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8.0 MASS TRANSFER AND KINETICS FOR FISCHER-TROPSCH REACTION SYSTEMS

The Fischer-Tropsch kinetics is complex due to interaction of large number of parameters such as pressure, temperature, fresh gas composition, residence time, heat and mass transfer, and catalyst (including catalyst type, catalyst support, catalyst activation during preparation and catalyst age). The composition of the reaction products of Fischer-Tropsch synthesis (ratio of alkanes to alkenes, chain length distribution, formation of water or carbon dioxide) is kinetically controlled and does not correspond to the composition predicted by thermodynamics [Bussemeier et al., 1986]. According to thermodynamics, the main products should consist of methane and carbon dioxide [Storch et al., 1951]. The hydrogenation of CO is achieved by simultaneous chemisorption of H_2 and CO on metallic sites of the catalyst surface. H_2 is believed to be chemisorbed dissociatively, whereas, CO is believed to be bounded to the catalyst metal (Co, Fe, or Ru) by C-metal bond. This bond results in weakening the C-O bond, ultimately resulting in hydrogenation of the CO molecule.

In heterogeneous catalysis, such as the Fischer-Tropsch synthesis, interactions of CO and H_2 with the catalyst particle are important and any factor hindering such an interaction may result in the reaction being diffusion limited. The mass transfer of the reactants to the external catalyst surface and the desorption of the products are believed to be relatively fast and do not determine the net reaction rate [Frohning et al., 1982]. This is explained by relatively high activation energy for the FT reaction (20 to 25 kcal/mol). However, under normal FT conditions the activity of the catalyst is dependent on the particle size of the catalyst [Dry, 1981]. This indicates that mass transfer within the pore structure of the catalyst is of considerable importance. The reactants dissolve in this liquid and diffuses into the catalyst pores where they react. The reaction creates a concentration gradient leading to an increase in the diffusion. Thus, the kinetics of the reaction and diffusion are coupled. Within the catalyst particle, the concentration of the

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reactants decrease (due to the reaction) and that of products increase as the distance from the catalyst surface increases. High concentration of products such as CO_2 and H_2O results in the oxidation of the inner catalyst surface. Since the catalyst is active only in reduced state, only the area surrounding the pore opening (up to a depth of 0.1 mm) remains active [Anderson et. al., 1964].

The FT reactions occur via a complex path and thus the published equations for the FT and the related methane synthesis do not present a uniform picture. Table 8.1 summarizes various kinetic equations reported for each of the four main metal catalyst [Dry, 1981]. Table 8.1 indicates that the order of reaction with respect to hydrogen varies from zero to two, and for CO from one to minus one. Vannice (1976) in his review reports that when the H_2/CO molecular ratio is between one and three, the order with respect of H_2 is about one and the order with respect to CO is between zero and minus one half. The partial pressure of water plays an important role in case of Fe catalysis due to the water-gas shift reaction. If water competes with CO and H_2 for catalyst sites, then it plays a role in the kinetics, this has been seen in case of Ni catalyst by Saletore (1977). Variation in the rate expression reveals the complex nature of the reaction. For Co, Fe or Ru catalysts, the apparent activation energies cover the range from 20 to 25 kcal/mol, indicating that the FT reaction is not diffusion limited. Dry (1981) reports that for FT reaction over iron catalyst, the rate is proportional only to the partial pressure of H_2 in case of a differential reactor; whereas in an integral reactor the rate is proportional to $pH_2 \cdot pCO/(pCO + a \cdot pH_2O)$; where a is a constant obtained from regression. Thus information generated in differential reactor would be of little value for designing commercial integral reactors.

When the F-T reaction is carried out in a slurry reactor syngas is bubbled through a slurry of finely divided catalyst suspended in heavy-oil medium whose composition may or may not change with time depending upon the product selectivity variation and time. In this three phase reactor, a discontinuous gas phase in form of bubbles moves relative to a continuous "homogeneous" slurry phase. The syngas reactants

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TABLE 8.1 Some Kinetic Equations for CO Hydrogenation over Various Metal Catalysts [Dry, 1981].

Metal	Kinetic equation	Ref. No
Ni	$r = kp_{CO}p_{H_2}^{0.5}$	[185]
	$r = kp_{CO}'(1 - kp_{CO})^2$	[186]
	$r = kp_{H_2}^{0.9}p_{CO}^{-0.2}$	[187]
	$r = kp_{H_2}^{0.25}p_{H_2O}^{-0.9}$	[188]
Co	$r = k$ (i.e. independent of reactant pressures)	[189]
	$r = kp_{H_2}^2 p_{CO}$	[190]
Fe	$r = kp_{H_2}^{0.5}p_{CO}^{0.6} - fp^{0.5}p_{H_2O}^{0.5}$	[56]
	$r = kp_{H_2}/(1 + ap_{H_2O}/p_{CO})$	[2]
Ru	$r = kp_{H_2}^2$	[191]
	$r = kp_{H_2}^{1.33}p_{CO}^{-0.33}$	[192]
	$r = kp_{H_2}^2 p_{CO}(1 - mp_{CO} + np_{H_2})^3$	[193]
	$r = kp_{H_2}^{1.5}p_{CO}^{-0.6}$	[157]

[185] = Lee et al., 1970
[186] = van Herwijnen et al., 1973
[187] = Luyten and Jurgens, 1945
[188] = Saletore and Thomson, 1977
[189] = Anderson et al., 1949
[190] = Brotz, 1949
[56] = Anderson et al., 1964
[2] = Anderson, 1956
[191] = McKee, 1967
[192] = Karn et al., 1960
[193] = Phung Quach and Rouleau, 1978
[157] = Ekerdt and Bell, 1979

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must diffuse through the liquid phase to the catalyst surface in order for the F-T synthesis to occur. The general approach has been to consider the specific mass transfer and kinetic rate processes that are taking place.

In the slurry, the effective H_2/CO ratio the catalyst sees is controlled by the vapor-liquid equilibrium, chemical reaction rate and mass transfer rate. Due to the reaction stoichiometry and the difference in the rates of diffusion for CO and H_2, the H_2/CO ratio at the catalyst surface may be different than the corresponding ratio in the inlet gas stream. Akgerman (1990) has examined variation in the H_2/CO ratio at the catalyst surface and the inlet gas stream as a function of the rate controlling step. If the reaction is slow and is the rate determining step, then the concentration is uniform and the effective H_2/CO ratio the catalyst surface sees is the ratio of solubilities of H_2 and CO in the liquid phase. Based on the available information in the literature, Akgerman (1990) estimated the solubility ratio of H_2/CO to between 0.5 and 1.12 (depending on the values of the Henry's Law constants) for a gas stream with H_2/CO ratio of 0.7 Thus for a chemical rate controlling the reaction, the effective H_2/CO concentration the catalyst surface sees is not much different from a gas phase reaction.

On the other hand, if the reaction is mass transfer controlled, then the effective ratio of H_2/CO at the catalyst surface depends on the diffusion coefficient of H_2 and CO in the liquid. Assuming that the ratio of the diffusion of H_2 to CO is 3.0, Akgerman (1990) estimated the mass flux ratio of H_2/CO to be between 1.5 and 3.36 (depending on the values of the Henry's Law constant) for a gas stream with H_2/CO ratio of 0.7. Thus for a mass transfer controlled reaction, the catalyst sees a higher H_2/CO ratio than in the gas phase. If the process is gas-liquid mass transfer controlled, then the H_2/CO ratio at the catalyst surface depends on the mass transfer coefficients of H_2 and CO. Based on various correlations for mass transfer coefficients available in the literature, Akgerman (1990) estimates the ratio of mass transfer coefficients of H_2/CO to be between 1.5 and 2.0 (depending on the mass transfer coefficient calculation) for a gas stream with H_2/CO ratio of 0.7. Thus for a gas-liquid mass transfer control, the H_2/CO ratio at the catalyst surface is higher than the ratio in the gas phase.

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Since the rate of mass transfer from the gas-liquid interface is proportional to the interfacial area, both the solids loading and bubble hydrodynamics are extremely important. Details of the bubble hydrodynamics is discussed in Section 6.0. An increase in the solids loading has been reported to result in a decrease in the gas-liquid interfacial area and results in a decrease in the effective rate of mass transfer [Akgerman, 1990].

The intrinsic kinetics associated with various F-T slurry reactions have been presented by Kuo (1981 and 1983) and Satterfield et al. (1983) and are summarized in Table 8.2 [Saxena et al., 1986]. In Kuo's (1981) expressions, r_{H_2} is the hydrogen conversion rate. The Satterfield et al. (1983) expression gives the rate of H_2 plus CO conversion based on the following F-T reaction:

$$n\ CO\ +\ (m+n)\ H_2 \longrightarrow C_nH_{2m}\ +\ n\ H_2O$$
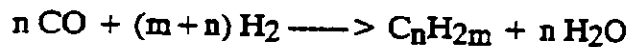
where n is 3 and m is 3.5, based on the average organic product composition of C_3H_7. At low conversion, the partial pressure of H_2O is negligible and the Satterfield et al. equation in Table 8.2 simplifies to the first order form proposed earlier by Anderson (1956). Kuo (1983) considered two consecutive reactions, the Fischer-Tropsch reaction (r_1) followed by the water-gas shift reaction (r_2). Earlier Kuo (1981) had included the loading factor for the active component (Fe) to stress the significance of the water-gas shift reaction. The interpretation of the intrinsic kinetic rate constant requires the concentration of the reactants in the liquid phase, catalyst loading, residence time of the reactants, the degree of mixing of the fluid phases and the order of reaction [Saxena et al., 1986]. Since the order of reaction with respect to H_2 changes with the extent of conversion, Saxena et al. (1986) recommend caution when applying intrinsic rate expressions to conversion results in a F-T slurry reactor, especially at high conversions.

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TABLE 8.2. Intrinsic Kinetic Rate Expressions for Fischer-Tropsch Catalyst. [Saxena et al., 1986].

Investigators	Kinetic Expression	Rate Constant
Satterfield et al. [1983]	$r_{H_2-CO} = a'b'P_{CO}P_{H_2}^2/(P_{H_2O} + b'P_{CO}P_{H_2})$	$a' = 2.39 \times 10^8 \exp(-19700/RT)$ $b' = 9.50 \times 10^8 \exp(-24000/RT)$ $a' = \mu mol/g \cdot cat\text{-}min\text{-}kPa$ $b' = kPa^{-1}$ $T = K, R = 1.987 \; cal/K$ P_{CO} = Partial pressure CO, kPa P_{H_2} = Partial pressure H_2, kPa P_{H_2O} = Partial pressure H_2O, kPa
Kuo [1981]	$r_{H_2} = k_c^-(1 - \varepsilon_g)(1 - V)w_{Fc}C_{HL}$	$0.84 < k_{H'} < 2.3$ ε_g = Gas holdup, cm^3 gas/cm^3 expanded slurry V = Volume fraction catalyst in slurry, cm^3 catalyst/cm^3 slurry w_{Fc} = Iron concentration in liquid phase, gFe/cm^3 liquid C_{HL} = Liquid phase H_2 concentration, mol H_2/cm^3 liquid k_c^- = cm^3 liquid/g Fe-s
Kuo [1983]	$r_1 = k_1[H_2][CO]/([CO] + k_3[H_2O])$ $r_2 = k_2 \{[CO][H_2O] - [H_2][CO_2]/k_4\}/$ $([CO] + k_3[H_2O])$	$k_1 = 0.50 \; cm^3$ liquid/g Fe-s $k_2 = 1.35 \; cm^3$ liquid/g Fe-s $k_3 = 0.2$ $k_4 = 37.5$ [ ] = Volumetric concentration in liquid phase

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9.0 CATALYST WAX SEPARATION

One of the challenges of operating the slurry reactors is the separation of sub-micron to 50 micron sized catalyst particles from the product wax. The separation of catalyst-wax is necessary to avoid catalyst loss and avoid problems in wax upgrading. Catalyst-wax separation may be performed inside or outside the slurry reactor [Zhou, 1991]. Zhou (1991) has recently reviewed various catalyst-wax separation techniques and concluded that internal filters are subject to plugging risks and not suited for commercial plants. The concept of using high gradient magnetic separation has a potential of being used inside or outside the reactor. This technique is feasible as F-T catalysts are ferromagnetic. Preliminary results indicate the solids content can be reduced down to less than 0.015 wt% [Zhou, 1991]. In a laboratory scale study, Mobil demonstrated that the solids content can be reduced from 0.13 wt% to less than 0.015 wt%. Thus the magnetic separation technique can be employed as a catalyst-wax separator and also as a polishing technology. Although the magnetic separation technology has been demonstrated to a limited extent at the laboratory scale and holds the potential for success at the commercial scale, it is expensive and the technology is still in infancy.

Other potential techniques to separate catalyst-wax mixture include: (a) vacuum distillation and thermal cracking of vacuum bottoms, (b) chemical conversion of iron catalyst, (c) sedimentation by gravity, (d) centrifuges, and (e) pressure filtration. The first two techniques are unsuitable for F-T synthesis as it results in loss of catalyst. Sedimentation, centrifuges and pressure filtration are more suited for catalyst-wax separation in the F-T synthesis. Gravity sedimentation or settling is a commonly used simple and inexpensive device for catalyst-wax separation. This usually requires a long settling time of 1 to 3 hours to reduce solids content of reactor wax down to 0.1 wt% [Zhou, 1991]. The British Greenwich F-T pilot plant operated a gravity settling system fairly well [Farley and Ray, 1964]. However the efficiency of separation was low and was considered to be due to hindered agglomeration. The mechanical difficulties of operating the pilot plant were mainly those of efficient separation of catalyst and wax to maintain a constant level in the reactor.

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Mobil used an on-line catalyst settling system and believes that it was demonstrated [Kuo, 1985]. Mobil's on-line catalyst-wax separation system can continuously withdraw up to 7 kg of clean reactor wax containing less than 0.1 wt% solids per day. The residence time in the settler is less than 3 hours. Mobil also noticed an increase in the catalyst size and is believed to be due to agglomeration or by the growth of heavy polymers on the outside of the catalyst particle. But the mechanism for the particle growth remains unclear [Kuo, 1985]. The particle growth was not reported in the British study [Farley and Ray, 1964]. Gravity settling can remove bulk of the catalyst from the wax, but a complete removal of catalyst particles could take an unacceptable long time. According to Zhou (1991), essentially no study was specifically specifically done on settling performance of the F-T catalyst-wax system.

Hydroclones offer a simple and inexpensive means of solid-liquid separation. Hydroclones have been used in the petroleum refining industry in somewhat similar fashion were catalyst fines are separated from the fractionator bottom slurries. Hydroclones have an advantage as the same size units employed in the pilot plant can be used in parallel in commercial plant, so there is no problem associated with scale-up. Hydroclones do not produce an overflow with very low solids content and therefore the overflow needs to be further clarified through other means, such as filtration to meet the solids content requirement for wax processing. Hydroclones may be used to recover bulk of the catalyst from the slurry stream to be recycled back to the reactor. A combination of hydroclone and filters appears the most feasible method to remove the solids from the wax in cost effective manner. Mobil had proposed such a system for removal of catalyst in their conceptual process design [Kuo, 1985]. However, the hydroclone system has not been demonstrated for F-T slurry separation [Zhou, 1991]. An investigation of the hydroclone efficiency, reliability and economics for the slurry F-T catalyst-wax separation is desirable.

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Centrifuges are similar to the hydroclones in the mechanism of separation, expect that external energy is used to generate the centrifugal force required for catalyst-wax separation. This is usually a batch operation and is not preferred for continuous use in commercial operations.

Pressurized filtration is another important solid-liquid separation technique. The advantages of filtration is that it has a high efficiency of solids separation with reasonable particle size. However, poor mechanical history, high capital and operating costs, intermittent operation and difficulty in catalyst recovery makes filtration undesirable to remove solids from a slurry with a high solids loading. However it is an excellent device for small scale operation and for use in series with hydroclones or settlers. Pressurized filters have been used in the Rheinpreussen-Koppers Demonstration Plant [Kolbel and Ralek, 1980].

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10.0   FUTURE DIRECTION

The F-T reactions are highly exothermic and non-nselective. The exothermicity of the reaction can be handled by a proper reactor design and depending on the H_2/CO ratio of the feed and the H_2/CO usage ratio, an appropriate reactor and catalyst with/without CO shift can be selected. The non-selective nature of the F-T reaction can be reduced by improvements in catalyst formulations which should be capable of yielding reproduceable results. Precipitated iron catalysts with promoters are perhaps the most promising catalyst. Programs funded by PETC and presentations at the Proceedings of the Liquefaction Contractors' Review Meeting (September 1991) indicate that significant efforts are in progress towards development of a poison resistant and selective catalyst with high activity, longevity and attrition resistance.

The source of H_2/CO for most demonstration and commercial facilities has been coal. Advanced coal gasifiers produce H_2/CO ratio of 0.5 to 0.67. The ratio can be modified to suit the H_2/CO usage ratio in gas phase reactor by adding a shift convertor prior to the reactor. The use of shift convertor is not necessary for slurry phase reactor as the catalyst will have CO shift capability.

The direction of the F-T reactor technology can be inferred from the work conducted at Sasol. The fixed bed ARGE reactor and the entrained bed reactor technologies have been commercially proven in mid 1950's. Although these technologies were proven, Sasol developed and commercialized bubbling fluidized bed technology in 1989 [Dry, 1990]. This development was undertaken to improve the economics of the technology. More recent information from Sasol i: :icates that the development of bubbling fluidized bed has been discontinued in favor of slurry reactor design. These developments indicate that slurry reactor promises to be the technology of the future for F-T synthesis. The most comprehensive study on the slurry reactor, other than the original Kolbel's work, has been made by Mobil. Mobil's work was hampered by a variety of operational problems and only

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a few runs lasted long enough to obtain reliable information on the product distribution and catalyst deactivation [Srivastava, et al., 1990].

Air Products and Chemicals is currently operating a process development unit at LaPorte, which is capable of producing about 9 tons/day of methanol in slurry phase reactor of 22 in ID. Air Products plans to operate this unit for F-T synthesis in mid 1992. The F-T test to be conducted at LaPorte is expected to provide additional insight into the future direction of the slurry reactor technology.

There are engineering problems associated with the design and scale-up that need to be resolved. These include: exploring the fouling of heat exchange tubes due to catalyst deposition, catalyst separation from the wax and a better understanding of heat and mass transfer and hydrodynamic parameters. The ability to understand and engineer these factors into a workable reactor design are critical to the commercialization of the F-T slurry reactor technology.

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11.0 SCALE-UP OF BUBBLE COLUMN FISCHER-TROPSCH REACTORS

The scale-up of a slurry reactor is a rather complex problem since in most cases reactor performance depends significantly upon the prevailing hydrodynamics, transport and mixing characteristics of the reactor. As discussed earlier (Section 6.0), the variations in these characteristics with the reactor scale-up are not well understood [Shah and Deckwer, 1985]. The applicability of the hydrodynamic and mixing models used for correlating the reactor performance can also depend on the scale of the reactor. Since the apparent reaction rate depends upon various transport resistances, as discussed in Section 8.0, the controlling resistance can also depend on the scale of the reactor. This implies that the reactor performance model used for small-scale reactor may not be useful for the large-scale reactor. On the other hand, German researchers have been successful in scaling up the laboratory scale reactor from 6 L suspension to 10,000 L suspension in a demonstration unit [Kolbel and Ralek, 1980]. The most difficult task was reported to be the uniform distribution of gas when the ratio of length to diameter of the reactor was decreased from 60:1 to 6:1.

The proper design and scale-up of a slurry reactor is facilitated by a good reactor design. A procedure normally followed for this purpose is shown in Figure 11.1 [Shah and Deckwer, 1985]. Application of the procedure requires the definition of throughput, nature of the reaction system, and the product yield structure desired. In addition it is desirable to have specifications on reactor geometry, adjustable reactor operating conditions and process data. The desired production rate, reactor geometry and the process data fix the bounds of the adjustable operating condition such as phase velocities, temperature, pressure and the direction of flow. The hydrodynamics is dictated by the reactor geometry, process data and adjustable operating parameters.

In design of a multiphase reactors, the non-adjustable are important parameters. These non-adjustable parameters include phase holdup, the interfacial areas, the heat and mass transfer properties, and the dispersion coefficients or the mixing parameters. These parameters are also dependent on the reactor geometry,
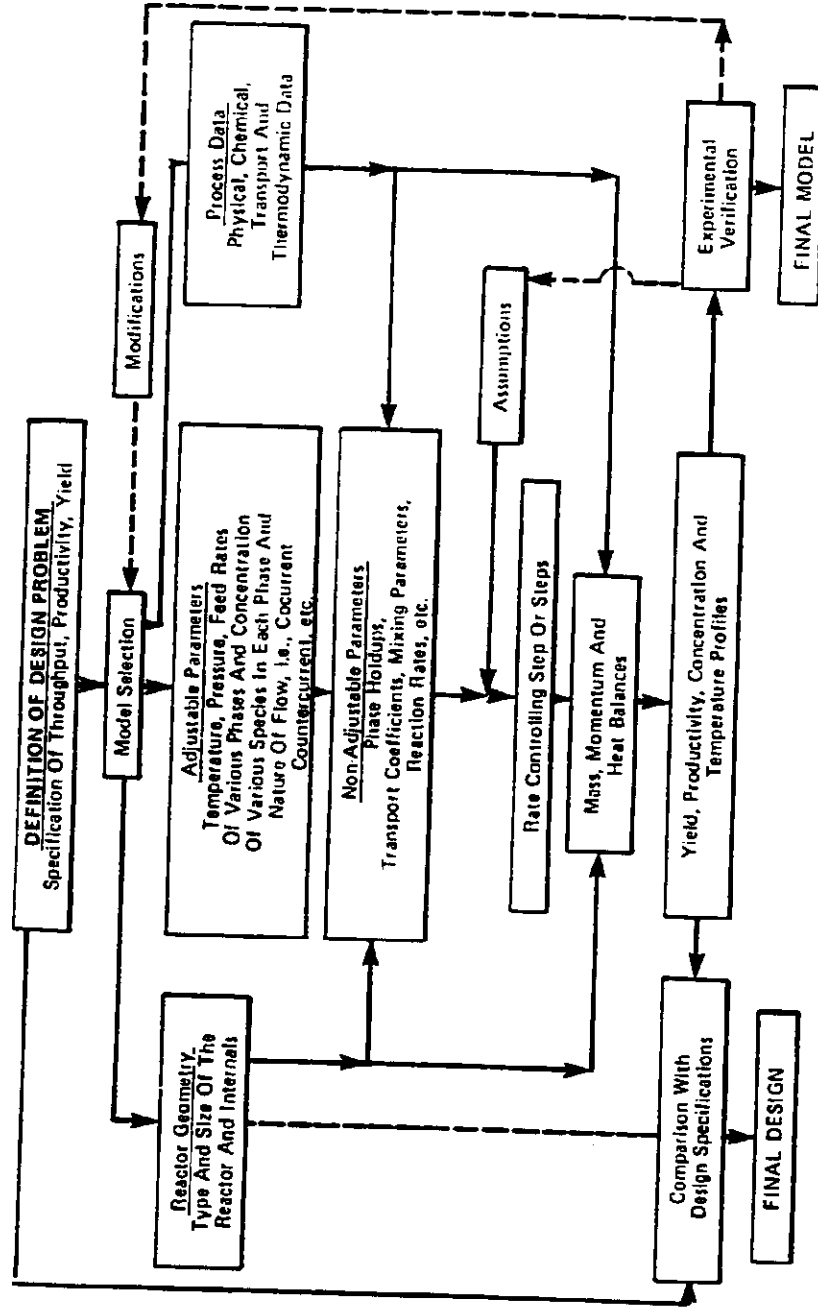
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FIGURE 11.1. A Flow Path for the Design and Modeling of Reactors [Shah and Deckwer, 1985].

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adjustable operating conditions and process data. Various methods of estimating these parameters have been discussed in detail in Section 6.0.

Based on the physical and chemical phenomena occurring within the reactor, reactor geometry and reaction parameters, the adjustable and non-adjustable, the reactor model equations are derived. As mentioned in Section 6.0, a number of researchers have been involved in developing reactor models to design slurry reactor for F-T synthesis [Bukur et al., 1990, Deckwer et al., 1980, Kuo, 1985, Saxena et al., 1986, Shah and Deckwer, 1985]. Work is also at progress at Viking Systems International to develop a model to design slurry reactor for indirect liquefaction [Prakash and Bendale, 1991]. The models can then be used to scale-up the reactor. Reactor scale-up usually implies an increase in the reactor diameter, length, and gas velocities. These changes are accompanied by changes in the flow regime, phase holdup characteristics, axial and radial mixing and transport coefficients (Section 6.0). Shah and Deckwer (1985) have reviewed the scale-up of slurry reactors and summarized the effect of scale-up variables on the non-adjustable parameters, and is shown in Table 11.1. Table 11.1 shows the effect of increase in a number of independent scale-up variables on the hydrodynamic, transport and mixing characteristics of the bubble column.

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TABLE 11.1 General Trends of Reactor Scaleup Variables on Bubble Column Reactor Dynamic Variables [Shah and Deckwer, 1985].

Effect on/Increase in	Reactor Length	Reactor Diameter	Gas Velocity	Liquid Velocity*	Solids Concentration
Flow regime	Generally no change except near transition boundary	May change	May change (around 5-10 cm/sec)	Most likely will not change under practical range of operation	May change at high solids concentration and for large particle size
Gas holdup	For very tall bubble columns holdup may decreases due to coalescence	Essentially no change	Increase	Essentially no change	May change-effect is complex and not well known yet
Gas-liquid interfacial area	May decrease in tall bubble column	Essentially no change if proper gas distributor is used	Increase	Essentially no effect unless liquid velocity is large	Effect not known - depends on particle size and concentration- decreases for larger particles and concentrations
Volumetric gas-liquid mass transfer coefficient ($k_l a$)	May decrease along column length	Essentially unchanged	Increase	No or small change	Effect is quite complex- depends on particle size and concentration usually decreases
Gas-phase dispersion	No observed effect	Increase	Increase	No observed effect	Not known - most probably only small effect under the practical range of operations
Liquid-phase dispersion	Observed increase along length	Increase	Increase	No observed effect	No effect under most practical conditions - may show effect for large particles
Solid-phase dispersion	No observed effect	Increase	Increase	No observed effect	Small effect at large concentrations
Slurry-wall heat	No observed effect	Increase	Increase	Some increase	Small increase may show optimum

*Generally liquid velocity range of 0-3 cm/sec is considered to be of most practical importance. Gas to liquid velocity ratio in most practical operations is of the order of 10 to 1.

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12.0   RECOMMENDATION

For the proposed pilot plant, the objective is to convert syngas generated from coal to hydrocarbon fuels. The H_2/CO ratio generated from modern coal gasifiers is in the range 0.5 to 0.67. Syngas rich in CO is unsuitable for fixed bed reactors as this leads to carbon deposition and different H_2/CO usage ratio. Thus for a fixed bed reactor a CO shift reactor would be needed to increase the H_2/CO ratio, in order to match the H_2/CO usage ratio with the feed H_2/CO ratio and to minimize catalyst deactivation due to carbon deposition. This would result in added cost. Use of slurry reactor, on the other hand, eliminates the need for the CO shift reactor. Furthermore when compared to a fixed bed reactor, a slurry reactor, has a high conversion per pass, requires about one fourth the cooling surface area, can operate over a wide temperature range (200 to 350°C compared to 200 to 250°C for fixed beds), and for a given throughput the volume of the reactor system is lower. Heat transfer limitations of the fixed bed also results in thermal gradients causing coke formation and catalyst deterioration. Due to low conversion and heat transfer limitations, gas recycle is required for fixed bed systems. This results in additional costs due to recompression of gas. Another drawback of the fixed bed reactor is that for catalyst regeneration and reloading of fresh catalyst requires shut down of the system. Whereas, a slurry reactor this step can be conducted without interruption. Thus both the capital and operating costs of the slurry reactor system is much more attractive, when compared to the fixed bed system. A review of the literature indicates that most of the current work on catalyst development is geared towards the slurry reactor technology. Very little has been reported in the recent literature on the development of catalyst for fixed bed reactor technology. Based on these findings, building of the fixed bed reactor system for the proposed pilot plant study is not recommended.

The three critical factors needed to further develop and commercialize the slurry phase F-T technology are: (1) the compactness of the reactor, (2) improvements in catalyst activity and selectivity, and (3) catalyst-wax separation. It is recommended

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that a more active and more selective catalyst such as those formulated by Air Products should be investigated. Further work needs to be done in catalyst pretreatment procedures, promoters, and examination of its effect on conversion, product distribution and selectivity. Improvements in the physical attrition, activity and poison resistance properties of the catalyst is also needed.

Based on the information available in the literature, the following conditions are recommended for operating and designing the slurry reactor:

Conditions	Operating Range	Normal Operation
Temperature, °C	200-380	250
Inlet Pressure, psig	300-550	400
Space Velocity, Nl/hr-kg Fe	1200-6000	3400
Catalyst Loading, wt%	4-50	15
Superficial Gas Velocity, cm/s	3-30	10

For compactness of reactor an increase in conversion along with high solids loading and higher superficial gas velocities is required. Although solids concentration of up to 20 wt% has been used, work at Air Products indicates that upto 50 wt% is feasible for methanol synthesis. Over 35 wt% solids has been theorized to make the slurry reactions mass transfer limited. For F-T synthesis it is expected that the solids loading will be less than that of methanol synthesis. Hence the recommended range for solids loading is 4 to 50 wt%. Increased solids loadings can result in an increase in viscosity of the suspension causing a decrease in the interfacial area and ultimately resulting in reduced conversion. Use of bimodal catalyst size distribution should also help in reducing the slurry viscosity.

Superficial gas velocities of up to 12 cm/sec have been successfully used in the F-T synthesis without loss in conversion. Air products has used superficial gas velocities of up to 22 cm/sec for methanol synthesis. With improvements in catalyst activity and reaction time, and with improvements in technology to limit

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backmixing, it is expected that velocities of up to 30 cm/sec should be possible. Therefore the recommended range for superficial gas velocity is 3 to 30 cm/sec.

A commercial slurry bed reactor would operate in churn turbulent flow regime. Based on the information available, the recommended diameter for the pilot plant slurry reactor should be at least 4" ID to avoid wall effects. The L/D ratio in the pilot plant studies is generally greater than 35, while in commercial reactor the L/D ratio is in the range 5 to 10. The recommended L/D ratio for the slurry reactor is between 40 and 50 for the proposed pilot plant.

The design of gas distributor for smaller and uniform bubbles is critical. Use of sintered metal plate has been found to be capable of producing small and uniform bubbles compared to gas nozzles. However, sintered metal plates require a greater pressure drop.

Catalyst-wax separation is another important factor that needs to studied. Three methods: (1) settling, (2) pressure filters and (3) hydroclones followed by pressure filters, appear to be promising conventional methods and needs to be evaluated. Catalyst particle size, slurry viscosity and differences in the densities of catalyst and wax determine the efficiency of separation. Since the catalyst size is of the order of sub-micron to micron range, use of solvent extraction coupled with the aforementioned physical separation is recommended for further evaluation.

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APPENDIX A: DISCUSSION WITH PROF. DRAGOMIR BUKUR

On 17 July 1991, a discussion was held with Prof. Bukur current status of bubble column slurry reactor technology for Fischer-Tropsch (F-T) synthesis, major design parameters, future direction, key technical issues to be resolved before commercialization occurs, and Kellogg's reactor concept. Almost 2-1/2 hours were spent on the subject. A summary is outlined below:

- Bukur thinks that a slurry F-T reactor should have a high superficial velocity--it is almost a necessity in cases where a high solid concentration is to be used. A magnitude of 8 to 10 cm/sec or even higher can be used. Regarding the effect of velocity on bubble size, it has been established that bubble size increases with increasing velocity and that larger bubbles are not desirable for enhanced mass transfer. But, there will always be smaller bubbles present in the swarm. Therefore, higher superficial velocity is the direction to take.

- He said that slug flow occurs only in small diameter reactors and should not be a concern for an 8" diameter PDU proposed for PETC.

- Catalyst pretreatment is very important, but no well-defined procedures have emerged yet. Pretreatment methods would most likely vary from catalyst to catalyst. Storage behavior of pretreated catalysts has not been studied. Bukur believes that it should be possible to store activated catalyst, if it is kept under reducing or inert atmosphere with wax. He does not expect storage temperatures to have any effect on activity. Pilot plant work by Mobil indicates need for pre-activation even for make up catalysts.

- Mobil's reported axial variation of catalyst concentration must be evaluated in the context of other variables. With an appropriate choice of conditions, such variation can be eliminated.

- Causes of bed slumping, observed in Mobil pilot plant work are unknown. This is related to catalyst agglomeration, which is a complex phenomenon and not well-understood at all. Furthermore, settling tendency is a strong function of particle size, more so than velocity.

- Foaming: He said that foaming decreases with an increasing superficial velocity and reactor diameter. He believes that foaming should not be a concern in design of an Indirect Liquefaction PDU.

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- Attempts are being made to develop new catalysts (Fe-containing zeolites) to control Schulz-Flory distribution up to a carbon number of 11. Supported catalysts have also been tried. These new catalysts may have promise but he does not know. UOP is active in new catalysts development.

- Rheinpreussen demonstration work is a bench mark in this technology. Others have confirmed most of Rheinpreussen's results except catalyst selectivity. Indeed, Mobil repeated these observations, but could not reproduce selectivity.

- His major concern regarding commercialization of Fischer Tropsch technology is separation of catalyst from wax product. Use of larger particles may not help as catalyst disintegrates during reaction. The catalyst size, therefore, decreases with a prolonged operation. Millipore is developing new filters which should be evaluated for possible applications with F-T technology.

- Bukur had following comments on MWK's suggested reactor design:

  New design concept, uncharted territory, which, to his knowledge, has not been studied by any group. Although design and scale-up principles of distillation towers are well known, this is a reactor with relatively high concentration of solids. It is not a distillation column.

  Internals (trays) and pumpback arrangement is complicated when compared to the simple bubble column reactor.

  Effect of catalyst removal from reactor and pumping on catalyst performance is a major concern to him. He feels that catalyst should not be removed from the reaction environment. By doing this, catalyst may get deactivated. Attrition due to pumping is another concern.

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APPENDIX B

LAPORTE METHANOL PLANT VISIT:

SUMMARY OF DISCUSSION AND OBSERVATIONS

On 13 Aug 1991, Kellogg members from PETC Pilot Plant project visited Air Products/DOE Alternate Fuels Demonstration Unit (AFDU) in LaPorte in order to obtain information regarding design features, Air Products' experience, and operating problems, if any. Such information would be useful in designing the pilot plant and avoiding potential pitfalls. Members from Kellogg and Air Product employees who met are as follows:

Kellogg	Air Products
R. Daze	Ed. G. Heydorn, LaPorte Pilot Plant, Manager, Host
G. Henningsen	John L. Henderson, Technology Manager
P. Sadhukhan	

Mr. Henderson took us around the plant. Outline of the discussion and observations from the tour are summarized.

- Air Products experience spans about 8 years.

- Early use of bubble caps as gas distributors did not work well - large bubbles and bubble coalescence were the main problems.

- Sparger worked very well - generating small bubbles, which are desirable. Also, with sparger, no plugging problems encountered. When spargers are restarted following catalyst settling during shutdown, they worked as well as before solids settling.

- Up to 50% solids (catalyst) concentration has been used successfully. With such a high solid loading, slurry looks like mud. The highest superficial gas velocity used with 50% solids concentration is 0.7 ft/sec (21.3 cm/sec).

- Latest reactor operating mode (LP-3) used coils inside and does not need any external circulation of slurry. No coil erosion or solids build-up problems have been observed. Earlier operations (LP-2) used coils outside with slurry circulation for heat transfer. Currently coils occupy only 4% of reactor cross-section. For Fischer-Tropsch synthesis outside coil should not be ruled out due to higher heat generation per mole of synthesis gas.

- A gamma ray device is used to measure reactor bed density - the device can slide vertically so measurement can be made at any axial position.

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- No foaming has been observed or detected in the reactor during operation.

- Catalyst was the "precursor" used for making pellets of fixed-bed methanol catalyst. Catalyst bulk activity in terms of (kg methanol formed)/(kg catalyst hr) is higher than in fixed bed.

- Early operations with ebullating bed mode did not work due to catalyst pellet disintegration.

- The company has developed procedures for testing catalysts in a stirred autoclave and for predicting activity expected in LaPorte reactor using the autoclave data. Such predictions and observations usually match well.

- Slurry pump is centrifugal (Lawrence pump) that worked well. Over the years, Air Products developed proprietary expertise in pump seals and slurry handling. Mineral oil return pumps are capable of handling slurry, albeit there are usually little solids in the oil collected.

- Plant has been operated successfully both in methanol and methanol-dimethyl ether modes.

- One more oxygenate mode run is planned before the proposed F-T operation. However, actual sequence of oxygenate and F-T operation is subject to change.

- Slurry reactor is essentially isothermal - - few degrees difference between the top and bottom locations. Isothermal in radial direction too.

- The plant is very well maintained.