

SIMULATION OF AN ACETYLENE HYDROGENATION REACTOR

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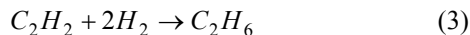
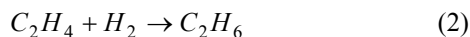
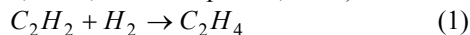
ABSTRACT

The kinetics of acetylene hydrogenation has been studied in a fixed bed reactor of a commercial Pd/Al₂O₃ catalyst. The experiments were carried out at 30, 50 and 70 °C with various feed compositions at atmospheric pressure. The experiments were repeated at 70 °C in the presence of the used catalyst to determine the effect of the catalyst deactivation where the corresponding deactivation rate constant was determined in order to predict the activity of the catalyst during each run. Two well known kinetic models were used for a nearly similar catalyst to predict the experimental data of this work and none of them were found satisfactory. A new model was then proposed to fit the experimental data. The hydrogenation reactor was also simulated at industrial operating conditions with the proposed kinetics for both plug and dispersion flows. The results of these simulations were almost close to each other in most cases.

1 INTRODUCTION

Polyethylene has been a key product for many industries since 1960's. The feed of the polymerization reactor, which comes from the olefin plant, is a mixture of hydrocarbons mainly consisting of ethylene. An undesired impurity in the ethylene stream is acetylene at approximately 0.3 to 2% of the effluent of the olefin plant which may lead to undesirable polymer properties. The amount of acetylene in the feed of ethylene polymerization reactor should not exceed 2-3 ppm (Bos et al., 1993). In order to reach the desired amount of acetylene for polymerization, it is selectively hydrogenated to ethylene in a multi-bed adiabatic fixed bed catalytic reactor.

There are three major reactions considered in this system (Bos et al., 1993; Westerterp et al., 2002):



Many catalysts have been studied for hydrogenation of acetylene. Catalysts based on nickel sulfide (Anderson et al., 1948), nickel or zinc based catalysts on alumina or silica (Muller et al., 1987), cadmium, calcium, barium, strontium or magnesium on Cr₂O₃ (Weisang and Engelhard, 1970) as well as copper on alumina, magnesia or silica (Taghavi et al., 1978) have been used in some cases. However, it was found that palladium is the most selective metal for acetylene hydrogenation and the most common commercially used catalyst is Pd/alumina (Brodzinski and Cybulski, 2000; Vincent and Gonzalez, 2001; Godinez et al., 1995; Vincent and Godinez, 2002). This catalyst is usually prepared either by ion exchange or by precipitation in order to produce a low dispersion, low metal content supported catalyst (Vincent and Gonzalez, 2001).

2 THEORY

2.1 Kinetics

The process of acetylene hydrogenation is consisted of adsorption of acetylene and hydrogen on the catalyst surface, chemical reaction between the adsorbed species, and desorption of the products from the surface (Vicent and Gonzalez, 2001). Bond (1962) proposed that since the enthalpy of adsorption of acetylene is higher than that of ethylene, the surface coverage ratio of acetylene to ethylene would be always high. Therefore, in this case it was expected that if a mixture of acetylene and ethylene is used, hydrogenation of ethylene would not start until all the acetylene in the mixture is consumed. However, the experiments conducted by Bos et al. (1993) and Brodzinski and Cybulski (2000) indicated that this assumption is not realistic and hydrogenation of ethylene cannot be completely prevented in any case. On the other hand, Al-Ammar and Web (1978, 1979), Menshchikov et al. (1975) and Mc Gown et al. (1978) proposed that the catalyst surface contains at least two different types of active sites. Furthermore, Brodzinski and Cybulski (2000) proposed a

model based on three active sites. They suggested that these sites are created on the palladium surface by carbonaceous deposits. Some of these sites can only take part in acetylene hydrogenation and others may be open to all the species in the gas phase. According to Brodzinski and Cybulski (2000), a type site may exist which is too small for the species other than acetylene to be adsorbed on. As compared to ethylene, acetylene is selectively hydrogenated on these sites by hydrogen atoms which are also adsorbed on these sites.

Different kinetic models have been proposed based on each of the above described mechanisms. Nevertheless, due to the complexity of the reactions in this system, none of the proposed kinetics can be considered as the best, yet. Among them, the kinetic expressions proposed by Boss et al. (1993), Brodzinski and Cybulski. (2000) and Menshchikov et al. (1975) seem to be more acceptable and have been used by other researchers (Westerterp et al., 2002; Vincent and Gonzalez, 2001).

2.2 Modeling

The acetylene hydrogenation system considered in this work consists of only Reaction (1) and (2). All other side reactions are neglected. The industrial reactor of acetylene hydrogenation operates at non-isothermal conditions. Therefore, in order to model such a reactor, the mass balance equations have to be coupled with the energy balance equation and to be solved simultaneously. Up to now most of the simulation studies in this field have been based on the plug flow assumption for the reactor. Moreover, the few researchers, who have considered the dispersion model, did not report temperature and concentration profiles in a large scale reactor or make a comparison between these two models (Vincent and Godinez, 2002; Szukiewicz et al., 1998). The acetylene hydrogenation reactor has modeled by both models in this study.

The mass and energy balance equations, assuming the plug flow pattern for the gas, are as follows:

$$\frac{dC_A}{dz} = \frac{\rho_c(1-\varepsilon)}{\varepsilon u} r_A \quad (4)$$

$$\frac{F}{A} C_P \frac{dT}{dz} = \rho_c \frac{1-\varepsilon}{\varepsilon} \sum \Delta H_i r_i \quad (5)$$

The second method of simulating this system is to take dispersion of the gas into consideration. In this case, the mass balance equation should be rewritten as follows:

$$\frac{D_A}{u} \frac{d^2 C_A}{dz^2} - \frac{dC_A}{dz} + \frac{\rho_c(1-\varepsilon)}{\varepsilon u} r_A = 0 \quad (6)$$

Thermal dispersion may be neglected in this case as the ratio of thermal dispersion coefficient to mass dispersion coefficient is very low and the energy balance equation would be the same as the previous case (Eq. 5) in the modeling. Therefore, mass balance equations (Eq. 6)

should be solved for all species together with the energy balance equation (Eq. 5), simultaneously. In order to solve the mass balance equation (Eq. 6), two boundary conditions are needed for each species. In this case, Dankwerts boundary conditions may be used (Fogler, 1999) as given below:

$$z = 0; \quad C_{A0} = -\frac{D_A}{u} \left(\frac{\partial C_A}{\partial z} \right)_{z=0^+} + C_A(0^+, t) \quad (7)$$

$$z = L; \quad \frac{\partial C_A}{\partial z} = 0 \quad (8)$$

Equations (5), (6), (7) and (8) form a set of boundary-value differential equations and could be solved by the finite difference method (Constantinides and Mostoufi, 1999).

3 EXPERIMENTAL

3.1 Catalyst and Gases

The catalyst was Pd/Al₂O₃ with commercial name of G58-B from Sud-Chemie which is currently used in many petrochemical complexes. Both new and used catalysts were employed in the experiments. The used catalyst was acquired from an industrial reactor being in service for six months before getting deactivated and taken out from the reactor.

The gases used in this work were 99.65% pure C₂H₂, 99.99% pure C₂H₄ and 99.99% H₂ along with 99.95% nitrogen. The latter was used as the diluting gas to prevent high conversion of acetylene during the experiments. In order to obtain the desired concentration of hydrogen in the mixture, hydrogen gas was premixed with nitrogen at a 1:9 ratio. To make such a premixed gas, the container was first vacuumed and then filled by a calculated amount of hydrogen and then slowly pressurized with nitrogen up to 6 barg.

3.2 Apparatus and Procedure

The experimental set-up for measuring the reaction rates of acetylene hydrogenation reactions is shown in Figure 1. The U-shaped micro reactor filled with 0.3 grams of finely pulverized catalyst with a mesh of 180 to 300 μm. Flow rates of the inlet streams were measured by three rotameters. The reactor was placed in a warm water bath equipped with temperature controller and heater. Compositions of both inlet and outlet streams of the reactor were analyzed by a gas chromatograph (GC) equipped with a FID analyzer. At the beginning of each run, the feed was analyzed by the GC before entering the reactor. During the experiments, the product gas from the reactor was also conducted to the same GC for determining its composition after the reaction. The feed flow rate varied between 30 to

110 mL/min and its composition was changed from high about 25% to less than 1% of acetylene content. The experiments were carried out at three different temperatures, i.e., 30, 50 and 70 °C.

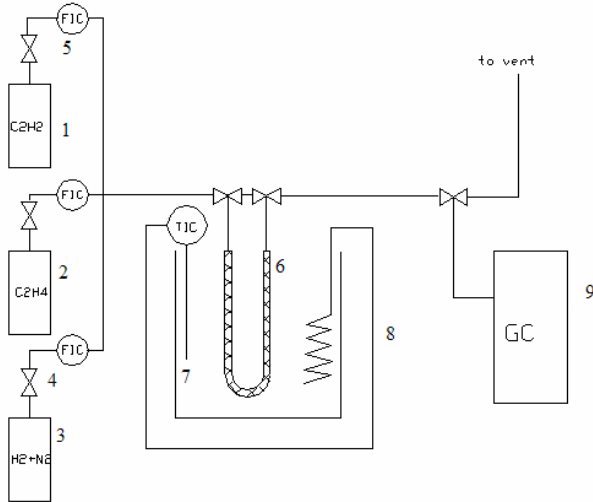


Figure 1: Simplified schematic diagram of the experimental set up of this study. 1-C₂H₂ container 2-C₂H₄ container 3- H₂+N₂ container 4-Pressure regulator 5-Rotameter 6-Reactor 7-Thermocouple and temperature indicator 8-Heater 9-Gas chromatograph.

In addition to the new catalyst, the experiments were repeated with the deactivated catalyst to obtain a deactivation coefficient for this catalyst. The experiments for the old catalyst were carried out only at 70°C because as the catalyst becomes deactivated in the reactor, the feed temperature is increased to counter this effect. The feed temperature used at industrial reactors at such conditions is usually between 70 to 80°C.

4 RESULTS AND DISCUSSION

4.1 Kinetics

Using the data of these experiments, the reaction rates of acetylene consumption and ethane formation in each case were found to be as follows:

$$r_{C_2H_2} = \frac{F_{C_2H_2(in)} - F_{C_2H_2(out)}}{m} \quad (9)$$

$$r_{C_2H_6} = \frac{F_{C_2H_6(out)} - F_{C_2H_6(in)}}{m} \quad (10)$$

The calculations and discussions done below are based on these reaction rates.

Initially, the two well known kinetic models of Bos et al. (1993) and Menshchikov et al. (1975) were considered as the base models and fitted the experimental data of this work to these models to obtain new kinetic parameters for

the catalyst employed in this study. By comparing the experimental data obtained in this work with the above mentioned models, it has been concluded (Ghoorchian, 2003) that the model of Bos et al. (1993) cannot predict the reaction rates of the catalyst employed in this study in the range and operating conditions of this study for either acetylene consumption rate or ethane formation rate. However, the model of Menshchikov et al. (1975) is able to predict the rate of acetylene consumption satisfactorily for the catalyst and conditions of this study while its rate of ethane formation still needs to be improved. Therefore, a new kinetic model is proposed here which consists of the acetylene consumption rate of the model of Menshchikov et al. (1975), for with new parameters which have been obtained in this study, and a new rate expression for ethane formation which better fits the experimental data. After simplifying, this new model is given as follows:

$$r_{C_2H_2} = \frac{48.01 \exp\left(-\frac{146.8}{T}\right) P_{C_2H_2} P_{H_2}}{\left[1 + 584.59 \exp\left(\frac{668.6}{T}\right) P_{C_2H_2}\right] \left[1 + 2.855 \exp\left(\frac{404.3}{T}\right) P_{H_2}\right]} \quad (11)$$

$$r_{C_2H_6} = \frac{202.67 \exp\left(-\frac{4784}{T}\right) P_{C_2H_4} P_{H_2}}{\left[1 + 0.0742 \exp\left(\frac{1502.7}{T}\right) P_{C_2H_4}\right]^{1.25} \left[1 + 2.89 \exp\left(\frac{400}{T}\right) P_{H_2}\right]} \quad (12)$$

4.2 Reactor Modeling

The two flow models coupled with each of the three kinetic models described in the Theory section were solved for an industrial-scale reactor. The operating conditions considered for the simulation are listed in Table 1. It is worth mentioning that in the industrial acetylene hydrogenation units, two reactors in series are employed for complete conversion of acetylene in the feed (Weiss, 1996). The values given in Table 1 are typical for the first hydrogenation reactor. Results of this simulation are shown in Figures 2a-d in terms of profiles of temperature, acetylene conversion, ethylene formation, and ethane formation along the reactor, respectively. In these figures, the results of simulation of the reactor by the two flow models, i.e., plug flow and dispersion flow, which are coupled with the kinetic model proposed in this study are shown.

Table 1: Input to simulation

Parameter	Unit	Value
C ₂ H ₂	%	4
C ₂ H ₄	%	93
C ₂ H ₆	%	1
H ₂	%	2
Reactor length	m	3

Inlet temperature	K	293
Pressure	Bar (g)	25

Figure 2a illustrates the temperature profiles of the reactor for the three kinetic models as coupled with the two flow models. It can be seen in this figure that all these models predict almost the same final temperatures for the reactor. In addition, the results of dispersion and plug models are actually close to each other. The exit temperature of the reactor is about 360 to 365 K according to all models which are close to the exit temperature of the product from the first hydrogenation reactor in the industrial acetylene converting units.

The corresponding acetylene conversion profiles are shown in Figure 2b. This conversion is calculated from the following formula:

$$X_{C_2H_2} = \frac{F_{C_2H_2}(in) - F_{C_2H_2}}{F_{C_2H_2}(in)} \quad (13)$$

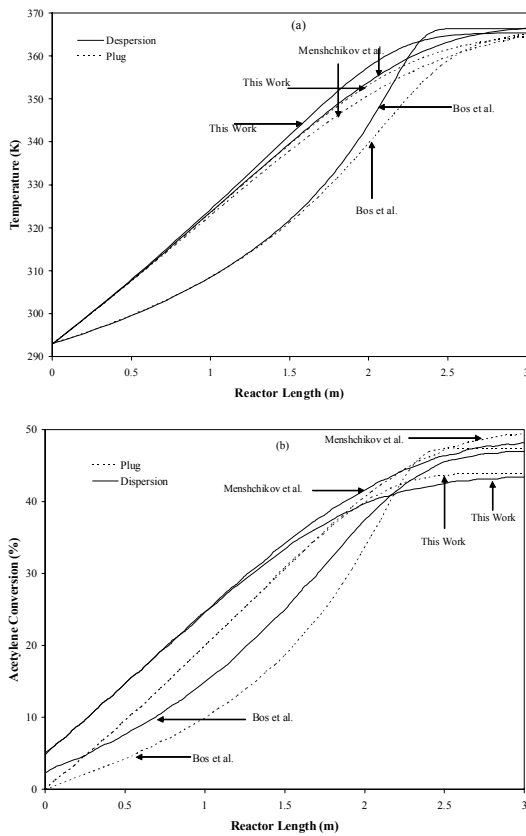


Figure 2: Simulation results for different flow patterns (a) temperature profiles. (b) acetylene conversion profiles.

It is also seen in Figure 2b that the profiles are close to each other and so do the exit conversions. This is an expected trend since all three kinetic models considered in this study provide quite the same acetylene hydrogenation rates. This figure illustrates that only about half of the acetylene is eliminated in the first hydrogenation reactor

and the rest of this task remains to be accomplished in the second reactor. The reason for not completing the conversion of acetylene in a single reactor is controlling the temperature, as discussed in the introduction section and shown in Figure 2a.

Ethylene formation can be calculated from:

$$X_{C_2H_4} = \frac{F_{C_2H_4} - F_{C_2H_4}(in)}{F_{C_2H_4}(in)} \quad (14)$$

The profiles of ethylene formation along the reactor are shown in Figure 2c. It can be seen in this figure that the kinetic model of Menshchikov et al. (1975) predicts the highest ethylene formation among the three models and the model developed in this work predicts the lowest. The difference between the predictions of the three models observed in Figure 2c is due to the fact that in the process of ethylene formation, two reaction rates (i.e., acetylene conversion and ethane formation) are involved.

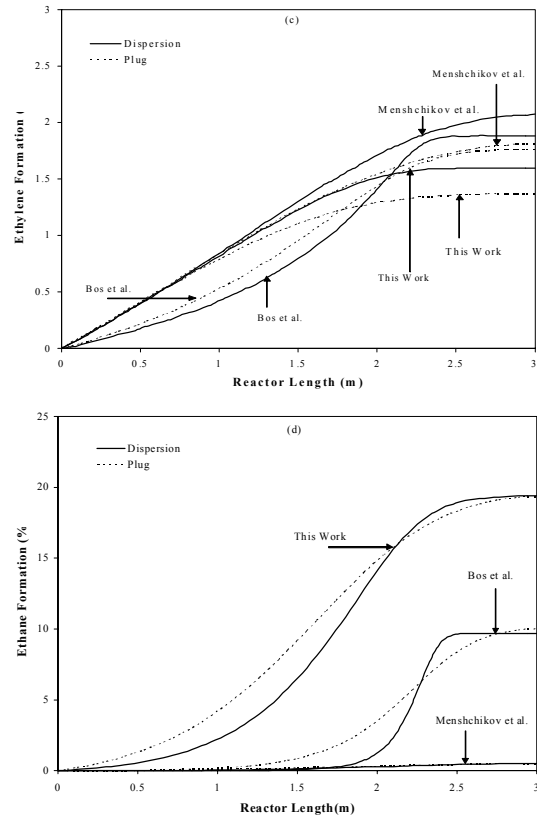


Figure 2: Simulation results for different flow patterns (c) ethylene formation profiles. (d) ethane formation profiles.

Although all three kinetic models considered in this study provide almost the same acetylene conversion rates, they are dissimilar in the rate of ethane formation. Therefore, different profiles are obtained from each kinetic model for ethylene formation. This figure also reveals that regardless of the kinetic model used in the simulation, the

plug model provides lower ethylene formations compared to the dispersion flow model. This is some thing that can be expected because in dispersion flow the back mixing phenomena helps the conversion of acetylene to be higher than that of plug flow. Consequently, the ethylene formation would be also higher in this case.

Ethane formation is calculated from:

$$X_{C_2H_6} = \frac{F_{C_2H_6} - F_{C_2H_6(in)}}{F_{C_2H_6(in)}} \quad (15)$$

Figure 2d shows the profiles of ethane formation along the reactor length for the models considered in this work. It is clear in this figure that each kinetic model predicts a different ethane formation rate as compared to another one. The discussions made for Figure 2c regarding the difference of the three kinetic models in terms of ethane formation rate are also valid here. In fact, the difference between these models, which is mainly originated from the difference in ethane formation rate, shows up noticeably in this figure. Since the reaction rates proposed in this work fits the experimental data better than the other two models (see Figures 2b, 3b and 4), the results of simulation with the new model can be more trusted for the employed catalyst and operating conditions of this simulation.

4.3 Catalyst Deactivation

As mentioned in the Experimental section, both active and deactivated catalysts were employed in this study. The new catalyst was used to find the proper kinetic for the system and the deactivated one was used to study the effect of using the catalyst for a long time and determine the deactivation coefficient for the catalyst. This deactivation coefficient can be used for analyzing the long term dynamic behavior of the acetylene hydrogenation unit and estimates the temperature evolution of the feed to the reactor during the catalyst useful life.

The rate of reaction incorporating catalyst deactivation can be obtained as follows:

$$r_{i,d} = r_i a(t) \quad (16)$$

from which the deactivation coefficient could be evaluated by using the experimental data of this work. There is no explicit expression for the deactivation rate of this catalyst in the literature. Therefore, although the future works might suggest a nonlinear relationship between the catalyst deactivation rate and the fraction of active catalyst, a first-order deactivation rate is assumed in this case:

$$\frac{da}{dt} = -k_d a \quad (17)$$

or

$$a(t) = e^{-k_d t} \quad (18)$$

The deactivated catalyst used in this study had been used in the corresponding industrial process for six months for which the deactivation coefficient was found to be

0.25. Based on this value, the deactivation rate constant is estimated to be

$$k_d = 2.772 (0.067) \quad \text{month}^{-1} \quad (19)$$

The figure in the parenthesis in Eq. (20) is the standard deviation of the calculated deactivation constant.

When the hydrogenation catalyst is used in an industrial reactor, it is gradually deactivated until it reaches the point of inefficiency. At this point, it should be replaced with fresh catalyst. However, up to this point the feed temperature is gradually being increased during the usage of the catalyst to counter the effect of deactivation. This increase in the temperature can raise the activity of the catalyst to some extent. Figure 3 demonstrates the effect of increasing the feed temperature on exit acetylene concentration with catalysts of different activities for the simulation parameters given in Table 1. The operating point of the fresh catalyst (acetylene concentration at the reactor exit for the catalyst of the activity equal to unity) is also illustrated in the same figure.

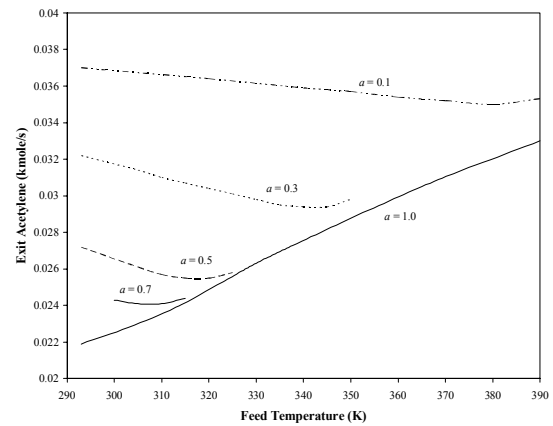


Figure 3: Effect of temperature on altering the activity of deactivated catalyst.

It is obvious from this figure that in neither case, increasing the temperature can lead to an exit acetylene concentration equal to that with a new catalyst being employed. In fact, the effect of increasing the feed temperature on the performance of a reactor containing deactivated catalyst is to decrease the acetylene concentration at the beginning, although such a concentration would not reach the concentration equivalent to the fresh catalyst. Nevertheless, further increase in the feed temperature even overturns this trend and results in decreasing the acetylene conversion in the reactor. This is because of the reverse effect of temperature on the concentration of feed components and the reaction rate. The higher the temperature, the higher will be the reaction rate but at the same time the feed concentration would become lower as the temperature increases. It is for this reason that increasing the feed temperature cannot be used

as the only way of dealing with deactivation of the catalyst during each run. Therefore, in practice, two reactors are used in series in order to help the catalyst in the second reactor of the process to reach the desired concentration of acetylene in the final product, in addition to increasing the feed temperature of the first reactor.

5 CONCLUSIONS

Selective hydrogenation of acetylene was studied in a fixed bed reactor of a commercial Pd/Al₂O₃. Using the experimental data of this work and existing kinetic models from the literature, a new kinetic expression for hydrogenation of acetylene was developed. The acetylene hydrogenation reactor was simulated with different flow models (i.e., plug flow and dispersion flow models) coupled with three different kinetic models (i.e., Bos et al., 1993; Menshchikov et al., 1975) and the new model developed in this study). It has been shown that although the profiles along the reactor length could be different, in most cases the differences between plug and dispersion flow models are small in terms of reactor outlet quantities. The effect of deactivation of the catalyst was studied experimentally with a used catalyst and the deactivation rate constant of the catalyst was evaluated. It was demonstrated by simulation that it is necessary to employ two hydrogenation reactors in series due to the following reasons:

(a) Practical temperature control of the reactor: Hydrogenation of the whole acetylene in the feed would result in an unacceptable increase in the temperature of the outlet of the reactor if a single reactor is to be employed.

(b) Reaching the desired exit concentration of acetylene: While the catalyst gets deactivated over the time, it is not possible to overcome the deactivation of the catalyst only by increasing the feed temperature, thus, a second reactor is needed to complete the process of acetylene hydrogenation up to the desired exit acetylene concentration.

NOMENCLATURE

a fraction of active catalyst
 A cross section area (m²)
 C_A concentration of component A (kmole/m³)
 C_p specific heat (J/kmole.K)
 D_A dispersion coefficient of component A (m²/s)
 E_a activation energy (J/kmole)
 F total molar flow rate of feed (kmole/s)
 F_i molar flow rate of species i (kmole/s)
 ΔH_j heat of reaction of reaction j (J/kmole)
 k reaction rate constant
 k_0 frequency factor
 k_d deactivation rate constant (s⁻¹)
 L reactor length (m)

m mass of catalyst (kg)
 P pressure (Pa)
 R gas constant (J/kmole.K)
 r_i reaction rate of species i (kmole/kg cat.s)
 $r_{i,d}$ reaction rate of species i for a deactivated catalyst (kmole/kg cat.s)
 t time (sec)
 T temperature (K)
 u superficial velocity (m/s)
 X conversion
 z distance along the reactor (m)

Greek Letter

ε bed voidage
 ρ_c catalyst density (kg/m³)

Subscripts

in inlet
out outlet

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