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Adsorption Behavior of Light Naphtha Components on Zeolite (5A) and Activated Carbon

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Abstract

Light naphtha is one of the products from distillation column in oil refineries used as feedstock for gasoline production. The major constituents of light naphtha are (Normal Paraffin, Isoparaffin, Naphthene, and Aromatic). In this paper, we used zeolite (5A) with uniform pore size (5A) to separate normal paraffin from light naphtha, due to the suitable pore size for this process and compare the behavior of adsorption with activated carbon which has a wide range of pores size (micropores and mesopores) and high surface area. The process is done in a continuous system- Fixed bed column- at the vapor phase with constant conditions of flow rate 5 ml/min, temperature 180^{o}C, pressure 1.6 bar and 100-gramweight of each adsorbent according to many other experiments on zeolite (5A) and choose the best conditions for comparison. The molecular sieve (5A) separated the normal paraffin (C_4 – C_8) from light naphtha feed with highest percentage removal reaching a (92.36 %) at the beginning of the process. Activated carbon separated naphthene and aromatics with highest percentage removal reaching a (95.3 %) for naphthenes and a (100 %) for aromatics at the beginning of the separation process. The study shows the difference in physical adsorption behavior and the effect of pore size on these processes.

Keywords: Light naphtha , Zeolite (5A) , Activated carbon , Adsorption process

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1- Introduction

Physical adsorption, a kind of adsorption depends on the potential of surface, shape, and structure of molecules. The principal thought of this type of separation is adsorbing at least one or more components from the blend without changing in compound synthesis[1]. The pore size of adsorbent is a very important factor. This belongs to the ability of components for penetrating to these tubes. Light naphtha, for the most part, comprises of (normal paraffin, isoparaffin, aromatics, and naphthenes)[2].As it is known that light naphtha derived from crude oil that contains many types of hydrocarbons from different families, the most common hydrocarbons types in petroleum fraction (Paraffin, Naphthene, and Aromatic)[3].

Zeolite is a porous media and consists of crystalline aluminosilicates with high surface area and property of molecular sieving[4]. The 5A zeolite has been generally utilized as an adsorbent for separating n-paraffins from oil feedstocks. Adsorption process dependent on 5A zeolite displays predominance in improving the usage proficiency of naphtha[5]. Separation of various group composition in naphtha through the adsorption procedure utilizing zeolites 5A has pulled in extensive enthusiasm from researchers[6], [7]. Zeolite 5A compound have LTA (Linde Type A) cross section structures with Ca cations[8].

The corresponding three-dimension (3D) channels with a diameter of 5.1 Å, which is between the size of linear and branched alkanes[9], [10]specify good shape selectivity in the separation of hydrocarbons.

The equilibrium of adsorption [11], [12]and kinetics[13],[14]of linear paraffins by using zeolite, 5A compounds have drawn in much researches attention. C_5−C_6 range n-paraffin separation, joined with the process of isomerization, was used in the formation of high-octane gasoline[15].

In 2002, UOP (Universal Oil Products) built up a simulated moving bed technique [16],[17]utilizing molecular sieve 5A compounds as the adsorbent and normal pentane as the desorbent to separate linear paraffin from naphtha. The fixed bed adsorption process was built up by East China University of Science and Technology, It is known as Molecular Sieve Fixed-bed Adsorption (MSFA) Technology by using Zeolite 5A as adsorbent and nitrogen as a desorbent[18],[19].In any case, the majority of the examinations on the normal paraffin separation simply focus on the adsorption range of new zeolite 5A, which differs after the adsorption/desorption cycles (regeneration). Activated carbon is solids adsorbent in the separation of hydrocarbons that can create distinctive values of hydrophobicity, as indicated by oxygenated group on the surface; this feature supports organic compound adsorption (aliphatic) with low molecular weight that might be available in the air, increasing its pollution concentrations[20].

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As a porous material, activated carbon has a high surface area and pore volume, which makes it a decent adsorbent in the two gas and liquid phase [21]. There is much research revealed in the writing about the adsorption of the volatile organic compound on activated carbons[22], [23] . Among these compounds is hexane (C_6H_{14}), which is a non-polar compound of a direct aliphatic chain and builds up the cooperation of a dispersive kind with the adsorbent.

In this project, we studied the adsorption selectivity of zeolite (5A) and compared this process with another adsorbent (Activated Carbon) by using light naphtha as a feedstock for this process because light naphtha contains multicomponent of hydrocarbons with a wide range of diameter sizes.

2- Experimental Work

2.1. Materials

Zeolite (5A) purchased from local market produced by (BDH) chemical ltd pool England, extrudate (60-80 mesh). Activated carbon purchased from the local market.

2.2. Physical Properties Of Zeolite (5A)

The physical tests achieved at petroleum research and development center as shown in Table **1**.

Table 1. Physical properties of molecular sieve (5A)

Test Name	Result
BET Surface Area	581.4885 m^2/g
Bulk Density	0.5848 g/cm^3
Crushing Strength	6.3198 N/mm
Pore Volume	0.354553 cm^3/g
Pore size	5Å

2.3. XRF for zeolite 5(A)

XRF test was done at the University of Baghdad – college of science department of geology. Table **2** shows the chemical composition of (5A) molecular sieve.

Table 2. Chemical composition of zeolite (5A)

Component	SiO_2	Al_2O_3	Na_2O	CaO	SiO_2
Weight %	36.89	24.56	2.190	12.29	36.89
SiO_2/Al_2O_3			2.54890		

2.4. Activated Carbon Test Result

Table **3** shows the chemical composition and physical properties of purchased Activated carbon.

Table 3. Chemical composition and Physical properties of Activated Carbon

Parameters	Units	Report value
Iodine number	Mg/gm	1027
Moisture	%	4.86
Ash	%	4.63
Apparent density	gm/ml	0.52
PH		9.2
Over size	%	4.12
Under size	%	2.69
Ball-pan hardness	%	99.38
Surface area	m^2/g	800
Pore volume	cm^3/g	0.676

2.5. Feedstock

Hydrogenated light naphtha supplied from Al-Dura refinery. Table 4.shows the chemical composition of light naphtha the test achieved at petroleum research and development center by gas chromatography according to ASTM (D 2887-02).

Table 4. shows chemical composition of hydrogenated light naphtha

Hydrocarbon name	Wt. %	N-paraffin name	Wt. %
N-paraffin	47.525	C4 (N-butane)	0.06585
Isoparaffin	32.902	C5 (N-pentane)	24.15975
Naphthene	19.44	C6 (N-hexane)	19.42429
Aromatics	0.133	C7 (N-heptane)	3.71383
		C8 (N-Octane)	0.16128

2.6. Adsorbents Activation

(5A) molecular sieve sample was activated at (400 ^oC) for four hours while activated carbon was heated at (200 ^oC) for (90 min) by using the electric furnace and transmitted after 24 hours to plastic cans with silica gel bags to keep the adsorbents dry.

2.7. Experiments

Experiments were performed in the vapor phase by pumping (5 ml/min) of light naphtha from the bottom of a fixed bed column and passed through (100 gram) of adsorbent material. The temperature of the experiment was 180 °C according to many other experiments on adsorption behavior of light naphtha on zeolite (5A).

The Fixed bed stainless steel column are with (440 mm) length and (38.1 mm) inside diameter (500 ml).

The stainless-steel column was followed by stainless steel condenser with an outside diameter (50 mm) and length (200mm) Fig. **1** shows flowchart for the adsorption process. The pressure of experiments was (1.6 bar). Time was set to zero when the pump began to work and record when the first drop appeared.

The samples were collected at dark glass collectors taken at every fixed time interval and the samples were analyzed by gas chromatography device to measure the weight percentage of hydrocarbons components. The adsorption capacity was calculated according to the equation (1)[24].

$$q = \frac{(C^{o} - Ci)}{M} \times Q \times t \quad (1)$$

Where,

q = Adsorption capacity, (g adsorbate /gadsorbent).
C^{o} = Inlet concentration of adsorbate, (g/ml).
Ci = Outlet concentration adsorbate, (g/ml).
M = mass of adsorbent, (gm)
Q = Volumetric flow rate, (ml/min).
t = Adsorption time, (min).

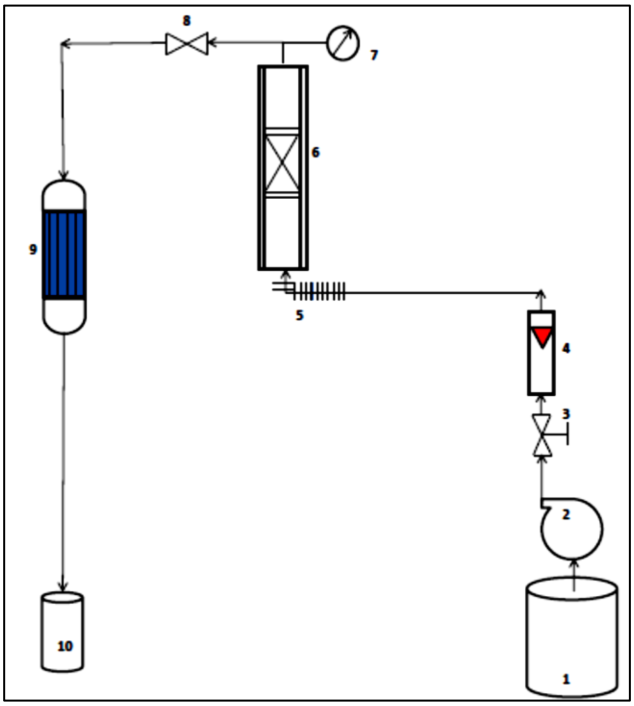

Fig. 1. Flow chart for adsorption separation process: 1- Naphtha container; 2- Fuel pump; 3- Gate valve; 4- Flow meter; 5- Heater tape around the stainless-steel pipe; 6- Fixed bed stainless steel column with heating jacket; 7- Pressure Gauge; 8- Needle valve; 9- Condenser; 10- sample collector.

3- Results and Discussion

3.1. Adsorption Behavior Of Hydrocarbons On Zeolite (5A)

The hydrocarbons in light naphtha majorly consist of (normal paraffin, isoparaffin, naphthene and aromatic) different group of hydrocarbons can be separated by the zeolites through adsorption shape selectivity. Normal paraffins with straight long chains have a smaller footprint than those of iso-paraffins, cyclanes, and aromatics.

The essential channels of zeolites A are 8-membered rings. At the point when the cations are exchanged by Ca^{2+}, the channel diameter is ~5.1 Å.

According to the quantum chemistry calculation, the diameters of normal paraffins are in the range of 4.8−5.0 Å, while the diameters are in the range of 6.1−6.6 Å for iso-paraffins and 6.7−7.4 Å for cyclanes and aromatics [25].

When light naphtha entered at vapor phase to a fixed bed column. Normal paraffin adsorbed in the microspores of zeolite (5A). The S shape of breakthrough curve Fig. **2** shows that the weight % of normal paraffin decreased while other components of hydrocarbon increased.

The decrease in weight percentage of normal paraffin belongs to the suitable diameter sizes for normal paraffin components to enter (5A) zeolite pores, On the other side the isoparaffin, naphthenes, and aromatics stayed in the stream because the diameter of the size for these hydrocarbons is larger than the pore size of (5A) zeolite[26].

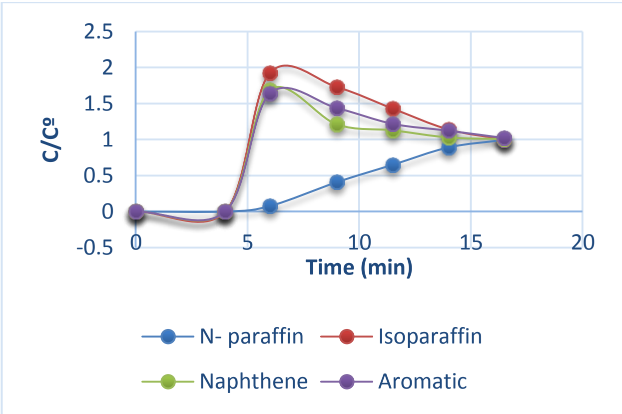

Fig. 2. Adsorption breakthrough curve of hydrocarbons separation using zeolite (5A)

The percentage removal of normal paraffin at the beginning of the adsorption process (first sample of non-adsorbed components 4 ml) reached to 92 % and weight % of isoparaffin increased by 92 % compared to the weight percentage of the inlet. on the other hand the isoparaffins behave like an inert on zeolite (5A)[27]. Naphthene weight percentage increased by 69 % while aromatic increased by 64 % compared to inlet weight percentage.

The increase in the weight percentage of isoparaffin, naphthenes and aromatics may depend on the inlet concentration of these types of hydrocarbons. Adsorption capacity will decrease as time passed until equilibrium (saturation) take place where q_{max} for total normal paraffin was 13.16 g/100g-adsorbent.

The results partly agree with previous studies with single hydrocarbon at the very dilute concentration[28]which proved that the adsorption of hydrocarbon mixture strongly depends on the temperature of the experiment and the inlet composition. Whereas the earliest time of saturation belongs to the high concentration of normal paraffin in the feed (47.525 %).

Normal paraffins with low carbon numbers that occupy zeolite 5A compound micropores can be partially interchanged with larger carbon numbers of normal paraffins.

The roll-up phenomenon[29]can be noticed in the low-carbon-number normal paraffin adsorption breakthrough curves as shown in Fig. **3**. The breakthrough curve of normal paraffin components on zeolite (5A)

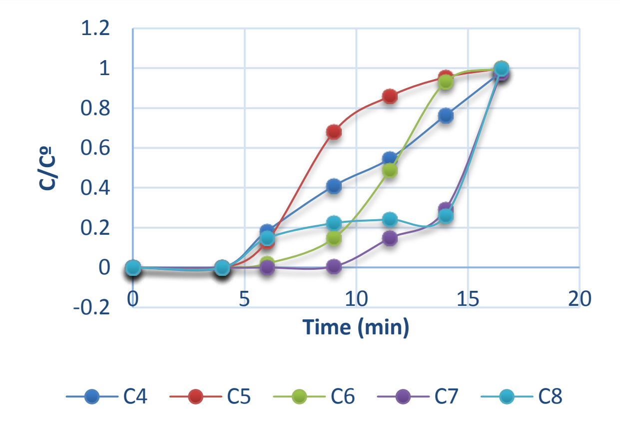

Fig. 3. Adsorption breakthrough curve of normal paraffins contents on zeolite (5A)

The percentage removal of normal butane C_4 at the beginning of the adsorption process (first sample of non-adsorbed components 4 ml) reached to 81.7 %, while normal pentane C_5 reached to 86.8 %, normal hexane percentage removal was 98 %, normal heptane 99 % and normal octane percentage removal was 85 %, from these results we notice that highest weight % components C_5, C_6 and C_7 will be preferred in the adsorption due to their high concentration at the inlet feed of light naphtha.

In addition, we noticed form adsorption breakthrough curve that the components with a large number of carbon atoms preferred in this type of separation this agree with yang et al.[29].

3.2. Adsorption Behavior of Hydrocarbons on Activated Carbon

Due to the high surface area and the wide range of pores size distribution of activated carbon, non-polar molecules of a certain volume and molecular weight such as hydrocarbons have a strong affinity to activated carbon. The variation in affinity includes activated carbon being an appropriate adsorbent when separating or purifying the gas phase [30].

In spite limit use of activated carbon in the separation of hydrocarbons mixture.

We notice that activated carbon generally adsorbed the naphthenes and aromatics from light naphtha feed when entered fixed bed column so there are increasing in weight percentage of isoparaffin and normal paraffin as shown in Fig. **4**.

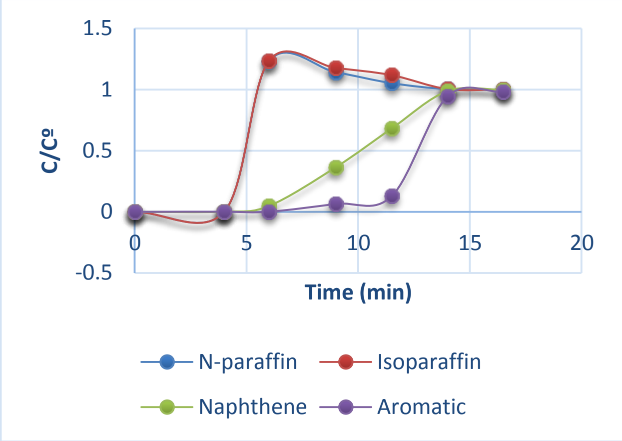

Fig. 4. Adsorption breakthrough curve of hydrocarbons separation using Activated carbon

At the beginning of the adsorption process (first sample of non- adsorbed components 4ml), we notice that isoparaffin and normal paraffin increased by 23 % compared to the inlet concentration.

From the other side, there is a decrease in weight % of naphthenes and aromatics comparing to inlet weight percentage.

The percentage removal of naphthenes reached to 95 % while the aromatics reached to 100 % whereas the inlet concentration of aromatic increased a little bit (0.133%) compared to other types of hydrocarbons.

The separation behavior belongs to the large pores size compared to the zeolite (5A) which allowed for hydrocarbons that have large diameter sizes to enter pores of activated carbon.

This may agree with Lillo-Ródenas et al. who referred to The percentage removal of hydrocarbons may also rely on porosity and the surface chemistry of the adsorbent used such as surface functional groups[31].

This adsorption behavior will continue until equilibrium (saturation) take place. For more details about normal paraffin concentration, we must discuss the results of normal paraffins contents.

We noticed at the beginning of the process (first sample of non – adsorbed components 4ml) the weight % of C_6, $C_7$and C_8 decreased while the C_4 and C_5 increased.

The weight % of normal butane C_4 increased by 281 % while normal pentane C_5 increased by 139 % this increase refers to the normal paraffin which has a large number of carbon atoms preferred in the separation process although the high concentration of C_5 normal pentane in the feedstock.

In addition, there is a decrease in weight % compared to inlet concentration for normal hexane C_6 by 97.5 %. While normal heptane percentage removal was 99.7 % and normal octane was 100 %.

Fig. 5 shows adsorption breakthrough curve for normal paraffin components.

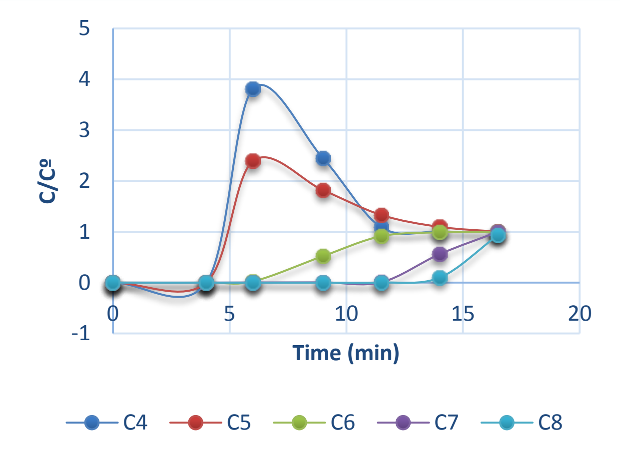

Fig. 5. Adsorption breakthrough curve for normal paraffin components

The adsorption behavior of light naphtha hydrocarbons may agree with Villa- cañas et al. that is referred to that the activated carbon surface chemistry plays a main role in removing components of hydrocarbons as it impacts both electrostatic and dispersive interactions between adsorbents and adsorbates, which are essentially interactions in the form of van der Waals[32]. The adsorption capacity calculated according to equation (1) for both naphthenes and aromatics where q_{max} was 5.55 g/100g - adsorbent and 0.0399 g/100g - adsorbent for naphthenes and aromatics respectively.

4- Conclusions

The study of hydrocarbon adsorption of light naphtha components at vapor phase in fixed bed adsorber refers to the ability to adsorb n-paraffin from light naphtha by (92 %) at process beginning and increases the percentage of (isoparaffin, naphthene and aromatic) because the components of normal paraffin has size diameter smaller than pores of zeolite (5A). From the other hand activated carbon which has a wide range of pore size generally adsorb naphthenes and aromatics by (95%) and (100 %) respectively, while the percentage of total paraffins increased (isoparaffin and normal paraffins).

For more details, we noticed that the normal paraffins components C_4 normal butane and C_5 normal pentane increased while other components C_6 (normal hexane), C_7 (normal heptane) and C_8 (normal octane) decreased. This indicates that adsorption behavior strongly depends on the inlet concentration and the structure of adsorbents.

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سلوك امتزاز مركبات النفثا الخفيفة باستخدام الزيولايت (5أ) والكربون المنشط

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الخلاصة

النفثا الخفيفية هي احد نواتج برج التقطير في المصافي النفطية تستخدم كمادة خام لانتاج البنزين . المركبات الرئيسية للنفثا الخفيفة هي (البرافينات الخطية , البرافينات المتفرعة , النفثينات و المركبات الاروماتية) . في هذا البحث تم استخدام الزيولايت (5A) بحجم مسامات واحد (5 انكستروم) لغرض فصل البارافينات الخطية من النفثا الخفيفة , ويعود هذا الى الحجم المناسب للمسامات في هذه العملية ومقارنة سلوك الامتزاز مع الكربون المنشط والذي يحتوي على مدى واسع من حجوم المسامات (مايكروبوروس و ميزوبوروس) بالاضافة الى المساحة السطحية العالية. العملية تمت بالنظام المستمرباستخدام (مفاعل الحشوه الثابتة) في الطور البخاري و بظروف ثابتة بجريان (5 مل/ دقيقة) , ودرجة الحرارة 180^{o}م و الضغط 1,6 بار و 100 غم وزن المادة الممتزة. المنخل الجزيئي (5A) قام بفصل المركبات البرافينية الخطية ($C_4 - C_8$) من وقود النفثا الخفيفة بأعلى نسبة ازالة وصلت الى (92,36 %) في بداية العملية. اما الكربون المنشط قام بفصل المركبات النفثينية والاروماتية بأعلى نسبة ازالة وصلت الى (95،3 %) للنفثينات و (100 %) للمركبات الاروماتية في بداية عملية الفصل. هذه الدراسة تبين الاختلاف في سلوك الامتزاز الفيزياوي وتاثير حجم المسامات على العملية .

الكلمات الدالة: النفثا الخفيفة ، الزيولايت (5 أ) ، الكربون المنشط ، عملية الامتزاز.