y = inter-crossbar spacing, distance between adjacent crossbars in a spacer, cm

Y = length of active membrane area in the direction of flow, cm

Greek Letters

λ = conventional Darcy friction factor, defined in eq 10

ν = kinematic viscosity of the fluid, cm^2/s

ρ = density of the fluid, g/cm^3

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Effect of the Noncylindrical Shape of Extrudates on the Hydrodesulfurization of Oil Fractions

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In this paper the results are presented of an investigation into the effects of noncircular cross sections of extrudates on the hydrodesulfurization of oil fractions. Twelve different types of catalyst particles of different porosities and compositions were prepared and tested under various process conditions. The results show that noncylindrical extrudates having a particle volume identical with that of cylindrical extrudates may lead to an activity which is higher than that of the cylindrical ones, depending on the degree of diffusion limitation. Just as for cylindrical extrudates, the activity of polylobal particles is related to the surface to volume ratio of the extrudates. This was found to hold both for diluted catalyst beds and undiluted beds at high mass velocities. When measured in undiluted beds at low mass velocities, the effect of polylobes tends to be overrated. Relative diffusion coefficients are in good agreement with data obtained by liquid–liquid diffusivity measurements. The advantages and disadvantages of polylobal shapes will be discussed.

Introduction

Several authors have reported that diffusion limitations play an important role in distillate hydrodesulfurization. Adlington and Thompson (1964) mentioned an effectiveness factor of 0.6 for a 1/8-in. pelletized catalyst in hydrodesulfurization under typical process conditions. Van Zoonen and Douwes (1963) showed that smaller particles and particles having larger mean pore radii displayed increased activities. A strong dependency of the hydrodesulfurization activity on the extrudate diameter was also reported by de Bruijn (1976) for the hydrodesulfurization of Kuwait vacuum gasoil. The shift to small catalyst particles in hydrodesulfurization units in the past ten years demonstrates that pore diffusion limitation also plays an important role in commercial operation. A further shift to even smaller particles is impracticable because of the strong increase of the pressure drop in the catalyst bed.

The use of noncylindrically shaped extrudates was proposed in order that with particles having a high outer surface to volume ratio a relatively low pressure drop might be achieved (Hoekstra 1970). Moreover, an improvement was obtained of the deposition of metals in residue hydrotreating as well.

Gustafson (1975) motivated the use of these extrudates in gasoil treating on account of their better diffusion performance. For trilobal catalysts a far better performance was reported than was to be expected from diffusion limitation effects only. On the other hand, a theoretical study of Suzuki and Uchida (1979) shows that the effect on the activities of noncylindrical extrudates (including trilobes) of applying the diffusion limitation theory is fully comparable with the effect on the activities of cylindrical particles.

For a better understanding of the possible advantages of noncylindrical shapes, various series of catalysts were prepared. These various series have different porosities and/or metal contents. Within each series only the shape or size of the extrudates was varied.

In the first section the results of hydrodesulfurization tests with vacuum gasoil and atmospheric residue are reported. Standard test methods were applied to measure the influence on the activity of shape and diffusion.

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Table I. Properties of Catalysts

nominal metal loading, MoO₃/CoO, wt %	nominal pore diameter, nm	sample no.	shape and size, in.	MoO₃, wt %	CoO, wt %	surface area, m²/g	pore vol, mL/g	length, mm	particle size, L_p, mm
12/4	6	A1	cyl. ¹/₁₆	12.9	4.1	293	0.43	4.1	0.341
		A2	cyl. ¹/₃₂	12.9	4.1	288	0.44	2.8	0.165
		A3	AQ	12.9	4.1	300	0.44	3.6	0.220
12/4	8	B1	cyl. ¹/₁₂	12.9	3.9	266	0.52	5.4	0.448
		B2	cyl. ¹/₁₆	12.7	3.9	274	0.54	4.6	0.333
		B3	cyl. ¹/₃₂	12.8	3.9	274	0.55	3.1	0.183
		B4	AQ	12.9	4.0	279	0.53	3.9	0.204
		B5	SQ	12.9	3.9	278	0.54	4.1	0.189
		B6	3-lobe	12.9	4.1	273	0.53	4.0	0.236
12/4	12	C1	cyl. ¹/₁₂	12.7	4.1	188	0.55	4.0	0.397
		C2	cyl. ¹/₁₆	12.6	4.0	184	0.56	3.4	0.310
		C3	cyl. ¹/₃₂	12.6	4.2	197	0.55	3.6	0.192
		C4	AQ	12.6	4.1	193	0.59	3.7	0.185
		C5	3-lobe	12.6	4.0	201	0.58	3.7	0.234
15.5/5.5	6	D1	3-lobe	15.5	5.5	297	0.43	3.3	0.254
		D2	as 3-lobe	15.7	5.5	294	0.45	3.1	0.181
		D3	cyl. with groove	15.5	5.5	289	0.45	3.1	0.186
		D4	cyl. ¹/₁₆	15.6	5.5	275	0.44	3.5	0.322
		D5	as D2 1.5×	15.6	5.5	291	0.44	3.6	0.256
		D6	as D3 1.5×	15.6	5.5	285	0.41	3.3	0.266
		D7	cyl. ¹/₃₂	15.4	5.5	278	0.43	3.2	0.172
		D8	triangle	15.6	5.5	286	0.48	3.2	0.236
		D9	ellipse	15.6	5.5	288	0.44	3.0	0.196
16/5	6	E1	cyl. ¹/₁₆	14.8	5.0	292	0.43	3.1	0.322
		E2	3-lobe	14.7	5.2	283	0.44	3.1	0.254
		E3	cyl. ¹/₃₂	14.8	4.8	287	0.42	2.3	0.176

Table II. Various Activity Test Methods

feedstocks	Kuwait vacuum gasoil		Kuwait atmospheric residue	
boiling range, °C	331–533		ibp 240 °C	ibp 241 °C
			10% 348 °C	10% 343 °C
			50% 506 °C	50% 496 °C
sulfur content, wt %	2.80		3.73	3.96
density (15/4), g/cm³	0.9206		0.9576	0.9586
viscosity, 210 °F cSt	7.58		28.3	26.0
test conditions:				
LHSV, m³/m³ h	1–3		0.8	1.0
temperature, °C	365		371	385
pressure, bar	41		51	101
H₂/oil ratio, nm³/m³	540		900	1000
type of test	50–75 mL dil/undil. downflow	850 mL undil. downflow	75 mL diluted downflow	500 mL undiluted upflow/ downflow
diam. reactor, mm	21	16	21	25
diam. thermocouple, mm	6	no	6	6
mass velocity, kg/m² s × 10⁻³	0.04–0.16	1.06–2.11	0.044	0.27

In the second section various types of tests applied are described. The influence will be discussed of the test method on the measuring results obtained with the various particle sizes and shapes. The advantages and disadvantages of polylobal catalysts are given in the third section, in which the several compromise choices of the optimum catalyst size and shape are considered.

Experimental Procedures and Results

Catalyst Preparation. The catalyst samples consist of γ-alumina loaded either with 12 wt % MoO₃ and 4 wt % CoO or with 15.5 wt % MoO₃ and 5.5 wt % CoO. The porosity and surface areas were considered to be variables. The porosities and surface areas were varied by standard procedures. The mean pore diameters, defined as $4 \times PV \times 10^3/SA$, of the various series of catalysts were 6, 8, and 12 nm, respectively. Within a series of catalysts the shape was varied by changing the die plates during the extrusion process. The various shapes are shown in Figure 1. The impregnations were carried out by standard methods in-

cluding drying and calcination after each step. Analytical data of the various series of catalysts are given in Table I.

Activity Tests. Prior to the activity tests, the catalysts were dried and presulfided. In the 50 or 75 mL tests the catalyst particles were diluted with carborundum (46 mesh). The standard procedures applied are as described by de Bruijn (1976).

Activity tests were carried out at various mass velocities, in upflow and downflow directions and with diluted and undiluted catalyst beds. Use was made of two feedstocks, viz., Kuwait vacuum gasoil and Kuwait atmospheric residue. For each series of catalysts the activity of crushed (20–60 mesh) catalyst was also measured. The test methods, process conditions, and feedstock properties are summarized in Table II. A typical value for the coefficient of variation for the rate constants in the various tests is 3.5%.

Activity Calculation. The activities of the catalysts are expressed as the reaction rate constants on weight basis
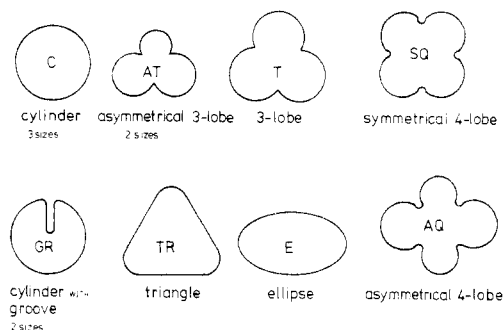
Figure 1. Various forms of catalyst particles. (The scale of the different drawings varies; sizes are given in Table I.)

Table III. Test Results with Kuwait Vacuum Gasoil (50–75 mL Diluted Catalyst Bed)

sample	particle size, L_p, mm	apparent reaction rate constant, k_w (1.65th order)
A1 crushed	0.055	6.7
A2	0.341	3.9
A2	0.165	6.2
A3	0.220	5.5
B2 crushed	0.055	7.9
B1	0.448	4.7
B2	0.333	5.9
B3	0.183	7.4
B4	0.204	6.6
B5	0.189	6.8
B6	0.236	5.9
C1 crushed	0.055	6.5
C1	0.397	5.7
C2	0.310	5.6
C3	0.192	6.7
C4	0.185	6.1
C5	0.234	6.6
D1 crushed	0.055	7.8
D1	0.254	5.8
D2	0.181	6.7
D3	0.186	6.4
D4 crushed	0.055	7.8
D4	0.322	4.8
D5	0.256	5.8
D6	0.266	5.3
D7	0.172	6.5
D8	0.236	6.3
D9	0.196	5.4
E1 crushed	0.055	8.2
E1	0.322	4.8
E2 crushed	0.055	8.0
E2	0.254	5.5
E3	0.176	6.1

using the experimentally found apparent orders of reaction of 1.65 for vacuum gasoil and 2.0 for atmospheric residue. The relative weight activities (RWA) were calculated from the rate constants of the sample and the standard catalyst. The results of the activity measurment in diluted catalyst beds are given in the Tables III and IV. The results obtained in the undiluted catalyst beds are given in the Tables V and VI. The differences in the rate constants of the crushed catalysts, presented in Table III, are not only due to differences in product properties but also to differences in preparation methods.

Liquid–Liquid Diffusivity Measurements. Five grams of catalyst particles, outgassed at 0.02 bar and 60 °C, were soaked in mesitylene for 2 h. The particles were placed in a spinning basket. The outer film of the mesitylene was removed by fast rotating in air for 3 s. The basket was placed in a reactor containing 60 mL of do-

Table IV. Test Results with Kuwait Atmosphere Residue (75 mL Diluted Catalyst Bed)

sample	particle size, L_p, mm	apparent reaction rate constant, k_w (2nd order)
B1	0.448	0.49
B2 crushed	0.055	1.05
B2	0.333	0.60
B3	0.183	0.75
B4	0.204	0.79
B6	0.236	0.69
C1	0.397	0.75
C2	0.310	0.81
C4	0.185	0.95
C5 crushed	0.055	0.98
C5	0.234	0.95

Table V. Test Results with Kuwait Vacuum Gasoil in Various Reactor Setups

sample	particle size, L_p mm	apparent reaction rate constant, k_w (1.65th order) 50–75 mL diluted cat. bed	850 mL undiluted cat. bed at high mass velocity	75 mL undiluted cat. bed at low mass velocity
D1	0.254	5.8	5.4	
D4	0.322	4.8	4.6	
D7	0.172	6.5	6.1	
E1	0.322	4.8		3.7
E2	0.254	5.2		5.4
E3	0.176	5.8		6.0

Table VI. Results with Kuwait Atmospheric Residue (500 mL Undiluted Catalyst Bed)

sample	particle size, L_p, mm	apparent reaction rate constant, k_w (2nd order)
B2	0.333	1.13
B3	0.183	1.39
B4	0.204	1.33
B6	0.236	1.26

Table VII. Relative Diffusion Rates

form/size	volume/ outer surface	relative diffusion rates from diffusion measurements	from activity tests in shaped and crushed form
st cat. of $^1/_{16}$ in.	0.333	1.00[a]	1.00[a]
$^1/_{32}$ in.	0.172	2.0	2.1
3-lobe	0.254	1.2	1.1
$^1/_{16}$ in.	0.322	0.8	0.5

[a] By definition.

decane, so that the catalyst was contacted with dodecane, with careful stirring.

The changes in concentration of mesitylene in dodecane as a function of time were measured by chromatographic analysis. The relative diffusion rates were calculated by comparing the slopes of the relation between ln $[(C - C_\infty)/C_0 - C_\infty)]$ and time. The results of the measurements relative to a standard catalyst are listed in Table VII.

Discussion

Theoretical Introduction. For a process in which the diffusion influences the overall activity, Thiele derived the
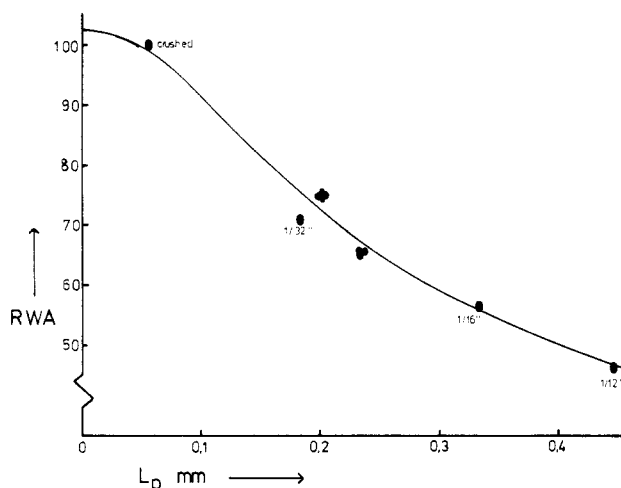
Figure 2. Relative weight activity as a function of particle size in resid tests. Catalyst series B.

following well-known equations for a first-order process (Thiele, 1939)

$$\phi = L_p(k_i/D)^{1/2} \qquad (1)$$

$$\eta = f(\phi) \qquad (2)$$

where η = effectiveness factor = k/k_i; k = apparent reaction rate constant; k_i = reaction rate constant without diffusion limitation; ϕ = Thiele number; D = diffusion rate constant; and L_p = particle size.

The reaction order and the shape of the catalyst do not very much influence the relation between η and ϕ, if the particle size is defined as proposed by Aris (1957)

$$L_p = V_p/S_p = \frac{\text{volume of the particle}}{\text{outer surface of the particle}} \qquad (3)$$

The shapes studied were spheres, cylinders, and flat plates. With the aid of computer simulations Suzuki and Uchida (1979) found that this L_p definition can also be successfully used in calculations of the effectiveness factors for non-cylindrical catalysts. For our calculations the generalized η–ϕ relation (Aris, 1975) was used. As indicated in the Introduction, the investigation will be discussed in three successive sections.

I. Relative Activities, Thiele Numbers, and Diffusion Rates for Noncylindrical Particles

The relative weight activities as a function of particle size (volume/outer surface) are plotted in Figure 2 for catalysts of the B series in atmospheric residue hydro-desulfurization. This figure shows that there exists a relation between RWA and L_p and that the polylobal samples fit the curve for cylindrical extrudates. The same applies to vacuum gasoil desulfurization for the various series of catalysts (Table III). The high relative weight activities for polylobal catalysts as reported in the literature (Gustafson, 1975) were not confirmed in these experiments.

With the η–ϕ relation the Thiele numbers for all samples were calculated, assuming an effectiveness factor of 1.0 for the crushed sample. These Thiele numbers being plotted as a function of L_p, the real effectiveness factor and the Thiele number for the crushed catalyst were estimated. With these values the calculations were repeated until the iteration was completed and the intrinsic activity was known.

The Thiele numbers as a function of particle size for catalysts of series B and D are given in Figure 3. Similar relations were obtained, in spite of differences in pore
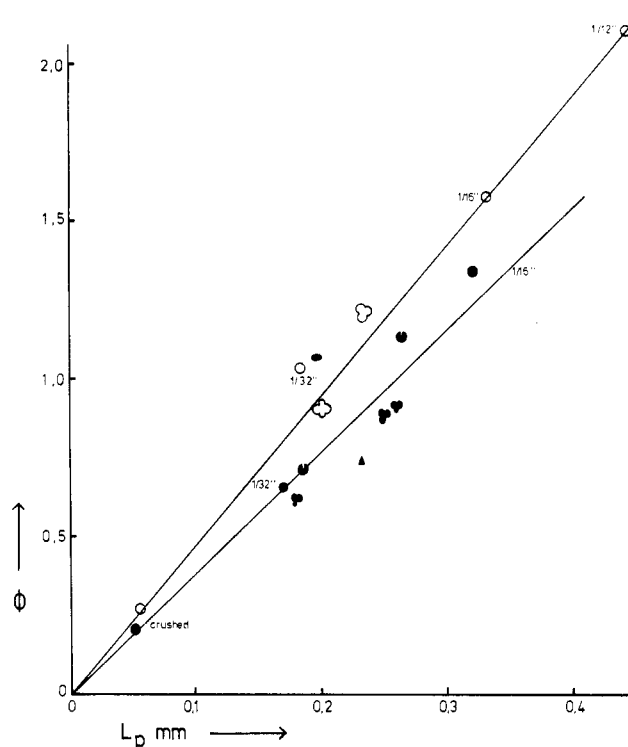

Figure 3. Thiele number as a function of particle size catalyst: series D, VGO tests, black marks; series B, resid tests, open marks.
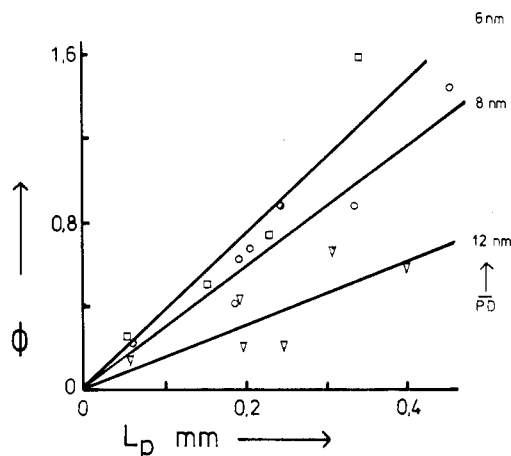

Figure 4. Thiele number as a function of particle size of catalysts having a different pore diameter; VGO tests: □, series D, $\bar{p}_d$ = 6 nm; PV = 0.55 mL/g, SA = 297 m^2/g; O, series B, $\bar{p}_d$ = 8 nm; PV = 0.54 mL/g, SA = 274 m^2/g; △, series C, $\bar{p}_d$ = 12 nm; PV = 0.47 mL/g, SA = 193 m^2/g.

diameters, metal loading, and feedstock tested. This figure supports the conclusions of Aris (1975) and Suzuki and Uchida (1979) that the volume to outer surface ratio of a particle is a reliable particle size parameter for cylindrical and noncylindrical particles.

No differences can be observed in the relations between particle size (L_p) and Thiele numbers for the different shapes of catalyst particles for various levels of diffusion limitations. Although the level of the effectiveness factor may be a function of the conversion, the conclusions will not change.

Figure 4 shows the lines for vacuum gasoil tests on three different series of catalysts with equal metal loadings, but different nominal pore diameters. From the slopes and the intrinsic activities the relative diffusion constants for each series of catalysts were calculated. Relative values of 0.6, 1.0 (by definition), and 3.3 were obtained for pore

diameters of 6, 8, and 12 nm, respectively. Hence, the relative diffusion constants not only are dependent on the pore volume but also on the surface area or pore diameter. Consequently, the mechanism of pore diffusion limitation satisfactorily accounts for the particle size effects.

This conclusion is supported by the strong influence of the type of feedstock on the relative diffusion constants. For atmospheric residue tests relative values of 0.05 k-(VGO)/k(resid) and 0.23 k(VGO)/k(resid) were calculated for catalysts with pore diameters of 8 and 12 nm. At a ratio of k(VGO)/k(resid) of about 6 the diffusion constants for resid will be 0.3 and 1.4. These values are lower than the values obtained for vacuum gasoil by a factor of about 3.

In Table VII relative diffusion rates calculated from activity tests with eq 1 are compared with the results of the diffusion measurements under nonreacting conditions. Both data show the same ranking. The polylobal catalyst (3-lobe) fits in the relation of diffusion constants vs. particle size for cylindrical extrudates. This observation is in agreement with the results of the activity measurements given in Figures 2, 3, and 4, the particle size L_p and not the particle form being determinative of the diffusion rate.

II. Influence of Test Method on the Measured Effect of Particle Size and Shape

The results and conclusions discussed up till now were based on activity measurements in small diluted catalyst beds containing 50 or 75 mL of catalyst. In small *undiluted* catalyst beds backmixing and channelling may occur and obscure the measured particle size effects (de Bruijn, 1976). This point was investigated by carrying out experiments in undiluted catalyst beds both at low and at high mass velocities.

Tests in Undiluted Small Catalyst Beds. Three catalysts of the E-series ($^1/_{16}$ and $^1/_{32}$-in. cylinders and trilobal extrudates) were tested with vacuum gasoil both in a 75-mL *undiluted* catalyst bed and in a diluted one. The activity data are given in Table V.

For a diluted bed a 14% higher activity was observed for the trilobal catalyst compared with the $^1/_{16}$-in. extrudates, which value is in line with the theoretical increase as calculated from L_p data. For the undiluted bed a 45% higher activity was measured which is not in line with the theory of Thiele.

A similar phenomenon has been reported by Gustafson (1975). However, because of enhanced channelling, the too small catalyst bed setup led to unrealistically low activity data on large particles as compared with the data on the small particles and polylobal particles. Hence, the above conclusion is based on incorrect activity data.

Test in Undiluted Catalyst Beds Under High Mass Velocities. Catalysts of series D have been tested in an *undiluted* bed of 850 mL catalyst. Details are given in Tables II and V. The highest mass velocity used was 2.2 kg/cm² s, which is near to commercial mass velocities.

The activity levels of the catalysts were, within the test accuracy, equal to those measured in the small *diluted* catalyst beds. From the particle size effect the Thiele numbers were determined by a graphical method. The results of tests in undiluted as well as diluted beds are shown in Figure 5. The effects of particle sizes on the effectiveness factors and Thiele numbers are the same with the two test methods. The same applies for tests with Kuwait atmospheric residue on catalysts from series B (Table VI). So the conclusions given in section I are also valid for high mass velocity tests in undiluted catalyst beds.

III. Advantages and Disadvantages of Polylobes

The shape and size of catalyst particles will influence properties such as activity, degree of packing (bulk den-
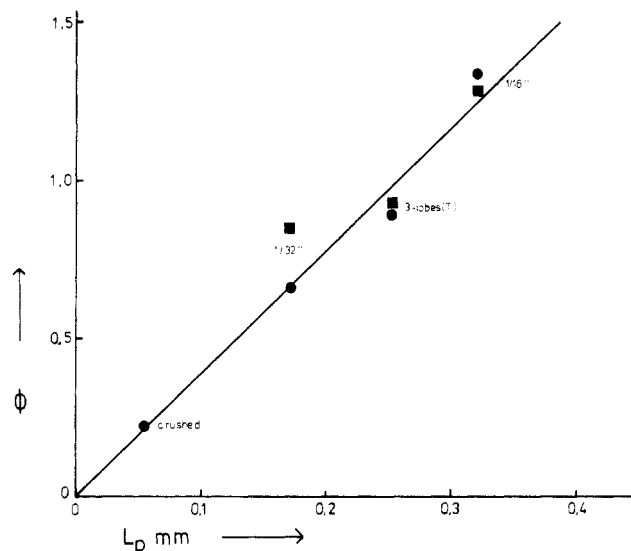

Figure 5. Thiele number as a function of particle size in VGO tests with small diluted and large undiluted catalyst beds: ●, 75 mL diluted catalyst bed; mass velocity 0.11–0.16 kg/m² s; ■, 850 mL undiluted catalyst bed; mass velocity 1.1–2.1 kg/m² s.

sity), pressure drop, strength, and liquid distribution. A large particle will often have a low activity, a high strength, and will cause a low pressure drop. A compromise choice will therefore have to be made of particle size and shape, depending on the prevailing process conditions. From the present paper, however, some more general rules may be derived.

In industry different methods are used for measuring the strength. The relative strength data obtained are not only dependent on catalyst properties such as shape, bed porosity, and intrinsic strength but also on the method applied. We observed different strength ranking for the noncylindrical and cylindrical extrudates to which the various methods were applied.

A full description of all parameters involved is beyond the scope of this paper. In general, it may be concluded that particular polylobes such as quadrulobes and trilobes have an acceptable strength. Besides, the measured strength is a derived parameter. Only the pressure drop and pressure drop increase during the commercial operation are important factors. Strength requirements are intended to prevent pressure drop increase due to breakage. This parameter can only be monitored in the commercial unit.

The pressure drop in the catalyst bed is related to length, diameter, shape, and bed porosity. Noncylindrical particles have a higher bed porosity. This contributes to obtain a lower pressure drop in the catalyst bed, as is indicated by the Carman equation. This influence of the porosity is also shown by pressure drop experiments with gas, carried out on the samples of series B and D.

Because of the higher bed porosities, the use of smaller cross sections also leads to the same or even lower pressure drops than for cylindrical shapes. Rather than the initial pressure drop, it is the pressure buildup with time due to fouling processes which must be considered to be of extreme importance.

The higher bed porosity of noncylindrical particles will generally have a favorable influence on this mechanism (Young, 1977). However, the ultimate requirements with regard to pressure drop and strength are determined by the type of process. The use as an additional parameter of polylobes allows making a better choice with respect to ΔP in the typical process.
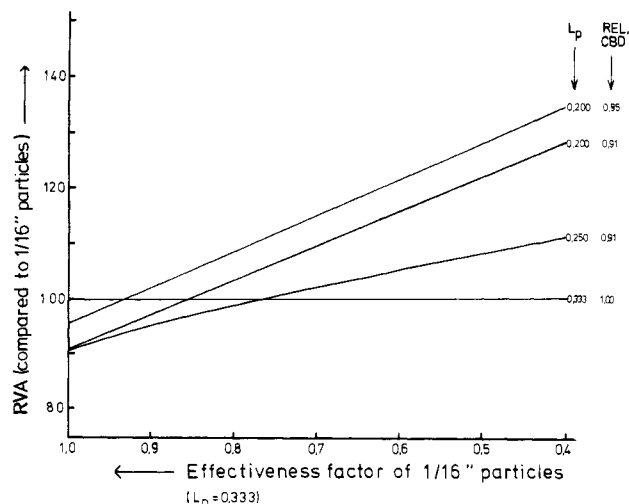
Figure 6. Relative volume activity as a function of the level of diffusion limitation.

Finally, a choice can be made as far as activity is concerned. Shape and size are determinative of the ratio of volume to outer surface of the particle and of the compacted bulk density. The activity is also influenced by diffusion limitation. The effects are illustrated in Figure 6, in which the relative volume activities of particles having two different sizes and various densities are compared with a standard catalyst of $1/16$-in. cylinders. Diffusion limitation will result in a decrease of the activity per unit weight. This decrease will be smaller for catalysts with lower L_p values and will result in higher *relative weight* activities of catalysts having L_p <0.333 mm.

Severe diffusion limitation may even result in higher *relative* volume activities despite the lower density of the catalysts. For light feedstocks, the effectiveness factor will be high (0.8–1.0). Then even lower volume activities may be expected for differently shaped extrudates. The relative weight activity will be not very different from that of $1/16$-in. cylinders. For heavier feedstocks such as vacuum gasoil and residue the use of polylobally shaped extrudates will often result in both higher weight and higher volume activities. So for activity reasons a noncylindrical shape is of advantage. The use of polylobes permits new choices as far as activity/pressure drop/strength are concerned. The choice is dependent on the diffusion limitation in the process. Both the catalyst formulation (e.g., activity, pore diameter, preparation method) and the process (feedstock and conditions) play a very important role in this choice.

Conclusions

The activity of noncylindrical extrudates can be predicted from the particle size by applying the diffusion limitation theory. The relation between activity and particle size, if defined as the ratio of volume to outer surface, was found to be independent of the shape of the extrudates.

The activity of noncylindrical extrudates may be higher than that of cylindrical particles because it permits the use in the catalyst bed of a smaller particle size at an equal pressure drop.

The activity advantage of polylobes over cylindrical particles depends on the degree of diffusion limitation. The combination of feedstock, catalyst properties, and operating conditions is determinative of the specific advantage of noncylindrically shaped extrudates.

In experiments with *small undiluted* catalyst beds, unrealistically high relative activities are observed for polylobal catalysts compared with cylindrical extrudates. However, the high activities are not observed at high mass velocities, which shows that these results are not applicable in predicting practical results in the HDS-unit.

Nomenclature

C = concentration, mol/m^3
C_∞ = concentration at equilibrium time, mol/m^3
C_0 = concentration at t = 0, mol/m^3
k = $[1/(n-1)][(1/(S^{n-1})) - (1/(S_0^{n-1}))] \times LHSV$, (m^3/m^3 h) $(1/(\text{wt } \%)^{n-1})$
k_w = as above, based on equal weights, (m^3/m^3 h) $(1/(\text{wt } \%)^{n-1})$
k_i = intrinsic rate constant
$LHSV$ = liquid hourly space velocity, m^3/m^3 h
n = order of reaction
S = sulfur content product, wt %
S_0 = sulfur content feedstock, wt %
RWA = k_w (catalyst)/k_w (standard catalyst)
CBD = compacted bulk density, kg/m^3
η = effectiveness factor
ϕ = Thiele number = $L_p(k_i/D)^{1/2}$
L_p = particle size = volume/outer surface, mm
D = diffusion rate constant = F (porosity, preparation method, pore diameter), m^2/s

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