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Adsorptive Desulfurization of Iraqi Heavy Naphtha Using Different Metals over Nano Y Zeolite on Carbon Nanotube

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Abstract

The present research was conducted to reduce the sulfur content of Iraqi heavy naphtha by adsorption using different metals oxides over Y-Zeolite. The Y-Zeolite was synthesized by a sol-gel technique. The average size of zeolite was 92.39 nm, surface area 558 m^2/g, and pore volume 0.231 cm^3/g. The metals of nickel, zinc, and copper were dispersed by an impregnation method to prepare Ni/HY, Zn/HY, Cu/HY, and Ni + Zn /HY catalysts for desulfurization. The adsorptive desulfurization was carried out in a batch mode at different operating conditions such as mixing time (10,15,30,60, and 600 min) and catalyst dosage (0.2,0.4,0.6,0.8,1, and 1.2 g). The most of the sulfur compounds were removed at 10 min for all catalyst types. The maximum sulfur removal was 56% using (Ni+Zn)/HY catalyst at 1.2 g dose for 24 h. The adsorption kinetics and isotherm of sulfur removal were studied, and results indicated that desulfurization adsorption kinetic was 2nd order, and Temkin and Freundlich models were the best representation isotherm.

Keywords: Carbon nanotube, Nano Y zeolite, naphtha, adsorption desulfurization

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1- Introduction

Naphtha usually contain a small amount of sulfur compound. This sulfur causes many problems such as the catalyst poisoning and deactivation in processing, corrosion of equipment and the oxidation of sulfur compounds to SOx during fuel combustion. Sulfur exists in amounts from thousands parts per million to about close to hundred [1]. Environmental regulations have been introduced in many countries around the world to reduce the sulfur content of distillate fuels to ultra-low levels (10 ppm). The aim of this research is lowering the transportation fuel engine’s harmful exhaust emissions and improving air quality [2].

A new selective adsorption process for removing sulfur, the main objective of the process is to selectively adsorb sulfur from fuels, and non-sulfur containing hydrocarbons such as aromatic, olefin hydrocarbons and cyclic paraffinic hydrocarbons remain unchanged. The adsorbent containing transition metal compound supported on silica gel has been used. The adsorption experiments were accomplished under ambient temperature from room temperature to 250°C and atmospheric pressure in the adsorption column. A unique characteristic can be adsorbed without using hydrogen gas [3].

Adsorptive desulfurization processes are considered among the most economically attractive techniques due to their simple operating conditions, availability of inexpensive and the re-generable adsorbents such as reduced metals, metal oxides, alumina, metal sulfides, zeolites, silica, and activated carbon This process occurs as the sulfur molecules attach to the adsorbent and stay there separate from the fuel [4].The main part of any adsorption process is a porous solid medium as it offers high surface area or high micropore volume that is translated into high adsorption capacity

The difficulty in adsorbent development is that it must preferentially adsorb the sulfur-containing compounds over competing hydrocarbons, namely aromatics have a high adsorption capacity that it will have a high ratio of sulfur removed per gram of adsorbent be capable of regeneration such that the adsorbent can be reused. There are many types of adsorbents that have been heavily explored in literature, primarily: supported metals, metal oxides, activated carbons, ionic liquids, and metal loaded zeolites [4].

A classical definition of zeolite is a crystalline aluminosilicate with a three-dimensional framework structure that forms uniformly sized pores of molecular dimensions. Y-type zeolites are among the most widely used zeolites in catalysis, especially for the conversion of hydrocarbons, and this thanks to a structure with large pores allowing the adsorption of a large variety of molecules, but also a thermal stability a remarkable opportunity to perform multiple structural modifications according to the reaction conditions [5].

Nanocrystalline zeolite particles are becoming an important material in many technical applications.

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Synthetic methods that minimize the zeolite crystal diameter, while providing a narrow particle size distribution, are of primary importance in these technical applications [6].

Modern research lacks the techniques to synthesize designed properties of many nanomaterials also the need to interface these nanomaterials with microscale and macroscale platforms. The synthesis of Zeolite or silica-based material has shown a particular interest in the carbon nanotube (CNT), due to its enhanced physical properties and particle size [7].

The objective of this work is to reduce the sulfur content of Iraqi heavy naphtha by adsorption. Nano Y zeolite was prepared with a multi-wall carbon nanotube (MWCNT) and transformed into HY type. Various types of adsorbents were prepared by dispersion of different metals over the zeolite such as Ni/HY, Cu/HY and Zn/HY for the batch adsorption desulfurization which carried out at different operating conditions. The adsorption isotherm and kinetics of desulfurization were studied.

2- Experimental Work

2.1. Materials

Heavy naphtha 600 ppm sulfur content was supplied from Midland Refineries Company/Al-Dura Refinery. Sodium Aluminate was provided by Kunshan Yalong Trading Co., Ltd China. Sodium Aluminate has a molecular weight of 62, purity % =50-56(Al_2O_3), 40-45 (Na_2O). Sodium silicate (Na_2SiO_3) was provided by SIGMA Aldrich Chemical has a molecular weight of 122, purity % =10.63 Na_2O, 26.5 SiO_2. Sodium hydroxide (NaOH) was provided by SIGMA Aldrich has a molecular weight of 40, purity % =99.5. Ammonium chloride was provided by Merck. The Multiwall Carbon Nanotubes (MWCNT) used in the experiment was supplied by ZHENGZHOU DONGYAO MATERIALS Company with purity 97 % and the surface area 231.856 m^2/g.

2.2. Synthesis of Nano y zeolite

The sol-gel and hydrothermal method were applied for the synthesis of NaY zeolite and according to the following steps. The aging solution was prepared from 4.07 g sodium hydroxide pellets dissolved in 19.95 g of deionized water. 2.09 g of the sodium aluminate solution is stirred in a 100 ml plastic bottle until dissolution, then 33g of sodium silicate was added and aging for 24 h. the stock solution was 131 g of deionized water was added to 0.14 sodium hydroxide with 13.1 g sodium aluminate and 1 g MWCNT, the mixture was stirred then 206 g of sodium silicate was added and the mixture was mixed with 1600 rpm mixer for 20 min. The solutions prepared in the previous steps were mixed in a polypropylene bottle and subjected to homogenization for 24h at room temperature; the product was centrifuged for 15 min for MWCNT separation and then the MWCNT was transferred to jacketed stainless steel autoclaves.

For crystallization, the autoclave was made from stainless steel and lined with polytetrafluoroethylene (PTFE), the mixture was heated at 100 ° C for 24 hours without agitation. The product was then filtered, washed with distilled water until neutralization (pH = 7) and dried in an oven at 110 ° C for 24 h. Then, it was calcined at 550 C for 3 h.

2.3. Ion Exchange of Nay Zeolite

The prepared NaY zeolite was transformed into HY type zeolite using the ion exchange method.10 g of Y Zeolite was mixed with 42.79 g of ammonium chloride and 200ml distilled water which reflexed at 70 C for 2 h. The solution was left at room temperature overnight to complete the ion exchange process then the solution was filtered, washed and dried at 110 C overnight. The produced zeolite was calcined in a furnace at 525 C for 3 hours.

2.4. Preparation of Metal Oxide-Based Catalyst

Different types of metal oxide-based catalysts were prepared. An aqueous solution of different metal salt was prepared by dissolving 0.66g of Zn $(NO_3)_2$, 0.72g of Ni $(NO_3)_2$, $2H_2O$ and 0.68 g of Cu $(NO_3)_2$. $2H_2O$ in 10 ml distilled water. 5 g of HY zeolite was placed in the vacuum flask for 15 min to discharge air from zeolite pores, then an appropriate volume of metal solution was added. The impregnation was carried out at room temperature for 2h. The impregnated zeolite was dried at 110^oC overnight and calcination at 550^oC for 3h.

2.5. Adsorption Desulfurization Process

100 ml of heavy naphtha was poured in a 150 ml flask with an initial sulfur concentration 600 ppm. The batch adsorption desulfurization experiments were conducted at different operating conditions such as catalyst dose range (0.2-1.2 g) and mixing time (10 min to 10 h) for various catalyst types.

3- Results and Discussion

3.1. Catalyst Characterizations

The identification of the phases formed by X-ray diffraction shows that the zeolite type Y is pure and well crystallized with a better yield of crystals is obtained from a gel initial stoichiometric composition. The X-ray diffraction of the material is given in Fig. **1**.

This spectrum shows the characteristic diffraction peaks of a Na-Y zeolite, as are presented in the collection of simulated powder diagrams for zeolites [8].

All the peaks of diffraction were indexed in the cubic system. The mesh parameter determined from the structural refinement is a = 24.678 Å. XRD analyses were carried out at room temperature using CuKα radiation nickel filter (λ= 1.5418A) and energy condition of (4 kV and 3 mA).

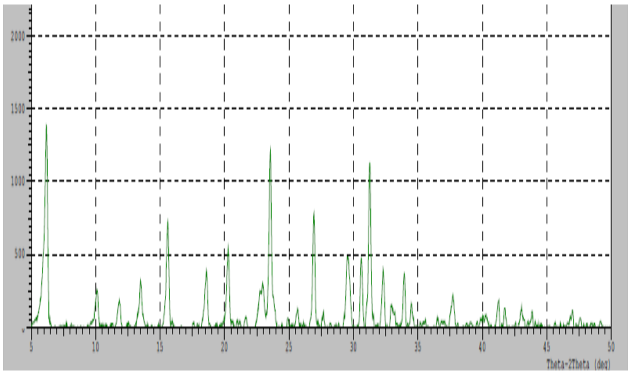

Fig. 1. XRD pattern for zeolite Y with the using of MWCNT

The AFM has been performed to the prepared zeolite with the use of MWCNT. The topography of the surface of the prepared zeolite was taken by the Atomic force microscope. These images show details about particle size distribution. AFM allowed a detailed observation of nanometer-size scale at crystal surfaces as shown in Fig. **2**

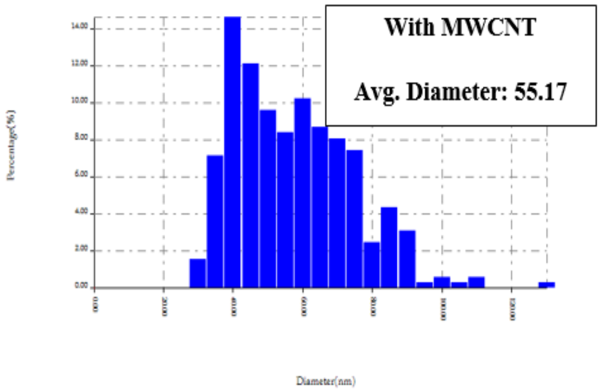

Fig. 2. The AFM analysis of NaY zeolite with MWCNT

The surface area and pore volume have been measured for prepared Y-Zeolite with MWCNT. The BET surface area of the zeolite was 558 m^2/g and the pore volume was 0.231 cm^3/g. located petroleum research and development center.

3.2. Adsorption Desulfurization of Naphtha

Sulfur removal from Iraqi heavy naphtha by adsorption desulfurization was carried out by using different types of metals oxides base zeolite catalysts. All experiments were conducted on a batch mode with various operating conditions like catalyst type, catalyst dose and mixing time.

a. Effect of Time on Sulfur Removal

The removal of sulfur from Iraqi heavy naphtha has been studied with the adsorption time using a different type of adsorbent. Fig. **3** shows the effect of adsorption time on the percentage of sulfur removal.

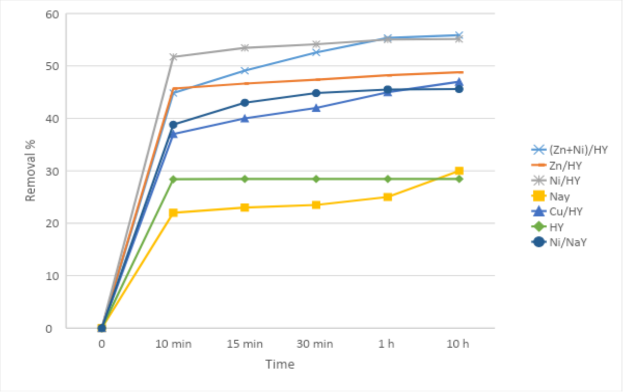

Fig. 3. Sulfur removal versus time for different metal oxide-based catalyst

It is clear that after 10 min a major sulfur removal was achieved for all adsorbent types. The sulfur removal as a function of time shows that the adsorption equilibrium was reached approximately 1 h. At 10 min the percentage of sulfur removal was 51.7% for Ni/HY catalyst, while the removal was 45.6% for Zn/HY, 45.2% for Zn+Ni /HY, and 37% for Cu/HY. The sulfur removal increased gradually with time, until 1h. The lowest sulfur removal % was achieved with NaY and HY pure Zeolite. In the case of HY, maximum removal was (28.4 %) at 10 min, and no change was detected after 10 h. Meanwhile, NaY showed the least sulfur removal among other types. At 10 min percentage was (22%) and slightly increased to (25%) at 1 h and (30 %) at 10 h. The best removal of sulfur achieved for the Ni/HY loading and Ni+Zn /HY, the sulfur removal in both of them after 1h was 55% and 55.3% respectively. As for the Zn/HY, after 10 hours the sulfur removal was 48.7%. After 10 h, all the catalyst types reach the maximum of their removal. The max removal of Ni+Zn /HY was 56.2% followed by Ni/HY were the removal was 55.1%.

b. Effect of Adsorbent Mass on Sulfur Removal

In order to optimize the mass of each catalyst type for the removal of sulfur from naphtha, a study was conducted with different adsorbent concentrations varied from 0.2 mg up to 1.2 mg in 100 ml solution.

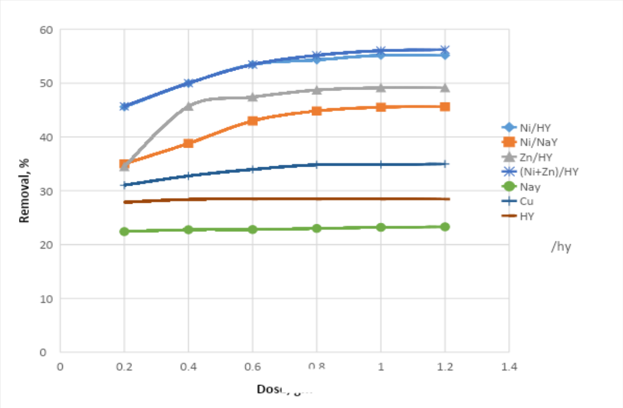

Fig. 4. Sulfur removal versus adsorbent dose for different metal oxide-based catalyst

Fig. **4**, shows the effect of catalyst dose on removal percentage. At 0.2 gm the percentage removal was 45.68 % for Ni/HY and Ni+Zn/HY, 34.48 % for Zn/HY, 34.5% for Ni/NaY, 31% for Cu/HY, 27.86 for HY and 22.4 for NaY. The sulfur removal increases with catalyst dose increase until it reaches 56% at 1.2 gm for Ni/HY and Ni+Zn/HY. For Cu/HY and Zn/NaY also the removal values were close at any dose, it starts with 31% removal at 0.2 g, 32.75 at 0.4 g, 33.96 for 0.6g and for 0.8, 1 and 1.2 the removal was 34%.

For pure HY and NaY alone without metal ions, the removal was the lowest due to the absences of the metal ion that can react with the sulfur ions in the sulfur compound [9]-[12]. The removal of both was constant 22.4% for NaY and 27.9% for HY.

For Zn+Ni /HY, the removal with dose 0.2g was 45.68% and at 0.4 the removal increase to 50% while at 0.6g the removal was 53.44%. At the rest of the doses, the removal didn’t change much.

The removal increase as the catalyst does increase at any catalyst type except for the NaY zeolite were the removal has no significant effect on NaY dose. The NaY zeolite base has lower performance than the HY base this is due to the presence of sodium ion in the catalyst the decrease its overall activity [8],[13]. At any catalyst dosing, the highest removal was achieved for Ni/HY and Ni+Zn /HY catalyst followed by Zn/HY then Ni/NaY while the lowest removal was for NaY zeolite.

c. Effect of Nickel Concentration on Sulfur Removal

The effect of Ni loading concentration on the percent of sulfur removal from Iraqi heavy naphtha has been studied as shown in Fig. **5**. It’s clear that the sulfur removal was increased with Ni concentration increase. The sulfur removal was 52% and 54% at 2% and at 3% respectively, while sulfur removal rise to 56% at 6%.

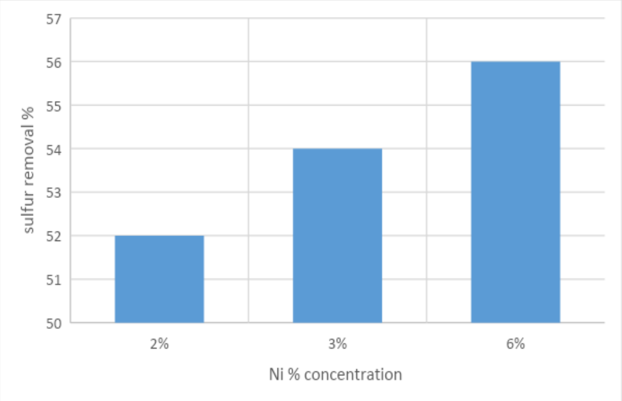

Fig. 5. Effect of NiO concentration over HY zeolite on sulfur removal

Results show that the Ni concentration increase has a slight effect on the percentage of sulfur removal. The Ni concentration increase from 2% to 6% led to increased sulfur removal 4% only. This result confers a favor to use low metal concentration on a carrier and it acceptable from an economic point of view.

3.3. Kinetic Study of Sulfur Adsorption

The kinetics of sulfur adsorption on Ni-based zeolite was analyzed to see the best-fitted adsorption order, the comparison between the experimental data and the predicted model are based on the values of the correlation coefficients (R^2) hence the R^2 value closer to the unit will indicate the correct model to correctly describe the kinetics.

For each temperature, the curve of ln (q_e -q_t) as a function of time t (Figure 6) is a straight line indicating that the adsorption kinetics is of the first order, from which the constant k_1, is estimated.

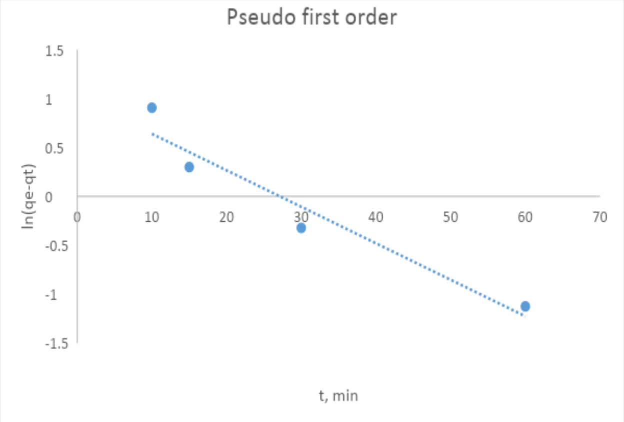

Fig. 6. Time vs ln (q_e-q_t) pseudo first order kinetic

In such circumstances, Figure 7 gives a linear relation, which allows the computation of q_e from the slope and k_2 to the ordinate.

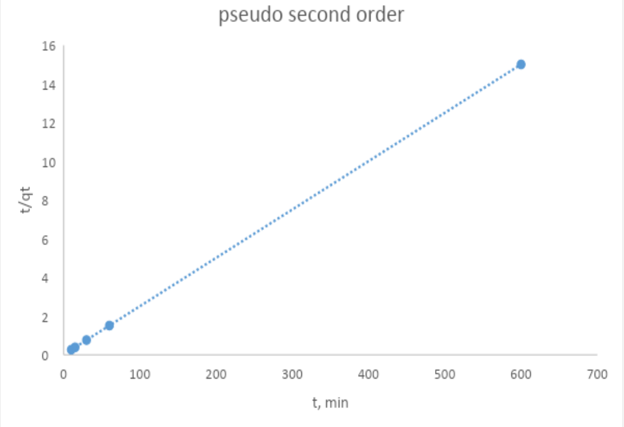

Fig. 7. Pseudo second-order relation

So the advantage of using this model lies in the fact that there is no need to know the capacity of equilibrium from the experiments since it can be determined from the model by trying both first and second-order, it's noticeable that the second-order is more accurate (R^2=1) than the first order R^2=0.9332.

3.4. Study of Adsorption Isotherms

The study of isotherms was carried out to try to understand the adsorption phenomenon. These isotherms have been established at a different catalyst. The batch process was carried in 100 mL of naphtha solution and the initial concentration of sulfur was 600 ppm. The mixture is kept shaken so that the sulfur spread evenly throughout the solution. The concentration of sulfur in solution is monitored as a function of time, for temperatures ° C.

a. Freundlich Model

Freundlich model, which gives an indication of the heterogeneity at the surface of the adsorbent, was applied to measure the adsorption capacity according to the following relation:

$$q_e = K_f . C_e^{\frac{1}{n}} \qquad (1)$$

Where: K_f = Freundlich isotherm constant (mg/g) n = adsorption intensity; C_e = the equilibrium concentration of adsorbate (mg/L) q_e = the amount of metal adsorbed per gram of the adsorbent at equilibrium (mg/g). The linear form of the Freundlich equation can be written in the form logarithmic according to the following relation:

$$log\ log\ q_e\ = log\ log\ Kf + \frac{1}{n} logC_e \qquad (2)$$

The experimental results obtained for isotherms given in Fig. **8**.

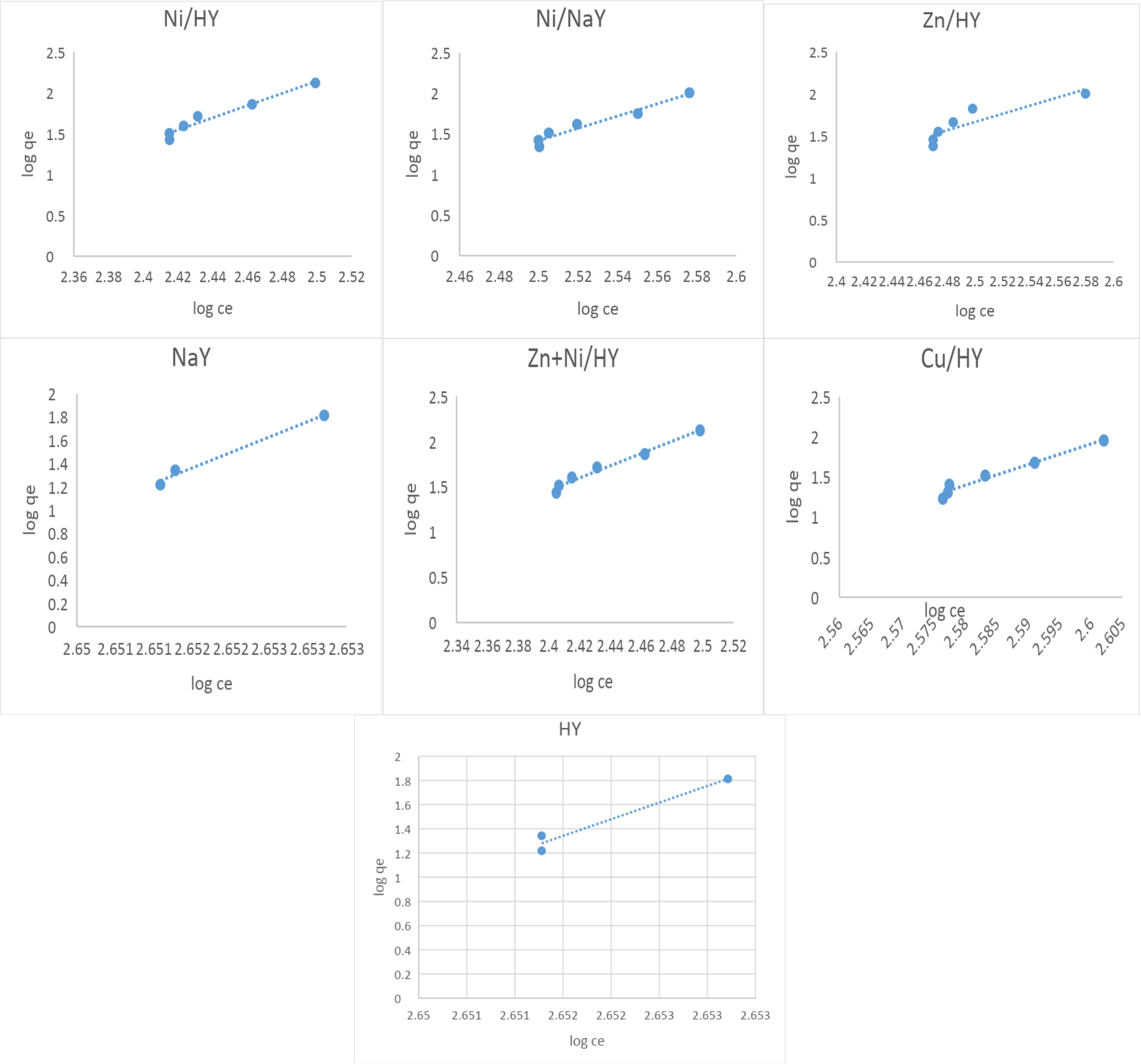

Fig. 8. Freundlich isotherm of adsorption process for all catalysts types

According to Figure 8, the three curves illustrating the isotherms show adsorption follows the Freundlich model, the experimental results can be correlated by the Freundlich equation and the correlation coefficients are close to unity. The Freundlich constants K and n were determined from the isotherms and their values are summarized in Appendix A. From table (1) the n value for Ni/HY, Ni/NaY, Ni+Zn/HY, Zn/HY lies between 1 and 10 means this model is favorable for these types of catalyst. While for Cu/HY, HY and NaY n value is greater than 10 means it's not favorable.

b. Langmuir isotherm

The Langmuir model allows determining if a monolayer is adsorbed and if there is no interaction between the adsorbed molecules. The Langmuir equation is valid for only one monolayer adsorbed with a well-defined number of adsorption sites uniform and identical energetically according to the following relation:

$$q_e = \frac{Q_0.K_L.c_e}{(1+K_L.c_e)} \qquad (3)$$

Where: C_e = the equilibrium concentration of adsorbate (mg/L^{-1}), q_e = the amount of metal adsorbed per gram of the adsorbent at equilibrium (mg/g). Q_o = maximum monolayer coverage capacity (mg/g) K_L = Langmuir isotherm constant (L/mg). The Langmuir equation can be written also in this form:

$$\frac{1}{q_e} = \frac{1}{Q_0} + \frac{1}{Q_0.K_L.C_e} \qquad (4)$$

By representing the experimental results, for the three temperatures studied, according to the Langmuir equation gives the isotherms of Fig. **9**.

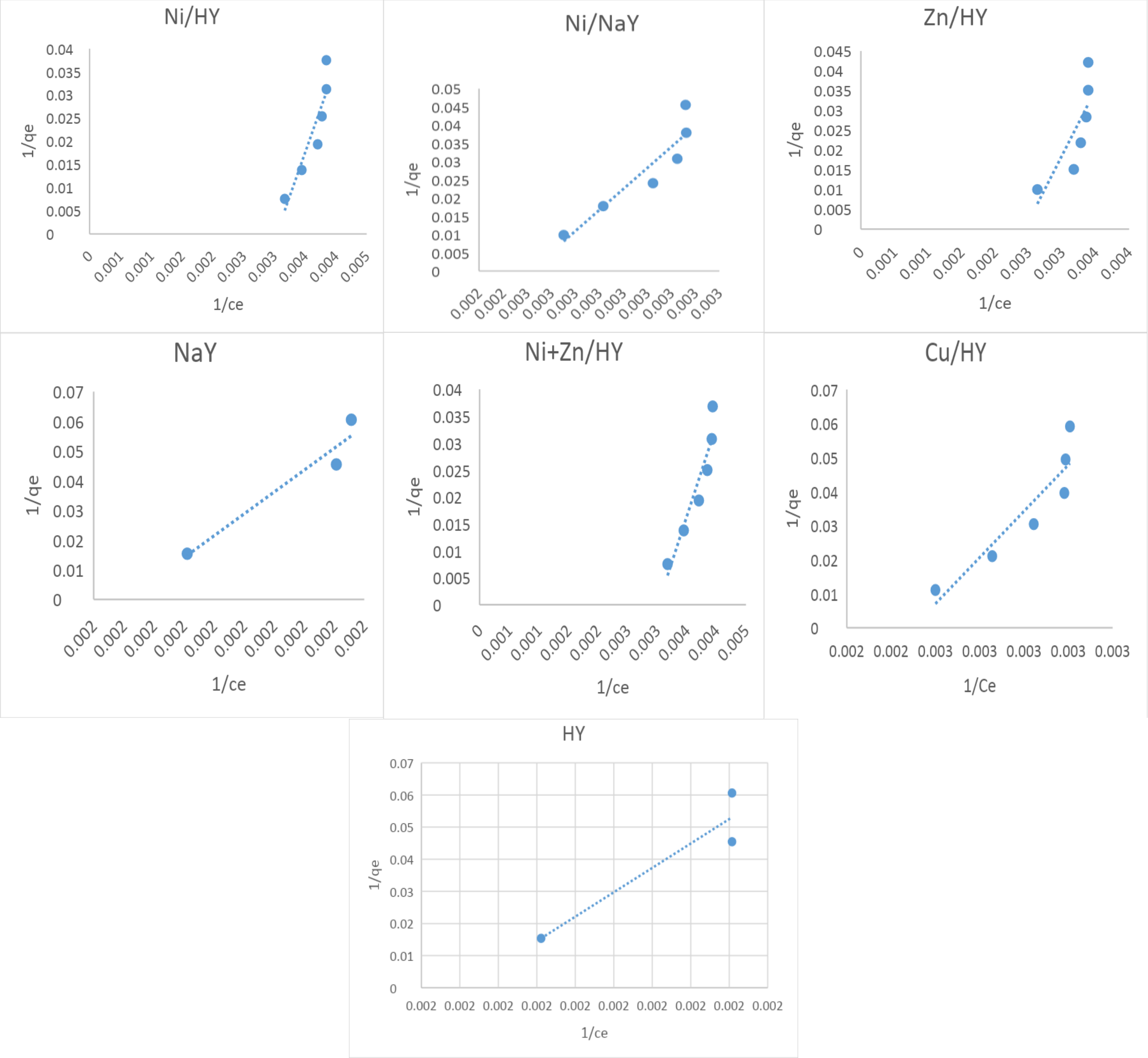

Fig. 9. Langmuir isotherm of adsorption process for all catalysts types

K_L: constant related to adsorption energy (Langmuir model). A value of R_L less than unity represents favorable adsorption and a value greater than unity represents unfavorable adsorption. The R_L shown in Table **1** was greater than 1 for all types for all catalysts which means that the model is also unfavorable.

c. Isotherm of Temkin

The Temkin isotherm assumes that the decrease in adsorption heat is linear and that adsorption is characterized by a uniform distribution of binding energies. The Temkin isotherm was used in the following form [9,10]

$$q_e = \frac{RT}{b} \ln(A_T C_e) \tag{5}$$

$$q_e = \frac{RT}{b} \ln A_T + \left(\frac{RT}{b}\right) \ln C_e \tag{6}$$

$$B = \frac{RT}{b} \tag{7}$$

$$q_e = B \ln A_T + B \ln C_e \tag{8}$$

A_T =Temkin isotherm equilibrium binding constant (L/g), b = Temkin isotherm constant, R= universal gas constant (8.314 J/mol/K), T= Temperature, B = Constant related to heat of sorption (J/mol).

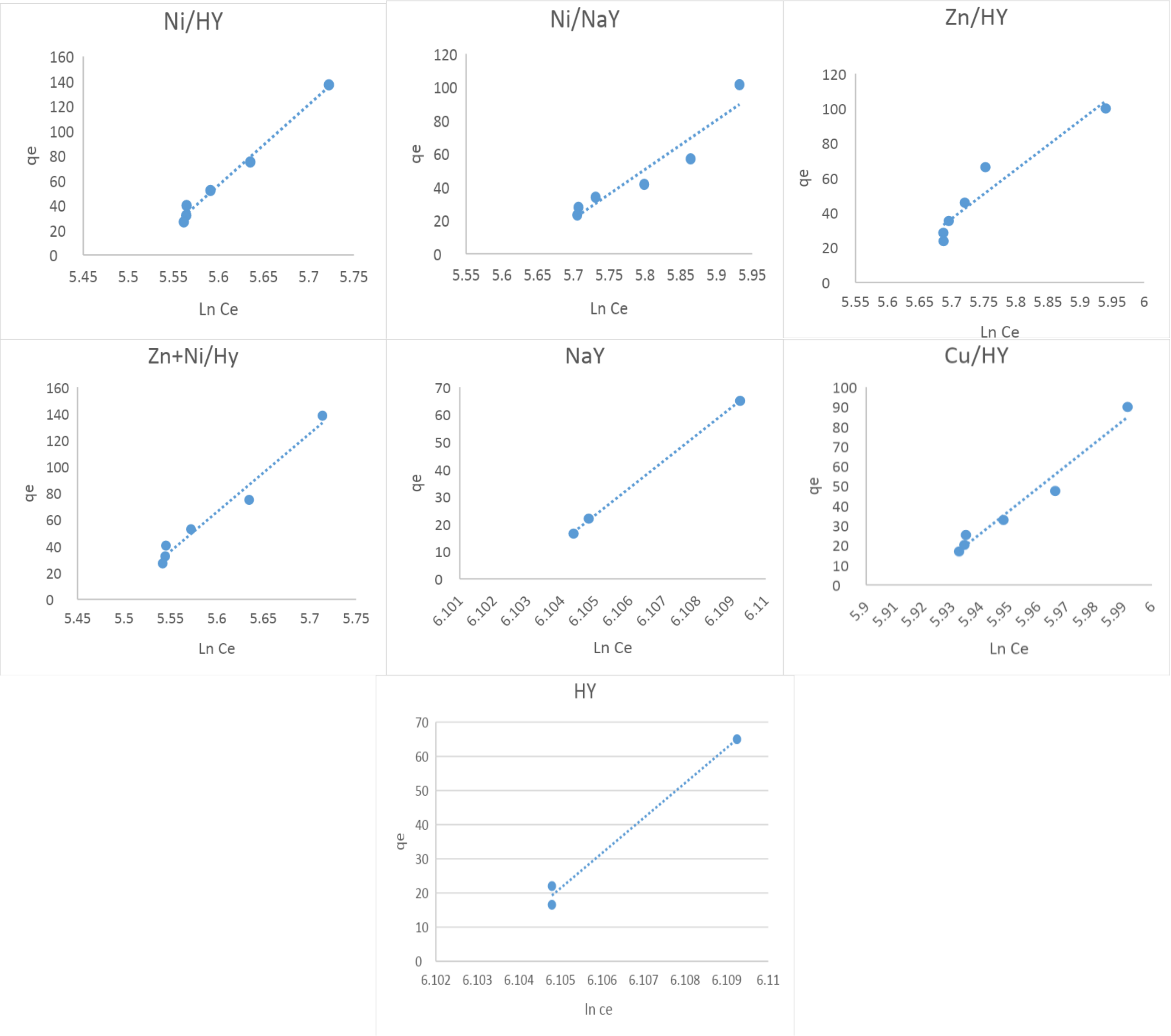

Fig. 10. Temkin isotherm of adsorption process for all catalysts types

The curve of q_e versus Ln Ce (Figure 10) allows the determination of the isothermal constants of B and A from the slope and interception, respectively. The experimental results can be correlated by the Temkin equation and the correlation coefficients are close to unity.

The Temkin constants A_T and b were determined from the isotherms and their values are summarized in Appendix A. All the isotherm result has been summarized in **Table 1**.

Table 1. The isotherm parameters

Isotherm	Parameters	Ni/HY	Ni/NaY	Zn/HY	Ni +Zn/HY	HY	Nay	Cu/HY
Langmuir	q_m	-8.733	-6.89	-12.34	-9.823	-0.123	-0.122	-1.486
	K_L	-3.04E-3	-2.5E-3	-2.44E-3	-3.01E-3	-2.43E-3	-2.22E-3	-2.47E-3
	R^2	0.8404	0.8595	0.6399	0.8939	0.9608	0.9382	0.8461
	R_L	4.22	1.81	1.70	3.91	1.69	1.40	1.76
Freundlich	k_f	2.9E-17	3.9E-18	2.8E-11	1.9E-15	0	0	5.30E-29
	n	7.47	7.5303	4.89	6.7373	263.155	264.76	25.776
	R^2	0.955	0.9505	0.7836	0.977	0.8917	0.9864	0.9641
Temkin	B	649.74	295.75	281.42	591.75	9681.2	9794.8	1145.3
	A_T	4.03E-3	3.59E-3	3.812E-3	4.13E-3	2.88E-3	2.24E-3	2.69E-3
	R^2	0.9897	0.9026	0.9141	0.9723	0.9894	0.9996	0.9655

4- Conclusions

The phase pattern by XRD indicated to Y-zeolite catalyst, in spite of using MWCNT for the crystallization step, which referred to the success of using MWCNT for crystal growth. The Y-zeolite was prepared with a narrow range particle size (55.17) nm by using MWCNT as a medium for crystal growth compared with the conventional method. Most of the sulfur removal was achieved after 10min for all types of catalysts 51.7% for Ni/HY. The sulfur removal increased with time, after 10h become slightly increased. Ni concentration increase doesn't highly effect on a percent of sulfur removal, increasing concentration from 2% to 6% increase sulfur removal just 2% low metal concentration is acceptable from an economic point of view. Experimental results have been applied by the models of Freundlich, Langmuir, Temkin. It has also is that the Freundlich and Temkin model better describes the adsorption of sulfur than the Langmuir model. The study of the kinetics of adsorption of sulfur made it possible to specify the order of the reaction. Indeed, the two applied kinetic models, a model of 1st, and 2nd order model. By comparing the coefficients of regression of the curves corresponding to the two kinetic models, the 2nd order is closest to the unit. So we can say that the kinetics of the reaction Sulfur adsorption is most likely of second order.

Nomenclature

AFM :	Atomic force Microscopy
BET :	Brunauar , Emmett, Teller
SEM :	Scanning Electron Microscopy
MWCNT :	Multi wall carbon Nano tube
XRD :	X-ray Diffraction

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امتزاز الكبريت من النافثا الثقيلة العراقية باستخدام معادن مختلفة فوق زيوليت نانو Y

حسام موسى جمعه و حسين قاسم حسين

قسم الهندسة الكيمياوية ، كلية الهندسة ، جامعة بغداد

الخلاصة

في البحث الحالي الذي اجريته لدراسه تاثير الامتزاز الانتقائي لازالة مركبات الكبريت في النافثا العراقيه الثقيله الذي يتم انتاجها من مصفى الدورة . وتمت عمليه الازالة باستخدام Y−Zeolite بتقنية sol−gel وتحميله بأكسيد فلزي مختلف. تم الكشف عن نمط الطور والبنية الكيميائية وحجم الجسيمات ومساحة السطح بواسطة XRD و AFM على التوالي. أظهرت النتائج أن متوسط حجم الزيولايت وكان (92.39) نانومتر وحجم الجسيمات (92.39) نانومتر .

تم تحسين المساحة السطحية لتكون (558) م 2 / جم مع حجم مسامي كبير قليلاً (0.231) كم 3 / غ تم الحصول على أنواع مختلفة وتركيزات من المحفزات القائمة على أكاسيد الفلزات مثل Ni / HY و Zn / HY و Cu / HY بواسطة طريقة التشريب لامتصاص الكبريت من النافثا الثقيلة العراقية. تم ازالة الكبريت بعملية دفعية في ظروف تشغيل مختلفة مثل وقت الخلط (10،15،30،60 و 600 دقيقة) وكمية العامل المساعد (0.2،0.4،0.6،0.8،1 و 1.2 g). تم إزالة مركبات الكبريت بعد (10 دقائق) لجميع أنواع العامل المساعد. كان الحد الأقصى لإزالة الكبريت 56 ٪ باستخدام العامل المساعد HY / (Ni + Zn) في جرعة g1.2 خلال hr24. جعلت دراسة الحركية الامتزازية لإزالة الكبريت من الممكن تحديد الترتيب الحركي. في الواقع ، معاملات الانحدار من الدرجة الثانية هي الأقرب إلى الوحدة. لذلك يمكننا القول أن حركية تفاعل امتصاص الكبريت هي في الغالب من الدرجة الثانية. تظهر الدراسة أن النماذج Temkin و Freundlich كانت أفضل لتمثيل النتيجة التجريبية isotherm.

الكلمات الدالة: الأنبوب النانوي الكربوني، زيوليت Yنانو ، النافثا ، إزالة الكبريت بالامتزاز