Adsorption separation Technique

Emsalem F. Hawege [1], Almaki A. abushina [1]

[1] Department of Chemical Engineering, Elmergib University, Al-homs Libya

emsalemfrg@yahoo.com
Cheamaki@yahoo.com

الملخص

على مدى العقود القليلة الماضية، كانت تقنيات الامتزاز إحدى الطرق المستخدمة بنجاح في فصل الخلائط الأزيوتروبية. في العمل الحالي، تمت دراسة فروع شجرة الأوكالبتوس (Eu–GAC) وممتصات السيليكا (الرمل) باستخدام وعاء الامتزاز الدفعي لقياس قدرتها على الامتزاز. تمت دراسة تأثير وقت التلامس وجرعة الامتزاز في تعزيز عملية الامتزاز. أظهرت النتائج أن عامل Eu–GAC كممتز يبدو أكثر كفاءة مقارنة بعامل الرمل بنسبة 98٪ تقريبًا.

Abstract

For the past few decades, adsorption techniques are one of the methods used successfully in separating azeotropic mixtures. In the present work, Eucalyptus tree branches (Eu-GAC) and Silica adsorbent (Sand) have been studied by using a Batch Adsorption vessel to measure their capability for adsorption. The effect of contact time and adsorption dose in enhancing the adsorption process has been investigated. The results showed that the Eu-GAC agent as adsorbent appears more efficient compared to the sand agent with almost 98%.

Keywords*:* Azeotropic mixture; Eucalyptus tree branches (Eu-GAC); Silica adsorbent (Sand); adsorbents doses.

1. Introduction.

The term "azeotropy" denotes a mixture of two or more components where the equilibrium vapor and liquid compositions are equal at a given pressure and temperature. More specifically, the vapor has the same composition as the liquid and the mixture boils at a temperature other than that of the pure components boiling points [1]. Azeotropic mixture is a special mixture with a boiling point higher or lower than its components. Therefore, the common distillation methods widely used have not led to separate the azeotropic mixture into pure components. Several methods such as decompression distillation and additive distillation have been applied to separate azeotropic, however, these processes have accompanied with wasting excessive energy. Although chromatography has an important role in the mixtures separation including azeotropic mixtures, it is just used for the analysis due to the disability to separate large amount of the mixture [2]. Some adsorbents, such as synthetic resins, dehydrating agents and more, are very expensive to achieve economically the separation process of azeotropic mixtures [3].

Benzene and cyclohexane classify as important materials since they constitute the raw materials of various petrochemical products. Because the boiling points of these compounds are very close, azeotropic and extractive distillation are frequently used for their separation by adding a third component. During the last decade, a number of polymer membranes have been developed for the separation of a benzene/ cyclohexane mixture. However, in the separation, including aromatic compounds with high adsorptivities, the polymer membranes become swollen due to the accumulation of highly adsorptive compounds in the membrane resulting reducing the separation factors. Thus, inorganic membranes such as zeolite are expected to be effective membranes to separate benzene and cyclohexane. Separation of benzene and cyclohexane by adsorption technique may be identified as an azeotropic mixture [4].

Separation of benzene and cyclohexane is recognized to be the most important and difficult processes in the petrochemical industry. Cyclohexane is produced by catalytic hydrogenation of benzene. The unreacted benzene occurred in the reactor's effluent stream and must be removed for pure cyclohexane retrieving. Separation of benzene and cyclohexane is difficult by a conventional distillation process because these components have close boiling mixtures at the entire range of their compositions. Presently, azeotropic distillation and extractive distillation are used for this separation. These two processes, however, suffer from complexity and high energy consumption. For all these reasons, the industry has always been eager to look for an alternative method to the conventional separation processes [5].

The investigation of the adsorption isotherm aimed to measure the adsorption capacity of the adsorbents (Eu-GAC and Sand) and to determine the equilibrium distribution of the solute concerned (e.g. benzene in the azeotropic mixture benzene-cyclohexane). In this study, the investigation of the efficiency of Eu-GAC and Sand to separate the azeotropic mixture benzene-cyclohexane was carried out.

2. Materials and methods.

2.1Materials.

In this study, we have used activated carbon obtained from Eucalyptus tree branches (Eu-GAC) and Silica adsorbent (Sand) from seaside of El-Khoms. The carbon was produced by thermal pyrolysis of Eucalyptus tree branches in the absence of oxygen. The activated carbon was used without any further treatment except drying in an oven at 100˚C before usage. The sand was washed with ethanol and then dried at 100˚C.

2.2. Method.

In this research, the azeotropic mixtures were prepared from commercial fine reagents according to the components and the ratios. Three types of azeotropic mixtures of benzene and cyclohexane were prepared containing 4%, 6% and 8% benzene in cyclohexane.

All readings were made on a UV-Visible spectrophotometer (model 8700 series Unicam UV/Vis) (Figure 1). The absorbance's of the solutions were measured at the wavelength (255 nm) [6], as benzene absorbs at this wavelength while, cyclohexane has no absorbance above 200 nm. The absorbance readings obeyed Beer's law in this range.

$A_i = S * C_i$ Where: A_i: absorbance (%), S: constant, slope of curve and C_i: concentration, mg / L.

Figure1: UV-Visible (Spectrophotometer).

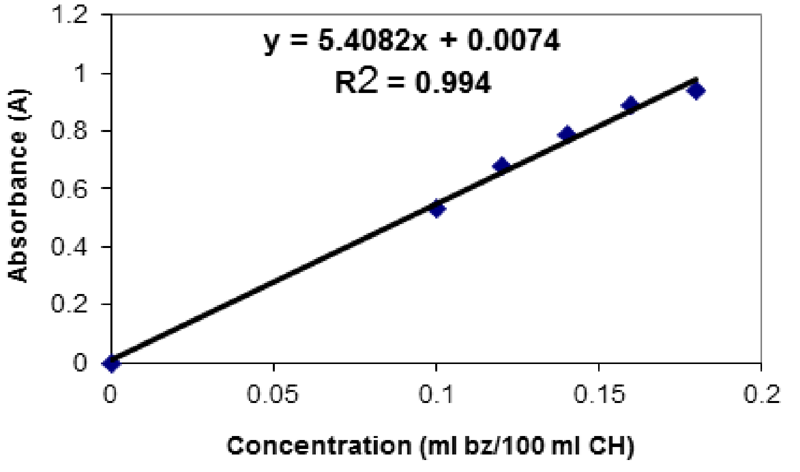

Figure 2: Calibration Curve for the Azeotropic Mixture, (Benzene-Cyclohexane).

2.3. Equilibrium Adsorption studies,

An azeotropic mixture prepared by mixing 0.1-0.18 ml (874 - 1572 mg/L) of benzene with cyclohexane to obtain 100ml solutions containing 4 %, 6 % and 8 % benzene solutions. The experiments were carried out using Eucalyptus (Eu-GAC) and Sand as adsorbents, and were performed at the following conditions:

1. The equilibrium isotherm measurements were carried out for different solution/solid ratios and at constant temperature of 25 ± 2˚C.
2. The equilibrium shaking time (contact time) was varied from one to 15 hours at an stirring speed of 200 rpm.
3. The experiments were conducted in 125 ml Erlenmeyer flasks on a magnetic stirring apparatus as showed in (Figure 3).
4. The ratio of solution/adsorbent was varied from 1 to 3 gm, and the samples were collected at the intervals time.
5. Samples were analyzed by UV/VIS spectrophotometer by measuring absorbance of the samples at λmax (255 nm) for benzene - cyclohexane.

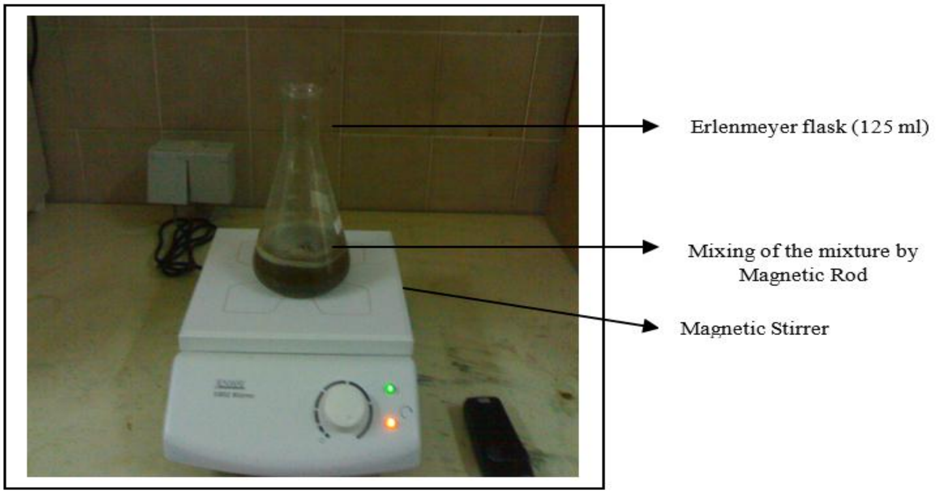

Figure 3: Stirred Batch Adsorption Vessel for Kinetic Studies

3. Results and discussion.

The investigation of the adsorption isotherm aimed to measure the adsorption capacity of the adsorbents (Eu-GAC and Sand) and to determine the equilibrium distribution of the solute concerned (e.g. benzene in the azeotropic mixture benzene-cyclohexane).

3.1 Effect of Contact Time.

Results in Figures 4 and 5 show that the adsorbed benzene increases with an increase the contact time. Other parameters such as dose of adsorbent and pH of solution were kept at optimum situation as well as the temperature was kept at 25˚C. The adsorption efficiency increases when contact time increases from one to 3 hours. Optimum contact time for adsorbed Benzene-Cyclohexane in both adsorbent agents (EU-GAC and Sand) was found to be 4 hours. It is clear that the EU-GAC agent was found to be more efficient than the sand. This result agreed with what was found by Othman [7].

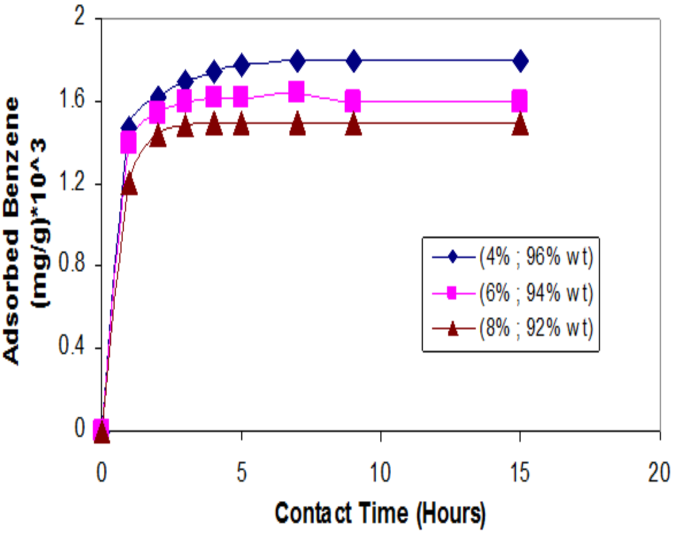

Figure 4: Effect of Contact Time on the Adsorption of Azeotropic Mixture, (Benzene-Cyclohexane) of different initial concentrations, by EU-GAC (1g; Mesh No 100) pH=3; Stirring Speed 200rpm; T= 25˚C, atm.

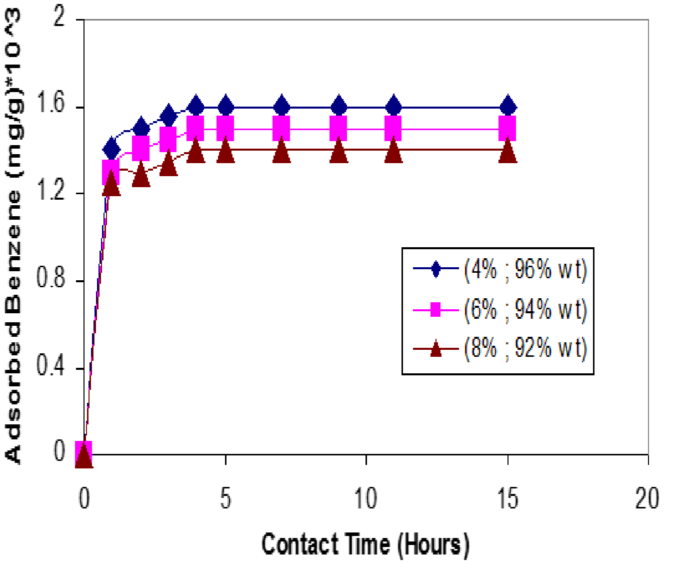

Figure 5: Effect of Contact Time on the Adsorption of Azeotropic Mixture, (Benzene-Cyclohexane) of different initial concentrations, by Sand (1g; Mesh No 100), pH=3; Stirring Speed 200rpm; T= 25˚C,1atm.

3.2 Effect of Adsorbent Dose

The adsorption of benzene on Eu-GAC and Sand adsorbents was examined by using various amount of adsorbents doses from 1g to 3g/100 ml. Figures (6, 7 , 8 and 9) present the adsorption efficiency of benzene on two adsorbents. From these figures, we can conclude that the adsorption efficiency of the adsorbents generally improved by increasing adsorbents dose. The percentage removal of azeotropic mixture from the solution increased from 80-95% to 87-97% as the adsorbent dosage increased from 1g to 3 g for Eu-GAC adsorbent. Whereas the percentage removal of azeotropic mixture from the solution increased from 70-90% to 75-93% as the adsorbent dosage increased from 1g to 3g for sand. This result is expected because of the increased adsorbent surface area and availability of more adsorption sites caused by increasing adsorbent dosage [8].

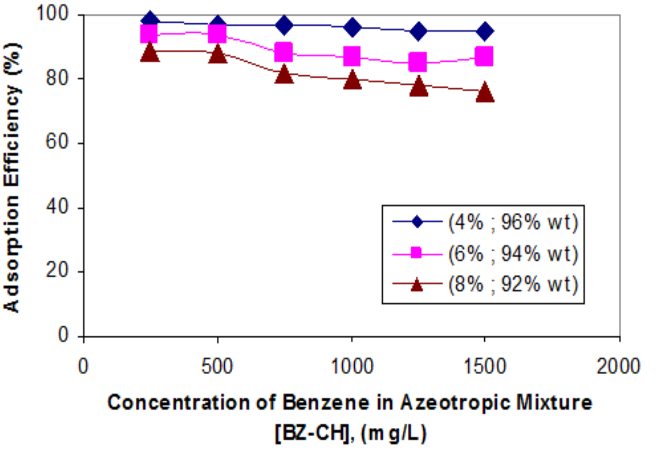

Figure 6: Adsorption percent of the Azeotropic Mixture, (Benzene-Cyclohexane) onto EU-GAC (1g ; Mesh No 100) with different compositions; T= 25 oC,1atm.

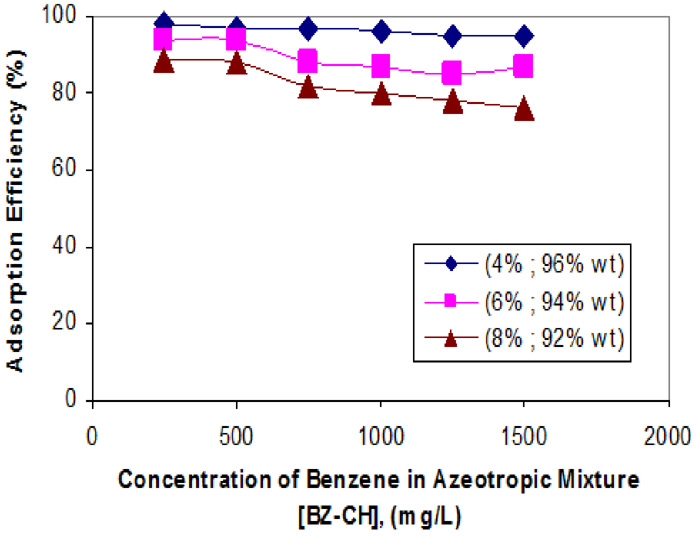

Figure 7: Adsorption percent of the Azeotropic Mixture, (Benzene-Cyclohexane) onto EU-GAC (3g ; Mesh No 100) with different compositions; T= 25 oC,1atm.

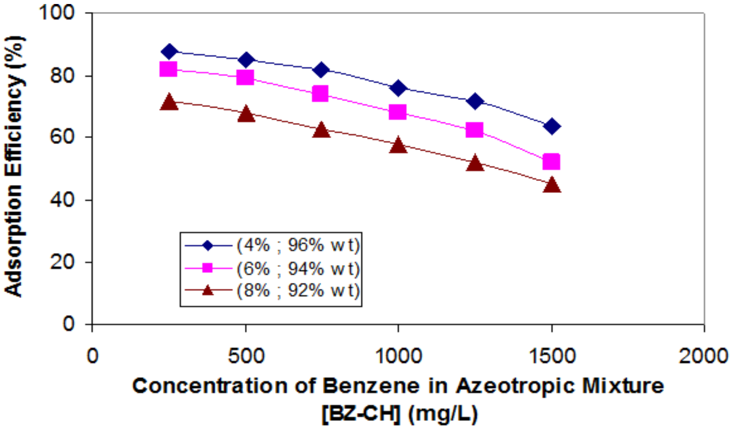

Figure 8: Adsorption percent of the Azeotropic Mixture, (Benzene-Cyclohexane) onto Sand (1g ; Mesh No 100) with different compositions; T= 25 oC,1atm.

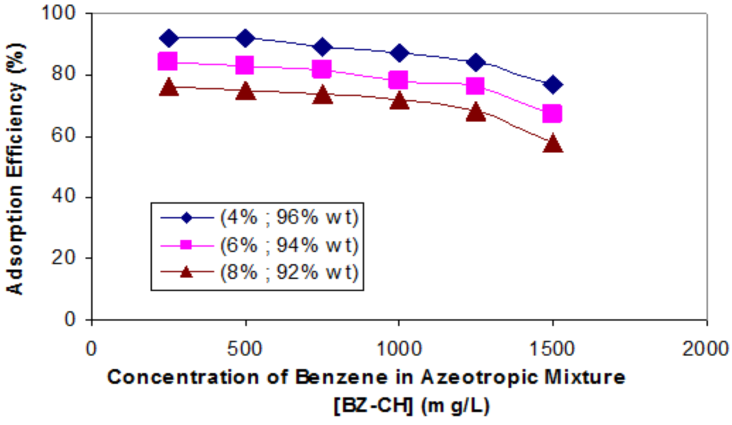

Figure 9: Adsorption percent of the Azeotropic Mixture, (Benzene-Cyclohexane) onto Sand (3g ; Mesh No 100) with different compositions; T= 25 oC,1atm.

4.Conclusion.

In conclusion, we have studied the adsorption isotherms and adsorption efficiency of the two adsorbents Eu-GAC and Sand to separate the azeotropic mixture benzene-cyclohexane in batch column techniques. The adsorption efficiency increased with an increase in contact time, the optimum contact time for both agents was three hrs. The dependence of the adsorption efficiency of benzene on the two adsorbents was studied and it was found that the efficiency is dependent on the dose of the adsorbent and increases with the increase in dose. The behavior of Eu-GAC as an adsorbent appears to us better than sand, it has shown high separation efficiency in the range between 80-97% at different doses.

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