

Part B- Hydrotreating of Gas Oil in Fixed- Bed down Flow Reactor with NiO-MoO₃-WO₃ [F-5] Catalyst for Reactor Modeling and Simulation

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Abstract

Hydrotreating of a mixture of FCC & Coke gas oil, 45:55 respectively was investigated by using FH-5 catalyst to improve the product quality. The experiments were performed in an adiabatic fixed- bed down flow reactor under operation conditions of 4.0 - 6.0 MPa of pressure, 300 - 450 m³/m³ of, hydrogen to gas oil ratio, 2.5 - 3.5 h⁻¹ of LHSV and 300 - 380 °C of temperature. The experimental data were used for reactor modelling and simulation. The set of ordinary differential equations such as material and energy balance equations were integrated along the reactor length using the fourth Rung-Kutta-Gill method using Fortran Program. The addition of quench is required for temperature control for inlet temperature higher than 330°C. Experimental were found to be agree reasonably well with calculated data at high inlet reaction temperature. Hydrocarbon saturation and other reaction must be taken into account due to their contribution to heat evolution. The simulation results showed agreement between experimental and calculated results.

Keywords: Hydrotreating reactor modelling; NiO-MoO₃-WO₃ catalyst.

1. Introduction

Hydrotreating [HDT] is a mild operation to saturate olefins and reduce the sulfur and / or nitrogen, oxygen and metal content without changing the boiling range of the feed. Hydrocracking converts most of the feed to product with boiling range lower than that of the feed [1]. The most important factors in determining the operating conditions needed for a given hydrotreater are the feed stock properties and the desired product properties [2, 3]. These factors set the general severity that is required in hydrotreating operation although other considerations such as catalyst type, heater limitation, or the values of pres-

sure and purity of hydrogen available will affect the actual conditions used [4]. Large-scale hydroprocessing trickle-bed reactors are normally considered to operate under adiabatic conditions because energy losses from the reactor usually negligible compared to the energy generated by the reactions[3]. The Aim of this Study To model and simulate the fixed bed reactor for hydrotreating with the feed of FCC and Coke gas oil. The model developed in this study will be used to facilitate the scale up of experimental unit and properly design in any future experiments of similar nature.

2. Material and Methods

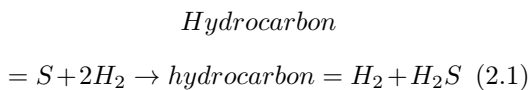
Plug flow Model

The following assumptions were made to the model derivation:

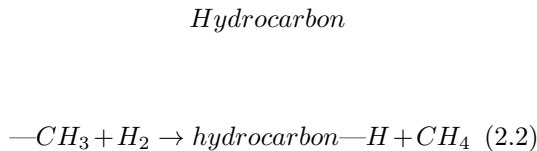
1. The main reactions were the hydrogenation of olefins (HCS), HCC, HDN and HDS.
2. Because the number of individual reactions is too large, it is customary to lump them into groups of reactions. We may write one equation for each of the following lumped reaction groups: 1 Hydrodesulfurization, 2 Hydrodenitrogenation, 3 Hydrocracking, 4 Saturation of unsaturated hydrocarbons.

The following lumped chemical reactions were considered:

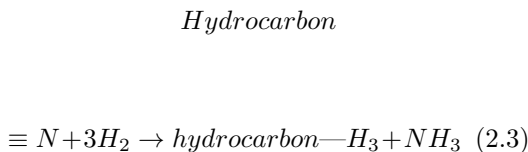
Sulfur-containing hydrocarbons:



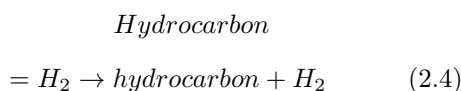
Hydrogenated hydrocrackable hydrocarbons:



Nitrogen-containing hydrocarbons, e.g., pyridines, quinolines:



Unsaturated hydrocarbons with double bonds:



3. The reactions occurred in the liquid phase in contact with the catalyst surface; it meant that the reactions occurred between dissolved hydrogen in the liquid phase and the other reactants in the feed.

4. Plug flow reactor was considered.
5. The reactor is concurrent and down flow.
6. The reactor operates adiabatically, so there is no radial transport of heat; the liquid volume in the reactor remains constant.
7. A gaseous reactant takes part in the reaction and its concentration in the liquid film is uniform and in excess.
8. Catalyst, liquid, and gas are in the same temperature, thus resistance to the transport of heat into the fluid phase and between the external fluid phase and particle surface are neglected.
9. It was assumed that the external mass transfer would be negligible.
10. Reaction occurs only at the liquid-solid interface. The reactor model consists of a set of ordinary differential equations such as material and energy balance equations for each reactant.

3. Material and energy balance

Material balance equation (3.1) , (3.2)

$$\frac{dC_L}{dz} = -\frac{\Re}{u_L} \quad (3.1)$$

Equation (3.1) can be converted to a dimensionless form by substitution of, $CR = \frac{C_L}{C_{L0}}$ and $Z = \frac{z}{dp}$. Thus, we obtain

$$\frac{dCR}{dZ} = -\frac{dpR_L}{C_{L0}u_L} \quad (3.2)$$

Where C_{L0} is the initial value of C_L

Energy balance equation (3.1) , (3.2)

$$\frac{dT}{dz} = \sum [(-\Delta H) \Re] \varepsilon_L / (\varepsilon_G \rho_G C_{pG} u_G + \varepsilon_L \rho_L C_{pL} u_L) \quad (3.3)$$

Again, Equation (3.3) can be converted to a dimensionless form by substitution of, $Z=z/dp$ $T_R=T/T_o$. In addition, after simplification we will have:

$$\frac{dT_R}{dz} = \sum [(-\Delta H) \mathfrak{R}_L] d_p \varepsilon_L / T_0$$

$$(\varepsilon_G \rho_G C_p u_G + \varepsilon_L \rho_L C_p u_L) \quad (3.4)$$

These equations were integrated along the reactor length using the fourth Runge-Kutta-Gill method. The integration of the ordinary differential equations stops, when the temperature exceeds a maximum value fixed as 420°C, in order to protect the catalyst in the bed from sintering. The amount of pure hydrogen gas needed to quench the reaction from a maximum temperature to one of reference temperature (inlet temperature reactor, for example) is calculated; then this amount of hydrogen is added to the mixture, and the integration of the ordinary differential equations is continued for a second bed. The power law models obtained for HDS, HCS and HDN reactions were used in the reactor modeling. In addition, for calculating the liquid hold-up, an expression proposed by Satterfield [7], was used. This equation is namely

$$h_L = \beta \left(\frac{G_L d_p}{\mu_L} \right)^{\frac{1}{3}} \left(\frac{d_p^3 g \rho_L^2}{\mu_L} \right)^{-\frac{1}{3}} \quad (3.5)$$

The oil viscosity, molecular and weight were taken from the literature (Maxwell's book)[8], the hydrogen density and, heat capacity, viscosity was taken from the literature (Perry's Chemical Engineering Hand book) [9], the specific heat, heats of reaction were taken from the literature Tharan [8].

4. Results and Discussion

The performance of trickle bed reactor was studied using experimental data in Table (4.1) and (4.2) (from Part A- Hydrotreating of a Mixture of FCC & Coke Gas Oil, 45:55 respectively in Fixed- Bed Down Flow Reactor with NiO-MoO₃-WO₃ [F-5] Catalyst for Diesel and Gasoline Pool) a summary of simulation results is shown in Table (4.3) -(4.6). From the results we can observed that, by increasing the reactor inlet temperature from 300 to 380°C, the difference between reactor inlet and exit temperature will increased which indicated increase of HDT rate also the small difference between reactor inlet and out let is an

indication of absorption of exothermic reaction. We can also observed that the reactor outlet gas composition changed with inlet temperature, the hydrogen concentration decreased with increasing of reactor inlet temperature while H₂S, NH₃ and CH₄ increased this also and indication of increasing of rate of HDT. Figure (4.1)-(4.4) show typical concentration profiles along the reactor length for the unsaturated hydrocarbons, sulfur, and nitrogen. It can be seen that a larger reactor length is required for the nitrogen removal and saturation of unsaturated hydrocarbons, a similar observation was reported by Cotta et al. [10] in the study of HDS and HDN of middle distillates over a commercial Ni-Mo/ -Al₂O₃. The results indicated that the rate of HCS >HDS >HDN. Figure (4.5) and (4.6) show typical temperature profiles along the reactor it can be seen that addition of quench is required for temperature control for inlet temperature higher than 330°C.

Figure (4.7) to (4.9) show both the experimental and calculated results of reaction temperature on product sulfur, nitrogen, and hydrocarbon saturation. From the figures we can observed that experimental were found to be agree reasonably well with calculated data at high inlet reaction temperature, where there is difference at low temperature this possible may be because the product is unstable at low temperature so that its properties will change during the period between production time and analysis. The effect of other reactions that were not consider in the model. At low temperature, the liquid hold up is very high and this may increase undesirable side reaction. We can also observe that although there is variation in calculated product sulfur, nitrogen data in tables (4.3) -(4.6). But in comparing these data with the experimental one in Figures (4.7) - (4.9) they seem almost constant and higher than experimental data, may be due to measurement range of the method we used to measure experimental data, as well as losses during measurement. In addition, the components of sulfur and nitrogen in the product is very low which make the measurement is very difficult. Also at low reaction temperature the overall reaction rate is effectively first order because the large quantity of relatively easy compounds [sulfur, nitrogen] mak the reaction kinetics of the smaller quantities of refractive compounds. To achieve deep hydrotreating e.g. 98% conversion at higher reaction temperature these

refractory compounds must be broken down, and its their kinetics which now set the overall rate, but in model we used average reaction order for example for sulfur we used 1.5 which is far from one and this is will affect the calculation in mass balance equation so that there is difference between calculated and experimental data at low temperature. Flinn et al.[11] reported a similar observation in their study “Now You Can Improve Residue Treating”, using lumping model, namely, they reported that this relationship does not quite represent the experimental data, because, the reaction appears to follow the rate constant k_1 up to a certain temperature, above which there is a bend in the corresponding curve and the reaction then proceeds according to k_2 . From the results we can observed that plug flow reactor model can be used to study the performance of gas oil HDT reactor.

The mode developed in this study can be used to facilitate the scale up of this experimental unit and to properly design any future experiments of similar nature such as: 1- Design of a commercial scale reactor from fundamental information and / or information from a laboratory scale reactor. 2-Scale up from pilot plant to a large-scale reactor. 3-Performance of the reactor for different feedstock and new catalysts.4-Effect of different reaction conditions on product distribution. 5- Optimization of steady state operating conditions.6- Better understanding of the system that may lead to process design improvements. The simulating results indicated that as the reaction temperature increases the percentage conversion would increase.

Disadvantage of this model are: Not account for the effects of the full range of process variables at low flow rate and temperature on product properties. For example at low temperature, there is deviation between calculated and experimental. Not account for all component reaction presence in gas oil. Can not-predict the effect of catalyst ageing and the changes in catalyst properties on activity and selectivity.

The model can be improved by doing more experiment in which most reactant concentration can be considered as well as individual component instead of lumped one Measure the model parameters instead of taking them from literature Re-evaluate the simplifying assumptions, which may result in imposing new simplifying assumptions or

Table 4.1: reaction order, rate constant, activation energy and frequency factor for HDT reactions by using catalyst

Catalyst	
Reactor	DFR
HDS	
n	1.47
A	27.85096
E_A	866.00914
HCS	
n	1.35
A	3.68422
E_A	336.78133
HDN_T	
n	1.59
A	30.0098
E_A	553.28362
HDN_B	
n	1.37
A	29.46623
E_A	577.33943

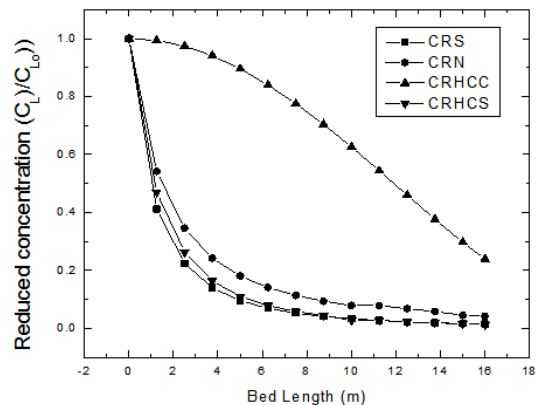
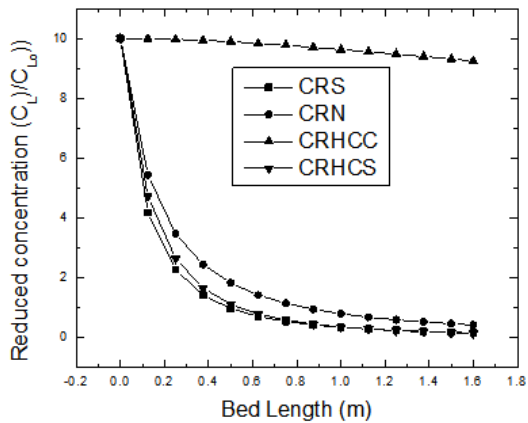
Table 4.2: Feed stocks properties and product quality

Exp. No	Feed	Feed	211G
P MPa		6	6
T °C		350	350
R H ₂ /gas oil		350	350
LHSV h ⁻¹		2.5	2.5
D g/cm ³	0.8924	0.8655	0.8655
S % (ppm)	0.39	[119]	[119]
Br gBr/100g	21.5	1.4	1.4
N ₂ ppm	749	150	150
Base N ₂ ppm	273	36	36
IBP (°C)	205	401 (°F)	120
10%	227	441	211
20%	239.5	463	225
30%	251.5	485	239
40%	269.5	517	253
50%	283.5	542	269
60%	301.5	575	284
70%	317	603	303
80%	335	635	319
90%	345	653	336
Final	>350	>662	>350

Table 4.3: reactor output data at inlet temperature of 300 °C FIXED BED1

ZZ, m	CR=	CRN=	CRHCC=	CRHCS=	T, K=
0.000E+00	1.000E+00	1.000E+00	1.000E+00	1.000E+00	5.732E+02
1.250E-01	4.167E-01	5.431E-01	9.994E-01	4.715E-01	5.963E+02
2.500E-01	2.262E-01	3.470E-01	9.976E-01	2.641E-01	6.071E+02
3.750E-01	1.415E-01	2.436E-01	9.942E-01	1.649E-01	6.126E+02
5.000E-01	9.671E-02	1.819E-01	9.895E-01	1.109E-01	6.158E+02
6.250E-01	7.023E-02	1.419E-01	9.839E-01	7.875E-02	6.178E+02
7.500E-01	5.328E-02	1.143E-01	9.775E-01	5.825E-02	6.191E+02
8.750E-01	4.180E-02	9.434E-02	9.707E-01	4.451E-02	6.201E+02
9.999E-01	3.366E-02	7.944E-02	9.634E-01	3.491E-02	6.208E+02
1.125E+00	2.769E-02	6.797E-02	9.558E-01	2.797E-02	6.215E+02
1.250E+00	2.317E-02	5.894E-02	9.479E-01	2.282E-02	6.220E+02
1.375E+00	1.967E-02	5.168E-02	9.398E-01	1.890E-02	6.224E+02
1.500E+00	1.691E-02	4.575E-02	9.315E-01	1.587E-02	6.229E+02
1.600E+00	1.510E-02	4.175E-02	9.247E-01	1.390E-02	6.232E+02

END OF REACTOR
 GAS COMPOSITION, MOL FRACTION %
 HYDROGEN H₂S NH₃ CH₄
 0.93807 0.02131 0.00911 0.03151



relaxing others.

5. Conclusion

The results obtained with power law model can be used to study the performance of gas oil HT catalysts reactor. Calculations indicated that dilution of the catalyst in this way yields good liquid contacting distribution and plug flow operation. Hydrocarbon saturation [HCS], HCC and other

Table 4.4: reactor output data at inlet temperature of 330 oC FIXED BED1

ZZ, m	CR=	CRN=	CRHCC=	CRHCS=	T, K=
0.000E+00	1.000E+00	1.000E+00	1.000E+00	1.000E+00	6.032E+02
1.250E-01	4.143E-01	5.415E-01	9.938E-01	4.705E-01	6.265E+02
2.500E-01	2.243E-01	3.455E-01	9.748E-01	2.633E-01	6.377E+02
3.750E-01	1.401E-01	2.424E-01	9.415E-01	1.642E-01	6.440E+02
5.000E-01	9.563E-02	1.809E-01	8.960E-01	1.104E-01	6.482E+02
6.250E-01	6.939E-02	1.410E-01	8.402E-01	7.834E-02	6.516E+02
7.500E-01	5.261E-02	1.135E-01	7.758E-01	5.793E-02	6.546E+02
8.750E-01	4.125E-02	9.369E-02	7.041E-01	4.425E-02	6.574E+02
9.999E-01	3.320E-02	7.886E-02	6.263E-01	3.469E-02	6.601E+02
1.125E+00	2.729E-02	6.746E-02	5.441E-01	2.779E-02	6.629E+02
1.250E+00	2.282E-02	5.847E-02	4.599E-01	2.266E-02	6.655E+02
1.375E+00	1.937E-02	5.126E-02	3.764E-01	1.877E-02	6.681E+02
1.500E+00	1.664E-02	4.537E-02	2.973E-01	1.575E-02	6.705E+02
1.600E+00	1.485E-02	4.138E-02	2.395E-01	1.380E-02	6.723E+02

END OF REACTOR
 GAS COMPOSITION, MOL FRACTION %
 HYDROGEN H₂S NH₃ CH₄

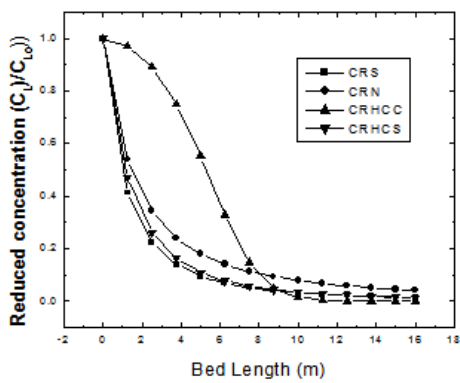


Figure 4.3a : Concentration profiles along reactor length at inlet temperature of 350 °C

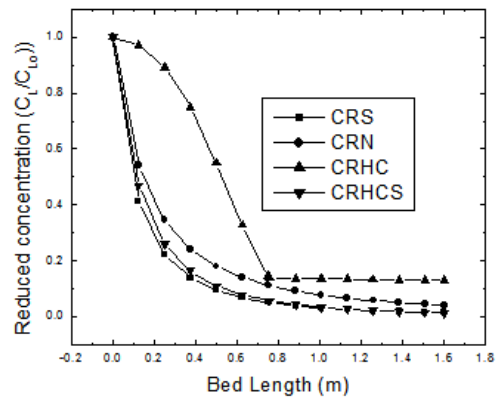


Figure 4.3b : Concentration profiles along reactor length at inlet temperature of 350 °C and addition of hydrogen

Table 4.5: Reactor out put data at inlet temperature of 350 oC FIXED BED 1

ZZ, m	CR=	CRN=	CRHCC=	CRHCS=	T, K
0.000E+00	1.000E+00	1.000E+00	1.000E+00	1.000E+00	6.232E+02
1.250E-01	4.128E-01	5.406E-01	9.725E-01	4.698E-01	6.470E+02
2.500E-01	2.230E-01	3.446E-01	8.918E-01	2.627E-01	6.597E+02
3.750E-01	1.391E-01	2.416E-01	7.491E-01	1.638E-01	6.689E+02
5.000E-01	9.490E-02	1.802E-01	5.512E-01	1.100E-01	6.774E+02
6.250E-01	6.879E-02	1.404E-01	3.288E-01	7.805E-02	6.853E+02
7.500E-01	5.211E-02	1.130E-01	1.472E-01	5.768E-02	6.915E+02
7.570E-01	5.133E-02	1.116E-01	1.392E-01	5.674E-02	6.917E+02

END OF BED 1
 QUENCH = .00000 KMOL/S
 GAS COMPSITION, MOL FRACTION %
 HYDROGEN H₂S NH₃ CH₄
 0.93807 0.02131 0.00911 0.03151

reaction must be taken into account in modeling and simulating gas oil hydrotreating reactor due to their contribution to heat evolution. The aromatic hydrogenation and HDO were not considered in the modeling because there were no available data otherwise; this problem is very complex due to the reversibility of the aromatic hydrogenation reaction.

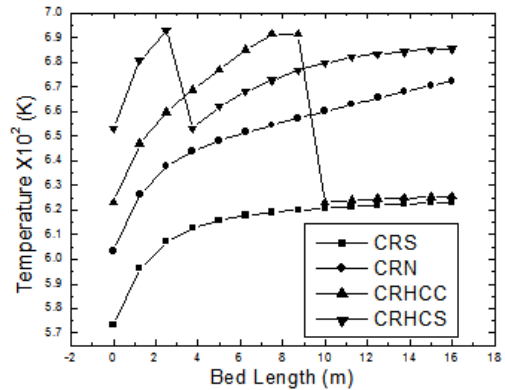


Figure 4.6 : Temperature profile along the reactor length

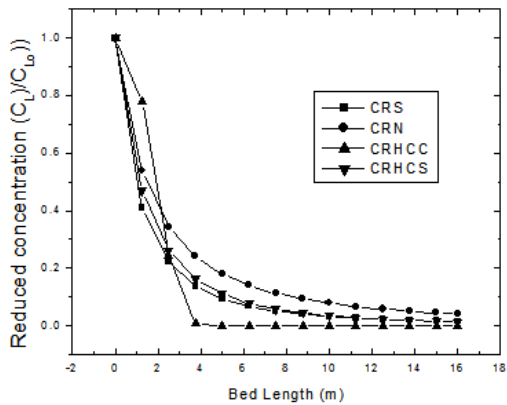


Figure 4.4a: Concentration profiles along reactor length at inlet temperature of 380 °C

Table 4.6: FIXED BED 2

ZZ, m	CR=	CRN=	CRHCC=	CRHCS=	T, K=
7.572E-01	5.133E-02	1.116E-01	1.392E-01	5.674E-02	6.232E+02
8.822E-01	4.043E-02	9.238E-02	1.379E-01	4.347E-02	6.240E+02
1.007E+00	3.266E-02	7.794E-02	1.365E-01	3.416E-02	6.245E+02
1.132E+00	2.693E-02	6.680E-02	1.351E-01	2.742E-02	6.250E+02
1.257E+00	2.259E-02	5.800E-02	1.337E-01	2.240E-02	6.253E+02
1.382E+00	1.922E-02	5.091E-02	1.322E-01	1.858E-02	6.256E+02
1.507E+00	1.654E-02	4.512E-02	1.308E-01	1.561E-02	6.258E+02
1.600E+00	1.490E-02	4.146E-02	1.297E-01	1.382E-02	6.259E+02

END OF REACTOR
 GAS COMPOSITION, MOL FRACTION %
 HYDROGEN H₂S NH₃ CH₄
 0.60508 0.02132 0.00912 0.36447

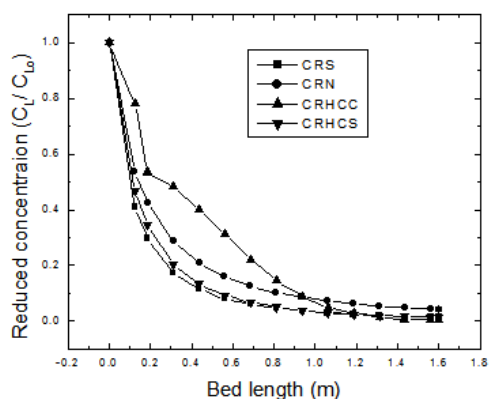


Figure 4.4b: Cocentration profile along reactor bed at 380 °C and addition of hydrogen

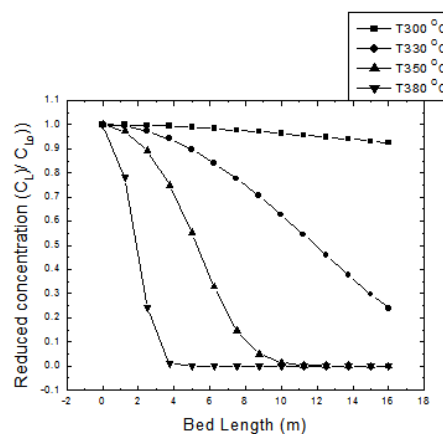


Figure 4.5: Effect of inlet temperature on hydrocracking

Table 4.7: Reactor output data at inlet temperature of 350 oC FIXED BED 1

ZZ, m =	CRS=	CRN=	CRCC=	CRHCS=	T, K=
0.000E+00	1.000E+00	1.000E+00	1.000E+00	1.000E+00	6.232E+02
1.250E-01	4.128E-01	5.406E-01	9.725E-01	4.698E-01	6.470E+02
2.500E-01	2.230E-01	3.446E-01	8.918E-01	2.627E-01	6.597E+02
3.750E-01	1.391E-01	2.416E-01	7.491E-01	1.638E-01	6.689E+02
5.000E-01	9.490E-02	1.802E-01	5.512E-01	1.100E-01	6.774E+02
6.250E-01	6.879E-02	1.404E-01	3.288E-01	7.805E-02	6.853E+02
7.500E-01	5.211E-02	1.130E-01	1.472E-01	5.768E-02	6.915E+02
8.750E-01	4.082E-02	9.318E-02	5.043E-02	4.404E-02	6.949E+02
9.999E-01	3.283E-02	7.841E-02	1.469E-02	3.452E-02	6.965E+02
1.125E+00	2.698E-02	6.705E-02	3.957E-03	2.764E-02	6.972E+02
1.250E+00	2.256E-02	5.811E-02	1.024E-03	2.254E-02	6.975E+02
1.375E+00	1.914E-02	5.094E-02	2.588E-04	1.866E-02	6.978E+02
1.500E+00	1.645E-02	4.508E-02	6.427E-05	1.566E-02	6.980E+02
1.600E+00	1.467E-02	4.112E-02	2.083E-05	1.372E-02	6.981E+02

END OF REACTOR
 GAS COMPOSITION, MOL FRACTION %
 HYDROGEN H₂S NH₃ CH₄
 0.55053 0.02134 0.00913 0.41900

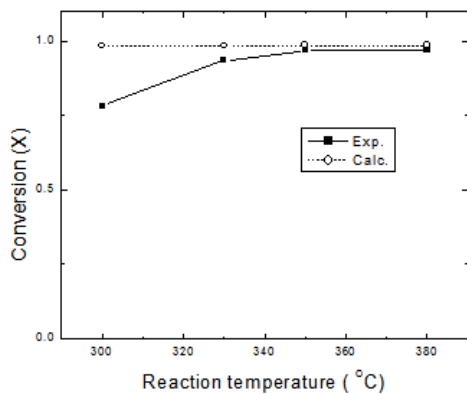


Figure 4.7: Effect of reaction temperature on product sulfur

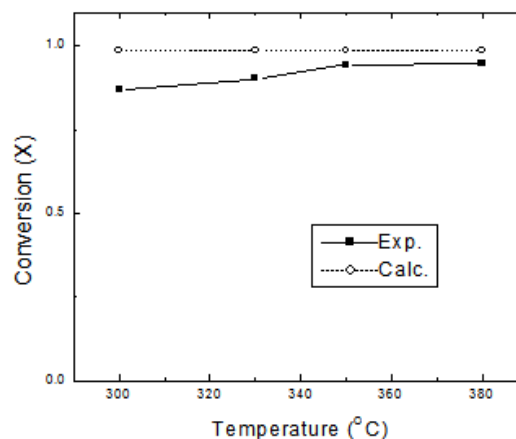


Figure 4.8 : Effect of reaction temperature on product hydrocarbon saturation

Table 4.8: Reactor out put data at inlet temperature of 380 oC FIXED BED 1

ZZ, m =	CR=	CRN=	CRHCC=	CRHCS=	T, K=
0.000E+00	1.000E+00	1.000E+00	1.000E+00	1.000E+00	6.532E+02
1.250E-01	4.106E-01	5.392E-01	7.805E-01	4.689E-01	6.811E+02
1.857E-01	2.966E-01	4.260E-01	5.399E-01	3.470E-01	6.932E+02

END OF BED 1

QUENCH = .00000 KMOL/S

GAS COMPSITION, MOL FRACTION %

HYDROGEN	H ₂ S	NH ₃	CH ₄
0.92551	0.00527	0.00191	0.06731

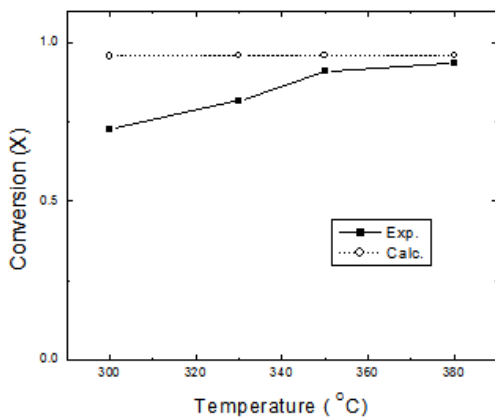


Figure 4.9 : Effect of reaction temperature on product nitrogen conversion

References

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Table 4.9: FIXED BED 2

ZZ, m=	CR=	CRN=	CRHCC=	CRHCS=	T, K=
1.860E-01	2.966E-01	4.260E-01	5.399E-01	3.470E-01	6.532E+02
3.110E-01	1.738E-01	2.863E-01	4.823E-01	2.059E-01	6.621E+02
4.360E-01	1.140E-01	2.076E-01	4.022E-01	1.336E-01	6.681E+02
5.610E-01	8.037E-02	1.586E-01	3.110E-01	9.240E-02	6.729E+02
6.860E-01	5.968E-02	1.257E-01	2.215E-01	6.698E-02	6.768E+02
8.110E-01	4.604E-02	1.025E-01	1.452E-01	5.036E-02	6.798E+02
9.360E-01	3.659E-02	8.545E-02	8.824E-02	3.898E-02	6.821E+02
1.061E+00	2.977E-02	7.253E-02	5.045E-02	3.090E-02	6.836E+02
1.186E+00	2.470E-02	6.246E-02	2.761E-02	2.498E-02	6.845E+02
1.311E+00	2.081E-02	5.446E-02	1.466E-02	2.053E-02	6.852E+02
1.436E+00	1.778E-02	4.798E-02	7.636E-03	1.712E-02	6.856E+02
1.561E+00	1.536E-02	4.265E-02	3.925E-03	1.445E-02	6.858E+02
1.600E+00	1.470E-02	4.116E-02	3.179E-03	1.373E-02	6.859E+02

END OF REACTOR
 GAS COMPSITION, MOL FRACTION %
 HYDROGEN H₂S NH₃ CH₄
 0.55053 0.02134 0.00913 0.41900

Table 4.10: Reactor out put data at inlet temperature of 380 oC FIXED BED 1

ZZ, m =	CR=	CRN=	CRHCC=	CRHCS=	T, K=
0.000E+00	1.000E+00	1.000E+00	1.000E+00	1.000E+00	6.532E+02
1.250E-01	4.106E-01	5.392E-01	7.805E-01	4.689E-01	6.811E+02
2.500E-01	2.211E-01	3.431E-01	2.409E-01	2.618E-01	7.053E+02
3.750E-01	1.376E-01	2.402E-01	9.204E-03	1.631E-01	7.165E+02
5.000E-01	9.373E-02	1.791E-01	1.050E-04	1.095E-01	7.197E+02
6.250E-01	6.790E-02	1.395E-01	0.000E+00	7.761E-02	7.214E+02
7.500E-01	5.143E-02	1.122E-01	0.000E+00	5.735E-02	7.225E+02
8.750E-01	4.030E-02	9.255E-02	0.000E+00	4.379E-02	7.233E+02
9.999E-01	3.242E-02	7.789E-02	0.000E+00	3.432E-02	7.238E+02
1.125E+00	2.664E-02	6.661E-02	0.000E+00	2.748E-02	7.242E+02
1.250E+00	2.228E-02	5.773E-02	0.000E+00	2.241E-02	7.245E+02
1.375E+00	1.891E-02	5.061E-02	0.000E+00	1.856E-02	7.247E+02
1.500E+00	1.625E-02	4.479E-02	0.000E+00	1.557E-02	7.249E+02
1.600E+00	1.450E-02	4.086E-02	0.000E+00	1.364E-02	7.250E+02

END OF REACTOR
 GAS COMPSITION, MOL FRACTION %
 HYDROGEN H₂S NH₃ CH₄
 0.55032 0.02135 0.00914 0.41919