

The application of monoliths for gas phase catalytic reactions

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Abstract

A general introductory review of the fundamental principles of monoliths as supports for catalytic gas phase reactions is presented. Monoliths are used because of low pressure drop and high mechanical strength required for the harsh conditions encountered in environmental applications. The chemical and physical properties of monoliths and the basics for mass transfer calculations and pressure drop are presented. Existing and emerging applications are briefly discussed. Reference citations are given for those requiring more depth. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

1.1. What is a monolith and how is it used in catalytic reactors?

Monolithic supports are uni-body structures composed of interconnected repeating cells or channels. They are most commonly composed of ceramic or metal materials but some can also be made of plastic. The most important physical characteristics when used as a catalyst support is the size of the channel through which the gaseous reactants and products traverse. The catalyst is composed of a high surface area inorganic oxide carrier, i.e., γ - Al_2O_3 , upon which catalytic metals or metal oxides are dispersed. Every catalyst company has their own proprietary technology for catalyzing the walls of the monolith but a typical method is to impregnate the Al_2O_3 (or other suitable carrier) with salts of the catalytic components. The catalyzed carrier is then milled to a particle size less than about 10 microns in an aqueous media sometimes with a small amount of acid. The slurry is usually about 30–40 wt.% solids but is adjusted to obtain the proper loading. The ceramic monolith is then dipped into the slurry and the wet gain recorded. After drying at 110°C a dry weight gain is obtained. If acceptable the catalyzed monolith is calcined to about 400–500°C in air in order to decompose the salts and to insure good bonding between

the carrier and the monolith. For metal monoliths it may be necessary to use a pre-coat of an inorganic oxide to insure good bonding between the carrier and the metal monolith. When deposited on the walls it is referred to as the catalyst washcoat. The exact procedures vary with the type of monolith and the specific application.

Reactants enter each of the channels, interact with the catalyst on the walls and the resulting products continue down the channel and exit. A cartoon of the monolith channel coated with a catalyst is shown in Fig. 1.

1.2. Advantages of a monolith compared to a packed bed

The number of channels, their diameters and wall thickness determine the cell density, expressed as cells per square inch (cpsi), which in turn allows the calculation of the geometric surface area; the sum of the areas of all the channel walls upon which the catalyst is deposited. This leads to one of the most important advantages of the monolith in that it has a large open frontal area resulting in very little resistance to flow and hence low pressure drop. The lower the pressure drop the lower the resistance to flow or back pressure on the system and hence lower the energy loss.

Metal monoliths can be made with even thinner walls, with open frontal areas approaching 90% resulting in larger channel diameters and offering even lower pressure drop than ceramics at comparable or greater geometric areas. Factors such as cost, weight, maximum temperature capability, heat management, etc. dictate which material is used in a specific application. Fig. 2 shows some typical ceramic and metal monoliths.

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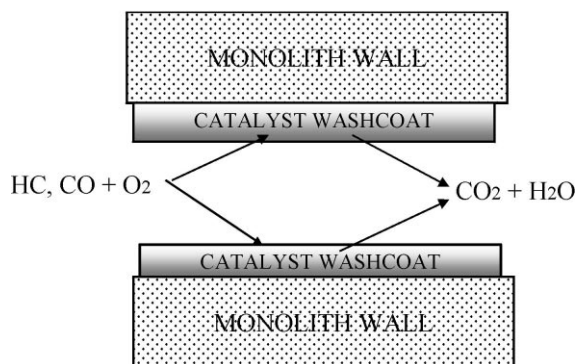


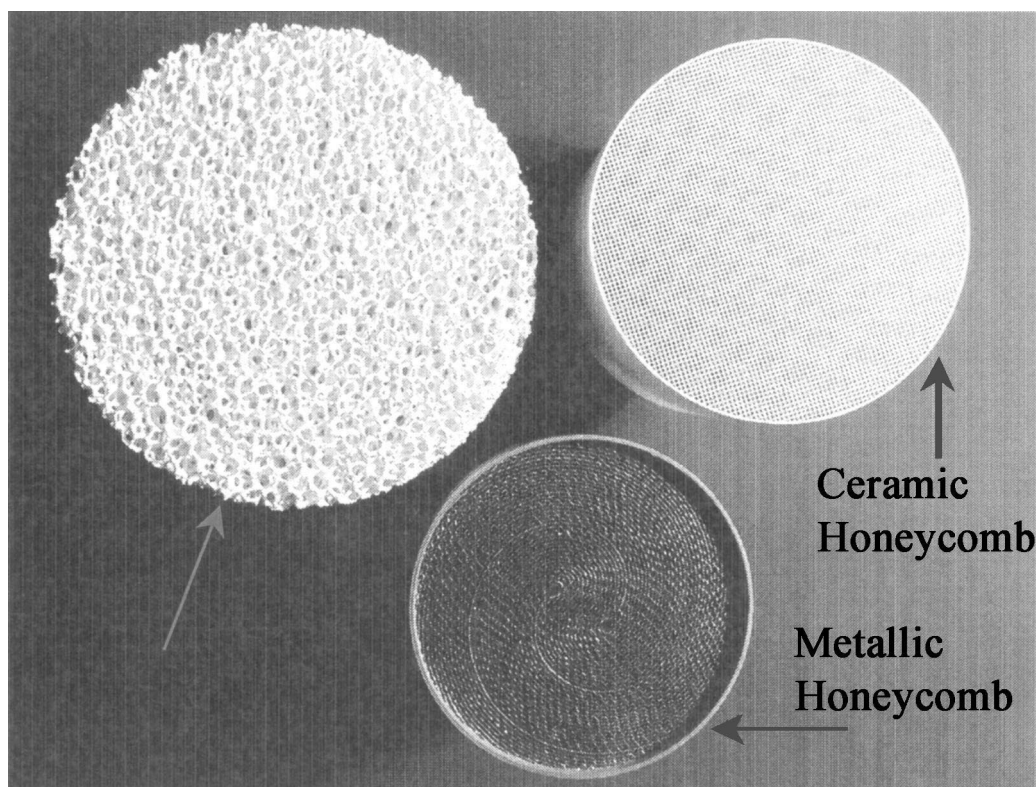
Fig. 1. Reaction in an idealized monolith channel.

The first large-scale use of the ceramic monolith came in mid-1970s when the catalytic converter was installed on new vehicles in US (1). The material of choice was an extruded multi-cell ceramic called cordierite ($2\text{MgO} \cdot 2\text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2$) with low thermal expansion with the resulting property of high resistance to fracture due to thermal shock. To implement this structure there was a need to develop an entirely new technology for depositing the catalytic component but the advantages greatly outweighed the cost associated with developing the new way of manufacturing

catalysts. The presence of the catalyst in the exhaust of the automobile in the form of an open structured monolith (or honeycomb) offers less resistance to flow decreasing power loss compared to a typical bed of particulate bead catalysts. The open structure allows their use in high dust environments, such as coal-fired power plants and diesel exhausts, without concern for plugging. For extreme operating conditions where there is a heavy accumulation of dust the monolith allows ease of cleaning by air lancing or chemical washing.

They offered high geometric surface areas with a lighter and more compact reactor than beads. Lighter weight allows more rapid warm up of the catalyst favoring conversion of pollutants in a shorter period of time once the engine is started. High geometric surface area favors high conversion of pollutants when the rate is controlled by bulk mass transfer a condition which exists for most operating conditions of the warmed up automobile. Because of their uni-body structure they are more resistant to mechanical vibrations and attrition experienced in normal driving than a packed bed of particulate beads. Their structure allows greater freedom of orientation in the exhaust.

By roughly around 1980 and still today essentially all automobile manufacturers design their catalytic converters using ceramic monoliths. There are, however, niche vehicular



Many options for monolith catalyst

Fig. 2. Ceramic and metal monoliths.

markets where metal monoliths are preferred over ceramics such as in heavy-duty trucks and in some high performance vehicles where monoliths with open frontal areas of 90% reduce the pressure drop close to zero. Today, the monolith is the support of choice for almost all environmental applications where high flow rates and low pressure are required with all the other benefits as indicated above. Monoliths are now taken on different forms tailored for specific applications. Metal heat exchangers or radiators are coated with a catalyst and function to convert dilute pollutant containing gas streams at high flow rates with minimal pressure drop. Ceramic and metal monoliths can be constructed with protrusions in the channel to enhance turbulence and hence increase mass transfer to the walls of the channel upon which the catalyst is deposited.

Certainly the successful application of the monolith as a support for the catalytic converter in the automobile gave great confidence to other industries to design their pollution abatement systems with them. Section 5 gives a brief summary of these with appropriate references for additional information. Section 6 highlights hydrocarbon fuel processing for the fuel cell as the next major application of monolithic structures.

1.3. Disadvantages of a monolith as a catalyst support

Although, the advantages of the monolith are many there are some disadvantages that prevent extensive use outside the environmental applications. The parallel channel monolith is essentially an adiabatic reactor limiting the control of temperature. For many exothermic or endothermic chemical and petroleum reactions selectivity is governed by temperature and therefore, these types of monoliths are not well suited. One can employ a metal heat exchanger or metallic foam to control temperature but the amount of catalyst on the walls in a given volume of monolith is much less than a comparable volume of small diameter beads or extrudates. Therefore, for chemical controlled reactions the monolith may not contain sufficient catalyst to yield the desired conversion efficiencies.

2. Chemical and physical properties of monoliths

Monolith materials as supports for catalysts in the automotive exhaust catalytic converter were required to meet

very severe operating conditions of temperatures over 1000°C with resistance to thermal shock. Ceramic materials such as cordierite, 2MgO–Al₂O₃–5SiO₂ (14% MgO, 35% Al₂O₃ and 51% SiO₂) have high melting temperatures 1465°C, resistance to oxidation, and can be made to have excellent thermal shock resistance (low expansion coefficients). This requirement stems from the rapidly changing temperatures experienced in the automobile exhaust. Today, ceramic monoliths can be made in a variety of sizes and materials both ceramic and metal; but one of the most important properties is the number of channels or cells per square inch (cpsi). This property coupled with wall thickness, dictates the geometric surface, a key factor for mass transfer controlled reactions and pressure drop (see Section 4). Table 1 gives the properties of commercially available ceramic monoliths.

It was the success of the cordierite monolith in the automobile exhaust application that gave catalyst companies confidence in considering them for use in other applications [1,2]. None of the other applications are as demanding as the automotive applications, so new designs and materials can be used. Some of the most important applications are listed in Section 5.

Metallic monoliths became available in the early 1990s and are used in niche markets such as close-coupled catalysts, ozone decomposition, motor cycles, restaurants, power plants, etc. The primary advantage is lower pressure drop and weight due to thin walls (0.0015 in. or 3.8 mm) with comparable cell densities to ceramics. Monoliths with open frontal areas approaching 90% provide high geometric surface areas while offering low resistance to flow i.e., back pressure. Because they are metals, the expansion coefficient is much greater than ceramics and therefore, requires special bonding techniques to produce an adherent washcoat. For applications where high temperature and corrosion are experienced such as the close coupled automobile catalyst, metals such as Fecralloy containing 73% Fe, 20% Cr, 5% Al plus small amounts of Ni and Si, with a melting temperature up to 1500°C are sometimes used. For ozone abatement from high flying commercial aircraft, lower melting temperature aluminum monoliths satisfy the temperature (about 200°C), pressure drop and weight requirements. Stainless steel monoliths with temperature capabilities up to about 800°C are used as pollution abatement catalyst supports in power plants.

Table 1
Ceramic monolith properties

Ceramic monolith sizes, cpsi /wall thickness (0.001 in.)	200/12 square channels	300/12 square channels	400/6.5 square channels	400/4.5 square channels	600/3.5 square channels	900/2.5 square channels	1200/2.0 square channels
Wall thickness (in.)	0.012	0.012	0.0065	0.0045	0.0035	0.0025	0.002
Hydraulic channel diameter (in.)	0.059	0.046	0.044	0.0455	0.0373	0.0308	0.0269
Geometric surface area (in. ² /in. ³)	47	54.7	69.6	72.8	89.7	111	129
Open frontal area (%)	68.9	62.9	75.7	82.8	83	85.6	86.6

3. Packaging

The monolith must be packaged in the exhaust of an automobile, power plant, restaurant, etc. The typical converter package consists of a resilient mat to hold the substrate, end seals to prevent gas leakage, a stainless steel can to house the system and sometimes a heat shield for highly exothermic applications to protect adjacent components [3].

A robust converter package provides positive holding pressure on the ceramic substrate, promotes symmetric entry of inlet gases and provides adequate frictional forces at the substrate/mat interface to resist vibration and back pressure loads [4] which otherwise would result in slippage of the substrate within the can. Metal monoliths can be designed with a metal shell or shroud welded around the monolith structure and thus serves as the can. This design allows the monolith and its shroud to be directly welded into the metal exhaust system.

The key mechanical properties of the substrate include strength, E-modulus and fatigue. The tensile and compressive strength [5,6] is important for withstanding packaging loads, in use vibrations; road shocks for mobile applications and temperature gradients. The E-modulus [7] represents the stiffness or rigidity of the monolith structure and controls the magnitude of the thermal stresses due to temperature gradients imposed by non-uniform gas velocity and exotherms. Low E-modulus, which reduces stress and increases substrate life, is more desirable.

4. Kinetics and pressure drop in monolith reactors

4.1. Kinetics

Kinetic analysis of the reaction rate on catalyzed monoliths follows the same chemical engineering principles as packed bed reactors. Because the reaction rate is many times very fast, the reaction itself is controlled by the transfer of the reactant species to the surface. Mass transfer controlled reactions can be obtained through the use of more active catalysts, higher catalyst loading or higher operating temperatures (Fig. 3). The basic test is to obtain the conversion versus operating temperature and determine the energy of activation, E_0 . If E_0 approaches zero, then the reaction is bulk mass transfer controlled. Considering a material balance across any reactor gives the following equation assuming one dimensional, plug flow, steady state operation,

$$\frac{d(vC)}{dz} = -r$$

where v is the velocity (cm/s); C the molar concentration (g mol/cm^3); z the length (cm); and r the rate of reaction ($\text{g mol/cm}^3 \text{ s}$).

When the conversion or the reactant concentration is low, the reactor is considered isothermal, hence

$$v \frac{dC}{dz} = -r$$

Reaction rate

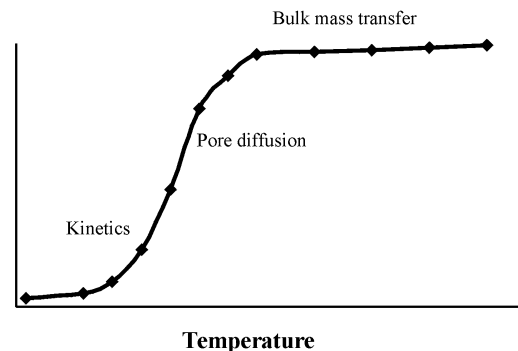


Fig. 3. Conversion versus temperature: rate controlling regimes.

For highly exothermic systems, a similar equation can be used assuming the temperature to be the adiabatic temperature rise. Otherwise, the effect of the temperature profile within the reactor must be considered and a heat balance equation must accompany the material balance. Assuming the reaction is known to obey first order kinetics then the reaction rate is expressed as

$$v \frac{dC}{dz} = -k'C$$

where k' is the apparent rate constant.

Integrating this equation between the reactor inlet (i) and outlet (o) gives

$$\ln \frac{C_o}{C_i} = -\frac{k'z}{v} = -k't$$

where t is the residence time (s) the reactant spends in the catalyst bed.

In reactor design, a useful expression is the space velocity defined as

$$\text{VHSV} = \frac{\text{volume flow rate of feed}}{\text{physical volume of catalyst}}$$

$$\text{VHSV} = \frac{1}{\text{time}}$$

The rate expression then becomes,

$$\ln \frac{C_o}{C_i} = \frac{-k'}{\text{SV}}$$

When the reaction rate is bulk phase mass transfer controlled, the following expression is obtained:

$$v \frac{dC}{dz} = -K_g a C$$

where K_g is the mass transfer coefficient (cm/s); a the geometric surface area per unit volume (cm^2/cm^3); and C the reactant gas phase concentration (g mol/cm^3).

Again integrating, this becomes

$$\ln \frac{C_o}{C_i} = -K_g a t$$

Assuming a constant temperature operation, a simplistic mass transfer model can be derived:

$$\text{fractional conversion} = 1 - \exp - (K_g a t)$$

Using the following dimensionless numbers,

$$N_{Sh} = \frac{K_g d_{ch}}{D}, \text{ Sherwood number}$$

$$N_{Sc} = \frac{D \mu}{\rho}, \text{ Schmidt number}$$

$$N_{Re} = \left(\frac{W}{A \varepsilon} \right) \frac{d_{ch}}{\mu}, \text{ channel Reynolds number}$$

where D is the diffusivity of the pollutant in air (cm^2/s); a the geometric surface area per volume of honeycomb (cm^2/cm^3); L the honeycomb length (cm); W the total mass flow rate to honeycomb catalyst (g/s); and A the frontal area of honeycomb (cm^2).

$$\text{fractional conversion} = 1 - \exp - \frac{N_{Sh} a / \varepsilon L}{N_{Sc} N_{Re}}$$

Correlations for N_{Sh} are available from the literature [8]. Based on the assumptions of mass transfer controlled reaction, an approximate reactor requirement can be calculated [1].

4.2. Pressure drop

Pressure drop is an important design parameter for any reactor since it represents an energy loss. In designing monolithic catalysts, the tradeoff is pressure and total geometric surface. The higher the total geometric surface area, the higher the conversion. The operating penalty, is usually, pressure drop. The basic equation for pressure drop can be derived from the energy balance (again assuming constant temperature) and results in the following expression:

$$\frac{-1}{\rho} \frac{dP}{dL} = \frac{2f v^2}{g_c d_{ch}}$$

where P is the total pressure (atm); f the friction factor, dimensionless; d_{ch} the honeycomb channel diameter (cm); g_c the gravitational constant (980.665 cm/s^2); L the length (cm); v the velocity in channel at operating conditions (cm/s); and ρ the gas density at operating conditions (g/cm^3). Correlations have been developed for catalyzed honeycombs [1] relating the apparent Fanning friction factor, f , to the channel Reynolds number (N_{Re}).

The velocity (v) in the channel is calculated using the mass flow rate (W), density (ρ), void fraction (ε) and area of honeycomb as follows:

$$v = W / (\rho A \varepsilon)$$

where A is the percent open frontal area of the honeycomb.

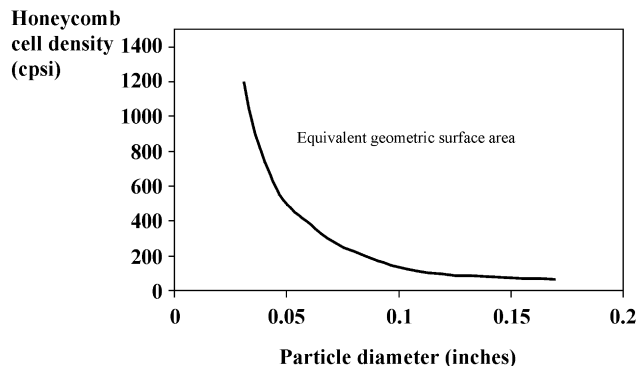


Fig. 4. Comparison of a monolith with particulate as a catalyst support at equivalent surface areas.

The equation simplifies to

$$\Delta P = \frac{2f L \rho v_{ch}^2}{g_c d_{ch}}$$

where d_{ch} is the hydraulic diameter of the honeycomb channel (cm); ρ the gas density at operating conditions (g/cm^3); μ the gas viscosity at operating conditions (g/s cm); and ε the void fraction of honeycomb, dimensionless.

With these fundamental expressions, various monolith options (such as cell density, wall thickness, etc.) can be considered and evaluated relative to other design constraints such as space, compressor capacity, etc.

4.3. General use of monoliths

Monoliths are a standard support material in environmental catalysis such as automotive catalysis, NO_x abatement, etc. [1]. However, in the classical chemical, petrochemical and petroleum industry, they are rarely encountered. For reactions that require high geometric surface area, the monolith is the most efficient support known at present. This is illustrated in Fig. 4 which compares the geometric surface area of commercial monoliths with standard spherical particulate, which is a common support for shape catalysts.

Fig. 4 shows that the particle size decreases exponentially in order to obtain equivalent geometric surface area. Some comparisons would be as follows:

Particle size (in.)	Monolith (cps)
1/8	64–100
1/16	300–400
1/31	900–1200

At the same time recall that the bed porosity is 40% for spherical particles while the monolith have open frontal areas approaching 85%. Therefore, the pressure drop of equivalent packed bed reactors using particulate is very high compared to monolith designs.

5. Applications

5.1. Three way catalysts

The three way gasoline catalyst converter (TWC) simultaneously converts CO, HC and NO_x to CO₂, H₂O and N₂ when operated in the stoichiometric air to fuel ratio in the exhaust of the internal combustion engine [1]. It is the most dominant application of the washcoated cordierite monolith due to the high geometric surface area, low pressure drop, mechanical integrity and thermal shock resistance. It is primarily used in the under-floor position in the exhaust (under the driver), however, it is now being utilized in a close coupled position 3–5 in. (7.5–12.5 cm) below the exhaust ports in order to obtain rapid conversion of hydrocarbons for cold start operation. Monoliths typically have 400–600 cpsi with wall thickness of about 0.045 in. (1.1 mm).

Metal monoliths are finding some use in the close couple position due to their higher thermal conductivity and equivalent or even faster light off than ceramics. In some instances, electrically heated monoliths are also used [9].

5.2. Diesel catalysts

The diesel oxidation catalyst converts liquid particulate (soluble organic fraction), and gaseous CO and HC in the exhausts of heavy-duty trucks, buses and passenger cars. Ceramic monoliths of varying cell densities are used depending on the duty cycle of the engine. Lower cell density monoliths (larger cell diameters) of 200 cpsi are used when the amount of dry particulate (soot) is expected to be high, for example, as in a two-cycle engine [10].

5.3. Ozone abatement in aircraft

High flying commercial airlines use catalyzed metal monoliths in the air intake system to decompose ozone present in make up air. The light weight of a metal monolith coupled with low pressure drop makes this a cost-effective technology for insuring a clean and safe environment in the cabin [1].

5.4. Natural gas engines

Ceramic monoliths are also used in the exhaust systems of natural gas fueled buses to catalytically convert liquid particulate, CO and hydrocarbons (non-methane) [11].

5.5. Ozone destruction on automobile radiators

A recent application of a catalytically washcoated monolithic heat exchanger (metal radiator) for the decomposition of ozone present in ambient air has been commercialized and installed on specific models (Model Year 2000) of Volvo and Nissan. The base metal oxide is coated on the radiator using special binders to insure an adherent catalyzed layer [12,13].

5.6. Small engines

Metal monoliths are catalyzed and used as exhaust surfaces for the destruction of CO and HC generated in small engines such as motorcycles, chain saws, lawn mowers, etc. [9].

5.7. Selective reduction of NO_x

Washcoated ceramic and metal as well as all catalyst monoliths have found wide use in power plants in the selective catalytic reduction (SCR) of NO_x using NH₃. For coal fired power plants where ash and dust are present in high concentrations, large cell density extruded monoliths composed of only catalyst (V₂O₅-TiO₂ or zeolites) are used. For low dust applications such as in gas turbines ceramic and metal monoliths washcoated are used. For high temperature SCR zeolites are coated onto ceramic or metal monoliths while for medium temperature applications the catalyst of choice is usually V₂O₅/TiO₂. Abatement of CO and HC emissions from power plants is also accomplished with both metal and ceramic monoliths [1].

5.8. Destruction of volatile organic compounds from restaurants

Volatile organic compounds are abated from chemical plants using high cell density metal and/or ceramic monoliths as catalyst supports for the oxidation of CO and HC. Quite recently, restaurants have begun using lightweight metal monoliths in the exhaust shroud of cooking operations to abate harmful oils and gaseous compounds associated with cooking greases and oils. Since the exhausts operate on chimney draft low cell density metals are used to minimize pressure drop in the vent [1,9].

5.9. Catalytic combustion

After many years of research and development catalytic combustion has now been commercialized for gas turbines replacing traditional burners. The technology combusts fuels with large excesses of air generating sufficient temperatures to operate the turbine, but with virtually no emission of CO, HC or NO_x. Very special design processes using the monolith ceramic and metal monoliths are used [14].

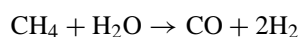
6. Emerging applications

6.1. Hydrogen generation for the fuel cell

The proton exchange membrane (PEM) fuel cell is being intensely investigated for homes and vehicles promising high efficiency and clean power generation. The PEM fuel cell requires H₂ for the anode. Fuel processing of hydrocarbons to make H₂ will likely involve the use of ceramic and/or

metal monoliths and/or heat exchangers catalyzed with the appropriate catalyst [15]. Ceramic and/or metal monoliths offer a wide variety of advantages over packed beds for almost all unit operations mainly related to reduced pressure drop, mechanical integrity, improved heat transfer and reduction in size. There is a disadvantage to the monolith, in that less active catalyst is present in a given volume than in a packed bed. The advantages and disadvantages for the use of monoliths for some of the major unit operations for generating H₂ from methane (i.e., natural gas) are discussed in the following subsections. It should be understood that what applies to natural gas will also apply to other fuels.

6.2. Steam reforming of hydrocarbons

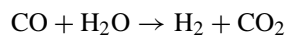


For the production of H₂ for ammonia and methanol plants, this reaction is carried out in a series of metal tubes all of which contain a packed bed of Ni/Al₂O₃ particulate. Because the reaction is very slow and highly endothermic it is necessary to supply large amounts of heat to maintain the reaction rates. This is accomplished by packing a series of small diameter metal tubes containing steam reforming particulate catalysts surrounded by heat. If the catalysts were deposited on a metal heat exchanger then heat transfer could be more efficient leading to greater reaction rates and a smaller reactor. Additionally, there would be a considerable decrease in pressure drop and a great improvement in mechanical integrity; a parameter of great importance for mobile source applications where mechanical perturbations can easily lead to attrition of particulate catalysts.

An alternative process, which utilizes catalyzed ceramic monoliths, is autothermal reforming which combines catalytic partial oxidation with steam reforming in one monolithic reactor. This eliminates the limitations of heat transfer since the heat of the partial oxidation reaction is directly utilized by the steam reforming reaction [16,17].



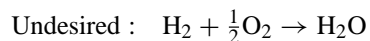
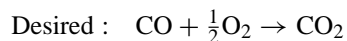
6.3. Water gas shift catalysts



This reaction is one of the slowest reactions in the entire hydrogen generation process so it requires large beds of particulate high temperature (Fe, Cr) and low temperature (Cu, Zn, Al) catalysts. For reduced pressure drop and increased mechanical integrity it would be favorable to prepare these catalysts as monoliths. The major disadvantage is the amount of catalyst that can be deposited on the walls of the monolith is significantly lower than the amount of particulate in the same volume. Therefore, the activity would not be sufficient

and a larger bed would be required. Clearly, water gas shift catalysts with significantly increased activity are needed.

6.4. Preferential oxidation of CO



The fuel cell anode, i.e. Pt/C, is poisoned by traces of CO so it is necessary to reduce the CO below to about 10 ppm. While pressure swing adsorption is commonly used in the large-scale chemical industry for hydrogen production, the high pressures prohibit it from being considered for the fuel processor. The technology of choice is the oxidation of residual CO (about 0.5%) using a highly selective catalyst for which there have been many publications [18–22]. Korotkikh and Farrauto's study [18] is the only one to utilize a ceramic monolith washcoated with a highly active and selective metal oxide promoted Pt catalyst which reduces the CO from 5000 to less than 10 ppm while oxidizing about 5000 ppm of H₂ (i.e. selectivity = 50%). This results in a substantial increase in temperature of the process gas, which can be conveniently controlled and utilized when the catalyst is deposited on a metal heat exchanger or on a series of ceramic monoliths with inter stage cooling. The monolith offers reduced pressure drop and excellent mechanical integrity for both transportation and stationary applications.

6.5. Other chemical applications

Applications for the production of chemicals [23] were also getting attention. Akzo-Nobel has announced the use of monoliths in the production of H₂O₂. The Boreskov Institute of Catalysis [24] is currently using extruded base metal oxide monoliths in combination with reduced amounts of precious metals in the production of nitric acid. Short contact time monoliths are under investigation for synthesis, gas generation and conversion of alkanes to olefins. Huff and Schmidt [25–27] primarily carried out the initial work. These studies demonstrated technical feasibility in oxidative dehydrogenation of paraffins to olefins using Pt deposited on monoliths. Other studies notably by Prof. V. Sadykov of the Boreskov Institute of Catalysis [28] report some advances; however, there are many engineering issues that must be solved before this technology can be moved towards commercialization.

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