



Zinc Oxide Hydrogen Sulfide Removal Catalyst/ Preparation, Activity Test and Kinetic Study

Karim H. Hassan*, Zuhair A-A Khammas**
and Ameel. M. Rahman***

Department of Chemistry, College of Science, University of Diyala, Baquba, Iraq,
E-Mails: * drkarim53@yahoo.com ** dr_zuhair52@yahoo.com
***Department of Biochemical Engineering, Al-Khwarizmi College of Engineering,
University of Baghdad, Jadiriya, Baghdad, Iraq,
E-Mails: explorerxp50@yahoo.com

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Abstract

Hydrogen sulfide removal catalyst was prepared chemically by precipitation of zinc bicarbonate at a controlled pH. The physical and chemical catalyst characterization properties were investigated. The catalyst was tested for its activity in adsorption of H₂S using a plant that generates the H₂S from naphtha hydrodesulphurization and a unit for the adsorption of H₂S. The results comparison between the prepared and commercial catalysts revealed that the chemical method can be used to prepare the catalyst with a very good activity.

It has observed that the hydrogen sulfide removal over zinc oxide catalyst follows first order reaction kinetics with activation energy of 19.26 kJ/mole and enthalpy and entropy of activation of 14.49 kJ/mole and -220.41 J/mole respectively.

Keywords: ZnO, H₂S, Absorbent; Reformate; COS, Hydrodesulfurization; Kinetics.

1. Introduction

The natural gas can be considered as the main raw material for fertilizer and petrochemical industries, in addition of being the main source of gas fuel. Hydrogen sulfide is poison material with unpleasant odor and corrosive in addition of being poison for most industrial catalysts [1]. Unfortunately, hydrogen sulfide is present in an appropriate amount in natural gas that affect the efficiency of catalysts used in fertilizer production. Accordingly it must be removed it from the feedstock.

Several chemical methods have been employed for hydrogen sulfide removal such as phosphate method, iron oxide method, the hydroxide method, activated carbon adsorption method, molecular sieve method and the zinc oxide method [2]. Of these ZnO catalyst method proved to be the most effective for sulfur compounds removal and H₂S in particular. From the standpoint of high H₂S removal efficiency, zinc oxide is

more attractive than iron oxide because of more favorable sulfidation thermodynamics [3].

Zinc oxide catalyst is used as a mixture of ZnO and alumina as a binder in addition to some fillers or binder materials. In general 90 wt % of ZnO is quiet acceptable.

The function of the catalyst depends on the chemical reaction between the catalyst and hydrogen sulfide to form zinc sulfide:



The quantity of hydrogen sulfide being adsorbed by zinc oxide depends on its value in the feed and on the degree of contact between it and the ZnO bed.

Many methods have been used for preparation of ZnO catalysts, but the most common were allocated between the thermal and chemical treatments. The thermal method involves the combustion of zinc metal with air in which ZnO

with surface area of $10 \text{ m}^2/\text{gm}$ was obtained [4]. Khammas et al. [5] have prepared zinc oxide catalyst from zinc oxide by-products purified by thermal methods treatments and the pure 95% ZnO was slurried with alumina and water and formulated to extrudates shape, dried at $130\text{-}150^\circ\text{C}$ and calcined at 700°C for two hours. More than 90% H_2S adsorption was achieved.

The chemical or wet method that is used to prepare catalysts by precipitation method is summarized by precipitating the zinc from its salts and then drying and calcining the precipitates at $300\text{-}350^\circ\text{C}$ to form ZnO [6].

Other important works on the ZnO catalysts was done by Susan et al [2]. They prepared a regenerable zinc oxide-titanium dioxide sorbents for high temperature H_2S removal from fuel gases.

In addition to ZnO catalyst, other sulfur compounds removal catalysts were studied by several investigators. Jiang et al. [7] studied the γ -alumina supported Na_2CO_3 , K_2CO_3 and CuO for flue gas desulphurization where regenerable adsorbents can recover sulfur as more valuable products such as sulfuric acid. They prepare all the adsorbents based on the principle of spontaneous monolayer dispersion of oxides or salts on high-surface-area supports and characterize them with XRD, BET, and XPS. They find an excellent correlation between the maximum adsorption capacity of SO_2 and the monolayer loading of Na_2CO_3 , K_2CO_3 , or CuO on γ -alumina.

Vohs and Halevi [8] developed a mechanistic studies of the interaction of SO_2 , H_2S and organo sulfur compounds, such as thiols and disulfides with metal oxide surfaces and makes use of TPD (Temperature Programmed Desorption) of probe molecules to measure reaction kinetics and energetic and surface sensitive spectroscopic probes such as XPS (X-Ray Photoelectron Spectroscopy) and HREELS (High Resolution Electron Energy Loss Spectroscopy) to identify stable surface intermediates.

The apparent kinetics [9] of H_2S removal by a ZnO-MnO desulfurizer were studied by thermogravimetric analysis. The experimental results show that the reaction is first order with respect to H_2S concentration. In the temperature range $200\text{-}400^\circ\text{C}$, the rate was controlled, at lower temperatures, by the grain surface reaction rate and, at higher temperatures, by the rate of intrapellet diffusion, respectively. The apparent kinetic behavior could be modeled by the equivalent grain model. The activation energies of surface reaction and solid diffusion were determined to be 11.842 and 20.865 kJ/mol,

respectively. An optimum reaction temperature was observed. Reasons for this and why the solid diffusion activation energy exceeded that of the surface reaction are proposed.

The aim of the present work is to prepare ZnO catalyst by a combined chemical treatment and test its activity and performance for the hydrogen sulfide removal from a natural gas.

2. Experimental

Materials

Chemicals:

Sodium bicarbonate NaHCO_3 solution of 1M, aluminum oxide Al_2O_3 , (it is possible to use aluminum hydroxide or aluminum nitrate instead), iodine, zinc metal, sodium thiosulphate, barium chloride, starch indicator and hydrochloric acid, all of which were of analytical grade (BDH).

Zinc nitrate $\text{Zn}(\text{NO}_3)_2 \cdot 4 \text{H}_2\text{O}$, since it is very dehydrated, it was prepared experimentally by dissolving zinc metal in dilute nitric acid.

Gases:

Nitrogen, compressed air, hydrogen was supplied from local gas factory supplier.

CoMo catalyst: Commercial cobalt molybdenum sulfur removal catalyst CoMo type (3E-124 HD).

Naphtha:

Heavy Naphtha that is produced in Daura refinery in Baghdad and certified to contain 0.07 % sulfur.

3. Catalyst Preparation

Since most of the ZnO catalysts consist of more than 90% of ZnO, we choose to prepare the one with 90 wt% ZnO and 10% of alumina to avoid problems associated with tableting and hardness. The required amount of zinc nitrate that give 90 wt % of ZnO was dissolved in water and then 10% alumina was mixed together and heated to $60\text{-}70^\circ\text{C}$ with stirring and then sodium bicarbonate precipitating agent is added gradually with stirring and monitoring the pH of the solution during the addition until it reaches a value of 7. The precipitate is left to settle for two hours and then filtered off and washed with distilled water for several times to remove any excess bicarbonate that may present.

The paste formulated as extrudates of $4 \times 5 \text{ mm}$ diameter in small laboratory extruder and then dried in an oven at 110°C for 12 hours and then cooled to room temperature and finally calcined in

a furnace at 450°C for two hours, the temperature of which was raised gradually at a rate of 5°C /minute.

4. Chemical Analysis and Characterization

ZnS was determined by treating the spent catalyst with dilute hydrochloric acid and the liberated hydrogen sulfide is then reacted with standard iodine solution [10-11]. The excess iodine was back-titrated with standard sodium thiosulphate in the presence of starch as indicator. For H₂S determination a measured volume of the gas containing the H₂S was passed into a solution of cadmium sulfate where yellow precipitate CdS is formed [10-11]. The precipitate thus formed is treated with dilute hydrochloric acid to liberate the hydrogen sulfide which is then analyzed as mention above.

The metal content of the catalysts were determined by standard atomic absorption method. Pore volumes and densities were determined by liquid impregnation method [12]. Hardness was determined with the ERWEKA.TBH28 hardness meter [13] while crushing value was determined by placing 25 gm of the sample under pressure of 18kN for 20 minutes and measure the amount of powder that pass through 53 micrometer mesh as a percent relative to the total weight.

5. Laboratory Catalyst Testing Unit:

Activities of the catalysts for adsorption of H₂S were determined by using a plant that consists of two units, the first one for generation of the

hydrogen sulfide by the hydrodesulphurization (HDS) of naphtha that contain 0.07 wt % of sulfur using a commercial cobalt molybdenum catalyst (CoMo), and the second one for the adsorption of the gas on ZnO catalysts. The first unit is a continuous flow plant (Fig.1). The reactor was made of stainless steel (316-heat resistant), length 800 mm, inside diameter, 19 mm. Heaters were in the form of four separately heat-controlled block shells. The reactor was packed with 110 ml of the CoMo catalyst between two layers of inert porcelain. Test conditions were: reactor temperature 612°K; pressure, 25bar; flow rate of naphtha, 0.08 liter/hour; flow rate of the gas produced, 18 liter/hour.

In a typical run the reactor was purged with N₂ gas and the temperature rose. After establishing steady state conditions, the reaction was started; the naphtha hydrogen mixture was pumped upwards into the reactor. The products were passed through a high pressure separator and condenser to separate the liquid desulfurized naphtha from the gas containing H₂S which leave the unit and enter the adsorption unit.

The second unit used for the adsorption of H₂S includes a quartz tube reactor of 17.5mm diameter and 350mm length placed in a heat controlled tubular furnace. The gas containing H₂S is passed from one side of the reactor and leave from the other side of it after being passed over catalyst bed. Test conditions were: reactor temperature 553-613°K ; pressure is atmospheric; flow rate of the gas produced were between 12-18 liter/hour which gives space velocity ranging between 800-1200 hr⁻¹ as 15ml catalyst volume was used.

Analysis of the gaseous products and the catalyst was carried out periodically whatever it is necessary.

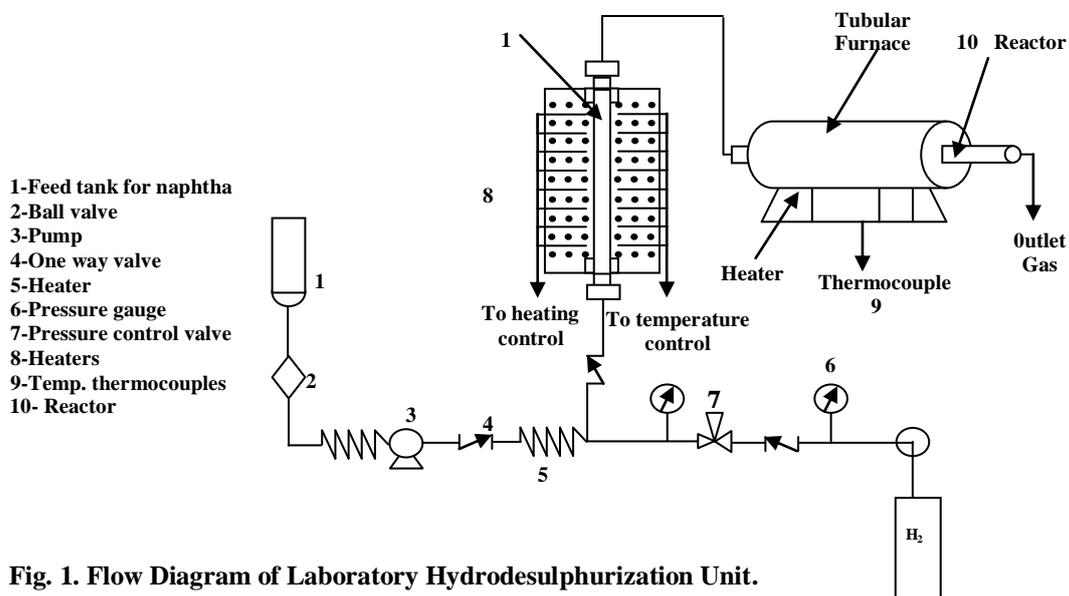


Fig. 1. Flow Diagram of Laboratory Hydrodesulphurization Unit.

6. Results and Discussion

The physical and chemical properties of the prepared ZnO catalyst given in Table (1) illustrates that they are quiet similar to the properties of the commercial catalyst. There is a slight difference in the values of hardness and crushing values which may be due to the purity of the material used in the preparation [14-15] and the extruder that is used in the formulation. The values of densities are quiet consistent with the expected differences between them, a slight yellow color was observed in the prepared catalysts resulted from purity of the raw material used and improper time of calcinations.

The capability of the catalyst for adsorption of the H₂S was observed by passing the gas containing H₂S on its surface as explained in the experimental section.

The flow rate of naphtha was 1cm³/min i.e, 60 cm³/ hour which is equivalent to 41.4 gm/hour considering the density of naphtha to be 0.6900 gm/cm³. On supposing 100 % sulfur removal from the naphtha that contains 0.07 wt% of sulfur means that the rate of sulfur passing over the ZnO bed is about 0.029 gm S/ hour or 0.030 gm H₂S / hour. By considering the flow rate of the gas leaving the HDS unit to be 0.3 liter/min or 18 liter/hour resulted that the gas leaving the HDS unit should contain 0.0017gm H₂S /liter.

Practically the amount of H₂S adsorbed was calculated by two means, the first one from the gas analysis for H₂S and the second one from the catalyst analysis after adsorption. The chemical analysis and calculations of the amount of H₂S in the gas leaving the HDS unit indicated that the value is 0.0016 gm H₂S/liter or 0.0288 gm H₂S/hour, this is the amount that is really passed over the catalyst ZnO bed at the previously described conditions.

The theoretical amount of H₂S that should be adsorbed by the catalyst was calculated by considering that we used a catalyst bed of 15 gm of 90% ZnO i.e the net is 13.5 gm of ZnO. Since 1 mole of ZnO (81gm) adsorbed 1 mole of H₂S (34gm) means that 13.5 gm must adsorb theoretically 5.66 gm of H₂S which means that the catalyst can operate for 196 hours before saturation is observed.

Passing the gas over the catalyst bed of 15 gm was continued for a period of 150 hours with continuous monitoring of the existence of H₂S in the exit gas by a solution of cadmium sulfate which shows no indication about its existence at all as the yellow precipitate of CdS did not form.

For 150 hours operation on the catalyst with rate of H₂S passing on it of 0.0288 gm / hour as calculated above, the mount of H₂S adsorbed on the catalyst should be 4.32 gm. Chemical analysis of the catalyst used after this period of time for sulfur revealed that the catalyst weight is 17.03 gm and contains 12.32 gm of ZnS, 3.21 gm of unreacted ZnO and 1.50 gm of alumina. This indicates that it consists of 79 wt% of ZnS and that the catalyst adsorbed 4.32 gm of H₂S during its operation, a value that is equal to the theoretical one and mean that catalyst is operated with more than 99% efficiency during this period of time and it is still active and can be used for further time as expected theoretically and experimentally from the left unreacted and unchanged ZnO after 150 hours of performance.

In industrial application the story is different from the theoretical one, as for example the catalyst of the Iraqi North Fertilizer Company unit was replaced by a new one even it contains only 75 wt% of ZnS to a void problems associated from sudden saturation that results from unexpected increase in the dose of H₂S. This is usually depends on the conditions of its use and the dose of H₂S that pass over and according to the origin and source of the gas which its compositions is sometime fluctuated and cause a problem.

Table 1
Properties of the Prepared and Commercial ZnO Catalysts.

Properties	Prepared Catalyst	Commercial Catalyst Two
Form	Extrudates	Extrudates
Color	White to yellow	White
Size (mm)	4 x 5	4-5 x 5-7
Packing density, gm/cm ³	1.22	1.18
Particle density, gm/cm ³	2.22	---
Skeletal density, gm/cm ³	3.23	---
Porosity	0.31	0.28
Pore volume, cm ³ /gm	0.25	0.23
Hardness, N	8	10
Zn, %	72	72.2
ZnO, %	89.7	90
Al ₂ O ₃ , %	10.3	10
Crushing value, % wt	9.5	8.5

7. Kinetic of the Hydrogen Sulfide Removal Over ZnO Catalysts

The experimental data were fitted (16-21) to a simple power-law rate equation (2) for first order:

$$\ln [H_2S_{in} / H_2S_{out}] = k / LHSV \quad \dots(2)$$

where H_2S_{in} is the concentration of hydrogen sulfide, gm/l in the inlet gas to the reactor and H_2S_{out} is the concentration of the hydrogen sulfide in the outlet gas from the reactor, k is the rate constant, with the unit of hr^{-1} and LHSV is the liquid hourly space velocity expressed as the ratio of the flow of feed to the volume of catalyst with the unit of hr^{-1} . and equation (3) for second order :

$$[1/H_2S_{out} - 1/H_2S_{in}] = k / LHSV \quad \dots(3)$$

With k , the second order rate constant expressed in $gm^{-1} l hr^{-1}$.

Tables (2-3) show that the concentration of H_2S in the gas increased with increasing the flow rate and the LHSV and this is due to the decrease in the contact time between the catalyst particles and the reacting gas, whereas Table (4) indicated that the temperature increasing resulted to a reasonable decreasing in the concentration of the H_2S and this is a usual phenomenon as the reaction is enhanced at high temperatures. These relations were shown in figures (2-4).

Table 2
The Effect of Flow Rate on the H_2S Content, gm/l in the Gas Leaving the Adsorption Unit at Different Temperatures with H_2S_{in} as 0.15 gm/l.

Flow rate, ml/min	553K	573K	593K	613K
0.25	0.0335	0.0253	0.0205	0.0154
0.375	0.0552	0.0455	0.0395	0.0326
0.500	0.0709	0.0615	0.0555	0.0481
0.625	0.0825	0.0735	0.0677	0.0603

Table 3
The Effect of LHSV on the H_2S Content at Different Temperatures with H_2S_{in} as 0.15 gm/l and Catalyst Volume of 15 ml..

LHSV, hr^{-1}	553K	573K	593K	613K
1	0.0335	0.0253	0.0205	0.0154
1.5	0.0552	0.0455	0.0395	0.0326
2	0.0709	0.0615	0.0555	0.0481
2.5	0.0825	0.0735	0.0677	0.0603

Table 4
The Effect of Temperature on the H_2S Content at Different LHSV with H_2S_{in} as 0.15 gm/l.

Temperature, K	1 hr^{-1}	1.5 hr^{-1}	2 hr^{-1}	2.5 hr^{-1}
553	0.0335	0.0552	0.0709	0.0825
573	0.0253	0.0455	0.0615	0.0735
593	0.0205	0.0395	0.0555	0.0677
613	0.0154	0.0326	0.0481	0.0603

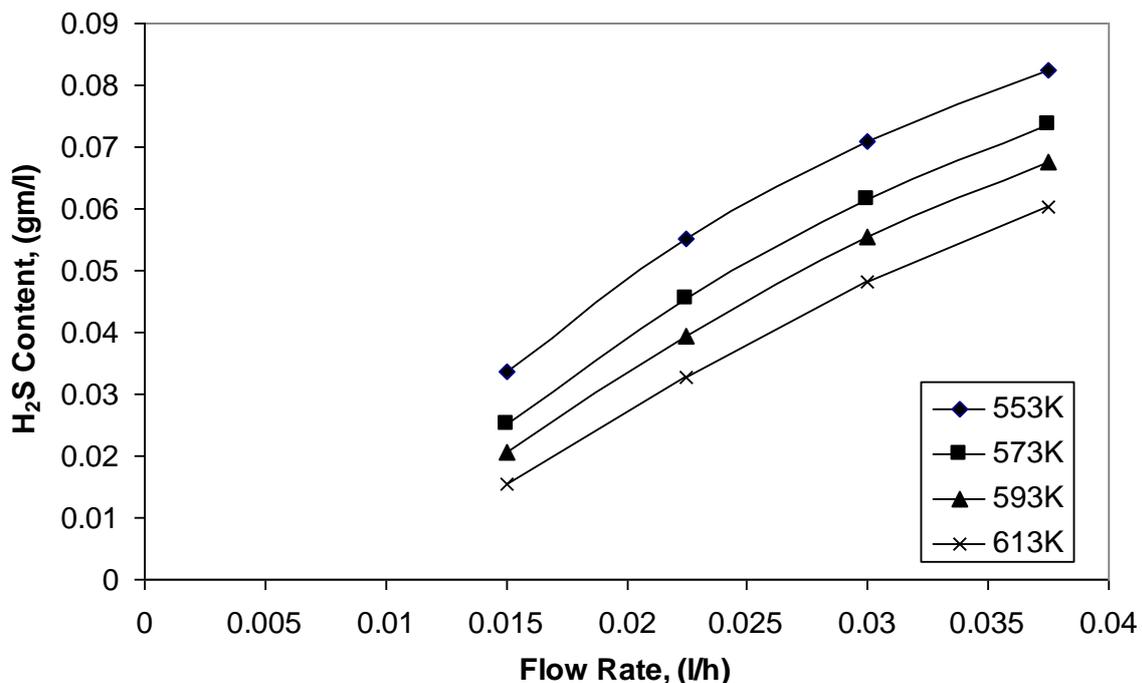


Fig. 2. The Effect of Flow Rate on The H_2S Content at Different Temperatures.

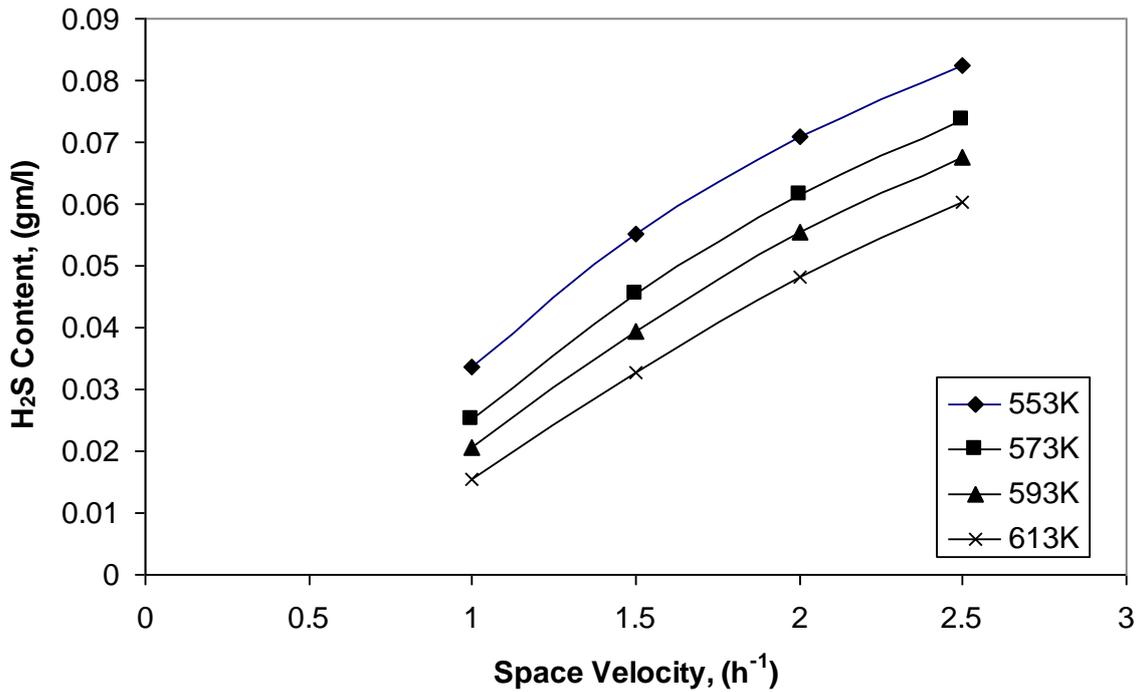


Fig. 3. The Effect of Space Velocity on The H₂S Content at Different Temperatures.

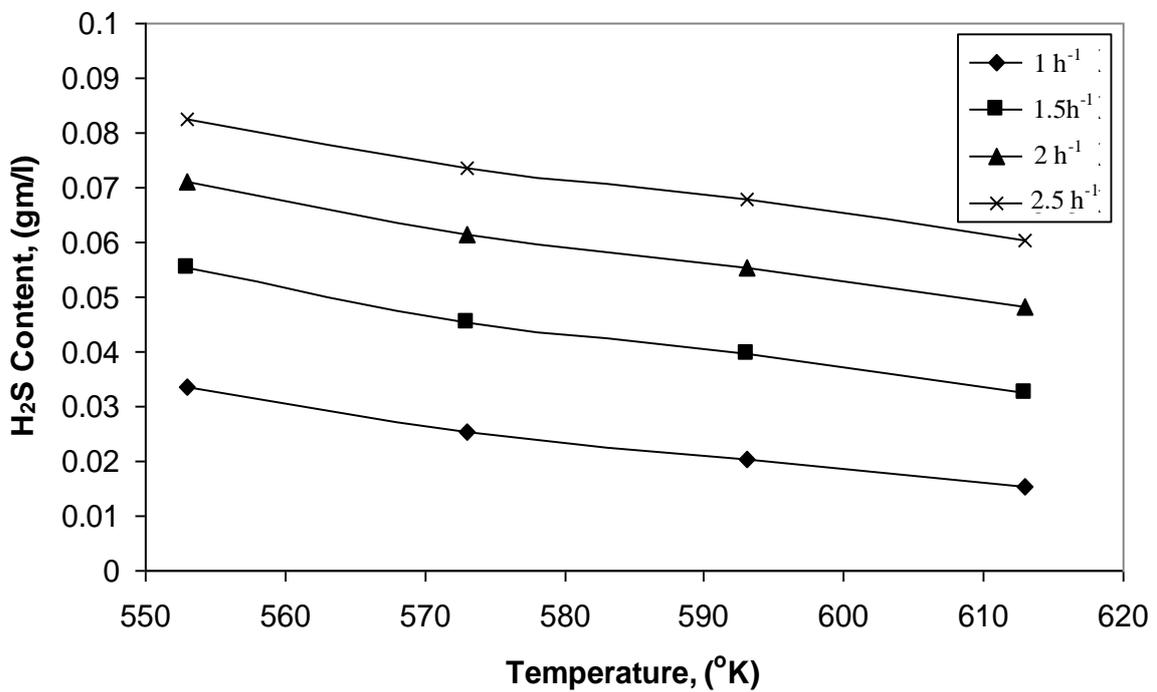


Fig. 4. The Effect of Temperature on the H₂S Content at Different Space Velocities.

To test the order of the reaction and hence calculate the thermodynamic parameters, the data in Tables (5) and (6) extracted from Tables (2-4)

were fitted in equations (2) and (3) to give the figures (5) and (6) respectively.

Table 5
The Data of $\ln (H_2S_{in}/H_2S_{out})$ vs $1/LHSV$ at Different Temperatures with H_2S_{in} as 0.15 gm/l.

1/LHSV, hr	553K	573K	593K	613K
1	1.499	1.781	1.989	2.277
0.67	0.999	1.193	1.333	1.525
0.5	0.749	0.891	0.994	1.139
0.4	0.598	0.712	0.796	0.911

Table 6
The Data of $[1/H_2S_{in}-1/H_2S_{out}]$ vs $1/LHSV$ at Different Temperatures.

1/LHSV, hr	613K	593K	573K	553K
1	23.184	32.859	42.114	58.268
0.67	11.449	15.311	18.649	24.008
0.5	7.438	9.593	11.351	14.123
0.4	5.454	6.939	8.104	9.917

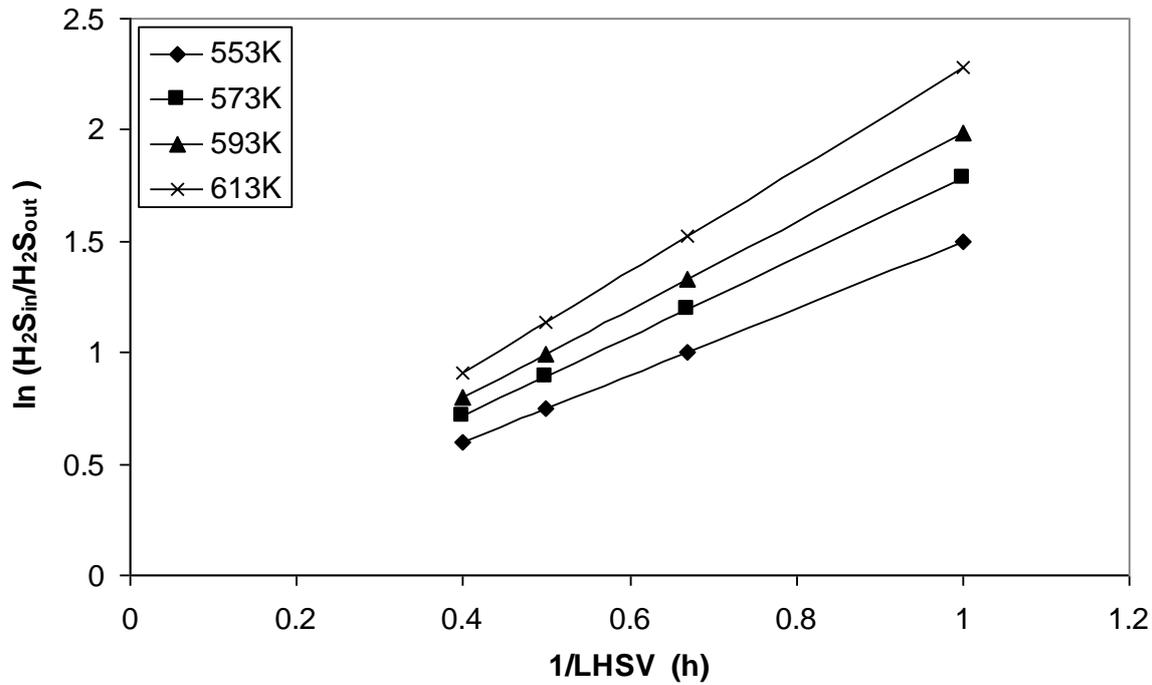


Fig. 5. Plot of $\ln (H_2S_{in}/H_2S_{out})$ vs $1/LHSV$.

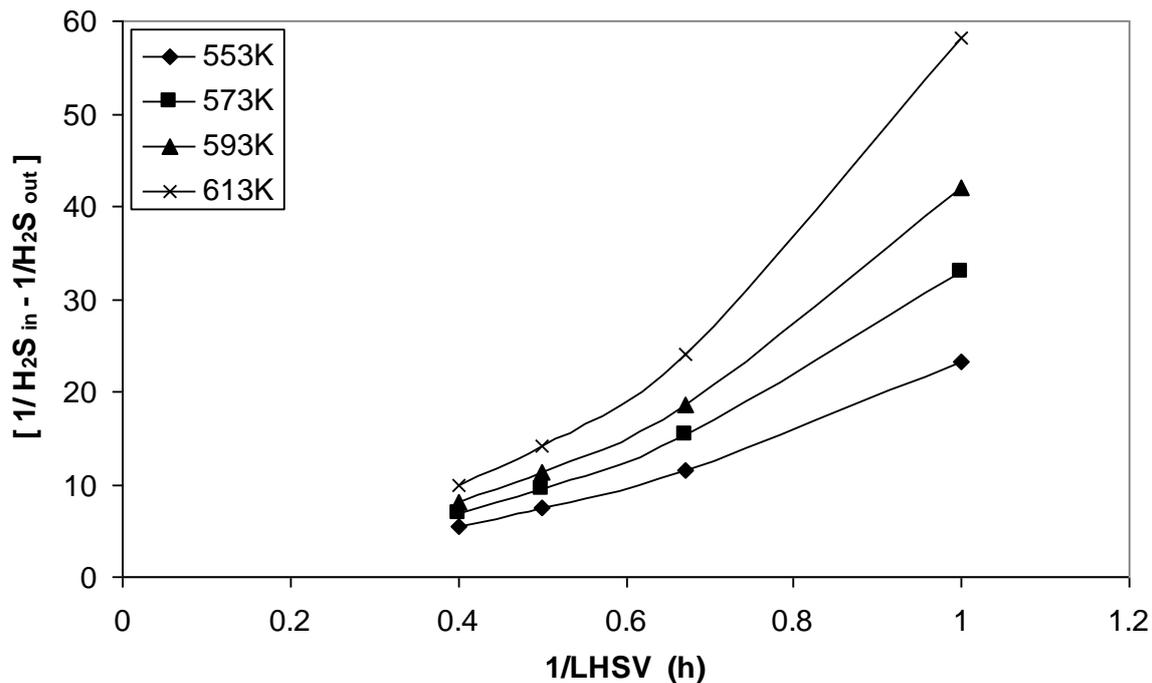


Fig. 6. Plot of $[1/H_2S_{in} - 1/H_2S_{out}]$ vs $1/LHSV$.

It is clear that figure (5) shows four straight lines that pass through the origin which is consistent with equation (2) while figure (6) show the otherwise as the four curves does not reflect or expressed equation (3) which suppose to give a straight line also. These results are well agree with those of Yanxu et.al for H₂S removal over ZnO-MnO bed (9) which proposed the reaction to follow solid diffusion mechanism and those of Kim et.al. for kinetic evaluation of H₂S biofiltration (22).

The slopes of the lines in figure (5) represent the rate constants values of 3216.342, 3307.672, 3374.491 and 3456.459 hr⁻¹ at temperatures of 553, 573, 593, and 613K respectively and is given in Table (7).

Table 7
The Effect of Temperature on the First Order Rate Constant, k Obtained from Equation (1).

Temperature, K	1/T	k,	lnk	ln(k/T)
553	0.00181	1.499	0.405	-5.911
573	0.00174	1.781	0.577	-5.774
593	0.00169	1.989	0.688	-5.697
613	0.00163	2.277	0.823	-5.595

The Arrhenius equation that satisfies the relationship between the rate constant and the reaction temperature

$$K=A_0\exp(-E_a/RT) \quad \dots(4)$$

with the E_a, the activation energy of the reaction, R is gas constant and A₀ is the pre-exponential factor.

So, by plotting the ln k values against the 1/T as shown in figure (7), the slope is (-E_a/R) from which the activation energy was calculated to be 19.26 kJ/mole.

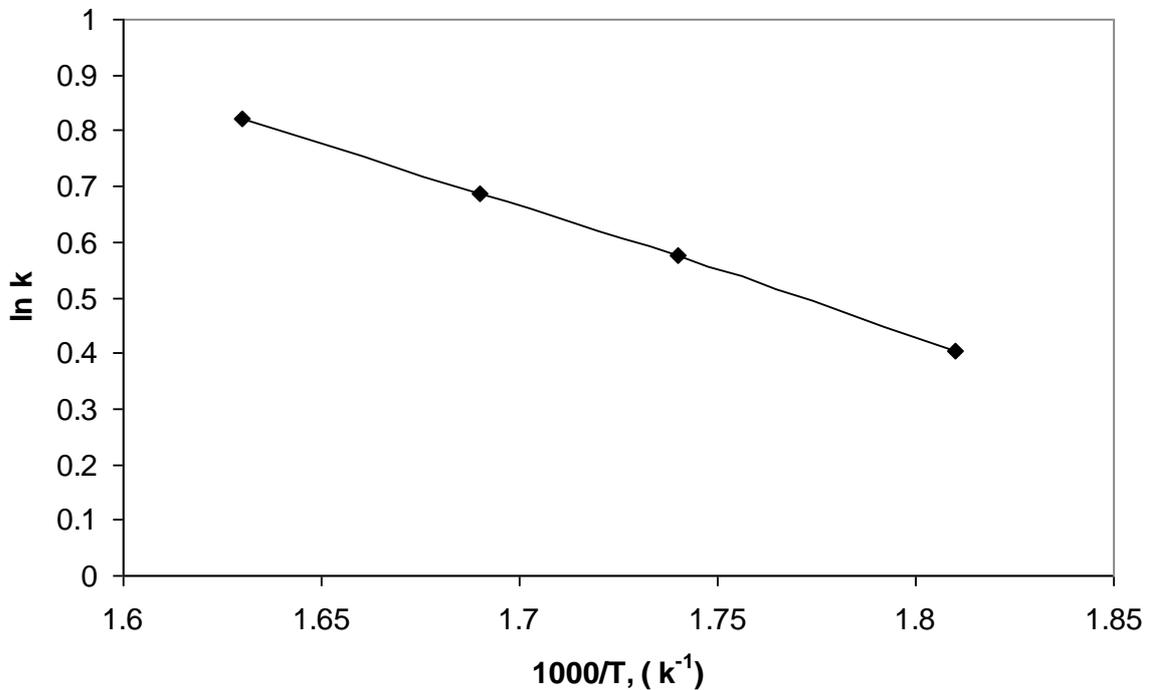


Fig. 7. Plot of lnk vs 1/T.

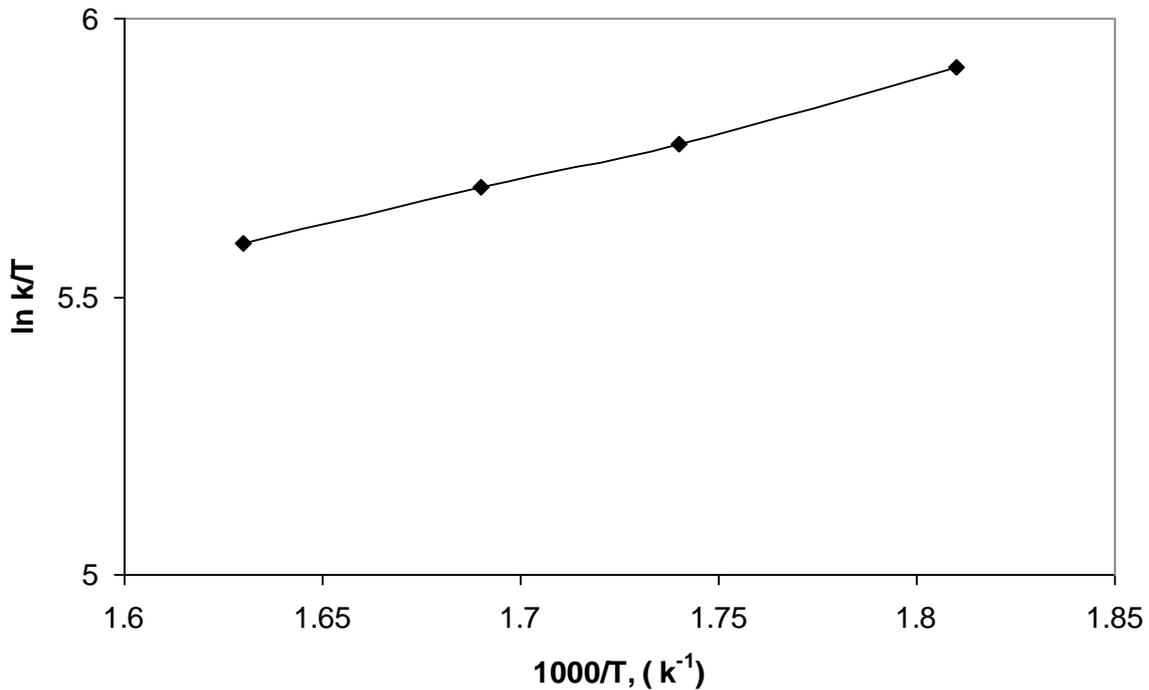


Fig. 8. Plot of $\ln(k/T)$ vs $1/T$.

8. Thermodynamics of the Hydrogen Sulfide Removal Over ZnO Catalysts

To calculate the enthalpy ΔH and entropy ΔS of activation the following equation which was obtained from the absolute rate theory was used.

$$k/T = k_T \cdot (F/h) \cdot \exp(\Delta S/R) \exp(-\Delta H/RT) \quad \dots(5)$$

where k_T is the transmission coefficient and is taken to be equal to 1, h is the Planck constant and F is the Boltzmann constant and R is the universal gas constant.

So a plot of $\ln(k/T)$ vs $1/T$ was a straight line and shown in figure (8) which gives a slope of $-\Delta H/R$ from which activation enthalpy was calculated to be 14.49 kJ/mole. The intercept of this line which is equal to $[(k_T \cdot F)/h] + [(\Delta S/R)]$ was used to calculate activation entropy ΔS value of -220.41 J/mole.K.

Conclusions

The chemical method for the combined preparation of pure ZnO and the catalyst was revealed that it is very simple, controllable, and not need sophisticated equipments compared to the thermal method which is solely limited for the preparation of pure ZnO.

Catalyst characterizations indicated very good similarities between the prepared and commercial catalyst and this was reflected upon the performance of the catalysts when tested in a laboratory performance unit and indicated that the catalyst can be operated with more than 99% efficiency.

Kinetic study indicated that the hydrogen sulfide removal over zinc oxide catalyst follows first order reaction with activation energy of be 19.26 kJ/mole and enthalpy and entropy of activation of 14.49 kJ/mole and -220.41 J/mole.K respectively.

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تحضير العامل المساعد اوكسيد الزنك المستخدم لازالة كبريتيد الهيدروجين، فحص الفعالية ودراسة الحركية

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* قسم الكيمياء / كلية العلوم / جامعة ديالى

** قسم هندسة الكيمياء الاحيائية / كلية هندسة الخوارزمي / جامعة بغداد

الخلاصة

جرى تحضير العامل المساعد اوكسيد الخارصين المستخدم في ازالة كبريتيد الهيدروجين من الغاز الطبيعي كيميائيا بطريقة الترسيب لبيكربونات الخارصين عند الدالة الحامضية 7 . تم إجراء اختبار الخواص الفيزيائية والكيميائية لهذه المادة . كما جرى فحص فعالية العامل المساعد لامتصاص كبريتيد الهيدروجين باستعمال منظومة ريادية تتكون من وحدتين . الأولى، لتوليد غاز كبريتيد الهيدروجين من ازالة الكبريت بالهيدروجين للنفثا . أما الثانية، لامتصاص كبريتيد الهيدروجين من قبل العامل المساعد المحضر . أشارت مقارنة الفحص بين العامل المساعد المحضر والتجاري إلى إمكانية الاعتماد على الطريقة الكيميائية كتدبير لتحضير العامل المساعد وبكفاءة عالية جدا . أظهرت دراسة حركية التفاعل أنه يتبع حركية المرتبة الأولى بطاقة تنشيط مقدارها 19.26 كيلوجول/مول وأن أنثالبية وأنتروبية التنشيط للتفاعل هي 14.49 كيلوجول/مول و -220.41 كيلوجول/مول . درجة كلفن وعلى التوالي .