

Mechanism and Kinetics of Hydro-De-Sulphurization of Hydrocarbon Distillates

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Abstract— The hydro-de-sulphurization of petroleum distillates is a catalyst-hydrodynamics flow-driven refining process in any of the heterogeneous reactors. This research used packed-bed catalytic reactor integral design model developed from first principles of balance; incorporating catalyst decay function model in the reactor. The model was integrated numerically to predict catalyst activity for the HDS reactions from 100 percent catalyst efficiency. The rate of cobalt-molybdenum-aluminum oxide ($C_o-M_oAl_2O_3$) decay for the research obeyed a first order reaction rate law as a function of time with a progressive conversion of distillates to sulphur-free products to serve as environmentally friendly downstream feeds.

Keywords— Catalytic reactor, Heterogeneous rate law, Hydro-de-sulphurization, Catalyst-decay, Petroleum light distillates, Energy balance

I. INTRODUCTION

The kinetic process includes a complex network of reactions, i.e HDS, HDN, HDO, HDM, hydrocracking, hydrogenation, and hydro-de-oxygenation involving hydrocarbon species of widely varying reactivity for each of these reactions as catalyst dependent.

The paper therefore focused on the adequate catalytic rate model for HDS process of sulphur-containing hydrocarbons. Kinetics for hydro treating of typical petroleum feed stocks is based on highly simplified treatments of hydrocarbon groups' properties.

Mechanism and Kinetics of HDS Process

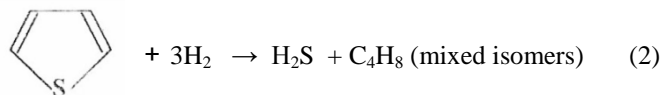
The petroleum – containing sulphur compounds include thiols, sulphides, disulphides, thiophenes and thiophene derivatives.

The approximate order of reactivity is $RSH > R-S-S-S-R' > R-S-R' >$ thiophene, the reactivity of thiophene decreases in the order thiophene, benzothiophen, dibenzothiophene, alkyl-benzothiophenes.

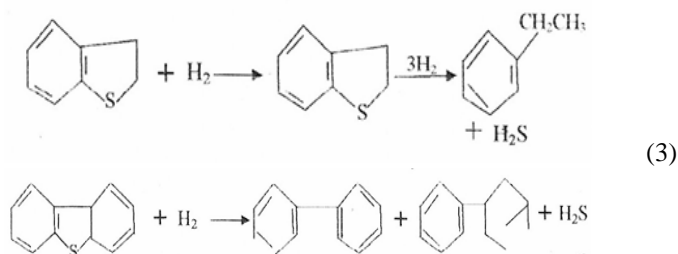
Thiols and sulphides react to form hydrogen sulphid and HC, eg.



Thiophene reacts largely to mixed isomers of butane



Benzothiophene and its derivatives are hydrogenated to thiophane derivatives before removal of the sulphur atom;



Proposed reaction network for HDS of DBT according to Gates et al. (1979) is illustrated in equation for biphenyl product.

According to Satterfield, (1991); Batholomew & Roberto (2006), HDS of dibenzothiophene (DBT) under typical industrial conditions occurs by reaction to mainly biphenyl (with small amount of phenyl cyclohexane).

TABLE 1. Typical impurity compositions of the petroleum residue ($b > 550^\circ C$)

Element	Khafji	California	Kuwait
Sulphur (w.t %)	4.3	1.73	3.66
Nitrogen (w.t %)	0.27	0.9	0.2
Nickel (ppm)	32	75	11
Vanadium (ppm)	93	63	38

II. MATERIALS

Catalysts cum Mechanisms

The materials are derived from the analytical problem framework of how sulphur contaminated products in parts per million i.e 50ppm(0.00005kmol) can be removed through Packed bed catalytic reactor.

The main HDS catalysts are based on molybdenum disulfide (MoS_2) together with smaller amounts of other metals. The nature of the sites of catalytic activity remains an active area of investigation, but it is generally assumed basal planes of the MoS_2 structure are not relevant to catalysis, rather the edges or rims of these sheet.

At the edges of the MoS_2 crystallites, the molybdenum centre can stabilize a coordinative unsaturated site (CUS), also known as an anion vacancy. Substrates, such as thiophene, bind to this site and undergo a series of reactions that result in both C-S scission and C=C hydrogenation. Thus, the hydrogen serves multiple roles—generation of anion vacancy by removal of sulfide, hydrogenation, and hydrogenolysis. A simplified diagram for the cycle is shown:

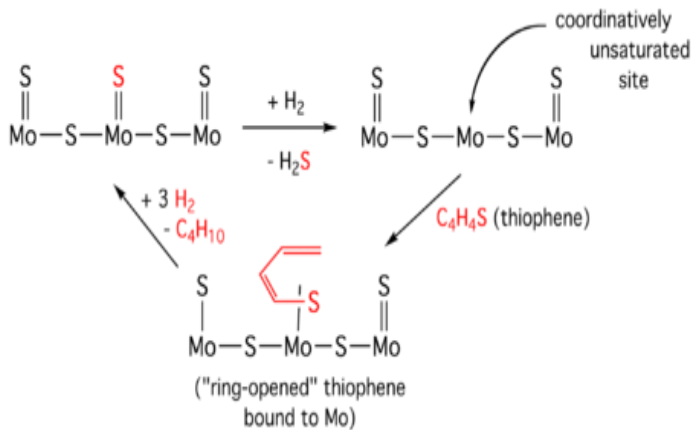


Figure 1. HDS cycle for thiophene (Chianelli, Berhault, Raybaud, Kasztelan, Hafner, & Toulhoat, 2002); (Daage, & Chianelli, 1994)

Catalyst

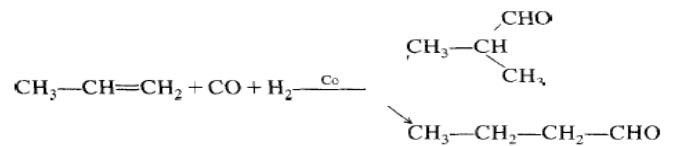
Catalysts have been used by human land for over 2000 years. The first observed uses of catalysts were in the making of wine, cheese, and bread. It was found that it was always necessary to add small amounts of the previous batch to make the current batch. However, it wasn't until 1835 that Berzelius began to tie together observations of earlier chemists by suggesting that small amounts of a foreign source could greatly affect the course of chemical reactions.

This mysterious force attributed to the substance was called catalytic. In 1894, Ostwald expanded Berzelius' explanation by stating that catalysts were substances that accelerate the rate of chemical reactions without being consumed. In over 150 years since Berzelius' work, catalysts have come to play a major economic role in the world market. In the United States alone, sales of process catalysts in 1996 were over \$1 billion, the major uses being in petroleum refining and in chemical production.

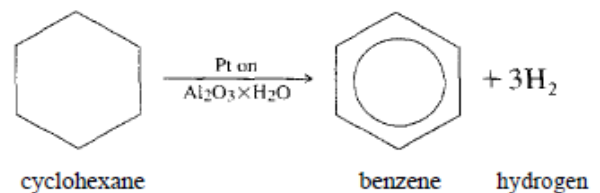
A catalyst is a substance that affects the rate of a reaction but emerges from the process unchanged. A catalyst usually changes a reaction rate by promoting a different molecular path ("mechanism") for the reaction. For example, gaseous hydrogen and oxygen are virtually inert at room temperature, but react rapidly when exposed to platinum. Commercial chemical catalysts are immensely important. Approximately one-third of the material gross national product of the United States involves a catalytic process somewhere between raw material and finished product. The development and use of catalysts is a major part of the constant search for new ways of increasing product yield and selectivity from chemical reactions.

Because catalyst makes it possible to obtain an end product by a different pathway (e.g. a lower energy barrier), it can affect both the yield and the selectivity

Homogeneous catalysis concerns processes in which a catalyst is in solution with at least one of the reactants. An example of homogeneous catalysis is the industrial Oxo process for manufacturing normal isobutyl-alcielhyde. It has propylene, carbon monoxide, and hydrogen as the reactants and a liquid-phase cobalt complex as the catalyst.



A heterogeneous catalytic process involves more than one phase; usually the catalyst is a solid and the reactants and products are in liquid or gaseous form. Much of the benzene produced in this country today is manufactured from the dehydrogenation of cyclohexane (obtained from the distillation of crude petroleum) using platinum-on-alumina as the catalyst.



Supports

Metal sulfides are "supported" on materials with high surface areas. A typical support for HDS catalyst is γ -alumina. The support allows the more expensive catalyst to be more widely distributed, giving rise to a larger fraction of the MoS_2 that is catalytically active. The interaction between the support and the catalyst is an area of intense interest, since the support is often not fully inert but participates in the catalysis.

III. METHODS

Rate Law Model for HDS Process

The rate law for catalyst deactivation function in a HDS process is given in equation (4) below:

$$\frac{1}{(-r_i)} = \frac{1}{\frac{K_T P_T}{[1 + K_T P_T + K_{H_2} S^{P_{H_2S}}]}} \quad (4)$$

Where

$$P_{H_2} = P_T (0.2) = 0.2 P_T$$

$$\frac{1}{(-r_{HDS})} = \frac{[1 + K_T P_T + K_{H_2} S^{P_{H_2S}}]^2}{P_T (0.2 P_T)} \quad (5)$$

$$= \frac{[1 + K_T P_T + K_{H_2} S^{P_{H_2S}}]^2}{0.2 P_T} = \frac{(a + bx)^2}{c}$$

Equation (5) is couched in a matrix form for resolution, and stated mathematically as follows:

Where, $a = 1 + K_T P_T$; $b = K_{H_2} S$, $x = P_{H_2} S$

$$c = 0.2 P_T^2 k$$

$$\frac{1}{(-r_{HDS})} = \frac{(a + bx)^2}{c} = \frac{a^2 + 2abx + bx^2}{c}$$

$$K \propto T \quad (6)$$

$$K = aT + b$$

$$\begin{bmatrix} \sum^n x & \sum^x x^2 \end{bmatrix} \begin{bmatrix} b \\ a \end{bmatrix} = \sum^y xy$$

$$\begin{bmatrix} 3 & 751 \\ 751 & 62817 \end{bmatrix} \begin{bmatrix} b \\ a \end{bmatrix} = \frac{4.9}{1231.1}$$

$$\frac{2253 \quad 564001 \quad 3679.9}{(2253 \quad 1885550 \quad 3695.7)}$$

$$\frac{0 \quad 37550 \quad -15.8}{}$$

$$\begin{bmatrix} 3 & 751 \\ 0 & 375550 \end{bmatrix} \begin{bmatrix} b \\ a \end{bmatrix} = \frac{4.9}{-15.8}$$

$$a = -15.8 / 375550 = -0.000042$$

$$3b + 751a = 4.$$

$$b = \frac{4.9 - 715(-0.000042)}{3}$$

$$b = \frac{4.93}{3} = 1.64$$

$$K = -4.2 \times 10^{-5}T + 1.64$$

For K_T

$$K_T = a_0 + a_1T$$

$$\sum K_T = \sum xy = 91$$

$$\sum xy = 22503$$

$$\begin{bmatrix} \sum^n x & \sum^x x^2 \end{bmatrix} \begin{bmatrix} a_0 \\ a_1 \end{bmatrix} = \sum^y xy$$

$$\begin{bmatrix} 3 & 751 \\ 751 & 62817 \end{bmatrix} \begin{bmatrix} a_0 \\ a_1 \end{bmatrix} = \begin{bmatrix} 91 \\ 832 \end{bmatrix}$$

$$\begin{bmatrix} 3 & 751 \\ 0 & 375550 \end{bmatrix} \begin{bmatrix} a_0 \\ a_1 \end{bmatrix} = \begin{bmatrix} 91 \\ 832 \end{bmatrix}$$

$$a_1 = \frac{832}{375550} = 0.0022154$$

$$a_0 = \frac{91 - 751(0.0022154)}{3}$$

$$a_0 = 29.78$$

$$K = 29.78 + 2.2 \times 10^{-3}T$$

For $K_{H_2}S^{(atm-1)}$

$$K_{H_2}S = \beta T$$

$$\begin{bmatrix} \sum^n x & \sum^x x^2 \end{bmatrix} \begin{bmatrix} \alpha \\ \beta \end{bmatrix} = \sum^y xy$$

$$\begin{bmatrix} 3 & 751 \\ 751 & 62817 \end{bmatrix} \begin{bmatrix} \alpha \\ \beta \end{bmatrix} = \begin{bmatrix} 48.6 \\ 11781 \end{bmatrix}$$

$$\begin{bmatrix} 3 & 751 \\ 0 & 375550 \end{bmatrix} \begin{bmatrix} \alpha \\ \beta \end{bmatrix} = \begin{bmatrix} 48.6 \\ 1155.6 \end{bmatrix}$$

$$\beta = \frac{11556}{375550} = 0.0031$$

$$\alpha = \frac{48.6 - 751(0.0031)}{3}$$

$$\alpha = \frac{46.29}{3} = 15.43$$

Hence,

$$K_{H_2}S = 15.43 + 3.1 \times 10^{-3}T \tag{10}$$

Hence,

At 370°C

$$K_{H_2}S = 15.43 + 3.1 \times 10^{-3}(370)$$

$$K_{H_2}S = 16.58 atm^{-1}$$

$$K_T = 29.78 + 2.2 \times 10^{-3}(370)$$

$$K_T = 30.594 atm^{-1}$$

$$K = 105 * [-4.2 \times 10^{-5}(370) + 1.64] \tag{8}$$

$$= 105 * 1.62446 = 170.5683$$

Again; $C_i = \frac{P_i}{RT}$

$$T = 370 + 273 = 643K$$

$$R = 8314J/molK$$

$$P_R = 20 atm$$

$$P_b = \text{bulk density}$$

$$CH_2S = 0.5 ppm = 0.5 \times 10^{-6}$$

$$P_{H_2}S = C_i + RT = 0.5 \times 10^{-6} [8314 * 643]$$

$$P_{H_2}S = 2.673 atm$$

$$P_{CH_3S} = C_{CH_3SH} RT = 50 \times 10^{-6} (8314 * 643)$$

$$P_{CH_3S} = 267.3$$

$$\frac{1}{(-r_{HDS})} = \frac{(a + bx)^2}{105ck}$$

$$a = 1 + K_T P_T = 1 + (30.594) \tag{20}$$

$$a = 611.88$$

$$b = K_{H_2}S = 16.58 atm^{-1}$$

$$c = 0.2 P_T^2 = 0.2(20)^2 = 80 atm^{-2}$$

$$\frac{1}{(-r_{HDS})} = \frac{a^2 + 2abx + b^2x^2}{105ck}$$

$$\frac{1}{(-r_{HDS})} = \frac{4680 + 253.62x + 3.44x^2}{k * 105}$$

$$\frac{1}{(-r_{HDS})} = \frac{4680 + 253.62x + 3.44x^2}{170.5683}$$

$$\frac{1}{(-r_{HDS})} = 27.44 + 1.5x + 0.020x^2 \tag{11}$$

$$\frac{1}{(-r_{HDS})} = \frac{1}{27.44 + 1.5x + 0.020x^2} \tag{12}$$

Equation (12) is the rate law for the mechanism cum kinetics of HDS process for catalytic activity

3.2 Catalyst Activity

The activity of catalyst [CoMo / Al₂O₃] decreases with time because of catalyst decay is a function of rate of reaction at t=0 and rate at t = t

$$a(t) = \frac{(-r_A)'(t)}{(-r_A)'(t=0)} \tag{13}$$

Where:

a(t) = Catalyst activity

$$(-r_A)'(t) = (-r_A)'(t=0) a(t) \tag{14}$$

$$(-r_A)'(t) = a(t)k(T) f n(C_A, C_B, \dots, C_p) \tag{15}$$

Where,

K(T) = Specific reaction rate, temperature-dependent process

C_i = Gas-phase concentration of reactants, products or concentrations

The rate of catalyst decay, (r_d), therefore, can be expressed in a rate law as:

$$-r_d = \frac{da}{dt} = p[a(t)kd(T)h C_A, C_B, \dots, C_p] \tag{16}$$

Where, p[a(t)] is some function of the activity?

k_d = The specific decay constant

h(C₁) = The functionality of (r_d) on the reacting species concentrations

Note that, the functionality of (r_d) can either be independent of concentration (i.e h=1) or will be a linear function of species concentration (i.e h = C_i)

The functionality of the activity term p[a(t)], in the decay law can take a variety of form , i.e

First order decay form

$$P[a(t)] = a \tag{17}$$

Second-order decay forms:

$$P[a(t)] = a^2 \tag{18}$$

$$\left\{ \begin{array}{l} \text{Rate of inf low of heat} \\ \text{int o differential element} \end{array} \right\} = \left\{ \begin{array}{l} \text{Rate of outflow of heat} \\ \text{from a differential element} \end{array} \right\} - \left\{ \begin{array}{l} \text{Rate of heat generator} \\ \text{due to chemical reaction in} \\ \text{differential element} \end{array} \right\}$$

The terms in equation (25) are defined as follows:

$$\left\{ \begin{array}{l} \text{Rate of heat inf low int o} \\ \text{differential element} \end{array} \right\} = (\rho_A V_o C P_A + P_{cat} V_{cat}) T \tag{26}$$

$$\left\{ \begin{array}{l} \text{Rate of outflow of heat} \\ \text{from a differential element} \end{array} \right\} = (\rho_A V_o C P_A + P_{cat} V_{cat}) (T + dT) \tag{27}$$

For this case, the functionality term p[a(t)] in the decay law will be first-order, because it is gas catalyst system beign used for this research.

Hence

$$(-r_A)(t) = ak_d[-r_A^1(t=0)] \tag{19}$$

Differentiating equation (19) gives equation (20)

$$\frac{-da}{dt} = kda[-r_A^1(t=0)] \tag{20}$$

Re-arranging equation (21) and integrating gives:

$$\int_{a_0}^a \frac{da}{a} = -k_d[-r_A^1(t=0)] \int_0^t dt \tag{21}$$

Since k_d = decay constant

$$[Ina]a_0^{a(t)} = -k_d[-r_A^1(t=0)]t \tag{22}$$

$$In \left\| \frac{a}{a_0} \right\| = -k_d[-r_A^1(t=0)]t$$

$$\frac{a(t)}{a_0} = e^{-k_d[-r_A^1(t=0)]t} \tag{23}$$

$$a(t) = a_0 \exp[-k_d(-r_A^1(t=0))t] \tag{24}$$

Where,

a(t) = Catalyst activity, at t = t

a₀ = Catalyst activity at t = 0 (is 100% efficiency)

3.3 Energy Balance of Reactor

Hydrogenation reaction which is the major reaction that characterizes the Hydrodesulphurization process is a highly exothermic reaction and as such it is expedient to develop a model that can be used to monitor the temperature variation at any point in the reactor.

The energy balance will he developed based on the principle of conservation of energy which is stated mathematically below.

The energy balance equation at steady state may he written as:

$$\text{Energy in} + \text{Energy produced} = \text{Energy out} + \text{Energy consumed} \tag{25}$$

The temperature profile along the reactor can he calculated using the following energy balance equation. The energy balance equation can be derived from first principle by applying the conservation principle on the differential element of the reactor as follows:

$$\left\{ \begin{array}{l} \text{Rate of of heat absorption due to} \\ \text{chemical reaction in differential} \\ \text{element} \end{array} \right\} = \sum_{t=1}^n (\Delta H_n) (-r_t) \rho_A dV_R \quad (28)$$

$$\left\{ \begin{array}{l} \text{Rate of accumulation of heat} \\ \text{in the differential element} \end{array} \right\} = 0 \quad (29)$$

Substituting equation (26) to (29) into equation (25), we have

$$(\rho_A v_o CP_A + P_{cat} v_{cat}) T = (\rho_A v_o CP_A + P_{cat} v_{cat}) (T + dT) - \sum_{t=1}^n (\Delta H_n) (-r_t) \rho_A dV_R \quad (30)$$

Where Cp_A = specific heat capacity of species A, Cp_{cat} – specific capacity of catalyst in the reaction, ρ_{cat} = density of catalyst, v_{cat} = volumetric flow rate catalyst, T = inlet temperature, dT = temperature change within differential element, ΔH_n = heat of reaction

$$(\rho_A v_o CP_A + P_{cat} v_{cat}) dT = \sum_{t=1}^n (\Delta H_n) (-r_t) \rho_A A_R dt \quad (31)$$

$$\frac{dT}{dt} = \frac{\sum_{t=1}^n (\Delta H_n) (-r_t) S_A A_R}{S_A \cdot V_o CP_A + S_{cat} V_{cat}} \quad (32)$$

IV. RESULTS AND DISCUSSIONS

4.1 Results

The results of the performance equations developed for the hydrodesulphurization of petroleum distillate is presented Table 1. Parameters for performance equation was simulated using MATLAB

TABLE 1. Performance parameters

S/no.	Parameter	Value	Unit
1.	Catalyst activity	Drops to 0	G
1.	Weight of Catalyst	43.93	G
2.	Reactor Length	6.93	M
3.	Space Time	13.94	S
4.	Space Velocity	0.072	1/s
5.	Heat Load	20.16	Kw/m ³
6.	Fractiona Conversion	0.90	Nil

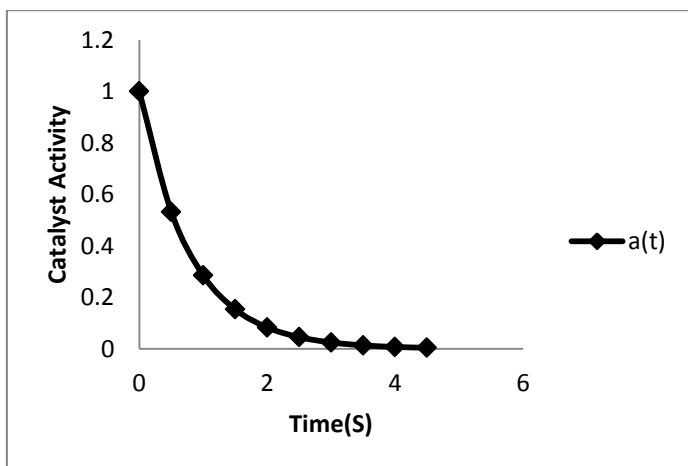


Figure 2 Plot of catalyst activity with time

Figure 2. depicts the profile of catalyst activity, a(t) as a function of time. As shown, the activity of the catalyst is

inversely exponentially related to the time since at time, t = 0, the catalyst activity there is deactivation of catalyst activity reduces exponentially to almost zero(0) as time increases to maximum.

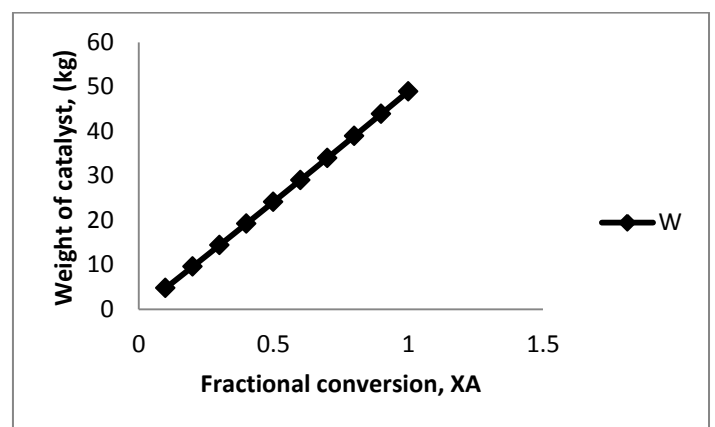


Figure 3. Pot of catalyst weight, W versus Fractional conversion, α_A

Figure 3 shows the plot of weight of catalyst with fractional conversion. The variation is linearly related. As fractional conversion increases, the weight of catalyst also increases. Hence, the weight of catalyst is a function of fractional conversion.

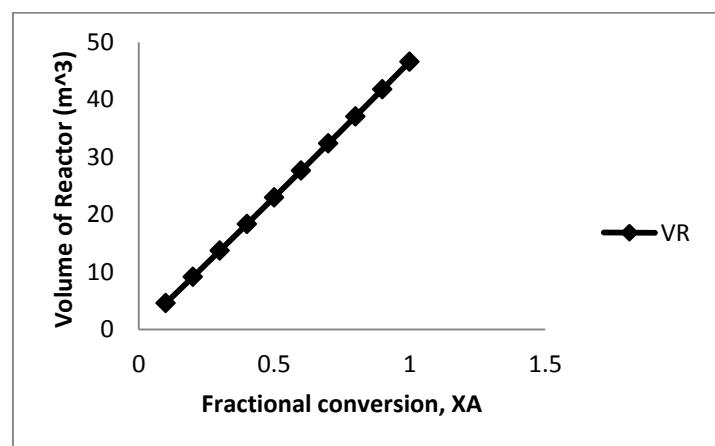


Figure 4. Plot of volume of reactor, V_R against Fractional conversion, α_A

Figure 4 depicts the profile of the volume of the reactor with fractional conversion. The volume of the reactor is proportionally and linear to related to fractional conversion.

As fractional conversion increases, the volume increases too. As $X_A=1$, $V_R=46.61$ Litres.

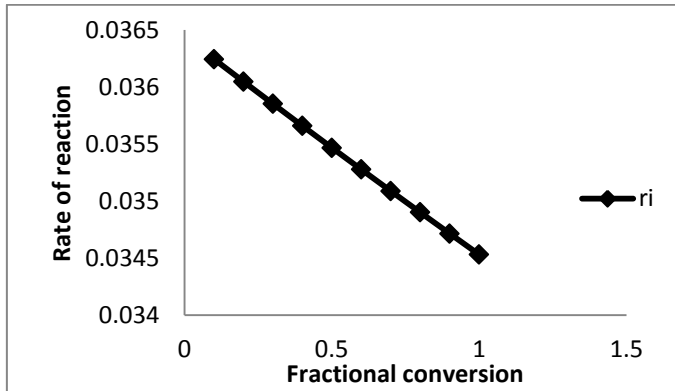


Figure 5. Plot of Rate of reaction, $(-r_A)$ versus Fractional conversion, α_A

Figure 5 shows the variation of the rate of reaction with fractional conversion. From the plot, it is seen that the driving force for the hydrodesulphurization is the rate of reaction. Hence, the rate of reaction is related to fractional conversion and decreases as fractional conversion increased.

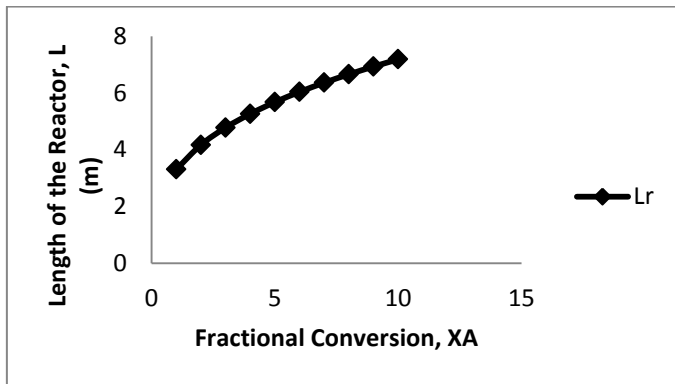


Figure 6. Plot of length of reactor L_R against fractional conversion, α_A

Figure 6 shows the variation of Length of reactor with fractional conversion. From the plot, it is seen that as the length of reactor increases, the fractional conversion also increases showing production of hydrodesulphurization. As seen in Figure 6, the length of reactor increases as the fractional conversion and at $\alpha_A = 1$, $L_R = 7.5m$

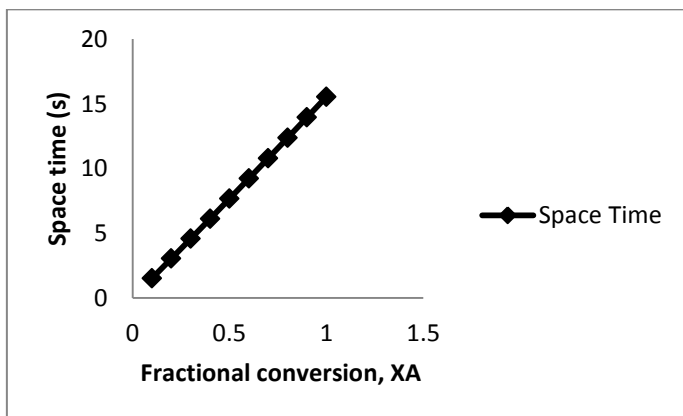


Figure 7. Plot of space time, τ versus fractional conversion, α_A

Figure 7 shows the variation of space time of reactor with fractional conversion. From the plot, it is seen that as the space time increases with the fractional conversion. Space time gives us the time it take a reactor to convert feed material to product. Thus figure 7 depicts this and its increment with fractional conversion.

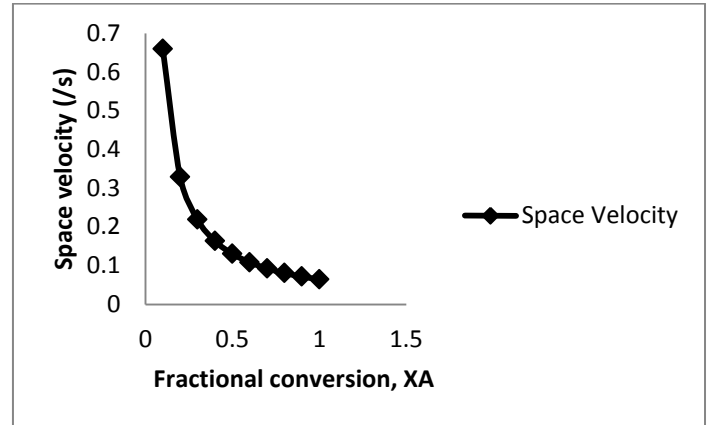


Figure 8. Plot of space velocity, S_v against Fractional conversion, α_A

Figure 8 shows the variation of space velocity of reactor with fractional conversion. From the plot, it is seen that the space velocity decreases with an increase in fractional conversion.

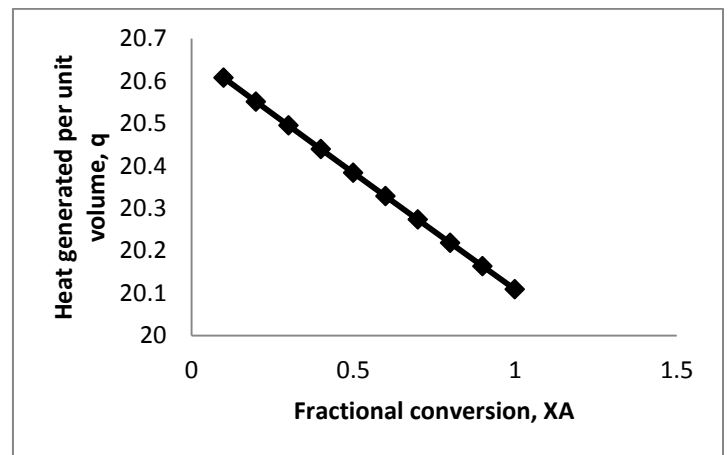


Figure 9. Plot of quantity of heat generated per unit volume versus fractional conversion

As shown in Figure 9, the rate of heat generated per unit volume decreases as fractional conversion increases. This implies that the smaller the value for q at higher conversion gives the best performance of the reactor.

V. CONCLUSION

Ideally, model for catalyst deactivation and heterogeneous rate law were put to test analytically for HDS process. The rate law was coupled with the decay function to achieve a progressive reaction model between the feed distillates and hydrogen involved in the sulphur depletion action.

The mechanism proved to be more effective in the decontamination of sulphur in distillates thereby making the products ultra-low-sulphur products.

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