J. Indian Water Resour. Soc.,
Vol. 37, No. 4, Oct., 2017

ADSORPTION STUDY FOR CHEMICAL OXYGEN DEMAND REMOVAL FROM AQUEOUS SOLUTIONS USING ALGINATE BEADS WITH ENTRAPPED ACTIVATED CARBON

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ABSTRACT

The aim of this research is to treat wastewater contaminants by reducing chemical oxygen demand (COD) concentration using adsorption process. This study, investigated and optimized the effect of operating parameters to reach an ideal treatment by using alginate beads with entrapped activated carbon (AG-AC). The removal efficiency affected by (contact time, pH, COD concentration, adsorbent dose, and stirring rate). The removal efficiency of COD concentration was about (77 and 89 %) after the adsorbent dose (30 g/L), pH (3), stirring rate (100 rpm), contact time (60 min) and temp (25 ^{0}C). The optimum operating parameters determined for increasing COD removal efficiency.

Keywords: *Alginate beads, Activated Carbon, Entrapped, Chemical Oxygen Demand*

INTRODUCTION

Water covers about two-thirds of the earth's surface to saturate all kinds of life activities but actually this amount of water isn't enough for all human activities, so wastewater reuse is the most important technique to reduce water shortages (Dzurik, 2003, Engelman et al., 1993, Lazarova et al., 2001, Lalzad, 2007, Selendy, 2011, Spellman and Bieber, 2016, Wintgens et al., 2005, Zubay, 2000).

Water pollutants are the most dangerous problem so the governments are facing and trying to find best economic techniques (Abdel-Gawad et al., 2016, Gonzalez-Serrano et al., 2004, Palanaippan et al., 2010, Risch, 2000, Van der Bruggen and Vandecasteele, 2003, Washington, 2004, Worch, 2012), one of these contaminants is organic compounds which have negatively effect on human health. It affects the nervous and the immunological system (Chang et al., 2008, Farhadi et al., 2012, Gaikwad and Mane, 2015, Joshi et al., Karunya et al., Lladó et al., 2016, Oh et al., 2013). The organic content of wastewater measured using parameters such as chemical oxygen demand (COD) (Dobrzyńska et al., 2004, Devi and Dahiya, 2008, Farhadi et al., 2012, Idris et al., 2012, Rakholiya and Puranik, 2012).

COD is a measure of the amount of oxygen required for oxidation of dissolved organic matter in water also it is important for the water quality (the high proportion of COD is an indicator of a high proportion of organic matters and low water quality.) (Hussain et al., 2002, Igbinosa and Okoh, 2009, Kang et al., 1999, Latif and Dickert, 2015, Mamais et al., 1993, Pai et al., 2007, Water, 2015, Worch, 2012). The most widely used in pharmaceutical industries are vitamin C (Ascorbic acid) and lactose, they are responsible for a high proportion of COD (Davarnejad and Nikseresht, 2016, Liu et al., 2016, Shi et al., 2011).

Various techniques employed in wastewater treatment such as coagulation, flocculation process, sedimentation, sand filters, aerobic and anaerobic biological contactors and able to achieve the treated effluents to allowable limits for reuse purposes. These methods are expensive, time consuming, complicated and requires skilled personnel, so it is necessary to investigate a new technology and economically feasible alternatives processes for effectively remove these pollutants (Karunya et al., Murali et al., 2013, Washington, 2004).

Activated carbon is an adsorbent material with a high adsorption capacity, which makes it able efficiently to adsorb many types of contaminants from water wastewater (Gonzalez-Serrano et al., 2004, Lin et al., 2005, Oliveira et al., 2002, Snyder et al., 2007, Yin et al., 2007). Activated carbon cost in wastewater treatment is not low enough and not easy because of dispersion of the powder. To overcome these problems and increasing the removal efficiency entrapped of activated carbon with other material using adsorption process (Lin et al., 2005, Siripattanakul-Ratpukdi and Tongkliang, 2012). It has become one of the best economics and effective process for wastewater treatment, thus this process has aroused great interest during the last years (Ali and Gupta, 2006, Faust and Aly, 2013, Noll, 1991, Rashed, 2013, Worch, 2012).

Alginate is an anionic polysaccharide which has important physical and chemical properties such as stable, chelating ability material, non-toxic, water-insoluble and has a high viscosity (Abdel-Gawad et al., 2016, Bezbaruah et al., 2014, Hill and Khan, 2008, Lee and Mooney, 2012). Calcium alginate is the most utilized entrapment matrix in environmental applications (Hill and Khan, 2008, Kim, 2014, Siripattanakul-Ratpukdi and Tongkliang, 2012, Siripattanakul and Khan, 2010). It is a porous material, allows contaminated aqueous solutions to pass through it and be in contact with the entrapped material (Abdel-Gawad et al., 2016, Bezbaruah et al., 2014, Brachkova et al., 2010, Hill and Khan, 2008, Roy et al., 1987, Siripattanakul and Khan, 2010).

This study aimed to removal of COD from an aqueous solution using alginate beads with entrapped activated carbon (AG-AC) of the wastewater, also to solve the difficulty of activated carbon separation. Supplement this study, application of alginate beads with entrapped activated carbon on each of vitamin C (Ascorbic acid) and lactose.

MATERIALS AND METHODS

Chemicals and reagents

Sodium Hydroxide (NaOH, 99% Pure), Sodium alginate

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Manuscript No.: 1449

$(C_6H_7NaO_6)^n$, 99% Pure), Potassium hydrogen phthalate ($C_8H_5KO_4$, 99.9 % Pure), Hydrochloric Acid (HCL, 38% Pure), Nitric acid (HNO_3 ,68% Pure), Potassium dichromate ($K_2Cr_2O_7$,99% Pure), Activated carbon (activated charcoal C, Pure), Sulfuric acid (H_2SO_4 ,97% Pure) , Mercuric (II) sulfate ($HgSO_4$,98% Pure), Silver Sulfate (Ag_2SO_4, Pure), Calcium Chloride ($CaCl_2$, 86% Pure).

Preparation of Chemical Oxygen Demand (500 mg\L)

Lightly crush and then dry potassium hydrogen phthalate (KHP) to constant weight at 110 °C. Dissolve 425mg in distilled water and diluted to 1000 ml, this solution has a theoretical COD of 500 mg/ L (Association et al., 1915).

Preparation of alginate beads

Alginate beads prepared by dissolving 2 g of Sodium alginate in 100 ml of distilled water. 2 % alginate solution added drop by drop to a 5% of Calcium Chloride solution. It takes out and washed several times with distilled water to use in the adsorption method.

Preparation of alginate beads entrapped with activated carbon (AG–AC)

After preparing of 2 % of Na-alginate, 1 g of activated carbon added to it. The solution added to 5% calcium solution, then alginate beads with entrapped activated carbon formed, shown in Fig. (1).

Initial COD concentration: About 30 g of wet alginate beads with entrapped activated carbon added to 1000 ml of different concentrations of COD, namely (100,200,300,400,500,600 and 700 mg/L) at contact time 30 min, temp 25 ± 3 °C, and at pH 3± 0.05 and the stirring rate fixed at 100 rpm.

Stirring rate: About 30 g of wet alginate beads with entrapped activated carbon added to 1000 ml of COD (500 mg\L) at contact time 60 min, temp 25 ± 3 °C, at pH 3± 0.05, at a different stirring rate (100, 200, 300,400 and 500) rpm.

Procedure of adsorption experiment

Alginate beads with entrapped activated carbon added to 1000 ml of COD containing different concentrations. Under various conditions which include: effect of (pH, dose, concentration, time and stirring rate) and mixing the adsorbent with solution, filter solution and take a certain amount of filtrate and reagents carefully in test tube, then in the digestion reactor, which set at 150 ^oC for 2 hours to oxidize the organics by the acid, potassium dichromate and catalyst.

COD concentration after adsorption evaluated by measuring the absorbance at a wavelength (600 NM) by a UV/V means is spectrometer (UV-1800). The percentage of COD removal calculated using the following equation:

$$\text{Sorption } [\%] = [C_o - C_e/C_o] \times 100 \qquad (1)$$

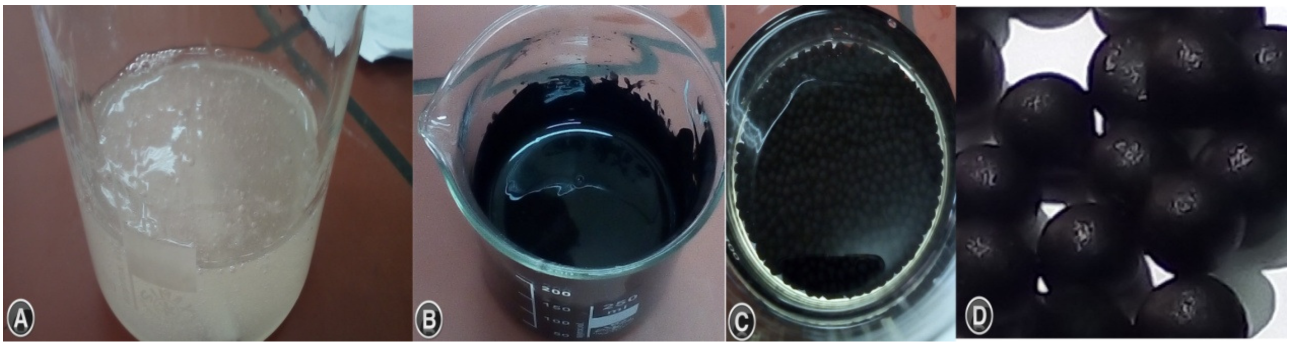

Fig. 1: Preparation of alginate beads with entrapped activated carbon (AG–AC).

Testing Parameter

COD estimated before and after addition of adsorbent.

Effect of different operating parameters

Contact time: About 30 g of wet alginate beads with entrapped activated carbon added to 1000 ml of COD (500 mg\L) at different contact times, namely; (15, 30,45,60,90,120,150 and 180 min), temp 25 ± 3 ºC and at pH 3 ± 0.05 and the stirring rate fixed at 100 rpm.

pH: About 30 g of wet alginate beads with entrapped activated carbon added to 1000 ml of COD 500 (mg\L) at contact time 60 (min), at different pH (3,5,7 and 9), temp 25 ± 3 °C and the stirring rate fixed at 100 rpm.

Adsorbent dose: Different weights of wet alginate beads with entrapped activated carbon were namely; (10, 20, 30, 40, 50 and 60 g) added to 1000 ml of COD 500 (mg\L) at contact time 60 min, temp 25 ± 3 ºC, at pH 3 ± 0.05 and the stirring rate fixed at 100 rpm.

Where C_o is the initial concentration (mg/L) of COD in solution and Ce is the equilibrium concentration (mg/L) of COD in solution. The amount of COD adsorbed by alginate beads with entrapped activated carbon calculated using the following equation:

$$q_e \text{ [mg /g]} = [[C_o - C_e] V] / m \qquad (2)$$

where q_e is the equilibrium adsorption capacity (mg/g), V is the volume of aqueous solution (L) and m is the weight of the adsorbent (g).

Adsorption isotherm

Langmuir and Freundlich isotherm models used to describing the equilibrium sorption data (Freundlich, 1907, Langmuir, 1918). Langmuir assumes monolayer coverage of adsorbate over a homogeneous adsorbent surface.

The linearized form of the Langmuir is given by the equation:

$$C_e / q_e = 1 / (K_L q_{max}) + C_e/q_{max} \qquad (3)$$

Where q_e (mg/g) is the amount of COD adsorbed by the alginate beads with entrapped activated carbon at the equilibrium, C_e (mg/L) is the COD concentration in the equilibrium solution, q_{max} (mg/g) is the maximum monolayer adsorption capacity, and K_L (L/mg) is the Langmuir constant related to binding sites affinity and adsorption energy. The plot of C_e/q_e versus C_e employed to generate the values of q_{max} and K_L.

The Freundlich isotherm is an empirical equation employed to describe heterogeneous adsorption surface and is given by:

$$\text{Ln } q_e = 1/n \ln C_e + \ln K_f \qquad (4)$$

Where K_f ((mg/g) $(mg/L)^{-1/n}$) and n (dimensionless) are Freundlich constant related to the adsorption capacity and adsorption intensity, respectively. (K_f) and (n) evaluated by plotting ln q_e and ln C_e.

Results and Discussion

COD removal using alginate beads with entrapped activated carbon

Effect of contact time

The effect on COD removal studied at different times (15,30,45,60,90,120,150 and 180 min) against 1000 ml of COD standard solution using 30 g from alginate beads with entrapped activated carbon at pH 3 ± 0.05, temp 25 ± 3 ºC, and the stirring rate fixed at 100 rpm. COD concentration is (500 mg\L) and the removal efficiency recorded as (148, 129,118,105, 95, 92, 90 and 89 mg\L) and the removal percentage was (70, 74, 76, 79, 81, 82, 82 and 82%) respectively, shown in Figure (2). As expected as time increase; the value of COD reduction increases because with an increase in time, increase in the contact between the solution to the larger surface area available of adsorbent as there are lots of free active sites for the adsorption (Amin, 2008, Rakholiya and Puranik, 2012). Fig. 2. The effect of contact time on the COD removal using (AG–AC).

Effect of pH

The effect of pH on 1000 ml of COD studied at pH (3, 5, 7 and 9), at contact time 60 min, temp 25 ± 3 ºC, at pH 3 ± 0.05 and the stirring rate fixed at 100 rpm. COD concentration reduced from (500 mg\L) to (105,181,284 and 387 mg\L) and the removal percentages were (79, 64, 43 and 23%) showing the best removal of COD at pH 3, shown in Figure (3). The pH is the most important factor affecting the adsorption

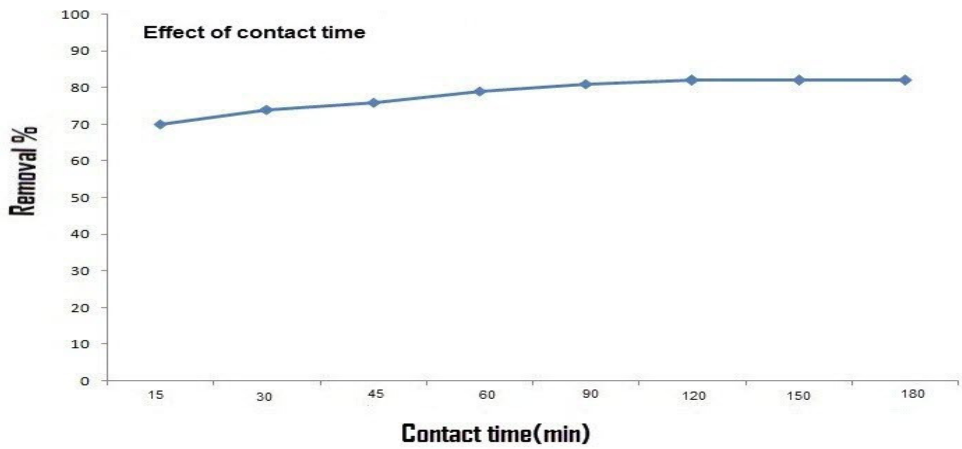

Fig. 2. The effect of contact time on the COD removal using (AG–AC).

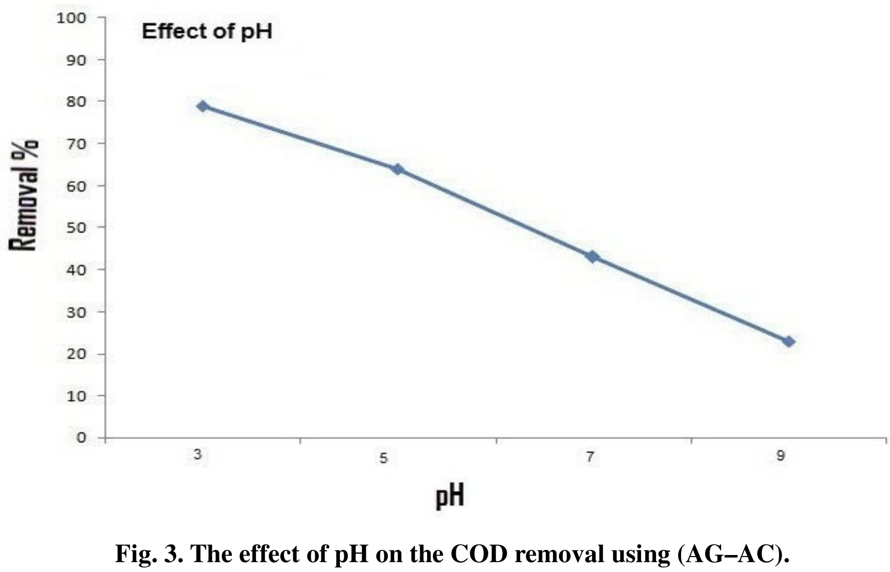

Fig. 3. The effect of pH on the COD removal using (AG–AC).

process to study the pH influence on the adsorption capacity of adsorbent. At lower pH levels, might be attributed to a larger number of (H)+ ions existence lead to increase the adsorption efficiency which in turn neutralizing the negatively charged the surface of adsorbent, thus reducing impediment to the spreading of organics. on another hand, at higher might be pH the adsorption capacity reduced due to the increase of hindrance to organic ions diffusion, because of that abundance of $(OH)^-$ ions (Aluyor and Badmus, 2008, Sun et al., 2013).

Effect of adsorbent dose

The effect on COD removal studied at pH 3 at contact time 60 min, temp 25 ± 3 ºC, at pH 3± 0.05 and the stirring rate fixed at 100 rpm, using different doses of alginate beads with entrapped activated carbon (10, 20,30,40,50 and 60 g) added to 1000 ml of COD (500 mg\L). COD concentration reduced from (500 mg\L) to (203, 162, 105, 99, 55 and 41 mg\L) and the removal percentage was (59, 68,79,80,89 and 92%), shown in Fig. (4). When the amounts of adsorbent increased (adsorbent dose increases), the removal efficiencies for pollutants increase; due to the number of active sites increased because of increased the surface area (Gaikwad and Mane, 2015, Garg et al., 2015, Hirunpraditkoon et al., 2011).

Effect of the initial COD concentration

The effect on the initial COD concentration removal using about 30 (g\L) alginate beads with entrapped activated carbon studied to 1000 ml of solution at contact time 60 min, temp 25 ± 3 ºC, at pH 3 ± 0.05 and the stirring of the solution fixed at 100 rpm. COD concentration reduced from (100, 200 ,300 ,400 ,500 ,600 and 700 mg\L) to (11, 29, 50, 82,105,135 and 164 mg\L) and the removal percentage was (89 ,86 ,83,80 ,79,78 and 77%), shown in Fig. (5). In the beginning of Adsorption process, the removal percentage for pollutants was higher because of the great number of available adsorption active sites of adsorbate molecules but it decreased with time

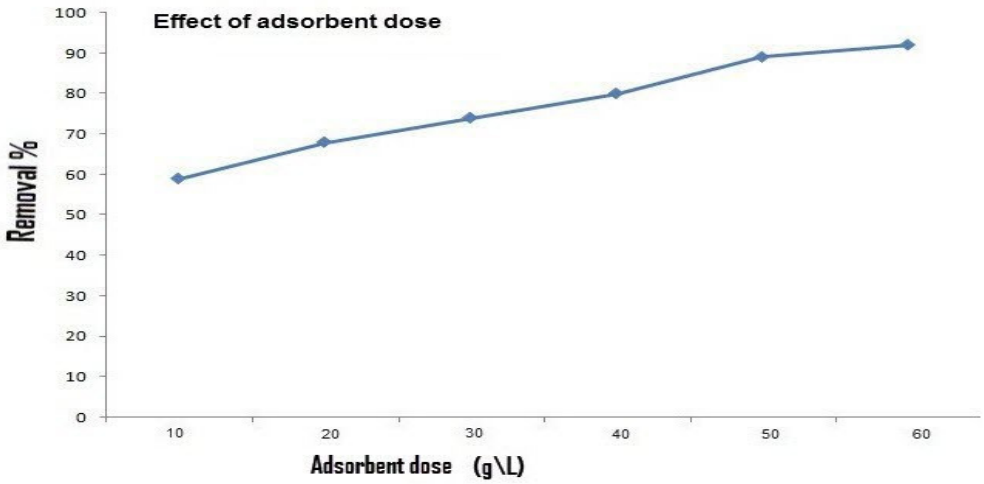

Fig. 4. The effect of adsorbent dose on the COD removal using (AG–AC).

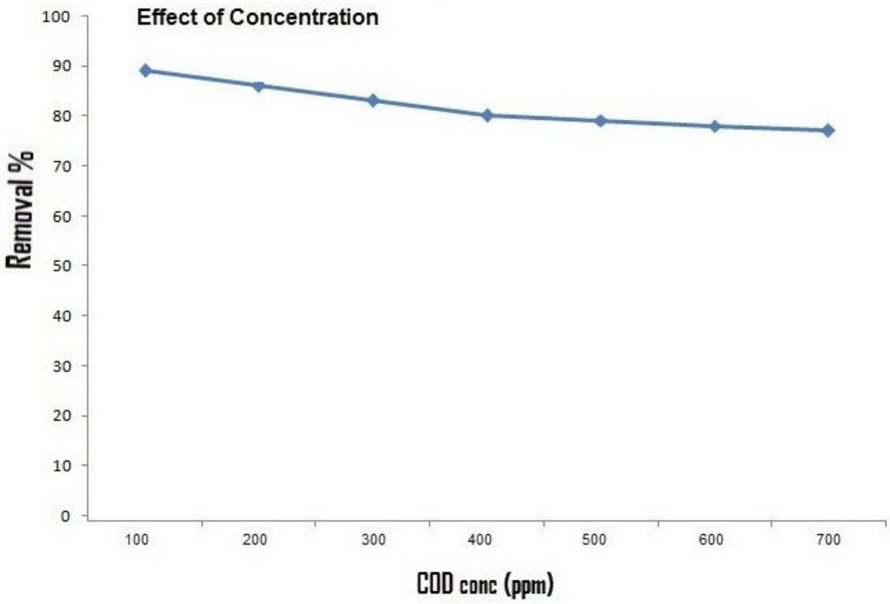

Fig. 5. The effect of the initial concentration on the COD removal using (AG–AC).

gradually due to the saturation and diminished of this sites, also caused a reduction in removal ratio (Devi and Dahiya, 2008).

Adsorption isotherm study for COD removal using alginate beads with entrapped activated carbon:

The sorption capacity of the adsorbent predicted and evaluated by Adsorption isotherm study (Ho and Chiang, 2001). The Freundlich and the Langmuir equations are the most common isotherms applications used for wastewater treatment (Abdel-Gawad et al., 2016, Weber, 1972). The most common used model for measuring the sorption of organic compounds from wastewater is the Freundlich isotherm (Abdel-Gawad et al., 2016, Nkansah et al., 2012). The Freundlich and Langmuir isotherm have acquired vogue because of their ability to fit a variety of sorption data (Kinniburgh, 1986).

Freundlich isotherm model, shown in Fig. (6) with correlation coefficient (R^2) around 0.998, (n) around 1.524, (K_f) around 1.61) showed better fit of adsorption data than Langmuir isotherm model, shown in Figures (7) with (R^2) around 0.932, (q_{max}) around 28.74, (K_L) around (0.0086) The Freundlich model is more appropriate than the Langmuir isotherm model.

Effect of stirring rate

The stirring rate effect on COD removal using alginate beads with entrapped activated carbon at different rpm (100,200,300,400 and 500) studied to 1000 ml of COD 500 (mg\L) at contact time 60 min, temp 25 ± 3 ºC, at pH 3 ± 0.05. The COD concentration reduced to (105, 91, 85, 83 and 85 mg\L) and the removal percentages were (79, 82, 83, 83 and 83%), shown in Figure (8). It is one of the important factors can significantly affect adsorption capacity and plays an important role in the removal efficiencies of pollutants. Increase in pollutant percentage removal was due to increase in stirring rate, enhanced the pollutants diffusion to the surface of the adsorbent (Bernard et al., 2013).

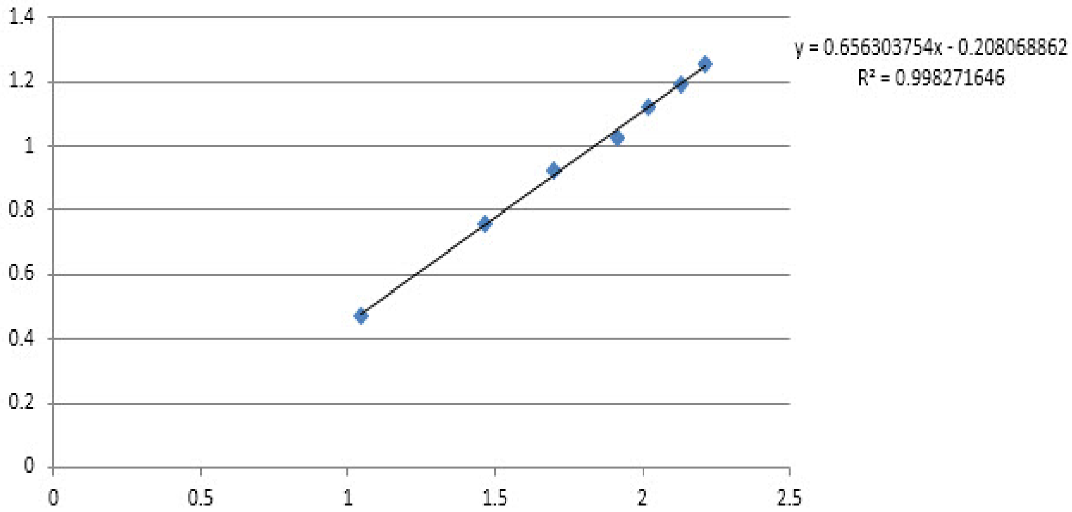

Fig. 6: Freundlich isotherm of COD removal using (AG–AC).

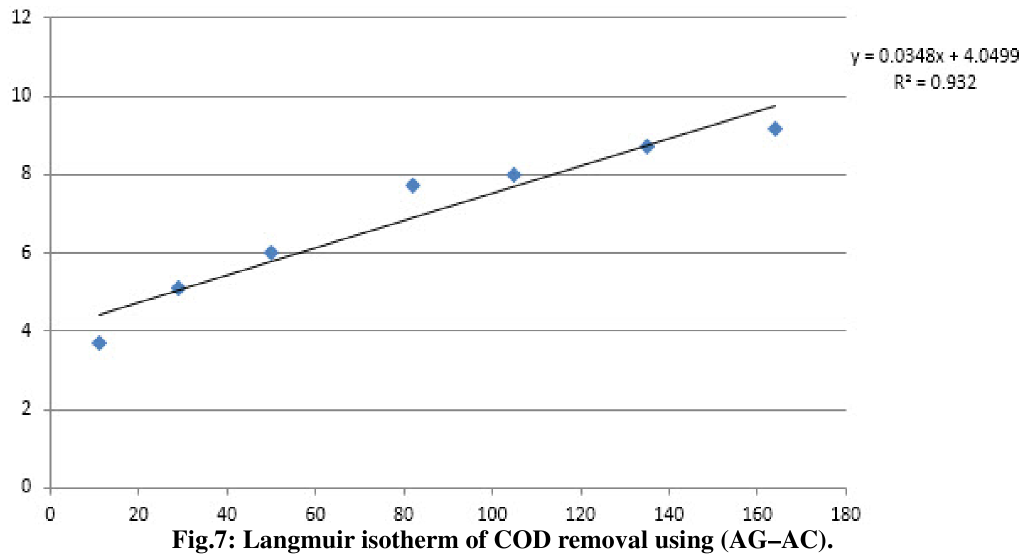

Fig.7: Langmuir isotherm of COD removal using (AG–AC).

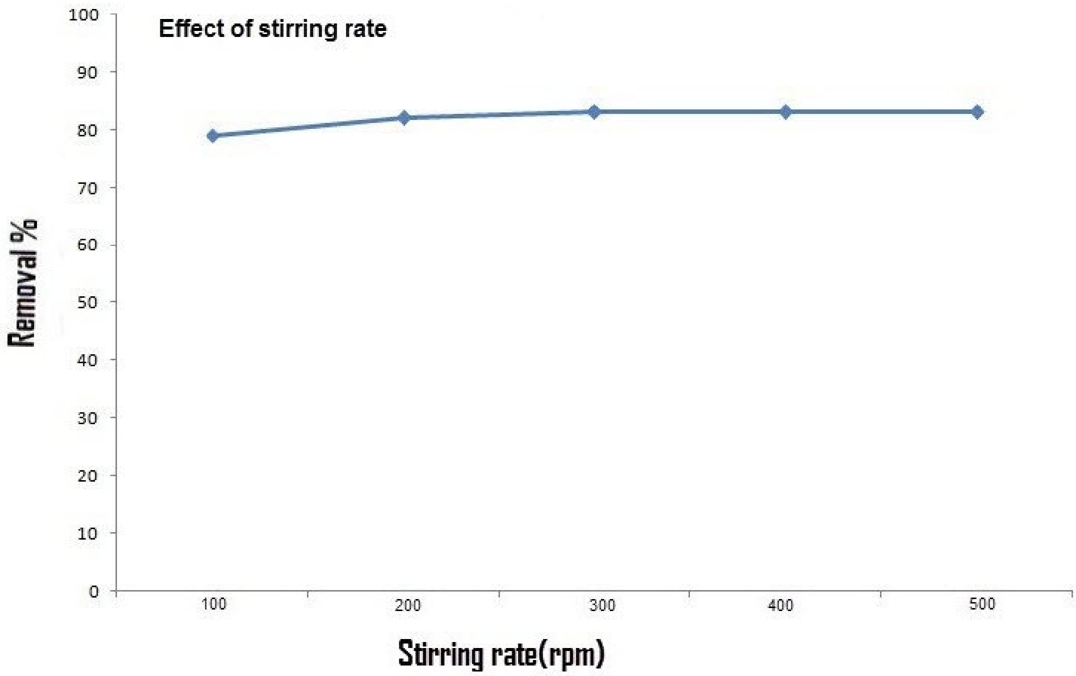

Fig. 8: The effect of stirring rate on the COD removal using (AG–AC).

Ascorbic acid removal using alginate beads with entrapped activated carbon:

The effect of alginate beads with entrapped activated carbon on ascorbic acid studied at contact time 60 min, temp 25 ± 3 ºC, at pH 3 ± 0.05 and the stirring of the solution fixed at 100 rpm, using about 30 (g\L) from adsorbent added to 1000 ml of ascorbic acid solution which its concentration is (500 mg\L). Ascorbic acid concentration reduced to (151 mg\L) and the removal percentage was (70%), shown in Figure (9).

Lactose removal using alginate beads with entrapped activated carbon:

The effect of alginate beads with entrapped activated carbon on lactose studied at contact time 60 min, temp 25 ± 3 ºC, at pH 3 ± 0.05 and the stirring of the solution fixed at 100 rpm, using about 30 (g\L) from adsorbent added to 1000 ml of lactose solution which its concentration is 500 (mg\L). Lactose concentration reduced to (269 mg\L) and the removal percentage was (46%), shown in Figure (9).

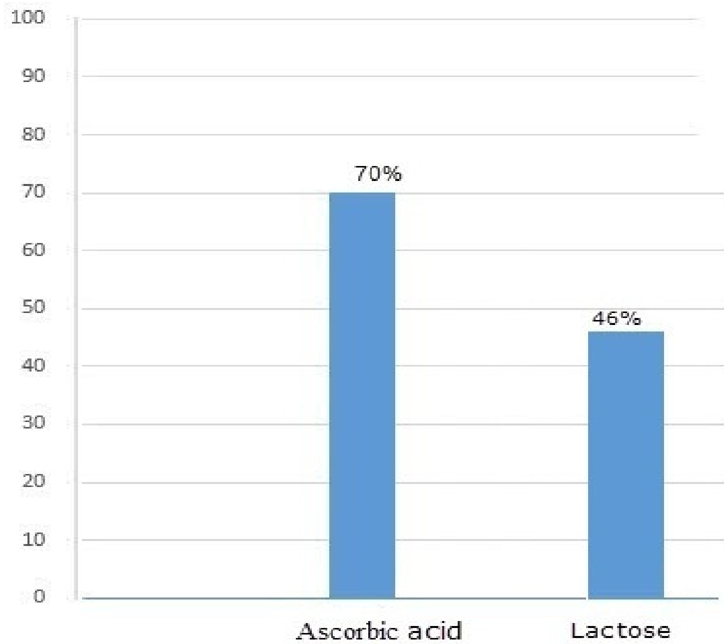

Fig. 9: Ascorbic acid & Lactose removal % using (AG–AC).

CONCLUSIONS

In this study, the results showed that alginate beads with entrapped activated carbon is capable of COD removal from aqueous solution. Various operating parameters on COD removal efficiency investigated and optimized. COD removal affected by the experimental parameters such as contact time, dosage, pH, temperature, stirring rate, initial COD concentration. The alginate beads with entrapped activated carbon be a cost-effective alternative and can lead to success in wastewater treatment and produce high-quality treated effluent.

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