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Title: Guidelines for Catalyst Testing in Laboratory Catalytic Reactors – Part A: Choice and Design of a Laboratory Reactor for Catalytic Studies

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ABSTRACT

The development of an industrial catalytic process starts very often at the laboratory, with experiments designed to screen several potential catalysts and the selection of an appropriate laboratory reactor for kinetic studies. It is thus very important to collect accurate and meaningful data on the laboratory scale (free from experimental artifacts and disguises) because the success of the industrial-scale reactor depends on the scale-up factors and parameters developed from the laboratory data. This article focuses on the elements that govern a proper choice of a laboratory catalytic reactor for experimental studies and a brief description of the different catalytic reactors that are available for catalytic studies.

Introduction:

The development of an industrial catalytic process starts very often at the laboratory, with experiments designed to screen several potential catalysts and the selection of an appropriate laboratory reactor for kinetic studies. It is thus very important to collect accurate and meaningful data on the laboratory scale (free from experimental artifacts and disguises) because the success of the industrial-scale reactor depends on the scale-up factors and parameters developed from the laboratory data [1-2]. This article focuses on the elements that govern a proper choice of a laboratory catalytic reactor for experimental studies and a brief description of the different catalytic reactors that are available for catalytic studies.

The paper will begin with some of the definitions of most frequently encountered terms in heterogeneous catalysis. Basic criteria and guidelines are then given for selection of laboratory reactors. This how-to primer focuses on the following areas of catalyst testing and development in laboratory and bench-scale catalytic reactors:

- What are some of the measures of specific reaction rates and reactivity for experimental catalysts?
- What are the basic choices of ideal flow reactors available for kinetic runs in the laboratory?
- How to ensure the absence of intraparticle and extraparticle transport (mass and heat) limitations (by use of diagnostic and mathematical criteria) so that the experiments are carried out under kinetic control?
- What are some of the experimental diagnostic tests for intraparticle and extraparticle gradients?

This article, Part A in this Series, deals with the factors that govern the choice and design of a laboratory catalytic reactor. As stated above, this is important from scale-up and industrial reactor design considerations. Part B in this series deals with the application of Weisz-Prater Criteria for intra-particle and extra-particle transport limitations [3] and is illustrated for two heterogeneously-catalyzed reactions – a gas-solid catalytic system for the synthesis of ethanol and higher alcohols from syngas over a 2% Rh-2.5% Fe/TiO₂ [4-5], and a gas-liquid-solid catalytic system for the synthesis of methanol from CO-rich syngas over a Cu/ZnO/Al₂O₃ catalyst [6-10].

Basic terms and definitions:

A typical supported metal catalyst (Figure 1) consists of metal nanoparticles (of the order 1-20 nm) distributed throughout the pore structure of a frequently inert inorganic support [2]. The metal particles can be tailored to reside at the exterior of the pore structure of each support particle or can be tailored to be at the center of the pore structure. These catalysts are termed as eggshell or cherry catalysts, respectively. The distribution of the active metal inside the catalyst pellet (which itself is a morph of several individual submicron-sized particles, each consisting of a complex architecture of interpenetrating pore networks) can take several forms, as shown in

Figure 2. The most common of these are egg-shell catalysts (Fig. 2-b) where the active metal is dispersed on the exterior of the pellet and the egg-yolk (also termed as cherry, Fig. 2-d) catalyst, where the active metal resides at the center of the pellet. The other two types are uniform (Fig. 2-a) where the active metal is distributed uniformly throughout the pellet, and the egg-white (Fig. 2-c), where the active metal forms the egg-white feature of the pellet, and can be distributed uniformly in the egg-white (region other than the egg-yolk) region.

The support particles are porous, and contain pores in the three basic size ranges. Pores in the size range of 2-5 nm (or, 20-50 Å) are termed as micropores, those in the size range of 5-50 nm (or, 50-500 Å) are termed as mesopores, and pores > 50 nm (> 500 Å) are called as macropores.

Typical supported metals are different than single crystals, which are crystals that can be cut and processed to expose various surface crystal planes. The crystal structures which are pertinent from the standpoint of catalytic chemistry are the body-centered cubic, face-centered cubic, and hexagonal structures (Figure 3).

Apart from supported metals and single crystals, other categories of heterogeneous catalysts are encountered in the literature. These include zeolites, metal oxides, mixed metal oxides, sulfides (typical hydrotreating catalysts), and materials such as nitrides, phosphides, and carbides. There are more novel materials in the literature that include the aluminophosphates (AIPO's), silicoaluminophosphates (SAPO's), MCM series, metal organic frameworks (MOF), and biologically-inspired materials including biomimetics. These novel materials are currently of mostly laboratory interest only.

The surface area, pore volume, and pore size distribution are important textural features of a supported-metal catalyst. The first property, surface area, is measured using physical adsorption of a suitable adsorbate, most typically N₂, at the liquid nitrogen temperature of 77 K, and the adsorption isotherm is analyzed using the BET (Brunauer, Emmett, and Teller, who originally in a landmark contribution, proposed a model for multilayer adsorption and arrived at an equation describing it, that allows for the calculation of monolayer coverage of the adsorbate) equation [2]. The resultant surface area is therefore called the BET-N₂ surface area. There are two common methods for the measurement of pore volume and pore size distribution of solid porous catalysts (or, materials). The first, called the Hg porosimetry method, is used for pores in a larger size range, above 10 nm (or, 100 Å). To determine the distribution of pores with diameter smaller than about 20 nm (or, 200 Å), the nitrogen desorption method is used which uses the Kelvin equation to relate the pore radius to ambient pressure.

While the overall surface area is important, often more crucial in catalytic science is the active metal surface area. Concomitantly, while the bulk loading of the metal is important, more important from the standpoint of catalytic reactions is the metal dispersion. The metal dispersion is a ratio of surface metal atoms to the total metal atoms [2]. The metal particle size is usually calculated from dispersion, and is simply the size (in nm or Å) of the metal cluster. The metal particle size and the particle size distribution is directly measured using high resolution transmission electron microscopy (HRTEM in bright and/or dark fields) or powder X-ray diffraction (PXRD), or is indirectly measured using titration of the surface atoms using probe molecules such as H₂, CO, or O₂, as in chemisorption measurements. Other tools based on

temperature-programmed methods and spectroscopy also are invaluable in physicochemical characterization.

Experimental Measures (or, Metrics) of Catalyst Performance:

Frequently, in a typical experiment, the reactants in the gas phase are contacted with a fixed catalyst charge (say, in a differential fixed-bed reactor system), at specified conditions of temperature, pressure, and space velocity (defined as volumes of gas at normal temperature and pressure conditions per unit volume or unit mass of the catalyst charge per unit time), and the catalyst performance features including conversion, selectivity, yield, specific reaction rates, areal reaction rates, and turnover frequencies are assessed.

- **Conversion, Selectivity, and Yield:** The conversion, selectivity, and yield are defined as follows, for a simple chemical reaction:



Let A be the limiting reactant and let N_{A0} be the number of moles of (limiting) reactant A fed to the reactor inlet (N_{B0} is the number of moles of reactant B). C is the desired product, and N_C is the number of moles of product C. The total moles of product are then $N_C + N_D = N_P$.

$$\text{Conversion} = \frac{N_{A0} - N_A}{N_{A0}} \times 100 \% \quad (2)$$

$$\text{Selectivity of product C} = \frac{N_C}{N_C + N_D} \times 100 \% \quad (3)$$

$$\text{Yield of product C} = \frac{N_C}{N_{A0}} \times 100\% \quad (4)$$

- **Specific Reaction Rate and Turnover Frequency:** The specific reaction rate can be defined on the basis of catalyst particle volume or mass (or, bed volume or mass), and the areal rate is frequently defined on the basis of overall surface area or the metal surface area. The turnover frequency (TOF), a very important performance feature, as it enables to compare data obtained on similar reactions and catalysts in different experimental tests and laboratories, is defined as molecules of reactant or product per surface metal atom per sec, and has the units of sec^{-1} . Thus TOFs (or areal rates, rates based on active metal surface areas) must be reported whenever possible in any proper catalytic study.

A balance between transport rates of reactants (to and from the catalyst surface, or the active site) and chemical reaction rates is the proper way to operate a commercial reactor. This is frequently called as the Weisz window for commercial operation, and is quantified to be $1 \mu\text{mol}/\text{cm}^3 \cdot \text{s}$. This reaction rate translates into a TOF of 1 s^{-1} , for many catalysts. However, for laboratory reactors, the TOF values are frequently 1-2 orders

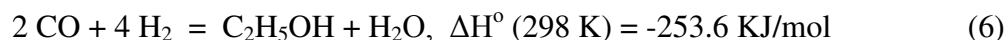
magnitude lower than the value for commercial operation, as diffusion and transport do not play a role in surface reaction kinetics. This mode of operation is referred to as kinetic control, and is used on the lab scale for estimation of intrinsic kinetics of catalytic reactions [11]. A simple definition of TOF can be given as follows:

$$\text{TOF (s}^{-1}\text{)} = \frac{\text{rate of reaction (mol/g cat. s)}}{\text{catalyst loading (g metal/g cat)}} \times \frac{\text{atomic weight of metal (g metal/mol)}}{\text{fraction of total metal atoms that are surface metal atoms}} \quad (5)$$

In the above formula, the catalyst loading (g metal/g catalyst) is the experimental loading, determined using a tool such as inductively coupled plasma – optical emission spectroscopy (ICP-OES) or inductively coupled plasma – mass spectrometry (ICP-MS).

Some basic guidelines for proper collection and analysis of reaction rates in terms of TOFs are as follows [12, 13]:

- The TOF data should be collected under surface-reaction control, i.e., in absence of extraparticle and intraparticle heat and mass transport limitations.
- The TOF and kinetic data should be collected in the absence of deactivation effects due to poisoning, coking, or sintering.
- The TOF data should be collected over wide ranges of temperature and partial pressures (of reactants), to enable easy comparison with data from other laboratories.
- For structure-sensitive reactions, i.e., for those reactions where the TOF is a function of the surface structure and the crystallite size, these effects of metal dispersion and crystallite size should be taken into account.
- Apparent Reaction Orders and Activation Energy: A key consideration in the experimental catalytic science is the determination of reaction orders with respect to reactants and the apparent activation energies of the reaction. Frequently, in gas-solid reactions, the TOF is modeled as a function of the partial pressures (or, concentrations) of the reactants. Consider the synthesis of ethanol (and light alcohols) from syngas (with a H₂:CO of 1) over 2%Rh-2.5%Fe/TiO₂ catalyst, in a fixed-bed microreactor system, at nominal conditions of 543 K and 20 atm. The overall reaction can be written as:



The reaction is thus highly exothermic. One approach to determine the activation energies and reaction orders with respect to H₂ and CO is from the plots of ln (TOF) vs. 1/T and from plots of ln (TOF) vs. ln (p_{CO}) or ln (p_{H2}). Frequently, in such cases, an inert such as He or Ar is used to dilute the feed, and the partial pressure of one of the reactants is varied while the other is held constant. For example, one strategy to determine the

apparent reaction order with respect to H₂ is to hold the partial pressure of CO constant at, say, 2 atm, and change the partial pressure of H₂. Of course, the partial pressure of the inert will change as well, to make up the total pressure to a nominal 20 atm.

- **Dispersion and Metal Particle Size:** The final term, dispersion, is the fraction of the total metal atoms that are on the surface, which are considered to be active sites. As stated above, dispersion is frequently determined using surface titration of metal atoms using probe molecules such as H₂, CO, or O₂, and is called chemisorption. For most metals, a simple empirical relationship exists between metal particle size and dispersion, as:

$$d_p = 1.1/D \quad (7)$$

where:

d_p = particle diameter (assuming spherical particles), and,
D = dispersion, fraction of total metal atoms that are surface metal atoms.

A detailed discussion of the several methods for the collection and analysis of laboratory data is available in many recent works [14-18] and is not re-reported here.

Guidelines for Catalyst Testing:

We will now consider the elements of a proper selection and design of a laboratory catalytic reactor. Frequently, in a typical laboratory experiment, the reactants in the gas phase are contacted with a fixed catalyst charge (say, in a differential fixed-bed reactor system), at specified conditions of temperature, pressure, and space velocity, and the catalyst performance metrics including conversion, selectivity, yield, specific reaction rate, areal rate, and the turnover frequency (TOF) are assessed.

The basic considerations in a catalyst testing program are as follows [12, 19]:

- Select appropriate laboratory reactor
- Establish ideal flow pattern
- Ensure isothermal operation
- Diagnose and minimize transport disguises
- Assess catalyst stability early

Choice and Selection of a Laboratory Reactor: A proper choice for the laboratory catalytic reactor often is governed by the process characteristics. For example, if one is trying to run reactions with relatively fast kinetics or trying to simulate continuous processes, a good choice is the continuous tube-based fixed-bed reactors. If the kinetics of the reaction are relatively slow, a proper choice is either the Berty or the Carberry reactor. For three-phase reactions, where a liquid is a dominant phase (say, as a process solvent), or with an unsupported catalyst, an ideal choice is a high-pressure stirred autoclave batch system. For three-phase reactions with a supported catalyst, a Robinson-Mahoney catalyst basket reactor is an ideal choice.

- Ideal Flow: Via-a-vis ideal flow, it is essential that ideal flows (plug or perfectly-mixed flow) be assured in laboratory reactor design and operation. The two flow patterns, plug flow and perfectly-mixed flow, are called ideal because they enable a simple mathematical description and easy data analysis. In plug-type flow, the fluid moves over the catalyst in the form of plugs, and is in turbulent flow. This type of flow generally ensures the absence of temperature, velocity, and concentration gradients, in the radial direction. Perfectly-mixed flow includes recycle reactors with a high recycle ratio and continuous stirred tank reactors. Usually, deviation from ideality occurs because of presence of gradients (incomplete mixing in a CSTR or axial mixing, wall effects, or catalyst bypassing in plug flow). These gradients should be avoided in laboratory reactors, to ensure ideality.
- Isothermal Conditions: Isothermal conditions are very important from the standpoint of data analysis for intrinsic kinetics. Operation of the laboratory reactor under low conversions or differential conditions, dilution of the feed with an inert, dilution of the catalyst bed with an inert such as SiC, are typically some measures taken to ensure isothermality. Where possible, one can also use a process solvent (typically a high-boiling organic solvent), to absorb the exothermic heat of the reaction.

For bench-scale studies, one also needs to have very accurate measurements of temperature, and well-placed, sensitive probes (thermocouples) are thus necessary. For example, in a differential fixed-bed reactor, it is very important that the thermocouple is in close and intimate contact with the catalyst bed, or errors will result in the experimental data. The temperature is the single most important variable that affects the reactant conversions and product selectivities, especially for gas-solid catalytic reactions with high apparent activation energies. For example, the apparent activation energy for the synthesis of ethanol from a mixture of CO and H₂ is about 100 KJ/mol.

- Catalyst Stability: The nature and extent of catalyst deactivation is also pivotal from the standpoint of the choice of laboratory reactors. If the deactivation is fast, a transport reactor, a reactor in which the catalyst particles are entrained in the fluid, is the clear choice, since the reactor residence times are small. For slow deactivations, a differential fixed-bed reactor (single-pass or recycle) is the proper choice.

A summary of a how-to guide for selection of ideal laboratory reactor for catalytic studies is also given in Table 1.

The discussion on intra-particle and extra-particle transport gradients warrants an in-depth treatment, and is given separately, in a companion article (Part B), following this article. A classification of the laboratory reactors are given in Figure 4. We will now examine a few representative types:

Reactors for Gas-Solid and Liquid-Solid Reactions:

Most assessments of catalyst activity in laboratory reactors are carried out in steady-state fixed-bed microreactor systems, operated in a continuous flow mode. A schematic of the typical fixed-bed microreactor system is given in Figure 5 [19]. In these laboratory reactors, a catalyst charge is placed at the center of a reaction tube, and the charge is held in place between two quartz wool

plugs. The porous catalyst may be diluted with an inert material like SiC to improve heat transfer. A thermocouple is placed in close proximity of the catalyst charge at the exterior surface of the reaction tube, or in a thermowell (or, a sleeve) provided inside the tube. The flow rate of the reactants to the reactor is controlled using mass flow controllers. An inert material may be placed before and after the catalyst bed. The fixed-bed reactor can also be used in a cocurrent downflow mode for gas-liquid or gas-liquid-solid reactions. This mode of operation is frequently called a trickle-bed.

The reactant and product composition is analyzed by using analytical instrumentation such as gas chromatographs. The fixed-bed continuous flow reactors can be operated in a differential (low conversions) or integral (high conversions) mode. The differential mode is more suited for kinetic studies. The integral mode is more suited for modeling of pilot and plant fixed-bed reactors, where heat and mass transfer effects are present. To ensure the ideality of plug flow in these types of reactors (integral design), the following criteria is generally used [1, 3]:

- Reactor diameter (D) should be at least 10-times the particle diameter (d_p)
- The bed length (L) for gas-solid systems should be at least 50-times the particle diameter (d_p) for integral operation.

A classification of the tubular (fixed-bed) reactors based on size is as follows [11]:

- Laboratory: 0.5 cm diameter tubular microreactor (0.1-1 g catalyst)
- Bench: 2.5 cm diameter by 30-50 cm long tubular reactor (50-200 g catalyst)
- Pilot: 7.5 cm diameter by 6-10 m long tubular reactor (20-100 kg catalyst)
- Plant: 1-6 m diameter by 20-50 m long tubular reactor (20-100 tons)

A variant of the fixed-bed reactor is called the fluidized-bed reactor, where the catalyst bed is “fluidized”, by the use of reactant gas flowing upward through a porous metal plate. At moderate-to-high gas velocities, the bed volume increases considerably, and the gas bubbles are no longer visible. Particle carryover to the reactor exit is avoided by operation at gas velocities lower than the entrainment velocities. This type of reactor circumvents the problems of pressure drop in the fixed-bed configuration, and is also more suited for exothermic reactions, due to the pseudo-homogeneous nature of the bubbling fluidized bed. It should be stressed, however, that the fluidized-bed reactors are more suited for industrial applications rather than studies at the laboratory scale.

The fixed-bed reactors can also be operated with internal or external recirculation of the fluid, to approach “gradientless” reactors, for gas-solid or liquid-solid reactions. The different approaches possible for internal or external recirculation are given in Figure 6. In the Carberry reactor, the catalyst is placed in baskets and is rotated at high speeds. In the Berty reactor, the reactor fluid is forced past the catalyst at high speeds, i.e., it is internally recirculated at high speeds. In a third variant, the fluid (gas or liquid) is externally circulated past the catalyst, using a suitable pump. As a major problem with these reactors is the gas-solid contact, two different designs of the Carberry reactors have been proposed, as shown in Figure 7.

Reactors for Gas-Liquid-Solid Reactions:

A packed bed reactor can be used in a trickle-flow mode, with concurrent downflow of gas and liquid past a fixed catalyst bed. The most common reactors for studying gas-liquid-solid reactions are the stirred autoclaves. In stirred tank reactors or stirred autoclaves, finely powdered catalyst (usually 1-200 μ in size) is stirred in the liquid phase. The gas phase is introduced through a gas-inducing impeller, at the tip of the impeller. The reactions take place in the liquid phase. Depending on the operation, the liquid phase can be a reactant, or just an inert (i.e., heat transfer medium).

The stirred autoclaves can be operated under unsteady state conditions, i.e., under batch or semi-batch conditions. These types of reactors are not suited for kinetic studies, where continuous flow reactors are more preferred. The batch and semibatch operation is more common in fine chemical and pharmaceutical industries.

A popular gas-liquid-solid gradientless reactor is the Robinson-Mahoney stationary basket reactor, in which the gas-liquid dispersion is forced through the stationary basket (Figure 8). In this type of reactor, the fixed annular catalyst basket has baffles inside and outside to control vortices. The rotating shaft is equipped with two impellers that draw fluid into the center of the annulus at the top and bottom and outward through the catalyst bed. The gradient-free design and long duration circulation capability for multiple phases has made the Robinson-Mahoney the most widely used design for supported, heterogeneous catalyst research with liquids. The gas-solid and gas-liquid-solid reactor designs can be summarized as follows:

- Fixed-bed Tube Reactor (Figure 9)
- High-pressure Stirred Batch Catalytic Reactor (Figure 10)
- Berty Stationary Basket Catalytic Reactor (Figure 11)
- Robinson-Mahoney Spinning Basket Catalytic Reactor (Figure 12)
- Harshaw Stationary and Falling Basket Catalytic Reactors (Figure 13)
- Carberry Spinning Basket Catalytic Reactor (Figure 14)

Some advanced reactor and testing types including pulse reactors, temporal analysis of products (TAP) reactors, and steady state isotopic transient kinetic analysis (SSITKA) are discussed elsewhere [19].

Most gas-solid catalytic reactions are accompanied by heat effects, thus, in addition to extraparticle and intraparticle diffusion, extraparticle and intraparticle heat transfer occurs. The presence of heat (temperature) and mass (concentration) gradients (Figure 10) confound the intrinsic kinetics of the reaction. The experimentalist must thus assure that the experiments are carried out in the absence of any transport gradients, to assure meaningful and reliable kinetic data from laboratory catalytic reactor system, that is amenable to scale-up. Part B in this series of articles focuses on the transport gradients and heat and mass effects in catalytic reactions.

For further information, the reader is referred to additional works in the area [20-26]. For novel reactor designs and future trends in catalytic reaction engineering (both not a part of this part), the reader is referred again to some recent works [27-30].

Conclusions:

The proper selection of a laboratory catalytic reactor and process operational conditions are important from the standpoint of collection of accurate and meaningful kinetic data. This is important from scale-up considerations and design of industrial-scale reactors.

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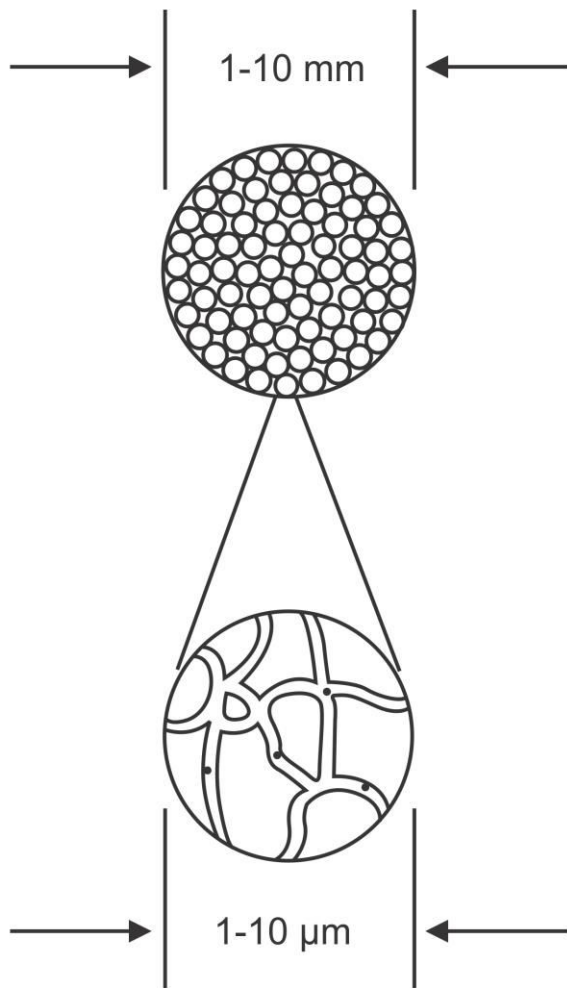


Figure 1. Schematic of a typical catalyst pellet comprised of porous particles (adapted from [2]).

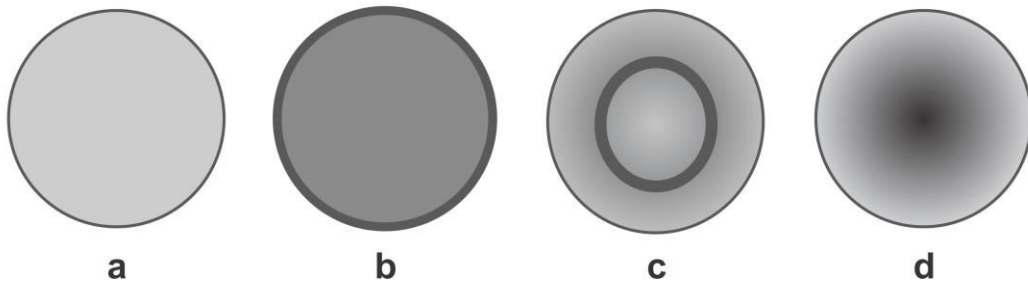


Figure 2. Schematic of possible distributions of active component in a catalyst pellet; (a) uniform, (b) egg-shell, (c) egg-white, and (d) egg-yolk.

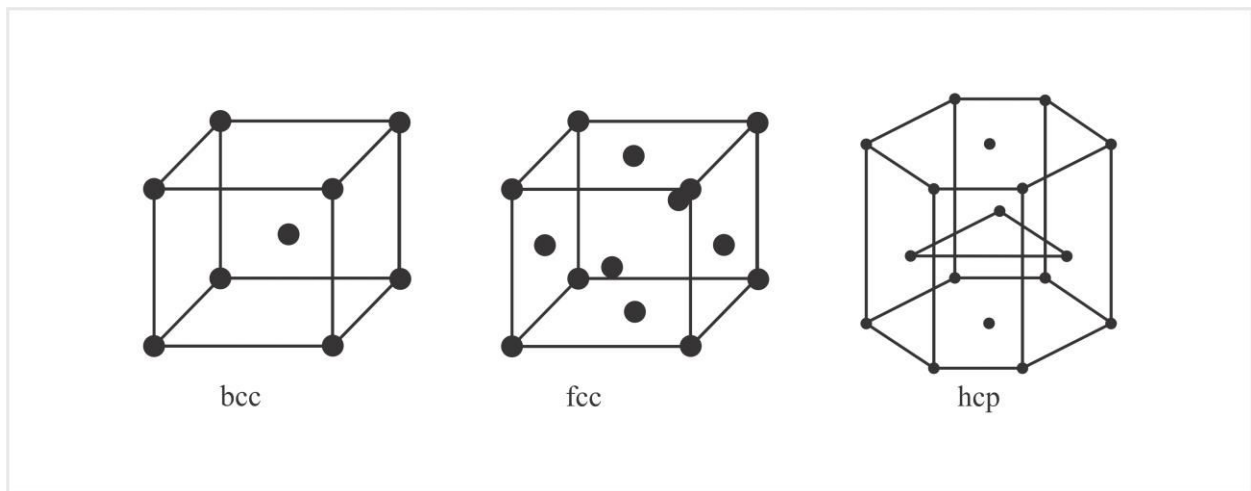


Figure 3. Schematic of BCC, FCC, and HCP crystal structures.

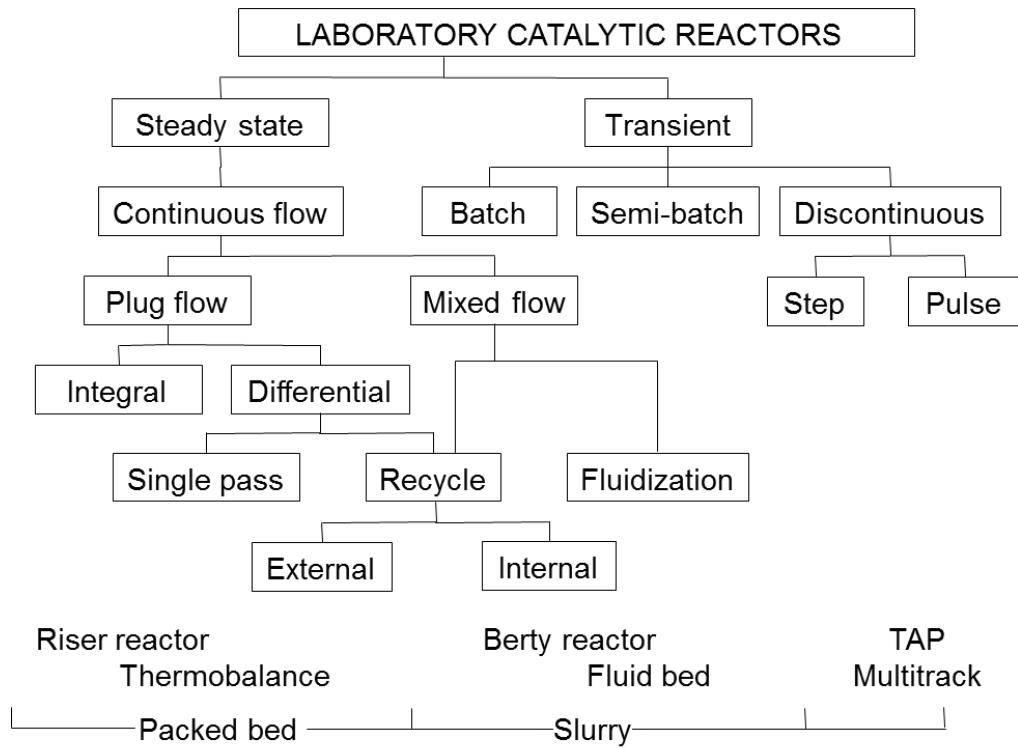


Figure 4. Classification of laboratory reactors.

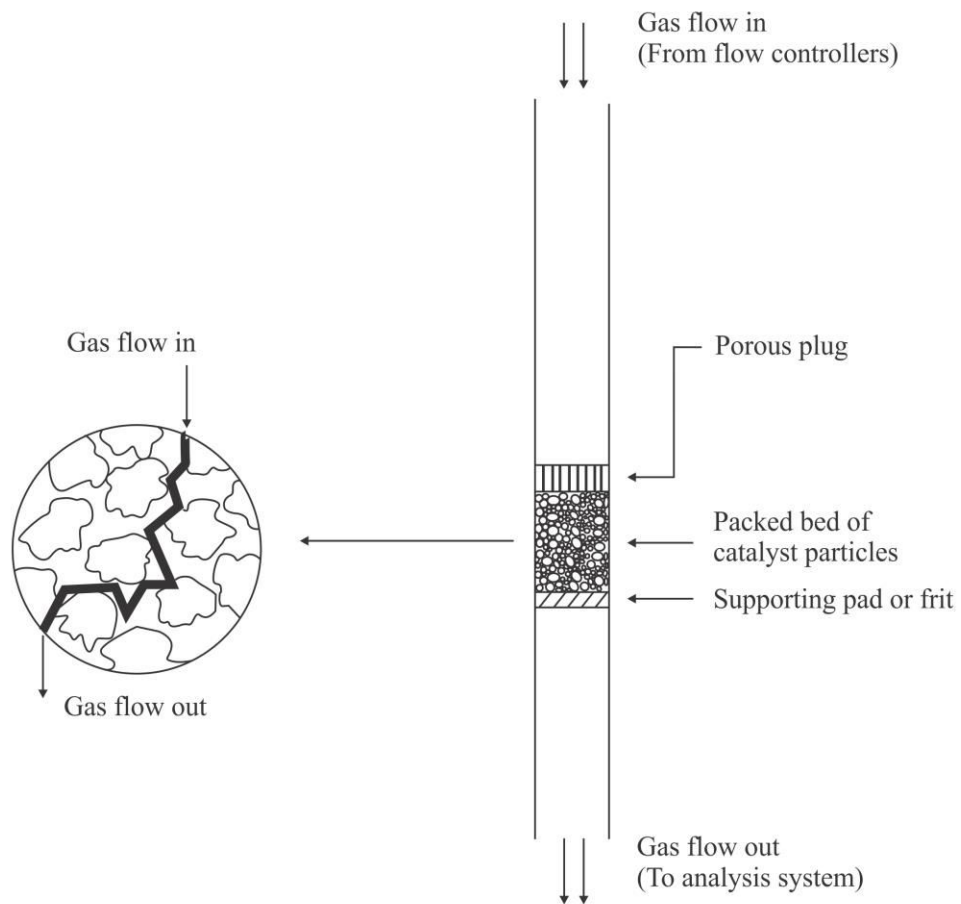


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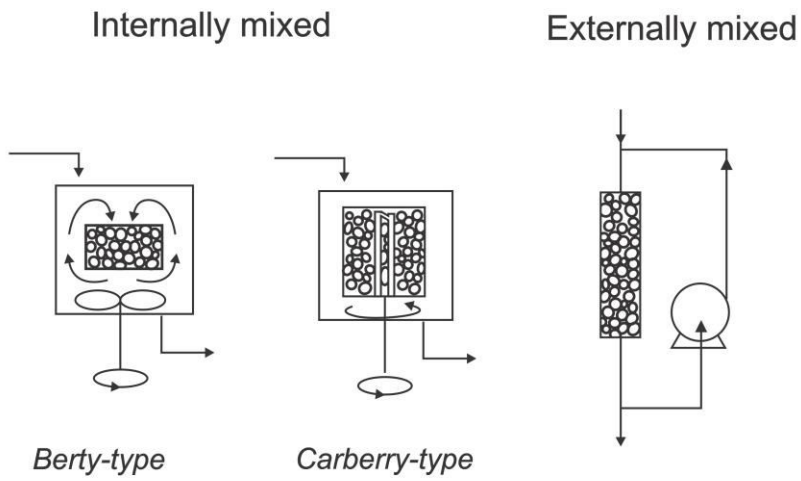


Figure 6. Schematics of CSTRs for heterogeneous catalysis (adapted from [3]).

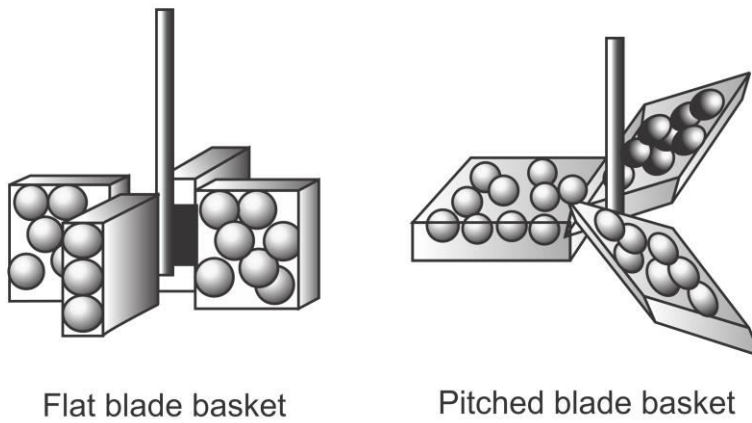


Figure 7. Basket designs for Carberry-type reactors: (a) flat blade; (b) pitched blade (adapted from [3]).

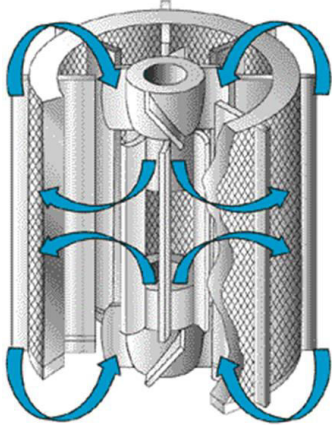


Figure 8. Schematic of a Robinson-Mahoney basket reactor (adapted from [26]).



Figure 9. Fixed-bed Tubular Reactor (adapted from [26])

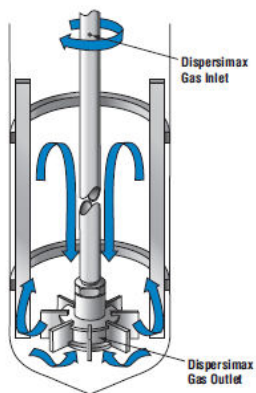


Figure 10. High-Pressure Stirred Batch Catalytic Reactor (adapted from [26])

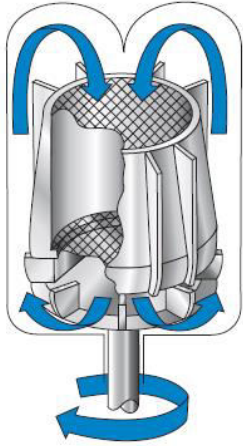


Figure 11. Berty Stationary Basket Catalytic Reactor (adapted from [26])

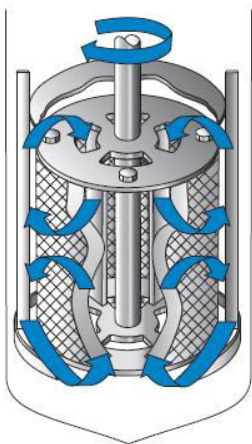


Figure 12. Robinson-Mahoney Spinning Basket Catalytic Reactor (adapted from [26])

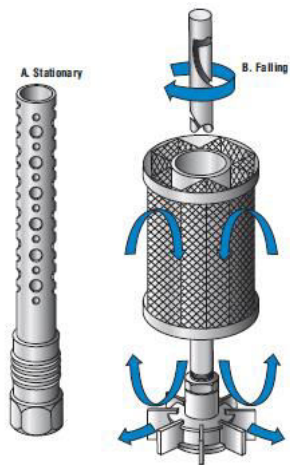


Figure 13. Harshaw Stationary and Falling Basket Catalytic Reactors (adapted from [26])

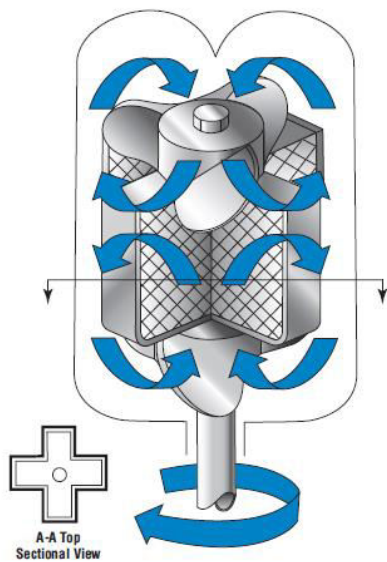


Figure 14. Carberry Spinning Basket Catalytic Reactor (adapted from [26])

Table 1. Some criteria for selection of laboratory reactors

No.	Criterion	Measures or methods to meet criterion
1	Satisfy purpose of measurement (i.e., application)	(1) Intrinsic activity/selectivity, (2) Kinetics of reaction and deactivation, (3) Mechanism, (4) Process simulation
2	Avoid catalyst deactivation (unless it is the object) and its complications; decide if catalyst decay is fast or slow	Fast decay causes activity and selectivity disguises and requires use of a straight-through (or, recirculating) transport reactor. Slow decay is best studied using CSTR or differential reactor.
3	Minimize extraparticle and intraparticle heat and mass transport gradients	Use fine catalyst particle, dilute the catalyst bed with a material like SiC, use differential conditions. Use a CSTR or a differential reactor, wherever possible. Minimize the film thickness (boundary layer thickness) by using moderate to high flow rates.
4	Maintain ideal flow patterns	Minimize mixing in a plug flow reactor and maximize mixing in a CSTR. Minimize gradients in a CSTR. Avoid gas and liquid hold-ups in case of a multiphase reactors.
5	Maximize accuracy of temperature and concentration measurements	Sensitive analytical methods and well-placed, sensitive probes; sufficiently high product concentrations.