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Two-stage semi-fluidized bed reactor: A comparative mini-scale experimental investigation

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In the present investigation, a two-stage semi-fluidized bed reactor (2-SFBR) has been evaluated at mini-scale and compared directly with a single-stage SFBR under identical operating conditions. The indigenously fabricated reactor comprises a Perspex tube, fitted with a 40-mesh stainless steel screen strainer as required. The spherical glass beads (~700 µm diameter) as solid phase provide the dry packed bed height of 100 mm in the semi-fluidized liquid phase reactor. The saponification reaction between equal flow rates of sodium hydroxide and ethyl acetate was carried out for both single- and two-stage SFBR configurations. Over the tested superficial flow-velocity range, the 2-SFBR achieved a 9-15% higher conversion of NaOH than the single-stage SFBR. The findings suggest that incorporating alternating fluidized and packed bed zones in multiple-stage SFBRs can significantly improve liquid phase reaction conversion. These insights are expected to facilitate the design and scaling of multi-stage SFBRs in future applications in the chemical and biochemical process industries. The present study emphasizes the future potential of two-stage SFBRs over other reactors for fast spontaneous reactions, especially prevalent in fertilizer and pharmaceutical industries.

Keywords: Conversion, Fluidization, Packed bed, Saponification, SFBR, Two-Stage

Introduction

Semifluidization, characterized by the concurrent existence of both fluidized and packed solids within a specific volume, has its own distinct identity in chemical and biochemical process engineering. By combining the favorable attributes of fluidization,such as enhanced mixing and reduced pressure drop, along with the stability of a packed bed, semifluidization has gained the attention of research fraternities since as early as 1959[1]. In this context, the hydrodynamic investigations into semifluidized beds outnumbered the studies on their application. Most of the hydrodynamic studies have primarily focused on variables such as column diameter, bed expansion ratio, and both minimum and maximum semifluidization velocities in two-phase as well as three-phase systems[2-8]. Investigations have also explored a variety of solid particles — regular or irregular, single or in homogeneous/ heterogeneous mixtures of a semifluidized bed to assess their suitability as the contacting media or as catalysts[9-15]. Beyond fundamental hydrodynamics, semifluidized beds have been investigated for practical applications including drying, ion exchange, adsorption, and in furnace operations[16-20]. A few number of works reported their applications as bioreactors. More recently, semifluidized bed reactors (SFBRs) have been studied extensively in the biotechnology domain, especially xanthan gum production, bioplastic synthesis, biodiesel manufacture, and effluent treatment[21-22].

On the other hand, the multi-stage phenomena are common in practice in continuous process industries to improve overall process efficiency. With the adoption of multi-stage operation, better heat transfer, mass transfer, and overall process performance can be achieved, along with reduced energy consumption. Multi-stage compressors, evaporators, fractionating columns, packed towers, and fluidized systems are some common examples. Analogous to these, multi-stage (in particular, two-stage) semi-fluidized bed systems can require lower power input than single-stage configurations under identical operating conditions[23-24]. Similar benefits may be attained by utilizing SFBRs. Multiple staging in SFBR can enhance the utilization of reactants while curtailing the energy demand associated with particle suspension. Despite extensive research on multi-stage configurations for conventional fluidized beds and other contacting devices, very little research has been

conducted on multi-stage SFBRs. Additionally, most of the studies on single-stage SFBRs were primarily focused on the hydrodynamic behaviour, bed expansion, pressure drop, and reaction characteristics[25-27]. Nevertheless, systematic investigations on staged SFBR configurations, specifically under liquid-phase reacting conditions, were bare minimum. Thus, the present investigation is intended to demonstrate the advantages of a two-stage SFBR over a single-stage SFBR in a liquid-phase reacting system under an identical operating environment, to ascertain the promisingness of stagingfor SFBR conversion performance. The findings of the present study will provide a strong foundation towards the design and development of multi-stage SFBRs for industrial-scale liquid-phase reaction systems.

Experimental Section

A mini-scale experimental set-up of 2-SFBR has been designed and fabricated for the present investigation. This section explicitly describes the experimental setup and the experimental procedure followed in this comparative conversion investigation.

Chemicals and reagents

Chemicals, including sodium hydroxide (NaOH) (CAS No. 1310-73-2), ethyl acetate (CAS No. 141-78-6), hydrochloric acid (CAS No. 7647-01-0), and phenolphthalein indicator (CAS No. 77-09-8), were procured from Merck (Mumbai, India).

Experimental set-up description

A detailed description of the experimental setup and its auxiliaries is presented in this subsection. Fig. 1 (a and b) represents the schematic and pictorial views of the experimental apparatus, respectively. The experimental setup has been primarily divided into three functional zones, as described hereafter (refer to Fig. 2). Moreover, the dimensions and material of construction of all the auxiliary parts, along with the sample particle's physical dimensions, are represented in Table 1.

(i) *Supply zone*: The reactants were stored in two fabricated reservoirs, each of 5 L capacity with submersible pumps at the bottom. The reservoirs were open to atmospheric pressure at the top. From each, a line from the pump outlet leads to a corresponding calibrated rotameter. These lines act as the provision for introducing liquid reactants (feed) to the bottom of the test section (reaction zone) via pipes connected to the rotameters.

(ii) *Test zone*: The test or reacting zone consists of two identical semi-fluidized bed test pipes (columns), each of internal diameter (I.D.) 12.7 mm and length 100 mm, fitted with a fixed 40-mesh stainless steel (SS) screen at one end. A green flexible plastic hose

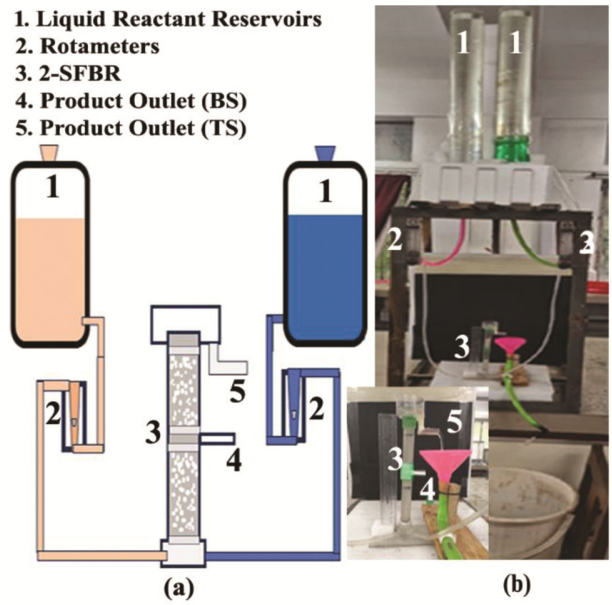

Fig. 1 — Experimental setup: (a) Schematic and (b) Pictorial representation

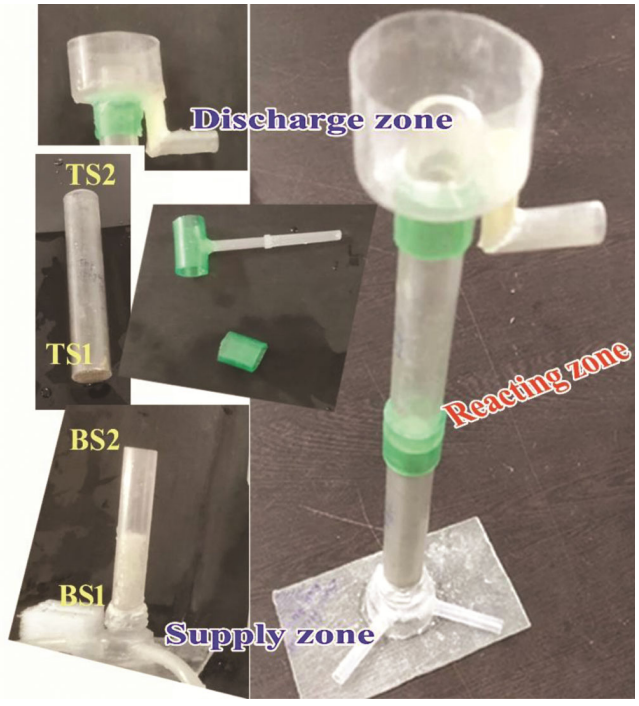

Fig. 2 — Two-Stage SFBR consisting of Supply zone, Reacting zone, and Discharge zone

Table 1 — Components of fabricated experimental setup, along with their materials of construction and dimensions

Components of set-up	Materials of construction	Dimensions	
Reaction zone (TS and BS)	Perspex (transparent)	I.D.: 12.7 mm Height: 100 mm each	
Screen	Stainless steel	40 mesh	
Two reactant reservoirs	Plastic (non-reactive &corrosion-resistant)	5 L capacity each	
Submersible pumps	--	1100LPH, 20 W each	
Two calibrated rotameters	Glass (with steel float)	*Flow rate capacity:18 LPH (max.)	
Packing bed materials	Size (diameter)	Bed expansion ratio	Dry bed height
Spherical glass beads (8 g per stage)	700 μm	2	50 mm per stage

connects the two test pipes- top section (TS) and bottom section (BS), allowing for fluid flow between stages. The solid phase comprises spherical glass beads (700 μm nominal diameter), with a total mass of 16 g. For all experiments, the total mass of solids is divided equally among the top and bottom test columns when operating in two-stage mode (8 g per stage), corresponding to a dry packed bed height of approximately 50 mm per stage. In the single-stage SFBR configuration, the two pipes are joined end-to-end (without the screen barriers) so that they act as one stage (i.e., BS1–BS2–TS2–TS1 arrangement). On the other hand, in the two-stage SFBR configuration, each pipe operates as a separate stage, each retaining its portion of the solid bed, separated by the screen (i.e., BS1–BS2–TS1–TS2 arrangement). Provision is made for withdrawing partially reacted flow from the bottom stage during two-stage operation. In all configurations, a bed expansion ratio of 2 was maintained (i.e., the fluidized (expanded) bed height is twice the initial packed bed height).

(iii) *Discharge zone*: At the top of the test zone, fluid from the reaction section is directed into a discharge pipe (I.D. 12.7 mm, height ~20 mm), which is fitted with a 40-mesh SS screen at its lower end. This discharge pipe is enclosed by a collecting chamber. The design ensures that the outlet stream is withdrawn with minimal effect from the hydrostatic head in the discharge zone.

Experimental procedure

Two reactants, ethyl acetate and aqueous sodium hydroxide solutions of strength 0.1 N, were pumped from their respective reservoirs to the bottom of the test section through two calibrated rotameters (capacity up to 18 LPH max.). Experiments were conducted under steady-flow conditions. For each run, the flow rates of both reactants were set equimolarly. Residence times were varied by adjusting flow rates. After initiation of flow, once steady-state conditions were reached (approximately after 60 s), the sample solutions of 10 mL each were collected for varied residence times from the outlet at the top of the test section. The collected samples were immediately mixed with 0.1 N aqueous hydrochloric acid solution to stop the saponification reaction in the collected samples. Consequently, both the top packed bed height and the partial fluidized bed height were measured during the experimentation through visual observation. Each collected sample was then titrated with aqueous sodium hydroxide solutions of strength 0.1 N, using phenolphthalein as an indicator, to quantify the conversion of sodium hydroxide within SFBR. The same procedure was repeated for both single-stage and two-stage SFBR configurations, maintaining identical solid loading and reacting section heights to allow direct comparison. All the experiments were performed in triplicate to assess the reproducibility of the results.

Result and Discussion

This section presents a comparative analysis of the performance of single-stage and two-stage SFBR under identical operating conditions.

Single-stage SFBR

Fig. 3 depicts the conversion of NaOH as a function of the superficial velocity of the reactant liquids in the saponification reaction. As the superficial liquid velocity increased up to 2.6 cm/s, the conversion increased despite the concomitant reduction in residence time in the fluidization region. This is attributed to improved mixing under fluidized bed conditions. Beyond this threshold superficial liquid velocity (~2.6 cm/s), as semi-fluidization commenced, further increment in liquid velocity led to a decline in fractional conversion. This decline correlates with an

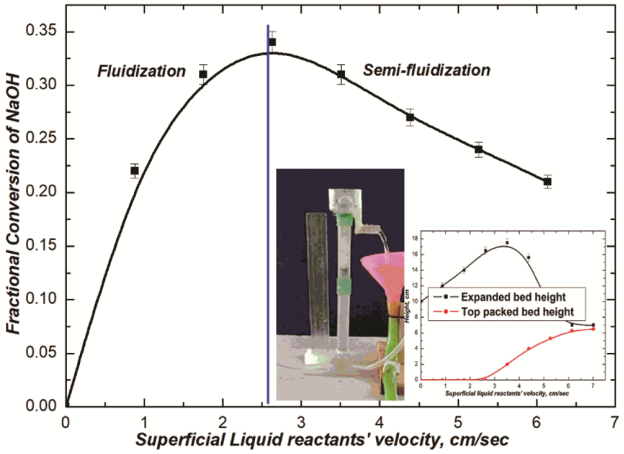

Fig. 3 — Variation in fractional conversion with superficial liquid reactants' velocity in a single-stage SFBR

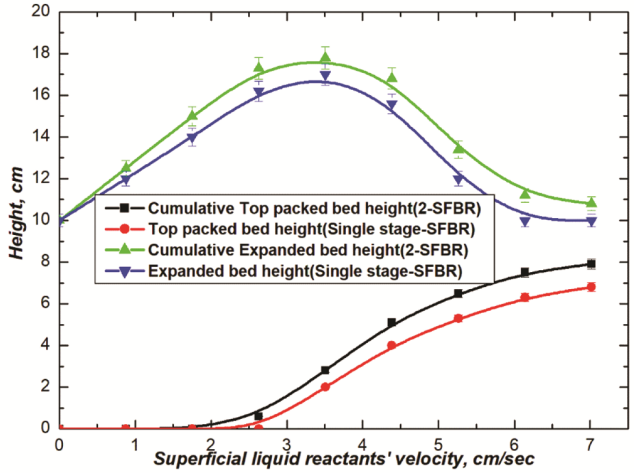

Fig. 4 — Comparative assessment of bed formation height with superficial liquid reactants' velocity

increase in the thickness of the top packed bed with the rise in superficial liquid reactants' velocity. On account of this, as more of the top packed bed becomes stationary (or less mobile), the contact of reactants with the active solid phase also decreases. Quantitatively, conversion fell by approximately 6-10% per centimeter of additional packed bed height under these conditions.

Two-stage SFBR

In the present range of investigation, the two-stage SFBR exhibited similar behaviour with respect to bed formation—both fluidized and semi-fluidized regimes across the tested superficial velocities. Compared to its single stage, a cumulative measure of bed formation (fluidized and packed regions) for the two-stageis is presented in Fig. 4. It was observed that the onset of fluidization and the development of the top packed bed occurred at lower superficial velocities in the 2-SFBR compared to the single-stage SFBR.

Fig. 5 presents a direct comparison of NaOH conversion in the saponification reaction between the two configurations (2-SFBR and single-stage SFBR). The two-stage reactor achieves substantially higher conversion levels across the tested superficial liquid velocity range. In the operational ranges studied, conversion in the 2-SFBR was observed to be 9-15% greater than in the single-stage reactor. The improvement may be attributed to the synergistic hydrodynamic effects of the staged arrangement in the 2-SFBR, unlike in the single-stage SFBR. The increased cumulative height of the fluidized and top-packed regions enlarges the contact pathway of the liquid through the reactor, while the intermediate separation between two stages redistributes the

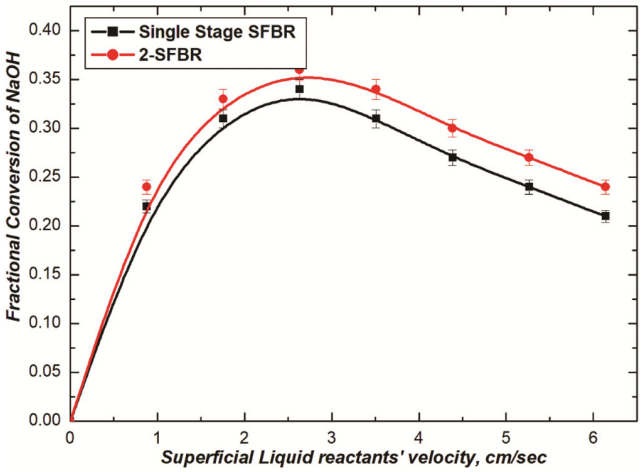

Fig. 5 — Comparison of single-stage and two-stage SFBR conversion

flowing liquid phase before it enters the second bed. Repetition of liquid redistribution facilitates improved solid-liquid contact, elevates mixing around the particles, enhances the available interfacial area for higher mass transfer, and reduces the tendency of channeling. This results in effective transport of reactant molecules to the reaction sites, which facilitates achieving higher reaction rates and conversion efficiency[26].

The laboratory-scale two-stage SFBR outperforms the single-stage SFBR in terms of conversion performance. These findings of the present study can act as a solid foundation for the development of multi-stage SFBRs, as its staged configuration and modular design provide enough flexibility for scaling up. For scaling up this reactor, some of the critical design

considerations will be the maintenance of the semi-fluidization regime, appropriate superficial liquid reactant velocity, solid phase nature, pressure drop, bed height, and liquid distribution in each stage. To validate the reactor performance under industrial operating conditions, pilot-scale studies of the two-stage SFBRs need to be undertaken in the future.

Conclusion

In this study, a mini-scale two-stage semi-fluidized bed reactor (2-SFBR) was fabricated and operated alongside a comparable single-stage SFBR to evaluate performance (conversion) in the saponification of ethyl acetate with sodium hydroxide. It can be concluded that 2-SFBR exhibits significantly higher conversion efficiency than the single-stage SFBR under equivalent solid loading, reactor height, and operating conditions. On average, the conversion of NaOH in the saponification reaction was increased by approximately 12% using the two-stage configuration. The repeated alternation of fluidized and top-packed bed behaviour—mimicking a plugflow-like pattern via stages—enhances reactant mixing, improves mass transfer, and leads to higher conversion. The key conclusions derived from this investigation include:

- The alternating fluidized and packed bed regions in semi-fluidized operation play a critical role in determining conversion.
- In the two-stage configuration, the cumulative top packed bed develops at lower superficial liquid velocities relative to the single stage, indicating earlier onset of semi-fluidization effects across stages.
- Although conversion improves with doubling the number of stages (from one to two), further improvements and optimization can be expected by exploring higher numbers of stages.
- Contacting pattern—how liquid and solid phases interact—is a major factor; since this study used regular spherical particles and only two stages, variation in particle shape, size, and more stages could yield further gains, which needs to be explored in the future in a broad sense.

Overall, the results of this work demonstrate that semi-fluidization can be effectively harnessed in multi-stage reactor configurations for very rapid liquid-phase reactions, especially in fertilizer, pharmaceutical, and chemical industries. However, for slow reactions as in biotech industries, such configurations of two-stage SFBR may not be able to provide satisfactory performance. Future investigations should explore scale-up, variation of solid phase materials, number of stages, and column and fluid medium characteristics to further enhance the performance of physical, chemical, and biological phenomena.

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Conflict of Interest

The authors declare that there are no conflicts of interest associated with this work.

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