STUDYING THE POSSIBILITY OF REMOVING SOME CHEMICAL CONTAMINANTS FROM SOLUTIONS USING SELECTED AGRICULTURAL RESIDUES

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ABSTRACT : This paper explores the feasibility of using the seeds of the *Leucaena leucocephala* in the preparation of activated carbon in view of the advantages that the plant can provide in terms of abundance, low cost and product quality. Activated carbon was prepared using *Leucaena* seeds and activated by H_3PO_4 chemical activation process using H_3PO_4: char ratios (w/w) of (1:1), (1:2) and (1:3) at (400°C). Surface properties were determined using FT-IR and FE-SEM techniques. Cadmium and PNP, PPD dyes were chosen as the prominent chemical pollutants to study the ability of the prepared carbon to adsorb them. The adsorption process was controlled and the factors affecting it were studied, which are: Mass of the adsorbent, Temperature, pH, Contact time, Initial concentration of the adsorbed material. It was found that the adsorption capacity increases with decreasing temperature for cadmium and dyes. The optimum PH value was between (pH=6, pH=5) for the mentioned adsorbents, Recorded (180 hours) as the best contact time. Positive value of ΔH (J\mole) indicate that the reaction is exothermic, negative value of ΔS (J\mole.K) indicate that the reaction is spontaneous and negative value of ΔG (J\mole) indicate that the reaction occurs spontaneously.

Key words : Activated carbon, Leucaena, adsorption, cadmium, dyes.

INTRODUCTION

Pollution is a global challenge facing scientists today. its impact on economic and social growth as well as potential health impacts on thousands, perhaps millions, of these city dwellers. It is defined as the change that occurs to the basic elements of the environment that the environment cannot treat it or neutralize in a timely manner (Azizullah *et al*, 2011). It is also defined as adding an element that does not exist to the group of elements of the ecosystem called pollutants, or increasing or decreasing one of the elements of this system in a negative way on its work. Water pollution is one of the most prominent concerns that have drawn attention due to its increasing seriousness (Kelly *et al*, 2007). Water pollution is an expression of the presence of an intruder in the water mass that disturbs the natural balance such as the presence of chemical, physical, organic or radioactive pollutants (Bhatnagar and Kumari, 2013). Chemical pollution is one of the most serious forms of water pollution.

These pollutants can be classified into inorganic pollutants such as heavy metals, and organic pollutants such as organic dyes. Cadmium is one of the trace elements in nature, its concentration increases only in the case of human activity, although the use of cadmium in general is decreasing due to its toxicity, but its environmental presence as a pollutant is still large, due to its industrial uses (Scheringer, 2009). The International Agency for Research on Cancer (IARC) classifies cadmium as a group 1 human carcinogen, exposure to which is associated with lung cancer. The consumption and release of cadmium compounds into the environment does not show any effective way to recycle it (Munisamy *et al*, 2013). The process of removing cadmium can include natural and chemical methods. Natural processes refer to the use of some plants such as Morinca, aloe vera, peanuts and others. The seeds of these plants can absorb and neutralize the positive charges in the solution,
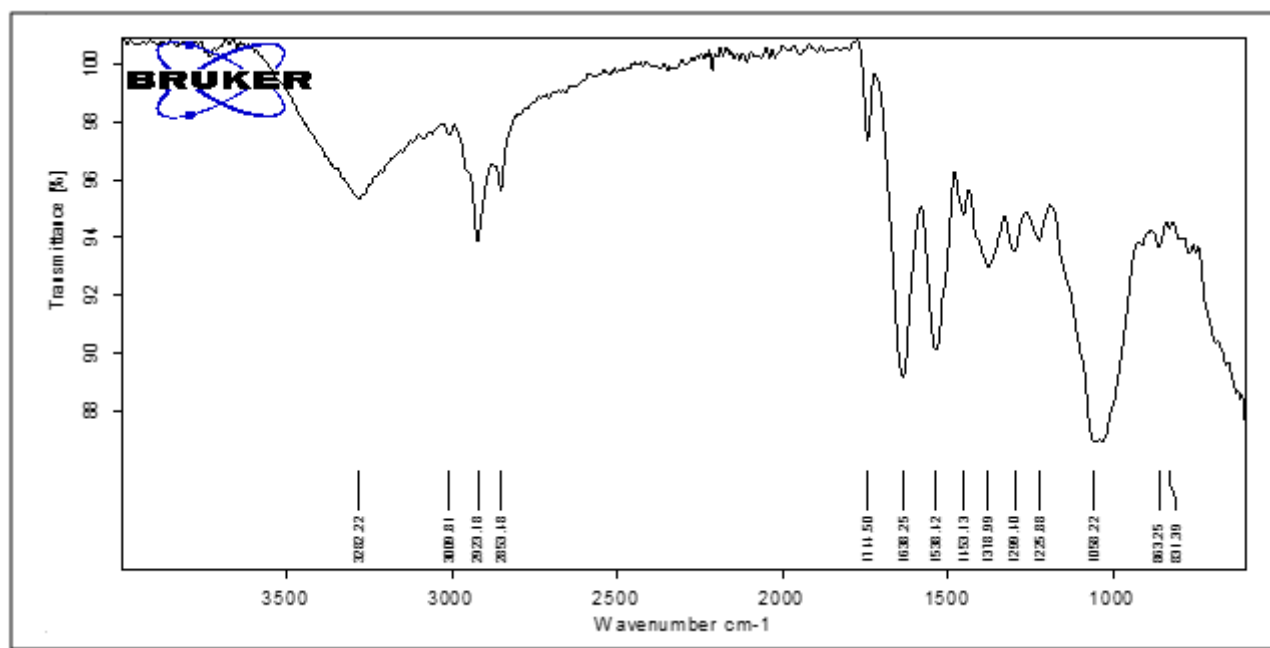
Fig. 1 : FT-IR Spectrum of pre-Treatment Leucaena Seed Powder (LOC).

which leads to the absorption of negative charges and mineral impurities in the waste water. Dyes are also considered one of the most dangerous water pollutants. Para-nitrophenol (pNPh), Para phenylene di amine (PPD) have varying toxicity effects (Muralikrishna and Manickam, 2017). Adsorption can be considered as one of the most important methods used in the treatment of water pollution. Recently, it has become popular to use activated carbon as one of the most widely used adsorbents in water purification for its unique properties such as large surface area, porous structure, and special surface effectiveness.

It is also a very inert material, thermally stable, but it is not without some disadvantages, including the high cost of some of its materials sometimes (Babel and Kurniawan, 2003). Using biomass in the preparation of activated carbon such as Leucaena can reduce production cost and overcome some of the other difficulties associated with the activated carbon technology. The Leucaena tree is wild and can be grown without the need for specific techniques (Babu Rajendran *et al*, 2015).

Aims of the study

In view of the lack of available and published information on activated carbon prepared from Leucaena; This research attempts to explore new possibilities of this raw material in the purification of polluted water.

MATERIALS AND METHODS

Preparing the raw material

One kilogram of Leucaena seeds were collected and washed with distilled water several times to get rid of impurities and suspended dust. It was left to dry at room temperature, then it was ground into a fine powder using an electric mill with a diameter of (15 cm) and the resulting powder was sieved using a sieve with a hole size of 30-200 μm, and then the fine powder was dried in a drying oven at a temperature of (70°C).

Preparation of activated carbon

25 g of the prepared powder was placed in three (500 ml) glass beakers, to which phosphoric acid was added in a ratio of (1:1), (1:2), (1:3) and they were mixed well using a glass stirrer until it was completely dissolved. The mixture was homogenized and then dried in a drying oven at (110°C) for 48 hours. Then it was placed in a ceramic crucible to be calcined in a firing furnace at (400°C) for three hours. Then the three samples were washed with distilled water until the PH of the washing water became 6 to 7 to remove the effect of activation on the surface of the activated carbon. Then it was ground to obtain a fine powder. The three models of activated carbon were named CHP1, CHP2 and CHP3, respectively.

Preparation of standard solution

The standard solutions of the samples were para-phenylenediamine diamine (PPDA), para-nitrophenol (PNP), cadmium ion (Cd^{+2}) and methylene blue dye (MB) with a concentration of (1000 mg/l) by dissolving (0.1g) of each of (PPDA), (PNP) and (MB) in (100 ml) of distilled water. Dissolving (0.16 g) of cadmium nitrate in (100 ml) of distilled water. Then (0.1N) of iodine solution was prepared by mixing (20 g) of KI dissolved in (40 ml)

of distilled water with (12.7 g) of I2 dissolved in a liter of distilled water, and from these standard solutions the required concentrations were prepared for the purpose of study of the factors affecting adsorption.

Diagnosis of carbon and raw material

The chemical surface functions of the activated carbon and the raw material were determined using infrared spectrophotometer (FT-IR) and the surface morphology was diagnosed using high-efficiency scanning electron microscope (FE-SEM).

Preconcentration

The adsorption of trace concentrations of cadmium ions (Cd^{+2}) on the surface of activated carbon (CHP) has been studied in two ways:

Batch method : Three standard solutions of different volumes were prepared (300,600,900 ml) of cadmium ion (Cd^{+2}) at a concentration of (1 mg/l) at (pH = 6) and one gram of activated carbon (CHP) was added to it. They were stirred continuously for three hours at room temperature, after which the solution was filtered and the cadmium concentration in the filtrate was measured using an atomic absorption device.

Column method : A suitable separation column with a diameter of 2 cm and a height of 40 cm was used, and activated carbon was placed inside it between two layers of fiberglass, with a height of 3 cm. Then, a solution of cadmium ions (Cd^{+2}) was gradually added with a concentration of (1 mg/l) and a volume of (3000 ml) at room temperature at (pH = 6) and a flow rate of (3 ml) per minute. Then the remaining cadmium ions were estimated in the solution descending from column using atomic absorption.

RESULTS AND DISCUSSION

Define surface functions

Infrared spectroscopy (FT-IR)

Pre-treatment Leucaena Seed Powder (LOC) was diagnosed using infrared spectroscopy (F.T.I.R) (Fig. 1). The infrared spectrum showed an absorption beam in the region (3000-2850 cm^{-1}) due to the vibration of the (C-H) bond. an absorption beam belonging to the group (CH3) in the region (1450-1375) cm^{-1}, an absorption beam of the group (CH2) at the region (1465) cm^{-1}, absorption beam belonging to the aliphatic (C-H) group at (3100-3000) cm^{-1} and absorption beam belonging to the aromatic (C-H) bond cm^{-1} at (900-690).

While when diagnosing carbon activated using phosphoric acid (CHP), prepared by burning process (400 °C) using infrared spectroscopy (F.T.I.R) (Fig. 2). The

Table 1 : The values of ash content and moisture for activated carbon samples

AC	C HP1	C HP2	C HP3
Ash content %	2	2	4
Moisture content %	3.6	5.6	5.6

infrared spectrum showed an absorption beam in the region (3105) cm^{-1} due to the vibration of the (O-H) bond, another absorption beam of the (C-H) bond at (1465) cm^{-1}, and an absorption beam of the (C-O) bond at (1633) cm^{-1}.

Scanning electron microscope

The scanning electron microscopy (SEM) technique was used to study and determine the surface formation of the raw material and the surface of the prepared activated carbon before and after adsorption. Fig. 3 shows that the surface of the raw material (LUC) is homogeneous, cohesive, soft and smooth compared to the surfaces of the activated carbon models.We notice that the surface has become rougher after activation and burning at high temperatures. It contains many pores and gaps of different sizes and shapes, as these pores can Providing excellent surfaces. The reason for the availability of these pores is due to the adsorption of pollutant particles, dissociation (LUC) at high temperatures, in addition to the evaporation of volatile compounds present in the raw material. From scanning electron microscopy (SEM) images of activated carbon with phosphoric acid (CHP) after adsorption of cadmium ions (Fig. 5) and compared to (SEM) images before adsorption (Fig 4). We find that the structural formation has changed significantly after adsorption, and that most of the large pores and gaps have been filled and covered with particles of adsorbed materials, which proves that the adsorption process has occurred on the surface of the adsorbents.

Ash and moisture content of activated carbon

Ash content was measured by placing (0.5 g) of the dried prepared activated carbon samples at (110°C) in an incubator for two hours and then placing them in a crucible of known mass and entering the incineration furnace at (800°C) for three hours. Then the moisture content was determined by placing (0.5 g) of activated carbon in a crucible and the whole was weighed and then dried in the drying oven at (110°C) several times until the mass became stable. The results shown in Table 1.

Experimental parameters

Adsorbent mass effect : The effect of activated carbon mass on adsorption was studied by adding different weights of (CHP) which are (0.01, 0.02, 0.03,
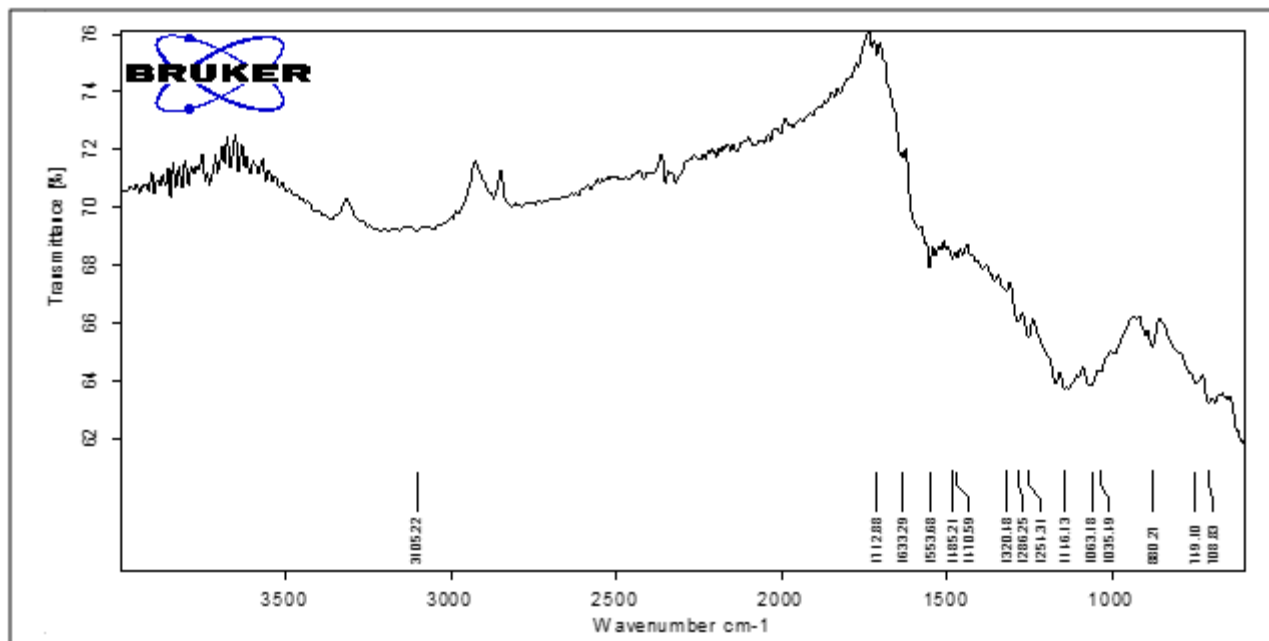
Fig. 2 : FT-IR spectrum of activated carbon with phosphoric acid (CHP).

Table 2 : Effect of adsorbent mass on adsorption.

The adsorbent surface	Adsorbent	Removal (%)									
		0.01g	0.02g	0.03g	0.04g	0.05g	0.06g	0.07g	0.08g	0.09g	0.1g
CHP	PPDA	87.4	90.2	92.6	94.6	95.4	96.3	96.8	97.2	97.4	97.4
	PNP	85.2	88	90.6	92.8	94.4	95.6	96.8	97.2	98	98.2
	Cd^{+2}	78.5	81.7	84.5	87.3	89.5	91.3	93	94.5	95.3	95.7

Table 3 : The effect of contact time on the rate of adsorption.

AC	Adsorbent	Removal (%)							
		15 min.	30 min.	45 min.	60 min.	90 min.	120 min.	150 min.	180 min.
CHP	PPDA	90.2	94.3	95.7	96.8	97.4	97.4	97.4	97.4
	PNP	90.4	95	96.2	97.3	98	98	98	98
	Cd^{+2}	86.2	91.4	93	94.2	95	95	95	95

…, 0.1g) to (50 ml) of (PPD), (PNP), (Cd^{+2}) with a concentration of (100 mg/l) was placed in a vibrating water bath at a temperature of (25°C) and for a time of (3h). From the results shown in Table 2. It can be seen that the removal percentage increases with the increase of the adsorbent surface mass. Keep increasing until it reaches a convergent value (Azouaou *et al*, 2010). Also, the adsorption efficiency increases with the increase in the adsorbent dose and the number of effective functional sites for bonding with the adsorbed material and continues to increase with the use of larger doses of the adsorbent material until a layer of ions is formed covering the surface

to prevent adsorption again (Wan Ngah and Hanafiah, 2008). From the percentage values, we find that the optimal dose of activated carbon was at (0.1g), where the highest adsorption rate of (CHP) was recorded for (PPD) 97.4%, for (PNP) 98.2% and for cadmium ion (Cd^{+2}) was 95.7% as shown in fig 6,7,8. (Wang *et al*, 2009).

The effect of contact time : It was studied by preparing several solutions of volume (50 ml) at a concentration of (100mg/l), for each of (PPDA) (PNP) (Cd^{+2}) and adding to it (0.1g) of (CHP). It was placed in a vibrating water bath at (25°C) and at different times
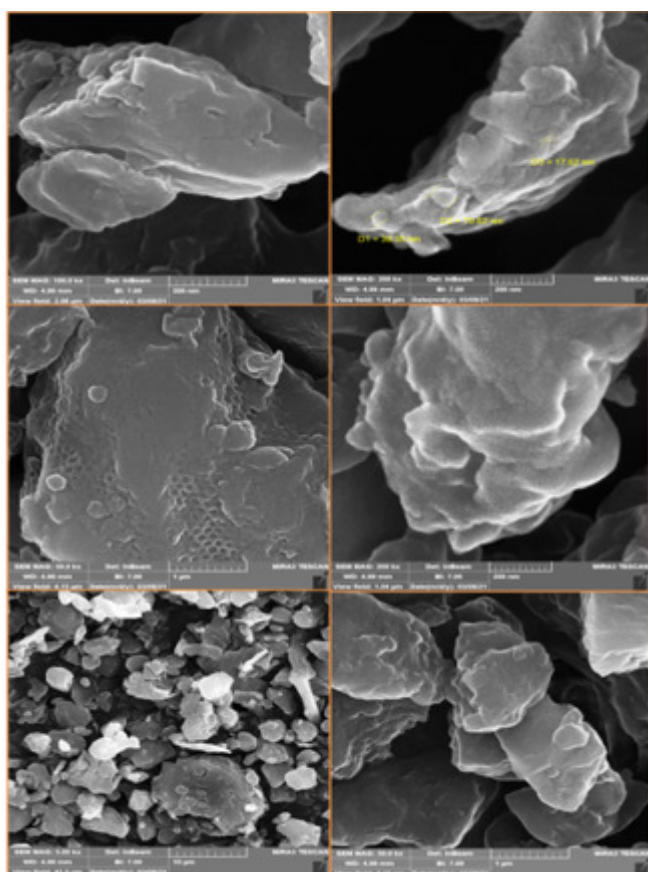
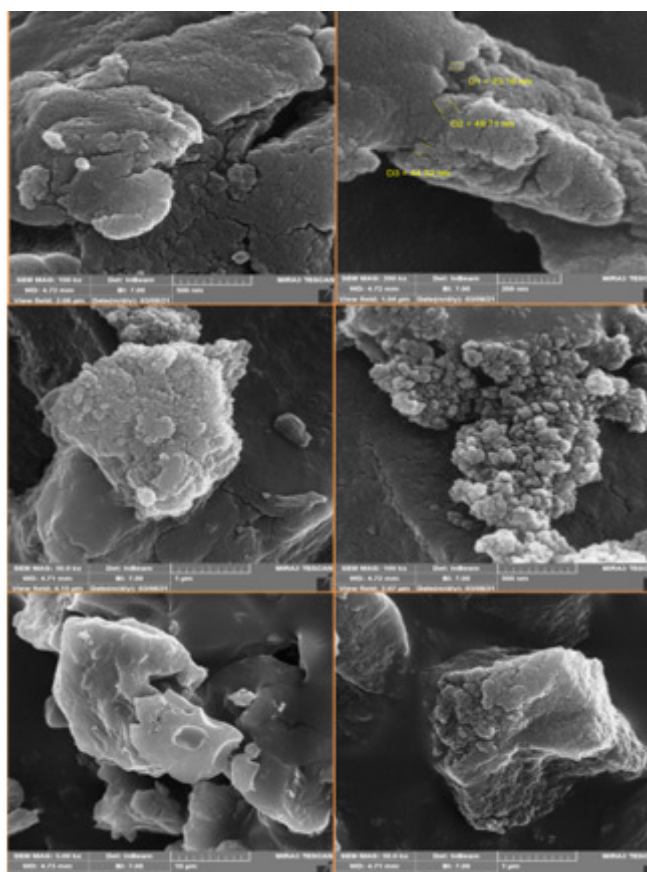**Fig. 3 :** SEM images of pre-treatment Leucaena seed powder (LUC).

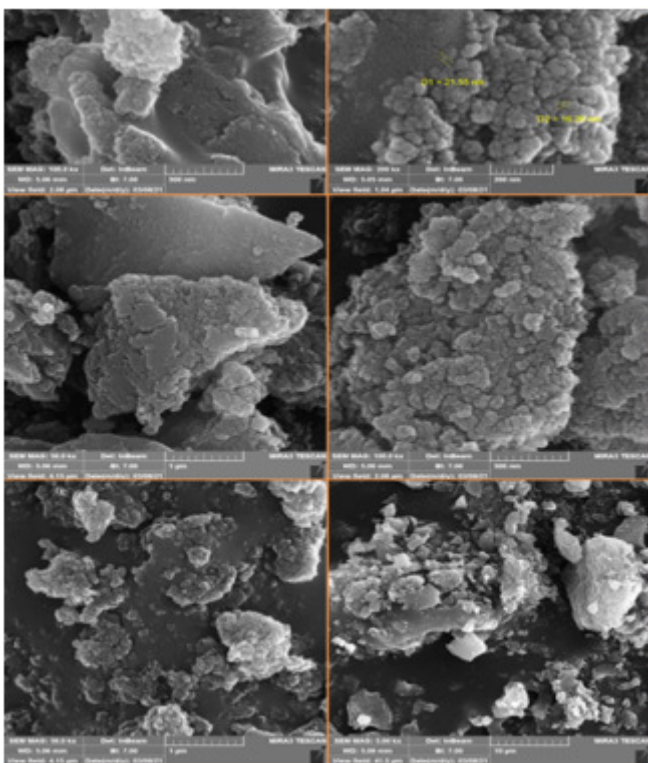**Fig. 4 :** SEM images of activated carbon with phosphoric acid (CHP) before adsorption.

Fig. 5 : SEM images of activated carbon with phosphoric acid (CHP) after adsorption.

Table 4 : The effect of temperature on the rate of adsorption.

AC	Adsorbent	Removal (%)		
		298K	**308K.**	**318K**
CHP	PPDA	97.4	97.7	97.8
	PNP	98	98.7	99
	Cd^{+2}	95	95.4	95.7

(15, 30, 45, 60, 90, 120, 150, 180 min). The solution was filtered after the specified time for each sample and the remaining concentration was measured (Table 3). It shows that adsorption occurs quickly during the first thirty minutes, due to the presence of a large number of vacant adsorption sites on the outer surface of the adsorbent material, which facilitates the rapid adsorption process during that time period (Babel and Kurniawan, 2003).

Then we note that the adsorption rate decreased after the thirty minute and the process became slow as the adsorption sites on the surface reached saturation, which causes a decrease in several adsorption sites. The results recorded that the adsorption time for (CHP) was at 90 min for PPDA by 97.4%, PNP by 98%, Cd^{+2} by 95% as shown in fig 9,10,11 (Abdel-Fattah *et al*, 2011).

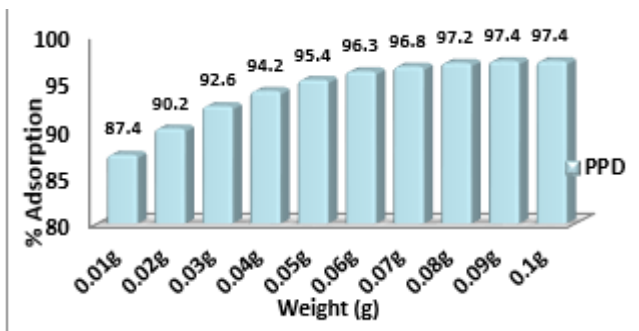**Effect of temperature :** Study by preparing several
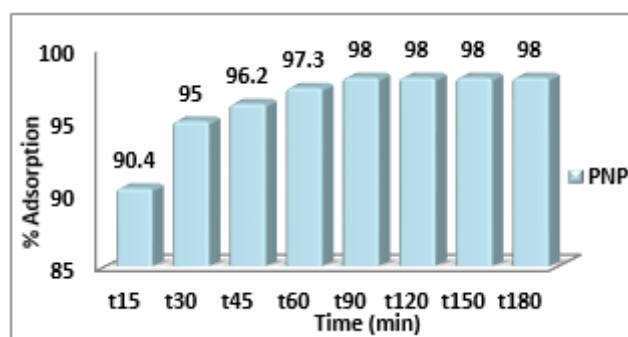
Fig. 6 : Effect of CHP mass on adsorption (PPD).

Fig. 7 : Effect of CHP mass on adsorption (PNP).

Fig. 8 : Effect of CHP mass on adsorption (Cd^{+2}).

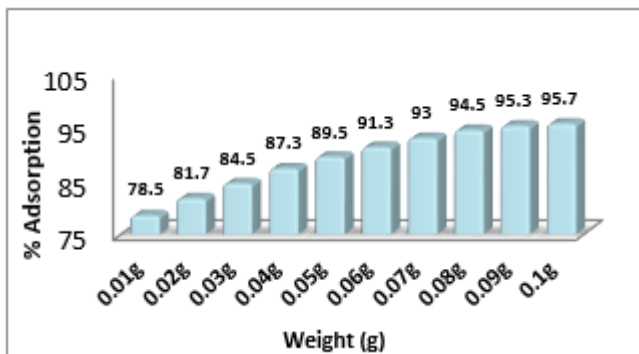**Fig. 9 :** Shows the effect of contact time on adsorption (PPDA) of CHP.

Fig. 10 : Shows the effect of contact time on adsorption (PNP) of CHP.

Fig. 11 : Shows the effect of contact time on adsorption (Cd^{+2}) of CHP.

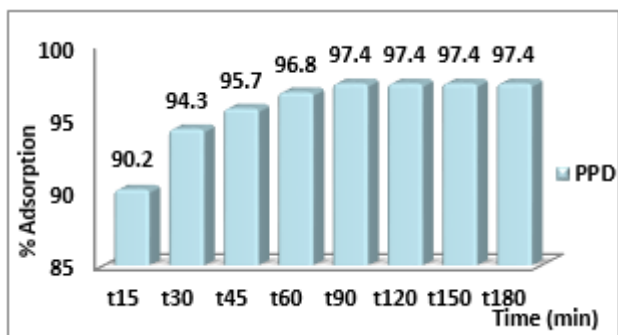**Fig. 12 :** Effect of temperature on adsorption (PPD) of CPH.
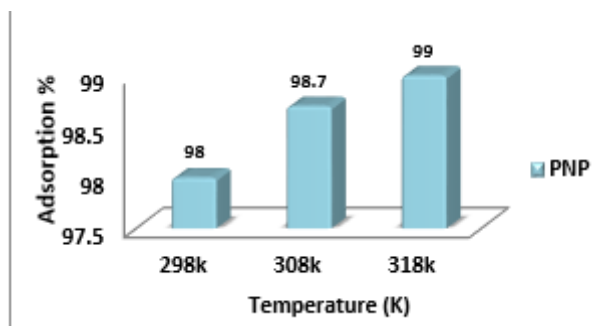

Fig. 13 : Effect of temperature on adsorption (PNP) of CPH.

Table 5 : The effect of pH on the adsorption rate.

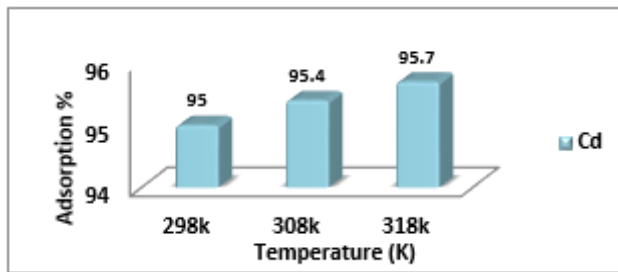| AC	Adsorbent	Removal (%)						
		pH=2	pH=3	pH=4	pH=5	pH=6	pH=7	pH=8
CHP	PNP	98.5	98.4	98.3	98.2	98	97.2	96.4
	Cd^{+2}	90.2	93	94.2	94.7	95	94.3	92.7

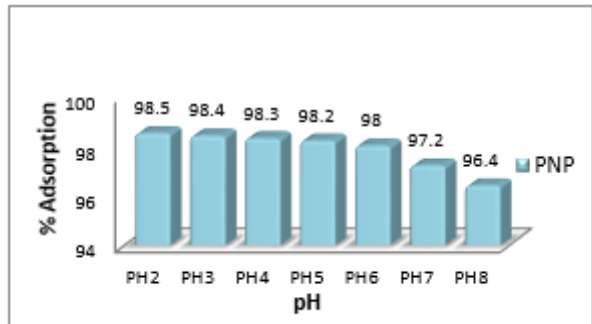**Fig. 14 :** Effect of temperature on adsorption (Cd^{+2}) of CPH.

Fig. 15 : Shows the effect of pH on PNP adsorption of activated carbon.
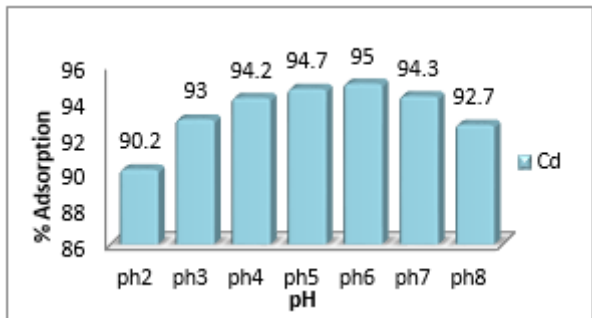

Fig. 16 : Shows the effect of pH on Cd^{+2} adsorption of activated carbon.
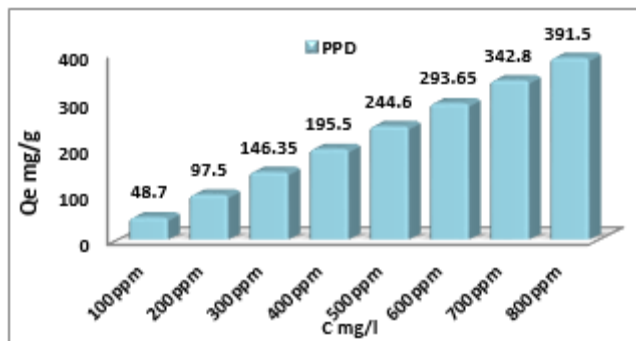

Fig. 17 : shows the effect of the initial concentration of (PPD) for adsorption on (CHP).
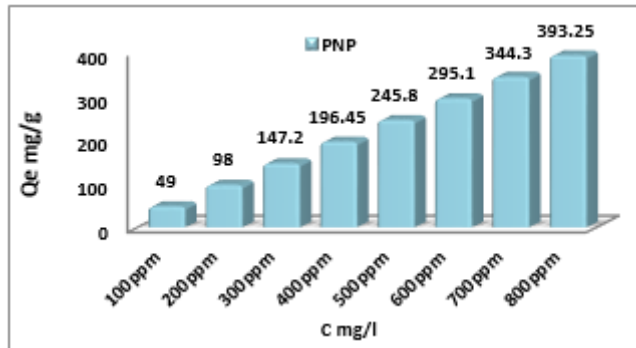

Fig. 18 : shows the effect of the initial concentration of (PNP) for adsorption on (CHP).
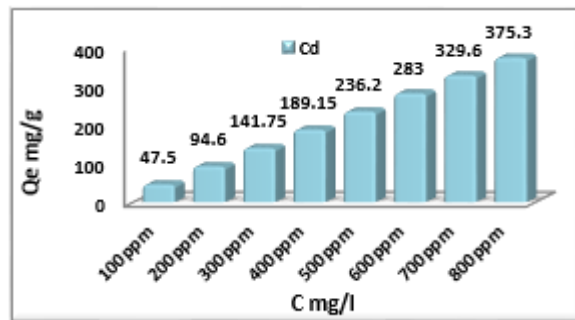
Fig. 19 : shows the effect of the initial concentration of (PNP) for adsorption on (CHP).

Table 6 : Shows the values of adsorption capacity before and after the equilibrium process.

	C^0	Ce	Qt	Qe	Temp	Time
Metal	100	97.4	14	13	25° C	3h
PPD	100	97.7	9	11.5	35° C	3h
	100	98	9.2	10	25° C	3h
PNP	100	99	8.3	5	35° C	3h
	100	95	5.9	25	25° C	3h
Cd^{2+}	100	96	6.1	20	35° C	3h

solutions of volume (50 ml) with a concentration of (100 mg/l) for each of (PPD), (PNP), (Cd^{+2}), adding to it (0.1 g) of (CHP). Then placed in a vibrating water bath at different temperatures (25, 35, 45, 55°C) for 3 hours.

The solutions were filtered and the residual concentration (Ce) measured at each temperature. From the results shown in Table 4. We note that the percentage of adsorption (Cd^{+2}), (PNP), (PPD) on (CHP) surfaces

Table 7 : Thermodynamic values for cadmium and dyes.

Metal	ΔH (J\mole)		ΔS (J\mole.K)		ΔG (J\mole)	
	25° C	35° C	25° C	35° C	25° C	35° C
PPD	4287	4267	10.35-	23.42-	1203-	2940-
PNP	2484	2464	3.620-	1.979-	-1384	-1854
Cd²⁺	2397	2458	-42.89	43.00-	1127-	1124-

increases with increasing temperature. This can be explained by the fact that the increase in temperature leads to a decrease in the viscosity of the solution and the release of functional groups for adsorption (Anayurt *et al*, 2009). Consequently, the pores on the surface of the adsorbent material increase, as well as the increase in the rate of diffusion of the adsorbed molecules on the surface of the adsorbent material due to the increase in its kinetic energy with the increase in temperature, and the possibility of an absorption process occurring in conjunction with the adsorption process, which is called sorption, which means that the process of adsorption and absorption occurs together (Argun *et al*, 2007).

Effect of pH : The effect of (pH) on adsorption was studied by preparing several solutions of (50 ml) volume at a concentration of (100 mg/l), the pH was adjusted at (2, 3, 4, 5, 6, 7, 8) for each of (PNP), (Cd⁺²), with the addition of (0.1g) of activated carbon. It was placed in a shaking water bath at a temperature of (25°C) for three hours, the solutions were filtered and the residual concentration was measured at each pH and then the percentage of adsorption was calculated (Krika *et al*, 2015). The amount and type of electrostatic charges carried by the adsorbent molecules directly control the pH of the medium. So, the percentage of adsorption is proportional to the pH of the aqueous medium. As we can see from the obtained results shown in Table 2. The percentage of the removal of para-nitro-phenol (PNP) decreased with the increase of pH, reaching the highest adsorption rate at pH = 2, after which the adsorption rate began to decrease significantly until there was a decrease in the adsorption rate at pH = 6 (Ho and McKay, 1998).

While the percentage of Cd⁺² ion adsorption increased with the increase in pH, reaching the highest percentage at pH = 6. The reason is that the surface of the adsorbent material acquires positive charges at low pH, so there is a strong attractive force between (PNP) (carrying negative charges) and the adsorbent material. While there is a repulsive force between the Cd⁺² ion (carrying a positive charge) and the adsorbent. The surface of the adsorbent material tends to acquire negative charges when the pH increases, and the opposite occurs (Abdel-Fattah *et al*, 2011). We also notice a decrease in the adsorption rate of the Cd⁺² ion at pH = 6, due to the formation of Cd (OH)$_2$ due to the presence of a large number of OH⁻ ions in the solution as shown in the figures 6,7 (Wan Ngah and Hanafiah, 2008).

Effect of Initial concentration : It was studied by preparing several solutions with a volume of (50 ml) and different concentrations (100, 200, 300, 400, 500, 600, 700, 800 mg/l of (PPD) (PNP) (Cd⁺²) and adding to it (0.1g) of (CHP), respectively. Then they were placed in a vibrating water bath at (25°C) during three hours. The solutions were filtered and the concentration was measured after adsorption and the amount of adsorbed (Qe) was calculated at each concentration, as shown in the Figs. 15, 16, 17 (Anayurt *et al*, 2009).

Calculation of thermodynamic

From Table 6, we get the values of K1, K2 by using the following equation:

$$\frac{t}{q_t} = \frac{1}{Kqe^2} + \frac{t}{qe} \tag{1}$$

To obtain the activation energy, Ea, the following equation is applied:

$$\ln\frac{k_1}{k_2} = \frac{k_a}{R}\left(\frac{T_2 - T_1}{T_1 \times T_2}\right) \tag{2}$$

To get ΔH for the studied ions, we get it from the following equation:

$$\Delta H = Ea - RT_1$$
$$\Delta H = Ea - RT_2 \tag{3}$$

To calculate ΔS, we use the following equation:

$$Ln\ Kd = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT_1} \tag{4}$$

where, the value of *Kd* is Qe/Ce.

Thus, we get the value of the change in free energy through the Gibbs equation for equilibrium:

$$\Delta G = \Delta H - T\Delta S \qquad (5)$$

The following results were obtained by applying the above equations as shown in Table 7 (Mustapha *et al*, 2019).

CONCLUSION

- Activated carbon was prepared using Leucaena seeds, and treated with phosphoric acid to increase its efficiency in the adsorption process.

- Surface diagnostics using FT-IR and FE-SEM technology

- The adsorption capacity of cadmium and dyes was determined by activated carbon with phosphoric acid (CHP). It was found that the adsorption capacity of cadmium and dyes increases with time, where it is greater at the time (180) hours. Also, the adsorption capacity of PPD dye is greater than that of PNP dye, which in turn is greater than cadmium over time when using the two surfaces.

- The adsorption capacity increases with decreasing temperature and decreases with increasing temperature for cadmium and dyes due to the separation of cadmium and pigments from the surface of activated carbon.

- The adsorption capacity increases at (pH = 6, pH = 5) for cadmium and pigments (PPD) and PNP) in relation to the acidity function.

- From the positive values of ΔH (J\mole) we conclude that the reaction is exothermic, and from the negative values of ΔS (J\mole.K) it shows that the reaction is spontaneous, as well as the negative values of ΔG (J\mole) indicating that the reaction occurs spontaneously.

Recommendations

- The use of prepared activated carbon in soil treatment and air purification.

- Manufacture of filters from coal to treat air, water and soil.

- The possibility of using the remains of other plants in the manufacture of absorbent surfaces.

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