

PREPARATION, CHARACTERIZATION AND EVALUATION OF OPTIMAL ACTIVATED CHARCOAL DERIVED FROM PLUM SEED COATS FOR THE REMOVAL OF LEAD (II) IONS^{*}

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ABSTRACT

In this study, the removal of lead by adsorption using charcoal of plum seed coats was conducted. The percent adsorptions of lead (II) ions on four different sizes of charcoal samples were determined. The optimum size (40 mesh) of the charcoal powder sample was observed on the basis of time duration to achieve the completeness and percent adsorption for lead ion. The FT-IR spectral measurements were done before and after adsorption of lead ion to know the efficiency of each particle size of the charcoal powder sample. The adsorption of Pb^{2+} on plum seed coats charcoal follows Freundlich isotherm. The straight line with value of slope equal to 1/n and log k as y-axis intercept was obtained. Lead is a highly toxic heavy metal and exposure to which can produce a wide range of adverse health effects. Adsorption is the adhesion of atoms, molecules, or ions from a gas, liquid or dissolved solid to a surface charcoal from nut shells and husks are particularly good as adsorbents. Hence, the activated charcoals are now widely used to remove pollutants from waste water by adsorption processes.

Keywords : Lead (II) ion, FT-IR spectrum, plum seed coats charcoals, adsorption capacity

1. INTRODUCTION

Humans have used water since the dawn of history, but the realization of its importance and of its danger to health is a relatively recent development. The extent of the human activities that influence the environment has increased dramatically during the past few decades and terrestrial ecosystems, fresh water and even the atmosphere are affected. The scale of socio-economic activities, urbanization, industrial operations, and agricultural production has reached the point where they also have a world-wide impact on water resources.[1]

Heavy metals such as lead can often be found in industrial waste water from sources such as smelling, electroplating, printing, metallurgy, and manufacturing of storage batteries, chemicals, paints, photographic materials, fuels, matches, explosives, etc., remains a matter of serious concern because of related health issues. Lead is a highly toxic heavy metal and exposure to which can produce a wide range of adverse health effects. Both adults and children can suffer from the effects of lead poisoning, but childhood least poisoning is much more frequent.[2]

Therefore, environmental engineers and scientists have investigated the method to treat the heavy metal-bearing waste water effectively and economically. Although free aqueous phase lead species can be removed by traditional techniques such metal precipitation, the presence of EDTA renders such processes either partially or completely in effective due to the formation of metal-chelate complex. In addition, several investigators report lead removal using different carbon based adsorption materials including the activated charcoals.[3]

The uses of activated charcoals are now widely used to remove pollutants from waste water by adsorption processes. Since commercially available activated charcoal is expensive, special attention on the preparation of activated carbon from several agricultural by-products has been given due to the growing interest in low cost activated charcoal from renewable agricultural resources. Many researchers have produced activated charcoal from natural resources such as bagasse, cassava peel, jute fiber, rice husk, coconut shell, etc.[4]

The advantage of using inexpensive natural resources as raw materials for manufacturing activated charcoal is that these raw materials are potentially less expensive to manufacture. The main purpose of this work is to evaluate the efficiency of activated charcoal produced from plum seed coat (seed husk) in the removal of aqueous lead species.

2. MATERIALS AND METHODS

2.1 Collection of raw material

In order to prepare charcoal, the raw material, the seed coats of plum seeds were collected from Singu village, Nyaung Oo Township, Mandalay Region, Myanmar. They were obtained as the by-products from the manufacturing of plum kernei for exportation.

2.2 Preparation of Sample Charcoal

The collected plum seed coats were firstly cleaned by washing with water and dried. In this way, the fleshy parts of plum fruit were completely removed.

Then, the dried raw materials were carbonized by heating in an earthen pot for about an hour at a brazier. The resulting carbonized products, i.e., charcoals were sprayed with water, dried in the desiccator and ground in motor with pastel. The resulting powdered samples were sieved to get four sizes. The size sieving was accomplished by using sieves (ENDECOTTS Ltd. London).

Finally, the sieved charcoal samples were activated by washing with boiled water, filtering and drying again.

2.3 Determination of the size of plum seed coats charcoal

Powdered and activated plum seed coats charcoal sample was sieved with different size mesh of the gauzes which are known to be 120 (+ 120) mesh, 80 (80 - 60) mesh, 60 (60 - 80) mesh, and 40 (40 - 60) mesh respectively.

2.4 Determination of the Optimum Particle Size for the Adsorption

2.4.1 Preparation of solutions

Exactly 10 g of pure $Pb(NO_3)_2$ were weighed in a weighing beaker and poured into a 500 ml volumetric flask. The weighing beaker was then rinsed with distilled water and poured into the volumetric flask containing $Pb(NO_3)_2$. The rinsing and pouring was repeated several times. About 100 ml of distilled water was added into this volumetric flask and shaken thoroughly to dissolve the $Pb(NO_3)_2$.

Finally, the required distilled water was filled up to the mark of the flask and mixed. Similarly 10 g of KI was weighed and dissolved with distilled water in a 1000 ml volumetric flask to obtain 1000 ml KI solution.

2.5 Determination of percent adsorption

Firstly, 50 ml of $Pb(NO_3)_2$ solution was placed in a beaker and then the Pb^{2+} content was precipitated as PbI_2 by treating with large excess of KI solution. The PbI_2 precipitate obtained was filtered by using a filter paper of know weight. The filtrate was again precipitated with KI solution and PbI_2 precipitate was again filtered. The precipitation and filtration were repeated again and again until no more precipitates were observed. The resulting PbI_2 precipitate with filter paper was dried in desiccator until no further weight loses. Then, the weight of PbI_2 and hence of Pb^{2+} was determined as the blank (without adsorption) weight of Pb^{2+} before adsorption.

Secondly, 10 g of 120 mesh charcoal sample was weighed and packed into a column. 50 ml of $Pb(NO_3)_2$ solution was poured down into the column which has already been packed with 120 mesh charcoal sample. Eluate was collected in a conical flask. When all the poured $Pb(NO_3)_2$ solution was completely eluted, from the resulting eluate, all the Pb^{2+} content was precipitated with KI solution as in the above procedure. The resulting PbI_2 precipitate was dried and weighed. In this way, the weight of Pb^{2+} after adsorption with 10 g of 120 mesh charcoal column. This procedure was also done in three times simultaneously by using columns of same size and average weight was determined.

Elution of 50 ml of $Pb(NO_3)_2$ solution was also done by using 80 mesh, 60 mesh, 40 mesh charcoal samples of same weight packing in the columns of same size respectively. In these processes, the procedure for each size charcoal was also repeated into three times and the average weight of Pb^{2+} was determined.

Finally, for each size of sample, adsorbed weight of Pb^{2+} was obtained from the weights of Pb^{2+} before and after adsorption and percent adsorption for each size of sample was calculated.

2.6 Determination of optimum size sample

From the values of percent adsorption and time taken for adsorption, the optimum size of charcoal for the adsorption was determined.

2.7 Examination of Charcoal Samples by FT-IR Spectrum

In order to confirm the adsorption of Pb^{2+} by charcoal sample, the FT-IR spectrum of each charcoal sample before and after adsorption were measured at the Department of Chemistry, University of Mandalay and comparison between the spectrum before adsorption and that after adsorption for each size of sample.

2.8 Determination of the Adsorption Capacity of the Pb²⁺ on Compromise Size of Plum Seed Coats Charcoal by Repeated Process

2.8.1 Procedure

Plum seed coats charcoal sample 10 g of compromise size (40 mesh) was accurately weighed and it was packed into the column. Then 50 ml of $Pb(NO_3)_2$ solution was poured down into the column. Remaining tasks of experiment were identical with that of the procedure

mentioned in above.

This procedure was recycled from the addition of 50 ml of $Pb(NO_3)_2$ solution into the column. The difference was only that adsorbent used in this procedure was not fresh, instead it was used repeatedly for each of experiments. Repeatation was performed for further five times.

Finally, for compromise size (40 mesh) of sample, adsorbed weight of Pb^{2+} was obtained from the weights of Pb^{2+} before and after adsorption and percent adsorption for compromise size of sample was calculated.

2.9 Verification of the Validity of Freundlich Isotherm

2.9.1 Procedure

Run I

2.2500g of Pb(NO₃)₂ was dissolved in distilled water to obtain 100ml of 2.25×10^{-2} gml⁻¹.

Step (1)

25ml of Pb(NO₃)₂ solution was placed in a conical flask. 25ml of distilled water was mixed. Then 3 drops of xylenol orange indicator was added. The solution turned red. Then about 10 ml of dilute nitric acid was added cautiously and with stirring, until the solution acquires a yellow colour. Now powdered hexamine was added until the colour is intensely red. This step ensures that the solution has the correct pH (about 6) for the subsequent titration. This solution was titrated with EDTA solution. When the colour changes to lemon yellow, the titration reached end point. Above procedure (step-1) was set as the blank titration.

Step (2)

30 ml of Pb(NO₃)₂ solution was poured down into the column packing with 10 g of 40 mesh plum seed coats charcoal sample. From this adsorption, eluate was obtained. In the resulting eluate, 25 ml of eluate was mixed with 25 ml of distilled water. Then the remaining tasks of experiment were described with that of the procedure mentioned in step (1). The amount of Pb²⁺ adsorbed on the plum seed coats charcoal sample was the difference between data from step (1) and step (2).

Run-II through Run-V

Procedure for each run was conducted as the same manner as mentioned in Run-I. Firstly, 2.2500 g of Pb(NO₃)₂ was dissolved in distilled water to obtain 100 ml of 2.2500×10^{-2} gml⁻¹ of Pb(NO₃)₂ solution. Then, the Pb(NO₃)₂ solution was diluted with distilled water to obtain the concentration 1.1250×10^{-2} gml⁻¹, 0.5625×10^{-2} gml⁻¹, 0.2813×10^{-2} gml⁻¹ and 0.14063×10^{-2} gml⁻¹ for runs, II, III, IV and V respectively. Just before the experiment for each run, the EDTA solution was standardized with diluted standard Pb(NO₃)₂ solution.

2.10 Determination of Adsorption Capacity of Reactivated Charcoal Sample

15 g of used plum seed coats charcoal sample was weighed and washed three times using totally 200 ml of distilled water. Then, the washed charcoal sample was dried and heated to reactivate.

Finally, 10 g of reactivated charcoal sample was packed into the column of the same size. The adsorption capacity of reactivated charcoal sample could be determined by using similar procedure. In addition, 15 g of used plum seed coats charcoal sample was boiled with 200 ml of distilled water for about 15 min. Then, the boiled charcoal sample was filtered, dried, and heated to reactivate.

3. RESULTS AND DISCUSSION

3.1 Results of Preparation of Charcoal

The activated charcoal powders were obtained by washing the powdered charcoal samples with boiling water, and drying.

3.2 Results of the Determination of Percent Adsorption

The mass of PbI_2 precipitate obtained from the precipitation of 50 ml of blank $Pb(NO_3)_2$ solution, i.e., the solution without adsorption with charcoal were given in the Table (1).

No.	Volume of blank solution	Precipitant	Mass of PbI ₂ precipitate (g)
1.	50 ml of Pb (NO ₃) ₂ solution	Excess of KI solution	1.3000
2.	50 ml of Pb(NO ₃) ₂ solution	Excess of KI solution	1.4000
3.	50 ml of Pb(NO ₃) ₂ solution	Excess of KI solution	1.4000

Table (1) The Mass of PbI₂ Precipitate Obtained from the Blank Solution

This means that the mass of PbI_2 before adsorption was assumed as 1.4000 g. The mass of PbI_2 precipitate obtained from the precipitations of solutions which were obtained from adsorption of each 50 ml of Pb (NO₃)₂ solution with each 10 g of plum seed coats

charcoal sample of 120 mesh, 80 mesh, 60 mesh, and 40 mesh sizes respectively were given in Table (2), (3), (4) and (5).

No.	Volume of Pb(NO ₃) ₂ (eluant) solution (ml)	Mass of adsorbent charcoal (120 mesh) (g)	Precipitant	Mass of PbI ₂ , precipitate (g)
1.	50	10	excess of KI	0.1000
2.	50	10	excess of KI	0.1050
3.	50	10	excess of KI	0.1050

Table (2) The Mass of PbI₂ Precipitate Obtained from Eluate Solution After Adsorption with Charcoal Sample of 120 Mesh Size

This result shows that the mass of PbI₂ after adsorption with charcoal sample of 120 mesh size was 0.1050 g.

Table (3) The Mass of PbI₂ Precipitate Obtained from Eluate Solution After Adsorption with Charcoal Sample of 80 Mesh Size

No.	Volume of Pb(NO ₃) ₂ (eluant) solution (ml)	Mass of adsorbent charcoal (80mesh) (g)	Precipitant	Mass of PbI2 precipitate (g)
1.	50	10	excess of KI	0.1998
2.	50	10	excess of KI	0.2000
3.	50	10	excess of KI	0.2000

It shows that the mass of PbI₂ after adsorption with charcoal sample of 80 mesh size was 0.2000 g.

Table (4) The Mass of PbI₂ Precipitate Obtained from Eluate Solution After Adsorption with Charcoal Sample of 60 Mesh Size

No.	Volume of Pb(NO ₃) ₂ (eluant) solution (ml)	Mass of adsorbent charcoal (60 mesh) (g)	Precipitant	Mass of PbI ₂ precipitate (g)
1.	50	10	excess KI	0.3951
2.	50	10	excess KI	0.4000
3.	50	10	excess KI	0.4000

The result states that the mass of PbI₂ precipitate after adsorption with charcoal sample of 60 mesh size was 0.4000 g.

Table (5) The Mass of PbI₂ Precipitate Obtained from Eluate Solution After Adsorption with Charcoal Sample of 40 Mesh Size

No.	Volume of Pb(NO ₃) ₂ (eluant) solution (ml)	Mass of adsorbent charcoal (40 mesh) (g)	Precipitant	Mass of PbI ₂ precipitate (g)
1.	50	10	excess KI	1.1000
2.	50	10	excess KI	1.1000
3.	50	10	excess KI	1.1000

This indicates that the mass of PbI₂ precipitate after adsorption with charcoal sample of 40 mesh size was 1.1000 g.

For 120 mesh charcoal sample, 1.0052 g of Pb(NO₃)₂ (or 0.6137 g Pb²⁺), 0.9298 g of Pb(NO₃)₂ (or 0.5650 g of Pb²⁺) was adsorbed and, thus, the percent adsorption could be observed 92.4990 %.

Initially, 10 g of $Pb(NO_3)_2$ was dissolved to obtain 500 ml of solution and, thus, 50 ml of $Pb(NO_3)_2$ solution was obtained from 1g of $Pb(NO_3)_2$. In experiment, the amount of $Pb(NO_3)_2$ recovered was 1.0052 g of $Pb(NO_3)_2$. Therefore, this method could assumed to be effective satisfactorily.

Similarly, for 80 mesh, 60 mesh, and 40 mesh charcoal samples, the mass of $Pb(NO_3)_2$ before adsorption, after adsorption, adsorbed by charcoal sample and percent adsorption can be tabulated in the Table (6).

Table (6) The Adsorption Data of Pb(NO₃)₂ or Pb²⁺ on Charcoal Sample of Different Sizes

No.	Size of charcoal sample	Mass of Pb(NO ₃) ₂ before adsorption (g)	Mass of Pb(NO ₃) ₂ after adsorption (g)	Absorbed mass of Pb(NO ₃) ₂ (g)	Percent adsorption (%)
1.	120 mesh	1.0052	0.0754	0.9298	92.4990
2.	80 mesh	1.0052	0.1436	0.8616	85.7143
3.	60 mesh	1.0052	0.2872	0.7180	71.4286
4.	40 mesh	1.0052	0.7898	0.2154	21.4286

According to these results, 120 mesh charcoal sample has highest percent adsorption and 40 mesh charcoal sample has lowest percent

adsorption. Since the adsorption is a surface phenomenon and if mass is the same, the smaller the particle size, the larger is the surface area and hence of greater adsorption. In addition, the contact also governs the amount of adsorption. Hence, the greater percent adsorption for samples 120 mesh, 80 mesh and 60 mesh may be due to their particle size and longer contact time.

3.3 Results of the Optimum Particle Size for the Adsorption

The results of percent adsorption, time taken for the adsorption, and size of sample charcoal powders were given in the Table (7).

Table (7) The Particle Size, Percent Adsorption, and Time Taken for the Adsorption of Various Size Charcoal Samples

No.	Size of charcoal sample	Percent adsorption (%)	Time taken for adsorption (hr)
1.	120 mesh	92.4990	102 hr
2.	80 mesh	85.7143	59.5 hr
3.	60 mesh	71.4286	32.5 hr
4.	40 mesh	21.4286	0.75 hr

For samples of 120 mesh, 80 mesh, and 60 mesh sized samples percent adsorption is satisfactorily good. However, the time duration for 50 ml solution to pass through the same length of column (26 cm) were about $4\frac{1}{4}$ days, $2\frac{1}{2}$ days, and $1\frac{1}{3}$ days respectively. The 40 mesh sized sample took only 45 minutes for 50 ml solution to pass through the same length of column.

For mixing the same mass of charcoal samples (of different sizes) with the same volume and concentration of $Pb(NO_3)_2$ solution and allowing the same time, the mass adsorbed by charcoal and percent adsorption for each size of sample were given in the Table (8).

Table (8) The Particle Size, Percent Adsorption at the Same Time Duration for Various Sized Charcoal Samples

No.	Size of charcoal sample	Mass of Pb(NO ₃) ₂ before adsorption (g)	Mass of Pb(NO ₃) ₂ after adsorption (g)	Adsorbed mass of Pb(NO ₃) ₂ (g)	Percent adsorption (%)
1.	120 mesh	1.0052	0.5394	0.4658	46.3390
2.	80 mesh	1.0052	0.5521	0.4531	45.0756
3.	60 mesh	1.0052	0.6250	0.3802	37.8233
4.	40 mesh	1.0052	0.8350	0.1702	16.9320

In this case, the percent adsorption of 120 mesh, 80 mesh, and 60 mesh sized charcoal samples are also found to be greater than that of 40 mesh size. But in fixed time, the difference was found to be not to great.

Therefore, for laboratory scale experiment, although 40 mesh size has least percent adsorption, it is assumed to be optimum particle size for adsorption.

3.4 Results for the Examination of Charcoals Before and After Adsorption

In order to confirm the adsorption of charcoal samples to Pb^{2+} ions, the comparison of FT-IR spectra of each charcoal sample before and after adsorption were given in Figure (1), (2), (3), (4) and (5) respectively.

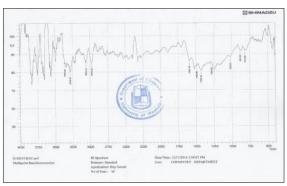


Figure (1) FT-IR Spectrum of Charcoal Sample

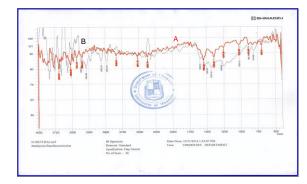


Figure (2) Comparison of FT-IR Spectra of 120Mesh Sized Charcoal Sample Before and After Adsorption of Pb²⁺ Ions

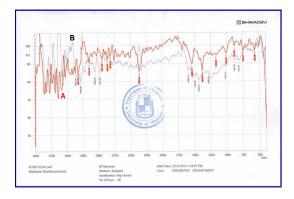


Figure (3) Comparison of FT-IR Spectra of 80 Mesh Sized Charcoal Sample Before and After Adsorption of Pb²⁺ Ions

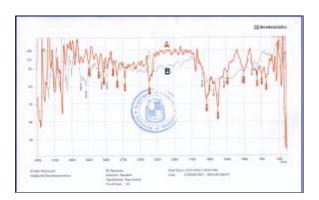
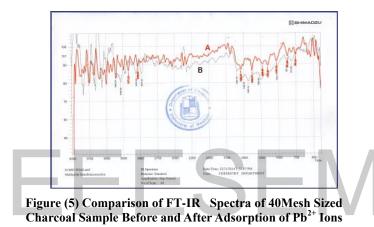


Figure (4) Comparison of FT-IR Spectra of 60 Mesh Sized Charcoal Sample Before and After Adsorption of Pb²⁺ Ions

2+



In these FT-IR spectra, for each charcoal sample, the changes in peaks were observed in before and after adsorption of Pb^{2+} ions and these changes were assumed to be caused due to the adsorption.

3.5 Results of the Adsorption Capacity of the Compromise Size (40 mesh) Plum Seed Coats Charcoal Sample

The mass of Pb^{2+} ion adsorbed by plum seed coats charcoal sample was determined in every adsorption experiment. The results were tabulated in Table (9).

Table (9) Adsorption	Capacity of the Co	mpromise Size (40 M	Mesh) Plum Seed Coats	Charcoal for $Pb(NO_3)_2$ or Pb^2	lons

No.	Size of charcoal sample	Mass of Pb(NO ₃) ₂ before adsorption (g)	Mass of Pb(NO ₃) ₂ after adsorption (g)	Adsorbed mass of Pb(NO ₃) ₂ (g)	Percent adsorption (%)
1.	40 mesh	1.0052	0.7898	0.2154	21.4286
2.	40 mesh	1.0052	0.8125	0.1927	19.1703
3.	40 mesh	1.0052	0.8512	0.1540	15.3203
4.	40 mesh	1.0052	0.8965	0.1087	10.8138
5.	40 mesh	1.0052	0.9473	0.0579	5.7600
6.	40 mesh	1.0052	0.9542	0.0510	5.0736

When percent adsorption was plotted against the number of adsorption repeated by the same adsorbent plum seed coats charcoal 10 g, a graph was obtained. This indicates that the adsorption capacity of plum seed coats charcoal tends to decrease with respect to the number of adsorption for Pb^{2+} .

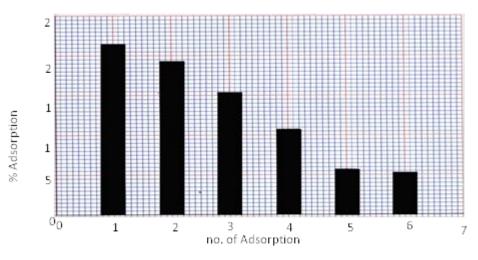


Figure (6) Percent Adsorption Versus Number of Repeatation

In accordance with this result, it can be noted that number of the effective repeatation can be done up to sixth time. Because adsorption efficiency of plum seed coats charcoal tended to decrease is starting from the sixth adsorption experiment.

3.6 Results for Verification of Validity of Freundlich Isotherm

The experimental data and calculated values were given in the Table (10).

No.	Conc. of Pb^{2+} (c) (mg ml ⁻¹)	log c	Adsorbed mass of Pb ²⁺ (y) (mg/g)	log y
1.	22.5000	1.3522	13.5000	1.1303
2.	11.2500	1.0513	9.0000	0.9542
3.	5.6250	0.7501	6.0268	0.7801
4.	2.8130	0.4492	3.6161	0.5582
5.	1.4063	0.1481	2.3438	0.3699

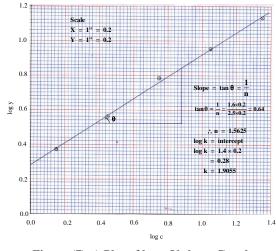


Figure (7) A Plot of log y Vs log c Graph

Thus a plot of log y versus log c should give a straight line with the slope of $\frac{1}{2}$ and intercepting at Y axis by log k.

3.7 Results of the Adsorption Capacity of Reactivated Charcoal Sample

The comparison of 40 mesh size plum seed coats charcoal sample before and after adsorption by reactivated with distilled water and boiled distilled water were given in the Table (11).

No.	Size of charcoal sample	Mass of Pb(NO ₃) ₂ before adsorption (g)	Mass of Pb(NO ₃) ₂ after adsorption (g)	Adsorbed mass of Pb(NO ₃) ₂ (g)	Percent adsorption (%)
1.	40 mesh (with distilled water)	1.0052	0.8152	0.1900	18.9017
2.	40 mesh (with boiled distilled water)	1.0052	0.8019	0.2033	20.2248

According to these results, percent adsorption of reactivated 40 mesh size of plum seed coats charcoal sample with distilled water was found to be 18.9017 % and then percent adsorption of reactivated 40 mesh size of plum seed coats charcoal sample with boiled distilled water was found to be 20.2248 %. So, it can be reused for adsorption process.

CONCLUSION

In this work, the activated charcoal sample was prepared from the seed coats of plum seed. The four different sized charcoal samples were made by powdering, sieving with specific sized sieves and activating. For each of these, 120 (+120) mesh, 80 (80-120) mesh, 60(60-80) mesh, and 40(40-60) mesh sized charcoal, percent adsorption was determined by packing sample in columns of same size and adsorption of it to Pb²⁺ ions by passing the Pb(NO₃)₂ solution into the column. Although the 120 mesh, 80 mesh and 60 mesh charcoal samples show satisfactorily good percent adsorption, their time duration to achieve the completeness of adsorption were found to be rather long. Hence, 40 mesh charcoal samples were sinked in equal amount of Pb(NO₃)₂ solution for equal time duration, the difference in percent adsorption was found to be not to great and filtering (passing through the column and adsorption) is more affected them the soaking (immersing and adsorption). In addition, the adsorptions of Pb²⁺ by charcoal powder samples were also confirmed by FT-IR spectra of before and after adsorption. When a fixed mass of charcoal sample was used to adsorb repeatedly, the percent adsorption firstly dropped decreases gradually. These show that the charcoal sample has great adsorptive power.

Furthermore, from the determination of weight of Pb^{2+} as $Pb(NO_3)_2$ adsorbed by unit mass of charcoal by using various concentration of $Pb(NO_3)_2$ solution, it can be realized that this adsorption follows the adsorption isotherm with the values of constants n and k are 1.5625 and 1.9055 respectively.

Moreover, the used charcoal sample could be reactivated by two different ways, its percent adsorption regained as closely as original value (20.2248 % and 18.9017 % compare with 21.4286 %). This is the evidence for the suitability of recycling this adsorbent.

The use of charcoal prepared from the plum seed coats is very efficient due to its low cost, great adsorptive capacity, reactivated property and ease to be prepared. In addition, it will help to reduce the environmental pollution.

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