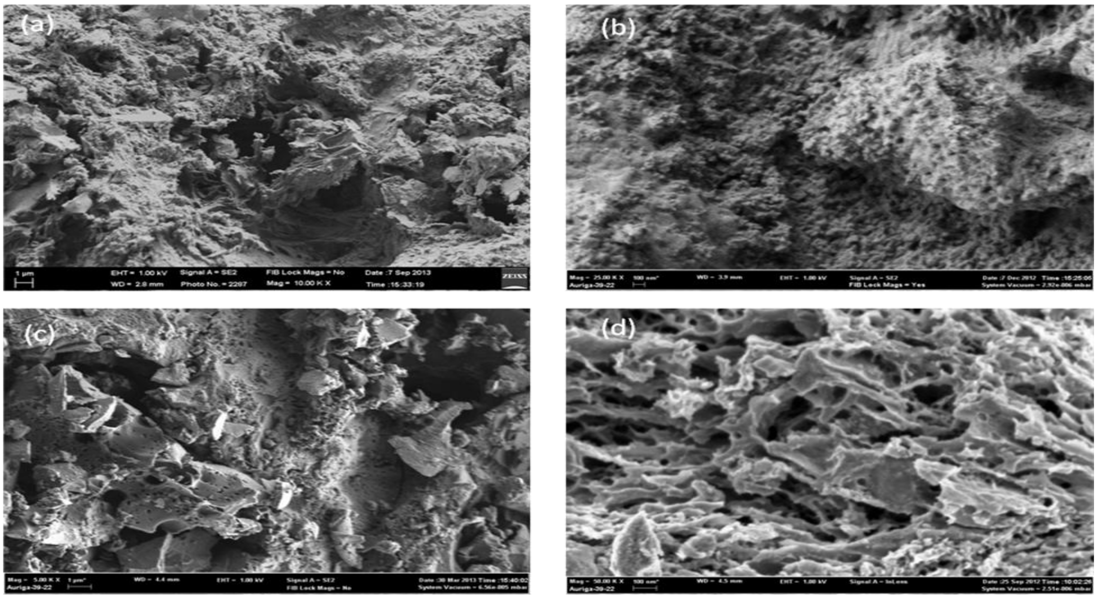

Fig. 4: FESEM image of (a-b) AC, (c-d) magnetic biochar.

Conclusions:

The effectiveness of Zn2+ removal from aqueous solution by different adsorbents was experimentally studied. The effect of various factors, namely pH, adsorbent dosage, agitation speed and contact time was statistically studied through analysis of variance (ANOVA). Statastical analysesis revealed that the ptimized condition are The statistical analysis revealed that that optimum condition for magnetic bochar and AC was at pH 10, dosage 0.09 g, agitation speed and time of 120 rpm and 60 min respectively. It is found that the removal efficiency of Zn^{2+} for an initial concentration of 10 mg/l using magnetic biochar was 95% and using activated carbon was 65%. Hence, magnetic biochar emerges as the most promising adsorbent in the removal of Zn^{2+} compared to activated carbon

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