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Modified Contactor for Experimental Studies of Mass Transfer and Chemical Reaction across a Liquid−Liquid Interface

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A modified stirred-cell type contactor has been designed and tested for its use in experimental investigations of mass transfer and chemical reaction in liquid−liquid systems. Its design overcomes the shortcomings of previously proposed contactors. It is easy to operate and has the ability to investigate the effect of a wide range of operating conditions. The mixing performance of the contactor was tested using a dye injection and a tracer technique. Experiments to evaluate mass-transfer coefficients across a liquid−liquid interface have been conducted to determine its effectiveness. The results verified that the contactor could be successfully employed as a useful experimental research apparatus.

Introduction

Many industrial processes involve mass transfer with and without chemical reaction across a liquid−liquid interface. These processes have been widely used in industry for manufacturing fine chemicals and pharmaceuticals, purification of chemicals, pollutant abatement, and selective separation. This has produced a need for fundamental mass-transfer and/or reaction kinetics experimental data for identification of transport parameters, intrinsic reaction kinetics, reaction network identification, process and equipment design and improvement, scale-up information, etc. (Levenspiel and Godfrey, 1974; Sotelo et al., 1990; Savage and Kim, 1985; Versteeg et al., 1987; Mills et al., 1992; Bart et al., 1990; Rodriguez Sevilla et al., 1993).

The mechanism of mass transfer across the free surface between two fluids is not fully understood, particularly when complex eddy motion is involved. For design of certain mass-transfer equipment, it is essential to understand properly the role and phenomena of convective mass transfer and kinetics; this has developed a need to study systematically all the involved variables. However, a reliable interpretation of liquid−liquid mass-transfer and reaction kinetics data requires a knowledge of the interfacial area for mass transfer, liquid phase mixing intensity, presence or absence of interface instability, magnitude of diffusional limitations, effect of interfacial contaminants, liquid−liquid thermodynamic equilibrium, rates of adsorption and desorption of surface-active solutes, and ionic equilibrium and ionic migration effects (Mills et al., 1992; Freeman and Tavlarides, 1980). Clearly, these and other related factors determine the selection of an appropriate contactor for investigation of liquid−liquid mass transfer and chemical reaction.

Therefore, an ideally designed experimental liquid−liquid contactor should satisfy the following requirements: (1) have a well-defined liquid−liquid interface area, (2) maintain a stable interface over a wide range of operating conditions, (3) provide independent control of the mixing intensity in each liquid phase, (4) provide well-defined hydrodynamics and flow characteristics, (5) maintain a clean interface between the liquid−liquid phases, (6) provide independent control of the parameters influencing transfer, (7) allow the user to operate efficiently over a wide range of operating conditions and contact time, (8) permit easier sampling of liquid phases, and (9) afford flexibility in handling and operating.

Many different types of experimental equipment for mass-transfer and chemical reaction studies have been developed for both liquid−liquid and gas−liquid systems such as stirred cell, packed-bed column, rotating disk, laminar jets, wetted wall column, string of disks or spheres, etc. (Mills et al., 1992; Al-Dahhan, 1988; Gordon and Sherwood, 1954; Lewis, 1954; Mayers, 1961; McManamey, 1961; Prochazka and Bulicka, 1971; Bulicka and Prochazka, 1976; Landau and Chin, 1977; Asai et al., 1983; Bart et al., 1990; Hancil et al., 1978; Colburn and Welsh, 1942; Vermijs and Kramers, 1954; Murdoch and Pratt, 1953; Bauer, 1974; Freeman and Tavlarides, 1980; Ward and Quinn, 1964; Duda and Vrentas, 1968; Quinn and Jeannin, 1961; Levenspiel and Godfrey, 1974; Danckwerts and Gillham, 1966; Danckwerts and Alper, 1975; Versteeg et al., 1987; Roberts and Danckwerts, 1962; Davidson and Cullen, 1957; Danckwerts, 1970; Stephens and Morris, 1951; Lynn et al., 1953; Bjerle et al., 1972; Scriven and Pigford, 1958; Raimondi and Toor, 1959; Nijsing et al., 1959; Chaing and Toor, 1959; Danckwerts and Kennedy, 1954; Govindan and Quinn, 1964). Mixed-liquid contactors are preferred since they more closely approach the operating conditions of industrial practice involving the mixing of liquid phases over vertical or horizontal surfaces, mechanical agitation, interaction in cocurrent or countercurrent flow through a packed bed, etc.

For liquid−liquid systems, the major shortcoming of investigations within packed-bed column (Colburn and Welsh, 1942) and/or rotating disk (Vermijs and Kramers, 1954) contactors is its undefined interface area. The liquid−liquid jet (Freeman and Tavlarides, 1980, Duda and Verntas, 1968; Ward and Quinn, 1964; Quinn and Jeannin, 1961), rising or falling drop (Bauer, 1974), and wetted-wall column (Murdoch and Pratt, 1953) contactors are limited to laminar flow. A properly designed stirred-cell type contactor provides a number of advantages over the other mentioned contactors. Hence, such a contactor has been used widely in mass-transfer studies with and without chemical reactions since it can provide a well-defined interfacial area, independent control of mixing intensity in each phase, and a uniform concentration in the bulk of each phase. However, the previously proposed stirred-cell type contactors that have been used for liquid−liquid mass transfer and chemical reaction studies have some shortcomings. Table 1 summarizes the essential features and short-

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Table 1. Essential Features and Shortcomings of the Current Stirred-Cell Type Contactors for Liquid−Liquid Systems

investigators	essential features and shortcomings
Gordon and Sherwood (1954)	The cell has a single shaft to agitate both phases dependently at the same speed. The interface area is equal to the cell cross sectional area and it is easily subjected to instability at relatively low agitation speed, since there are no baffles or interface plate.
Lewis (1954)	The cell has central shafts (paddles) for independently stirring each phase (the shaft for the lower section is mounted inside the shaft for the upper section). These shafts are introduced through the top of the cell's upper section, and, hence, the shaft for agitating the lower phase crosses the interface or the interface plate. The two phases are separated by a circumferential plate; this together with the thick central plate formed the annular interface area. The cell has no baffles. It was found that the interface was subjected to breaking up even at low agitation speeds (Bulicka and Prochazka, 1976). The cell is suspended in a constant-temperature bath.
Mayers (1961), McManamey (1961)	The cell is a compromise between Lewis and Gordon and Sherwood cells described above. The stirrers are located close to the interface which is easily subjected to wave motion and to breaking up even at low agitation speed.
Prochazka and Bulicka (1971)	The cell has central shafts (the shaft for the lower section is mounted inside the shaft for the upper section) for independently stirring each phase. The shafts are introduced through the top of the cell's upper section, and, hence, the shaft for agitating the lower phase crosses the interface or the interface plate. The two phases are separated by a thick central plate where the interface is formed between the central plate and the wall. Unlike the Lewis cell, impellers with 45° inclined blades are used. The stirrer is surrounded by two cylindrical grids mounted on the central plate (the inner grid is higher than the outer one). Wave motion of the interface occurs at relatively low to moderate agitation speeds. A thick central plate causes less mixing intensity near the interface compared to that produced in the bulk for a given agitation speed (Al-Dahhan, 1988). The cell is suspended in a constant-temperature bath.
Bulicka and Prochazka (1976)	It is a modified version of the Prochazka and Bulicka cell (1971). The shaft is surrounded by one cylindrical grid and stator equipped with 12 vertical baffles mounted on the central plate. The perforations of the cylindrical grid are omitted over a 4 mm wide strip on each side of the central plate and over the height of the impeller. The thickness of the central plate is 8 mm; hence, the bulk mixing does not represent the mixing intensity near the interface. It was found that its configuration provided more instability than what they claimed (Al-Dahhan, 1988).
Landau and Chin (1977)	Its configuration is similar to that of the Bulicka and Prochazka cell (1976). Both phases are independently agitated using central shafts (the shaft for the lower section is mounted inside the shaft for the upper section). The annular interface is formed between a thick central tapered ring and the wall. The stirrers are housed in a perforated shell with vertical baffles. It was found that this configuration produced rippling and breaking up (Al-Dahhan, 1988).
Hancil et al. (1978)	Both phases are mixed by a vibrating perforated plate and are dependently mixed. The flow is directed by the shape of the perforation which was oriented toward the interface. In this cell the turbulence in each phase was affected by frequency, amplitude, and distance of the plate from the interface. There was neither a central plate nor baffles. Hence, the interface is easily subjected to wave motion and breaking up.
Asai et al. (1983)	Both phases are independently mixed. The upper phase impeller shaft is introduced from the top, while the impeller shaft of the lower phase is introduced from the bottom. However, the upper shaft is extended and crossing the interface to the lower phase near its impeller to reduce vortex formation near the center of the interface. The stirrers are glass paddles with two flat blades. The cell has four glass baffles attached to the wall. It is subjected to a surface wave motion, but the authors assumed that the effects of wave motion on mass-transfer rate could be neglected.

comings of the current liquid−liquid stirred cell. It is noteworthy to mention that Levenspiel and Godfrey (1974) developed a stirred-cell type gradiantless contactor for a gas−liquid system and suggested that their contactor could be used for a liquid−liquid system. Unfortunately, for a liquid−liquid system, their contactor, as well as the other gas−liquid stirred cells (Danckwerts and Gillham, 1966; Danckwerts and Alper, 1975; Versteeg et al., 1987), provided an unstable interface when using low to moderate agitation rates. A detailed discussion of the disadvantages of the current contactors is given by Al-Dahhan (1988). In this study the Levenspiel−Godfrey (1974) contactor was modified to provide a more effective research contactor for investigating liquid−liquid systems.

Modified Stirred-Cell Contactor

The modified stirred-cell contactor is constructed from transparent acrylic plastic so that the interface stability could be easily observed. It consists of two identical sections, which are separated by an interface plate as shown in Figure 1. The volume of each cell section is 392 cm^3. Each cell section has two ends. One end accommodates the impeller shaft, sampling port, and inlet and outlet openings for the continuous operation mode. The other end holds the interface plate by acrylic flanges. Gaskets, 113 mm i.d. and 5 mm in width, are used between the flanges and the interface plate to prevent leakage. In this investigation, only one size and shape of the interface has been utilized; however, the flange design provides flexibility for testing different interface sizes and shapes. The interface plate is constructed from a 0.8 mm thick, stainless steel plate. It has three symmetrical, annular openings which give a total interfacial area of 31.6 cm^2. The interface plate has been chosen to be thin enough to permit the turbulence eddies from both phases to reach the interface and rigid enough to avoid any vibration which might produce interface rippling.

Mixing in each cell section is critical for the mass-transfer and chemical reaction studies in this type of contactor. The mixing in each phase must provide an

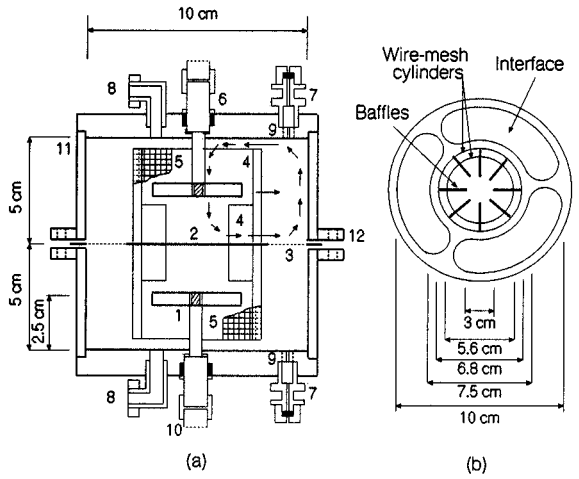

Figure 1. (a) Side view section of the developed cell assembly: (1) impeller, (2) interface plate, (3) interface, (4) baffles, (5) wire-mesh cylinders, (6) impeller shaft sealing, (7) sampling port, (8) inlet stream, (9) outlet stream (for continuous and semibatch operating mode options), (10) motor shaft, (11) cell body, (12) interface flange. (b) Top view of the interface, baffles, and wire-mesh cylinders configuration.

overall bulk or convection flow, homogeneity, and interface stability. Suitable configuration of perforated wire-mesh cylinders and baffles around the impeller is required to achieve the desired mixing with sufficient stability of the interface. The hydrodynamic conditions of the mixing within the designed stirred cell and the flow patterns of the mixed phase in each compartment are affected by the following: (1) the impeller type, size, and position away from the interface; (2) the perforated wire-mesh cylinders and baffles configurations around the impeller; (3) the fluid properties (Edwards et al., 1985; Oldshue, 1983; Bates et al., 1966). A pitched-blade turbine with four 45° inclined blades was found to produce the desired flow pattern (Al-Dahhan, 1988). Different impeller sizes along with the different configurations of baffles and perforated cylinders were tested for sufficient mixing and interface stability. In all these tests, the position of the impeller in each section was established at the half-distance of the section height. An unstable interface was found to occur at relatively low agitation speeds when using the pitched-blade turbine dimensions proposed by Bulicka and Prochazka (1976), Strake and Karcz (1985), Holland and Chairman (1966), and Oldshue (1983). A pitched-blade turbine, having a length equal to 41.6 mm and a width equal to 0.2 L (8.3 mm), was found to provide ample mixing with sufficient interface stability over an adequate range of agitation speeds.

The configuration of baffles and perforated wire-mesh cylinders around the impeller is important for directing a proper flow pattern and for stabilizing the interface over the desired agitation speeds. Wire-mesh cylinders, having 2 mm openings, were utilized. Such cylinders serve to limit the size of eddies, thus facilitating the formation of a uniform turbulent flow structure throughout the cell compartments. They also help eliminate any tangential velocity component of the bulk flow. The baffles were designed to stabilize the interface and transform the tangential flow to a vertical one. Different configurations of baffles and wire-mesh cylinders were tested for interface stability over the desired range of agitation speed and for fluid mixing using a pitched-blade turbine fixed in the middle of the cell section. The configuration that was found to be the best but not the optimum, as shown in Figure 1, consists of two wire-mesh cylinders with the same height mounted on the central solid part of the interface plate, eight stainless steel baffles (4 mm width with the same height as the cylinders) mounted between these two cylinders, and another eight baffles (13 mm in width and 20 mm high) vertically fixed inside the inner cylinder and equally spaced and aligned to those baffles mounted between the cylinders. Other tested configurations involved two wire-mesh cylinders without baffles and two wire-mesh cylinders with the following: only four baffles inside the inner cylinder, four baffles between the cylinders with another four inside the inner cylinder, 12 baffles between the cylinders with another 12 inside the inner cylinder, and the later configuration with an annular disk around the inner baffles. The designed cell's flow pattern can be described as a downward flow from the impeller zone toward the interface plate where it is diverted into upflow to the upper part of the compartment; there it flows back down into the impeller zone as illustrated by the arrows in Figure 1. This flow pattern determines the inlet and outlet stream positions in the case of a continuous operation mode (Figure 1). Samples are drawn manually from each section. The total volume of drawn samples is negligible compared to the phase volume (less than 1% of the initial phase volume).

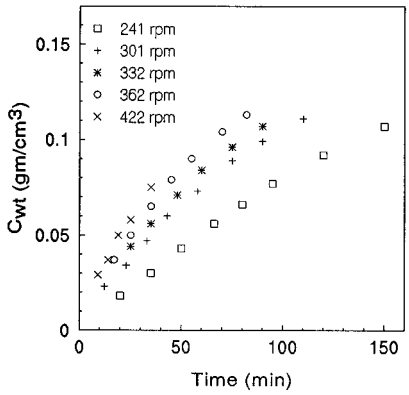

Figure 2. Measured water concentrations in the *n*-butanol phase with time, when both phases were agitated at the same speed.

The cell's support structure is an important feature of the developed cell. It is designed to provide the desired flexibility in handling and operating the cell while investigating a wide range of operating conditions (for a detailed mechanical design, see Al-Dahhan, 1988).

Mixing performance tests were performed in a batch mode to verify that the designed cell provides adequate mixing and fluid uniformity. This was accomplished using dye injection and tracer technique tests. These tests demonstrated that the mixing performance was sufficient to provide a uniform fluid throughout the cell compartments over a wide range of agitation rates (Al-Dahhan, 1988).

The mass-transfer operations were performed using a mutual partially-miscible water/*n*-butanol binary system in batch operation mode. Figure 2 shows an example of the experimental data of the measured water concentration in the *n*-butanol phase (C_{wt}) with time when both phases were agitated at the same speed. A detailed mass-transfer study using this contactor can be found in Al-Dahhan and Wicks (1996) and Al-Dahhan (1988) which confirmed how well the modified cell could be utilized as a useful research tool.

Closing Remark

The modified contactor is flexible enough to overcome the shortcomings of previous experimental contactors. Its general features are summarized as follows: (1) it provides sufficient mixing and phase uniformity in each cell section at low to high agitation rate, (2) the mixing intensity (agitation rate) in each phase can be controlled independently, (3) it has a well-defined interface area, and different area sizes and configurations can be easily mounted and tested as well, (4) a stable interface can be obtained over a desired range of agitation, (5) interface stability can be observed during the operation, (6) its design and support structure permit easy and wide flexibility in varying its hydrodynamic conditions by easily replacing the baffles and wire-mesh cylinders configuration, impeller of different type, size, and position within the cell section, and the interface plate of different sizes and shapes, (7) it can be easily operated in batch, continuous, and semibatch modes, (8) it provides easy sampling of liquid phases, (9) it would be utilized for gas−liquid systems, and (10) a temperature control bath or jacket can be easily added to evaluate the effect of temperature.

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