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Short communication

Solid–liquid mass transfer studies in trickle bed reactors

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ABSTRACT

Solid–liquid mass transfer was studied in a trickle bed reactor packed with copper cylindrical particles. Diffusion-controlled dissolution of copper in acidified dichromate solution was used to study the solid–liquid mass transfer. The effect of liquid and gas throughputs and particle diameter on solid–liquid mass transfer was investigated.

Keywords: Diffusion; Mass transfer; Hydrodynamics; Packed bed; Multiphase reactors; Turbulence

1. Introduction

Trickle bed reactor (TBR) implies a reactor in which a liquid phase and a gas phase flow co-currently downward through a fixed-bed of catalyst particles where reaction takes place. The liquid and gas phase flow co-currently upward in certain cases. The hydrodynamics and heat and mass transfer will be different for co-current upward flow and co-current downflow operations (Saroha and Khera, 2006).

Solid–liquid mass transfer rate has been experimentally determined by many investigators using the following techniques:

1. Dissolution of sparingly soluble solids into liquids (Sato et al., 1972; Goto and Smith, 1975; Sylvester and Pitayagulsarn, 1975; Lemay et al., 1975; Specchia et al., 1978; Hirose et al., 1976; Satterfield et al., 1978; Ruether et al., 1980; Sedahmed et al., 1996).
2. Electrochemical technique (Chou et al., 1979; Delaunay et al., 1982; Rao and Drinkenburg, 1985; Gabitto and Lemcoff, 1987; Latifi et al., 1988; Rode et al., 1994).
3. Chemical reactions with significant solid–liquid mass transfer resistance (Morita and Smith, 1978).
4. Ion exchange followed by an instantaneous irreversible reaction (Yoshikawa et al., 1981).
5. Dynamic absorption (Tan and Smith, 1982).

Technique 1 is used by most of the investigators but this technique appears to underestimate the solid–liquid mass transfer rate in low interaction regime, perhaps due to incomplete catalyst wetting (poor wetting of the packing by the liquid). The wetting efficiency has a significant effect on solid–liquid mass transfer. Most of the mass transfer correlations proposed by various investigators for the low interaction regime are generally in terms of Sh.ΦSc$_{-1/3}$ where Φ is the wetting efficiency. The term Φ was introduced because it is difficult to measure 'k' and 'a' separately in a TBR. The solids are completely wetted in the high interaction regime and wetting efficiency equals unity. The electrochemical technique facilitates direct and instantaneous measurements of solid–liquid mass transfer and is thus very useful in pulsing flow conditions. The electrochemical technique suffers from maldistribution of current and potential at the surface of catalyst.

2. Experimental setup

The diffusion-controlled dissolution of copper in acidified dichromate solution was used in the present study to determine solid–liquid mass transfer. The schematic diagram of the experimental setup is shown in Fig. 1. Experiments were performed in a 2.54 cm (i.d.) perspex column packed with copper wires oriented at random. The diameters of three types of copper wires used in the present study were 0.05 cm, 0.1 cm and 0.165 cm and the length of the each element of copper wire was 1.2 cm. The column length was made up of three sections—copper cylindrical particles in the middle zone and glass spheres of 5 mm diameter at the top and bottom of the cylindrical particles. The working section (copper cylindrical

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Nomenclature

a	surface area of particles per unit volume of packed bed
a_s, a_w	geometrical surface area and wetted area per unit volume of bed
A	cross-sectional area of column
C	concentration of liquid
C_S	concentration of liquid at the particle surface
C_Z	concentration at distance Z
$C_{Z+\Delta Z}$	concentration of liquid at distance $Z+\Delta Z$
k	mass transfer coefficient
Q_L	volumetric flow rate of liquid
Re	Reynolds number
Sc	Schmidt number
Sh	Sherwood number
t	time spent by the liquid in copper section
u_G	superficial gas velocity
u_L	superficial liquid velocity
ϵ	void fraction

particles) of the column (40 cm) was preceded by 30 cm high inlet section packed with glass spheres of 5 mm diameter to avoid channeling. A similar section of glass spheres followed the middle section of copper particles. The packing was supported on a perspex disc fitted at the base of the column.

Experiments were conducted with the down flow of liquid; acidified solution of potassium dichromate was used as the liquid phase. Potassium dichromate solution was stored in a glass storage vessel at a height and was gravity fed to the column. Metallic parts were avoided in the experimental setup to avoid corrosion problem and undue loss of dichromate. The liquid flow from the storage vessel was experimentally measured by collecting the volume for a fixed interval of time. A constant pressure was maintained in the dichromate storage vessel to eliminate the effect of height of liquid in the vessel by inserting a glass dip tube nearly upto the bottom of vessel. Dichromate solutions of three different concentrations were used: 0.5 M H_2SO_4 + 0.003 M $K_2Cr_2O_7$, 1 M H_2SO_4 + 0.003 M $K_2Cr_2O_7$ and 2 M H_2SO_4 + 0.003 M $K_2Cr_2O_7$. The change in dichromate solution concentration with time during each run was determined by withdrawing samples at regular time intervals from the outlet of the column and titrating against standard ferrous ammonium sulfate using diphenylamine as an indicator. All the experiments were performed at ambient conditions of temperature and pressure. The reproducibility of the experimental results was found to be within ±5%.

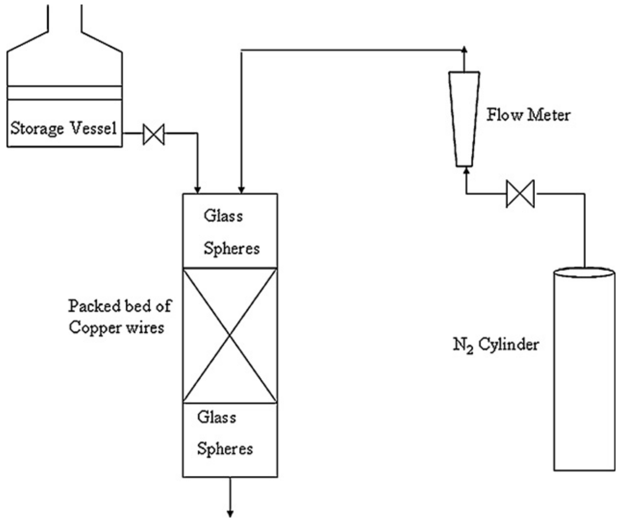

Fig. 1 – Experimental setup for the determination of solid–liquid mass transfer coefficient.

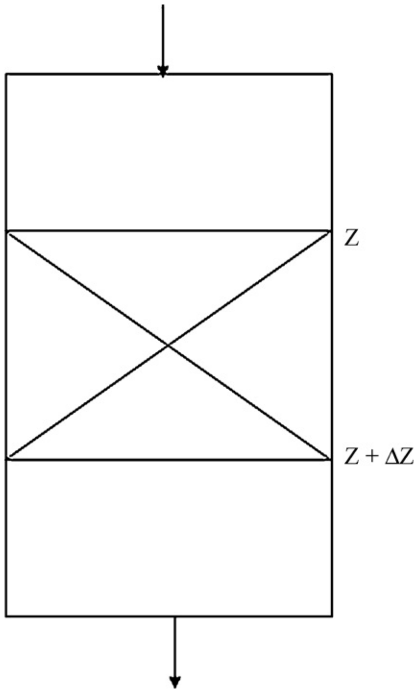

Fig. 2 – Packed bed configuration.

The experiments were also performed with co-current downflow of liquid and gas to study the effect of flow of gas on solid–liquid mass transfer. Nitrogen, due to its inert nature, was fed from a cylinder and the flow rate was measured by a flowmeter. Superficial velocities of nitrogen gas ranged from 6 to 17 cm/s while those of dichromate solution were in the range of 0.5–4.0 cm/s.

The fractional wetted area was evaluated by applying the following correlation of Onda, Sada, Kido and Tanaka reported by Hirose et al. (1976):

$$\frac{a_w}{a_s} = 1 - \exp\left\{-0.225\left[\frac{-Re_L}{(6(1-\epsilon))}\right]^{0.43}\right\}$$

The reaction between copper and chromate is a very fast redox reaction. Since the objective of the present study is to determine liquid–solid mass transfer, concentration of dichromate ions was kept very low (0.003 M) to make diffusion control as the rate-determining step. In such a situation time required for the reaction is very low as compared to the time required for the diffusion of the ion from bulk of the liquid to the interface. So it can be assumed that the process is diffusion controlled.

Mass transfer coefficient was determined from the dichromate concentration–time data. Plug flow is assumed in the packed bed and axial dispersion is neglected (Fig. 2). Making a mass balance of chromate ions in the packed bed

$$Q_L.(C_Z - C_{Z+\Delta Z}) = (\Delta Z.A).a.\,k.(C - C_S)$$

Dividing by ΔZ and taking the limit $\Delta Z \to 0$

$$-Q_L.\,dC/dZ = A.\,a.\,k.(C - C_S)$$