

Preparation and Formation of Zeolite 5A from Local Kaolin Clay for Drying and Desulphurization of Liquefied Petroleum Gas

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Abstract

This work deals with preparation of zeolite 5A from Dewekhala kaolin clay in Al-Anbar region for drying and desulphurization of liquefied petroleum gas. The preparation of zeolite 5A includes treating kaolin clay with dilute hydrochloric acid 1N, treating metakaolin with NaOH solution to prepare 4A zeolite, ion exchange, and formation. For preparation of zeolite 4A, metakaolin treated at different temperatures (40, 60, 80, 90, and 100 °C) with different concentrations of sodium hydroxide solution (1, 2, 3, and 4 N) for 2 hours. The zeolite samples give the best relative crystallinity of zeolite prepared at 80 °C with NaOH concentration 3N (199%), and at 90 and 100°C with NaOH concentration solution 2N (184% and 189%, respectively). Zeolite 5A was prepared by ion exchange of zeolite 4A prepared at 90°C and 2N NaOH concentration with 1.5 N calcium chloride solution at 90 °C and 5 hours, the ion exchange percentage was 66.6%. The formation experiments included mixing the prepared powder of 5A zeolite with different percentages of kaolin clay, citric acid and tartaric acid to form an irregular shape of zeolite granules. Tartaric acid binder gives higher bulk crushing strength than that obtained by using citric acid binder with no significant difference in the surface area. 7.5 weight% tartaric acid binder has the higher bulk crushing strength 206 newton with surface area 267.4 m²/g. Kaolin clay binder with 15 weight% gives the highest surface area 356 m²/g with bulk crushing strength 123 newton, it was chose as the best binder for zeolite 5A. The prepared granules of 5A zeolite were used for the adsorption experiments of H₂O, and H₂S contaminants from LPG. Different flow rates of LPG (3, 4, and 5 liter/minute) were studied. It was found that H₂O is the strongly adsorbed component and H₂S is the weakly adsorbed component. The best flow rate in this work for H₂O, and H₂S adsorption is 5 liter/minute of LPG. The adsorption capacity for H₂O was 7.547 g/g and for H₂S was 1.734 g/g.

Keywords

LPG, Drying and sweetening, Kaolin clay, Zeolite 5A, Formation, Adsorption

Introduction

Liquefied petroleum gas (LPG) is the fuel of choice for inhabited areas and for leisure applications. LPG consists of a mixture of propane and butane in

various ratios depending on country and season. Natural gas and LPG contain sulfur components, either naturally occurring, or added deliberately as odorant to odorize the

otherwise odorless gas. The sulfur removal task for LPG is more challenging than it is for natural gas. Propane, butane and heavier hydrocarbons are potential competitors for the sulfur compounds for adsorption sites, decreasing the adsorption capacity. Sulfur levels in LPG can be much higher when compared to natural gas^[1].

Impurities such as H₂S in light hydrocarbons usually cause hazardous odors, corrosion problems and air pollution; therefore elimination of these impurities is very important^[2]. Zeolite adsorbents are used to dry the natural gas or liquefied petroleum gas to prevent freezing and corrosion in pipeline, remove sulfur compounds from the natural gas or liquefied petroleum gas to prevent corrosion in burners, remove compounds that are obnoxious or toxic such as the odoriferous hydrogen sulfide and mercaptans that form sulfur dioxide pollutants when burned for home cooking and heating^[3].

Zeolites are generally available in bound forms where the zeolite crystals (1–5µm) are formed in particle shapes (beads, pellets, extrudates) using a binder material (clay, alumina, polymers, etc.). The purpose of the bound forms (diameters of 0.5–6.0 mm are common) is to reduce the pressure drop in adsorbent columns^[4].

Fixed bed adsorption has been used widely in separation and purification of gases and liquids. The breakthrough curves are perhaps the most common basis for assessing the behavior of adsorbents^[5]. An examination of the breakthrough curve gives an estimate of the adsorptive capacity of the bed^[6]. The breakthrough curve for a particular component is obtained by measuring its concentration in the effluent streams as a function of time until equilibrium is obtained. The concentration of a

component in the effluent streams at the beginning is zero until breakpoint, at which the concentration rises gradually to the initial value of the inlet stream^[7].

Inoue & Tsunoi^[8] produced shaped zeolite from a mixture of zeolite, poly carboxylic acid binders, kaolin clay binder, and water. The binder amount was about 1-10 wt. %. Tartaric acid binder gives crushing strength 12N/mm², while kaolin clay gives crushing strength 5 N/mm².

Kikkinides, *et al.*^[9] studied the removal of H₂S from pre-dried natural gas contains 1000 ppm H₂S and 5% CO₂ by zeolite 5A. The purified adsorption product contains one ppm H₂S.

Chantawong & Harvey^[10] synthesized zeolite A from kaolin by hydrothermal treatment with various concentrations of NaOH solutions (0.5-4N) crystallized at 100 °C for 1 to 5 hours. Solid to liquid ratio was 1 gram metakaolin to 20 milliliter NaOH solution. The best crystallinity of zeolite was obtained at 2N NaOH solution with 4 hours.

Ugal, *et al.*^[11] studied the preparation of zeolite 4A from Iraqi kaolin. Metakaolinite was treated with 8M sodium hydroxide solution with a solid: liquid ratio of 1: 5 at 90 °C for 4 h. The obtained zeolite powder was mixed with 15 % raw metakaolinite, and calcined for 4 h at 500° C. The crushing strength of the prepared zeolite was 16 Newton and the adsorption capacity was 23 H₂O/100g.

The aim of this study was preparation of 5A zeolite from local kaolin clay by preparing 4A zeolite and study the effect of different temperatures and concentrations of sodium hydroxide solutions on the degree of relative crystallinity, studying the formation of the prepared 5A zeolite with different contents of

kaolin clay, citric acid, and tartaric acid, finally, studying the adsorption performance of the prepared 5A zeolite granules in the drying and desulfurization of liquefied petroleum gas.

Experimental Work

Materials

Liquefied Petroleum Gas (LPG)

LPG was supplied by Midland Refineries Company/Al-Durra Refinery. The analysis of the LPG was done by Al-Durra Refinery and tabulated in Table 1.

Table 1, LPG analysis

Test		Result
Chemical analysis (vol. %)	Ethane	0.14
	Propane	34.62
	iso Butane	19.96
	n. Butane	45.22
	iso Pentane	0.06
Specific gravity		0.564
Vapor pressure @ 37.8°C, kg/cm ²		7.36
95% of LPG volatilized at, °C		-1

Kaolin Clay

Kaolin clay was supplied by The State Company of Geological Survey and Mining. It is available in Dewekhala Quarry in Al-Anbar region. Table 2 shows the chemical composition of kaolin clay.

Table 2, Chemical composition of local kaolin clay

Component	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	Na ₂ O	L.O.I
Wt%	49.7	34.1	1.8	1.1	0.5	12.3

Chemicals

The chemical compounds are tabulated in Table 3.

Table 3, Chemical Compounds

compound	company	purity
hydrochloric Acid	Gainland Chemical Company GCC, UK	35-36%
Sodium Hydroxide	RIEDEL-DE HAËN AG SEELZE-HANNOVER	98%
Calcium Chloride	Weifang Bell Chemical Co., Ltd., China	98%
Silver Nitrate	Tianjin Yinlida Chemicals Co., Ltd., China	99%
Citric Acid Anhydrous	Fluka AG, CH-9470 Buchs, Switzerland	99.5%
Tartaric Acid Crystals	HOPKIN AND WILLIAMS, England	99.5%
Silica Gel	THOMAS BAKER CHEMICALS, India	water adsorption capacity 30% of its weight

Preparation of 5A Zeolite

- 1- Local kaolin clay was crushed and sieved to the grain size -75 micron. The sieved kaolin mixed with hydrochloric acid solution 1N with a solid to liquid ratio (3 g: 10 ml), the mixture was agitated and heated at 100°C for 30 minutes^[12].
- 2- The acid treated clay was washed with deionized water and filtered several times until the washing water was free from chloride ions. This was showed by adding several drops of 0.1N silver nitrate solution to the filtrate.
- 3- The dried treated kaolin clay was calcined at 550°C for 2 hour to convert kaolin to metakaolin^[13].
- 4- The metakaolin was grained, sieved to the particle size -75 micron, treated with different concentration of sodium hydroxide solutions (1, 2,

3, and 4N). The metakaolin powder to sodium hydroxide solution ratio was (1 g: 5 ml) ^[14]. The mixture was aged for 24 hours at room temperature ^[15]. The reaction mixture was agitated and heated at different temperatures (40, 60, 80, 90, and 100 °C) for 2 hours^[16]. The mixture was filtered and washed with deionized water to remove the excess of alkali, decanted and filtered several times till the pH reaches 10.5, then dried at 100°C for 4 hours and milled to the particle size -63 micron.

5- Sodium zeolite 4A was converted to calcium zeolite 5A by ion exchange with 1.5N calcium chloride solution by heating at 90°C for 5 hours then filtered and washed several times with deionized water until removing all the ions of chloride by adding drops of 0.1 N silver nitrate solution to the filtrate. The extent of exchange was determined using equation (1) by chemical analysis of zeolite before and after ion exchanging with calcium chloride solution ^[17].

$$\% \text{ exchange} = \frac{\text{quantity of Na}^+ \text{ removed}}{\text{original quantity of Na}^+} \times 100 \dots(1)$$

Pelletizing of Zeolite 5A

Different types of binders were used in the formation of zeolite granules. These are tartaric acid, citric acid ^[8], treated kaolin clay ^[18] as shown in Table 4.

Table 4, The mixtures of zeolite 5A and binders

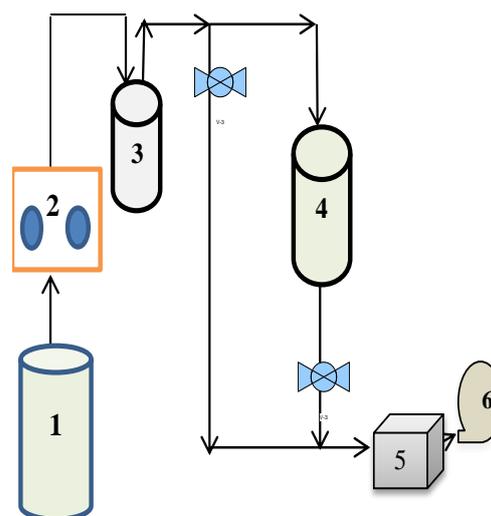
binder	Treated kaolin clay -63 micron			Citric acid crystal			Tartaric acid Crystal		
	10	15	20	2.5	5	7.5	2.5	5	7.5
Wt%									

Water is used in an amount 60% weight of the mixtures of zeolite and binder. The formation of the pastes of zeolite and binder was done by using

the Oedometer instrument. The paste after compression was left at room temperature to the next day to let the excess water in the paste be vaporized slowly then dried at 100°C for 24 hours. Zeolite disks contained organic binder, citric and tartaric acid, were calcined at 350°C for 2 hours, while zeolite disks contained kaolin clay as a binder calcined at 550°C for 2 hours. The disks were crushed into small piece and sieved to the grain size -2.36 + 0.85 mm. The zeolite granules were tested for determining surface area, bulk crushing strength, and bulk density. The zeolite-binder mixture which gives the best properties was chosen for purification of liquefied petroleum gas.

Adsorption Performance Experiments

The adsorption performance experiments were carried out in the laboratory unit consist flow gas control board, adsorption column, and hygrometer. The adsorption unit has adsorption column of 2.5 cm diameter and 37.5 cm height. Figure (1) shows the flow diagram of adsorption unit.



1- LPG cylinder, 2- flowmeter and pressure gauge, 3- water container, 4- adsorption column, 5- hygrometer, 6- H₂S gas sampling balloon

Fig. 1, Flow diagram of the adsorption process

Different flow rates of LPG (3, 4, 5 liter/minute) were studied.

Test Methods

Chemical Composition

The chemical composition of prepared zeolite samples was done by the laboratories of the State Company of Geological Survey and Mining, Ibn Sina State Company, and Petroleum Research and Development Center.

X-ray Diffraction

X-ray diffraction analysis was done in the State Company of Geological Survey and Mining by using Maxima XRD-7000 diffractometer/Japan.

BET Surface Area

The surface area of zeolite granules was determined in the Petroleum Research and Development Center by using surface area analyzer-Therm/USA.

Bulk Density

Bulk density is determined in the Petroleum Research and Development Center by using tap density.

Bulk Crushing Strength

The bulk crushing strength of the zeolite granules was obtained by using the testing device CRUSH BK – CRUSH STRENGTH from MA materials technologies, USA) determined in the Petroleum Research and Development Center. This device is used for measuring the crush strength of solid materials, their size can be as small as 1 mm and up to about 20 mm.

Concentration of H₂O and H₂S

The concentration of H₂O in LPG was measured by hygrometer device Shaw, ALPHA moisture system and H₂S was measured by the Tutwiler method (UOP) 9-59.

Results and Discussion

Preparation of Zeolite 5A

The chemical analysis of kaolin clay before and after the treating with the dilute hydrochloric acid and metakaolin calcined at 550°C is shown in Table 5.

Table 5, The chemical composition of locally kaolin clay before and after treating with dilute HCl, and metakaolin

Weight %	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	L.O.I	Na ₂ O
Kaolin clay before washing	49.70	34.10	1.80	12.30	0.50
Kaolin clay after washing	49.56	34.62	0.17	12.47	0.06
metakaolin	37.50	53.20	-	3.14	0.09

The Fe₂O₃ percentage was reduced by 90%. The iron contaminants occur in the kaolin as goethite, hydrogoethite, they may also enter into the kaolinite lattice, in which case the iron is uniformly distributed and firmly bound throughout its mass^[19], in this case it is difficult to remove iron from the clay.

Due to the dehydroxylation, SiO₂ and Al₂O₃ percentages increased from 49.56% and 34.62% to 53.20% and 37.50% respectively, while L.O.I% was decreased from 12.47% to 3.14%.

For evaluation the relative crystallinity of prepared 4A zeolite at different temperatures and NaOH concentrations, X-ray diffraction analysis was done and compared with standard sodium zeolite 4A. The strongest relative intensities peaks for reference zeolite 4A and the prepared samples of zeolite 4A were determined at the values of 2θ = 7.18°, 10.17°, 12.46°, 16.11°, 21.6°, 23.99°, 27.11°, and 29.94°. The values of the strongest intensity peaks were used for determination the area under the peaks of the strongest intensities. The values of the area under the peaks were used for estimation of relative crystallinities% by equation (2)^[20].

$$\text{relative crystallinity of 4A} = \frac{S_x}{S_r} \times 100 \quad \dots(2)$$

Where

S_x = sum of integral peak intensities for the sample 4A

S_r = sum of integral peak intensities for the reference 4A

Figure (2) shows the effect of temperatures and NaOH concentrations on the relative crystallinity.

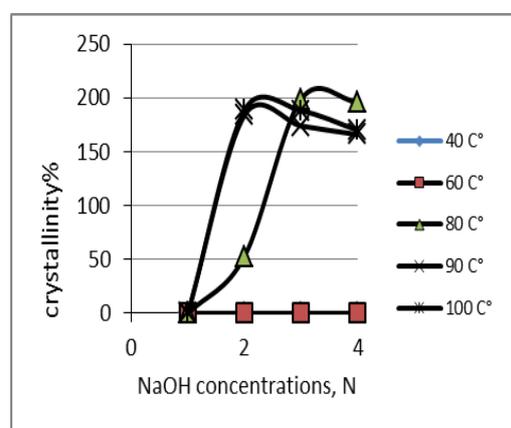


Fig. 2, The effect of temperatures and NaOH solutions concentration on the relative crystallinities% of the prepared samples of zeolite 4A

Figure (2) shows that no relative crystallinity was observed at temperatures 40 and 60°C with all NaOH concentrations. When the temperature was increased (80, 90, 100 °C) the crystallinity of 4A was appeared except with 1N NaOH concentration.

Higher relative crystallinities were obtained at 80 °C and 3N NaOH concentration (199%), at 90°C and 2N NaOH concentration (184%), and at 100°C with 2N NaOH concentration (189%). The crystallinity begin slightly to decrease at NaOH concentration of 3N at higher temperatures (90 and 100°C) and 4N at 80°C. This decrease may be due to the increase in the concentration of OH⁻ which can cause as consequence a change in the

resulting zeolite composition which may lead to the formation of sodalite in place of zeolite A [21]. Sodalite structurally similar to zeolite A but it is more compact and has much less void space available to water molecules [22]. The negative effect of increasing the concentration of NaOH also pointed out by Haden, *et al.* [23], Wongwiwattana [24], and Chantawong, *et al.* [10].

It could be said that the best temperature of crystallization and NaOH concentration for synthesis 4A was 90°C and 2N respectively. Although 100°C gave slightly higher crystallinity than 90°C, temperature 90°C was preferred in order to reduce the observed bubbling and solution vaporization at temperature 100°C. Temperature 80°C gives moderately higher degree of crystallinity but it was not the choice for the best operating temperature because it needs to high concentration of NaOH solution (3N).

Table 6, The chemical composition of 4A zeolite prepared at 90°C and 2N

component	SiO ₂ %	Al ₂ O ₃ %	Na ₂ O %
Weight%	44.17	28.13	12.63

The chemical analysis of calcium ion exchange zeolite is shown in Table 7, while figure (3) shows the X-ray diffraction of calcium-exchanged zeolite 5A.

Table 7, Chemical composition of calcium zeolite 5A

component	SiO ₂	Al ₂ O ₃	Na ₂ O	CaO
Weight %	42.70	25.32	4.23	11.20
SiO ₂ /Al ₂ O ₃	2.86			

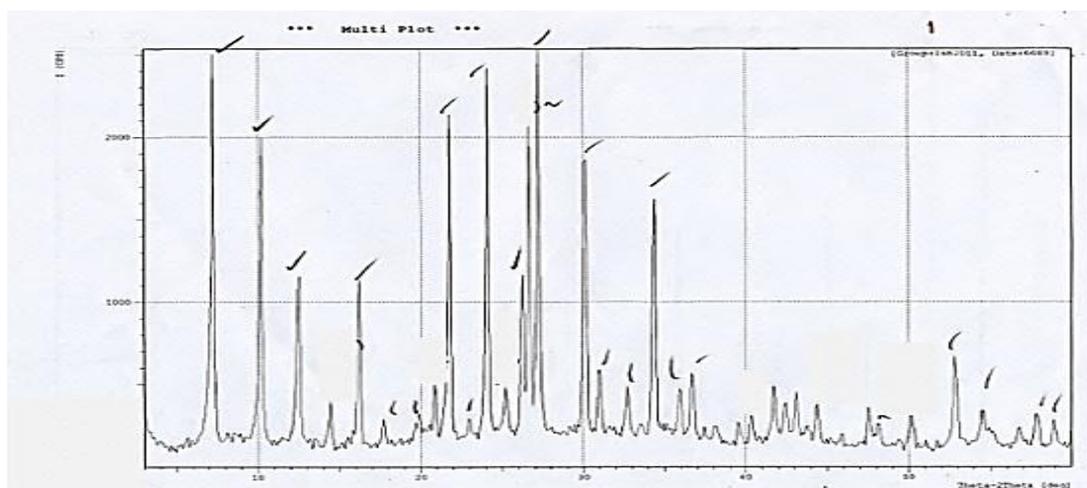


Fig. 3, X-ray diffraction pattern of zeolite 5A

In the exchange operation of calcium for sodium at least about 25% of the sodium originally present in the zeolite 4A zeolite should be replaced by calcium to obtain effective pore size (approximately 5Å). The percentage of ion exchange of this study estimated by equation (1) was 66.6%. This result is within the range of the prepared ion exchange 5A zeolite by Pacaud, *et al.* [18].

In the formation of 5A zeolite the dried formed 5A zeolite with kaolin clay binder was calcined at 550 °C, while those formed with citric acid and tartaric acid were calcined at 350 °C. The selected calcination temperatures is compatible with temperature at which the zeolite 5A is thermally stable, the temperature at which the clay binder undergoes an irreversible phase change, the temperature which is sufficiently high to burn off the poly carboxylic acid, and the minimum temperature for calcination is that temperature at which the loss of the water of hydration of zeolite will be affected. Zeolite 5A is thermally stable at 700°C but at 800°C converted to amorphous material. Kaolin clay binder converts to an amorphous (metakaolin) binder at 550°C to improve the mechanical strength and reduce the infiltration of water and solution through the formed sample.

The prepared shaped bodies with polycarboxylic acids binders are calcined at 350°C which is sufficiently high to burn off the organic acid.

After drying, calcination, crushing, and sieving of the prepared bounded zeolite to the particle size (-2.36mm) – (+0.85 mm), the physical properties (surface area, bulk crushing strength, and bulk density) were determined and the results are shown in Table 8.

Table 8, The result of binder types and quantity on the physical properties of 5A zeolite granules

Binder weight %	Surface area (m ² /g)	Bulk crushing strength (Newton)	Bulk density (g/cm ³)
Citric acid 2.5	-	5	0.619
Citric acid 5	289.6	9	0.615
Citric acid 7.5	258.2	97	0.623
Tartaric acid 2.5	-	16	0.580
Tartaric acid 5	270.5	120	0.549
Tartaric acid 7.5	267.4	206	0.553
Kaolin clay 10	-	81	0.612
Kaolin clay 15	356	123	0.597
Kaolin clay 20	284.6	181	0.592

Table 8 shows that citric acid binder gives low crushing strength with 2.5 weight% and 5 weight% (5 and 9 Newton respectively). Increasing the binder content to 7.5 weight% the bulk crushing strength highly increased (97 Newton) resulting in surface area decreasing.

Tartaric acid gives low bulk crushing strength with 2.5 weight% (16 Newton), and increasing the tartaric content up to 7.5 weight%, increases crushing strength up to 206 Newton. Samples with tartaric acid binder have higher bulk crushing strength than those obtained by using citric acid binder in the same percentages with no significant difference in the surface area. The good results of tartaric acid binder may be due to the hydroxyl groups in the organic binder. The organic binder with short chain (tartaric acid) adsorbed on the surface of particles and during the drying, water eliminated from hydroxyl group produces tridimensional hydrogen bonds (among the molecules of binder distributed on the surface of the particles). The development of the stronger tridimensional structure and mechanical strength improves proportionally to the amount of organic binder. Tartaric acid has two hydroxyl groups, while citric acid has only one. Citric acid has longer chain compared with tartaric acid, it may be during drying does not adsorbed at the surface of particles but they are able to form tridimensional hydrogen bonds^[24].

Samples with 10, 15, and 20 wt. % kaolin clay binder were prepared. Sample with 10 wt. % gives the lowest bulk crushing strength 81 Newton, while with 15 and 20 wt. % gives 123 and 181 Newton respectively. Increasing the bulk crushing strength for 15 and 20 wt. % was accompanied with a decreasing of the surface area from 356 to 284.6 m²/g. The addition

of a clay binder may reinforce the mechanical properties of the adsorbent, resulting in significant reduction in the adsorbent properties as mentioned by Pacaud, *et al.*^[18] and Shams & Mirmohammadi^[26]. The best result among the tested samples of zeolite with different percentages of binders was obtained with 15% kaolin clay and 7.5% tartaric acid. The using of 15% clay gives surface area 356m²/g and bulk crushing strength 123 N, while 7.5% tartaric acid binder gives lower surface area 267.4 m²/g and higher bulk crushing strength 206 N. This agreed with Inoue, *et al.*^[8]. Since the adsorption processes depends mainly on surface area and the bulk crushing strength 123N was acceptable, it could be desired that 15% kaolin clay binder is the choice. Furthermore kaolin is locally available and so cheap.

The 15% kaolin clay percentage is less than that chosen by Golovko, *et al.*^[27] and Ahmed^[28], they choose kaolin clay with 20-25weight% as the most suitable binder. This result agrees with the result obtained by Mahdi^[29] who choose 10-20% clay binder.

Adsorption Performance of the Prepared 5A Zeolite Granules

The prepared granules of 5A zeolite were used for the adsorption of H₂O, and H₂S contaminants from LPG. The inlet concentration of H₂O was 500 ppm, and H₂S was 209 ppm. Figures (4) to (6) show the effect of different flow rates (3, 4, 5 liter/minute) on the breakthrough curve of adsorbed H₂S and H₂O.

Figures (4) - (6) show that the adsorption of H₂O at any flow rates is stronger than the adsorption of H₂S. The breakpoint time for H₂O is higher than that for H₂S, for example, at 5 liter/minute, the break point time increases for H₂S and H₂O from 15 minute to 130 minute respectively.

This may be due to the higher affinity and smaller diameter of H₂O than H₂S and hence enhancing the adsorption of the H₂O^[30].

Since the H₂O has stronger adsorption affinity than H₂S, H₂O displaced H₂S from the surface of adsorbent and H₂S concentration increased in the effluent stream and became higher than the inlet gas concentration (i.e. $C/C_0 > 1$), see figure (4) and (5), this displacement of a relatively weakly adsorbed component by a more strongly adsorbed component is sometimes referred to as roll-up effects mentioned by Chi & Lee^[31]. Figures (4) and (6) also show that the breakpoint appears earlier by increasing the flow rate. This may be due to the reduction in thickness of the surface film which considers as a resistance for the mass transfer and consequently an increase the mass transfer rate and hence causes increasing of the adsorption rate as mentioned by Nasir^[32], furthermore Ali^[33] mentioned that increasing flow rate will ease the penetration and the passage of the adsorbate molecules through the particles.

Dynamic adsorption capacity of zeolite 5A is calculated from summation of accumulative adsorption after a given time by equation (3).

$$qi = \frac{C_0 - C_i}{M} * Q * t \quad \dots (3)$$

Where,

q_i = The quantity of the H₂O or H₂S adsorbed per unit mass of 5A zeolite adsorbent at any given time, grams adsorbate/gram adsorbent.

C_0 = Initial concentration of adsorbate (H₂S or H₂O), grams/liter.

C_i = Effluent concentration of adsorbate at a given time, grams/liter.

M = Mass of adsorbent, grams.

Q = Volumetric flow rate, liter/minute.

t = Time of sampling, minutes.

Figures (7) - (9) show the accumulative adsorbed of H₂S and H₂O verse time at different flow rates.

These figures indicate that the amount of H₂O adsorbed is higher than that of H₂S at any time and flow rate. The lower uptake of H₂S in zeolite can be explained by hindrance of the water molecules strongly adhered to the adsorbent surface; even an extremely dilute amount of water can substantially decrease the adsorption capacity for other gases on zeolites and lower their intracrystalline diffusivity^[34]. This result is agree with the work Chi & Lee^[31] and Kikkinides, *et al.*^[9]. The capacity of zeolite 5A for H₂O adsorption decreases with flow rate increasing from 20.61 to 7.54 g/g of zeolite because increasing in the flow rate decreases the contact time between the water vapor and adsorbent. This result agrees with Zangana^[35]. Because of the decreasing the adsorption capacity of H₂O the adsorption capacity of H₂S increases with flow rate increasing, for example, increasing the flow rate from 3 to 5 liter/minute increases the capacity of H₂S from - 2.69 to 1.76 g/g of zeolite^[36].

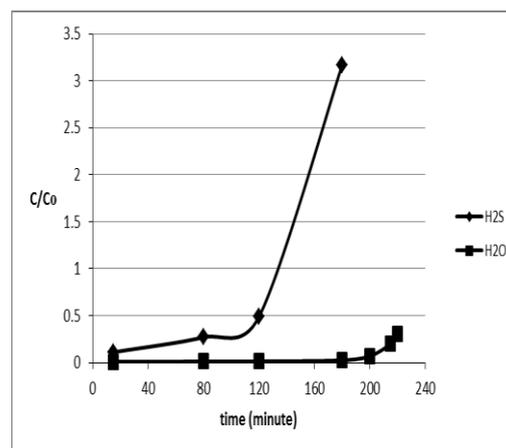


Fig. 4, Breakthrough curve for adsorption of H₂S and H₂O at 3 liter/minute

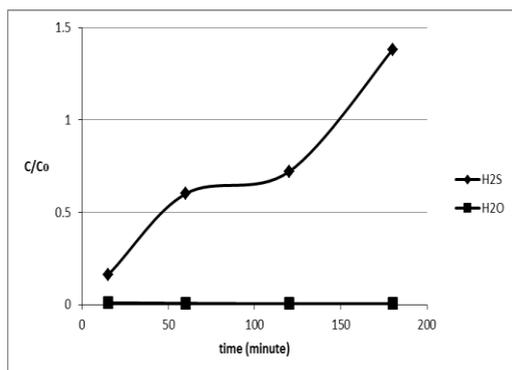


Fig. 5, Breakthrough curve for adsorption of H₂S and H₂O at 4 liter/minute

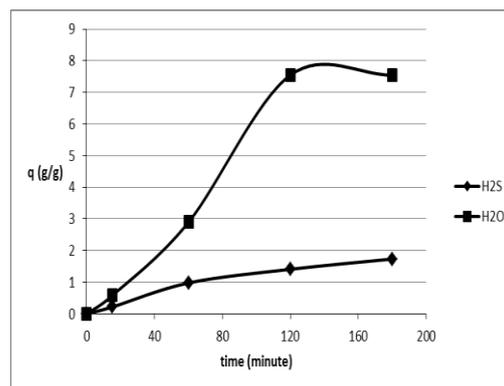


Fig. 9, The accumulative adsorbed of H₂S and H₂O verse time at flow rate 5 liter/minute

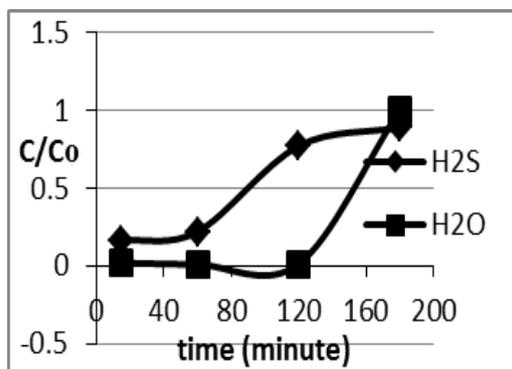


Fig. 6, Breakthrough curve for adsorption of H₂S and H₂O at 5 liter/minute

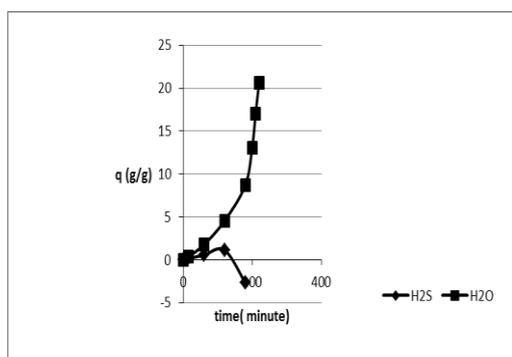


Fig. 7, The accumulative adsorbed of H₂S and H₂O verse time at flow rate 3 liter/minute

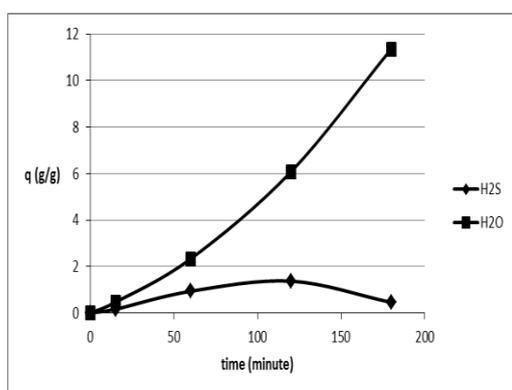


Fig. 8, The accumulative adsorbed of H₂S and H₂O verse time at flow rate 4 liter/minute

Conclusions

- 1- X-ray diffraction analyses show that temperatures 40 °C and 60 °C did not show any relative crystallinity of zeolite 4A. At all other temperatures with 1N NaOH concentration no crystallinity was observed. Temperature 90 °C with 2N NaOH concentration was selected to prepare zeolite 4A from kaolin.
- 2- The percentage of ion exchange of zeolite 4A with calcium chloride solution was 66.6%.
- 3- The formation experiments of 5A zeolite with organic binder showed that tartaric acid binder gave higher bulk crushing strength than that obtained by using citric acid binder in the same percentages with no significant difference in the surface area. 7.5% tartaric acid gives the higher bulk crushing strength 206 N with surface area 267.4m²/g.
- 4- The prepared zeolite with 15wt. % kaolin clay binder gave the higher surface area 356 m²/g, and bulk crushing strength 123 N.
- 5- The adsorption of H₂O and H₂S from LPG using the prepared granules of 5A zeolite showed that H₂O is the strongly adsorbed component compared with H₂S. This affinity toward H₂O affected on the breakpoint time for H₂O which appeared very late.

- 6- The capacity of the prepared zeolite 5A for adsorption of H₂S increases with flow rate increasing, while for H₂O the capacity decreases with flow rate increasing.

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