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OPTIMUM ADSORPTION OF LEAD FROM WASTE WATER USING BEANS HUSK

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Abstract: This study is aimed at determining the optimum adsorption of lead from waste water onto activated carbon prepared from bean husk. The optimization was designed using response surface methodology. Box–Behnken design was employed to generate a matrix and the factors considered were pH (2–10), temperature (25–65 °C) and contact time (20–120 minutes). It generated 12 experimental runs and the selected responses were adsorption capacity and removal efficiency. Run nine (9) gives the highest adsorption capacity (19.8 mg/g) and removal efficiency (99%) while run five (5) gives the lowest adsorption capacity (7 mg/g) and remover efficiency (33%). The result indicates that the Optimum condition for the adsorption Pb (II) from waste water were pH (10), temperature (65^{0}C) and Contact time (120 minutes). This gave Adsorption Capacity of 19.940mg/g and 99.698% Removal Efficiency of Pb from waste water. There was good agreement between experimental value and predicted value. The study also showed that activated carbon from beans husk is an effective adsorbent for the removal of Pb (II) from waste water.
Keywords: Beans husk, Optimum, Adsorption capacity, Removal efficiency

INTRODUCTION

Discharge of pollutants containing heavy metals into water systems is one of the most serious environmental problems globally (Das et al., 2007; Ibrahim et al., 2010). With the rapid industrialization in developing and developed countries, large volumes of wastes containing heavy metals are generated and directly or indirectly discharged into water ecosystems thus posing significant danger to human health.

Pollutants enter aquatic systems via numerous pathways, including metal finishing, electroplating, painting, dying, photography, surface treatment and printed circuit board manufacture (Papageorgiou et al., 2006). Heavy metals can also enter water bodies via mining activities, agricultural run–off and domestic effluent which lead to increase in metallic species released into the environment (Churong et al., 2013). The presence of toxic and polluting heavy metals in wastewaters from industrial effluents, water supplies and mine waters and their removal has received much attention in recent years. The number of heavy metals that industrial wastewaters often contain is considerable and would endanger public health and the environment if discharged without adequate treatment.

Among all the water pollutants, heavy metal contaminations are posing a serious threat for human society. Heavy metal is a general collective term applying to the group of metals and metalloids with an atomic density higher than 6 g cm^{-3}. However, it is only a loosely defined term, which is widely recognized and usually applied to the metal elements associated with pollution and toxicity problems. Three categories of heavy metals viz. toxic metals, precious metals and radionuclides are of environmental concern. Substantial amount of various toxic metals is released into water system by many types of industries, such as mining and smelting of minerals, the surface finishing industry, energy and fuel production, fertilizer and pesticide industry and subsequent application, metallurgy, iron and steel, electroplating, electrolysis, electro–osmosis, leatherworking, electric appliance manufacturing, photography, aerospace and atomic energy installation etc. For example, mining industries release heavy metal ions such as lead (Pb (II)), mercury (Hg (II)), silver (Ag(I)), chromium (Cr (III)), arsenic (As (V)), cadmium (Cd (II)), palladium (Pd (II)), zinc (Zn (II)) and aluminum (Al (III)) to the environment.

Water pollution by heavy metals has been a major concern for chemists and environmental engineers (Ekpete, 2017). Heavy metals are of concern because of their toxicity, bio–accumulating tendency, threat to human life and the environment (Dorris et al., 2000).

Throughout history, human progress has depended on access to clean water and on the ability of societies to harness the potential of water as a productive resource (HDR, 2006). Water for life in the household and water for livelihoods through production are two of the foundations for human. Therefore, there is a growing concern that the world is facing a crisis of shortage of clean water that if left unchecked, will derail progress towards the Millennium Development Goals and hold back human development.

In cooperation with the U.S. Environmental Protection Agency, the Agency for Toxic Substances and Disease Registry (ATSDR) has compiled a Priority List for 2011 called the ATSDR 2011 Substance Priority List. Based on the list, lead is ranked as second hazardous heavy metals among the substances after arsenic (ATSDR, 2011).

Lead is of concern because once it gets into the environment, it bio–accumulate and bio–magnify as it go through the tropic levels of the food chain. Furthermore, metals being inorganic, they are non–biodegradable. It is

therefore important that they are excluded from circulation in the ecosystem due to various neurological, reproductive and systemic impacts on humans and negative impacts on other animals especially the aquatic species.

Conventional physicochemical methods for metals remediation include chemical precipitation, filtration, coagulation, evaporation, ion exchange, membrane separation and solvent extraction. However, application of such processes is always expensive and ineffective in terms of energy and chemical products consumption, especially at low metal concentrations of 1–100 mg/L (Bian et al., 2015). Therefore, there is a great need for an alternative technique, which is both economical and efficient. Adsorption has been shown as the most appealing as an economic and environmentally friendly procedure to remove heavy metals in wastewater (Ahmad et al., 2009). Activated carbon is the most popular material used as an adsorbent. However, it is quite expensive. The search for alternative adsorbents to replace the costly activated carbon is highly encouraged. Adsorption, based on live or dead adsorbent, has been regarded as a cost–effective biotechnology for the treatment of complex wastewater containing heavy metals at high volume and low concentration (Amboga et al., 2014).

In addition, converting beans husk into value added products such as adsorbents would serve as a way to mitigate the disposal challenges posed by this waste materials to the environment. Also, the industries involved in conversion of this waste materials would serve as an indirect way of revenue generation and simultaneously for job creation. This research is part of that process of developing an alternative technology for utilizing cheap effective and available adsorbent for the adsorptive removal of lead from wastewater.

Optimization using response surface methodology can be used to determine the optimum conditions involved in a process (Onu et al., 2014 and Ositadinma et al., 2019). It is different from the method of one factor at a time (OFAT) which involves keeping all other parameters constant while varying one factor. OFAT method uses a large number of experiments in determining the optimum condition. It is time consuming and does not show the interactive effects of the independent factors unlike optimization using response surface methodology (RSM). Design of experiment using RSM is an enhanced systematic experimentation that takes into consideration all the process parameters involved simultaneously (Onu et al., 2014 and Ositadinma et al., 2019).

Hence the aim of this work is to use response surface methodology to optimize the process parameters for the optimum adsorption of lead from waste water onto activated carbon prepared from bean husk.

MATERIALS & METHOD

Materials

The beans husk was collected from the local market in Oyingbo, Lagos, Nigeria.

Apparatus and Reagents used

The following apparatus and reagents were used: analytical grade hydrochloric acid (Epoxy Oilserv, 30% w/w purity); analytical grade sodium hydroxide (Epoxy Oilserv, 98% purity); analytical grade Lead (II) nitrate (Indian Platinum, 98% purity); distilled water; Rotary shaker (Bioevopeak SHK–O0310111, China); Weighing balance (AL Mettler Toledo GmbH); Furnace (Bioevopeak FNC–TB1700, China); Oven (Gallenkamp, England); pH meter (HANNA Instrument pHep®); Beaker (Pyrex, England); Conical flask (Pyrex, England); Measuring cylinder (Pyrex, England); Atomic absorption Spectrophotometer (Perkin Elmer Analyst 200); Scanning electron Microscope (Model Jeol–JXA 840 A, Japan); Water bath shaker (Grant OLS 200); Filter paper (Whatman)'Stop watch (Electronic Timer,TIME–Q118, China); Sieve (B.S.S. 200–100); FTIR Spectrometer (Nicolet Avator 330, England).

Adsorbent Preparation

The beans husk was prepared by adopting the method of Ositadinma et al., (2019). It was washed thoroughly with distilled water to remove dust and soil, dried in sunlight for 2 days and kept in an oven at 70°C for 24hours.

Carbonization

The carbonization process was done by the procedure adopted by Sandip et al., (2017). The beans husk was heated in the Muffle Furnace at 450°C for 30min then permitted to cool. The beans husk was then crushed with blender and sieved to a size smaller than 850 µm. The yield of carbon is defined as the ratio of final weight of the obtained product after carbonization to the weight of dried precursor initially used was calculated using:

$$\text{Yield}(\%) = \frac{\text{product}}{\text{reactant}} \times 100 \quad (1)$$

Activation

The activation process was done by the procedure adopted by Hanum et al., (2017). The carbonized beans husk was impregnated with 1M HCL at carbon to acid ratio of 1:3(w/v) for 24 hours. Afterwards it was placed in a furnace and heated at 650°C for 30minutes. The resulting sample was allowed to cool and watched with distilled water until neutral pH was reached.

Moisture Content

1 g of activated carbon was placed oven and heated at 105–110°C for 1.5 hr (Hanum et al., 2017). Then, sample was cooled in and the weight of dried sample was measured. Moisture content was calculated as follow:

$$\text{M} = \frac{\text{weight of dried sample}}{\text{weight of original sample}} \times 100 \quad (2)$$

Ash Content

1 g of activated carbon was heated in a muffle furnace at 750°C for 1.5 hr hr (Hanum et al., 2017).

The sample was cooled and the weight of the ash was measured

$$A = \frac{\text{weight of ash sample}}{\text{weight of sample}} \times 100 \quad (3)$$

Scanning Electron Microscope (SEM)

To determine the surface morphological composition of the prepared adsorbent. The SEM analysis was carried out at the magnifications X–900, and X–10,000.

Adsorbate

Lead (II) nitrate (Indian Platinum, 98% purity) was used as the adsorbate and was obtained from luth Lagos. It was prepared by dissolving 1 g of Pb(NO3)2 in 1 litres of distilled water.

Design of Experiment

Design Expert 13 was used to design the experiment. It was employed to check for the interdependence of more than one factor by identifying their overall effect (Olufemi et al.,2018). Box–Behnken design was employed.

Box–Behnken design

The main factors (pH, temperature and contact time) were selected, as well as their factor levels, coded as –1 (low) and +1 (high), as seen in Table 3.2 Box–Behnken design was employed and a matrix generated. It generated 12 experimental runs. The selected responses were adsorption capacity and removal efficiency.

Table 1. Input factors with their code levels using Box–Behnken design

Factors	Units	Low	High
Ph	–	2	10
Time	Min	20	120
Temperature	°C	25	65

Batch adsorption Process

2 g of beans husk derived activated and 40ml of 1000 mg/L lead solution was fixed in all the batch sorption experiment on a water bath shaker (Grant OLS 200) at 120rpm. Process optimization was done by altering the pH (2–10), contact time (20–120 minutes) and temperature (25–65°C). The final concentration of lead was determined through the use atomic absorption Spectrophotometer (Perkin Elmer Analyst 200). The % removal of Lead (II) ion and the adsorption capacity of the beans husk was calculated using the following equation,

$$q_e = \frac{C_0 - C_t}{M} \times V \quad (4)$$

$$\text{Removal (\%)} = \frac{C_0 - C_t}{C_0} \times 100 \quad (5)$$

M = mass of activated carbon in gram
V = volume of test solution in liter
C_o = initial concentration of lead
C_t = final concentration of lead
q_e is the amount of solute removed or adsorbed

RESULTS & DISCUSSION

Physical Properties of the Adsorbent

Table 2 presents the physical properties of the adsorbent with values of the ash content, moisture content and pH.

Table 2. Physical Properties Beans Husk Adsorbent

Properties	Bean Husk
Ash Content (%)	3.5
Moisture Content (%)	8.8
pH	6.8

Scanning Electron Microscopy (SEM)

Scanning electron microscopy has been extensively used to study the surface morphology of the Activated carbons.

The SEM images of the HCL impregnated activated carbon before and after adsorption are shown in the figures below. The SEM analysis was carried out at a magnification of 9,000X Before adsorption and 8,000X after adsorption, the surface morphology of activated carbon has uneven cavities and fine open pores which indicate its ability to absorbed metal ions from wastewater.

The large pores observed is due to the fact the activating agents promote the contact area between the carbon and the activating agent. The HCL activated carbon clearly showed partially developed honey comb like highly defined pores and cavities in its surface. However, the pores are not–uniform.

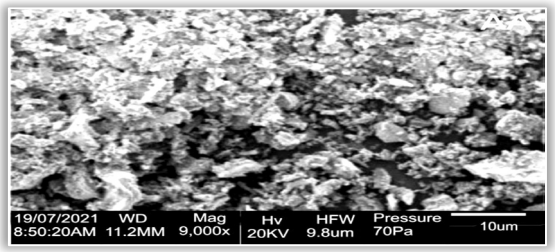

Figure 1. SEM of beans husk before Adsorption

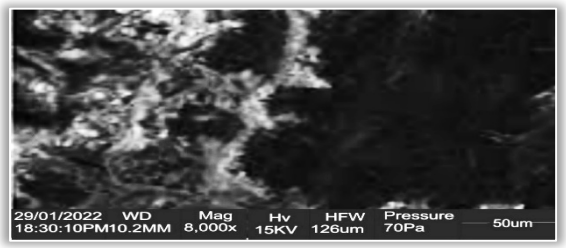

Figure 2. SEM of beans husk after Adsorption

During carbonization process, pores are developed in the carbon and promote the diffusion of HCL molecules into these pores and thereby increase the HCL–carbon reactions which would then create more pores in the activated carbon. This would enhance the surface area and pore volume of the activated carbon so prepared. The SEM image after adsorption shows smaller pores as a result of adsorption Pb on the beans husk.

Optimization Process

The result of the experimental runs in the optimization process indicated that the best adsorption conditions are at pH of 6, contact time of 120 minutes and temperature of 65°C. This gave the highest adsorption capacity of 19.8(mg/g) and removal efficiency lead. The result

equally revealed that the three factors optimized have great effect on the adsorption of lead from waste water.

The model summary values suggested that a linear model best fitted the optimization process. The R–squared values for the quadratic and 2FI models is slightly greater than of linear. But we focus on the model maximizing the adjusted R^2 predicted R^2. The quadratic model was aliased and aliases are false signals of any sort present hence the linear model was suggested.

Table 3. Experimental Design Matrix for the Optimization Studies

Std	Run	pH	Time (Min)	Temperature (°C)	Adsorption Capacity (mg/g)	Removal Efficiency (%)
4	1	10	120	45	18.2	91
6	2	10	70	25	18.5	92.5
10	3	6	120	25	17.6	88
3	4	2	120	45	12	60
7	5	2	70	65	7	35
11	6	6	20	65	13.6	68
1	7	2	20	45	12.4	62
2	8	10	20	45	16	80
12	9	6	120	65	19.8	99
8	10	10	70	65	16.8	84
5	11	2	70	25	10	50
9	12	6	20	25	9.6	48

Table 4. Model Summary Statistics for adsorption capacity

Source	Std. Dev.	R^2	Adjusted R^2	Predicted R^2	
Linear	2.63	0.7027	0.5913	0.3312	Suggested
2FI	3.24	0.7184	0.3805	–0.6219	Not Suggested
Quadratic	3.79	0.7687	0.1521	–2.7000	Aliased

Table 5. Model Summary Statistics for removal efficiency

Source	Std. Dev.	R^2	Adjusted R^2	Predicted R^2	
Linear	13.16	0.7027	0.5913	0.3312	Suggested
2FI	16.20	0.7184	0.3805	–0.6219	
Quadratic	18.95	0.7687	0.1521	–2.7000	Aliased

Analysis of Variance (ANOVA) for Adsorption Capacity and Removal Efficiency

The ANOVA in Table 5 and 6 was used to analysis the result and validate the adsorption model.

Table 6. ANOVA for Adsorption Capacity

Source	Sum of Squares	Df	Mean Square	F–value	p–value	
Model	130.98	3	43.66	6.30	0.0168	significant
A–pH	98.70	1	98.70	14.25	0.0054	
B–Contact time	32.00	1	32.00	4.62	0.0638	
C–Temperature	0.2813	1	0.2813	0.0406	0.8453	
Residual	55.41	8	6.93			
Cor Total	186.39	11				

Table 7. ANOVA for Removal Efficiency

Source	Sum of Squares	Df	Mean Square	F–value	p–value	
Model	3274.56	3	1091.52	6.30	0.0168	significant
A–pH	2467.53	1	2467.53	14.25	0.0054	
B–Contact time	800.00	1	800.00	4.62	0.0638	
C–Temperature	7.03	1	7.03	0.0406	0.8453	
Residual	1385.17	8	173.15			
Cor Total	4659.73	11				

The lack of fit test and the adequacy of the regression models were equally performed. A significance level of 5% was used hence P–values greater than 0.05 are considered insignificant while those at 0.05 or less are significant. Hence, only the interactions of A, B and C are significant. The model F–value of 6 implies that the model is significant agreeing with the P–value being less than 0.05. There is only a 1.68% chance that an F–value this large could occur due to noise. There is only a 1.68% chance that an F–value this large could occur due to noise. The P values check the significance of the factors and equally help to understand the pattern of the mutual interactions between the test variables (Shrivastava, 2008).

Optimum Model Equations

The generated model equations for the adsorption process in terms of coded factors are:

$$\text{Adsorption Capacity (mg/g)} = 4.29+3.51A+2B+0.1875C \quad (6)$$

$$\text{Removal Efficiency (\%)} = 71.46+17.56A+10B+0.9375C \quad (7)$$

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as –1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients. The positive sign of a factor indicates that there will be increase in the response when there is an increase in the factor while negative sign will lead to decrease in the response (Kumar, 2008). The generated model equations for the adsorption process in terms of actual factors are:

$$\text{Adsorption Capacity (mg/g)} = 5.80104+0.878125A+0.040B+0.009375C \quad (8)$$

$$\text{Removal Efficiency (\%)} = 29.00521+4.39063A+0.2000B+0.046875C \quad (9)$$

The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor. This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space.

Diagnostics Case study

Table 7 shows the diagnostic case study of adsorption capacity and removal efficiency of Pb.

Table 8. Diagnostic case study of adsorption capacity and removal efficiency of Pb

Run Order	Adsorption Capacity			Removal Efficiency		
	Actual Value	Predicted Value	Residual	Actual Value	Predicted Value	Residual
1	18.20	19.80	–1.60	91.00	99.02	–8.02
2	18.50	17.62	0.8833	92.50	88.08	4.42
3	17.60	16.10	1.50	88.00	80.52	7.48
4	12.00	12.78	–0.7792	60.00	63.90	–3.90
5	7.00	10.97	–3.97	35.00	54.83	–19.83
6	13.60	12.48	1.12	68.00	62.40	5.60
7	12.40	8.78	3.62	62.00	43.90	18.10
8	16.00	15.80	0.1958	80.00	79.02	0.9792
9	19.80	16.48	3.32	99.00	82.40	16.60
10	16.80	17.99	–1.19	84.00	89.96	–5.96
11	10.00	10.59	–0.5917	50.00	52.96	–2.96
12	9.60	12.10	–2.50	48.00	60.52	–12.52

The residual values represent the closeness of actual to the predicted value. When the predicted value is greater than the actual, there will be a negative residual but when the value of actual is greater than the predicted, we have a positive residual.

Error Graph

The Predicted vs Actual plot in Fig. 3 and the Normal plot of Residuals in Fig. 4 were used to determine if the residuals follow a normal distribution.

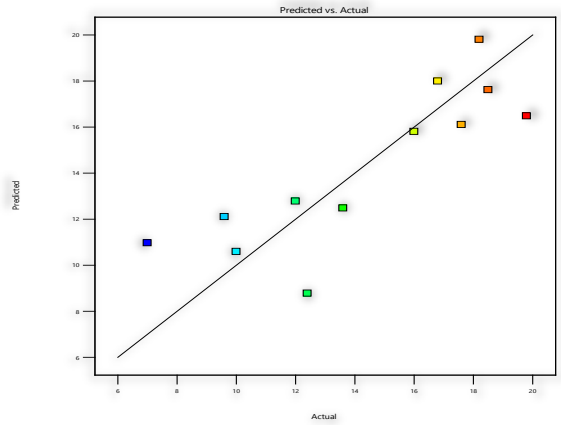

Figure 3. Predicted vs Actual plot

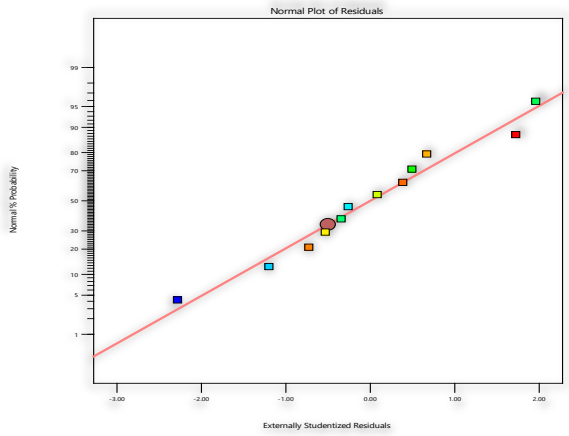

Figure 4. Normal plot of Residuals

It is assumed to have followed a normal distribution as the points closely aligned to the straight line of the plot thereby confirming the good relationship between the experimental values and the predicted values of the response and the adequacy of the suggested model in predicting the response variables in the experimental values (Ositadinma, et al., 2019). From the plot of predicted versus actual, the closer the points to the normal line, the greater the R–squared and vice–versa

Model Graph

The 3–D response surface plots are graphical representation of the interactive effects of any two variables factors. Response surface estimation serves as a function of two factors at a time, maintaining other factors at fixed levels. This is more helpful in understanding both the main and the interaction effects of those two factors. These plots can be easily obtained by calculating from the model, the values taken by one factor where the second varies with constraint of a given response value. The response surface curves were plotted to understand the interaction of the variables and to determine the optimum levels of each variable for maximum response. The nature of the response surface curves shows the interaction between the variables. The elliptical shape of the curve indicates good interaction of the two variables and circular shape indicates no interaction between the variables (Ositadinma et al., 2019). There was a relative significant interaction between every two variables, and there was a maximum predicted efficiency as indicated by the surface confined in the smallest ellipse in the contour diagrams. It was also observed from contour and 3D representation that increase in contact time, temperature and pH increases adsorptio capacity and removal efficiency.

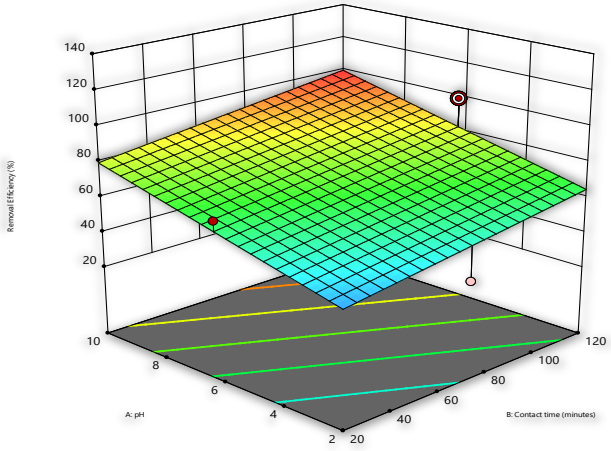

Figure 4. Interactive effect of pH and contact time

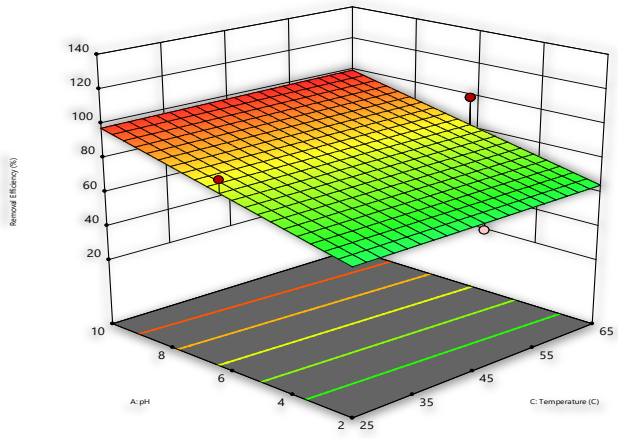

Figure 5. Interactive effect of pH and temperature

Numerical Operation Studies on Adsorption Capacity and Removal Efficiency

The optimization study for adsorption capacity and removal efficiency was obtained from Design expert software (13). The three selected factors which are pH, Temperature, and Contact time were all set to "maximize" with their respective upper and lower limit as shown in table 8. Optimum value suggested for pH was

10, temperature value was 65°C and Contact time was 120 minutes. There was agreement between actual and predicted value.

Table 9. Selected factors used for optimization showing their respective ranges

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A: pH	maximize	2	10	1	1	3
B: Contact time	maximize	20	120	1	1	3
C: Temperature	maximize	25	65	1	1	3
Adsorption Capacity	maximize	7	19.8	1	1	3
Removal Efficiency	maximize	35	99	1	1	3

CONCLUSIONS

Optimization of factors for the adsorption Pb (II) was successfully carried out using the Box–Behnken design in the design of expert. 12 experimental runs were generated. run nine (9) with experimental condition of (65°C, 120 mins and pH 6) gives the highest adsorption capacity and removal efficiency of 19.8(mg/g) and 99% respectively while run five (5) with experimental condition of (65°C, 70mins and pH 6) gives the lowest adsorption capacity and remover efficiency of 7(mg/g) and 33% respectively. A linear model with a high correlation coefficient was suggested in describing the interactive effects of the process parameters The numerical values for the optimum adsorption of Pb (II) from waste water was optimized to be pH (10), temperature (65°C) and Contact time (120 minutes). There was agreement between actual and predicted optimization value. The Activated carbon was prepared from beans husk via the chemical method using HCL as activating agent and was also characterized to determine the basic properties and surface morphology of activated carbon. The ash content, moisture content and pH value of the beans activated carbon are 3.5%, 8.8% and 6.8 respectively. It can be concluded that a waste material like beans husk is an effective and suitable adsorbent for removing Pb (II) ion from aqueous solution, and a probable cost–effective adsorbent for treating Pb (II) contaminated water.

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