IJCPE
Iraqi Journal of Chemical and Petroleum Engineering

Iraqi Journal of Chemical and Petroleum Engineering
Vol.9 No.3 (September 2007) 17-24
ISSN: 1997-4884

University of Baghdad
College of Engineering

Separation Benzene and Toluene from BTX using Zeolite 13X

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Abstract

This work deals with the separation of benzene and toluene from a BTX fraction. The separation was carried out using adsorption by molecular sieve zeolite 13X in a fixed bed. The concentration of benzene and toluene in the influent streams was measured using gas chromatography. The effect of flow rate in the range 0.77 – 2.0 cm3/min on the benzene and toluene extraction from BTX fraction was studied. The flow rate increasing decreases the breakthrough and saturation times. The effect of bed height in the range 31.6 – 63.3 cm on benzene and toluene adsorption from BTX fraction was studied. The increase of bed height increasing increases the break point values. The effect of the concentration of benzene in the range 0.0559 – 0.2625g/cm3 and toluene in the range 0.144 – 0.21 g/cm3 was studied. The increasing of inlet solute concentration increases the slope of the breakthrough curve. The amount of toluene adsorbed in the packed bed at any time is higher than that of benzene while it decreases after the saturation time. The best operating conditions in this work for benzene and toluene adsorption are 0.77 cm3/min of feed and 31.6 cm bed height of zeolite 13X.

Introduction

Generally in the oil industries aromatics are recovered by liquid – liquid extraction of reformate using selective polar solvents like dimethylsulfoxide (DMSO) [1], N – formylmopholine (NFM) [2], and sulfolane [3]. The aromatics and solvent are separated by distillation and then xylenes are separated from the aromatics mixture by extractive distillation. Adsorption is the fixation of molecules by reversible reaction on the surface of a solid. The adsorption of compound on zeolite is the sum of three different phenomena; these are chemisorption, forming the first layer at low partial pressures, physisorption, due to the formation of multiple layers by hydrogen bonding in the alumina pores and capillary condensation, where localized condensation takes place at temperature above that of the bulk fluid's dew point [4]. Contrary to other adsorbents like activated alumina and silica; zeolites have a high adsorption capacity at low partial pressure. Adsorption capacity decreases with increasing temperatures, but zeolite keep their efficiency for drying up to 100 ^{o}C, whereas alumina has it is more favorable adsorption characteristics below 50 ^{o}C [5].

The present study is a trial to separate benzene and toluene from BTX fraction supplied from Arab company for detergent and chemicals by adsorption technique using molecular sieve zeolite 13X.

Experimental Work

This investigation includes the study of effect of feed flow rate in the range 0.77 – 2.0 cm3/min, bed length in the range 31.6 – 63.3 cm, concentration of benzene in the range 0.0559 – 0.2625 g/cm3 and concentration of toluene in the range 0.144 – 0.21 g/cm3 on the benzene and toluene separation from BTX fraction by using zeolite 13X in the packed bed column.

Materials

Feed Stock

The feed stock used in this study is BTX fraction supplied from Arab Company for Detergent and Chemicals which is extracted from the reformate of Beiji Refinery using sulfolane extraction process. The properties of BTX fraction are given in Table (1).

Table (1) Properties of BTX Fraction

No.	Properties	Values
1	API Gravity	30.62
2	Specific gravity at 60 oF/60 oF	0.8728
3	Aromatic content, wt % Benzene Toluene C8 – Aromatics C9 – 10 Aromatics	 6.90 22.25 40.52 30.32

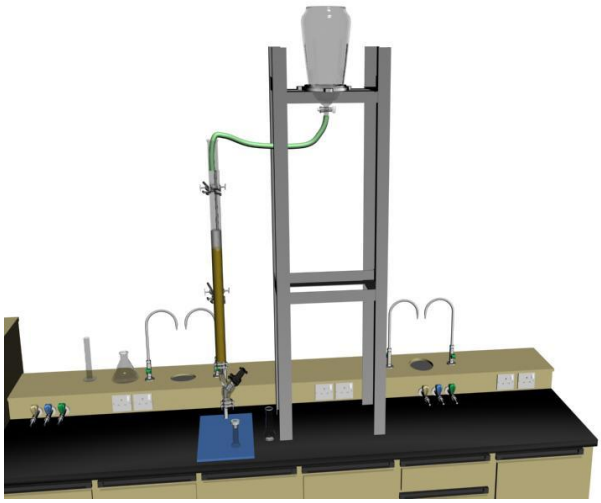

Fig. 1: Experimental apparatus

Zeolite

The adsorption column is packed with 13X molecular sieve obtained from "Rhone Poulenc" with bulk density 0.64 g/cm3, extrudate diameter 1.6 mm, total pore volume 0.285 cm3/g, apparent porosity 0.36, bed void fraction 0.4 and normal pore diameter 10 Ao.

Adsorption Equipment

An experimental apparatus shown in Fig. (1) is constructed for adsorption of BTX fraction by molecular sieve 13X. It consists of 2.5 liter glass container for feed, connected with the adsorption column by a plastic tube. The Q.V.F column of adsorption has 1.5 cm inside diameter and 100 cm long. It is packed with 13X molecular sieve adsorbent. The bottom of adsorption column was fitted with piece of cloth to support the zeolite bed. To reduce the channeling and bad distribution of feed through the bed, balls of polyvinyl chloride were used. These balls are non – reactive with adsorbent and adsorbate. The average length of polyvinyl chloride balls in top of bed is 9 cm and in the bottom is 8 cm. Balls of the bottom of adsorption column protect the adsorption bed from the mechanical force and attrition. Glass receiver is used for collection of effluent.

Gas Chromatography Analysis

The gas chromatography apparatus of type Philips Varian 3300 was used for analysis of benzene and toluene in effluent. The carrier gas used is nitrogen purity of 99 % supplied from Al-Mansour Company. The recorder packed model 621 type was used and the integrator packed model 602 type was used.

Results and Discussion

Effect of Feed Flow Rate on Benzene and Toluene Separation

The effect of feed flow rate in the range 0.77 – 2.0 cm3/min on the benzene and toluene separation from BTX fraction was studied. Figures (2,3, and 4) show the breakthrough curves for adsorption of benzene and toluene using flow rate 0.77, 1.00 and 2 cm3/min, respectively, while figure (5) and (6) show the effect of the flow rate on the adsorption of benzene and toluene respectively.

Examining these figures, it can be seen that at low flow rate the amount of benzene and toluene adsorbed is higher than that obtained with higher flow rate at a given time.

The flow rate increasing decreases the break point time and saturation times, because the increasing in the flow rate leads to decreasing the contact time between

the adsorbate and the adsorbent along the adsorption bed. For example, at flow rates 0.77, 1.00, and 2.00 cm3/min benzene reaches the breakthrough concentration C/Co = 0.1 at times 25, 10, and 5 min respectively. Using the flow rates 0.77, 1.00, and 2.00 cm3/min and time 25 minutes the values of C/Co for toluene are 0.64, 0.849, and 1.022 respectively.

The short break point time of toluene is because toluene has the higher concentration in the influent stream therefore higher driving force between adsorbate and adsorbent was obtained.

As shown in Fig. (5) and (6) the best volumetric flow rate was Q = 0.77 cm3/min.

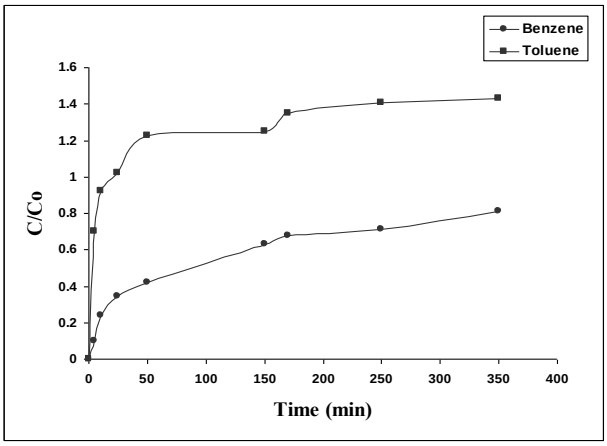

Fig. 4: Breakthrough curves for adsorption benzene and toluene at Q = 2 cm^3/min and Z = 19 cm

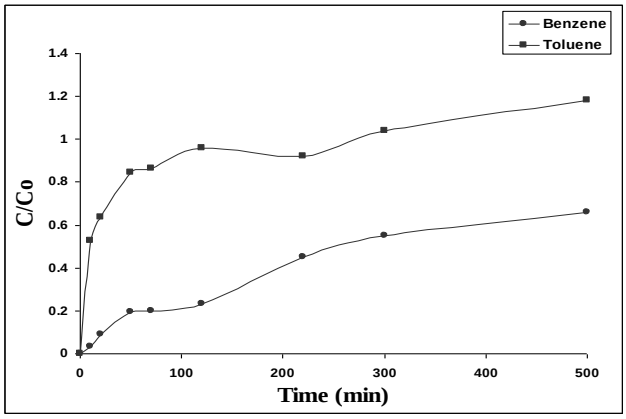

Fig. 2: Breakthrough curves for adsorption benzene and toluene at Q = 0.77 cm^3/min and Z = 19 cm

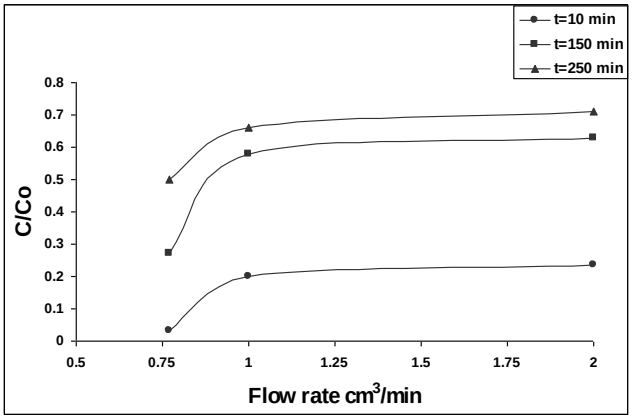

Fig. 5: The effect of feed flow rate on benzene adsorption at constant bed height Z = 19 cm

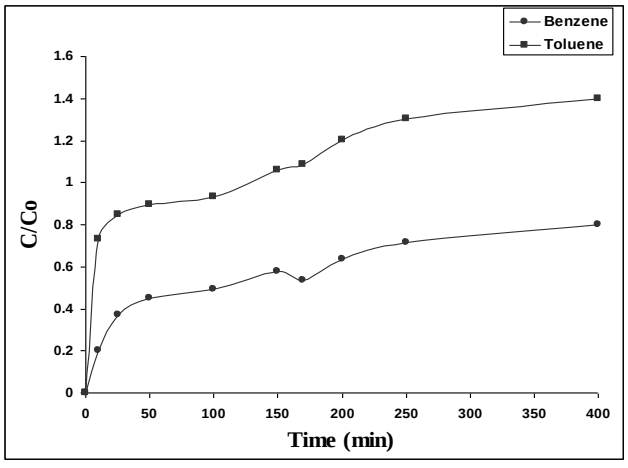

Fig. 3: Breakthrough curves for adsorption benzene and toluene at Q = 1 cm^3/min and Z = 19 cm

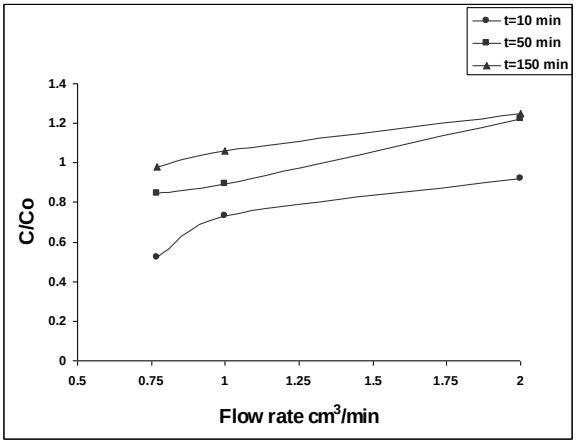

Fig. 6: The effect of feed flow rate on toluene adsorption at constant bed height Z = 19 cm

Effect of Bed Height on Benzene and Toluene Separation

The effect of bed height in the range 31.6 – 63.3 cm on benzene and toluene separation from BTX fraction using constant feed flow rate 1.00 cm3/min was studied. as shown from Figures (7 – 11).

Examining these figures, it can be seen that the break point time values increase by bed height increasing knowing that increasing the bed height will be accompanied by an increase in the bed cost.

At bed heights 31.6, 44.3, and 63.3 cm, benzene reaches the breakthrough concentration C/Co = 0.1 at times 2, 3, and 5 min, respectively. For toluene at bed height 31.6, 44.3, and 63.3 cm and time 5 minutes the values of C/Co are 0.3, 0.151, and 0.130 respectively.

The use of long bed height will give additional spaces for benzene and toluene molecules to be adsorbed, further more increasing the bed height will give a sufficient contact time for these molecules to be adsorbed on the zeolite 13X.

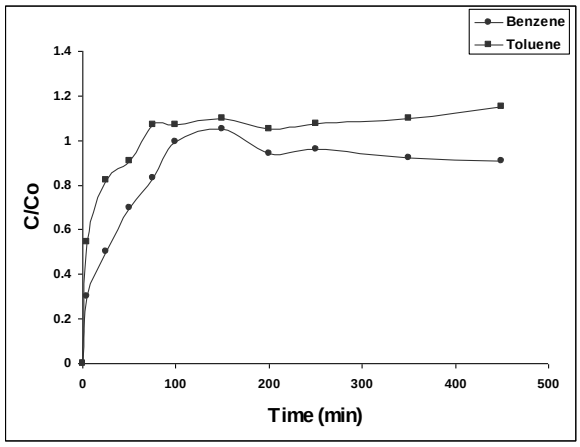

Fig. 7: Breakthrough curves for adsorption benzene and toluene at Q = 1.0 cm^3/min and Z = 31 cm

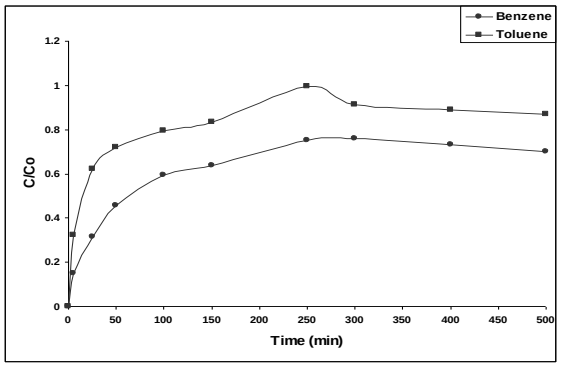

Fig. (8) Breakthrough curves for adsorption benzene and toluene at Q = 1.00 cm^3/min and Z = 44.3 cm

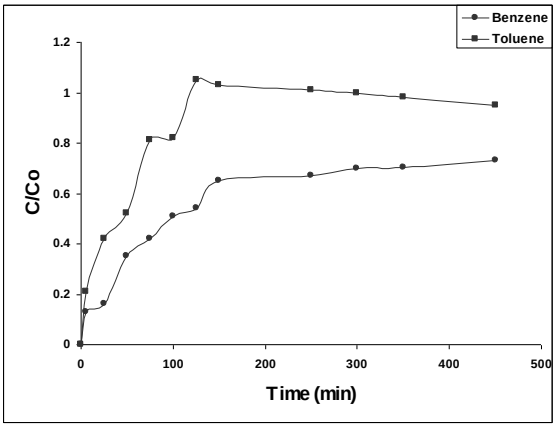

Fig. 9: Breakthrough curves for adsorption benzene and toluene at Q = 1.00 cm^3/min and Z = 63.3 cm

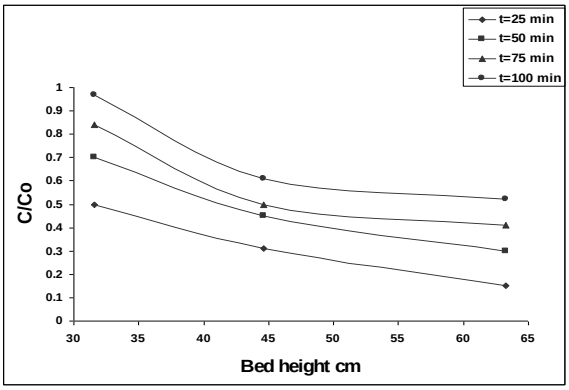

Fig. 10: The effect of bed height on benzene adsorption at constant feed flow rate Q = 1 cm^3/min

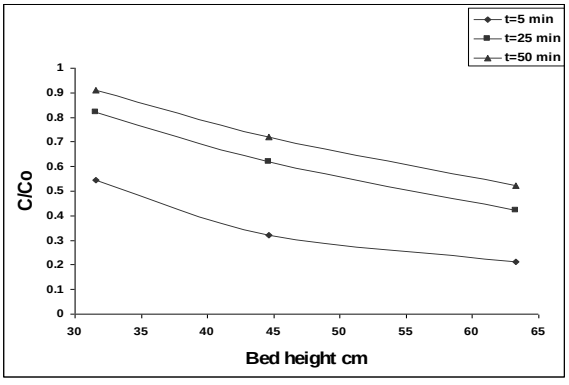

Fig. (11) The effect of bed height on toluene adsorption at constant feed flow rate Q = 1 cm3/min

Effect of Feed Concentration on Benzene and Toluene Separation

The effect of concentration of benzene in the range 0.0559 – 0.2625 g/cm3 and concentration of toluene in the range 0.144 – 0.21 g/cm3 was studied, as shown in Figures 12 and 13 , respectivly.

These figures show that the increasing in inlet concentration will lead to increase driving force and consequently increasing the adsorption rate and leads to quick saturation of the adsorbent with benzene and toluene there by decreasing the breakthrough time of benzene and toluene.

The increasing the inlet solute concentration increases the slope of the breakthrough curve. For example, at benzene initial concentration 0.0559, 0.105, 0.175, and 0.2625 g/cm3 and time 25 min the values of C/Co are 0.315, 0.495, 0.638, and 0.765, respectively. For toluene initial concentration 0.144, 0.181, and 0.21 g/cm3 at time 50 min the values of C/Co are 0.551, 0.181, and 0.21 g/cm3 ,respectively.

As shown in Fig. (13) when P and m-xylenes begin to adsorb some of the toluene is re-adsorbed. This leads to rollup the concentration of toluene in the effluent stream to a level above that of feed.

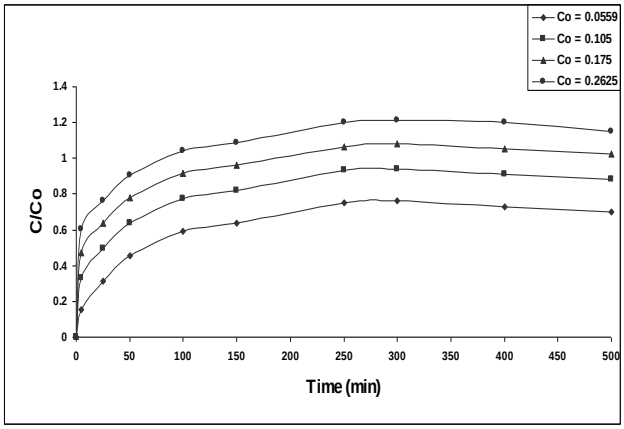

Fig.12: Breakthrough curves at different initial concentration of benzene at Q =1 cm^3/min &Z = 44.3 cm

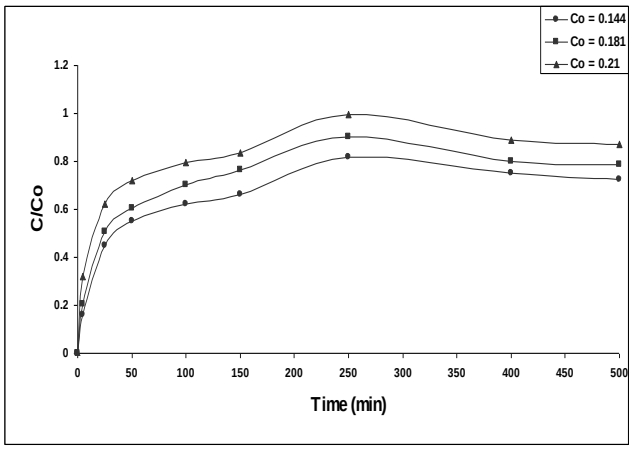

Fig. 13: Breakthrough curves at different initial concentration of toluene at Q = 1 cm^3/min & Z = 44.3 cm

CONCLUSIONS

1. The time required to reach adsorbent saturation is increased by decreasing flowrate.
2. The time of break points increases with bed length increasing for benzene and toluene extraction from BTX fraction.
3. The amount of benzene and toluene adsorbed by molecular sieve 13x increases with increasing the feed stock concentration.
4. The best operating condition in this work for benzene and toluene adsorption are 0.77 cm3/min of feed and 31.6 cm bed height of zeolite 13x.

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