Assessment of the Use of Slurry Reactors in Fischer-Tropsch Synthesis

by

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Introduction

The major technological problems of Fischer-Tropsch processes are the rapid and efficient removal of the reaction heat to avoid local overheating of the catalyst (and hence excessive methane formation), and the ability to operate with synthesis gas having low H_2-to-CO ratio to reduce cost and increase thermal efficiency of production, without leading to excessive carbon formation. This paper briefly reviews the various reactor types that have been developed to solve these problems, compares some of the key features of the three major reactor technologies, and focuses on the ability of slurry-phase operation to handle low H_2-to-CO feed ratios (Table 1). Although slurry-phase operation appears attractive, commercialization of slurry bubble column reactors still awaits reliable scale-up and design rules. There is limited knowledge of the factors that influence the flow regime, the degree of mixing in both liquid and gas phase, and the design parameters in slurry bubble column reactors.

Reactor Developments

To solve the problems associated with Fischer-Tropsch synthesis, various types of reactors were developed, differing mainly in the way heat is removed and gas and catalyst are contacted. A classification based on catalyst mobility and number of phases in the process is shown in Table 2. While some of these reactor technologies soon appeared unsatisfactory (the lamellar and concentric tube reactor [1] and the trickle bed reactor [2]) or too expensive (the tube wall reactor [3]), other technologies were not given the opportunity to demonstrate their eventual competitiveness, often because of the unfavorable economics of the coal liquefaction route at the time they were developed. These alternative technologies deserve reconsideration.

However, an objective assessment of their relative merits is extremely delicate, because such an assessment mainly depends on the basis for comparison. While some authors compare reactor technologies at identical operating conditions [1,4], always limited by the range of the least flexible technology and sometimes even including identical catalyst particle sizes [4], others prefer to optimize each technology separately to meet a given productivity [3]. Recognizing that these different approaches might result in conflicting conclusions, an attempt has been made to compare the three major reactor technologies--entrained fluidized bed, tubular fixed bed, and the developing slurry-phase operation--in the most general terms (Tables 3.A and B), as distilled from a survey of several sources [1,3-12]. The general conclusion from this comparison is that slurry-phase operation definitely appears to be a most attractive alternative to the existing technologies, especially when compared to the tubular fixed-bed technology.

Hydrogen-to-Carbon Monoxide Ratio in Slurry-phase Operation

The ability of slurry-phase reactors to accept syngas of low H_2-to-CO ratio, as supplied by so-called "second generation" coal gasifiers (Texaco, Shell-Koppers, BGC Lurgi [11]), is basically due to the intrinsic water-gas shift activity of the catalysts used, the high degree of backmixing in the liquid phase, and the differences in diffusivities and solubilities of the reactants in wax media [13, 14].

The necessity of high activity toward the water-gas shift, which essentially translates into low usage ratios, is best understood in the relationships between the feed (I), usage (U) and exit (E) H_2-to-CO ratios, and the hydrogen (X_{H}), carbon monoxide (X_{CO}), and syngas ($X_{CO + H}$) conversions (Table 4). The set of Equations 1 to 3, or its equivalent 1' to 3', readily combines into Equation 4, which expresses the exit H_2-to-CO ratio solely as a function of the syngas conversion and the feed and usage ratios. A comparison of the nominator and denominator in this expression then leads to the conclusions (5), which essentially state that the exit ratio will exceed the feed ratio whenever the usage ratio is lower than the feed ratio. This observation, reported by Satterfield and Huff [13] in different terms, has been illustrated in Figure 1 for several values of the usage ratio at a feed ratio of 1.5 (note that I = E at $X_{CO + H_2} = 0$).

It should be emphasized that Relations 1 through 5 generally apply to the gas-phase effluent of any type of reactor, provided an "overall" usage ratio has been calculated, e.g. from the exit conversions. Local values of the usage ratio may differ, however, depending upon local temperatures and concentrations; and Relation 5b, if valid at the reactor outlet, may not be met throughout the reactor. The high degree of mixing usually obtained in the liquid phase of slurry reactors, however, allows the assumption of uniform conditions in the liquid phase, and hence of a constant value for the usage ratio thoughout the reactor. If mass (and heat) transfer limitations are negligible, the liquid phase concentrations furthermore correspond to saturation at the exit gas-phase concentrations [13]. It may therefore be stated that in Fischer-Tropsch slurry reactors, with sufficient liquid mixing and negligible mass transfer limitations, the actual H_2-to-CO ratio in the liquid phase will exceed the feed ratio (hence decreasing the risk of possible carbon formation) whenever U is lower than I. The latter condition was indeed fulfilled for the catalysts used in slurry Fischer-Tropsch synthesis (Table 5).

Even when resistance to mass transfer is considered, the statement still holds, because of the higher diffusivity and solubility of hydrogen (as compared to carbon monoxide) in wax media. This has been extensively discussed by Stern and co-workers [14] for the methanation reaction:

$$2H_2 + 2CO \longrightarrow CH_4 + CO_2 \qquad (6)$$

in slurry phase, as illustrated in Figure 2. Similar considerations are believed to hold for slurry-phase methanol synthesis.

Identification of Scale-up Problems

Flow Regimes

One of the major reasons why slurry-phase operation of Fischer-Tropsch synthesis has not yet been commercialized is the limited knowledge of the factors that determine the flow regime. An approach to characterize the various flow regimes in two-phase bubble column reactors as a function of superficial gas velocity and reactor diameter has been presented by Deckwer and co-workers [20] (Figure 3), who claim its applicability to three-phase systems for sufficiently small catalyst loads (<16 wt%) and particle sizes ($d_p < 50$ µm). The transition range in this representation is believed to additionally depend on the dispersion height, the gas distributor, the liquid velocity, and the physicochemical properties of the slurry (liquid) phase. Figure 3 also illustrates the operational ranges (vertical lines and dots) of the slurry phase studies summarized in Table 6. Although most studies apparently pertain to the homogeneous flow regime, "excessive foaming" and "gas bubble slugging" were observed by Mobil workers [13] in a 5.2-cm diameter column at gas velocities above 0.4, resp. 1.5 cm/sec (asterisk in Figure 3), hence revoking the general reliability of Deckwers' representation.

Kölbel and Ralek [7], on the other hand, tried to characterize the flow regimes as a function of the solid content of the slurry phase, C_S, as represented in Figure 4 together with the operational data from Table 6. No mention was made of the reactor geometry and gas distributor used, but if their terminology is properly understood, the upper region of "big bubble formation" probably coincides with Deckwers' heterogeneous flow regime. The observation that the homogeneous flow regime, considered to be the region between the shaded areas, narrows with increasing solid concentrations is important. On the other hand, the minimal superficial gas velocity required for catalyst fluidization appears uncertain, as British researchers found that a gas velocity of 2.75 cm/sec is insufficient to maintain effective fluidization at a solid load of ~5.3 wt% [25].

If it is true that industrial applications (high U_G and d_R desired for high capacity) imply operation at churn-turbulent conditions, it is important to realize that most correlations for important design parameters have been derived only under bubble-flow conditions.

Backmixing

Another source of uncertainly in predicting large-scale operation is the extent to which backmixing needs to be taken into account. Although there is general agreement on the plug flow behavior of the gas phase in bubble columns, controversy still exists regarding the degree of backmixing in both liquid and solid phase, despite their recognized importance in predicting reactor performances--especially at high conversions [26,27]. Bukur [28] emphasizes that the degree of mixing in the liquid phase depends on the magnitude of the axial mixing parameter:

$$N_L = \frac{D_L E_L}{U_G^o L} \tag{7}$$

in which the axial dispersion coefficient for the liquid phase, D_L, may be estimated from [20,29]:

$$D_L = 3.676\ U_G^{0.32} d_R^{1.34} \tag{8}$$

to yield:

$$N_L = \frac{3.676\ U_G^{0.32} d_R^{1.34} \varepsilon_L}{U_G^o L} \tag{9}$$

It can be conceived from Equation (9) how dramatic the impact of the L/d_R-ratio is on the liquid phase (and related solid phase) mixing. The considerable range of L/d_R-ratios used in Fischer-Tropsch investigations (see Table 6 for L_R/d_R-values) therefore partially accounts for observed divergencies in selectivity.

Design Parameters and Physicochemical Properties

Apart from the inevitable shortcomings of the kinetic models applied, remaining uncertainties in scale-up arise mainly from the limited availability, applicability, and/or reliability of correlations about some essential parameters, such as gas hold-up, diffusion, solubility, mass transfer (k_La), and dispersion coefficients. Correlations pertaining to these parameters often apply only to the specific range of operational conditions (such as reactor geometry, gas distributor, liquid medium, solid concentration, temperature) for which they were derived. Considerable inaccuracy might result upon extrapolation to other conditions; Figure 5 illustrates this for gas hold-up correlations and values obtained at different conditions in bubble columns.

The limited reliability of some other parameter correlations is probably most revealing in the case of diffusion coefficient estimations. The two diffusivity correlations usually recommended [20,30]--the Sovova equation and the Wilke-Chang correlation--indeed predict values which, under typical Fischer-Tropsch conditions, may differ as much as 120% [12], obviously also affecting diffusivity-dependent parameters such as k_L [20]. The limited availability of data appears especially crucial when estimating solubilities, though essential in determining the liquid phase concentration of the reactants. Solubility coefficients for hydrogen and carbon monoxide are most often estimated from the data of Peter and Weinert [30] in molten paraffin. The composition of the "wax" media used in slurry-phase reactors may differ considerably, however, depending on the operating conditions and time onstream [4,6,15]. It has not been investigated how this affects the gas solubilities.

Conclusions

The slurry-phase bubble column reactor undoubtedly appears to be an attractive reactor technology for Fischer-Tropsch processing, considering its favorable economics and ability to solve the major problems (temperature control, low H_2-to-CO ratios) associated with Fischer-Tropsch synthesis. Its ability to handle low H_2-to-Co ratios depends, among other factors, on the intrinsic water-gas shift activity of the catalyst used; an elucidation of the synthesis mechanism can only contribute to a better understanding of the factors that influence the H_2-to-CO usage ratio.

However, a better understanding of the reactor hydrodynamics is required to establish sound scale-up and design rules. The flow regimes, although dependent upon dispersion height, gas sparger, and liquid medium, have not been studied in terms of their dependency on these parameters. There is still a considerable disagreement concerning the degree of mixing in both liquid and solid phases, to which the reactor geometry (L/d_R) appears of essential importance. More reliable data are needed on essential design parameters (gas hold-up, bubble size) and on the physical properties (diffusivity, solubility) of the liquid media used.

List of Symbols

a	specific gas-liquid interfacial area $[cm^2/cm^3{}_R]$
C_s	solids concentration [wt% of slurry]
d_p	catalyst particle size $[\mu m]$
d_R	reactor diameter [cm]
D_L	axial dispersion coefficient for the liquid phase $[cm^2/s]$
E_g	gas hold-up
E_L	liquid hold-up
E	H_2/CO ratio at reactor outlet
I	H_2/CO feed ratio
k_L	liquid-side mass transfer coefficient [cm/s]
k_La	volumetric gas-liquid mass transfer coefficient $[s^{-1}]$
ξ	dimensionless axial distance in reactor
L	expanded height of slurry [cm]
L_R	reactor height [cm]
N_L	axial mixing parameter, given by Equation (7)
$\theta_{G,i}$	dimensionless gas-phase concentration of component i
$\theta_{L,i}$	dimensionless liquid-phase concentration of component i
ρ_L	density of the liquid phase $[g/cm^3]$
τ_G	gas space time [s]
τ_L	liquid space time [s]
U	H_2/CO usage ratio [moles H_2 consumed/moles CO consumed]
U_G	superficial gas velocity [cm/s]
U_G^o	superficial gas velocity at reactor inlet [cm/s]
X_{CO}	carbon monoxide conversion
X_{H_2}	hydrogen conversion
X_{CO+H_2}	syngas conversion

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- BRIEF REVIEW OF THE DIFFERENT REACTOR TECHNOLOGIES APPLIED SO FAR TO THE FISCHER-TROPSCH SYNTHESIS.

- COMPARISON OF THE INDIVIDUAL MERITS OF THREE MAJOR TECHNOLOGIES:

    - FLUIDIZED BED WITH CATALYST ENTRAINMENT

    - TUBULAR FIXED BED

    - SLURRY PHASE BUBBLE COLUMN OPERATION

- ELABORATION ON THE ABILITY OF SLURRY PHASE OPERATION TO HANDLE LOW H_2/CO - SYNTHESIS GAS.

- IDENTIFICATION OF THE DIFFICULTIES ENCOUNTERED IN SCALING-UP SLURRY PHASE BUBBLE COLUMN REACTORS, WITH RESPECT TO:

    - FLOWREGIMES

    - DEGREE OF BACKMIXING

    - DESIGN PARAMETERS

    - PHYSICAL PROPERTIES

- RECOMMENDATIONS AND CONCLUSIONS

TABLE 1. OUTLINE

Reactors With Stationary Catalyst:

Two-Phase:
- lamellar reactor
- tubular reactors:
  - concentric tube reactor
  - tube wall reactor (PETC)
  - fixed bed reactor (Ruhrchemie/Lurgi: ARGE)

Three-Phase:
- trickle bed reactor (BASF, U.S. Bureau of Mines)

Reactors With Mobile Catalyst:

Two-Phase:
- fluidized bed reactor:
  - without catalyst entrainment (HRI: Hydrocol)
  - with catalyst entrainment (Kellog: Synthol)

Three-Phase:
- slurry reactor:
  - bubble column reactor (Kölbel)
  - continuous stirred tank reactor
- ebullated bed reactor (U.S. Bureau of Mines)

TABLE 2. Reactor technologies applied to Fischer-Tropsch synthesis.

Technology	Tubular fixed bed reactor	Fluidized bed with entrainment	Slurry phase bubble column
Operating conditions			
temperature [°C]	220-250	320-340	260-275
pressure [bar]	25	20-23	11-12
H_2/CO feed ratio	1,3-2,0	2,0-2,8	0,6-0,7
recycle/feed ratio	2,2-2,5	2,0-2,5	/
catalyst: particle size [mm]	1-5	0,05-0,2	0,03-0,07
charge [ton]*	highest	800	90
Operability			
temperature control	poor	good	good-excellent
pressure drop at high U_G	small	medium	medium up to high
flexibility	intermediate	low	high
catalyst: continuous replacement	very difficult	simple/expensive	feasible/problematic
losses	none	2-4% per day	small
lifetime	6-9 months	44 days	unknown

TABLE 3A. Comparison of the tubular fixed bed, fluidized bed with catalyst entrainment and slurry phase bubble column operation.

Technology	Tubular fixed bed reactor	Fluidized bed with entrainment	Slurry phase bubble column
Conversion/yields			
syngas conversion [%]	50-70	80-85	> 90
space time yield [kgHC/m_R^3 , hr]	comparable to slurry phase	highest	comparable to fixed bed
catalyst efficiency [kgHC/$m^3_{cat.}$.hr]	lowest	highest	intermediate
Selectivity			
CH_4 - formation	low	high	as fixed bed or lower
gasoline production	lowest	high	comparable to fluidized bed
product range	full range	restricted to low molecular weight	full range
Profitability			
relative investment cost* (reactors, vessels, heat exchangers)	intermediate	100	45
relative catalyst cost*	highest	100	51
thermal efficiency [%] (steam generation, BFW heating)	lowest	85	91

*Based on conversion of 793 10^3 std m^3 of syngas/hr (UOP, 1982)

TABLE 5B. Comparison of the tubular fixed bed, fluidized bed with catalyst entrainment and slurry phase bubble column operation.

$$X_{CO+H_2} = \frac{X_{CO} + I X_{H_2}}{1 + I} \qquad (1)$$

$$U = I \frac{X_{H_2}}{X_{CO}} \qquad (2)$$

$$E = I \frac{(1 - X_{H_2})}{(1 - X_{CO})} \qquad (3)$$

$$X_{CO} = \frac{I - E}{U - E} \qquad (1')$$

$$X_{H_2} = \frac{U}{I} \left(\frac{I - E}{U - E} \right) \qquad (2')$$

$$X_{CO+H_2} = \frac{(I - E)\ (1 + U)}{(U - E)\ (1 + I)} \qquad (3')$$

$$E = I \frac{(1 + U) - \frac{U}{I} (1 + I)\ X_{CO+H_2}}{(1 + U) - (1 + I)\ X_{CO+H_2}} \qquad (4)$$

$$E = 0 \text{ as } X_{CO+H_2} \nearrow \text{ if } U = I \Longrightarrow X_{H_2} = X_{CO+H_2} = X_{CO} \qquad (5a)$$

$$E \nearrow \text{ as } X_{CO+H_2} \nearrow \text{ if } U < I \Longrightarrow X_{H_2} < X_{CO+H_2} < X_{CO} \qquad (5b)$$

$$E \searrow \text{ as } X_{CO+H_2} \nearrow \text{ if } U > I \Longrightarrow X_{H_2} > X_{CO+H_2} > X_{CO} \qquad (5c)$$

TABLE 4. Some useful relations between the feed (I), usage (U) and exit (E) H_2/CO - ratio, and the hydrogen (X_{H_2}), carbon monoxide (X_{CO}) and syngas (X_{CO+H_2}) conversion.

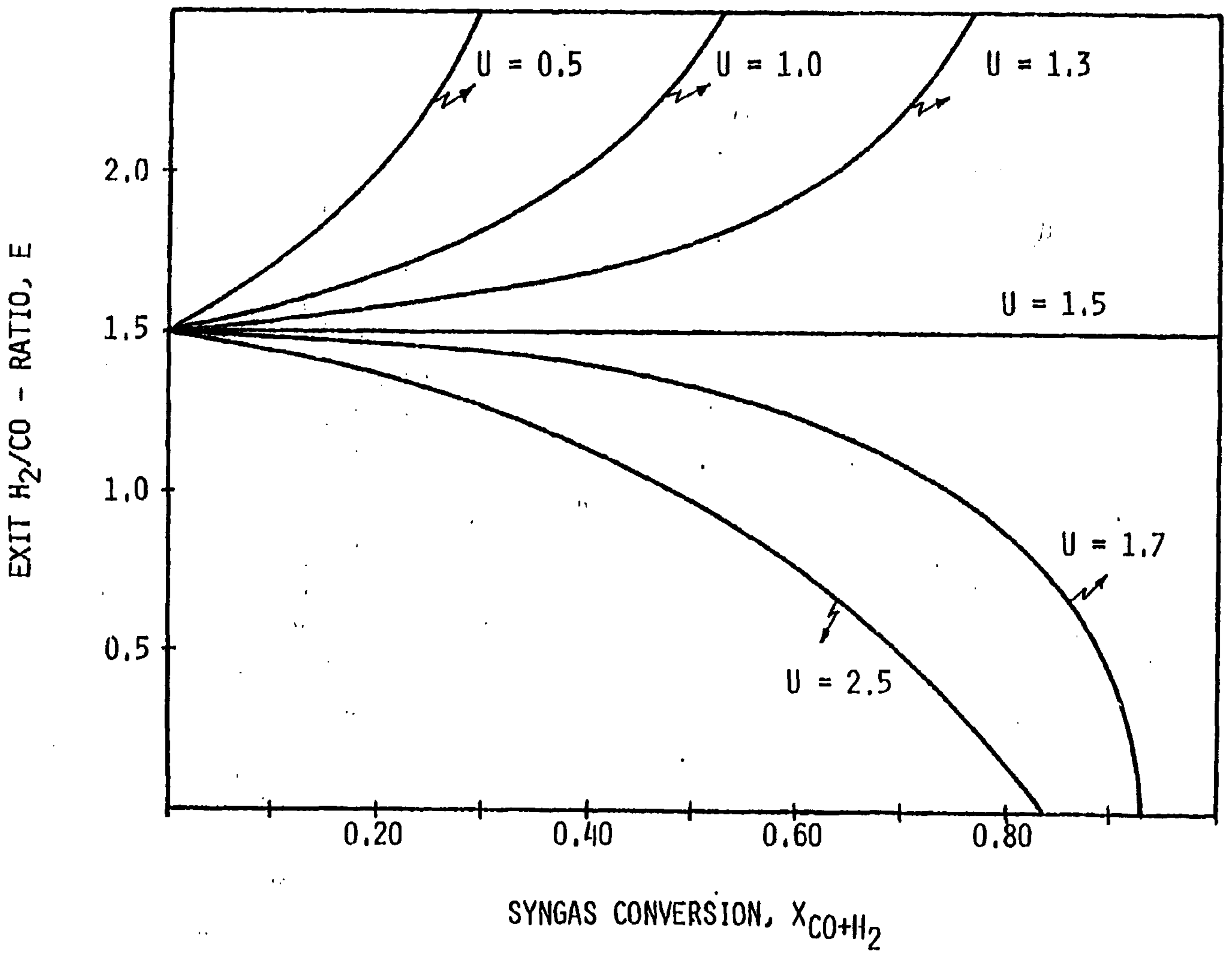


FIGURE 1. Relation between the exit H_2/CO - ratio and the syngas conversion for assumed values of the usage ratio and feed H_2/ CO - ratio of 1.5.

Authors	I	U	X_{H_2}	X_{CO+H_2}	X_{CO}	Catalyst
Schlesinger et al. (1951)	1.0	0.67	0.28	0.35	0.42	pptd. Fe catalyst:
	1.0	0.61	0.48	0.64	0.79	Fe/Cu/K_2O = 100/10/1
Hall et al. (1952)	3.27	2.82	0.571	0.592	0.662	fused Fe catalyst:
	2.44	2.34	0.924	0.935	0.962	with 0.7% K_2O, 3.4% MgO
Schlesinger et al. (1954)	1.0	0.81	0.639	0.714	0.789	nitrided fused Fe catalyst
Kolbel et al. (1956)	0.67	0.63	0.86	0.89	0.91	unsupported pptd. Fe:
	0.67	0.63	0.85	0.88	0.90	Fe/Cu/K_2O = 100/0.1/0.05-0.5
Mitra and Roy (1963)	1.33	1.19	0.671	0.705	0.750	pptd. Fe catalyst: Fe/Cu/
	1.30	1.24	0.896	0.915	0.939	MgO-(CaO)/K_2O/Kieselguhr: 100/10/4/3.5/50
Farley and Ray (1964)	0.703	0.616	0.327	0.354	0.373	pptd. Fe catalyst:
	0.662	0.565	0.538	0.594	0.631	Fe/Cu/K_2O = 100/1.6/2
Kunugi and Sakai (1968)	0.59	0.59	0.808	0.808	0.808	pptd. Fe catalyst:
						Fe/Cu/K_2CO_3 = 100/0.3/0.6-1.2
Forney et al. (1975)	1.00	0.62	0.45	0.59	0.73	Fe catalyst with:
						Fe/Cu/K_2O = 100/10/1
Mobil (1982)	0.687	0.580	0.602	0.668	0.713	"I-B" with
	0.673	0.635	0.839	0.868	0.888	Fe/Cu/K_2O

TABLE 5. Feed and usage ratio of hydrogen to carbon monoxide for several slurry phase studies of the Fischer-Tropsch reaction.

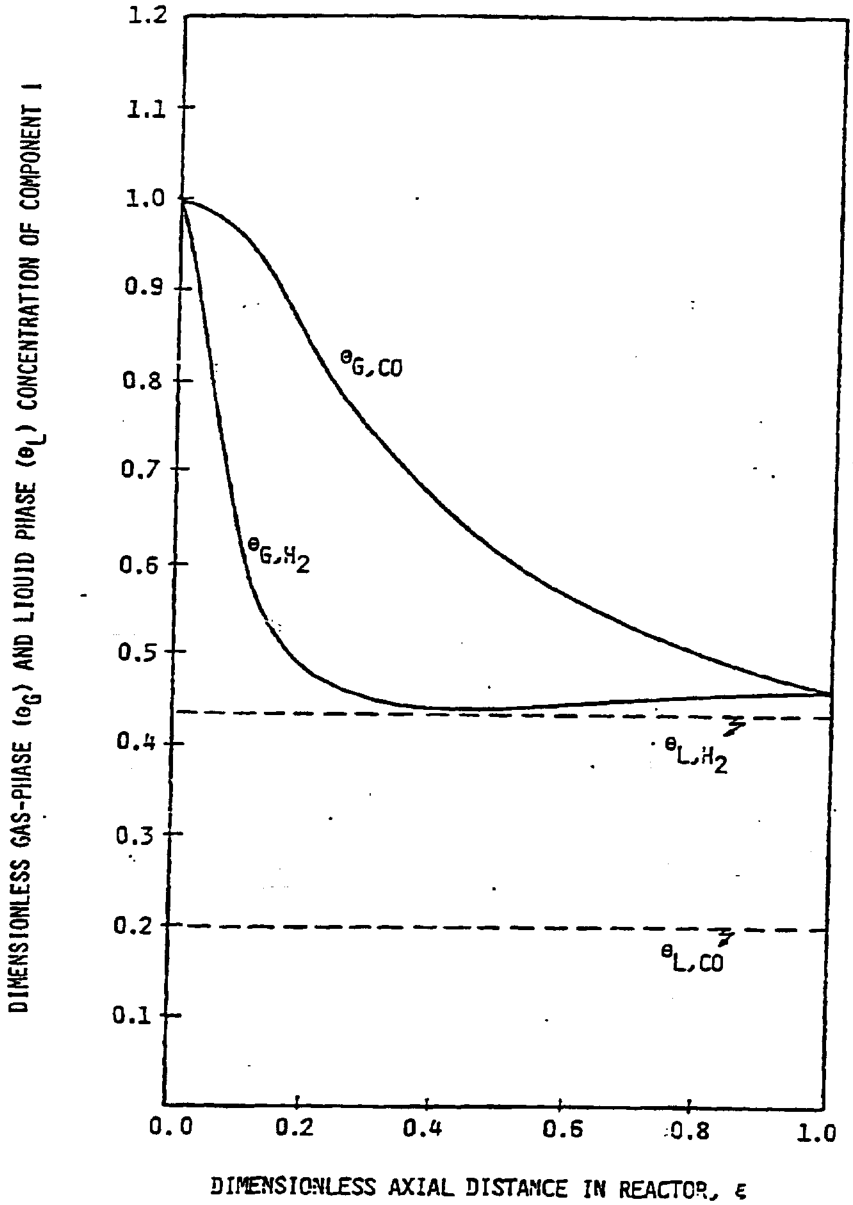
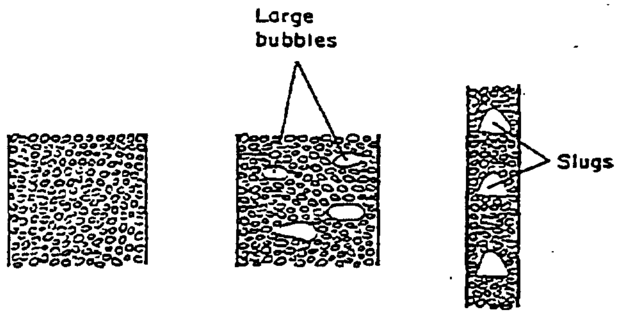


FIGURE 2. Dependence of the gas and liquid concentrations of H_2 and CO on the axial reactor distance for the reaction: $2H_2+2CO \rightarrow CH_4+CO_2$ at 270°C with τ_G=70s and τ_L=700s (Stern et al., 1983)



(a) Homogeneous (Bubbly flow)

(b) Heterogeneous (Churn-turbulent flow)

(c) Slug flow

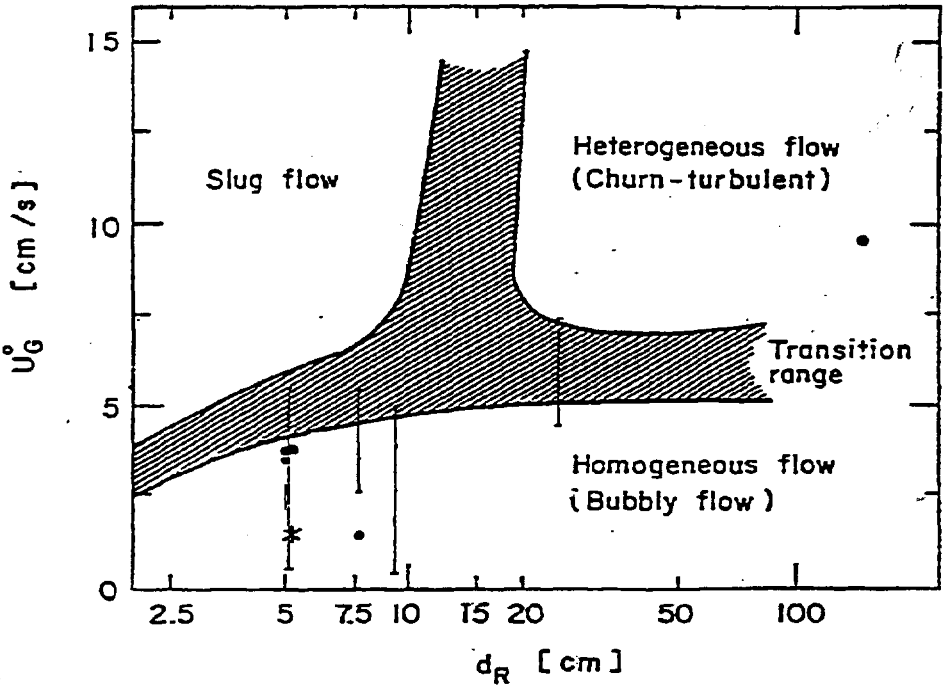


FIGURE 3. Assumed dependency of flow regimes on superficial gas velocity and reactor diameter ($d_p \leq 50\,\mu m$, cat. load ≤ 16 wt%) according to Deckwer et al. (1980).

Authors	L_R [cm]	d_R [cm]	L_R/d_R [/]	U_G° [cm/sec]	cat. load [wt.%]	d_p [μm]
Kölbel et al. (1956)	860	155	5.55	9.5	28.4*	30
Quicker et al. (1981)	135	9.5	14.21	0.5-5	0	/
Hall et al. (1952)	120	5	24	2.2-2.9	22.2-55.6	75
Farley and Ray (1964)	853.4	24.8	34.4	4.5-7.4	~8*	1-3
Schlesinger et al. (1951)	304.8	7.6	40	2.7-5.5	25-30.8	61
Schlesinger et al. (1954)	304.8	7.6	40	1.5	21.1	?
Forney et al. (1974)	304.8	7.6	40	2.7	42.1*	61
Mitra and Roy (1964)	213.4	5.1	42	1.5-2.2	16-18	?
Mitra and Roy (1964)	304.8	5.1	60	0.9-2.2	16-18	?
Kölbel et al. (1956)	300	5	60	3.5	11.2*	?
Calderbank et al. (1963)	457.2	5.1	90	0.6-5.5	1.5-4.4*	?
Kunugi and Sakai (1968)	550	5	110	3.8	4.8	?
Sakai and Kunugi (1974)	600	5	120	3.8	10.1	?
Mobil (1982)	730	5.2	140.4	3.9	14.3	?

*Calculated assuming $\epsilon_g = 0.053\ U_G^{1.1}$ and/or $\rho_L = 0.85$ g/ml

TABLE 6. Reactor dimensions, superficial velocity and catalyst concentration and size used by several authors.

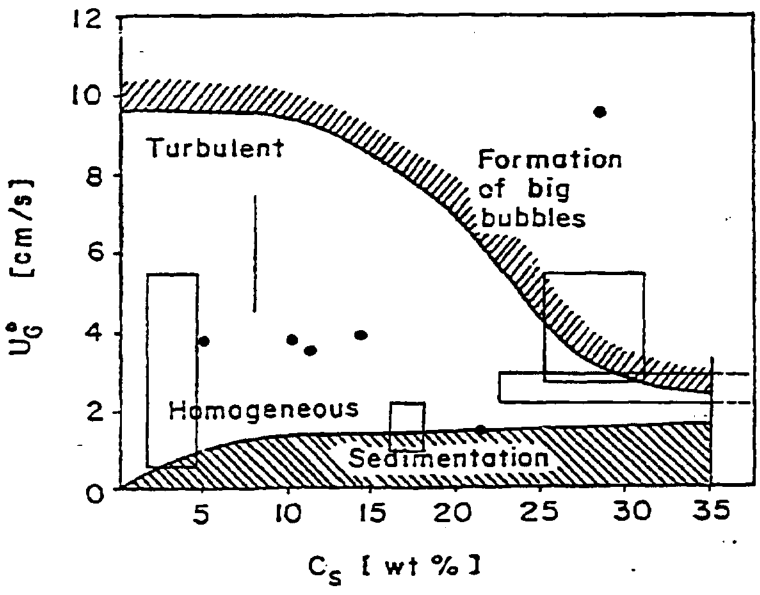


FIGURE 4. Operational range diagram for a bubble column reactor with suspended solid matter (d_p = 30 µm, $\rho_s \approx 2$g/cc, $\rho_L \approx 1$g/cc), according to Kölbel and Ralek (1980)

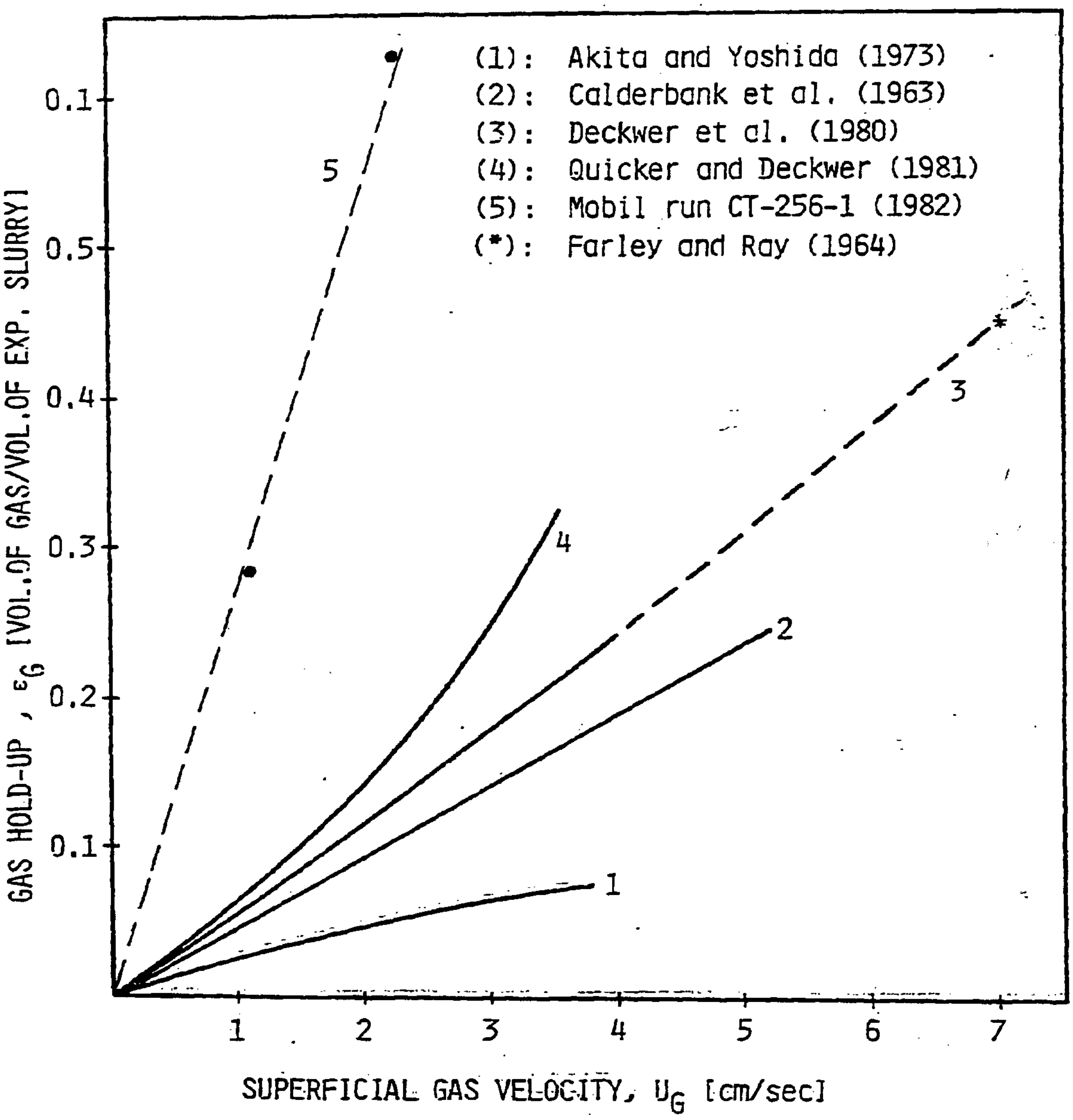


FIGURE 5. Gas holdup as a function of the superficial gas velocity according to several authors.