

## Using ozone for activation of manufactured porous media to improve the removal efficiency of heavy metals from industrial wastewater

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### ABSTRACT

Adsorption processes are being widely used by various researchers for the removal of heavy metals from waste water and activated carbon has been frequently used as an adsorbent. Despite its extensive use in water and wastewater treatment industries, activated carbon remains an expensive material. In recent years, the need for safe and economical methods for the elimination of heavy metals from contaminated water has necessitated research interest toward the production of low cost alternatives to commercially-available activated carbon. Activated carbon taken from pine cone (ACPC) was used as adsorbent to remove Fe<sup>+2</sup> ions from industrial wastewater. In this study, ozonation, ozon pumping to the porous media, was the effective reason of removal efficiency increasing. Ozonation has been conducted on the adsorption process of removing Zn<sup>+2</sup> under specific conditions, which is the same conditions and pollutant that were studied in the aforementioned research, where the removal efficiency before and after ozonation was compared and found it reached to 99.55% instead of what was it been by 96%. Batch adsorption experiment was conducted to examine the effects of adsorbent dosage, contact time, pH and stirring rate on adsorption of Fe<sup>+2</sup> from the wastewater. The obtained results showed that, the adsorption of the metal ions was adsorbent dosage, contact time, pH and stirring rate dependent. The optimum adsorbent dosage, stirring rate and pH, were found to be at 3 g, 250 rpm, 60 ppm initial concentration of Fe<sup>+2</sup> and pH 6 respectively. Environmental parameters were studied in this work including pH, total dissolved solid and total suspended solid. Kinetic studies were evaluated by Langmuir, Freundlich and Elvich isotherm models. Freundlich isotherm afforded the best fit to the equilibrium data. Maximum adsorption of Fe (II) was 97% observed in specific condition.

**Keywords:** Adsorption, Activated carbon, Pine cone (ACPC), Ozonation.

**Article type:** Research Article.

### INTRODUCTION

Soil pollution by heavy metals has become a major environmental concern in recent decades, resulting from the increase in the intensity of industrial activity and some agricultural practices (Nriagu & Pacyna 1988; Gaballah & Kilbertus 1998). This fact favors that metal concentration in some soils rises to potentially phytotoxic levels (Kabata Pendias & Pendias 2001; Celik & Demirbas 2005). Heavy metals in the soil are subjected to adsorption-desorption processes, which affect their geochemical behavior, particularly in terms of transportation, distribution and impact on biological systems. Due to heavy metal are not biodegradable, their presence in soil, rivers and lakes can lead to their accumulation in living organisms (Argun & Dursun 2008). Pine bark can be considered as a low cost by-product from the forest activity because it is simple to process and is abundant in nature. It contains

several organic compounds such as lignin, cellulose and tannins (Bermúdez Alvite & Touza Vázquez 1999; Miranda *et al.* 2012). Activated carbons, on the other hand, are among the effective adsorbents because of their excellent sorption capacity. Their use has been often prohibited mainly due to their relatively high cost, low selectivity and regeneration problems; they can be regenerated by thermal desorption of the adsorbed substances or by liquid phase extraction in the case of soluble adsorbed species, although the former method is not very environmentfriendly and leads to partial mass loss of the adsorbent (Mohammadi *et al.* 2010; Şvab *et al.* 2011; Jasinska *et al.* 2011; Campean *et al.* 2012; Dabioch *et al.* 2013; Eliyas *et al.* 2013; Hadi *et al.* 2014; Kamil *et al.* 2016). The microporous nature of the majority of porous activated carbons is well suited to many applications, including molecular sieving, sorption and catalytic reactions of small molecules. Of course, there are numerous other potential applications, such as sorption of dyes, in which the presence of wider pores, preferably in the mesoporous range, would be advantageous (Asouhidou *et al.* 2009). Dyes and heavy metals are two contaminants commonly found in the wastewater of several industries. Specifically, heavy metals are discharged by industries such as agrochemical, petrochemical and fertilizers, whereas dyes are found principally in effluents of dye manufacturing industries, electroplating factories, distilleries and food companies (Hernandez Montoya *et al.* 2011; Trevino Cordero *et al.* 2013). It was further reported that an anionic dye favored the electrostatic interactions with heavy metals or might create new specific sites for the adsorption process (Tovar Gomez *et al.* 2012). For treating contaminated effluents many technologies have been developed over the years. Most important of those techniques depend on chemical precipitation, filtration, ion-exchange, reverse osmosis and membrane systems. However, all these techniques have their inherent advantages and limitations in application. In the last few years, adsorption has been shown to be an alternative method for removing dissolved metal ions from liquid wastes (Bayat 2002). Adsorption is a mass transfer process in which substances present in a liquid or gas phase are adsorbed or accumulated on a solid and thus removed from the liquid or gas; (Crittenden *et al.* 2005). Application includes the purification of drinking water, removal of harmful pollutants from waste water. It also has application in air pollution control and many processes in chemical engineering;( Ebrahim 2008; Mckay 2001). A porous solid material of large surface area per unit mass in which adsorption of liquids or gases occurs on its surface is known as "adsorbent". The material to be adsorbed on the adsorbent is known as "adsorbate" (Bhatia 2001; Ahmed 2006). There are two general types of adsorption: physical (physisorption) and chemical (chemisorption) adsorption. These kinds depend on the nature of the force between the adsorbate and the adsorbent; (Mckay 2001).

## MATERIALS AND METHODS

### Activation of Adsorbent Material, Activated carbon taken from pine cone (ACPC).

Activated carbon taken from pine cone (ACPC), that prepared in the aforementioned research, was activated by ozon treatment and washed with deionized water and then heated to 450 °C for 4 hours.



**Fig. 1.** Activated carbon derived from pine cone (ACPC).

Sieve Analysis using US standard sieve type was achieved using sample weighing 350 g from ACPC media. Fig. 2 shows the graphical representation of results.

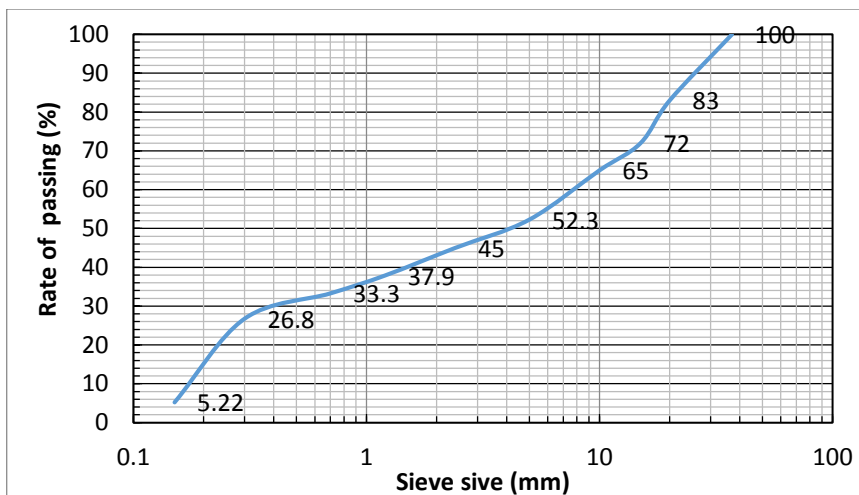


Fig. 2. Illustration of sieve analysis of ACPC.

where:

Effective size  $D_{10}$ = 0.54 mm,  $D_{25}$ = 1.25 mm,  $D_{30}$ = 1.5 mm,  $D_{60}$ = 2.4 mm,  $D_{75}$ = 2.95 mm.

Uniformity coefficient (Cu) = 4.44

Coefficient of curvature (Cc) = 1.736

Sorting coefficient (Ct) = 1.54.

**Batch adsorption test on ACPC**

The adsorption of heavy metals by the adsorbents was studied using batch experiments. 0.5, 1, 2.5 and 3 g of ACPC was added to 50 mL of different concentrations of heavy metal standard solutions (50, 60, 70 and 80) ppm of  $Fe^{+2}$ , while the pH of the solution was in range (4-10) using 1.0 N HCl and NaOH. The solution was sonicated for 20 min at 250 rpm and then allowed to cool and followed by equilibration at room temperature for 24 hours. The Langmuir adsorption model is valid for single-layer adsorption. It is based on the following assumption:

1. The maximum adsorption corresponds to a saturated monolayer of solute molecules on the adsorbent surface.
2. The energy of adsorption is constant.
3. There is no transmigration of adsorbate in the plane of the surface;(Lucas & Cocero 2004). The Langmuir isotherm equation is:

$$q_e = \frac{q_m b C_e}{(1+b C_e)} \quad \text{----- (1)}$$

where:

$q_e$ : is the sorbed metal ions on the biomass ( $mg\ g^{-1}$ ).

$q_m$ : is the maximum sorption capacity for monolayer coverage ( $mg\ g^{-1}$ ).

$b$ : is a constant related to the affinity of the binding site.

$C_e$ : is a metal ions concentration in the solution at equilibrium ( $mg\ L^{-1}$ ).

In order to evaluate the parameters ( $q_m$  &  $b$ ) it is better to rewrite eq. (1 a) as:

$$\frac{C_e}{q_e} = \frac{1}{b q_m} + \frac{1}{q_m} C_e \quad \text{----- (1 a)}$$

The Langmuir isotherm is used most frequently to describe the adsorption isotherm which is limited by the assumptions of uniform energies of adsorption on the surface of adsorbent. It is based on the following four assumptions; (Sharker & Acharya 2006):

- The surface of the adsorbents is uniform, that is, all the adsorption sites are equivalent.
- There is no interaction between molecules adsorbed on neighboring sites.
- All adsorption occurs through the same mechanism.
- Molecules are adsorbed at the define sites on the adsorbent surface.

Weber & Chakkravorti (1972) reported that the essential characteristics of a Langmuir isotherm equation could be expressed in terms of a dimensionless constant separation factor or equilibrium parameter, 'R<sub>s</sub>' which is defined by the following equation:

$$R_s = \frac{1}{1+bC_i} \quad \text{----- (2)}$$

The separation factor as described above was calculated using the Langmuir model constants. This separation factor yields the type of isotherm as shown in Table 1.

**Table 1.** Value of separation factor and type of isotherm (Weber & Chakkravorti 1972).

Value of R <sub>s</sub>	Type of isotherm
R <sub>s</sub> > 1	Unfavorable
R <sub>s</sub> = 0	Linear
0 < R <sub>s</sub> < 1	Favorable

Freundlich equation is derived to model the multilayer sorption and for the sorption on heterogeneous surfaces. The Freundlich isotherm theory says that the ratio of the amount of solute adsorbed onto a given mass of sorbent to the concentration of the solute in the solution is not constant at different concentrations (Zhihui *et al.* 2009) The equation may be written as:

$$q_e = K_F C_e^{1/n} \quad \text{(Non-linear form) ----- (3)}$$

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad \text{(Linear form) ----- (4)}$$

where:

K<sub>F</sub>: constant indicates the relative adsorption capacity of the adsorbent (mg g<sup>-1</sup>).

n: constant indicative of the intensity of the adsorption.

Both the constants K and n, being indicative of the extent of adsorption and the degree of non-linearity between solution and concentration, respectively. Linear Freundlich form is obtained by plotting log q<sub>e</sub> versus log C<sub>e</sub>. From the figure the adsorption coefficients could be evaluated as explained in figure (3.1). Elvich equation model bases on a kinetic principle assuming that the adsorption sites increase exponentially with adsorption, which implies a multilayer adsorption. The model is expressed as:

$$\frac{q_e}{q_m} = K_E C_e \exp\left(-\frac{q_e}{q_m}\right) \quad \text{----- (5)}$$

Where K<sub>E</sub> is the Elovich equilibrium constant (l/mg) and q<sub>m</sub> is the Elovich maximum adsorption capacity (mg/g). These constants can be calculated from the slope and intercept of the plot for ln (q<sub>e</sub>/C<sub>e</sub>) versus q<sub>e</sub>. Table 2 represents the linear form of the above models and could be considered as good tools to evaluate the required parameters.

**Table 2.** Linear forms of single component isotherm models.

Isotherm	Linear form	Plot
<b>Langmuir</b>	$\frac{C_e}{q_e} = \frac{1}{q_m b} + \frac{1}{q_m} C_e$	$\frac{C_e}{q_e}$ vs. $C_e$
<b>Freundlich</b>	$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e$	$\ln q_e$ vs. $\ln C_e$
<b>Elovich</b>	$\ln \frac{q_e}{C_e} = \ln K_E q_m - \frac{q_e}{q_m}$	$\ln \frac{q_e}{C_e}$ vs. $q_e$

**RESULTS AND DISCUSSION**

**Activation of ACPC by ozonation**

In this test, the ozone was pumped through the porous media (ACPC) to improve the removal efficiency of Zn<sup>+2</sup>, which studied in aforementioned research in batches under the same conditions, 3 g of porous media which has

pH = 7 was added to 50 ml of contaminated solution for batch tests at 25 °C and 20ppm of Zn<sup>+2</sup>. In Figs. 3 and 4, the removal efficiencies of Zinc increase with increasing of ozone pumping time until it reached to the point at which the removal efficiencies gradually decreased (Rawaa *et al.* 2019).

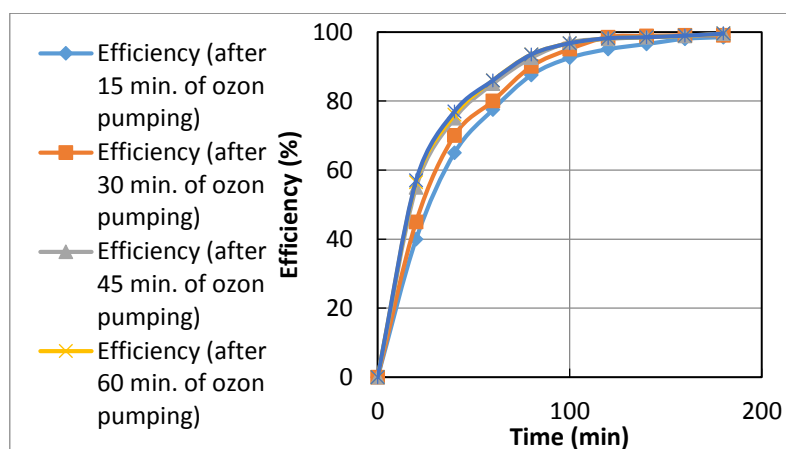


Fig. 3. Effect of ozonation on removal efficiency.

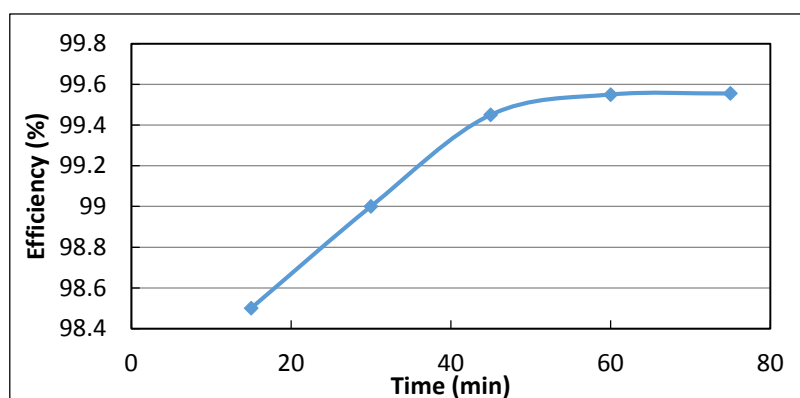


Fig. 4. Ozon pumping time with removal efficiency.

### Effect of Initial Iron Concentration

Initial Iron concentration is important parameter for removing metal ions from industrial wastewater. Figs. 3 and 4 show the effect of initial Fe<sup>+2</sup> concentration on the percent removal efficiency under specific conditions (contact time = 180 min; speed = 250 rpm; adsorbent dose = 3 g 50 mL<sup>-1</sup>; pH = 6; T = 25 °C). As shown in this Fig., the percentage removal decreased significantly from 97% to 80% with raising in initial Fe<sup>+2</sup> concentration from 60 to 80 mg L<sup>-1</sup>. This is because, at low metal ion/adsorbent ratios, metal ion adsorption involves higher energy sites giving higher adsorption efficiency. On the other hand, as the metal ion/adsorbent ratio increases, the higher energy sites are saturated and adsorption begins on lower energy sites, resulting in decreases in adsorption efficiency (Ekpete *et al.* 2010).

### Effect of contact time

The effect of contact time on removal of metal ions was studied for a period of 180 min. three g of the adsorbent (ACPC) was added to different conical flask containing 50 mL of wastewater, the flask was closed and placed in a rotary shaker, and agitated at 250 rotation per minute (rpm), for each of the different contact times chosen (20, 40, 60, 80, 100, 120, 140, 160, and 180 min). The content of each flask was filtered and analyzed after each agitation time (Onyeji *et al.* 2011).

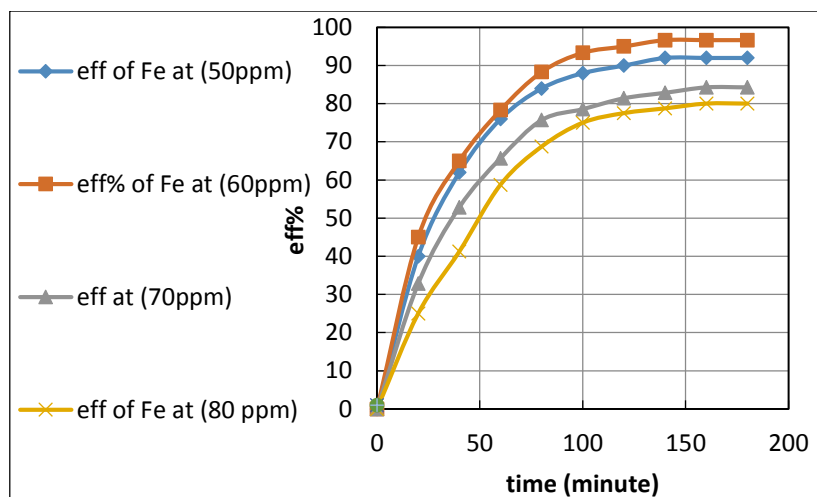


Fig. 5. Effect of Fe<sup>2+</sup> concentration on removal efficiency.

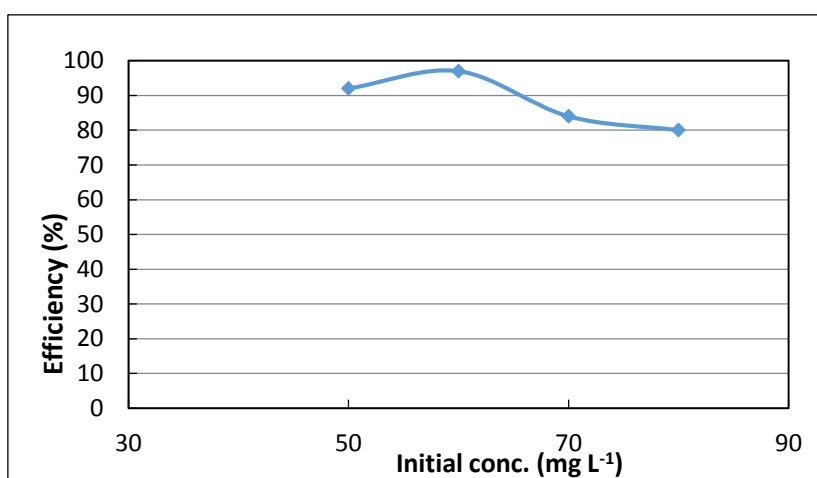


Fig. 6. Initial concentration with the removal efficiency of Fe<sup>2+</sup>.

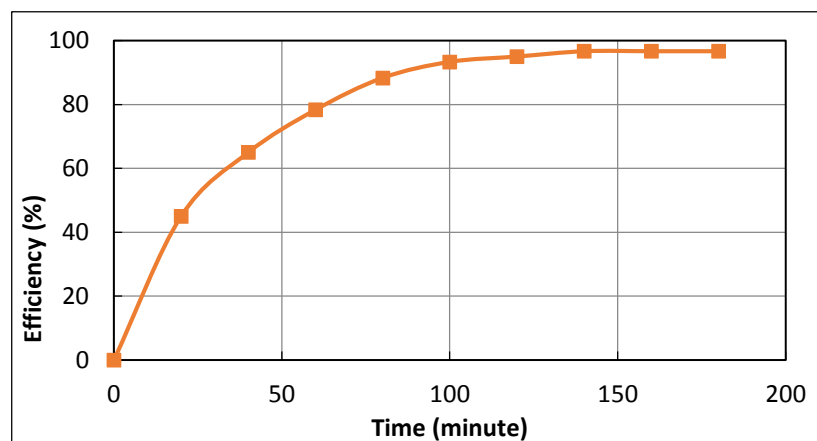


Fig. 7. Effect of contact time on removal efficiency of iron.

### Effect of pH

Over a pH range of 4-10, the effect of pH on adsorption on metal ions was studied. For this particular study, 50 mL of wastewater was measured into different 250 mL conical flask and 3 g of the ACPC being the optimum adsorbent from the previous experiment, was added and agitated at 250 rpm for 180 minute.

### Effect of adsorbent dosage

Different dosages of the adsorbents (0.5-3 g) were added in different conical flasks containing 50 mL of wastewater solution, corked and agitated in a shaker for 180 minute at a speed of 250 rotations per minute (rpm)

at a room temperature of 25C. The content of each flask was then filtered and analyzed after the agitation time (Norhafizahbinti *et al.* 2011).

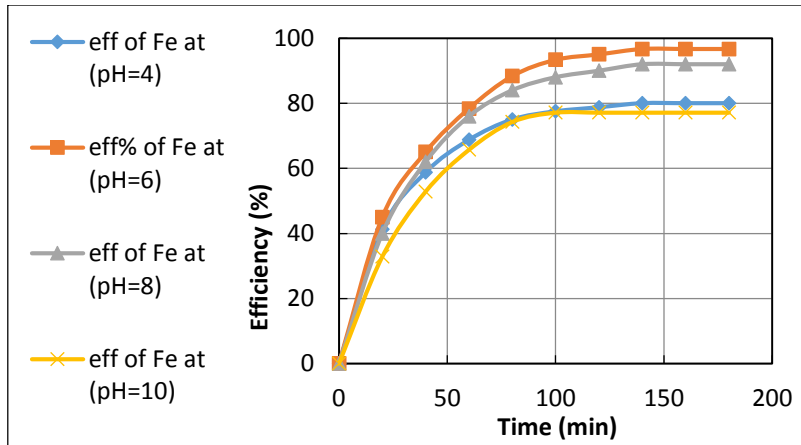


Fig. 8. Removal efficiency of Iron with ACPC media at different pH solution at specific conditions.

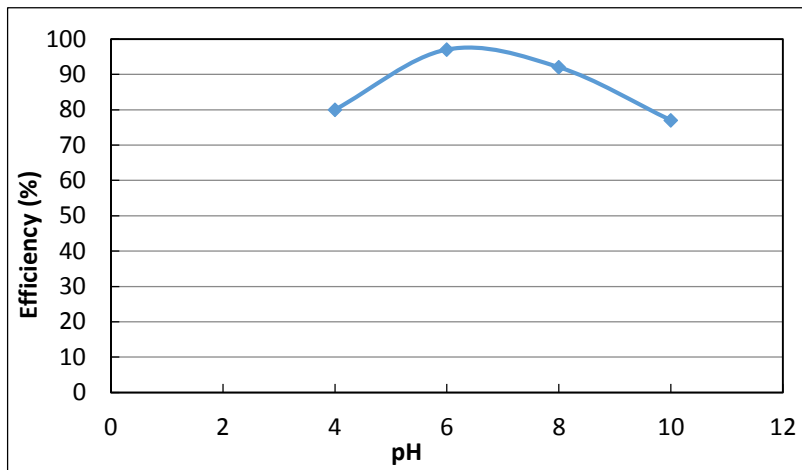


Fig. 9. Effect of pH varying on removal efficiency of Fe at specific conditions.

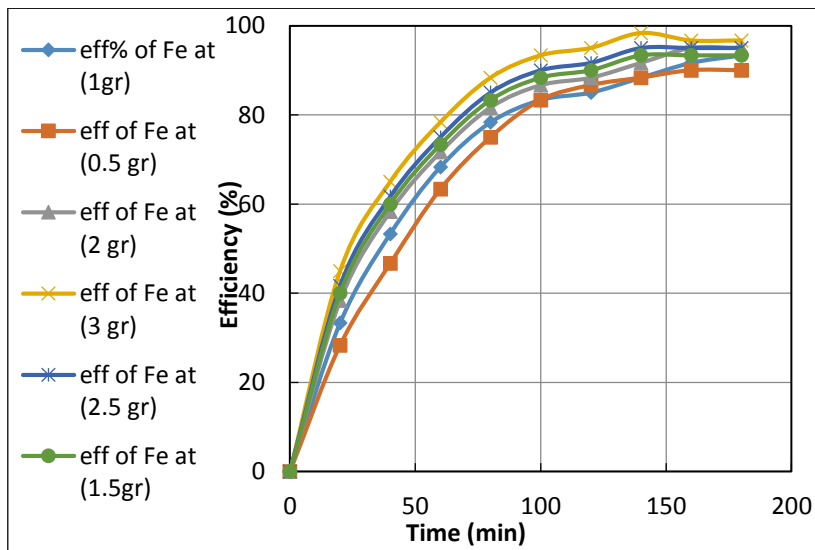


Fig. 10. Effect of ACPC dosage on removal efficiency of Fe<sup>+2</sup>.

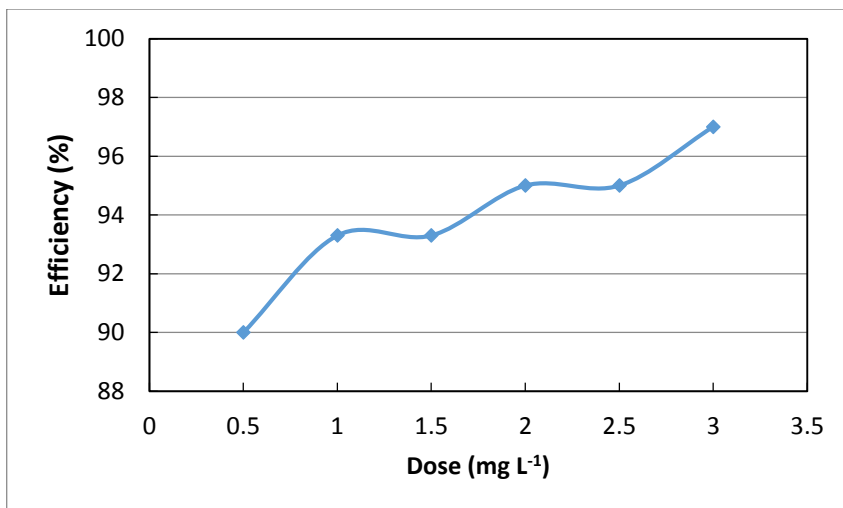


Fig. 11. Adsorbent dose with removal efficiency of iron.

**Sorption isotherms**

Adsorption isotherms are very powerful tools for the evaluation of adsorption process. Adsorption isotherms determine the relationship between the equilibrium concentration and the amount of adsorbate adsorbed by the unit mass of adsorbent at a constant temperature. Langmuir, Freundlich, and Elvich isotherms models are widely used to investigate the adsorption process. The model parameters can be seen further, providing understanding on adsorption mechanism, surface properties, and an affinity of the adsorbent. The Langmuir, Freundlich, and Elvich adsorption constants are estimated from isotherms and their correlation coefficients are given in Table 1. The linearized Langmuir, Freundlich, and Elvich of Fe<sup>+2</sup> ions are given in Figs. 12, 13 and 14.

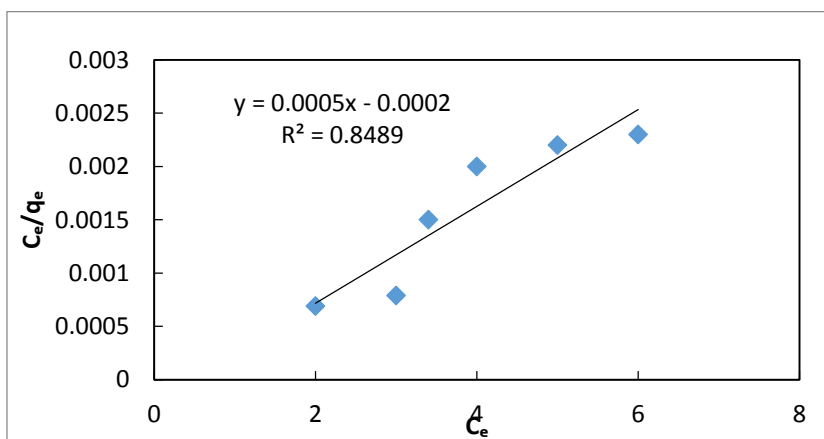


Fig. 12. Langmuir model.

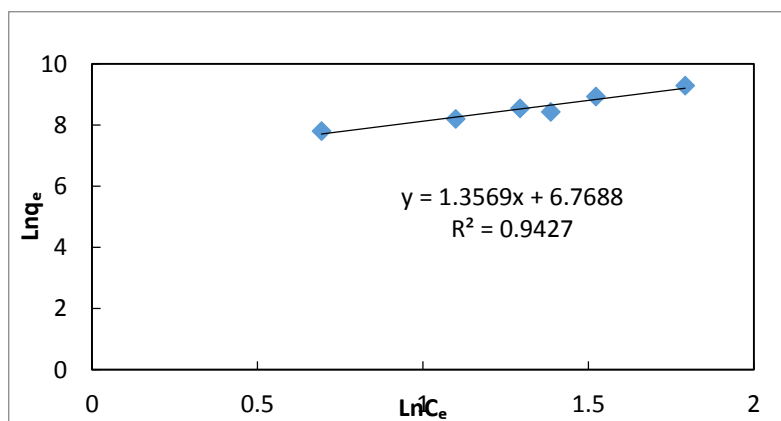


Fig. 13. Freundlich model.



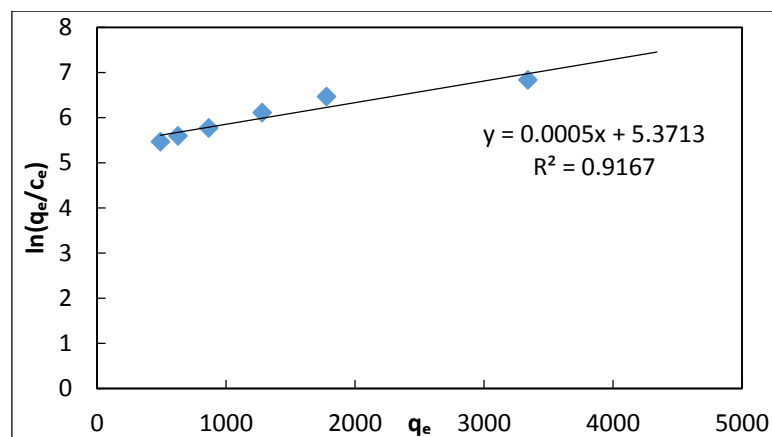


Fig. 14. Elvich model.

Depending on the results, it is clear that the Freundlich isotherm model provides the best correlation in comparing with other isotherm models for  $Fe^{+2}$  sorption.

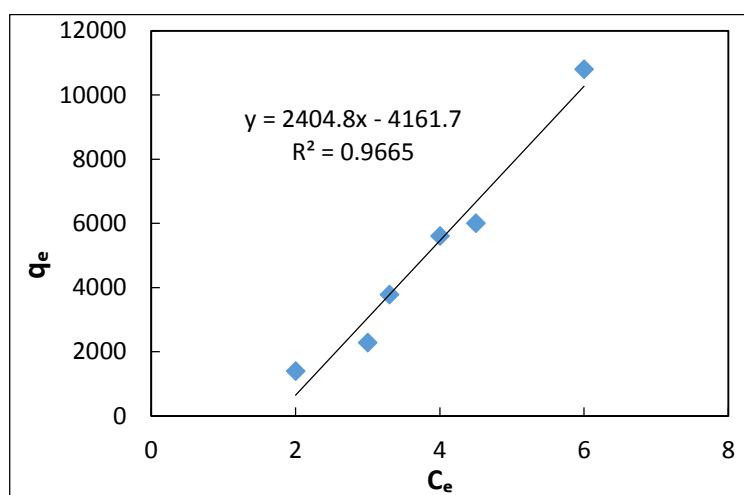


Fig. 15. Comparison of the experimental results with the  $q_e$  values obtained by Freundlich isotherm model for  $Fe^{+2}$  sorbed by ACPC.

**Environmental parameters**

Total dissolved solids, turbidity and total suspended solid were also tested for their important role on the soil and plants. Figs. 13-15 show the variations of TDS, turbidity and TSS concentrations under specific conditions: (180 min, pH 7, 20 ppm of initial values of  $Zn^{+2}$  and 3g of ACPC).

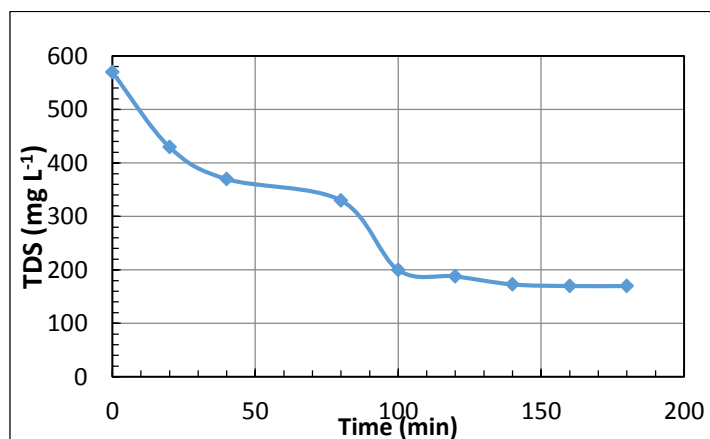


Fig. 16. Variation of the total dissolved solids ( $mg L^{-1}$ ).

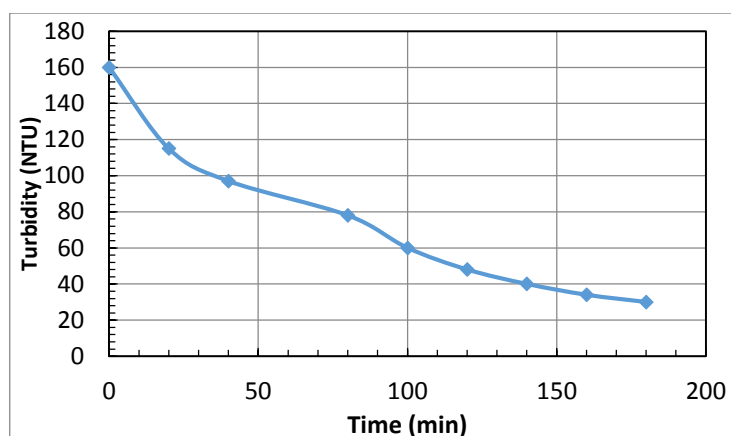


Fig. 17. Variation of the turbidity (NTU).

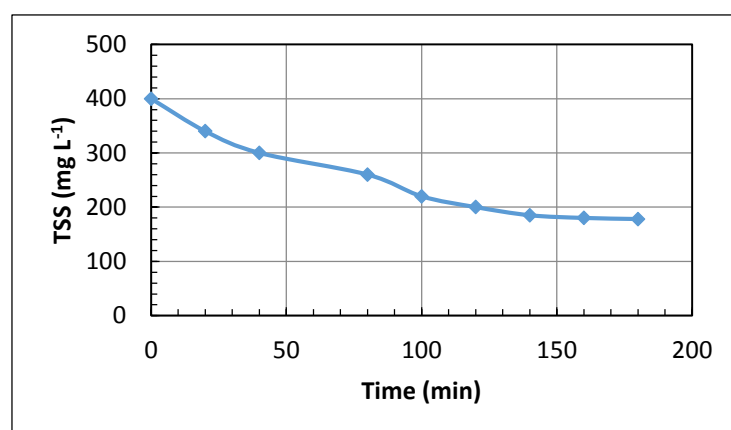


Fig. 18. Variation of the total suspended solids (mg L<sup>-1</sup>).

## CONCLUSION

1. Ozone pumping was found to be a good activated factor for adsorption process.
2. Results showed that low cost adsorbents can be fruitfully used for the removal of Fe<sup>+2</sup> with a concentration of 60 mg L<sup>-1</sup>.
3. It was found that the percentage removal of heavy metals was dependent on the dose of low cost adsorbent (ACPC) and adsorbent concentration.
4. The contact time necessary for maximum adsorption was found to be three hours.
5. The optimum pH range for heavy metal adsorption was 6.
6. Further studies are on the way to investigate PCAC as a suitable adsorbent in a continuous system.

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