

Performance of the monolithic stirrer reactor: applicability in multi-phase processes

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Received 27 February 2004; received in revised form 9 July 2004; accepted 27 July 2004
Available online 15 September 2004

Abstract

The novel monolithic stirrer reactor is a promising replacement for a conventional slurry reactor in multi-phase reactions. In this reactor monolithic structures are used as stirrer blades, creating a catalytic stirrer. The most important advantage of the monolithic stirrer reactor is the easy catalyst handling. The influences of different parameters on the performance of the monolithic stirrer reactor in the selective hydrogenation of 3-methyl-1-pentyn-3-ol were investigated. The decreasing catalyst layer thickness with increasing cell density proved to be beneficial for the performance of the monolithic stirrer reactor. An increasing stirrer speed caused an increase in the activity because of the higher flow through the monolith channels and the improved gas–liquid mass transfer. If the length of the monolithic stirrer blades was smaller than 0.03 m the performance of the monolithic stirrer was enhanced.

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Keywords: Multiphase reactors; Catalyst support; Selectivity; Catalysis; Mass transfer; Hydrogenation

1. Introduction

Heterogeneously catalysed gas–liquid reactions are very common in the chemical industry. A wide range of reactor types is available for this type of reactions. In the production of fine chemicals the stirred tank reactor is mostly applied, because it can be used for a variety of reactions. There are a few drawbacks with respect to the handling of the slurry catalyst and scale-up of such a process. The most important of these being the separation of the catalyst from the reaction mixture, attrition and agglomeration of the catalyst particles.

An alternative is to combine the mixing and catalytic function by fixing the catalyst to a stirrer. Carberry (1964) introduced the stirred basket reactor as a laboratory reactor for the measurement of kinetics without external mass transfer limitations in the gas phase. The stirred basket reactor is also

used for measurements of kinetic parameters in solid catalysed gas–liquid phase reactions; examples from the literature include different hydrogenation reactions (Kenney and Sedriks, 1972; Turek and Winter, 1990) and the oligomerisation of propylene (Peratello et al., 1999). Various researchers tried to overcome the internal mass transfer limitations associated with pellets, by using more permeable catalyst supports. Bennett et al. (1991) mounted different configurations of monolith blocks on a stirrer shaft to measure the kinetics of the oxidation of propane in the gas-phase. Liakopoulos et al. (2001) used spinning baskets filled with two monolith pieces to determine the gas-phase kinetics of the oxidation of acetaldehyde.

The use of catalytic stirrer reactors for the chemical industry has not received much attention. The use of a spinning basket reactor type filled with rather large catalyst pellets for hydrodechlorination in the industrial cleaning of liquid waste streams was proposed by Schioppa et al. (2001). It was shown that internal mass transfer limitations play an important role in the use of pellets. A patent reported the use

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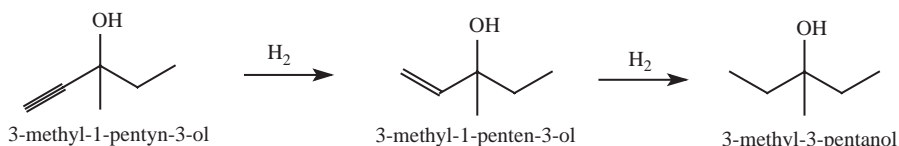


Fig. 1. Hydrogenation of 3-methyl-1-pentyn-3-ol.

of a spinning basket reactor filled with monolith pieces in the polymerisation of organosilicon compounds (Kolaczowski, 1994). It was claimed that the performance of the spinning basket filled with a monolithic catalyst was promising in comparison with baskets filled with a pelleted catalyst.

A monolithic stirrer reactor in which monolithic structures are mounted on the stirrer shaft as impeller blades can be an attractive replacement for the traditional slurry reactors. It makes the separation of the catalyst easier, prevents attrition of the catalyst, and the safety can be improved when viscous liquids are used because the reaction can be stopped by stopping the impeller. These advantages together with the possibility of using the stirrer in existing stirred tank reactors make the concept interesting for the fine chemical industry. Edvinsson et al. (1998) showed that the principle of a monolithic stirrer reactor concept works for low viscosity liquids. The open structure of the monolithic stirrer blade enables a high throughput of reactants and a large geometrical area. However, this preliminary study concentrated only on the hydrodynamic aspects of the novel reactor type.

In this paper we will demonstrate the industrial possibilities of the monolithic stirrer reactor in a heterogeneously catalysed gas–liquid reaction. The hydrogenation of 3-methyl-1-pentyn-3-ol, shown in Fig. 1, was used as a model reaction for a typical fine chemical hydrogenation of a functionalised alkyne. The reaction is catalysed by a Pd on silica catalyst (Spee et al., 2001; Nijhuis et al., 2003). Different aspects of the operation and design of the monolithic stirrer reactor are examined, like the influence of the stirrer speed, the cell density, and the length of the monolithic structures.

2. Experimental

2.1. Catalyst preparation

Monolithic structures are coated with a washcoat of silica to function as a carrier for the palladium. The application of the coat layer of silica onto the cordierite monolith samples was done with a slurry coating method based on the description in the review from Nijhuis et al. (2001). Before use the monolith substrates were calcined at 1273 K. A silica slurry was prepared from water with 18.5 wt% Davisil grade 643 (Aldrich) as the silica source and 7 wt% Ludox AS40 (Aldrich) colloidal silica as a binder. The pH of the slurry mixture was brought to 9 with a NaOH solution. This mixture was ball milled for approximately 20 h. After dip-

Table 1

Characteristics of the monolithic structures with different cell densities			
Cell density (cpsi)	Channel diameter (mm)	Wall thickness (mm)	Specific surface area (m ² /m ³)
50	2.98	0.61	924
200	1.49	0.31	1848
400	1.09	0.18	2703

ping the monolithic structures in the silica slurry for approximately 1 min, the channels were opened with pressurised air. The monoliths were then dried overnight at room temperature, before calcining at 723 K for 4 h.

All the monolithic structures used were cylindrical with a diameter of 0.043 m. The influence of the length was investigated by using structures with a length of 0.01, 0.02, 0.03, 0.04, 0.05, 0.06, and 0.08 m. The standard length of the monoliths used in the other experiments was 0.04 m. The different cell densities of the monolithic structures used were 50, 200, and 400 cpsi (cells per square in.). Table 1 shows the characteristics for these cell densities.

After the application of the silica layer 2 wt% palladium was deposited by means of an exchange procedure (Spee et al., 2001; Nijhuis et al., 2003). For this purpose 4.2 wt% palladium (II) acetate (Alfa Aesar, 47.4 wt% palladium) on silica was dissolved in toluene. The silica coated monoliths were added to the palladium acetate solution. The yellow/brown solution discoloured under continuous circulation for 24 h at room temperature while the monoliths coloured yellow/orange. After the impregnation the toluene solution was shaken out of the channels and the monoliths were dried overnight at room temperature. The acetate was removed by calcining at 523 K for 4 h. The catalyst was reduced in situ at room temperature in hydrogen atmosphere for 2 h before the catalyst performance testing.

2.2. Reactor set-up

The hydrogenation of 3-methyl-1-pentyn-3-ol was performed in a monolithic stirrer reactor as shown in Fig. 2. Different stirrer configurations were used. In the so-called standard configuration, which is shown in Fig. 2, two monolithic pieces were attached to the stirrer shaft. The other configurations use four monolithic structures mounted on the stirrer shaft: two monoliths above and parallel to the standard two monoliths, Fig. 3a, two monoliths perpendicular above the standard two monoliths, Fig. 3b, and four monolithic structures in one plane, Fig. 3c. The pieces of monoliths were

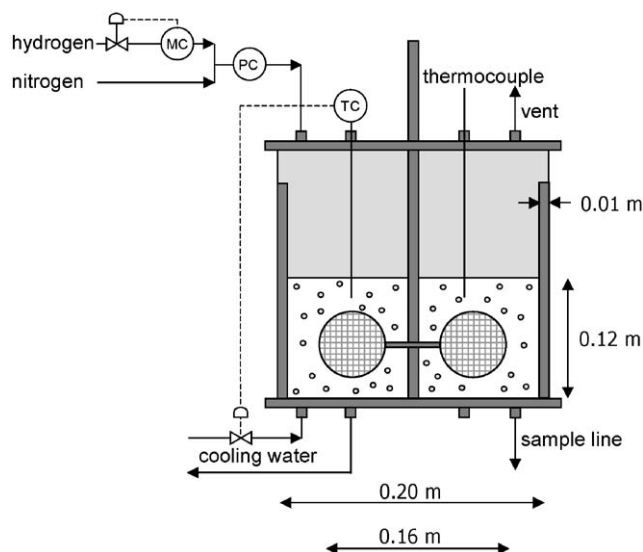


Fig. 2. Schematic representation of the standard monolithic stirrer reactor set-up.

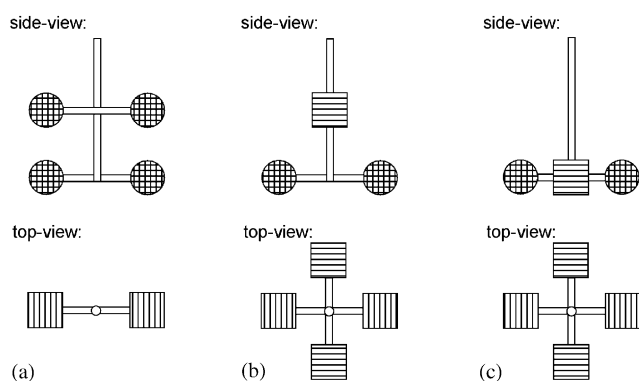


Fig. 3. Schematic drawings of the different stirrer configurations containing four monolith pieces: (a) straight above each other, (b) perpendicular above each other, and (c) four in one plane.

always mounted on the stirrer so that the tips were 0.16 m apart.

Depending on the configuration the reactor contained 2500 or 4000 g of solvent ethanol to which at the start of the reaction 200 mol/m³ of the alkyne was added. The conditions for catalytic testing were 1.2 bara hydrogen pressure and a temperature of 298 K. The stirrer rate was varied from 0–800 rpm. The course of the reaction was followed with a Varian CP 3380 gas chromatograph equipped with an 1177 FID detector, Varian CP8400 auto sampler, and a CP-SIL-8 column (length 60 m, internal diameter 0.25 mm, and film thickness 0.25 μm).

Fig. 4 shows the course of the hydrogenation of 3-methyl-1-pentyn-3-ol at a stirrer speed of 290 rpm. As can be seen the alkyne, 3-methyl-1-pentyn-3-ol, reacts with hydrogen to form the alkene, 3-methyl-1-penten-3-ol. When nearly all the alkyne is converted overhydrogenation of the alkene to

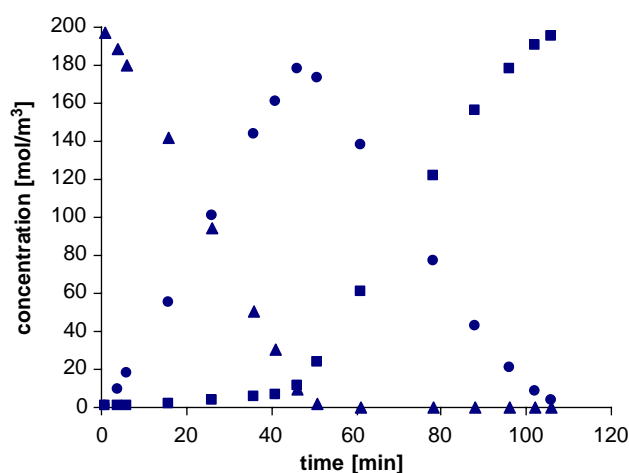


Fig. 4. Hydrogenation of 3-methyl-1-pentyn-3-ol with a 400 cpsi Pd on silica coated monolith with the standard configuration at 298 K, stirrer speed of 290 rpm, 1.2 bara H₂, 200 mol/m³ alkyne: (▲) alkyne concentration, (●) alkene concentration, and (■) alkane concentration.

form the alkane starts. The performance is expressed in terms of both activity and selectivity. The activity is calculated by determining the rate of alkyne hydrogenation, i.e. the slope of the alkyne concentration against time. To determine the selectivity the rate of alkene formation is divided by the rate of alkyne hydrogenation.

3. Results and discussion

3.1. Results of the silica coating of the monolithic structures

Table 2 shows the results of the catalyst preparation of the monoliths with different cell densities. The total amount and the weight percentage of silica on the two monoliths used in the catalytic testing is shown. The density of the silica coat layer was estimated to be 850 kg/m³. With this value an estimation of the thickness of the coat layer is made. These values, shown in Table 2, were in agreement with visual observations with the help of scanning electron microscopy (SEM). The total amount of silica that was deposited by the slurry coating method is approximately 2 g for all the cell densities. The thickness of the coat layers increases with decreasing cell density. This is related to the preparation method of the silica coat layer. An increase in channel diameter with a decrease in cell density results in

Table 2

Amount silica deposited after coating for two monolithic structures, length 0.040 m and diameter 0.043 m, with different cell densities

Cell density (cpsi)	Silica coat layer (g)	Silica coat layer (wt%)	Palladium dispersion (μm)	Palladium dispersion (%)
50	1.88	2.66	20	22
200	2.40	4.16	13	10
400	2.15	3.52	5	5

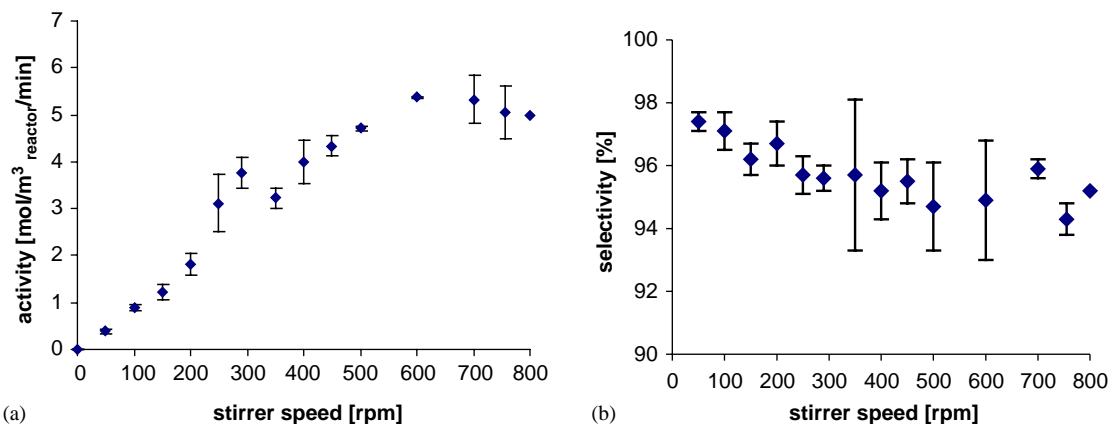


Fig. 5. Performance of a 400 cps Pd on silica coated monolith as a function of the stirrer speed at 298 K, 1.2 bara H₂, 200 mol/m³ alkyne: (a) activity and (b) selectivity.

lower flows during the emptying of the channels, leaving more silica on the monolith channel walls at low cell densities. The dispersion of the palladium catalyst, also shown in Table 2, decreased with increasing cell density. It is thought that this trend is related to the drying of the monolith structures after the exchange of palladium acetate. Due to the decrease in channel diameter the drying at room temperature slows down, resulting in migration of the not yet bonded palladium.

3.2. Influence of the stirrer speed

Fig. 5a shows the activity of the 400 cps monoliths in the hydrogenation of 3-methyl-1-pentyn-3-ol as a function of the stirrer speed. The overall effect of the increasing stirrer speed is that the activity increases. This trend can be explained by an increasing flow through the monolithic channels with increasing stirrer speed (Kritzinger et al., 2002). The mass transfer is increased with this increasing throughput. Another effect that plays an important role is the gas–liquid mass transfer of hydrogen. At stirrer speeds above 200 rpm the turbulence created by the stirrer entrains hydrogen bubbles in the liquid phase. This leads to a strong increase in the exchange area between the gas and liquid phase. A step in the increase of the activity can be seen above a stirrer speed of 200 rpm.

The effect of the stirrer speed on the selectivity towards 3-methyl-1-penten-3-ol is shown in Fig. 5b. Overall a decrease in selectivity is observed with an increasing stirrer speed. This decrease in selectivity is related to the increase in hydrogen mass transfer. A higher mass transfer rate leads to a larger availability of hydrogen and over-hydrogenation towards the alkene can take place more easily.

3.3. Influence of the monolith cell density

In Table 3 the performance of the different cell densities in the hydrogenation of 3-methyl-1-pentyn-3-ol is presented. The cell densities used were: 50, 200, and 400 cps. The per-

Table 3

The activity in the hydrogenation of 3-methyl-1-pentyn-3-ol for the different cell densities

Cell density (cps)	Activity (mol/m ³ reactor/min)	(mmol/m ² monolith/min)	Selectivity (%)
50	0.91 ± 0.12	25.0 ± 6.0	96.0 ± 0.1
200	1.43 ± 0.12	20.5 ± 1.8	96.2 ± 0.4
400	3.76 ± 0.34	37.6 ± 3.4	95.8 ± 0.5

Experiments performed at with the standard configuration at 298 K, 1.2 bara H₂, 200 mol/m³ alkyne.

formance is expressed in terms of activity per reactor volume, the activity normalised for the monolith surface area, and the selectivity towards 3-methyl-1-penten-3-ol. The activity per reactor volume clearly increases with increasing cell density. The increase in surface area with the increase in cell density, as shown in Table 1, makes the catalyst more easily accessible. In other words, the mass transfer of the reactants towards the catalyst layer is enhanced. When the activity is expressed per square meters of monolithic surface area it can be seen that the activity of the 400 cps monoliths is higher than for the 200 and 50 cps monoliths. The thicker catalyst layers in the case of the 200 and 50 cps cause internal mass transfer limitations, resulting in a lower activity. The palladium dispersion starts to play a role in the activity of the 50 and 200 cps. Since the dispersion of the 50 cps is higher, this cell density shows a higher activity per square meter of monolith surface. While in the case of the 400 cps the catalyst layer is so thin that external mass transfer determines the activity and dispersion does not play a role. The selectivity towards the alkene was for all cell densities approximately 96%. There is no clear influence of the cell density on the selectivity towards the desired alkene, because the hydrogen mass transfer dominates the reaction.

3.4. Influence of the length of the monolithic structures

The influence of the length of the monolithic structures on the performance was investigated. The lengths of the

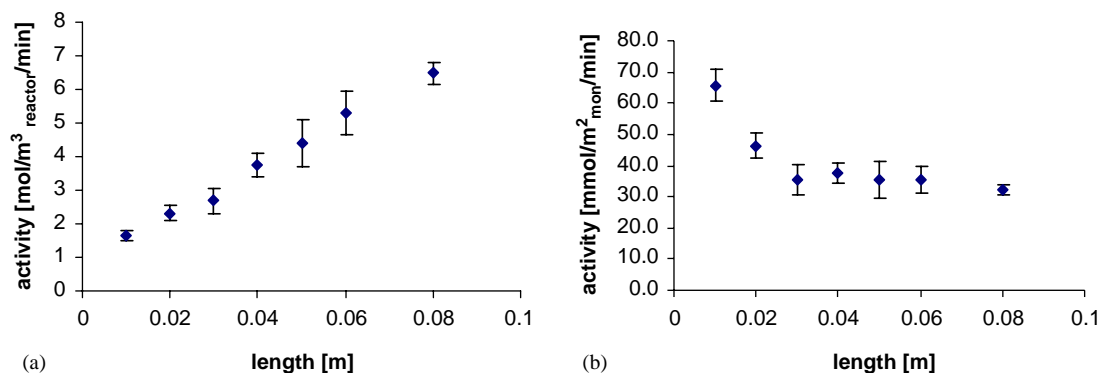


Fig. 6. Activity of 400 cps Pd on silica coated monoliths as a function of the length of the monolithic structures at 298 K, 1.2 bara H₂, 200 mol/m³ alkyne: (a) activity per reactor volume and (b) activity normalised for the surface area available.

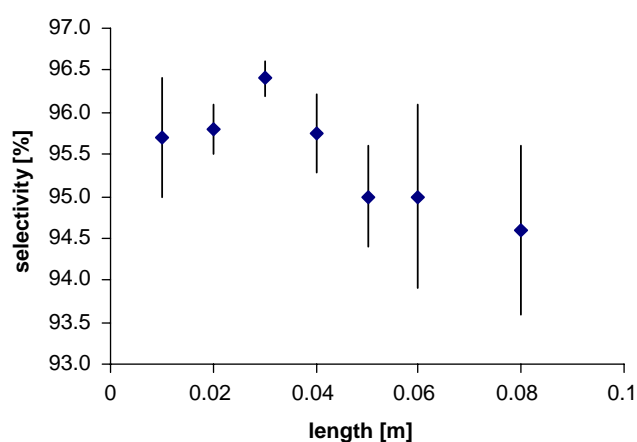


Fig. 7. Selectivity of 400 cps Pd on silica coated monoliths as a function of the length of the monolithic structures at 298 K, 1.2 bara H₂, 200 mol/m³ alkyne.

monoliths used were: 0.01, 0.02, 0.03, 0.04, 0.05, 0.06 and 0.08 m. Fig. 6a shows the activity per reactor volume, while Fig. 6b shows the activity normalised for the specific surface area available. The activity per reactor volume increases with the length of the monolithic structures. This is a direct consequence of the increase of the total amount of catalyst with the increasing monolith length. When the activity is normalised for the surface area the activity decreases with increasing length of the monolithic structures until at a length of 0.03 m the activity becomes constant. External mass transfer limitations might play a role here. In the case of the short monoliths, the flow in the monolith channel is still in development towards fully laminar flow. At the entrance region the velocity profile of the liquid is flat and in the case of the monolithic stirrer reactor the liquid enters with an angle. This so-called entrance effect is beneficial for the mass transfer, resulting in higher activities for shorter monolith structures.

Fig. 7 shows the effect of the monolith length on the selectivity towards 3-methyl-1-penten-3-ol. The selectivity towards the alkene decreased with the increase in length of

Table 4

The activity in the hydrogenation of 3-methyl-1-pentyn-3-ol for different stirrer configurations

Configuration	Activity (mol/m ³ _{mon} /min)	Selectivity (%)
Straight above (Fig. 3a)	45.3 ± 1.7	94.6 ± 0.7
Perpendicular above (Fig. 3b)	50.8 ± 4.7	96.5 ± 0.6
Four in one plane (Fig. 3c)	78.1 ± 5.6	96.4 ± 0.8
Standard (Fig. 2)	94.9 ± 7.1	95.8 ± 0.5

Experiments performed at 298 K, 1.2 bara H₂, 200 mol/m³ alkyne.

the monolithic structures, from a value of 96.7–95.6%. This is possibly related to the formation of the laminar flow in the monolith channels. With increasing length of the monolithic structures the laminar film for mass transfer increases (Shah and London, 1978), resulting in increasing external mass transfer limitations and a lower selectivity.

3.5. Comparison of the different stirrer configurations

Table 4 shows the results for the hydrogenation of 3-methyl-1-pentyn-3-ol with the different configurations of the monolithic stirrer containing four monoliths. Both the activity per monolith volume and the selectivity are shown. For comparison reasons the standard monolithic stirrer with two monolithic structures is also shown again. When the results are compared for the perpendicular and straight placement of an extra pair of monoliths above the standard two monoliths it can be seen that the placement of the monoliths straight on top of each other shows a slightly lower activity and selectivity. Multiple experiments showed that this decrease in performance is reproducible. This could be an indication that the macro-mixing is better when the monoliths are placed in the perpendicular way. The primary flow created by a pair of monoliths is in the radial direction. By placing the monoliths perpendicular on top of each other there might be a better interaction between the two radial flow regimes created by the different monolith pairs. The stirrer configuration containing four monoliths in one plane shows the highest activity for

Table 5

Comparison of the activity normalised for the amount of palladium and the maximum yield of alkene for a slurry reactor and the standard monolithic stirrer reactor

Catalyst	Activity (mol/g _{Pd} /min)	Maximum yield (%)
Slurry Davisil 643	0.46	90.2
Monolith	0.41	88.5

Experiments performed at 298 K, 1.2 bara H₂, 200 mol/m³ alkyne.

all the configurations containing four monoliths, because of the higher monolith concentration. This means that a higher activity can be easily obtained by using a monolithic stirrer with multiple monolithic pieces.

3.6. Comparison between the monolithic stirrer reactor and a slurry reactor

There have been earlier attempts presented in literature to use a catalytic stirrer reactor in the selective hydrogenation of 3-methyl-1-pentyn-3-ol. Spee (1999) used silica coated glass stirrer blades and found an activity of 0.0003 mol/g_{Pd}/min. The low surface area of the glass stirrer blades resulted in this low activity. Nijhuis et al. (2003) used a monolithic stirrer reactor with all silica monoliths as carrier for the palladium catalyst. The surface area was increased by using monolithic structures as stirrer blades. Activities up to 0.013 mol/g_{Pd}/min were found. In comparison with the results of Spee (1999) with the same palladium exchange method the activities increased significantly. But all these activities were still much lower as the values found for the slurry catalysts. The use of the all-silica monoliths caused severe internal mass transfer limitations. With the monolithic stirrer reactor as developed in this research much higher activities were obtained, up to 0.405 mol/g_{Pd}/min. This higher activity is reached by combining the high surface area of the monolithic structures with a thin easily accessible coat layer on the walls of the channel. Is this activity high enough to make the performance of the monolithic stirrer reactor comparable to that of a slurry reactor? Nijhuis et al. (2003) performed experiments in which the Davisil 643 is used as a slurry catalyst. This Davisil silica is used as the silica source in the coating of the monolithic structures in this work and thus forms a good basis for the comparison of the monolithic stirrer reactor with a conventional slurry reactor. Table 5 shows a comparison between the performance of the Davisil 643 silica used as a slurry catalyst and the monolithic stirrer reactor. This time the activity is expressed per gram of palladium, in both cases the palladium on silica loading was 2 wt%. The activity in the monolithic stirrer reactor is that of a 400 cpsi monolith at a stirrer speed of 600 rpm. The selectivity is expressed as the maximum yield of alkene. Both the activity and yield obtained with the two different reactor types are comparable. This makes the monolithic stirrer reac-

tor an interesting replacement for the conventional slurry reactors.

4. Conclusions

The monolithic stirrer reactor shows interesting characteristics in the hydrogenation of 3-methyl-1-pentyn-3-ol. Mass transfer characteristics play an important role in this fast selective hydrogenation reactor. The thinner silica coat layers with increasing cell density result in better internal mass transfer characteristics for the 400 cpsi, in comparison with the 200 and 50 cpsi monoliths. The activity of the 400 cpsi monoliths, in which internal diffusion limitations no longer play an important role, increases as a function of the stirrer speed. The increasing stirrer speed causes an increasing flow through the monolithic channels and an increasing gas–liquid mass transfer. When the lengths of the monolithic blades are varied a strong increase in this external mass transfer is observed at decreasing length below 3 cm. The laminar flow in the monolith channels is not yet developed. This entrance effect is beneficial for the performance of the monolithic stirrer. Placing more monolithic blades in one plane on the stirrer shaft is an easy way to improve the activity of the reactor. Combining the effect of the short monoliths and the placement of multiple blades could lead to an even more efficient monolithic stirrer reactor.

The performance of the monolithic stirrer reactor is comparable to that of the conventional slurry reactors. The most important advantage that can be obtained by using a monolithic stirrer reactor in industrial applications is the easy separation of the catalyst and reaction medium.

Acknowledgements

This research is financially supported by the Dutch Scientific Organisation NWO-STW and DSM. Corning Inc. is thanked for the supply of the monolithic samples. Shyreen Dahoe and Eugene van Brakel are gratefully acknowledged for their valuable contribution to this work.

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