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Study on Utilization of Acid Treated Coconut Shell Carbon on Removal of Lead from Industrial Effluent

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Abstract

Lead is one of the major pollutants in wastewater. It is used as a major raw material in battery manufacturing and industrial effluent from this industry can contain high concentration of lead. In this research, removal of lead from this industrial effluent has been investigated by using coconut shell sulphonated and nitrated carbon from carbonized coconut shell powder. Carbonization temperatures were 300ºC, 400ºC, 500ºC, 600ºC, 700ºC respectively at resident time of 10 minutes. Determination of yield %, the most suitable temperature, as well as ash content, moisture content, volatile matter content, fixed carbon content and iodine adsorption activity of carbonized carbon were studied. The most suitable carbonization temperature was found to be 500ºC. Carbonized powdered carbon (500ºC) was activated with various concentrations of sulphuric acid and nitric acid for 24 hour. Effect of various parameters such as acid concentration (1N, 1.25N, 1.5N, 1.75N, 2.0N) for activation on adsorption efficiency of sulphonated and nitrated carbon was characterized by iodine adsorption activity and methylene blue decolorizing power. It was found that carbon activated by 1.5N sulphuric acid and nitric acid had the most favorable for adsorption. Effect of pH of battery industrial effluent and amount of sulphonated and nitrated carbon on removal of lead from battery industrial effluent were determined. Amount of lead in industrial effluent before and after treatment were measured by Atomic Absorption Spectrophotometer (AAS). It was found that the maximum removing efficiency of lead by 200 mg of sulphonated carbon was 96.3% and that of nitrated carbon was 97% for industrial waste water at pH 6. The result indicated that acid treated carbonized coconut shell powder could be used as high capacity adsorbent for the removal of lead from industrial effluent.

Keywords: Carbonized Carbon, Fixed Carbon Content, Sulphonated Ccarbon, Nitrated Carbon

Introduction

Coconut trees grow in rainforests and other tropical of climates. The green empty coconuts find their way in the cities refuse. The shells are not much use other than for use as fuel. On the other hand, Lead is one of the major pollutants in wastewater. It is used as a major raw material in battery manufacturing and industrial effluents from this industry can contain high concentration of lead about 0.5-25 mg/L. Lead II ions which was very toxic even in low quantities to the receiving environment.

All lead compounds are considered as a cumulative poisons. Acute lead poisoning can effect nervous system and gastrointestinal track. Lead removals are attempted using adsorption

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techniques. Activated carbon is well known that can be prepared from a variety of carbonaceous precursors such as wood, lignite and nut shells. Coconut shell was selected to make activated carbon because it has the same chemical composition of hard wood. It has high fixed carbon content and low ash content. It is cheap and readily available in high quality.

Objectives

(i) to evaluate the most suitable sulphuric acid and nitric acid concentration for activation

(ii) to study the characteristics of sulphonated carbon and nitrated carbon

(iii) to study various operating parameters such as pH of industrial effluent, dosage of sulphonated carbon and nitrated carbon and removing efficiency of lead from battery industrial effluent.

Materials and Methods

Raw Materials

In this Research, Waste coconut shells were purchased from the Hlaing Thar Yar Township, Yangon Region. Battery industrial effluent were collected from Shwe-Pyi-Tha township.

Methods

Carbonization of Coconut Shell

Waste coconut shells were sun-dried for a week. All of the outer fibers were removed and inside of the shells were cleaned and then dried in an oven at 105°C for 2 hr. Then the dried coconut shells were crushed, ground and sieved by 35 mesh (500 µm) screen.

100 g of ground coconut shells were placed in a preweighed crucible and carbonized in a muffle furnace at various temperatures (300°C, 400°C, 500°C, 600°C, 700°C) for 10 minutes respectively. Carbonized carbon was ground into powder and sieved by 230 mesh (63 μm) screen. Then, the carbonized powdered carbon was stored in an airtight screw capped bottle.

Determination of Yield Percent of Carbonized Carbon from Waste Coconut Shells

100 g of ground coconut shells were placed in a preweighed crucible and carbonized in muffle furnace at 300˚C for 10 minutes. The crucible was allowed to cool and the weight of the carbonized coconut shell was determined. The experiment was repeated for three times and the average percent was determined. The same experiments at 400˚C, 500˚C, 600˚C and 700˚C were made. The yields of carbonaceous matters at various temperatures were determined as follows. These results are recorded in Table (1) and Figure (1).

$$\%\ \text{Yield of carbonized carbon} = \frac{\text{Weight of carbon residues}}{\text{100 g of ground coconut shells}} \times 100$$

Determination of Iodine Adsorption Value of Carbonized Carbon and Sulphonated Carbon and Nitrated Carbon

0.2 g of the carbonized carbon (300˚C) was placed in 250 ml dried conical flask. 40 ml of 0.1 N iodine solution was then added. The flask was shaken properly for 4 minutes and then filtered. The filtrate was titrated against 0.05 N standard sodium thiosulphate solution ($Na_2S_2O_3$) using starch indicator. The end point was indicated by change in color of solution from blue to colorless. A blank test was carried out without sample under the same condition. From difference, the concentration of iodine adsorbed by carbon at room temperature was calculated as amount of iodine adsorbed in milligrams. Each experiment was made three times. The same experiment for various carbonization temperatures 400˚C, 500˚C, 600˚C and 700˚C were made respectively. These results are shown in Table (2) and Figure (2).

The same procedure was employed for the determination of the iodine adsorption activity for sulphonated carbon and nitrated carbon at various concentrations (1.0 N, 1.25 N, 1.5 N, 1.75 N and 2.0 N). The iodine adsorption activity was calculated as follows. Their respective results are shown in Tables (4), (5) and Figure (3).

$$\text{Iodine Adsorption Value (mg/g)} = \frac{(\text{ml of } 0.05\text{N } Na_2S_2O_3 \text{ Blank} - \text{ml of } 0.05\text{N } Na_2S_2O_3 \text{ Test})\,(12.7)\,(0.05)}{\text{Weight of sample.}}$$

Determination of Moisture Content

1.0 g of dried carbonized carbon at 500˚C was weighed and placed in a cleaned, dried and previously weighed watch glass. The sample was placed in a hot air oven and dried at 105˚C to constant weight for one and half hour. After drying was completed, sample was removed from an oven and placed in a desiccator for about 30 minutes and weighed accurately. The same experiment was conducted for sulphonated and nitrated carbon and their moisture contents were calculated as follows. These results are shown in Tables (3) and (5).

$$\text{Moisture Content (\%)} = \frac{\text{Loss in weight (g)}}{\text{Weight of sample (g)}} \times 100$$

Determination of Volatile Matter Content

1.0 g of powdered carbonized carbon at 500˚C was taken in a cleaned, dried and previously weighed porcelain crucible. The crucible was placed in a muffle furnace maintained at 925˚C. It was taken out after exactly
7 minutes. After first cooling in air, the crucible was cooled in a desiccator and weighed again. The loss in weight of the powder reported on percentage basis given volatile matter content in the sample. The same experiment was conducted for sulphonated carbon (1.5 N, sulphuric acid) and their volatile matter contents were calculated as follows. These data are tabulated in Tables (3) and (5).

$$\text{Volatile Matter (\%)} = \frac{\text{Loss in weight due to removal of volatile matter}}{\text{Weight of sample}} \times 100$$

Determination of Ash Content

The residual carbonized carbon powder from the above step was then heated in a muffle furnace at 750˚C for half an hour. After first cooling in air, the crucible was cooled in a desiccator and weighed again. The heating, cooling and weighing cycle was repeated till a constant weight was obtained. Ash was almost white in color. The same experiment was conducted for sulphonated and nitrated carbon (1.5 N, sulphuric acid and nitric acid) and their ash contents were calculated as follows. These data are tabulated in Tables (3) and (5).

$$\text{Ash (\%)} = \frac{\text{Weight of ash left}}{\text{Weight of sample}} \times 100$$

Determination of Fixed Carbon Content

From the data of moisture contents, volatile matter contents and ash contents, fixed carbon contents of the carbonized carbon at 500ºC and sulphonated and nitrated carbon (1.5 N, sulphuric acid and nitric acid) can be calculated as follows. These data are shown in Tables (3) and (5).

Percent of Fixed Carbon = 100 - % of (Moisture +Volatile matter +Ash)

Preparation of Sulphonated Carbon and Nitrated Carbon

Sulphonated carbon was prepared by treating the carbonized powdered carbon with various concentrations of sulphuric acid (1.0 N, 1.25 N, 1.5 N, 1.75 N and 2.0 N) in 100 ml beaker for 24 hr. After 24 hr, each of the flask contents was filtered through Whatmann filter paper No.41 and each treated carbon residue was washed repeatedly till no more acid was left and air dried. The carbon was termed as sulphonated carbon. (SC) The same experiment was conducted for nitrated carbon by treating the carbonized powdered carbon with various concentrations of nitric acid. The carbon was termed as nitrated carbon.(NC)

Determination of Methylene Blue Adsorption by Sulphonated Carbon and Nitrated Carbon

0.1 g of sulphonated carbon or nitrated carbon sample was weighed and transferred to 125 ml glass stoppered flask. 10 ml of methylene blue solution (2 mg/l) was added to the glass stoppered and shaken for 5 minutes. After the first 10 ml was decolorized, methylene blue solution (1 ml at a time) was continued to add till the blue color had disappeared for 5 minutes. The removal treatment was performed at ambient temperature. The number of experiments for removal of methylene blue was then conducted thrice. The absorbance after methylene blue adsorption was measured by using UV-Visible spectrophotometer (UV-721G) at a wavelength of 665 nm and then concentration was obtained from calibration curve (absorbance Vs. concentration). The removing efficiency was calculated as follows. These results are shown in Table (5).

$$\text{Removing Efficiency (\%)} = \frac{[\,(C_o - C_e)\,]}{C_o} \times 100$$

where; C_o = Concentration of methylene blue in the sample solution before treatment

C_e = Concentration of methylene blue in the sample solution after treatment

Effect of pH on Removal of Lead from Industrial Effluents by Treatment with Sulphonated Carbon and Nitrated Carbon

100 ml of lead containing industrial effluents (pH 1.5) was taken in five 500 ml Erlenmeyer flasks and the pH in various flasks were adjusted from pH 4 to pH 8 using sodium hydroxide (conc.) solution. 100 mg of sulphonated carbon was added to each flask and stirred by magnetic stirrer slowly and allowed to interact for 60 minutes with occasional stirring shown in Figure (3.5). After an hour, each of the flask contents was filtered through Whatmann filter paper No.41 and filtrates were analyzed for residual concentrations of lead. The lead concentrations before and after treatment were detected by using Atomic Absorption Spectrophotometer (AAS). The results are shown in Table (6) and Figure (4). The same experiment was conducted for nitrated carbon The lead concentrations before and after treatment were detected by using Atomic Absorption Spectrophotometer (AAS). These results are shown in Tables (6) and Figure (4).

Effect of Dosage of Sulphonated Carbon on Removal of Lead from Industrial Effluents

100 ml of lead containing industrial effluents (pH 6) was taken in five 500 ml Erlenmeyer flasks. Various amount of sulphonated carbon (100 mg, 150 mg, 200 mg, 250 mg, and 300 mg) were added to each flask and stirred by magnetic stirrer slowly and allowed to interact for 60 mins with occasional stirring. After an hour, each of the flask contents was filtered through Whatmann filter paper No.41 and filtrates were analyzed

for residual concentrations of lead. The lead concentrations before and after treatment were detected by using Atomic Absorption Spectrophotometer (AAS). The results are presented in Table (7) and Figure (5). The same experiment was conducted for nitrated carbon The lead concentrations before and after treatment were detected by using Atomic Absorption Spectrophotometer (AAS). These results are shown in Tables (7) and Figure (5).

Determination of Total Solid (TS)

100 ml of well mixed industrial effluents sample (pH 1.5) was placed in a preweighed, cleaned and dried watch glass and then evaporated by using steam bath at 103-105˚C. The residue was cooled to room temperature in a desiccator and thereafter dried to constant weight in an oven at 103-105˚C. The weight of residue obtained was termed as total solid of unadsorbed effluents. The same experiment was conducted for treated industrial effluent. These results are shown in Tables (8).

$$\text{Total Solid (mg dm}^{-3}) = \frac{(B - A) \times 1000}{\text{Sample volume in ml (100)}}$$

A = Weight of dried watch glass

B = Weight of dried watch glass + Total Solid

3.5.2 Determination of Dissolved Solid (DS)

100 ml of well mixed industrial effluents sample (pH 1.5) was filtered and placed in a preweighed, cleaned and dried watch glass and then evaporated by using steam bath at

103-105˚C. The residue was cooled to room temperature in a desiccator and thereafter dried to constant weight in an oven at 103-105˚C. The weight of residue obtained was termed as dissolved solid of unadsorbed effluents. The same experiment was conducted for treated industrial effluent. These results are shown in Tables (8).

$$\text{Dissolved Solid (mg dm}^{-3}\text{)} = \frac{(C - A) \times 1000}{\text{Sample volume in ml (100)}}$$

A = Weight of dried watch glass

C = Weight of dried watch glass + Dissolved Solid

Determination of Suspended Solid (SS)

Using the data of total solid and dissolved solid, suspended solid can be calculated as follows. The result is shown in Table (8).

Suspended Solid = Total Solid – Dissolved Solid

Results

Table (1) Effect of Carbonization Temperature on Yield Percent of Carbonized Carbon

Temperature (ºC)	Amount of Sample (g)	Resident Time (min)	Yield (% w/w)	Literature Value* (% w/w)
300	100	10	33.0	47.2
400	100	10	29.5	30.6
500	100	10	27.9	24.3
600	100	10	23.8	22.2
700	100	10	22.8	16.7

Experiments were conducted at the Laboratory of Industrial Chemistry Department, West Yangon University.

*http://www.scienta_africana uniportjournal_info

Table (2) Effect of Carbonization Temperature on the Iodine Adsorption Activity of Carbonized Carbon

Carbonization Temperature (˚C)	Amount of Carbonized Carbon (mg)	Iodine Adsorption Value (mg/g)	Literature Value (mg/g)**
300	200	659	270
400	200	723	330
500*	**200**	**760**	**520**
600	200	710	480
700	200	685	350

Experiments were conducted at the Laboratory of Industrial Chemistry Department, West Yangon University .

* Most Suitable Condition

**http://www.scienta_africana uniportnournal_info

Table (3) Characteristics of Prepared Carbonized Carbon

Carbonization Temperature - 500˚C
Carbonization Time - 10 minutes

Parameters	Prepared Carbonized Carbon	Literature Value*
Moisture (% w/w)	8	9.7804
Volatile Matter (% w/w)	50	63.4799
Ash (% w/w)	7.8	15.5520
Fixed Carbon (% w/w)	34.2	11.1877

Experiments were conducted at the Laboratory of Industrial Chemistry Department, West Yangon University.

* http://www.ethesisi.nitrkl.ac.in/2401/1/The-Final-Thesis.pdf

Table (4) Effect of Concentrations of Sulphuric Acid and Nitric Acid on Iodine Adsorption Activity of Sulphonated Carbon (SC) and Nitrated Carbon (NC)

Amount of Carbonized Carbon (mg)	Concentration of Sulphuric Acid or Nitric Acid (N)	Iodine Adsorption Value (mg/g)		Yield of Sulphonated Carbon (SC) (% w/w)	Yield of Nitrated Carbon (NC) (% w/w)
		Sulphonated Carbon (SC)	Nitrated Carbon (NC)		
100	1.00	835	858	79.0	78.0
100	1.25	985	990	78.5	76.5
100	1.50*	1040	1091	77.5	75.0
100	1.75	925	975	76.4	72.0
100	2.00	875	895	74.0	70.0

Experiments were conducted at the Laboratory of Industrial Chemistry Department, West Yangon University.

* Most Suitable Condition

Table (5) Characteristics of Prepared Sulphonated Carbon and Nitrated Carbon

Carbonization Temperature - 500˚C
Carbonization Time - 10 mins
Sulphuric Acid Concentration - 1.5 N
Nitritic Acid Concentration - 1.5 N

Parameters	Prepared Sulphonated Carbon (SC)	Prepared Nitrated Carbon * (NC)	Literature Value**
Moisture (% w/w)	5.3	5.0	3-10 max**
Volatile Matter (% w/w)	28.0	25.0	-
Ash (% w/w)	2.6	2.0	≤8max**
Fixed Carbon (% w/w)	64.1	68.0	-
Iodine Adsorption Value (mg/g)	1040.0	1091.0	900-1100***
Removal Efficiency of Methylene Blue (% w/w)	88.2	89.3	87.5****

Experiments were conducted at the Laboratory of Industrial Chemistry Department, West Yangon University.
*MostSuitable Condition
** http://www.ijrce.org/download-pdf-file=32-35.pdf
*** http://www.err.wikipedia.org/wiki/Activated Carbon
****http://www.banglajol.info/index.php/DUJS/article/download/11491/8417

Table (6) Effect of pH on Removal of Lead from Battery Industrial Effluent by Treatment with Sulphonated Carbon and Nitrated Carbon

Amount of Sulphonated Carbon – 100 mg
Volume of Industrial Effluents – 100 ml

pH of IndustrialE ffluent	Amount of Lead in Industrial Effluent (ppm)				Lead Removal (%)		Acceptable Range of Lead in Industrial Effluent** (ppm)
	Before Treatment with SC	After Treatment with SC	Before Treatment with NC	After Treatment with NC	SC	NC	
4	6.5	3.17	6.5	2.77	51	57	<0.3
5	6.5	2.82	6.5	2.72	56	58	
6*	**6.5**	**2.25**	**6.5**	**1.96**	**65**	**70**	
7	6.5	2.44	6.5	2.10	62	67	
8	6.5	2.66	6.5	2.32	59.	64	

Experiments were conducted at the Laboratory of Universities' Research Center, University of Yangon.

* Most Suitable Condition
** Notification the Ministry of Science, Technology and Environment No.3, B.E 2539 (1996), published in the Royal Government Gazette, Vol.113 Part13D

- Activated carbon with (1.5 N) sulphuric acid for activation time (24 hr).

Table (7) Effect of Amount of Sulphonated Carbon and Nitrated Carbon on Removal of Lead from Industrial Effluent

Volume of Battery Industrial Effluents – 100 ml
pH of Industrial Effluents – 6

Amount of (SC)• or (NC)• (mg)	Amount of Lead in Industrial Effluents (ppm)				Lead Removal (%)		Acceptable Range of Lead in Industrial Effluents** (ppm)
	Before Treatment with SC	After Treatment with SC	Before Treatment with NC	After Treatment with NC	SC	NC	
100	6.5	2.25	6.5	1.96	65	70	<0.3
150	6.5	1.74	6.5	1.04	73	83	
200*	**6.5**	**0.24**	**6.5**	**0.2**	**96**	**97**	
250	6.5	1.81	6.5	1.53	72	76	
300	6.5	1.96	6.5	1.96	69	69	

Experiments were conducted at the Laboratory of Universities' Research Center, University of Yangon.

* Most Suitable Condition

** Notification the Ministry of Science, Technology and Environment No.3, B.E 2539 (1996), published in the Royal Government Gazette, Vol.113 Part13D.

• Activated carbon with (1.5 N) sulphuric acid or nitrated acid for activation time (24 hr).

Table (8) Charactristics of Industrial Effluent Before and After Adsorption with Sulphonated Carbon and Nitrated Carbon

Sulphuric Acid Concentration – 1.5 N
Nitric Acid Concentration _ 1.5 N
Volume of Battery Industrial Effluents – 100 ml

Parameters	Before Adsorption With (SC)	After Adsorption with (SC)	Before Adsorption with (NC)	After Adsorption with (NC)*	Literature Value**
pH	1.5	7.0	1.5	7.0	5.5-9.0
Total Dissolved Solid (mg/L)	2000.0	1600.0	2000.0	1224.0	<5000
Total Suspended Solid (mg/L)	150.0	72.0	150.0	60.0	<150
Lead (mg/L)	6.5	0.24	6.5	0.20	<0.3

Experiments were conducted at the Laboratory of Industrial Chemistry Department, West Yangon University.

- Most Suitable Condition

** Notification the Ministry of Science, Technology and Environment No.3, B.E 2539 (1996), published in the Royal Government Gazette, Vol.113 Part13D.

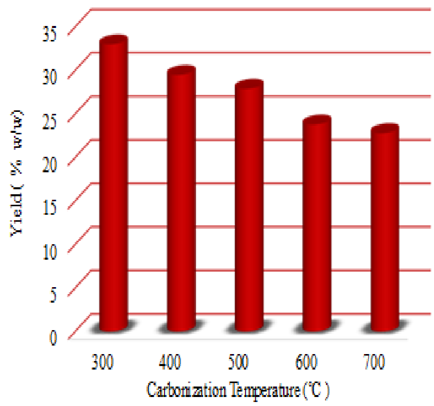

Figure (1) **Effect of Carbonization Temperature on Yield Percent of Carbonized Carbon**

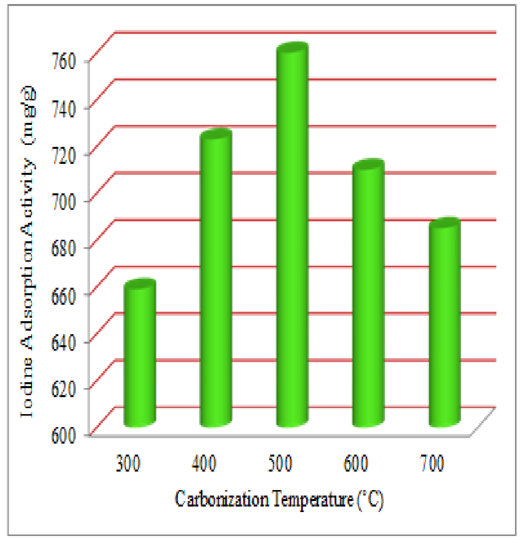

Figure (2) **Effect of Carbonization Temperature on the Iodine Adsorption Activity of Carbonized Carbon**

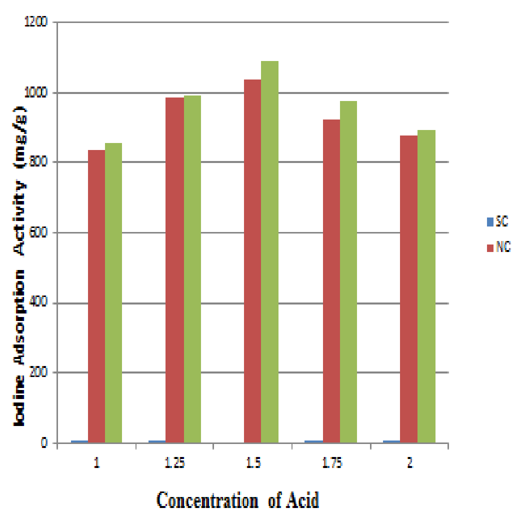

Figure (3) Effect of Concentrations of Sulphuric Acid and nitric acid on Iodine Adsorption Activity of Sulphonated Carbon (SC) and Nitrated Carbon (NC)

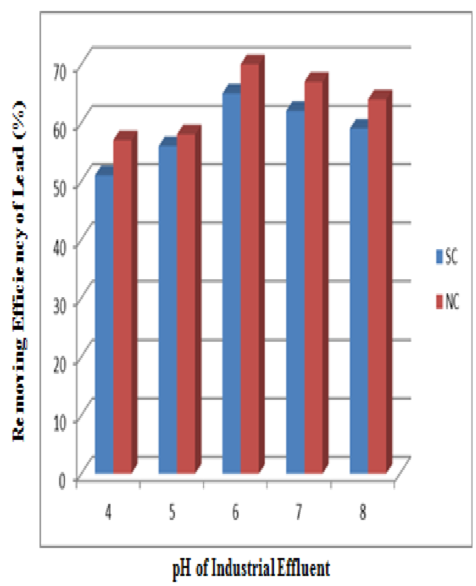

Figure (4) **Effect of pH on Removal of Lead from Industrial Effluents by Treatment with Sulphonated Carbon**

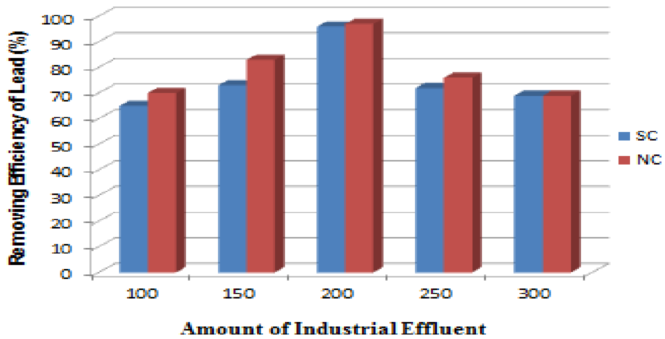

Figure (5) Effect of Amount of Sulphonated Carbon and Nitrated Carbon on Removal of Lead from Industrial Effluents

Discussion

Table (1) and Figure (1) show that effect of carbonization temperature on yield percent of carbonized carbon. It was found that reduction in yield percent of the carbonized product occurred with increasing temperature. The effect of carbonization temperature on the iodine adsorption activity of carbonized carbon prepared by various temperatures (300ºC, 400ºC, 500ºC, 600ºC and 700ºC) were studied. The results from Table (2) and Figure (2) show that carbonization of coconut shell at 500˚C gave the highest adsorption activity of the test adsorbate iodine (760 mg/g).

The effect of carbonization condition on the percentages of moisture content, ash, volatile matter and fixed carbon contents are shown in Table (3) and the Results were compared with the literature value. It was found that fix carbon content of prepared carbonized carbon was higher than literature value. Sulphonated carbon and Nitrated carbon from prepared carbonized carbon powder could be produced by chemical activation with various concentrations of sulphuric acid and nitric acid. Table (4) and Figure (3) show that the adsorption activity of iodine by prepared sulphonated carbon and nitrated carbon activated with various concentrations (1 N, 1.25 N, 1.5 N, 1.75 N, 2 N) of sulphuric acid and nitric acid. It was observed that carbon activated by 1.5 N sulphuric acid and 1.5 N nitric acid had the most favorable adsorption activity due to highest iodine adsorption value (1040 mg/g) and (1091 mg/g) indicating that the formation of more micropore in the product. The characteristics of prepared sulphonated and nitrated carbon are shown in Table (5). From the study of results 1.5 N acid treated carbon could be used in treatment of battery industrial effluent. Removal of lead from industrial effluents by sulphonated carbon and nitrated carbon depends on pH of the industrial effluents and amount of adsorbent. Results in Table (6) and Figure (4) indicate that pH 6 of battery industrial effluent was the most favorable condition for lead adsorption. Table (7) and Figure (5) show that the effect of various amount of sulphonated carbon and nitrated carbon on removal of lead from industrial effluents. It was found that the removing efficiency of lead by 200 mg of sulphonated carbon and nitrated carbon was 96.3 % and 97 %. It indicated that prepared sulphonated carbon and nitrated carbon could be effectively used in industrial effluents treatment. Table (8) indicates that the characteristics of industrial effluent before and after adsorption with sulphonated carbon and nitrated carbon. When the results in Table (8) were compared with standard values, it was found that the results fall within the allowable range of industrial effluent.

Conclusion

In this research, sulphonated carbon and nitrated carbon were prepared from the waste coconut shell by carbonization and activation with sulphuric acid and nitric acid. It was used as adsorbent for removal of the heavy metal, lead from industrial effluent. Prepared sulphonated carbon and nitrated carbon under most suitable condition was characterized by moisture, volatile matter, ash and fixed carbon contents, iodine adsorption activity and removal efficiency of methylene blue. As the results were compared with literature value, it was found that moisture content, ash content, iodine adsorption activity and removal efficiency of methylene blue were within the acceptable range. Using this sulphonated carbon and nitrated carbon, adsorption of lead from battery industry effluent was performed at different pH of industrial effluent and adsorbent dosages. Prepared sulphonated carbon and nitrated carbon showed good adsorptive properties at pH 6 of industrial effluents and 200 mg of absorbent. Effectiveness of sulphonated and nitrated carbon treatment operation on industrial effluent was investigated before and after adsorption. It was found that the amount of lead, suspended solid and dissolved solid of treated industrial effluents fall within the allowable range. Thus, the sulphonated carbon and nitrated carbon produced from waste coconut shell carbon powder could be used as adsorbent for number of polluted problems, such as, treatment of drinking water, removing color from industrial effluents and removal of heavy metal.

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