Improving Adsorption Process onto Activated Carbon by Applying Local Low-Cost Waste Materials for Decolourisation of Wastewater

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Abstract

Adsorption is as an alternative technology to remove colour from wastewater.

In this study, the capable of improving the run time through increasing the effectiveness of adsorption of activated carbon was investigated and assessed with more economical for dye removal from wastewater by using of waste and ecofriendly material as weight ratio to the granular activated carbon fixed bed using continues system.

Two types of experiments were carried out, batch and continuous flow(fixed bed column system) experiments. In batch study, the adsorption data were modeled with Langmuir and Freundlich isotherms, and the statistical analysis was done by using SPSS(V.15) program. The results showed that the Langmuir isotherm model was fitted the adsorption equilibrium data well compared with Freundlich model with favorable type and maximum monolayer adsorption capacity of(25mg/g) and(R^2=0.993).

Column experiments carried out with using of glass beads as different weight ratios to the commercial activated carbon (0%, 5%, 10%, and 15%) at similar operation conditions.

It was found that adding 5% glass beads weight ratio to the activated carbon bed increases operating time of activated carbon-column by 23.5%, while adding 10% and 15% glass beads weight ratio causes the operating time to decrease by about 38.5% and 61.5% respectively and therefore makes the adsorption process not efficient compared with pure(0% ratio) carbon bed.

Avery high degree of correlation of the experimental adsorption rate data was provided by this model suggesting that this model could be used in design applications.

الخلاصة

يعتبر الامتزاز التكنولوجيا البديلة لإزالة اللون من مياه الفضلات.

في هذه الدراسة تم التحري وتقييم إمكانية تطوير(زيادة) زمن التشغيل من خلال زيادة كفاءة الامتزاز لعمود حشوة الكربون المنشط وبأكثر اقتصادية لإزالة صبغة المثيلين الزرقاء من مياه الفضلات باستخدام مواد مخلفة كنسب وزنية لطبقة الكاربون المنشط عند تطبيق تجارب النمط المستمر.

تم اجراء نوعين من التجارب تضمنت تجارب دفعية(batch) وتجارب النمط المستمر(continuous system).

استخدم البرنامج الإحصائي(SPSS V.15) لتحليل النتائج التجريبية، وأظهرت النتائج للتجارب الدفعية إن علاقة التوازن للمادة المازة هي من النوع المفضل كما إن علاقة التوازن تتطابق جيدا مع نموذجي لانكمير وفراندلش ولكن بتثبيت أقوى للبيانات مع لانكمير (R^2=0.993) وبقابلية امتزاز قصوى مقدارها(25mg/g).

أجريت تجارب النمط المستمر باستخدام حبيبات الزجاج وبنسب وزنية نسبة إلى طبقة الكربون المنشط(0%, 5%, 10%, 15%) وتحت نفس ظروف التشغيل.

وجد ان اضافة نسبة وزنية(5%) من مادة حبيبات الزجاج الى عمود الحشو الثابت للكربون المنشط يؤدي الى زيادة زمن تشغيل عمود الامتزاز بنسبة(23.5%)، أما إضافة(10%, 15%) من حبيبات الزجاج كنسبة وزنية الى عمود الحشو يؤدي الى تقليل زمن التشغيل بنسبة(38.5%, 61.5%) على التوالي، وهذا يجعل عملية الامتزاز غير كفوءة بالمقارنة مع استخدام الكربون المنشط التجاري بمفرده(0%).

تترابط البيانات التجريبية المستحصلة من هذه الدراسة بدرجة عالية جدا مما يشير إلى إمكانية استخدام هذا الموديل في التطبيقات التصميمية.

1.Introduction

Dyes production industries and many others industries which used dyes and pigments generated wastewater, characteristically high in color and organic content(Garg et al.,2004).

Dyes are widely used in paper and pulp, textile and dyeing, leather, printing industries and food process industries. Among these various industries textile ranks first in usage of dyes for coloratic of fiber, so that, the discharge of colored wastewater is posing a serious environmental concern due to their poor biodegradability, carcinogenicity and toxicity
(Lee et al.,1999; Hao et al.,2000; Papic et al.,2000;Pala and Tokat,2002).

Different process for color removal typically include physical, chemical and biological schemes.

Some process, such as electrochemical techniques and ion pair extraction, are relatively new for textile waste treatment, while others are been used in the industry for along time.

Adsorption has been found to be superior to other techniques for removal dyes from the waste effluent.

The process of adsorption has an edge ever the other methods due to its sludge free clean operation and completely removal dyes, ever from the diluted solution(Ozacar and Sengil,2003;Shaobin et al.,2005).

Activated carbons are excellent adsorbents and thus are used to purify, decolorize, detoxicate, filter or remove dissolved substances(Cheremisinoff and Ellerbusch,1978; Rengaraj et al.,1999; Juang et al.,2002).

A number of studies have also been performed using activated carbon prepared from agricultural waste for the removal of dyes from aqueous solution.

The waste materials include coconut tree flower and jute fiber(Senthilkumaar et al.,2006), oil palm fiber(Tan et al.,2007), palm kernel shell(Jumasiah et al.,2005), corncob(Preethi et al.,2006), and bagasse pith(Amin,2008).

Basic dyes are the brightest class of water soluble dyes used by the textile industries, and methylene blue is one of the most frequently used dyes in all industries(Reid,1996).

The aim of this study is to investigate the potential of improving the fixed bed adsorber of continuous flow system to make it more efficient and/ or economical by increasing the adsorption efficiency with increasing the run-time of column through reduction the losses of adsorption process through the micro pores of fixed bed activated carbon particles by adding filler material like glass deads.

The experiments carries out batch and fixed bed column. Batch studies were conducted to estimate the adsorption capacity of the adsorbent.

In this work, Langmuir and Freundlich models were used to evaluate the adsorption capacity of activated carbon.

Four experiments in a fixed-bed column system with different weight ratio of glass-activated carbon were done to investigate the main
objective of this work.

2. Experiments

2.1 Materials and Methods

2.1.1 Adsorbent: The commercial activated carbon was supplied by(unicarbo, Italian, Lmt.Com.) to Iraqi local markets was used in this study.

The characteristics of commercial activated carbon were listed in Table(1).

Table (1): **Characteristics of the Activated Carbon**

Base	Coconut shell
Bulk density	$0.3 * 10^3$ Kg/m^3
Particle density	$1.5 * 10^3$ Kg/m^3
BET surface area	$650 * 10^3$ Kg/m^3
Particle porosity	0.4
Bed porosity	0.2
Ash content (%)	5 (max)
Iodine No (mg/g)	1100 – 1130
pH	10.2 -10.6
Moisture content(%)	5

Activated carbon was washed with distilled water, dried in oven at 120C$^{\circ}$ for 24h(Yamin et al.,2007).

This time was usually enough to remove any undesired moisture within the particles.

Finally, activated carbon was sieved to desired particles size(500 µm) and stored in desiccators for cooling.

The glass beads are a waste product, easily available, and economically advantageous.

The desired size of glass beads was obtained by crushing and sieved to get the particle size of(100 µm) to be also used in present study.

The same procedure of washing and drying of granular activated carbon was applied for the glass beads particles.

2.1.2 Adsorbate: Methylene blue dye(MB, chemical formula: $C_{16}H_{18}CLN_3S$) and a molecular weight of $373.9*10^{-3}$kg mol^{-1}(Hameed et al.,2006).

This basic cationic dye was supplied by Iraqi markets. Its molecular structure is shown in Figure(1).

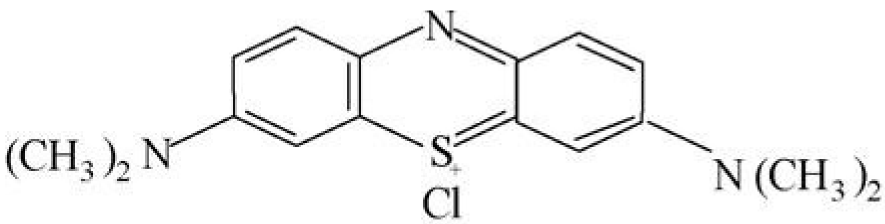

Fig.(1): Molecular Structure of Methylene Blue(Mohammad et al.,2009).

The methylene blue dye solution was prepared with concentration of 0.03kg/m^3.

The PH solution was adjusted with 0.1M HCL using PH meter to its effective adsorption PH value.

Absorbance measurements were made with using(UV-VIS-1650 PC SHIMATZU) spectrophotometer at corresponding wavelength for maximum absorbance(λ_{max}=664nm). Standard solution of the dye was taken and the absorbance was find at different wave lengths to get the linear calibration curve used in the determination of equilibrium MB concentrations.

2.2 Adsorption Studies

The adsorption of MB onto activated carbon was studied using two types of experiments, **batch and column experiments**.

2.2.1 Batch Experiments was carried out to study the adsorption isotherms of MB.

The experiments were adjusted at the initial PH of 5 for MB adsorption(Yamin et al.,2007; AL-Taliby,2009).

This experiments were carried out by adding granular activated carbon ranging in weight(0.1, 0.25, 0.5, 1.0, 1.5, 2.0)gm which are placed in individual six flasks of(100 ml) volume.

The same volume of dye solution with concentration of 0.03kg/m^3 is added to each of the flasks and the mixture was agitated by Wrist shaker for 24h hours at 25C◦. Along with these flasks, one blank was also run. After this period of shaking, samples were filtered using filter paper, in order to minimize the interference of the carbon fines with the analysis, and the filtrates were analyzed for residual concentrations of MB using a double beam UV-visible spectrophotometer at λ_{max}=664nm.

The adsorption isotherms of MB onto glass beads of size(100 µm) with the same conditions were investigated in the same procedure used for adsorption isotherms of MB onto activated carbon.

The amount of adsorption at equilibrium, q_e(mg/g), was calculated by the following equation(Ho et al.,1996; Rahmani et al.,2004).

$$q_e = \frac{(C_o - C_e)V}{m} \quad \text{...(1)}$$

Where:

q_e (mg/g) : is the amount of MB adsorbed in adsorbent at equilibrium.

C_o and C_e (mg/l): are the initial and equilibrium concentration of MB solutions respectively.

m (g) : is the amount of adsorbent .

V (l) : is the volume of MB solution.

2.2.1.1 Adsorption Isotherms

In order to determine the adsorption of the adsorbents(activated carbon) for removal of MB, study of adsorption isotherm was carried out and tested against the Langmuir and Freundlich isotherm models(Freundlich,1906; Langmuir,1918; Aksu and Yener,2001;; Ho et al.,2002; Rengaraj et al.,2002; Mahvi et al.,2004; Somasundaran,2006).

Langmuir isotherm is based on the assumption of uniform energy of adsorption on the surface of the adsorbent. The total monolayer capacity of the adsorbent is equal to **Q**, a Langmuir constant.

$$q_e = \frac{QbC_e}{1 + bC_e} \quad \text{……………………………………………..(2)}$$

The rearranged Langmuir isotherm is represented by following equation:

$$C_e/q_e = 1/Q.b + C_e/Q$$

Where, C_e is the equilibrium concentration of dye (mg/l), q_e is the amount of dye adsorbed at equilibrium(mg/g), Q(mg/g) and b(l/mg) are the Langmuir constants related to maximum adsorption capacity and energy of adsorption respectively. Hence, a plot of C_e/q_e versus C_e yield a straight line with **Q** calculated from the slope and the value of **b** as its intercept.

The essential characteristics of the Langmuir isotherm can be terms of a dimensionless equilibrium(**R_L**) which can be defined by(Hall et al.,1966; Weber and Chakravorti,1974; Gupta et al.,1990).

$$R_L = \frac{1}{1+bC_o} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{(4)}$$

where R_L is the a dimensionless separation factor, $C_\circ$ is initial dye concentration(mg/l), and b is the Langmuir constant(l/mg). The parameter R_L indicates the shape of the isotherm accordingly:

Values of R_L	Type of Isotherm
$R_L > 1$	Unfavorable
$R_L = 1$	Linear
$0 < R_L < 1$	Favorable
$R_L = 0$	Irreversible

Freundlich isotherm is an exponential equation can be written as:

$$q_e = K.C_e^{\frac{1}{n}} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{(5)}$$

or its linear form

$$\log q_e = \log K + \frac{1}{n}\log C_e \qquad \ldots\ldots\ldots\ldots\ldots\ldots\text{(6)}$$

Where, K is the constant indicative of the relative adsorption capacity of the adsorbent(mg/g), and $\frac{1}{n}$ is the constant indicative of the intensity of the adsorption.

The Freundlich equation possesses two constant, K and $\frac{1}{n}$.

High and low values of K and $\frac{1}{n}$ indicate high adsorption throughout the concentration range studied whereas high values of $\frac{1}{n}$ and low values of K show low adsorption. When $\frac{1}{n}$ =1, the adsorption is favorable.

The values of K and $\frac{1}{n}$ can be obtain from the intercept and slope, respectively, of the linear plot of experimental data of $\log q_e$ versus log C_e as shown in Figure (3).

Fixed Bed Column Experiments 2.2.2

2.2.2.1 Experimental Arrangements

The schematic representation of experimental equipments is shown in Figure(2).

2.2.2.1.1 Adsorption Column

Continuous flow adsorption studies were conducted in a vertical glass column made of Pyrex glass tube(0.8m) height, and(0.05m) internal diameter.

The activated carbon bed was confined in the column by the means of the glass spheres placed on the top and bottom of bed to ensure a uniform distribution of influent MB solution through the carbon bed.

The cylindrical tanks were used as container, the first one as a storage tank($100*10^{-3}m^3$); the second one as a feed tank($50*10^{-3}m^3$); each tank was fitted with gate valves.

The bed column was 0.05m height of granular activated carbon with flow rate of $1.70*10^{-6}m^3$/sec, MB concentration=$0.03kg/m^3$, PH=5 for all experiments.

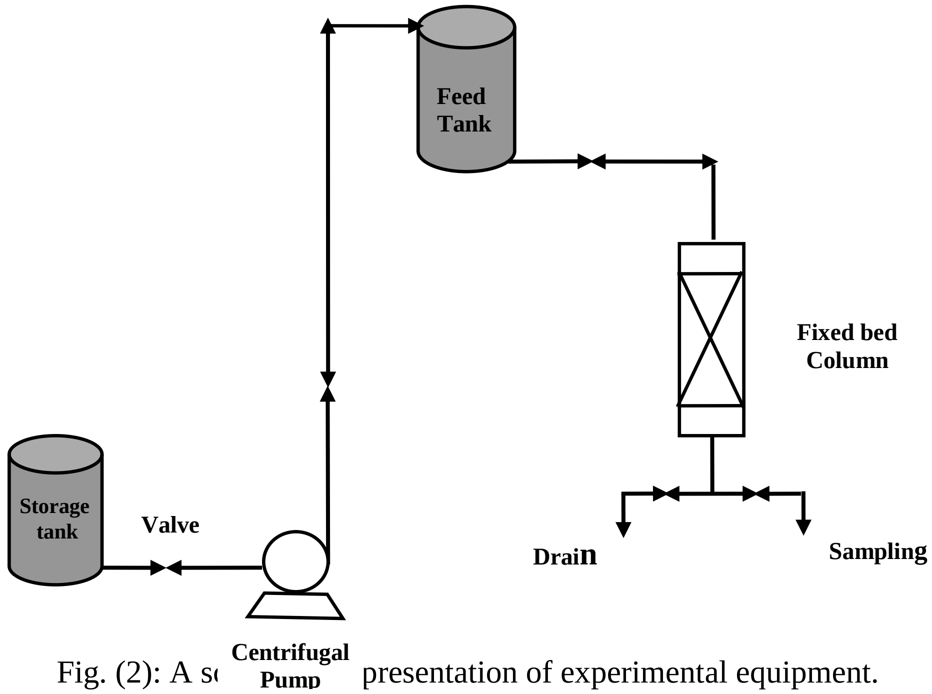

Fig. (2): A s presentation of experimental equipment.

2.2.2.1.2 Preparation of Samples with Different Glass Beads - Activated Carbon Ratios.

Different glass beads- commercial activated carbon weight ratios were used starting from 0%, 5%, 10%, 15% of glass beads. The glass beads with size of 100µm were mixed with activated carbon particles with size of 500 µm. Each sample was mixed by a shaker for 1 hour.

2.2.2.1.3 Experimental Procedure

After preparing the MB solution with desired concentration, the solution was pumped by means of centrifugal pump(KF-2), (SAER, ITALA) with capacity of $0.05m^3$/h from the storage tank to the feed tank and then to the top of the column by gravity flow.

The flow rate was adjusted to the design value by the volumetric method.

At first, every 15 minutes sample was taken from the outlet of the column, and after MB appeared in the sample, the time interval of sampling increases until equilibrium state was reached.

To achieve the measurement of the breakthrough curves for MB four experiments were done, once with pure activated carbon, and other three with different glass beads-activated carbon weight ratio(5%, 10%, 15%).

All conditions of experiments are summarized in Table(2).

Table (2) Column system-data

Experimental data									Data of results
Experimental No.	**Adsorbent**	**Adsorbate**	**Adsorbate concentration (kg/m^3)**	**Bed depth (m)**	**Flow rate ($\overline{Q}$) (m^3/sec)**	**Particle Size of Activated carbon (μm)**	**Particle Size of Glass Beads (μm)**	**Glass Beads – activated carbon ratio (%)**	**Saturation time (min.)**
1	Activated carbon	Methylene Blue	0.03	0.05	$1.70*10^{-6}$	500	100	0	1560
2	=	=	=	=	=	=	=	5	2040
3	=	=	=	=	=	=	=	10	960
4	=	=	=	=	=	=	=	15	600

3. Results and Discussion

3.1 Batch Experiments

The Langmuir, Freundlich, and the equilibrium adsorption isotherms of MB adsorption onto commercial activated carbon are shown in Figures(3),(4), and(5) respectively.

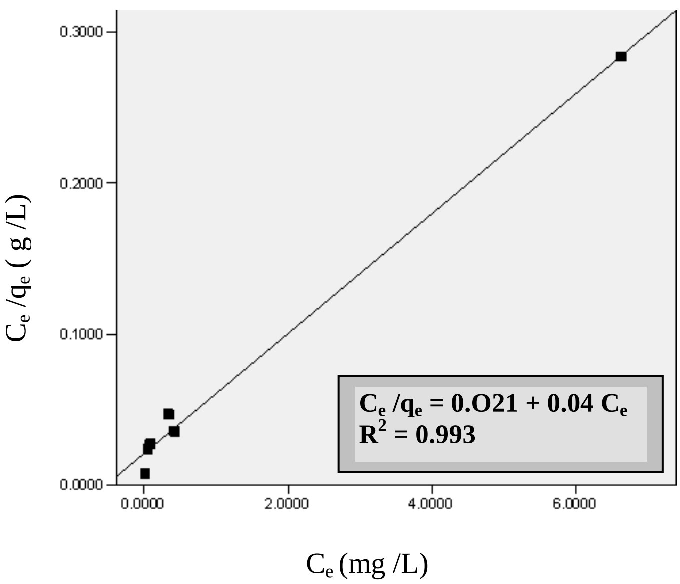

Fig.(3): Langmuir Adsorption Isotherm of Methylene Blue onto Commercial Activated Carbon at 25 ± 1 C^o, C_o = 0.03 $kg/m^3$and pH = 5 .

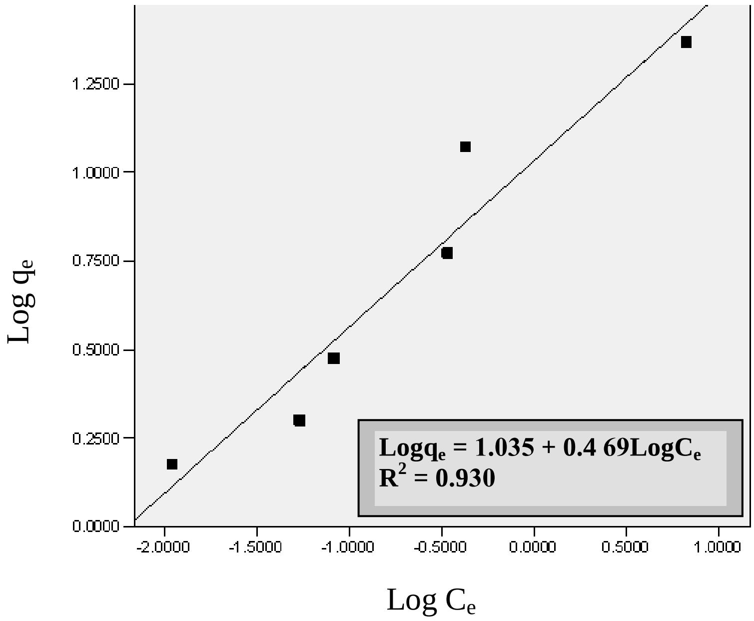

Fig.(4): Freundlich Adsorption Isotherm of Methylene Blue onto Commercial Activated Carbon at 25 ± 1 C^o ,C_o = 0.03 kg/$m^3$and pH = 5.

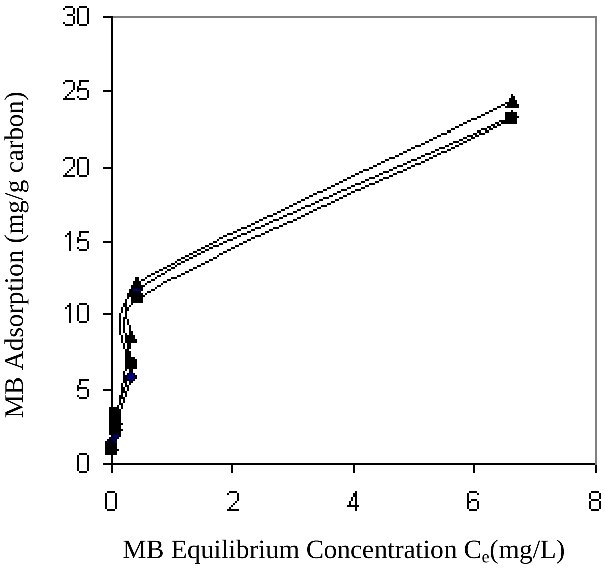

Fig.(5) : Equilibrium Adsorption Isotherm of Methylene Blue onto Commercial Activated Carbon at 25 ± 1 C^o ,C_o = 0.03 kg/m3, pH = 5 .

The obtained experimental data was correlated with Langmuir and Freundlich models. The parameters for each model were obtained by statistical regression using (SPSS V.15) program and they are summarized in Table(3).

The correlation coefficients(R^2) were significant at the 0.05 probability level and it was higher for Langmuir than Freundlich. This indicates that the Langmuir isotherm is clearly the better fitting isotherm to the experimental data. This attributed to the homogeneous nature of the surface of the activated carbon used.

The results also demonstrate the formation of monolayer coverage of dye molecule at the outer surface of adsorbent. Similar observation was reported by the adsorption of acid orange 10 dye onto activated carbon prepared from agricultural waste bagasse(Tsai et al.,2001), and by the adsorption of direct dyes on activated carbon prepared from sawdust (Malik,2004).

The equilibrium isotherm for activated carbon used in this study is favorable type, for being convex upward.

The value of(**R_L**) also indicated that the Langmuir isotherm was favorable for adsorption of MB onto the activated carbon(**R_L**<1).

Similar results were observed by(Weber and Chakravorti,1974; Han et al.,2005; Tan et al.,2007).

The equilibrium data for methylene blue adsorption on activated carbon well fitted to the Langmuir equation with maximum monolayer adsorption capacity of(25mg/g).

Table (3): Langmuir and Freundlich parameters of isotherm for activated carbon used.

Adsorbent	Langmuir isotherm (95 % confidence level)						Freundlich isotherm (95 % confidence level)				
	Q (mg /g)	b (L /mg)	Correlation coefficient (R)	Determination coefficient (R^2)	Standard error of the estimate (SE)	Separation Factor (R_L)	Intercept (K) (mg/g)(L/ mg)$^{1/n}$	Slope (1/n)	Correlation coefficient (R)	Determination coefficient (R^2)	Standard error of the estimate (SE)
Granular activated carbon	25	1.90	0.997	0.993	0.010	0.0172	10.8392	0.469	0.964	0.930	0.137

The result of adsorption isotherm of MB onto glass beads shown that the concentration of MB remains the same for different weight of glass beads. This proves that the beads are in inert material and there is no adsorption occurs onto it.

3.2 Column Experiments

The results of MB adsorption onto commercial activated carbon fixed bed using continuous flow were presented in the form of breakthrough curves which showed the loading behaviors of MB to be adsorbed from the solution expressed in terms of reductive concentration defined as the ratio of the outlet MB concentration to the inlet MB concentration as a function of time($C/C_\circ$ V_S. time).

To study the effect of different glass beads-commercial activated carbon, weight ratios were investigated for MB adsorption onto commercial activated carbon by adding different weight ratios of(100µm particle size)glass beads to the activated carbon bed(500µm particle size).

Four experiments were conducted using different weight ratios of(0%, 5%, 10%, and 15%). The experimental breakthrough curves are presented in Figure(6), and the data of results tabulated in Table(4).

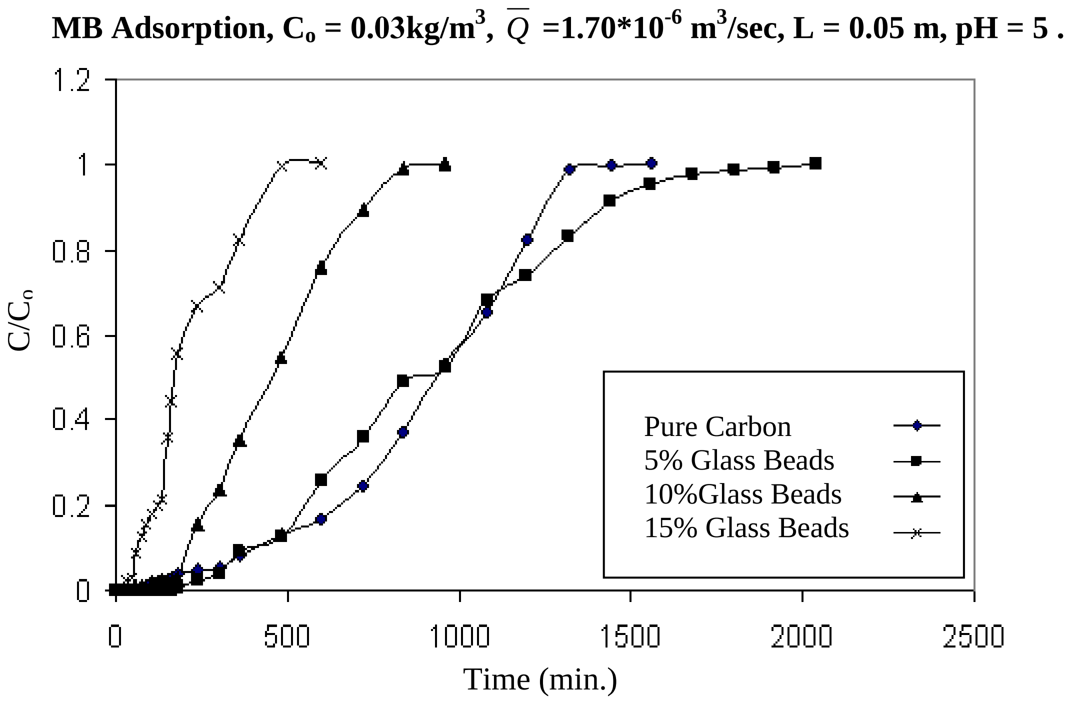

Fig.(6): Experimental Breakthrough Curves for MB Adsorption onto Commercial Activated Carbon at Different Glass Beads Ratios.

All experiments were conducted at constant conditions, bed depth(0.05m), initial MB concentration(0.03kg/m^3), flow rate(1.70*10^{-6}m^3/sec), particle size of granular activated carbon(500µm), particle size of glass beads(100 µm) and solution PH of 5 .

Figure(6) shows that a significant increase in the operating time by (23.5%) achieved by adding 5% glass beads weight ratio to the activated carbon bed.

Increasing the glass beads ratio to 10%, 15% caused the operating time to decrease by about 38.5%, and 61.5% respectively, and these additions were achieving lower operating time and removal efficiency than pure(0% ratio) activated carbon bed.

Knowing that the matching between the two mechanisms; static hold-up and operating hold-up will save weight of activated carbon and minimize the losses in surface area for adsorption. Then, 5% ratio increases the adsorption efficiency of the activated carbon through the reduction of dead zones between particles in fixed bed, and reduction of losses of adsorption-process through the micro pores.

Table(4): The Experimental Breakthrough Data for Adsorption of MB onto Commercial Activated Carbon at Different Glass Beads- Activated Carbon Ratios, $C_◦$=0.03 Kg/m^3,Q=1.70*10^{-6}m^3/sec, Bed Depth=0.05 m, PH= 5 .

0% Glass beads		5% Glass beads		10% Glass beads		15% Glass beads	
Time (min.)	C/C◦	Time (min.)	C/C◦	Time (min.)	C/C◦	Time (min.)	C/C◦
0	0	0	0	0	0	0	0
15	0	15	0	15	0	15	0
30	0	30	0	30	0	30	0.0244
45	0	45	0	45	0	45	0.0282
60	0	60	0	60	0.00887	60	0.0885
75	0	75	0	75	0.0091	75	0.1254
90	0.0018	90	0	90	0.0099	90	0.1525
105	0.0192	105	0	105	0.0122	105	0.17952
120	0.0210	120	0	120	0.0164	120	0.1992
135	0.0221	135	0	135	0.0177	135	0.2115
150	0.0233	150	0	150	0.0182	150	0.3584
165	0.0314	165	0	165	0.0199	165	0.4452
180	0.0390	180	0.00277	180	0.0282	180	0.5564
240	0.0466	240	0.0255	240	0.1528	240	0.66584
300	0.0533	300	0.0410	300	0.2369	300	0.7125
360	0.0823	360	0.0911	360	0.3541	360	0.8214
480	0.1303	480	0.1256	480	0.54821	480	0.9951
600	0.1705	600	0.2564	600	0.75894	600	1
720	0.2451	720	0.3598	720	0.8952		
840	0.3747	840	0.4874	840	0.9901		
960	0.5329	960	0.5236	960	1		
1080	0.6512	1080	0.6811				
1200	0.8212	1200	0.7421				
1320	0.9854	1320	0.8331				
1440	0.9953	1440	0.9154				
1560	1	1560	0.9554				
		1680	0.9771				
		1800	0.9878				
		1920	0.9908				
		2040	1				

4. Conclusions

On the basis of the results obtained, it can be safely concluded:

♦ The ability of commercial granular activated carbon to adsorp column system.

By this study, it can be shown that the potentialities of activated carbon in colored wastewater treatment and adsorption process can be an alternative treatment methods.

♦ In batch experiments, the equilibrium isotherms for methylene blue dye onto the granular activated carbon are of favorable type.

The adsorption data was analyzed by Langmuir and Freundlich adsorption isotherms; indicating the appropriateness of the experiments.

So that, the Langmuir model gives the best fit for experimental data with higher determination coefficient and the equilibrium-parameter, $\mathbf{R_L}$ was which confirmed that the Langmuir model is favorable.

♦ In continuous flow experiments, a glass beads cheap, easily available material and disposed as waste was used to develop the work of activated carbon fixed bed adsorber column.

Adding 5%(100μm particle size) of glass beads as a weight ratio to the activated carbon(500μm particle size) increases the operating time of column by 23.5% compared to 0% ratio(activated carbon only).

Increasing the glass beads ratio to 10% and 15% decrease the operating time by about 38.5% and 61.5% respectively, and therefore makes the adsorption process not efficient compared with pure carbon bed(0% ratio).

♦ The model parameters would be useful for fabrication for the removal of methylene blue from colored wastewater.

5. Recommendations

Based on the results achieved in this study, the following recommendations can be forwarded:

♦ Using cheap, low cost materials instead of glass beads such as sand and sawdust.

♦ It is recommended to conduct further works with study the effect of using different glass beads-granular activated carbon in continuous system to remove other pollutants such as heavy metals and pesticides from either domesticor or industrial wastewater.

♦ It is recommended to remove organic matter by using biological treatment before using of glass beads-activated carbon columns.

♦ This study may be extended for using another type of adsorbent than activated carbon.

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