Residence time distribution and liquid holdup in Kenics® KMX static mixer with hydrogenated nitrile butadiene rubber solution and hydrogen gas system

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ABSTRACT

A Kenics® KMX static mixer that has curved-open blade internal structure was investigated to study its hydrodynamic performance related to residence time distribution and liquid holdup in a gas/liquid system. The static mixer reactor had 24 mixing elements arranged in line along the length of the reactor such that the angle between two neighboring elements is $90°$. The length of the reactor was 0.98 m with an internal diameter of 3.8 cm and was operated cocurrently with vertical upflow. The fluids used were hydrogen (gas phase), monochlorobenzene (liquid phase) and hydrogenated nitrile butadiene rubber solution (liquid phase). In all the experiments, the polymer solution was maintained as a continuous phase while hydrogen gas was in the dispersed phase. All experiments were conducted in the laminar flow regime with the liquid side hydraulic Reynolds number in the range of 0.04–0.36 and the gas side hydraulic Reynolds number in the range of 3–18. Different polymer concentrations and different operating conditions with respect to gas/liquid flow rates were used to study the corresponding effects on the hydrodynamic parameters such as Peclet number (Pe) and the liquid holdup (ε_L). Empirical correlations were obtained for the axial dispersion coefficient (D_a) and liquid holdup in liquid system alone and for the gas/liquid system separately. It was observed that the Peclet number decreased with the introduction of gas in to the reactor while in the liquid system alone, an increase in viscosity decreased the Peclet number. The liquid holdup was empirically correlated as a function of the physical properties of the fluids used in addition to the operating flow rates.

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1. Introduction

Static mixers are tubular devices inside which there are static mixing elements aligned in a particular configuration. Static mixers are widely used in the chemical process industry not only for polymer processing applications but also for systems where multiphase reactions are performed. They offer many advantages over the conventional reactors (such as packed bed reactors, fluidized bed reactors) especially when used for gas/liquid systems. The main advantages are that they assure: plug flow behavior, enhanced heat and mass transfer, generation of very high interfacial surface area while other advantages include low maintenance cost, low space requirements and easy installation. The purpose and the performance of the static mixer depend on the type of internal element geometry used for a particular system (Visser et al., 1999; Baker, 1991; Cybulski and Werner, 1986; Myers et al., 1997). Based on the geometry of the internal structure, the static mixers are broadly classified into five categories viz., open designs with helices, open designs with blades, corrugated plates, multilayers designs and closed designs with channels or holes (Thakur et al., 2003). For gas/liquid and liquid/liquid systems where generation of a large interfacial area is required, helical elements, open designs and corrugated plates are used. Among these three types, if the system under study is highly viscous, internal structures with open designs are commonly used. The two most important commercial open blade designs available and used for many applications in the chemical process industry are Sulzer® SMX and Kenics® KMX. The Sulzer® SMX element has intricate open blade design with the blade being flat while the Kenics® KMX design has identical structure except that the blades are concavely curved. With respect to mixing, Heniche et al. (2005) showed that the Kenics® KMX structure is better than the Sulzer® SMX at the expense of a higher pressure drop.

Knowledge about the residence time distribution (RTD) in static mixers is very important when it is used as a chemical reactor (Streiff and Rogers, 1994). Different residence time distribution models for static mixers with helical elements have been proposed for both Newtonian and non-Newtonian flow (Nigam and Naumann, 1985; Pustelnik, 1986; Hobbs and Muzzio, 1997). Li et al. (1998) published a RTD model for flow of non-Newtonian fluids in static mixers with Sulzer® SMX internal structure. Madhuranthakam et al. (2009a)

Table 1
Physical properties of the working fluids (at 25 °C and atmospheric pressure).

Property	MCB	2.5% (w/w) Polymer	5.0% (w/w) Polymer	Hydrogen gas
Density, ρ (kg/m³)	1106	1163	1166	2.901
Viscosity, μ (Pa s)	0.004	0.0739	0.1613	11.2×10^{-6}
Surface ension, σ (N/m)	0.02926	0.02826	0.02781	–

have showed that the RTD in the Sulzer$^{®}$ SMX static mixer with water alone and air/water systems could be modeled using the plug with axial dispersion (PFAD) model. They have proposed an empirical correlation for the Peclet number as a function of gas side and liquid side Reynolds numbers. However, very high Reynolds numbers are used in their investigation. With respect to the liquid holdup data in the static mixers, most of them are used in a homogeneous regime (where both the phases are in the same flow regime) where they use a "no-slip velocity" condition which may only be true for limited cases (Heyouni et al., 2002). Depending on the type of reaction, the ratio of the flow rate of gas to liquid could be in the range of 0.1–6 which makes the flow regime to be in homogeneous or transition or heterogeneous regime. When the gas/liquid flow is either in transition or heterogeneous regime, modeling of the liquid holdup using the "no-slip velocity" condition could lead to large errors. Considerably less literature is available on the hydrodynamic behavior in the Kenics$^{®}$ KMX static mixer (with concavely curved-open design structure) when used for gas–liquid systems. The hydrodynamic information such as RTD, liquid holdup, heat and mass transfer coefficients in the SM are very important not only for the design of the reactor but also for further scale up of the process. In the present study, RTD and liquid holdup in the Kenics$^{®}$ KMX static mixer is studied in both liquid and gas/liquid systems. With different concentrations of the polymer solution (liquid phase) and different operating conditions, RTD and liquid holdup experiments were conducted. Empirical correlations are obtained for the axial dispersion coefficient and liquid holdup as corresponding functions of the operating conditions and physical properties of the fluids used.

2. Experimental

2.1. Experimental fluids

The working fluids used in the study are: mono chlorobenzene (supplied by Fisher Scientific), hydrogenated nitrile butadiene rubber solutions (obtained from hydrogenation of nitrile butadiene rubber solutions), hydrogen gas (99% pure, supplied by Praxair) and *n*-butylamineacetate (CH_3COOBu, prepared by titrating acetic acid (supplied by Fisher Scientific) with tributyl amine (nBu_3N, supplied by Fisher Scientific)). The physical properties of the working fluids are presented in Table 1. The density and viscosity of the working fluids are estimated by using the correlations obtained by Pan and Rempel (2004) while the surface tension is measured using an axisymmetric drop shape analysis (ADSA) system.

2.2. Experimental set-up

The experimental set up consists of a static mixer with twenty four mixing elements and the internal structure of the element has Kenics$^{®}$ KMX geometry (concave open blade supplied by Kenics$^{®}$ Company, Massachusetts, USA). Numerical simulations related to hydrogenation of nitrile butadiene rubber in static mixers performed by Madhuranthakam et al. (2009b) showed that a reactor with 20

mixing elements would give conversions above 95% and hence, with a safety design factor of 20%, 24 elements are used in the hydrodynamic study. The reactor was configured vertically up, with the gas and the liquid entering cocurrently at the bottom and leaving at the top. The liquid saturation and mass transfer rates will be better in a vertical upflow configuration compared to a downflow configuration especially when the reaction locus is in the liquid phase (continuous phase) which is generally true in the case of post polymerization processes (Larachi et al., 1991; Wu et al., 1996; Moreira et al., 2004). Fig. 1 shows the reactor set up used to study the RTD and liquid holdup characteristics in the static mixer with Kenics$^{®}$ KMX geometry. The reactor is made up of stainless steel with 3.8 cm ID 80S. The total length of the reactor is 1.22 m while the length of the active zone (where the mixing elements are present) is 0.98 m. The geometry of the internal element used in the reactor is shown in Fig. 2. These elements are arranged in the reactor so that each element is at an angle of 90° with its neighboring element. The intricate net shaped internal structure splits a single flow into several partial flows and these partial flows in turn mix together after one complete rotation which is obtained after every four elements in this type of reactor. Two reciprocating pumps (supplied by Milton Roy Company) were used to separately pump the polymer and the tracer solutions.

2.3. Methods

The pumps are primed before pumping the polymer solution and the tracer into the reactor. The required flow rates are set by adjusting the pump settings. The liquid holdup is measured by using the conventional method of volumetry technique. In this method, the desired gas and liquid flow rates are set at the desired settings and run for at least two residence times till steady flow is assured. The pumps and the gas flow are suddenly turned off and simultaneously the valves at the top and bottom of the reactor are closed. Using the three way valve at the bottom of the reactor the polymer solution in the reactor is drained and the volume of the drained solution is measured. The ratio of the volume of the polymer solution drained to the volume of drained polymer solution when there is no gas flow gives the liquid holdup (ε_L). RTD experiments were performed with a polymer solution system alone and with a hydrogen gas–polymer solution system. An organic salt solution of *n*-butylamineacetate (CH_3COOBu) was used as tracer in the RTD experiments. In the RTD experiments also, the reactor was initially operated for at least two times of the residence time until the flow becomes steady and then tracer was pumped from the bottom of the reactor. Discrete samples are collected at the sampling ports present at the first and twenty fourth elements. A continuous measurement of the conductivity was not possible because, the gas bubbles in the gas/polymer mixture caused a zigzag concentration profile. The conductivity of the samples is measured using a conductivity cell (probe, supplied by Sensorex). A step input in the tracer is used in the RTD experiments. Because of the low concentration of the organic salt (tracer), experiments with pulse input were found to be unfruitful. Hence, a step input is used in the RTD experiments. The advantage with the step input over the pulse input is that there is no need to know about the total amount of tracer used in the polymer solution over the experimental time period. The major disadvantages with the step input are that the computation of the exit age distribution involves differentiation of the data (that can lead to large errors) and a large amount of tracer is required for each experiment. Mathematically, the step input is represented by Eq. (1).

$$C_{TO}(t) = 0, \quad t < 0$$
$$C_{TO}(t) = C_o, \quad t \geqslant 0 \tag{1}$$