

# Fundamental principles of laboratory fixed bed reactor design

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A few important, foundational principles enable more effective discovery and development of heterogeneous catalysts. Numerous experts have documented and illustrated these principles over the past several decades, but many current practitioners fail to heed their sage advice. We revisit the concepts, which we categorize as the bed-scale phenomena of isothermality, contacting pattern ideality, isobaricity, and wetting efficiency, and the particle-scale phenomena of internal and external temperature and concentration gradients. In addition, we introduce a new public domain, web-based tool for quickly estimating the magnitude of the effects of these phenomena. Our objective is to motivate and equip our colleagues to teach these principles and to apply sound reaction engineering practices in their laboratory work.

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## Introduction

‘Those who cannot remember the past are condemned to repeat it’ [1]. This article intentionally deviates from the review of current advances to emphasize our current opinion, undergirded by foundational work that has accumulated over the past five decades. We feel this topic is perennially relevant. Just as each generation must teach the next generation the fundamentals of reading, so each generation of reaction engineering and catalysis practitioners must teach the next generation the fundamentals of our discipline.

In particular, this article targets an audience that lives at the interface of two disciplines, chemical reaction engineering and heterogeneous catalysis. Our objective is to provide a service to that community by highlighting the

fundamental principles that must govern the experimental evaluation of heterogeneous catalyst systems at the laboratory scale. In our experience, researchers often ignore these fundamental principles. We hope that, by drawing attention to this admittedly mature knowledge base, we will remind those who have forgotten, equip those who want to collaborate in our effort to educate the next generation, and teach those who never learned these foundational principles. We begin with brief comments addressing the historical context.

## Historical context

The field of heterogeneous catalysis can trace its roots to the first half of the nineteenth century, as ably described by Robertson [2]. On the other hand, chemical reaction engineering (CRE) evolved to become a formal discipline within the field of chemical engineering in the 1940s and 1950s. Early markers of this discipline’s formalization include the inaugural European Symposium on Chemical Reaction Engineering in 1957 (ISCRE History; URL: [http://iscre.org/iscre\\_history.htm](http://iscre.org/iscre_history.htm)) and the publication of Levenspiel’s *Chemical Reaction Engineering* in 1962 [3]. Levenspiel emphasized in the preface of his seminal text that ‘the goal is the successful design of chemical reactors...’ In spite of the goal stated by Levenspiel and shared by many other CRE textbook authors, the topic of laboratory reactors is inconspicuous in those textbooks. Although some CRE textbooks acknowledge the importance of selecting laboratory reactors that will generate accurate data useful for reactor design and provide heuristics to assist in selecting laboratory reactors [4,5], many provide no explicit mention of the importance of or methodology for selecting and designing laboratory reactors.

## Earlier reviews

Many have provided guidance regarding the selection, design, and operation of laboratory reactors. Using a gas-liquid reaction over a powdered catalyst relevant to his work at Mobil, Weekman focused on the decision process for selecting a laboratory reactor, providing heuristics for choosing from a menu of several potential options [6]. When Rase and Perkins [7] introduced their implementation of Hougen’s recirculation reactor concept [8], they emphasized the importance of eliminating temperature and diffusion gradients in order to independently study the effects of each system variable. Smith emphasized the reactor design dilemma caused by non-isothermal heterogeneous catalytic reactors [9]. Although these papers focused on concepts rather than quantitative criteria,

numerous authors tackled individual components of the macroscopic problem, but, in 1971, Mears provided the first comprehensive review with quantitative guidance for laboratory fixed bed reactor applications [10<sup>\*\*</sup>]. Dommeti *et al.* later showed the limitations of the Mears criterion for interphase transport [11<sup>\*</sup>]. More recent publications have expanded the scope to address additional factors important in trickle bed reactors, such as reviews by Sie [12<sup>\*</sup>], Mary *et al.* [13], Mederos *et al.* [14], and Bej [15].

### Scope of this review

The 45-year-old paper by Mears provides the launching point for our primer, which will specifically address laboratory scale fixed bed reactors, including both integral reactors and recycle reactors. Analogous concepts and principles apply to other systems, such as stirred tank slurry reactors [16<sup>\*\*</sup>] and single-phase tubular flow reactors [17], but those systems are outside of the scope of our current review.

Common laboratory fixed bed reactors include integral reactors (tubing packed with catalyst) and recycle reactors, which can use an external recycle scheme based on a packed tubular reactor coupled with a recycle pump or compressor or an internal recycle scheme. Internal recycle reactors typically used for gas–solid systems have varied designs and are informally named after their inventors (Berty [18], Caldwell [19], and Carberry [20]), but all have the objective of testing commercial-scale catalyst particles contained in a porous basket. We differentiate these reactors based on the mixing efficiency and the superficial velocities that can be achieved in the catalyst beds, with higher velocities preferred in order to minimize external transport gradients. Users of these reactors must carefully evaluate these reactors to ensure the performance is sufficient to generate data of the required quality for a particular application [19,21–25]. For gas–liquid–solid systems, the Robinson–Mahoney design [26] is the most popular option for testing fixed bed catalysts under well-mixed conditions.

In an effort to miniaturize laboratory fixed bed reactors, one new vista for laboratory fixed bed reactors involves reactors with channel-to-particle diameter ratios approaching unity. Recent publications [27–29] build on the old idea of a ‘single pellet string reactor’ [30,31] by shrinking the reactor diameter to approach the diameter of the catalyst particles, resulting in so-called packed bed microreactors or microchannel fixed bed reactors. Although our present review does not address these cases explicitly, we refer interested readers to these publications. Although the principles in our review equally apply to these cases, the specific correlations for phenomena such as gas–liquid mass transfer or axial dispersion and the evaluation of the importance of wall effects are special cases that must be addressed accordingly.

Next, we will review the guiding principles for laboratory fixed bed reactors and point the reader to useful references while avoiding the reproduction of quantitative expressions previously published in the literature. We close by illustrating the application of these principles to an example system.

### Guiding principles

In this section, we address bed-scale (interparticle) phenomena first, followed by particle-scale (interphase and intraparticle) phenomena.

#### Bed-scale phenomena

Researchers should design and operate their laboratory scale reactors to achieve isothermality, contacting pattern ideality, and isobaricity on the scale of the catalyst bed. The catalyst in gas–liquid–solid systems should be fully wetted.

#### Isothermality

For integral fixed bed reactors, isothermality is typically the most difficult performance criterion to satisfy. Mears provides a useful criterion, defining isothermality to be achieved when the average reaction rate at the cross-section of the hot spot in an integral reactor is within 5% of the rate at the wall temperature [32]. This criterion applies equally to all possible fluid phase scenarios. The primary challenge to implement this criterion involves choosing appropriate correlations for the wall heat transfer coefficient and the bed effective thermal conductivity that apply for the potentially lower linear velocities and smaller particles used in the laboratory setting. For recycle reactors, isothermality is achieved by attaining a sufficiently high recycle ratio so that the single pass adiabatic temperature rise is negligible [23].

#### Contacting pattern ideality

For integral fixed bed reactors, the ideal contacting pattern is plug flow. Mears provides a conservative criterion for plug flow [33]. In addition to relaxing the Mears plug flow criterion, Gierman observed that, in the limit of low flow rates, the Bodenstein number asymptotically reaches a constant value [34<sup>\*</sup>]. Combining those two factors, Sie offers an alternative plug flow criterion based on Gierman’s assumption and observation [12<sup>\*</sup>]. Unlike the criterion from Mears, this latter criterion does not require an estimate of the axial dispersion coefficient since that value is implicit in the constant Bodenstein number at low flow rates.

Sie emphasizes that the above criteria assume a randomly packed bed with no variability in the velocity profile as a function of the bed radius [12<sup>\*</sup>]. In fact, laboratory scale reactors are very prone to wall effects because of the greater void fraction near the wall. Sie proceeds to show that, for systems in which the only fluid is a gas, the traditional rule of thumb that the tube to particle diameter

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