Industrial Catalysis

Laboratory tour

group Industrial Catalysis

October 1999

	Set-up	Person	Poster
1	Multitrack (TAP)*)	Guido Mul	Multitrack.pdf
1a	Six-flow reactor *)	Freek Kapteijn	Javier.pdf
2	TEOM *)	Weidong Zhu	Adsorp4.pdf
3	TPR / TPS	Dick van Langeveld	
4	Raney-nickel autoclave *)	Gerard Ruigrok	
5	FCC set-up *)	Erick Gamas	Marion1.pdf Marion2.pdf
6	Rotating monolith *)	Igor Babich	Igor.pdf
7	Hydrodynamics monolith	Johan Heiszwolf	
8	Slurry autoclave+Berty or Monolith pilot*)	Xander Nijhuis	Pilot1.pdf Pilot2.pdf
9	Diesel exhaust cleaning*)	Barry van Setten	Barry.pdf

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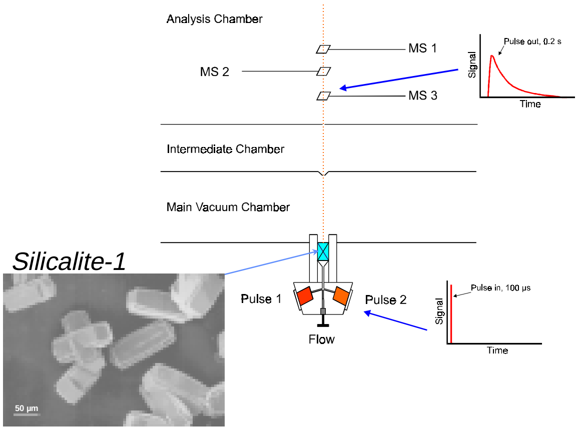
Transient Measurement of Adsorption and Diffusion in Microporous Materials

Introduction

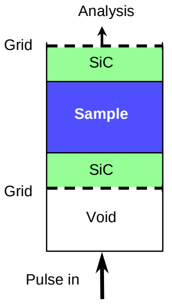
The transient adsorption behavior of gases on microporous materials was studied using the Multitrack system, a new type pulse reactor for catalyst testing. Schematically Multitrack can be represented as:



Adsorption, desorption and diffusion parameters of gases can be simultaneously determined by modeling the measured pulse responses. For the adsorption behavior of a number of gases on silicalite-1 this new method is demonstrated.

Model

The differential equations describe the pulse responses measured. The marked parameters are estimated using the Levenberg-Marquardt minimization method. The bed properties are determined separately by measuring and modeling a pulse of a non-adsorbing gas.



Gas phase in bed

$$\varepsilon_b \frac{\partial C_z}{\partial z^2} = D_{gas} \frac{\partial C_z}{\partial z^2} - \underset{\uparrow}{k_a} N_s C_z (1-\theta_{z,R}) + \underset{\uparrow}{k_d} N_s \theta_{z,R}$$

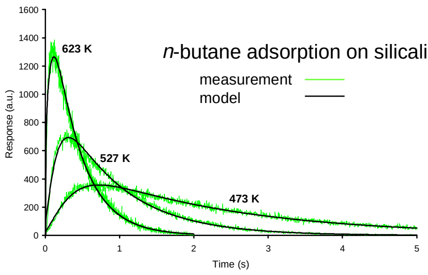
Adsorbed phase in particle

$$\frac{\partial \theta_{z,r}}{\partial t} = \underset{\uparrow}{D_{pore}} \frac{\partial^2 \theta_{z,r}}{\partial r^2}$$

Adsorption on outside of particle

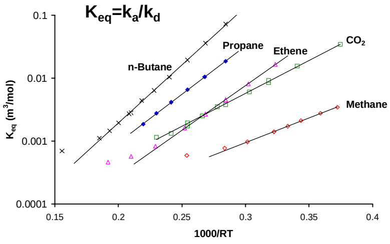
$$-\underset{\uparrow}{D_{pore}} \frac{\partial \theta_{z,r}}{\partial r} = \underset{\uparrow}{k_a} C_z (1-\theta_{z,R}) - \underset{\uparrow}{k_d} \theta_{z,R}$$

The model used is able to describe the measurements well. The effect of the temperature on the adsorption behavior is clearly visible.



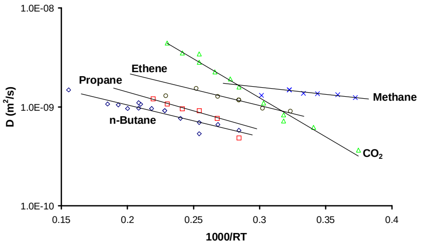
Results

From the rate constants for adsorption and desorption the equilibrium constant for adsorption can be calculated, which yields the heat of adsorption, visualized in a van ‘t Hoff-plot.



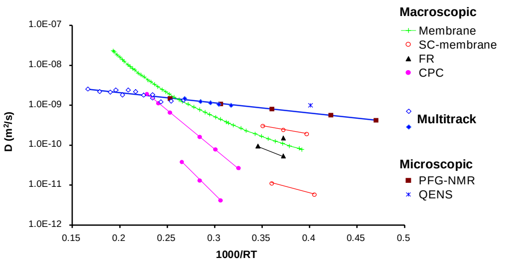
	$-\Delta H_{ads}$ (kJ/mol)
Methane	**18.1**
Ethene	**32.7**
Propane	**35.5**
***n*-Butane**	**42.5**
CO_2	**24.1**

The determined diffusivity is shown in the figure below, together with the activation energies for diffusion.



	$E_{act, diff}$ (kJ/mol)
Methane	**3.1**
Ethene	**7.5**
Propane	**8.7**
***n*-Butane**	**7.4**
CO_2	**18.1**

Compared to other methods, the presented one is the first macroscopic method that yields diffusivities in excellent agreement with those obtained from microscopic methods. This agreement can be explained by the absence of carrier-gas influences or external transport limitations in the current method. These aspects easily affect the extraction of the diffusivity in conventional macroscopic methods.



Conclusions

- The presented method yields simultaneously the intrinsic adsorption and diffusion parameters of gases in microporous materials.
- The adsorption parameters obtained are in good agreement with reported values.
- The diffusivities are in good agreement with diffusivity values obtained using microscopic methods

Adsorption of Butane Isomers in Silicalite-1

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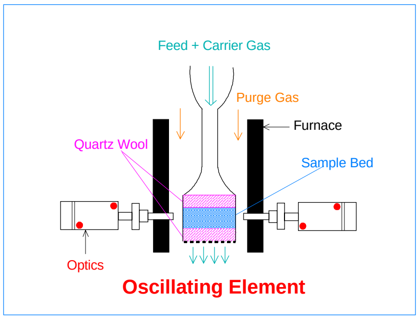
Introduction

Adsorption data of components are of the utmost importance for a good design of separation units based on differences in adsorption strengths and/or in diffusivities. Often data are obtained by either a volumetric method or a gravimetric method. Both methods have their advantages and disadvantages, and generally large corrections have to be made on the raw experimental data.

Recently, a new method has been commercialized in which the mass change of a vibrating tube is determined by frequency measurements of this vibration, the so-called tapered element oscillating microbalance (TEOM) mass analyzer (Chen et al., 1996). The main feature of the TEOM is an oscillating element that is based on inertial forces, instead of weight, to measure the amount adsorbed. An important advantage of this technique is that the gas which has to adsorb flows through a packed bed of adsorbent material. So, external mass transport limitations and heat effects are nearly absent in this technique and it can be used not only for equilibrium measurements, but also for transient uptake and desorption experiments.

Standard Operating Range of the TEOM Sample size: 100 mg; Gas flow rate: 500 ml.min^{-1};Temperature: 300-900 K; Pressure: 0-10 bar.

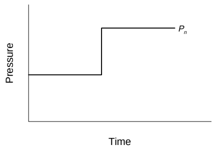
TEOM Principle



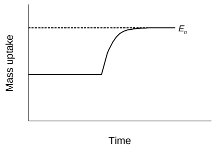
Mass uptake

$$\Delta m = K_0\left(\frac{1}{v_1^2}-\frac{1}{v_0^2}\right)$$

Adsorption Measurements



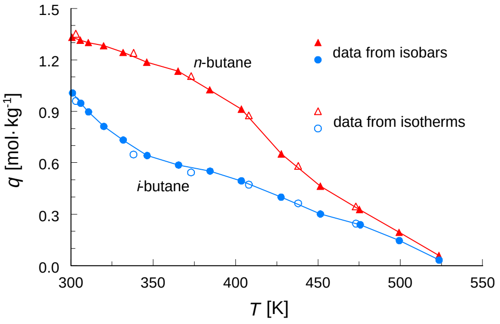
Pressure is changed and then held constant during adsorption at the set point P_n



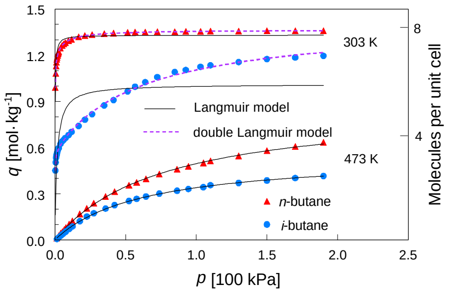
Mass uptake data are acquired and analysed in real time to determine kinetic parameters and the equilibrium uptake E_n

Isobars at 50 kPa

i-butane: stepwise adsorption behavior



Isotherms



Isotherm Models

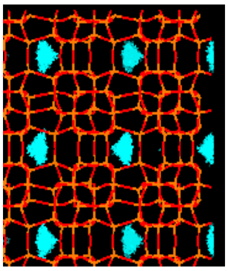
Double Langmuir: $q = q_{sat,1}\frac{K_1 p}{1+K_1 p} + q_{sat,2}\frac{K_2 p}{1+K_2 p}$

Langmuir (high T): $q = q_{sat}\frac{Kp}{1+Kp}$

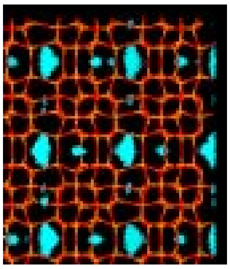
Adsorbate Location in Silicalite-1

Density distribution of *i*-butane in silicalite-1 micropores (Vlugt et al., 1998)

303 K and 0.1 kPa



303 K and 100 kPa



Comparison with Literature Data

Isosteric Heat of Adsorption [kJ·mol^{-1}] at Zero Coverage

	TEOM	Calor.[1]	G. C.[2]
methane	19		20
ethane	31	32	30
propane	41		41
n-butane	53	52	50
i-butane	48	49	49

1: Calorimetry (Thamm, 1987).
2: Gas Chromatography (Hufton and Danner, 1993).

Conclusions

- The TEOM is a powerful technique for measuring adsorption parameters in microporous materials.
- The adsorption of *i*-butane in silicalite-1 reveals some discrete structural heterogeneity for light alkanes in silicalite-1.
- The molecular simulation confirms that *i*-butane preferentially occupies the intersections in silicalite-1, while at higher pressures and lower temperatures it starts to fill the channels.
- Other applications: catalyst deactivation; diffusivity from uptake/desorption; and binary mixture adsorption.

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A Novel Bench Scale FCC Riser Reactor
- Short Contact Time Experiments -

Introduction

The fluidized catalytic cracking (FCC) process is one of the major processes in a modern refinery. Small changes in the selectivity of the process have a substantial influence on refinery economics. This makes FCC catalyst and feedstock testing a very important issue. Simulation of the industrial situation on a laboratory scale must be carried out in such a way that the original concepts of the process are mimicked in a well defined reaction system. To be able to make the translation from laboratory tests to the industrial process, a test reactor should provide intrinsic kinetic data, and has to meet a number of conditions:

- minimal mass and heat transport limitations;
- well defined contact time of oil and catalyst with optimal contact efficiency;
- unambiguous mathematical description of the physical situation;
- preferably isothermal operation.

The Microriser

The microriser is a bench scale entrained flow reactor that has been designed to provide intrinsic kinetic data of fluidized catalytic cracking under industrially relevant conditions.

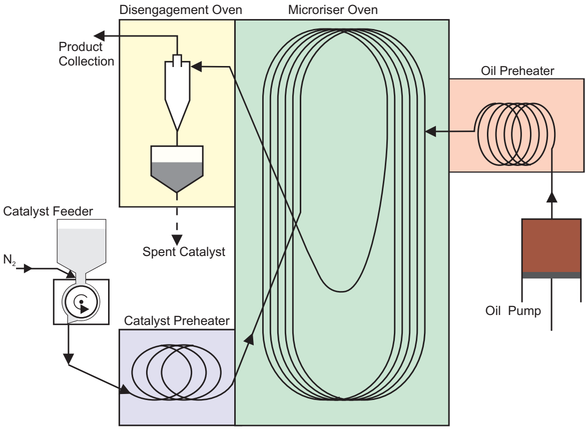

Figure 1: Outline of the microriser

The reactor is a folded tube with an adjustable length of maximal 20 m.

Hydrodynamics

The flow behaviour of the microriser has been studied with a cold flow model, using a typical FCC catalyst and air as carrier gas. When the carrier gas flow rate was decreased at a given catalyst flow rate, several transitions in flow behaviour were observed.

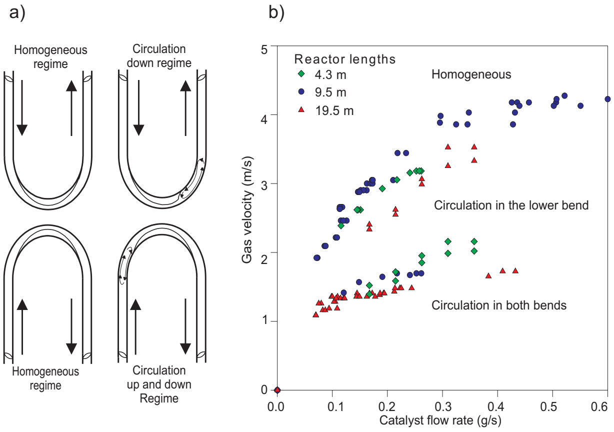

Figure 2: a) Different flow regimes observed in the microriser, b) flow map of the different flow regimes

Residence Time Distributions

The residence time distributions of the catalyst are given below.

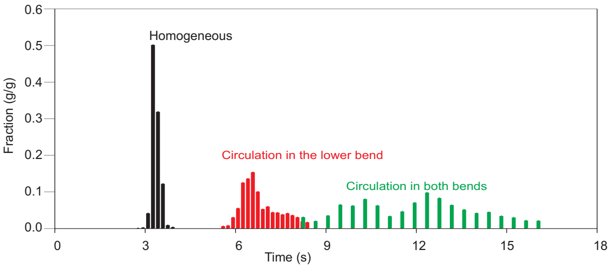

Figure 3: Residence time distributions in the different flow regimes

Modelling showed that the hydrodynamics in the homogeneous regime can be described with the plug flow model.

Cracking Experiments at Different Contact Times

Cracking experiments have been performed with a regular Kuwait vacuum gasoil (VGO) and two typical commercial cracking catalysts A and B. Catalyst A was presteamed and artificially deactivated, catalyst B was an equilibrium catalyst originating from an industrial unit.

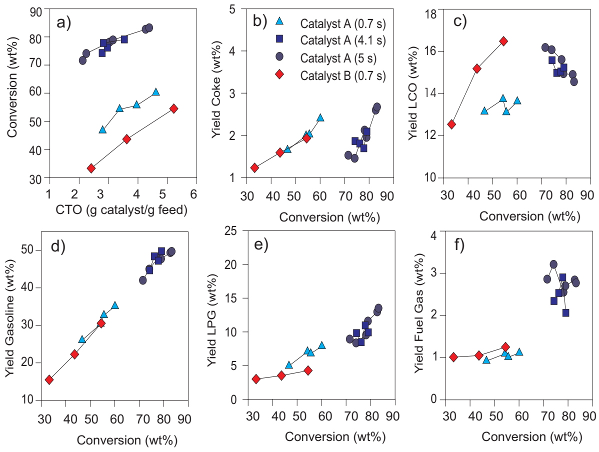

Figure 4: Results of cracking experiments with the microriser; a) conversion plot, b-f) yield plots

Major Observations

- Activity of catalyst A is higher than the activity of catalyst B.
- Results after 4 s are invariant with residence time.
- The majority of the coke is already formed at 0.7 s.
- The LCO (Light Cycle Oil) yield shows a series type of reaction.
- The gasoline and LPG yields at 0.7 s are in good agreement with yields at ≥ 4 s.
- Fuel gas production depends on residence time.

Conclusions

- Three different flow regimes have been observed: homogeneous flow, circulation down, and circulation up and down.
- The microriser is a plug flow reactor when operated in the homogeneous flow regime.
- For FCC, the experimental framework is more relevant at shorter residence times. Data obtained at more than 4 s were invariant with residence time.

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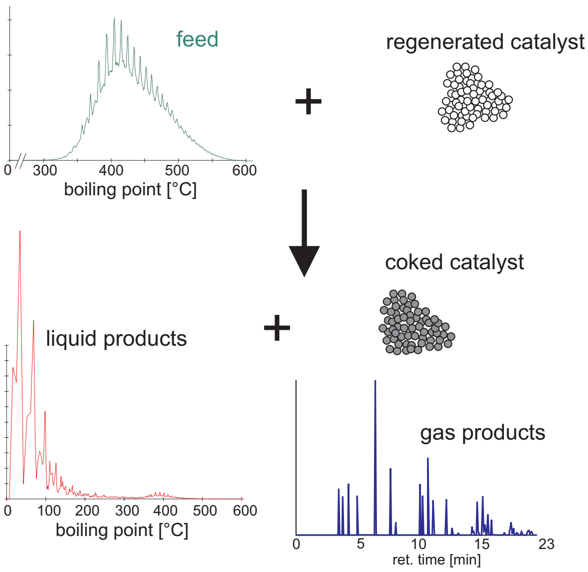
Kinetics of Catalytic Cracking

Introduction

Fluid catalytic cracking (FCC) is an important refinery process since the 50's. Research on reaction mechanisms and kinetics is still an ongoing process. One of the difficulties in catalytic cracking is the large amount of products involved. Cracking of even a simple paraffinic feedstock yields numerous products and largely increases its complexity. Therefore, usually different products are lumped into groups that are treated as a single component in a simple model to describe the reactions involved.



Objective

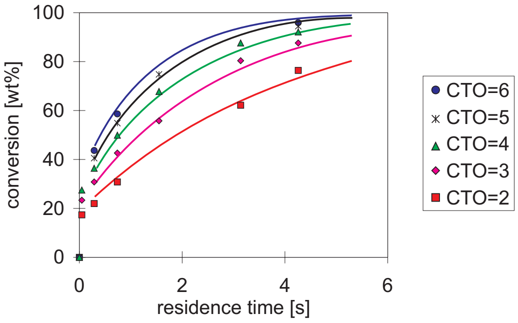
Development of a simple lumped kinetic model to describe catalytic cracking for residence times from zero to five seconds, a realistic range for industrial riser reactors.

Experimental

Experimental data were obtained using the microriser, a laboratory scale entrained flow reactor.

feedstock: Hydrowax (paraffinic, no contaminants)
catalyst: FCC equilibrium catalyst
residence time: 0.05 - 5 [s]
catalyst/oil, CTO: 2 - 8 [$kg_{cat}kg_{feed}^{-1}$]

Results

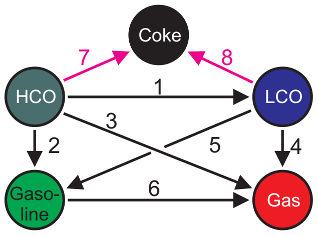


- Initially sharp increase of conversion;
- Additional conversion increase is more gradually.

Model Development

Assumptions:

- All reactions are catalytic with first order kinetics;
- Coke formation and conversion are deactivated according to different mechanisms;
- The reactions are described by a five lump model:



Deactivation functions:

$\Phi_{coke} = \exp(-\alpha \cdot \tau)$

$\Phi_{conv} = \exp(-k_d \cdot CoC)$

Example, reaction rate of HCO:

$r_{HCO} = \Phi_{conv} \cdot (k_1 + k_2 + k_3) \cdot y_{HCO} + \Phi_{coke} \cdot k_7 \cdot y_{HCO}$

Model Results

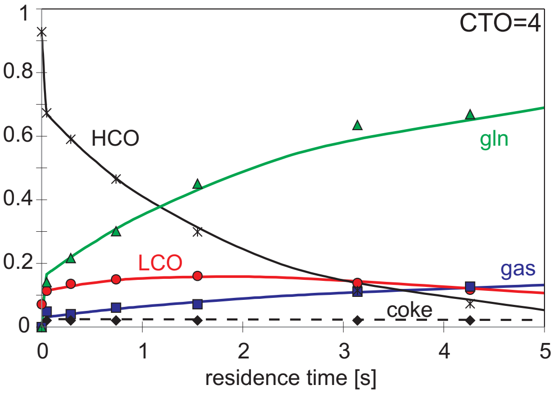
Parameters:

$k_1 = 8.1$
$k_2 = 17$
$k_3 = 3.5$
$k_4 = 0$

$k_5 = 15$
$k_6 = 0.61$
$k_7 = 0.27$
$k_8 = 2.7$

$k_d = 11$
$\alpha = 97$
$SS_{res} = 3\ 10^{-4}$

Description of regenerated data:



- Experimental data are well described for all residence times.

Conclusions

- A five lump model was sufficient to describe the cracking of Hydrowax.
- First order reaction kinetics can be applied.
- The developped model describes the data of the regenerated catalyst well.
- Formation of coke mainly occurs at the initial contact of catalyst and feedstock.

Shell International Oil Products is acknowledged for financial support and for the supply of feed and catalyst.

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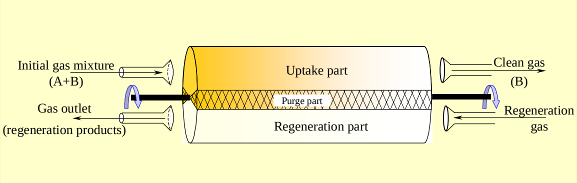
Rotating monolith reactor

Principle of the rotating monolith reactor

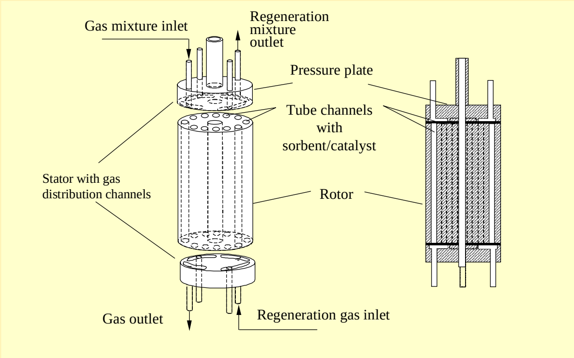
The main principle of the rotating reactor is based on the continuous separation of the gas mixture due to simultaneous operation of uptake and regeneration processes. The reactor is divided in different sections, "uptake" and "regeneration", which are separated by purge sections. Due to the rotation of the reactor the sorbent enters the "uptake" and "regeneration" section subsequently. In the "uptake" section one of the components of the gas mixture is adsorbed by the sorbent. As a result, the outlet gas is free from this component. The release of trapped gas occurs in the "regeneration" section due to purging by regenerative gas mixture.



Advantages continuous operation;
constant composition of cleaned gas and regeneration products ;
heat released at one stage is easy transferred to another;
low pressure drop;
no attrition of sorbent;

Limitation uptake and regeneration processes should be feasible at the same temperature

Design of the rotating monolith reactor



Experimental results

Separation of n-paraffins from paraffins mixture

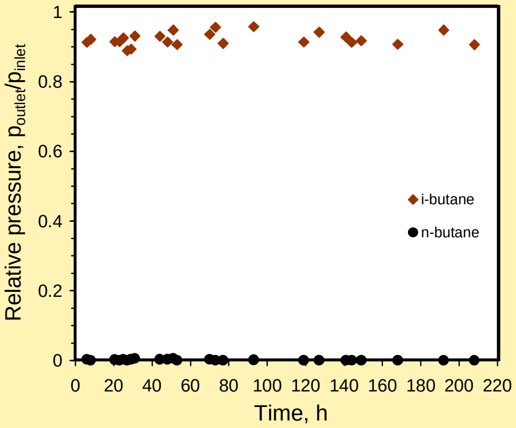
Uptake process: n-butane is adsorbed by zeolite from gas mixture of n- and i-butane

Regeneration process: purge-swing removal of n-butane from zeolite

Experimental conditions:

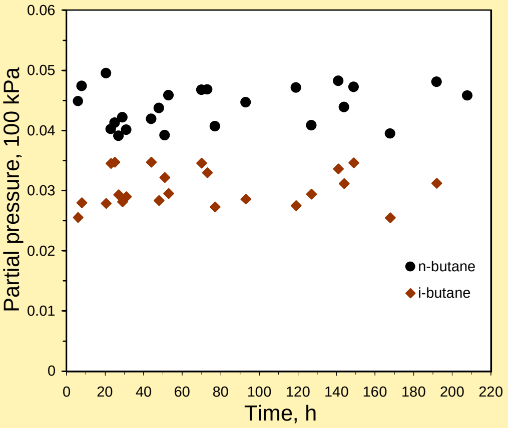
Sorbent	zeolite 5 A
Temperature	373 K
Gas flow	N_2 90 %; n-Butane 5 %; i-Butane 5 % 200 ml min^{-1}
Rate of rotation	0.25 min^{-1}
Regeneration	purge-swing desorption; N_2 flow 200 ml min^{-1}

Separation section

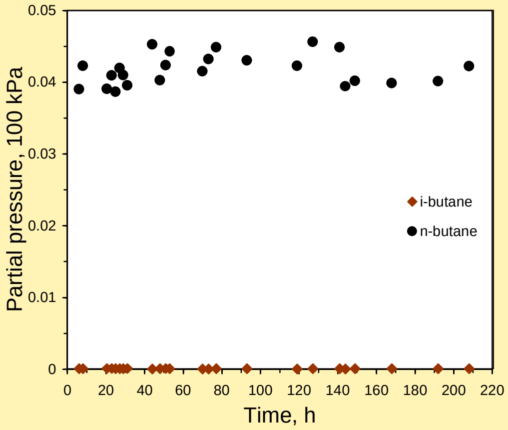


In fixed-bed reactor the same amount of zeolite will be saturated within 6 h.

Purge section



Regeneration section



Processes to be tested in the rotating monolith reactor

High temperature H_2S removal from fuel gas

Uptake reactions: $MnO + H_2S \longrightarrow MnS + H_2O$

$MnAl_2O_4 + H_2S \longrightarrow MnS + Al_2O_3 + H_2O$

Regeneration reaction: $MnS + H_2O \longrightarrow MnO + H_2S$

$MnS + Al_2O_3 + H_2O \longrightarrow MnAl_2O_4 + H_2S$

Regenerative CO_2 capture by calcium-based sorbents

Uptake reaction: $CaO + CO_2 \longrightarrow CaCO_3$

Regeneration reaction: $CaCO_3 \longrightarrow CaO + CO_2$

Direct conversion of CH_4 to synthesis gas:

Step 1: $CeO_2 + x\,CH_4 \longrightarrow CeO_{2-x} + x\,CO + 2x\,H_2$

Step 2 : $CeO_{2-x} + x\,CO_2 \longrightarrow CeO_2 + x\,CO$

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Demonstration of monolithic catalysts in three-phase reaction systems

Introduction

Although monolithic catalyst supports are widely used in exhaust gas cleaning, their application to other types of catalytic processes is limited. One of the few exceptions is in the production of hydrogen peroxide using the anthraquinone process. Monolithic catalysts, however, can be used as an attractive alternative in three-phase operation to both slurry and trickle bed catalytic reactors. Table 1 lists some of the advantages and disadvantages of monolithic catalysts in three-phase reactions.

Table 1. Monolith features

Advantages
- Low pressure drop (< 100 mbar / m)
- Large geometrical surface area
- Maldistribution impossible if inlet distribution is adequate
- Easy Scale-up
- No catalyst separation steps necessary

Disadvantages
- Heat transfer
- Catalyst preparation
- Experience is limited

Aim

To demonstrate the feasibility of the use of monolithic reactor systems in three-phase reaction systems.

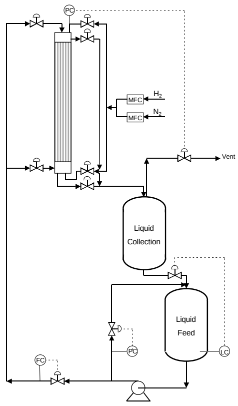
Experiments

Small size (43 mm diameter, 50 mm length) monolithic catalysts are developed and tested using small scale batch autoclave reactors. Promising catalysts are then tested in a larger scale pilot-reactor system (10 or 43 mm reactor diameter, 2 m length). This set-up allows for the investigation of practical problems like:
- monolith stacking
- liquid distribution and redistribution
- heat effects

To allow for a fair comparison with a conventional reactor, it is also possible to use the set-up with a trickle bed.

Pilot-scale reactor for testing monolithic catalysts

A flowscheme of the set-up is shown in the figure below. The system is capable of carrying out the experiments in a co-current upflow or downflow mode or with gas and liquid in countercurrent flow. The liquid is recycled batchwise, making it possible to simulate longer reactor lengths.



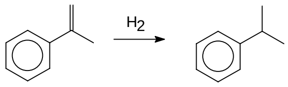
Flow scheme of pilot set-up

Specifications:
- *65 bar*
- *500 K*
- *liquid up to 90 l/h*
- *gas up to 12 Nm³/h*
- *0-100 % H_2 in N_2*

Model Reactions

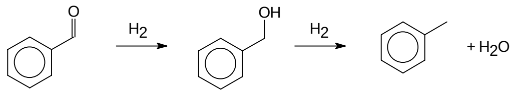
The pilot-scale experimental program currently involves three reaction systems:

- The hydrogenation of α-methyl styrene



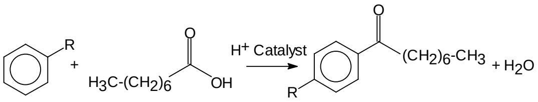
This is a very fast reaction and is used to determine gas-liquid and liquid-solid mass transfer parameters, which will show an increased mass-transfer rates compared to a trickle bed-system.

- The selective hydrogenation of benzaldehyde



It is expected that the better plug-flow behavior of the monolithic reactor system will show higher selectivities to benzyl alcohol than the conventionally used trickle-bed reactor.

- Acylation of aromatics over a solid-acid catalyst.



This reaction will be carried out in an internally finned monolith with a countercurrent nitrogen stream. The water produced in this reaction inhibits the catalyst and restricts the conversion of the equilibrium reaction. The countercurrent water removal will demonstrate a conversion enhancement.

Demonstration of Monolithic Catalysts

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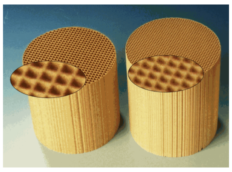
Introduction

A Monolithic reactor is a reactor in which the catalyst is deposited on a metal or ceramic structure with uniform parallel channels.



Monolithic Structure

Monolithic Structures

Advantages of a Monolithic structure:
- No filtering of catalyst necessary
- No attrition of catalyst
- Extremely low pressure drop
- Safety
- Poor radial heat conductivity: no hot spots

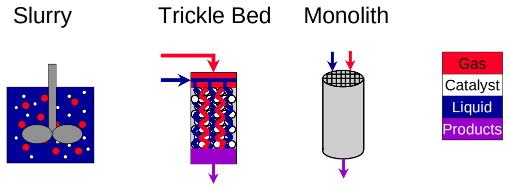
Disadvantages of a Monolithic structure:
- Little practical experience
- No interconnectivity between channels
- Poor radial heat conductivity

Applications

Currently well known applications:
- Automotive application - exhaust converter (400.000/year in The Netherlands)
- Selective catalytic reduction catalysts in power stations

Possible new applications
- Two Phases: Acylations, Esterifications
- Three phases: Hydrogenations, oxidations

Typical Gas-Liquid-Solid Reactors

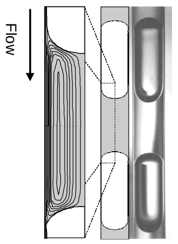


Comparison of reactors

	Slurry	Trickle Bed	Monolith
Separation	--	++	++
Pressure Drop	n.a.	--	++
Internal Mass transfer	++	-	+
Heat Transfer	++	-	-
Catalyst Loading	+	++	+
Catalyst Efficiency	++	--	+

-- very bad, - bad, + good, ++ very good, n.a. not applicable

Taylor Flow

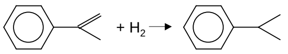


High mass transfer due to
- Thin film
- Internal circulation in liquid slug
- Large G-L transfer area

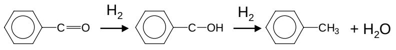
High selectivity due to
- Separated liquid slugs (plug flow)

Model Reactions

Measurement of Mass Transfer:
Hydrogenation of α-methylstyrene (AMS)
- very fast reaction, mass transfer limited in hydrogen
- zero order in AMS, first order in hydrogen
- Catalyst : Ni Catalyst (egg shell)



Measurement of Selectivity:
Selective hydrogenation of benzaldehyde
- Catalyst : Ni Catalyst (egg shell)



Operating Conditions

	Monolith	Trickle Bed
Length (m)	2	0.25
Diameter (mm)	10	47.5
Support	400 cpsi cordierite 9 wt% γ-Al_2O_3	1.7* 5 mm γ-Al_2O_3 extrudates
Catalyst	1 wt% Ni (eggshell)	7wt% Ni (eggshell)
Porosity Reactor	0.9	0.4
Superficial velocity(m/s)	0.2	0.01
	AMS	Benzaldehyde
Temperature	373K	410K
Pressure	10 bar	15 bar

Slurry conditions are same (particle size 50 µm)

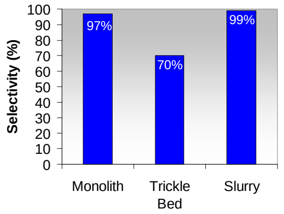
Results

Mass transfer measurements - AMS hydrogenation
Activity per

	Trickle Bed	Monolith
Reactor Volume ($mol/m^3_{reactor}/s$)	3.4	5.3
Catalyst Mass ($mol/g_{catalyst}/s$)	$4.0 \cdot 10^{-6}$	$8.6 \cdot 10^{-6}$
Nickel amount ($mol/g_{nickel}/s$)	$5.3 \cdot 10^{-5}$	$6.3 \cdot 10^{-4}$
Activity Catalyst in Slurry measurement ($mol/g_{catalyst}/s$)	$3.56 \cdot 10^{-5}$	$1.38 \cdot 10^{-5}$

Selectivity Measurements - Benzaldehyde hydrogenation

Selectivity at 50% Conversion



Conclusions

Monolithic Reactors are superior to Trickle bed and Slurry reactors in Gas-Liquid-Solid configuration

This is demonstrated by
- An increased activity in mass transfer limited reactions
- An increased selectivity in sequential reactions (A → B → C)

Acknowledgements

Natasja Romijn and Dennis Sterzenbach are kindly acknowledged for their work.

Combustion of Diesel Soot with Supported Molten Salt Catalysts

Researcher: Drs. B.A.A.L. van Setten (Barry)
Status: OIO
Started: July 1996
Support: STW, EEC Brite-Euram III program

The combustion of particulates from diesel exhaust with a Supported Molten Salt Catalyst is studied. A combined particulate filter/catalyst support is under

Background

One of the best ways of preventing diesel soot emissions is by placing a particulate trap in a diesel engine's exhaust pipe. The accumulated soot can then be oxidized with the excess air available in the diesel exhaust gas. Unfortunately exhaust pipe temperatures are generally too low to ignite the soot that accumulates there. The filter must therefore be regenerated to avoid plugging. This can be done periodically by heating or else continuously with the aid of an oxidation catalyst. The first option is not reliable because it can lead to disastrous thermal-runaways and it has a fuel penalty. The second option might offer a possible good solution.

Problem

While many solid materials are good oxidation catalysts for gas and liquid substrates, they show no activity with soot. The low activity of solid oxidation catalysts is a result of poor contact with the solid soot.

The Solution

The contact problem may be solved by using a liquid-phase catalyst. Different low-volatility eutectic mixtures with low melting points are currently being developed. They are composed of components, which are intrinsically good oxidation catalysts.

Experimental Work

The performance of molten salt catalysts is tested with lab-scale micro-flow equipment and thermal-analysis equipment. Printex U synthetic soot is used as a substitute for diesel soot. The molten salt catalysts are tested with and without support. The catalysts are tested for their tolerance for high-temperatures, high-temperature steam, SO_2, NO_x and mechanical compression. At present different ways of depositing the molten salt on the support are being developed. The big challenge at the moment is the deposition of the molten salt on large open-pore ceramic foam filters.

Results

For supported molten salts the soot oxidation rates where determined with flow analysis. The determined rates are very promising, since they are in the same order of magnitude as found for the most catalytic fuel additives. The catalyst is stable in high temperatures (>1000 K) and in high-temperature steam. The catalyst deteriorates alumina and SiC supports, which affects the mechanical stability of the soot trap.

Future Work

Future research will focus on: the mechanical stability of catalytic foams, the mechanism of soot combustion by means of a liquid-phase catalyst and the performance of catalytic foams with diesel exhaust gas.