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# Laboratory investigation of major parameters of sweetening process

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## ABSTRACT

Gas sweetening is a basic process in the gas refinery complexes. Undoubtedly, mixing of nano, sweetening and gas fuel can make revolution in the separation industries. Nano catalysts are adapted in this paper to vanish

 $H_2S$  as the toxic, corrosive and pyrophoric contaminant. The important feature which is considered is to improve the adsorption efficiency of hydrogen sulphide from hydrocarbon fuels such as methane gas by applying the Al2O3 as nano catalyst. Totally, the optimum conditions to eliminate the hydrogen sulphide from methane gas are evaluated in this paper, experimentally. In this paper, Al2O3 nano particles are synthesized and are contacted with flow of sour methane. The synthesized nano particles are char-

acterized by SEM and TEM. The process performance of  $H_2S$  removal from methane gas on Al2O3 nano particles is illustrated by the ratio of outlet concentration per feed concentration. The effects of operating con-

ditions such as operating temperature and pressure, the amount of  $H_2S$ 

concentration in feed stream, size of nano catalyst and the bed diameter are

investigated in this paper. This work studies the adsorption of  $H_2S$  from

natural gas with an emphasis on the influence of the operating and geometric parameters on process efficiency.

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#### **INTRODUCTION**

Desulphurization of gas is an important process used in a gas refinery to reduce the sulphur concentration and production of fuel products such as gasoline, jet fuel, kerosene, diesel and heating gas and oil<sup>[1,2]</sup>. So, the resulting fuels meet environmental protection standards<sup>[3]</sup>.

The challenge of fulfilling the world's growing transportation energy needs is no longer a simple issue of producing enough liquid hydrocarbon fuels<sup>[4,5]</sup>. This chal-

## **KEYWORDS**

Methane; Sour; Nano; Catalyst; Efficiency.

lenge is instead accentuated by a complex inter play of environmental and operational issues<sup>[6]</sup>. Environmental issues include societal demands that liquid hydrocarbon fuels be clean and less polluting. The emergence of new refining processes and the increasing use of new forms of energy production, e.g., fuel cells, exemplify operational issues. Together, these trends are driving the need for deep desulphurization of diesel and jet fuels. Nanotechnology (sometimes shortened to "nanotech") is the manipulation of matter on an atomic and molecular scale. The earliest, widespread descrip-

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tion of nanotechnology has been referred to the particular technological goal of precisely manipulating atoms and molecules for fabrication of macro scale products, also now is referred to as molecular nanotechnology. A more generalized description of nanotechnology was subsequently established by the National Nanotechnology Initiative, which defines nanotechnology as the study and application of fine particles which are sized from 1 to 100 nanometres in all of the science fields.

# MATERIALS AND METHODS

Al2O3 nano particle is a common ingredient and has a huge variety of applications. Zinc is an essential mineral and is non-toxic in low concentration.

## Synthesis method of nano-sized Al2O3

The Al2O3 nanopowder in this work was obtained via precipitation method using AlCl3·6H2O (Merck), Al powder (M.A. University), HCl (Merck) and NH4OH (Merck). The Al powder has a spherical shape with an average diameter about 37.5 µm. Aluminum chloride hexahydrate was first dissolved in aqueous HCl. The Al powder was then gradually added to the solution. The ratio of the raw materials was chosen under the conditions that were found to be the optimum in the work of Shojaie-Bahaabad. According to their work, the molar ratios of Al/AlCl3 and HCl/H2O were 1.81 and 0.18, respectively. The precursor solution was then continuously stirred at 100°C for 4 h to completely dissolve the starting materials. NH4OH was added to solution and the pH value of the solution was adjusted at 9. The obtained precipitate was washed using distilled water to remove the anion impurities, and finally dried at 80°C for 48 h. The obtained dried precipitate was ground into powder, and then the powder was calcined at different temperatures. For more comparison, one specimen was prepared via sol-gel method. The crystalline structure of the samples was determined by Xray diffraction (XRD), using Philips X-pert model with Cu K-α radiation. Differential thermal analyses (DTA) and thermo gravimetry (TG) were used with a rate of 10 °C/min with the STA 1460 equipment. The powder morphology was investigated using a Phillips XL30 scanning electron microscope (SEM). Fourier transformation infrared spectroscopy analysis (FTIR) was carried out in a Nicolet Nexus 6700 for studying the chemical groups of the dried precipitate and calcined powder. The obtained produced substance has light yellow colour, and can been characterized by SEM and TEM. Produced spherical particles with the average diameter of 40 -60*nm* in size are observed approximately and finally the crystal is pure Al2O3 with hexahedral structure. Figure 1 shows TEM and SEM photos of produced nano particles a) in the scale of 50*nm* and b) in the scale of 500*nm*.



Figure 1-a) : The TEM picture.



Figure 1-b) : The SEM picture.

## Set up description

One laboratory cylindrical vessel equipped with the nano-sized Al2O3 catalytic fixed bed is applied for  $H_2S$  adsorption process, in this work. The process

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Figure 1-c) : The SEM picture.

temperature is adjusted by one steam jacket around the vessel. Methane stream from a tank reservoir is mixed by  $H_2S$  and is fed into the bed containing Al2O3 nano particles. The inside diameter is adjusted between 2 to 6 cm and the height of the vessel is chosen 10 to 50 cm, respectively. All the instruments and equipments are made of stainless steel. Figure 2 shows briefly the mentioned adsorption experimental setup constructed to remove hydrogen sulphide from methane gas by using Al2O3 nano catalyst.

Methane gas flow rate is controlled by the flow meter and adjusted by valve after passing a filter, then is mixed by the adjusted amount of hydrogen sulphide and compressed to the reactor. The bed height of catalyst can be verified by some separate smaller metallic beds which are located in the vessel. Measuring the hydrogen sulphide concentration in the feed and the discharge flow, defines the performance of the process.

### **RESULTS AND DISCUSSION**

Anyone knows, the hydrogen sulphide is corrosive and toxic, severely. Meanwhile, this component is in several industrial. We know the current technologies use huge resources of energy for removing the hydrogen sulphide component. Therefore, the researchers try to enhance the performance of sweetening process. So, in this paper the Al2O3 are applied as nano catalysts for removal of  $H_2S$ . This metal oxide is not expensive comparing with the other metal oxides. So, several experiments are designed to evaluate the performance of sweetening process in this paper, operationally and economically. These experiments were tested to determine operational conditions that would optimize the amount of H2S removed from gas in order to gas sweetening.

Some major parameters are considered experimentally in the gas sweetening process by nano particles. The effects of operating conditions, properties of catalytic bed and Al2O3 catalyst are investigated on the process performance. The ratio of  $H_2S$  concentration in the product stream on the initial concentration in the input stream ( $C/C_0$ ) represents the process performance. The purpose of the experiments is to decrease the amount of hydrogen sulphide below the 4 ppm in the outlet stream. Experimental results are presented in the following Figures.

#### The effect of operating temperature and pressure



Figure 2 : The schematic of process.













Both, temperature and pressure are two important parameters in separation processes. Five  $30 \circ_{\rm C}$ ,  $40 \circ_{\rm C}$ , 50 °C, 60 °C and 70 °C are examined during different pressures and the amount of  $C/C_{a}$  is measured. So, to find the best operating temperature, the operating pressure is changed from 3atm to 9atm using 40 and 60 nm in diameter catalyst. The feed contains 100ppm H,S with  $1.67 \,\mu m^3 / s$  flow rate and the reactor has a bed with 50cm height. Figure 3 and 4 shows that the 3atm operating pressure is not effective even at 70 °C temperature, since the amounts of  $C/C_0$  are higher than 0.04. Also, the amount of  $C/C_0$  reaches below the 0.04 just when the range of operating temperature is adjusted between 30 °C to 70 °C and 7 atm is the operating pressure. The operating pressure 9atm decreases the amount of  $C/C_0$  near the 0.05 not below the 0.04 at all experimental operating temperatures. So, running the process under 9atm seems to be more feasible. According to the results in Figure 3 and 4, providing operating temperatures above the 50 °C is just effective on decreasing the amount of hydrogen sulphide at 7atm. Although the outlet concentration is the same at 30 °C and 70 °C, but the consumed energy in steam jacket to provide the operating temperature of 70 °C is higher comparing with 50 °C. So, the temperature of 50 °C and pressure of 7 atm are preferred as the best operating conditions.

Figure 3 and 4 shows the decrease in the outlet  $H_2S$  concentration by the temperature augmentation. Temperature varies from 30 °C to 70 °C. The adsorption progress is obtained by temperature rise. The values of  $C/C_0$  are changed from 0.22 to 0.034 and from 0.184 to 0.0028 when catalyst with 60 nm and 40 nm in diameter are used at 7atm, respectively.

### CONCLUSIONS

Low temperature gas sweetening applying nano

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catalyst has been investigated and has not commercialized yet. Nano catalyst development in various areas proposes to perform many processes economically and efficiently. The optimum operating conditions and reactor characteristics for hydrogen sulphide removal with Al2O3 nano catalyst are investigated experimentally in this work. The process performance is considered as the ratio of the outlet concentration of  $H_2S$  per the inlet concentration and is presented as value of  $C/C_0$ . Experiments are held in the cylindrical reactor in different temperature, pressure, bed height, bed diameter and Al2O3 catalyst diameter to find the best condition to reach the C/C0 value of 0.04 (approximately) for H2S at the product.

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