Al-Khwarizmi Engineering Journal, Vol. 9, No. 3, P.P. 58- 69 (2013)

Al-Khwarizmi
Engineering
Journal

Simulation of a Wet Sulfuric Acid Process (WSA) for Utilization of Acid Gas Separated from Omani Natural Gas

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(Received 2 April 2013; accepted 6 October 2013)

Abstract

In this study, a proposed process for the utilization of hydrogen sulphide separated with other gases from omani natural gas for the production of sulphuric acid by wet sulphuric acid process (WSA) was studied. The processwas simulated at an acid gas feed flow of 5000 m^3/hr using Aspen ONE- V7.1-HYSYS software. A sensitivity analysis was conducted to determine the optimum conditions for the operation of plant. This included primarily the threepacked bed reactors connected in series for the production of sulphur trioxidewhich represented the bottleneck of the process. The optimum feed temperature and catalyst bed volume for each reactor were estimated and then used in the simulation of the whole process for two cases namely 4 and 6 mole% SO_2 stream fed to the first catalytic reactor. The 4mole% SO_2 gaves the highest conversion (98%) compared with 6 mole% SO_2 (94.7%). A valuable quantity of heat was generated from the process. This excess heat could also be transformed into power in a turbine or used as a heating media in neighbouring process units.

Keywords: *Simulation, Acid Gases, WSA, Sulphuric Acid Production.*

1. Introduction

Most of natural gases contain H_2S as a major impurity. H_2S isa highly toxic and corrosive gas and considered as one of the majorsources for the environmental problems such as acid rains. Therefore, in order to utilize these fuels for chemical processing or energygeneration, the H_2S concentration in these gases must be reducedto very low levels [1].

Acid gas removal process is a very important industrial operation, which has been described elsewhere. The main processes used are based on absorption, and the selectivity of the solvent with respect to acid gasses is based on an affinity of the chemical or physical type. Adsorption is also used for intensive purification. Gas permeation has a substantial potential, but today, industrial applications are limited. The different processes for separation of acid gases from natural gas is well represented by Zulkifli[2]. Numerous different methods exist for the treatment of gaseous effluents that containboth acid compounds H_2S and CO_2.The selection of an optimum method for theelimination is not easy and, in many cases, to obtain the degree of purification of gas desired, it is necessary to combine different processes.

For the evaluation of the methods of utilization, the following variables, amongothers, must be taken into account [3]:

- Concentration of H2S in the gas to be utilized.
- Pressure, temperature and composition of the gas.
- Ratio of H_2S to CO_2.
- Volume of gas to be processed.
- Total H_2S load
- Off-gas specifications for the process output.
- Economic considerations.
- Environmental implications.

The acid gas after separation from Omani natural gas contains hydrogen sulfide, carbon dioxide, water vapor and small quantity of methane [4].

The aim of this study is to perform a case study on the application of the WSA process for the utilization of H_2S in the acid gas to produce sulfuric acid using wet sulfuric acid process (WSA) and to determine the optimum conditions for this process.

2. The WSA Process

The WSA (Wet gas Sulphuric Acid) processis an effective and in some cases superior alternative to the Claus process for the production of sulphur from sulphur containing gases[1].It is a process for the conversion of sulphurous streams into sulphuric acid developed by Haldor Topsøe A/S, Denmark. Since its introduction in the 1980s, the WSA process were recognised as an efficient process for recovering sulphur from various sources in the form of commercial quality sulphuric acid. The WSA process is a catalytic process producing 93–98 wt% acid [5].Up to 2009, Topsøe WSA plants have been in operation foralmost 30 years, and close to 80 plants were sold[6]. It has found widespread application in the metallurgical, steel industry (coking plant), power and cellulose industry, but the majority of applications are within the refinery and petrochemical industry[7].

In spite of its unique features andability to treat gases without any prior drying, the WSA technology has had certainrestrictions with regards to SO_2 concentration in the feed gas, and the overall conversionof SO_2 by catalytic means has been limited to approximately 99.7%.The WSA process is applied in all industries where the removal of sulphur is an issue.Typical applications include [5-8]:

- H2S-containing gases from amine units.
- Sour water stripper gases.
- Regeneration of spent sulphuric acid from alkylation.
- Treatment of stack gases from boilers fired by heavy residues and petroleum coke.
- In the viscose fibre industry for treatment of hydrogensulphide and carbon disulphide containing off-gases.
- In the non-ferrous metallurgical industry for treatmentof SO_2 containing off-gases from a variety of process units.

There are many advantages why one should use the WSA process for acid-gas treatment. The process does not generate any waste products or waste water and does not use any absorbents. Firstly the process produces sulfuric acid, which is a more valuable compound in comparison to elemental sulfur. Secondly the process uses the water content in the process gas and air introduced, which enables production of high purity concentrated sulfuric acid.

The block diagram for the WSA process is shown in Figure 1.

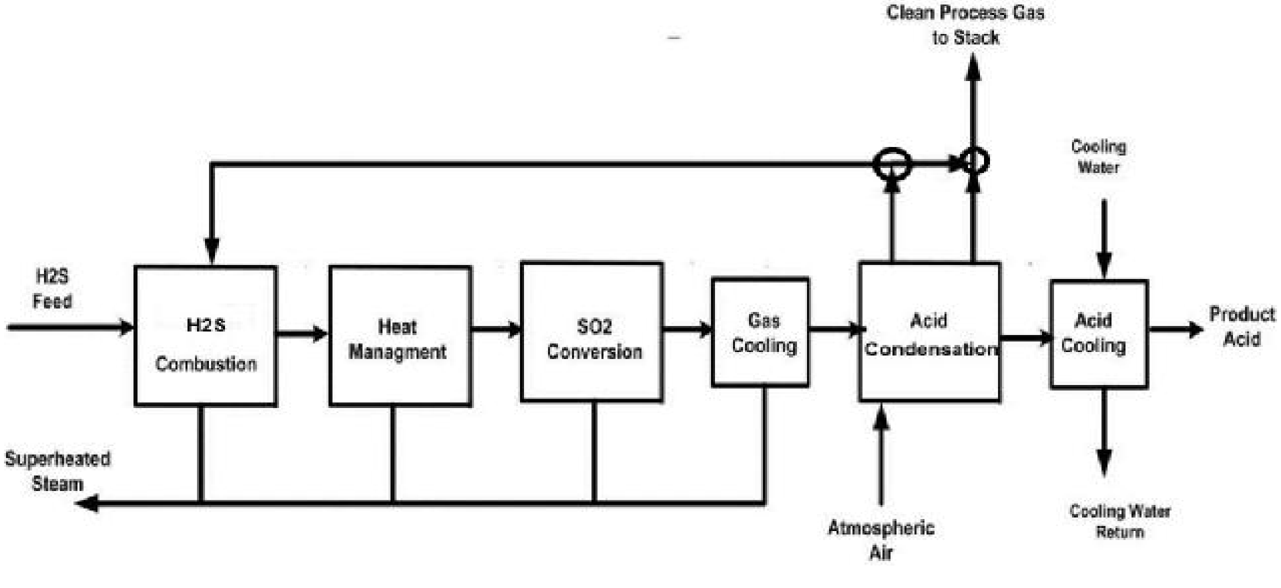

Fig. 1. A Block Diagram of the Various Steps in the WSA Process.

The inlets sulphurous gases are fed to a combustor where they are mixed with combustion air. In the combustor the hydrogen sulphide isconverted to sulphur dioxide according to thefollowing exothermic reaction[9]:

$$H_2S + 3/2\ O_2 \rightarrow H_2O + SO_2 \qquad ...(1)$$

In case there is some methane in the acid gas (as in our case), it will be combusted according to the following reaction:

$$CH_4 + 2O_2 \rightarrow CO_2 + 2H_2O \qquad ...(2)$$

The process gas leaving the combustor iscooled in a waste heat boiler. The cooledSO2 containing gas is fed to the SO2 converterwhere it is converted to SO3 in three adiabaticcatalytic beds. In the converter the SO2 isoxidized to SO3 according to the following reaction:

$$SO_2 + 1/2\ O_2 \leftrightarrow SO_3 \qquad ...(3)$$

The reaction is a temperature and pressuredependent equilibrium reaction. In order toachieve a high conversion the reaction is cooledin two inter-beds[8]. After the third catalyst bed theprocess gas is cooled by boiler water. During thiscooling part of the formed SO3 reacts with thewater vapour present in the process gasaccording to the following exothermic hydrationreaction:

$$SO_3\ (g) + H_2O\ (g) \rightarrow H_2SO_4\ (g) \qquad ...(4)$$

Being a single-contact process, the SO2/SO3 equilibrium curve limits the conversion totypically 99.4 - 99.7%[6]. This limitation can be overcome by scrubbing the tail gas withcaustic or hydrogen peroxide, but this means additional investment and operatingcosts.
After the SO2 converter the process gas is fed tothe WSA condenser were it is cooled by ambientair. During the cooling the remaining hydrationreaction and condensation of the sulphuric acidtakes place:

$$H_2SO_4\ (g) \rightarrow H_2SO_4\ (l) \qquad ...(5)$$

The condensed hot sulphuric acid is furthercooled in a heat exchanger cooled by coolingwater. The acid is of commercial quality. Theclean process gas leaves the condenser at the top.The heat released by all the exothermic reactions in the WSA plant is recovered in the form of steam.

Because of considerations concerning construction materials in the WSA condenser,is not possible to handle gases with sulphuric acid dew points higher than around260°C[6]. This corresponds to a content of SO_2 inlet to the SO_2 converter of some 6 – 7vol%. This limitation of course can be overcome by dilution of the gas withatmospheric air, but this will increase the process gas volume and thereby thedimensions of the plant.

3. Simulation of WSA Process

WSA process is simulated with AspenONE-7.1-Engineering (HYSYS) using the actual composition of theacid gas produced after separation from Omani natural gas [4] as shown in Table 1:

Table 1,
Feed acid gas composition.

Component	**Composition Mole%**
H_2S	16.7
CO_2	73.0
CH_4	0.20
H_2O	10.1

Although some authors refer to the H2S acid gas separated from natural gas by amine process but actually there is no data available in literature about this application. In any case, all the required operating data necessary to complete the simulation are collected from different sources [6,9,10].The air introduced to the process is assumed to be at 40 oC and with 70% relative humidity according to Omanclimate.

The most important part of this process is the SO_2 oxidation reactors. There are commercially three reactors in series. Each reactor is occupied by different quantity of catalyst. The SO_2 oxidation catalyst is based on a silica carrier (diatomeous earth) impregnated with a mixture of sodium, potassium and vanadium [10].The VK-WSA catalysts are specially developed for use in humid process gases [6]. The top layer of catalyst in the first bed consists of 25 mm daisy shaped particles which provide good capacity for accumulation of dust without creating excessive pressure drop. The remaining catalyst consists of either 9 mm or 12 mm daisies. Due to the exothermal reaction, the outlet temperature from the first bed may reach around 600°C. The gas is cooled by superheating of high pressure steam and the gas flows to the second bed. After the second bed the gas is cooled again by superheating of high pressure steam and the conversion is continued in the third bed. At the outlet of the third bed the conversion has reached more than 95%. The gas is then cooled to around 300°C by production of high pressure steam, whereby part of the SO_3 reacts with H_2O to form H2SO4 vapour.

The typical properties of the VK-WSA series catalyst specially manufactured for the WSA process is shown in Table 2. [11]:

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Typical properties of the VK-WSA series catalyst.

Catalyst	**VK-WSA**	
Description		
Diameter (outer/inner), (mm)	25/9	12/4
Length , (mm)	22-28	10-14
Chemical composition		
V_2O_5, (wt%)	6 - 8	
K, (wt%)	7 - 12	
Na, (wt%)	1 - 2	
Physical properties		
Bulk density (kg/m^3)	420-440	350-370
Attrition loss (%)	4 - 6	2 – 4
Operating conditions		
Gas concentration, SO_2 (mole%)	0 - 10	
Minimum O_2/SO_2 (mole/mole)	0.6	

The thermal and electrical behaviour “K-Na-V” catalyst series for SO_2 oxidation (produced specially for the WSA process) is studied by Guo et al.[12]. There results showed the following reaction kinetics as a function of the partial pressures:

$$r_{SO_2} = \frac{k_1 P_{SO_2} P_{O_2}{}^{m} (1-a)}{(P_{SO_2} + k_2 P_{O_2}{}^{m} + k_3 P_{SO_3})} \qquad ...(6)$$

$$a = \frac{P_{SO_3}}{K_p \; P_{SO_2} \; P_{O_2}{}^{0.5}} \qquad ...(7)$$

$$k = A \;\; \exp(-\frac{E}{RT}) \qquad ...(8)$$

$$\log K_p = \frac{4905.5}{T} - \;\; 4.6455 \qquad ...(9)$$

The parameters used in above equations are shown in Table 3.

Table 3,
Parameters used in the reaction rate equation [12].

Parameter	**Operating temperature range, ^{o}C**	
	380 - 470	**470 - 580**
A_1	3.035 x 10^{7}	15.63
E_1[kJ/kmole]	1.69 x 10^{5}	7.89 x 10^{4}
A_2	1.943 x 10^{-7}	1.111 x 10^{-5}
E_2 [kJ/kmole]	-7.21 x 10^{4}	- 5.57 x 10^{4}
A_3	3.021 x 10^{4}	5.019 x 10^{4}
E_3[kJ/kmole]	6.912 x 10^{4}	6.897 x 10^{4}
m	0.65	0.55

Many references stated that;the process which leave the last catalyst bed will typically contains SO_3 plus water vapour[5,7,8]. This means that there are no remarkable reaction between SO_3 and water vapour during the three catalytic reactors connected in series. After the last conversion stage, the gas is cooled and most of the SO_3 reacts with water vapour and forms gas phase sulphuric acid and then the process gas goes to the WSA condenser where final hydration and condensation of acid takes place[5]. Only Almqvist et al. [14] assumed that the reaction which produces vapour sulphuric acid occurrs through the three catalytic packed reactors connected in series.

The reaction kinetics for the formation of gaseous sulphuric acid was studied by different authors [15-18] . Only Jayne et al. [18] studied the effect of temperature on this reaction kinetics and theirwork is represented by the following equations:

$$-r_{SO3} = k_4 \; C_{H2O}{}^{2} \;\; C_{SO3} \qquad . ..(10)$$

$$k_4 = 1.41 \, x \, 10^{7} \; exp(13.5/RT) \qquad ...(11)$$

Where R is in Kcal/mole.K and rSO_3 in cm6/mole.s

We try to introduce this kineticsinside the three catalytic packed reactors connected in series used for the oxidation of SO_2 in the simulation case using equations 10 and 11.We found that we need to increase the volume of reactors ten times to allow the vapour phase sulphuric acid formation to occur in these reactors. In this simulation we assume that all the vapour phase formation of sulphuric acid occurs after the third catalytic packed reactor according to different authors [5,7,8] . After the third reactor the effluent is cooled to 270 oC and then introduced to the condenser which represented in the simulation by a conversion reactor where we assumed that all SO_3 will convert to H_2SO_4.

In this simulation we try to apply two different concentration of SO2 fed to the catalytic packed reactors namely 4 and 6 mole% by regulating the flow of air fed to the system. More than 6 mole% SO_2 will not be applicable since there is no enough oxygen for oxidation of SO_2. Less than 4% will reduce the temperature in the reactor and reduce the efficiency of the heat utilization. In any case 4-6 mole% SO_2 was normally used in commercial production of sulfuric acid by WSA process [5,7].

5. Results and Discussion

The simulation sensitivity results for the effect of input temperatures for the three catalytic reactors on the fractional conversion of SO_2 are shown in Figure 2 and 3 for 4 and 6 mole% SO_2 respectively. The optimum feed temperature for 4% will be 430, 450 and 430 0C for the three reactors respectively. The optimum feed temperature for 6% will be 430, 460 and 440^{o} Crespectively.

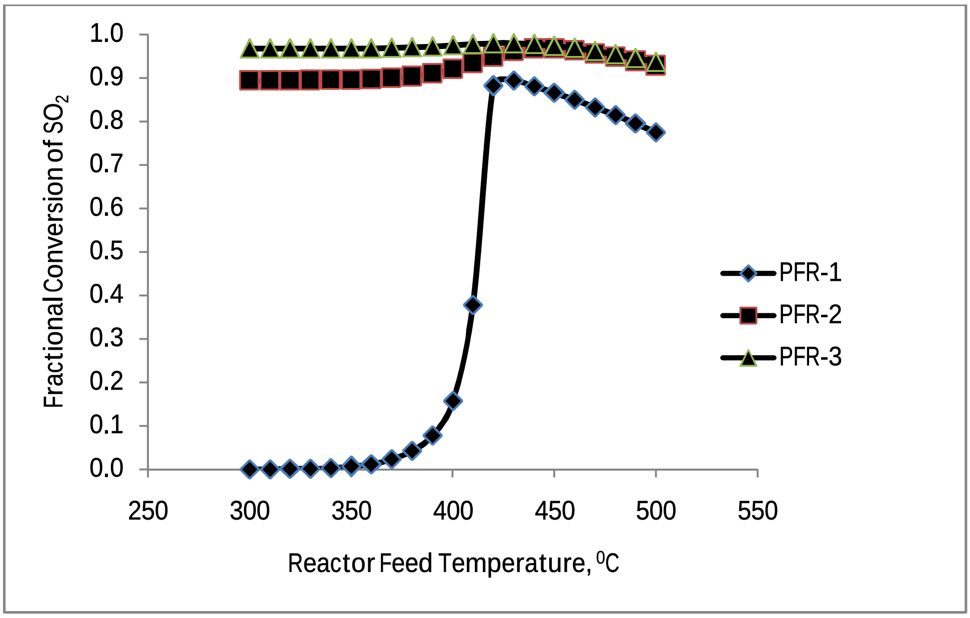

Fig. 2. Effect of Reactor Feed Temperature on the Cumulative Fractional Conversion of SO_2 in the Three Plug Flow Reactors (PFR's) Connected in Series at 4 Mole% SO_2 in Feed.

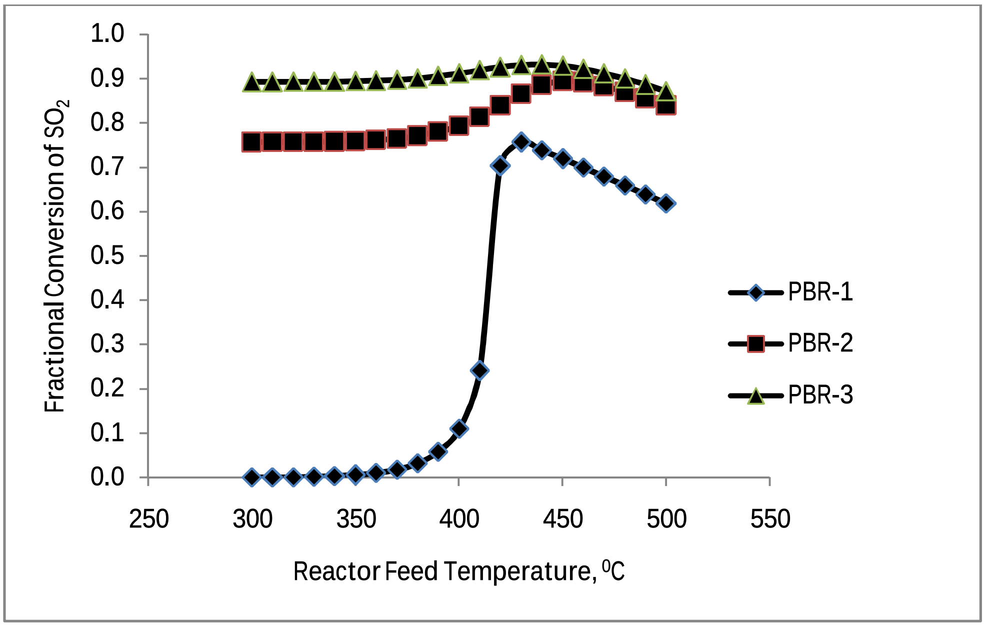

Fig. 3. Effect of Reactor Feed Temperature on the Cumulative Fractional Conversion of SO_2 in the Three Plug Flow Reactors (PFR's) Connected in Series at 6 Mole% SO_2 in Feed.

The effect of reactor volume for the three catalytic reactors on the fractional conversion of SO_2 were shown in Figures 4 and 5 for 4 and 6 mole% SO_2 respectively. The optimum volumes for 4 mole % SO_2 is 3.5, 4.5 and 5.5 m3 for the three reactors respectively. The optimum reactorvolumes for 6% is 4.5, 5.5 and 6.5for the three reactors respectively.

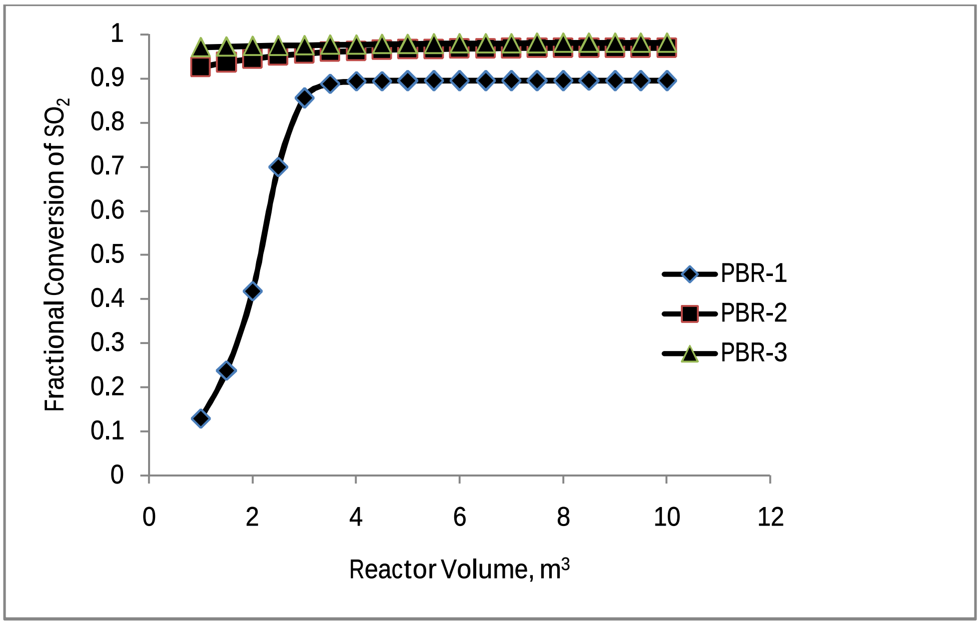

Fig. 4. Effect of Reactor Volume on the Cumulative Fractional Conversion of SO_2 in the Three Plug Flow Reactors (PFR's) Connected in Series at 4 Mole% SO_2 in Feed.

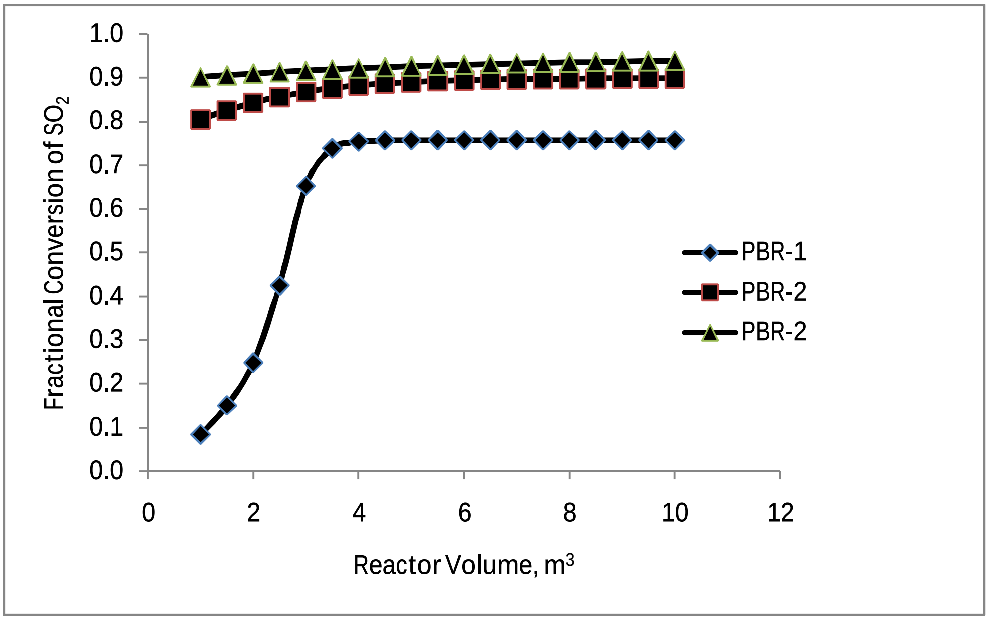

Fig. 5. Effect of Reactor Volume on the Cumulative Fractional Conversion of SO_2 in the Three Plug Flow Reactors (PFR's) Connected in Series at 6 Mole% SO_2 in Feed.

The mole fraction of different component throughout the first plug flow reactor is shown in Figure 5 and 6 for 4 mole% and 6 mole% SO_2 respectively.

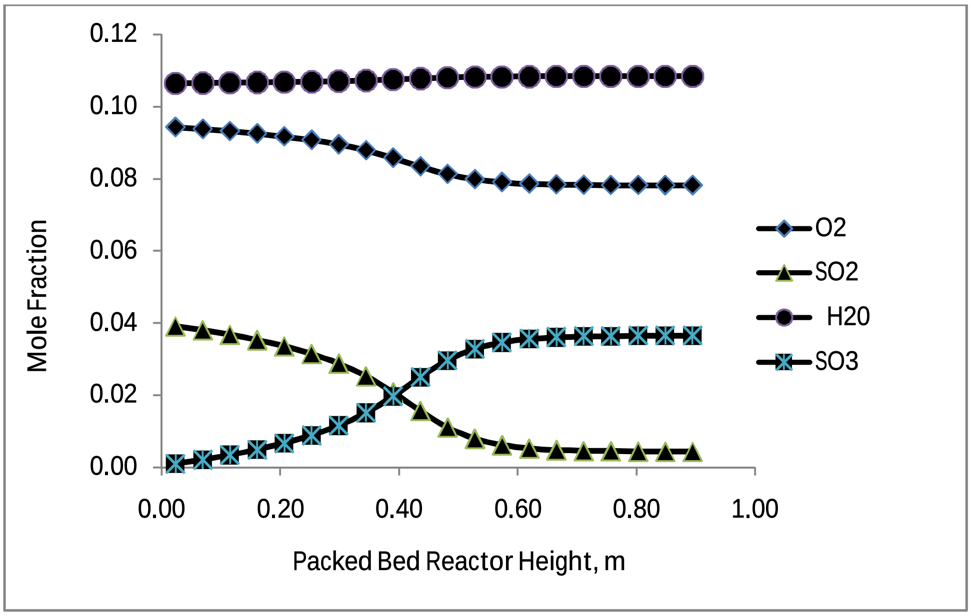

Fig. 6. The Distribution of Mole Fraction of Different Component throughout the First Plug Flow Reactor (PBR-1) at a Feed of 4 Mole% SO_2.

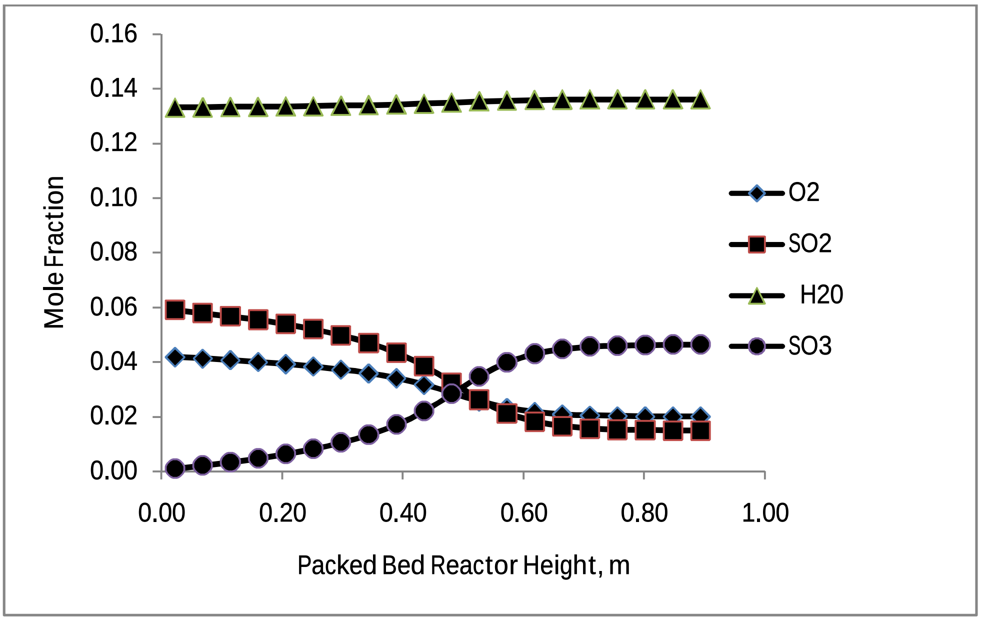

Fig.7. The Distribution of Mole Fraction of Different Component Throughout the First Plug Flow Reactor (PBR-1) at a Feed of 6 Mole% SO_2.

The optimum conditions with respect to the volume of reactors and the feed temperature to the reactors were used in the simulation for the two cases (4 mole% and 6 mole% SO_2).The main results for the two simulation cases are shown in Table 4

Table 4,
Main results of the process simulation.

	Conversion at discharge from PFR's			Total heat generated from the whole Process	wt% H_2SO_4	Production Rate of H_2SO_4kg/h
	PBR-1	PBR-2	PBR-3			
4% SO_2	0.894	0.967	0.980	9.95 x 107kJ/h	98.7	3620
6% SO_2	0.7736	0.9107	0.947	1.084 x 108kJ/h	98.0	3527

As shown in Table 4, the equilibrium conversions will reach a maximum value that cannot be exceeded even if we increase the volume of catalytic reactor which is the normal case for equilibrium reversible reaction. In general, the equilibrium conversion that can be reached for 4 mole% SO_2 is relatively higher than 6 mole% SO_2. The presence of excess oxygen raises the SO_2 equilibrium conversion as mentioned before, but it is also an essential pre-requisite for maintaining the activity of the vanadium catalyst. In any case working with 4 mole% SO_2 will reduce the treatment efforts for the off-gas produced from this process.

Haldor TopsØe A/S gave guarantee of a maximum of 2 kg SO_2 emission per ton 100 % sulfuric acid produced for one of the cases studied [14]. Actually this value cans hardlyachieved in our case where about 16 kg of SO_2 is produced per ton 100% sulfuric acid produced. This implied that extra tail gas treatment was necessary in order to achieve desired sulfur dioxide content in the stack gases.

The overall process is exothermal and large amount of steam is produced in the heat exchangers, which is used for preheating of the feed gas and for addition of water if that is necessary. The excess heat can also be transformed into power in a turbine or used as a heating media in neighbouring process units[14].No remarkable difference in the heat generated and production capacity and concentration of sulphuric acid for both cases were observed (as shown in Table 4).

The complete simulation flow sheet for the process using Aspen-ONE-HYSYS is shown in Figure 8.

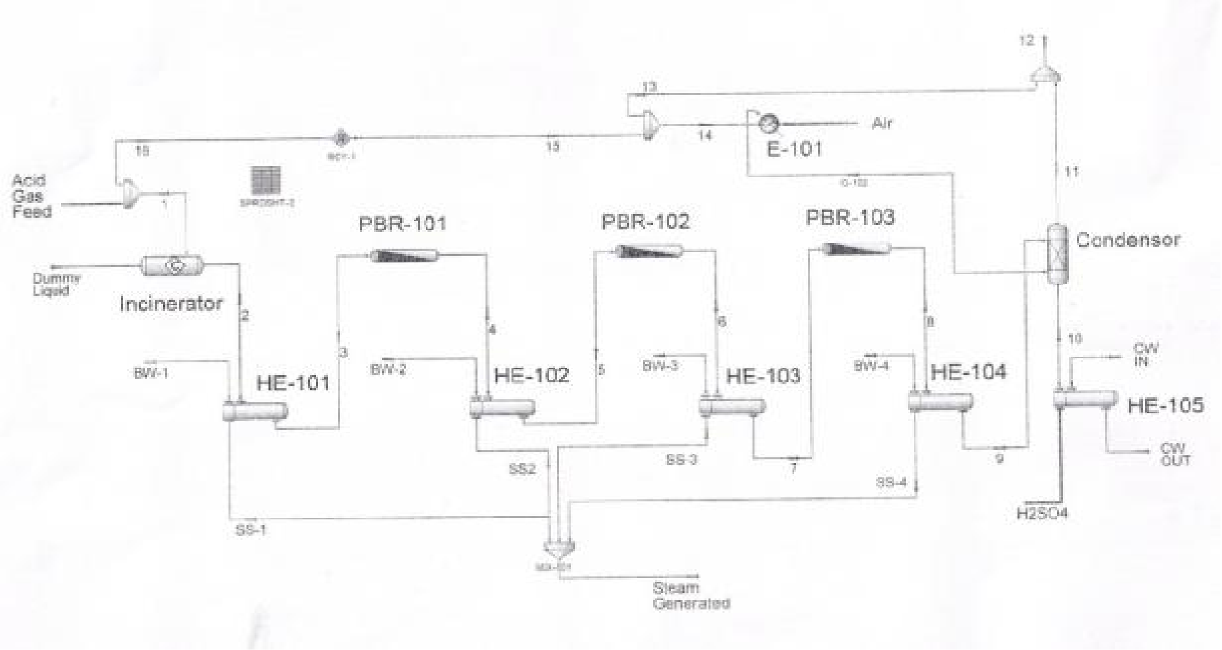

Fig. 8. Simulation Flow Sheet for WSA Process.

6. Conclusions

The following conclusions can be withdrawn from this study:

- Wet sulphuric acid process (WSA) was used successfully for the production of sulfuric acid by utilization of hydrogen sulphide separated from Omani natural gas.
- A sensitivity analysis was conducted to determine the optimum conditions for the plantoperation. This includes primarily the three packed bed reactors connected in series for the production of sulphur trioxide which represent the bottleneck of the process.
- The optimum feed temperature and catalyst bed volume for each reactor for production of SO_3 was found and then impended for the simulation of the whole process for two cases namely 4 and 6 mole% SO_2 stream fed to the first catalytic reactor. The 4 mole% SO_2 gives the highest conversion (98%) compared with 6 mole mole% SO_2 (94.7%).
- A valuable quantity of heat was generated from the process. This excess heat could also be transformed into power in a turbine or used as a heating media in neighboring process units.

Symbols

A	Frequency factor
C_{H20}	Water vapour concentration [mole/cm^3]$C_{SO3}$$SO_3$ concentration [mole/cm^3]
E	Activation energy[kJ/kmol]
K_p	Rate constant
k	Equilibrium constant[atm^{-5}]
m	Partial pressure exponent
P	Pressure[atm]
P_{SO2}	Partial pressure of SO_2[atm]
P_{O2}	Partial pressure of O_2[atm]
P_{SO3}	Partial pressure of SO_3[atm]
R	Gas constant [= 8.314 kJ/kmol K]
r_{SO2}	intrinsic rate of reaction[kmol/kg cat s]
r_{SO3}	rate of reaction for vapour sulphuric acid formation [cm6/mole.s]
T	Temperature [K]

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محاكاة لعملية أنتاج حامض الكبريتيك بالطريقة الرطبة (WSA) لاستغلال الغاز الحامضي المفصول من الغاز الطبيعي العماني

احمد جواد علي
قسم الهندسة الكيمياوية / كلية الهندسة / جامعة صحار

الخلاصة

في هذه البحث، تمت دراسة عملية مقترحة لاستغلال كبريتيد الهيدروجين المفصول مع غازات أخرى من الغاز الطبيعي العماني لإنتاج حامض الكبريتيك بالطريقة الرطبة . (WSA) تم محاكات هذه العملية عند تدفق حجمي للغاز الحامضي بحدود 5000 m3/h باستخدام برنامج AspenONE-V7.1- HYSYS. تم كذلك اجراء تحليل الحساسية لتحديد الظروف المثلى لتشغيل المصنع .وهذا يشمل في المقام الأول المفاعلات الثلاثة المحملة بالعامل المحفز والمربوطة على التوالي لإنتاج ثالث أكسيد الكبريت التي تمثل عنق الزجاجة بالنسبة للعملية .تم التوصل الى درجة الحرارة المثلى لتلقيم كل مفاعل على حدة وكذلك تحديد كمية العامل المحفز لكل المفاعل حيث تم أستخدامها لاحقا في محاكاة العملية برمتها لحالتين هما 4 & 6 mole% SO_2 كتيار تغذية للمفاعل الأول 4% SO_2 .أعطى أعلى نسبة تحول (89 ٪) مقارنة مع 6% SO_2 (94.7%) تم توليد كمية معتبرة من الحرارة نتيجة للعمليات داخل الوحدة .ويمكن أيضا تحويل هذه الحرارة الزائدة الى طاقة داخل توربينات أو يمكن استخدامها في عمليات التسخين لوحدات مجاورة.
