Design of a Slurry-foam Reactor System for the Manufacture of Precipitated Calcium Carbonate

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Abstract

Design methodology for a system of slurry-foam reactors in series for the manufacture of precipitated calcium carbonate is presented. Contributions of both the storage and foam sections of the slurry-foam reactors, towards the conversion of reactants, have been taken into account in the development of the mathematical model. Values of liquid holdup and solid-liquid mass-transfer coefficient, required for reactor(s) simulation, have been estimated from dimensionless correlations, developed using the experimental data for carbonation of hydrated lime in slurry in a semibatch slurry-foam reactor. Surface coefficient values and the initial particle-size distributions of hydrated lime determined have been experimentally. A design chart has been presented in the form of a figure.

Key words: Design, precipitated calcium carbonate, series reactors, slurry-foam reactor.

Introduction

A slurry-foam reactor consists of a shallow

Manuscript received July 20, 2008.

pool of reactant slurry and a tall column of slurry-foam above it. The promising features of this reactor are large gas-liquid contact times and interfacial areas, low liquid holdups and low to moderate pressure drops. Precipitation reactions incorporating a pure gas and a reactant slurry can be conveniently carried out in such a reactor: fouling problem is less even with a sticky material because of the presence of a surfactant in the liquid and only a marginal cost is incurred for the surfactant used in generation of a stable foam as the solvent can be completely recycled back into the reactor after separation of the product solid. The exit gas can also be recycled to the reactor helping approach the zero-discharge goal with respect to the "greenhouse" gas. During the recent two and half decades, many important experimental studies on gas absorption with chemical reaction and successful theoretical predictions of foam-reactor performance have been published (Biswas and Kumar, 1981; Bhaskarwar and Kumar, 1984; Asolekar et al., 1988; Subramanyam 1999: et al.,

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Sharma et al., 2005, to name a few). Subramanyam et al. (1999) presented a design methodology for a gas-liquid foam-bed reactor system and demonstrated it with an example. However, conversion of reactants only in the foam section of the reactors was considered in the model. In the present work, the design methodology for a slurry-foam reactor system incorporating sparingly soluble fine reactive particles of hydrated lime for the of manufacture precipitated calcium carbonate (PCC) has been presented taking into account, for this fast reaction system, the contributions of both the storage and foam sections towards the conversion of reactants.

A model for slurry-foam reactors in series

For production of PCC in industrial scale using continuous slurry-foam reactor. volumetric flow rate of slurry being sufficiently high, conversion obtainable in a single pass is likely to be small. This necessitates the use of multiple foam reactors in series or in parallel. A convenient multi reactor configuration is the reactors-in series as it obviates the need for recirculation of partially converted slurry. Figure 1 shows a series of N_r slurry-foam reactors in continuous operation. Slurry and gas flows are crosscurrent to each other. The exit gas from each reactor flows into a header pipe. This may be

recycled back to the reactors after removal of moisture by condensation and making up with fresh gas. PCC can be separated from product slurry using a rotary vacuum filter and the solvent can be recycled back for preparation of fresh-lime-slurry feed to the reactors.

Formulation

For p^{th} reactor, the mass balance equations for species A and B, and the volume balance equations for dissolving particles in slurry can be written as given below. It is assumed that, initially each of the N_r reactors contain V_{sl} m³ of slurry. Operation of reactors is started with the flow rate of Q m³/s of slurry into the system with a total loading of hydrated lime equal to $C_B^{T}(0)$. The conversion in the p^{th} reactor at the end of reactor operation time t can be calculated with a step size Δt from a knowledge of the concentration of component $B (C_{B_{(p)}}^{T})$, subscript 'm' indicating the average concentration, has been omitted here for brevity).

$$X = \frac{V_{l}.C_{B}^{T}(0) + \left[\sum(Q.(\frac{V_{l}}{V_{sl}})\Delta t)C_{B}^{T}_{(p-1)}\right] - V_{l}.C_{B}^{T}_{(p)} - \left[\sum(Q(\frac{V_{l}}{V_{sl}})\Delta t)C_{B}^{T}_{(p)}\right]}{V_{l}.C_{B}^{T}(0) + \left[\sum(Q.(\frac{V_{l}}{V_{sl}})\Delta t.)C_{B}^{T}_{(p-1)}\right]}$$
(1)

, where $C_{B_{(p)}}^{T}$ is the total molar loading of unreacted *B* (both dissolved in solution and also present as particles) at time 't' per



Make-up gas

Figure 1. Slurry-Foam reactors in series

volume of the solvent (water in this case).

$$C_{B_{(p)}}^{T} = C_{B_{(p)}} + \sum_{i} (n_{p_{i(p)}} \frac{\pi d_{p_{i(p)}}^{3}}{6}) \frac{\rho_{B}}{M_{B}} \frac{V_{sl}}{V_{l}}$$
(2)

Conversion obtained in N_r reactors in series during time period t can be written as

$$X = \frac{N_r V_l C_B^{T}(0) + t Q_l(\frac{V_l}{V_{sl}}) C_B^{T}(0) - \sum_{p=1}^{N_r} V_l C_B^{T}(p) - \sum_{p=1}^{r} (Q_l(\frac{V_l}{V_{sl}}) \Delta t) C_B^{T}(N_{r,r})}{N_r V_l C_B^{T}(0) + t Q_l(\frac{V_l}{V_{sl}}) C_B^{T}(0)}$$

(3)

The first term in the numerator indicates amount of component B (k mol) in the slurry filled in all the N_r reactors before the reactors start operating. The second term signifies amount of *B* (k mol) entering the first reactor in the series (which flows through the successive reactors) over the time period *t*. The third and fourth terms indicate total amount of *B* (k mol) left out in all the reactors after time *t* and discharged from N_r th reactor during the reactor operation time *t*, respectively.

Component material- balance equations:

At the beginning of a time interval Δt ,

the p^{th} reactor contains $V_l C_{A (p)}$ k mol of Aand during the entire time interval Δt , receives $Q.(V_l/V_{sl})$. $\Delta t C_{A, (p-1)}$ kmol of A from the $(p-1)^{\text{th}}$ reactor. Assuming that the entire quantity $Q.(V_l/V_{sl})$. $\Delta t C_{A, (p-1)}$ is added to the p^{th} reactor at the beginning of the time interval instead of over the entire time interval and applying similar arguments for component Band volume of particles, the average concentration of components A, B, and volume of particles of i^{th} size fraction in the slurry after mixing are calculated as follows.

Material balance for component A:

$$C_{A,m_{(p)}} = \frac{C_{A_{(p-1)}} . Q.(\frac{V_l}{V_{sl}}) . \Delta t + C_{A_{(p)}} . V_l}{Q.(\frac{V_l}{V_{sl}}) . \Delta t + V_l}$$
(4)

Material balance for component B:

$$C_{B,m_{(p)}} = \frac{C_{B_{(p-1)}} . Q.(\frac{V_l}{V_{sl}}) . \Delta t + C_{B_{(p)}} . V_l}{Q.(\frac{V_l}{V_{sl}}) . \Delta t + V_l}$$
(5)

Volume balance of particles of ith size fraction:

$$v_{pi,m_{(p)}} = \frac{v_{pi_{(p-1)}}(Q\Delta t.n_{pi}) + v_{pi_{(p)}}(n_{pi}V_{sl})}{(Q\Delta t + V_{sl})n_{pi}}$$
(6)

The model developed (Jana, 2007) for a semibatch slurry-foam reactor comprising of material balance equations for components Aand B and the volume balance equation for sparingly soluble particles of component B(since the component B is continuously supplied by dissolution of the solid to liquid phase), is extended to each of the N_r reactors in series operating in a continuous mode. These equations along with equations (2)-(6) are solved simultaneously using a fourth order Runge-Kutta method with a step size ' Δt ', equal to10⁻⁵ s to predict the concentrations and volume of particles of *i*th size fraction prevailing at the end of the time step. For calculation for the next time step, the initial concentrations and particle volumes are estimated for each reactor using equations (4) to (6). The above procedure is repeated for subsequent Δts to predict the conversion using equation (3) as a function of time.

Physico-chemical parameters: D_A , D_B , k_2 , k_s , $d_{pi}(0), k_l a_b, k_{sl}$, and $\overline{\varepsilon}_l$; equipment parameters, *viz.*, r_c , d_o , p_t , H_N , p' and operational variables: $Q, C_B^{T}(0), Q_G, h, X, H \text{ and } N_r \text{ are needed for}$ simulation of reactors using the set of equations as mentioned above. Values of D_A , D_B and k_2 have been taken from literature and those of k_s and $d_{pi}(0)$ have been measured experimentally. Values of $k_l a_b$ have been calculated using the correlation given by Sada et al. (1985). Data for k_{sl} and $\overline{\varepsilon}_{l}$ have been estimated from dimensionless correlations developed using the experimental data, operational variables, and physico-chemical and equipment parameters. Effective of slurry, μ_{eff} , viscosity required for

estimation of these parameters has been obtained from the correlations given by Capuder and Koloini (1984).

Q is calculated using the desired plant capacity and an assumed value for $C_B^{T}(\theta)$ (for a solids loading of 15 percent by mass). Values for d_o and p' are assumed based on an allowable pressure drop for flow through the distributor plate and N_H is calculated for a chosen radius of the foam reactor. Smaller diameter holes on the gas-distributor plate produce smaller gas bubbles leading to a larger interfacial area but at the cost of a larger gas-pressure drop. Values of Q_G are taken 10 percent in excess of that corresponding to the weeping velocity which in turn is estimated using the relevant equipment parameters and operational variables [9].

$$V_{ow} = 0.0229 \left(\frac{\sigma_{l.}g_{c.}}{\mu_{G}}\right) \left(\frac{\mu_{G}^{2}.\rho_{l.}}{\sigma_{l}g_{c}\rho_{G}^{2}d_{o}}\right)^{0.379} \left(\frac{1}{d_{o}}\right)^{0.293} \left(\frac{2A_{a}d_{0}}{\sqrt{3}p^{3}}\right)^{2.8/(z/d_{o})^{0.724}}$$

(7)

, where $A_a = \pi (r_c - 4p')^2$

Total area of orifices on the distributor plate, A_o , has been calculated using the equation [9] $A_o = 0.907 (d_o/p')^2 A_a$

Weeping velocity, Q_{ow} and superficial velocity of gas, u_G are thus obtained as

 $Q_{ow} = V_{ow} A_o; \quad Q_G = 1.1 Q_{ow}; \quad u_G = Q_G / A_c$ (8) Height of the slurry-pool (*h*) in the storage section is assigned a value of a few centimeters to keep the pressure drop low.

Design of a slurry-foam reactor system is presented below for the manufacture of 50 tons of precipitated calcium carbonate per day using hydrated lime and pure carbon-dioxide gas as the raw materials and Triton X-100 as the surface active agent. Design data (Jana, 2007) are shown in Table 1. Suitable values for radius of the column, r_c and solids loading (as mentioned above) are assumed and values of the variables / parameters: u_G , Q, μ_{eff} , $k_I a_b$, k_{sl} and $\overline{\varepsilon}_l$ are estimated. Height of foam

8	
Rate of production of PCC	: 50 tons/day
Desired conversion of lime	: 99 percent
Purity of hydrated lime	: 92.28 percent hydrated lime, 7.72 percent CaCO ₃
Slurry concentration	: 150 kg hydrated lime/m ³ solvent
Slurry flow rate	$: 3.0784 \text{ x } 10^{-3} \text{ m}^3/\text{s}$
Concentration of surfactant (Triton X-100): 1.64 kg/m ³	
Surface coefficient	$: 0.57 \times 10^{-4} \text{ m/s}$

Table 1. Design data for slurry-foam reactors in series

Column, required for 99 percent (or higher) conversion of hydrated lime in a given number of reactors in series is then predicted using equations (2)-(6) along with the material balance equations for components A,B and the volume balance equation (Jana,2007) for suspended particles of componentB. Figure 1 shows a design chart prepared



Figure 1. Variation of the number of slurry-foam reactors in series with the height of foam bed (Design Chart I)

$Q = 3.0784 \times 10^{-3} \text{ m}^{3}/\text{s}$	
$d_0 = 1.0 \times 10^{-6} \text{ m}$	u _G = 16.1 x 10 ⁻² m/s
$C_B^{T}(0) = 2.0243 \text{ kmol/m}^3$	$C_{\rm S} = 1640 \; {\rm ppm}$
$d_p = 6.74 \ \mu m$	k _S = 5.7 x 10 ^{−5} m/s

using the results of simulation. Heights of foam columns have been plotted against the number of reactors required in series for 99 percent conversion of hydrated lime under the operating conditions mentioned above. It is seen that the number of reactors and the height of foam columns reduce with an increase in the column-radius. One can choose an aspect ratio $(H/2r_c)$ of about, say 4, and find out the combination of H and r_c for which a reasonably small number of reactors, say not more than 10, are required.

Conclusion

Single-stage model of a semi-batch slurryfoam reactor has been used to develop the performance equation for a system of continuous reactors in series. A methodology for the design of reactors in series has been demonstrated with an example for the manufacture of 50 tons of precipitated calcium carbonate per day using hydrated lime and pure carbon-dioxide gas as raw materials and Triton X-100 as the surfaceactive agent. The design chart presented in the form of a figure permits selection of the best design among the various alternatives.

Notation

- A substance A, present in gas phase as well as dissolved in liquid
- A_a active area, for flow, on the distributor plate, m²
- A_c cross-sectional area of foam reactor, m²
- A_o total area of orifices on distributor plate, m²
- *B* substance *B* present in slurry as dissolved solute as well as dissolving particles

- $C_{A_{(p)}}, C_{B_{(p)}}$ concentrations of components A and B (dissolved), respectively in reactor 'p' at the start of a time step Δt prior to mixing with the feed slurry (fresh feed or inflow from $(p-1)^{\text{th}}$ reactor), k mol m⁻³
- $C_{A,m_{(p)}}$, $C_{B,m_{(p)}}$ mean concentrations of components *A* and *B*, respectively in reactor 'p' at the start of a time step Δt , k mol m⁻³
- C_{AG0} concentration of A in gas phase entering each reactor
- $C_B^{T}(0)$ initial total loading of *B* per unit volume of solvent in fresh feed , k mol m⁻³
- $C_{B}^{T}(p)$ total (dissolved in liquid as well as undissolved particles) loading of *B* per unit volume of solvent in reactor '*p*' at any time *t*, k mol m⁻³
- $C_{Bf,p}$ total loading of reactant *B* in reactor '*p*' in the drainage stream at any time *t*, k mol m⁻³
- C_s concentration of surfactant, ppm
- D_A liquid-phase diffusion coefficient of component A, m² s⁻¹
- D_B liquid-phase diffusion coefficient of component B, m² s⁻¹
- d_o diameter of holes on the gas distributor plate, m

- $d_{pi_{(p)}}$ particle diameter in the i^{th} size fraction
 - in slurry in reactor 'p', m
- d_p average diameter of lime particles, m
- *H* height of foam column, m
- H_N number of holes in the gas distributor plate, dimensionless
- *h* height of liquid in the storage section, m
- k_2 second-order reaction rate constant, m³ (k mol)⁻¹ s⁻¹
- $k_l a_b$ volumetric overall liquid-phase masstransfer coefficient, s⁻¹
- k_s gas-liquid mass-transfer coefficient in presence of surface resistance and chemical reaction in foam section, ms⁻¹
- k_{sl} solid-liquid mass-transfer coefficient, m s⁻¹
- M_B molecular weight of component *B*, kg $(\text{kg mol})^{-1}$
- $n_{pi_{(p)}}$ number of particles in the i^{th} size fraction per unit volume of slurry, m⁻³
- N_r total number of reactors in series
- p_t thickness of distributor plate, m
- p' pitch on the distributor plate, m
- Q flow rate of slurry into/ from the storage section, m³ s⁻¹
- Q_G flow rate of gas into the storage section, m³ s⁻¹
- Q_{ow} flow rate of gas at weeping velocity, m³ s⁻¹
- r_c radius of foam column, m

 u_G superficial velocity of gas, m s⁻¹

- $v_{pi_{(p)}}$ volume of particles of i^{th} size fraction in reactor 'p' at the start of a time step Δt prior to mixing with the feed slurry (p=1) or with inflow from $(p-1)^{th}$ reactor, m³
- $v_{pi,m_{(p)}}$ mean volume of particles of *B* (after mixing) of i^{th} size fraction in reactor '*p*' at the start of a time step Δt , k mol m⁻³
- V_l volume of solvent in slurry, m³
- V_{sl} volume of slurry, m³
- V_{ow} weeping velocity of gas, m/s
- *X* percentage conversion of lime in slurry
- *z* length of liquid path on the distributor plate, m

Greek Letters

- $\overline{\varepsilon}_l$ average liquid hold-up in the foam section, dimensionless
- μ_{eff} effective slurry viscosity, kg (m.s)⁻¹
- μ_G viscosity of gas, kg (m . s)⁻¹
- μ_l viscosity of liquid, kg (m. s)⁻¹
- ρ_B density of component *B*, kg m⁻³
- ρ_l density of liquid, kg m⁻³
- ρ_G density of gas, kg m⁻³
- σ_l surface tension of liquid, N m⁻¹

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