

**Effects of Adding Alcohols, Electrolytes and Carboxyl Methyl Cellulose (CMC) on  
Design Parameters in Internal Loop Airlift Bubble Column**

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**Abstract**

The effect of gas velocity, liquid-phase properties, solid-particle concentration and the static liquid height on gas hold-up ( $\epsilon_g$ ) and mass transfer coefficient ( $K_{La}$ ) were studied in a three phase internal loop airlift bubble column (ILBC) (the ratio of the draft tube to column diameter equal to 0.5). Air was used as a gas phase. Water and seven aqueous solutions of 10% concentration methanol, ethanol, NaCL, acetic acid, 50% glycerol and 2% CMC were used as the liquid phase. Polyethylene-non-porous-solid particles with a concentration of (50,100) Kg/m<sup>3</sup> were used as solid phase. Superficial gas velocity varied from 0.01 m/s to 0.1 m/s and air dispersed into the center of the draught tube by using a porous gas distributor. The results showed that ( $\epsilon_g$ ) and ( $K_{La}$ ) increase with increasing gas velocity and decrease with increasing solid particle concentration, static liquid height, viscosity and surface tension of liquid-phase.

**Keywords:** Alcohols; Electrolytes; Hydrodynamics; Mass transfer; Bubble columns.

**Introduction**

Bubble column reactors belong to the general class of multiphase reactors which consist of three main categories namely; the trickle bed reactor (fixed or packed bed), fluidized bed reactor, and the bubble column reactor[1,2]. Slurry columns similar to fluidized bed columns, in that a gas is passed through a column containing solid catalyst particles suspended in a fluid. In slurries the catalyst is suspended in a fluid, in fluidized beds the suspending fluid is the reacting gas itself[3,4]. In (SC) an attempt is made to realize intensive and intimate contact between a gas-phase component and a finely dispersed solid. With respect to this purpose (SC) are related to packed bed columns with the different gas-liquid flow regimes that can be realized (such as trickle flow, pulsed flow, dispersed bubble flow etc....). Also there is a lot of similarity with three-phase fluid bed systems. The latter systems share many properties with (SC), but the main difference is the fact that in fluid beds with upward fluid flows the drag force acting on the solids by the gas and liquid flow is on the average balanced by the net weight of the particles, while in (SC) the overall liquid-solid slip velocities are practically zero and particles remain suspended by the action of the turbulence, in the liquid phase[5,6].

Deckwer and Schumpe,[7] studied the effect of various design parameters. They found that mixing time decreases initially with increasing gas velocity and then

increases in the higher range of velocity. Guerin et al., [8] studied the effect of gas flow rate on mixing time. They found that the mixing time does not decrease proportionally with the increase of gas flow rate, and this is a main difference between BC and mechanically stirred columns. Solid-mixing and solid replacements are important factors in cases where the solids have a short lifetime[9].

Fan and Chern,[10], studied solid-mixing in a gas-liquid-solid system. They reported three-states of solid mixing, these states are complete segregation, partial intermixing and complete intermixing. Particle size in (SC) can be small to very small, even down to the submicron range. The effect of average particle size ( $dp$ ) and solid density on the critical superficial gas velocity ( $V_{gc}$ ) is given as[11]:

$$V_{gc} \propto d_p^{0.47}, V_{gc} \propto \left[ \frac{\rho_s - \rho_L}{\rho_L} \right]^{0.95} \quad (1)$$

The pressure drop in (SC) is usually more or less independent of the gas flow and close to the hydrostatic pressure. Of course there is also a pressure drop required for the gas distributor.

Slurry columns can be classified according to the phases where the reactants are present. Generally (SC) can be classified according to[6]:

- The chemical system.
- The contacting pattern and mechanical hold –up-fractions devices.

Hydrodynamics of (SC) includes the study of mixing and the volumetric liquid side mass transfer coefficient at the gas-liquid interface ( $K_{La}$ ). For the design of (SC), whether agitated only by the flowing gas or assisted by one or more stirrers, the conditions at which the particles are just suspended are very important. Therefore, generally only a minimum suspension criterion (M.S.C) is considered [12].

Roy et al., [13] studied by applying the pressure drop technique a large variety of gas solid and liquid systems including non-aqueous systems and particles

$$V_g (\text{min, actual}) = 1.25 \frac{D_c}{0.0508} \cdot \exp(3W_s) \cdot V_g (\text{min, theo}) \quad \text{for } w_s \leq 0.1$$

$$\text{Where } n = 0.2 \dots \text{for } d_p < 100 \mu\text{m} \quad (2)$$

$$n = 0.5 \dots \text{for } d_p > 200 \mu\text{m}$$

The average hold-up fractions of gas, solids and liquid should satisfy the equation:

$$\epsilon_g + \epsilon_s + \epsilon_L = 1 \quad (3)$$

In contrast to the three-phase fluid-beds where the relation between the three-phase hold ups can be rather complex, in slurry columns with the much smaller particles and slip velocities, the relation between ( $\epsilon_i$ ) and ( $\epsilon_s$ ) is often simple as it is fixed by the feed ratio of solids liquid phases, or liquid and solids volumes are constant (in batch systems). The bubble hold-up is much more difficult to predict, first of all because of the different regimes that might prevail both in stirred vessels and slurry sparger columns. Shah and Godbole, [14] studied the regimes in three-phase flow sparger. They showed that there are three regimes. The regimes are:

- Uniform is bubbling at low gas velocity.
- Churn-turbulent-flow at higher-gas velocities, with a mixture of large and small bubbles.
- Slugging in small diameter columns, where the large bubbles are comparable to the column diameter.

Koide et al., [17] studied the three phase flow with different solid concentrations; they showed that the effect of solid particles on reducing the  $\epsilon_g$  value in the transition regimes is larger than that in heterogeneous regime. Akita and Yoshida, [18] studied the gas hold up in BC and proposed a correlation for gas hold up in terms of bond and Galilo numbers.

$$\frac{\epsilon_g}{(1 - \epsilon_g)^4} = K \cdot B_o \cdot G_a \cdot F_r \quad (4)$$

Miller, [19] studied the gas hold up in three phase system and proposed for the churn turbulent regime in three

with different degree of wetting. Shah and Godbole, [14] studied the (M.S.C) for different column heights. Koide found that the effect of solid particles on reducing ( $\epsilon_g$ ) value in the transition regime is larger than that in heterogeneous regime. Fuku, and Chern, [15] studied the (M.S.C) for a column with a draft tube; they showed that with a draft-tube applied in the system, a much smaller volumetric flow rate of gas was required to suspend that same amount of solid, compared with normal column. Narayanan et al., [16] studied the (M.S.C) in sparged vessels with a stagnant liquid medium, and gave a relation to minimum gas velocity to suspend the particles as in this equation:

phase fluidization to use the Akita's equation using liquid/solid suspended bulk properties instead of liquid properties.

$$\epsilon_g = \frac{1}{8} Ln \left[ 1 + 8C_g^{7/24} \left[ \frac{\mu_B}{\rho_B} \right]^{\frac{1}{6}} \left[ \frac{\sigma}{\rho_B} \right]^{\frac{1}{8}} V_g \right] \quad (5)$$

Koide and Horibe, [20] studied the effect of using solids in the draught tube in a solid suspended bubble column. For an adequate description of mass transfer with chemical reaction in slurry columns, reliable data on the following two types

a- parameters which are specific for slurry columns ( $K_G$ ,  $K_L$ ,  $K_{La}$ ,  $K_{Ga}$ ).

B- Parameters which are not specific for the type of reactor applied (intrinsic reaction kinetics) [3].

Akita and Yoshida, [18] showed that a better empirical equation of ( $K_{La}$ ) can be obtained by using ( $\epsilon_g$ ) instead of ( $V_g$ ) in the BC:

Koide and Horibe, [20] studied the bubble column with draught tube. Koide et al., [21] studied BC with draught tube and with gas dispersion into the annulus, the values of ( $K_{La}$ ) and ( $\epsilon_g$ ) are much larger than those in the bubble column without draught tube, when a liquid with frothing ability is used.

$$\frac{K_{La} \cdot \sigma_L}{\rho_L \cdot D_i \cdot g} = 2.11 \left[ \frac{\mu_L}{\rho_L \cdot D_i} \right]^{0.5} \left[ \frac{g \cdot \mu_L^4}{\rho_L \cdot \sigma_L^3} \right] \dots \times \varepsilon_g^{1.18} \quad (6)$$

### Purpose

The purpose of this study is to clarify experimentally the effects of, gas velocity and properties of the liquid and solid particles on  $\varepsilon_g$  and  $K_{La}$  in the solid suspended bubble column with a draught tube in liquid solid batch operation.

### Experimental Section

A schematic diagram of the experimental setup in this work is shown in Figures 1a and 1b. A Plexiglass column of 0.09 m inside diameter and about 1.30 m total height with draught tube dimensions of 0.045 m inside diameter and 0.09 m total height was used. The draught tube was fitted with three support legs in the upper and the lower end of the column so as to locate it in a central position at any distance above the base. The column consists of two main sections namely, the gas inlet section and the liquid recycling testing section. The gas inlet section consists of a gas distributor. At the bottom of this section, two lines are connected together before entering the distributor section each line has a valve to be opened or closed as required. One of these lines is the air inlet flow. Air compressor supplied the line with the desired amount of air needed; the amount of air was measured using a gas meter. The other line is the nitrogen gas inlet flow. The nitrogen gas was supplied from a cylinder. A gate valve was used in the nitrogen flow, which must be shut off when the air was spared to the column, and must be opened during the desorption process. The liquid testing section contains two openings, one for liquid out-flow and the other for liquid in flow. The circulation of liquid in the column was achieved using a dosing pump placed in the recycling line. A ball valve placed in the middle of the recycling line was used to take various samples at various times to measure the concentration of the dissolved oxygen during the operation. The column was filled with water to the desired height above the distributor (0.3, 0.5, and 0.7) m. Then the solid particles (polyethylene 3mm particle diameter) were added to the liquid in the column. The concentration of solid particles to each level of static liquid were (50, 100) kg/m<sup>3</sup> respectively. Compressed air at (100-150) psig was supplied using a reciprocating compressor. The desired air flow rate was set-up using gate valve and the amount measured with a gas meter. The dissolved oxygen concentration in the liquid phase was measured using oxygen meter, which consists of a probe metal electrode. The liquid phase (batch) consists of the following systems (only water, water and solid,

water, alcohols and solids, water, NaCl and solid, water, acetic acid and solids, water, CMC and solids). The gas distributor (figure 1c) was constructed from a ceramic material and the type is the porous gas distributor. The distributor has an equivalent pore diameter of 0.1 mm and free section of 0.80%.

### Results and Discussion

*Gas holds up ( $\varepsilon_g$ ) and solid hold up ( $\varepsilon_s$ )*

The average gas hold up  $\varepsilon_g$  was calculated from equation (7) Using the data of the static slurry height ( $H_L$ ) and the height of the aerated slurry ( $H_F$ ) which were determined by visual observation:

$$\varepsilon_g = \frac{H_F - H_L}{H_F - (V_i / S_o)} \quad (7)$$

$(V_i / S_o)$  In equation (7) is a correction term for the volume of the draft tube [21].

The solid hold up was calculated from equation (8) Using the data of static liquid height ( $H_L$ ) and the height of slurry after adding solid particles  $\bar{H}_F$ .

$$\varepsilon_s = \frac{\bar{H}_F - H_L}{\bar{H}_F} \quad (8)$$

The experimental gas hold up was found by measuring the difference between initial liquid height and final liquid height. Since it was rather difficult to read directly the level of the aerated liquid the values of gas hold up thus obtained probably involves an error of about 5%, established via repeated measurements. Figure (2a) shows the effect of gas velocity for water system with and without solid particles. The gas hold up was found to increase with increasing gas velocity, because the large bubble holdup increases with increasing gas velocity leading to the increase of the overall holdup. But the effect without solid particles is larger than that with solid particles. When the gas velocities above 0.01m/sec and the liquid phase without solid particles, bubble size is independent of gas flow rate. The buoyant force of the bubble is opposed by viscous drag of the fluid, and if bubble size is independent of gas flow rate, the velocity of bubble rise will show similar independence. Hence increased gas flow rate will increase the gas hold up, and ultimately the liquid will see to be filled with bubbles. Figure (2b) shows, in slurries the presence of solid particles in the liquid might enhance bubble coalescence into larger bubbles and therefore reduces the value of gas hold up. The solid particles retard the bubble rise velocity and prevent

increases in bubble size. Figure (3a) shows, the effect of the static liquid height on gas hold up. It is clear that increasing the static liquid height will decrease the gas hold up. As the static liquid height is increased, the bubbles have time to coalesce further and ultimately reduce gas hold up. In our experimental studies the maximum static liquid height used was 0.70 m. In slurries the presence of solid particles in the liquid might enhance bubble coalescence into larger bubbles and therefore reduce gas hold up. Figure(3b) shows the effect of using different liquid phase (alcohols and electrolytes) respectively, on gas hold up. Low electrolyte concentrations have no noticeable effect on the surface tension of the solution. However the ionic forces in the liquid bulk reduce the bubble rise velocity and the bubble coalescence. As a result, the gas hold-up increase. For high electrolyte concentration, the interfacial tension increases, resulting in increased bubble size and reduce gas-holdup.

#### Mass transfer coefficient

The physical absorption of oxygen in the air by the liquid was employed to determine the mass transfer coefficient. A material balance of oxygen in the liquid gives:

$$K_{La} = \frac{2.303(1 - \varepsilon_g - \varepsilon_s)}{t} \cdot \text{Log} \frac{C_{Sa} - C_i}{C_{Sa} - C_o} \quad (9)$$

Rearranging equation (9) gives:

$$\text{Log} \frac{C_{Sa} - C_i}{C_{Sa} - C_o} = \frac{K_{La}}{2.303(1 - \varepsilon_g - \varepsilon_s)} \cdot t \quad (10)$$

Plotting the left hand side of equation (10) with (t), the average slope of the plot will give the term  $K_{La}/2.303(1 - \varepsilon_g - \varepsilon_s)$ . the values of  $(\varepsilon_g)$  and  $(\varepsilon_s)$  were determined as mentioned in (7) and (8) respectively, then  $(K_{La})$  can be calculated. Figure (4a) shows that the mass transfer coefficients increase with increasing gas velocity. The axial dispersion coefficients  $(D_L)$  increase with increasing gas velocity and therefore increase  $(K_{La})$ . Figure (4b) shows the effect of solid particle concentration on  $(K_{La})$ . The presence of solid particle in the liquid will decrease the axial dispersion coefficient and it enhances bubble coalescence. The bubble size will be larger and occupying larger space in the column and therefore reduces  $(K_{La})$ . At a higher gas velocity (0.1 m/sec, the effect of solid particles on  $(K_{La})$  will be less than in low gas velocities (0.03m/sec). Figures (5a) and (5b) show the effect of static liquid height on the mass transfer coefficient. As the static liquid height is increased, however the bubble has time to coalesce further and ultimately decreases the axial dispersion

coefficient and the mass transfer coefficient. Figure6 shows the effect of liquid phase properties on  $(K_{La})$ . As mentioned before, the ionic forces in the liquid bulk reduce the bubble rise velocity and the bubble coalescence, so that the mass transfer coefficient is increased.

#### Conclusions

- (i) The presence of suspended solid particles in the bubble column with a draught tube and the ratio of the draught tube diameter to column diameter equal to 0.5 reduce the values of holdup  $\varepsilon_g$  and the volumetric liquid-phase mass transfer coefficient  $k_{La}$ . The reduction of  $\varepsilon_g$  and  $k_{La}$  values due to an addition of solid particles to the column increases with increasing solid concentration and liquid phase (water, glycerol and solids, Newtonian and water, CMC non-Newtonian) viscosity.
- (ii) The gas hold-up and mass transfer coefficient in bubble column with a draught tube, where gas is dispersed into the center of the base of the inner draught tube using a pours multi hole distributor increase with increasing gas velocity, for  $V_g$  equal or less than 0.1m/sec, only for the case of (water, glycerol and solids) system, the mass transfer coefficient reaches its maximum at gas velocity of 0.02 m/sec.
- (iii) When the static liquid height is increased, the bubble has time to coalesce further and ultimately decreases the axial dispersion coefficient and reduces both gases hold up and mass transfer coefficient.

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### Nomenclature

$C_i$	Concentration of dissolved oxygen at any time, p.p.m
$C_0$	Initial concentration of dissolved oxygen, p.p.m
$C_{Sa}$	Saturated concentration of dissolved oxygen, p.p.m
$C_S$	Solid particle concentration, $K_G/m^3$
$D_C$	Column diameter, m
$D_i$	Diffusivity of oxygen in solution, $m^2/sec$
$D_L$	Axial dispersion coefficient (liquid), $m^2/sec$
$D_p$	Average particle size, m
$g$	Acceleration of gravity, $m/s^2$
$H_L$	Static slurry height, m
$H_F$	Level of aerated slurry, m
$\bar{H}_F$	Level of liquid phase & solids, m
$K_L$	Liquid phase mass transfer coefficient, m/s
$K_{La}$	Overall mass transfer coefficient, based on aerated slurry volume, 1/s
$S_C$	slurry column
$t$	Time, min
$V_g$	Gas velocity, m/sec
$V_{gc}$	Critical gas velocity, m/s
$W_S$	Mass of solid, /kg liquid

### Greek symbol

$\varepsilon_g$	Gas hold up
$\varepsilon_s$	Solid hold up
$\rho_L$	Liquid phase density, $kg/m^3$
$\rho_S$	Solid phase density, $kg/m^3$
$\mu_L$	Liquid phase viscosity, CP
$\nu_L$	Kinematic viscosity of liquid phase, $cm^2/s$
$\sigma_L$	Liquid phase surface tension dyn/cm

### Dimensionless groups

$Bo$	Bond number, $(G \cdot D_C^2 \cdot \rho_L) / \sigma$
$Fr$	Froud number, $Vg / (g \cdot D_C^{0.5})$
$Ga$	Galilo number $Vg \cdot D_C^3 \cdot \rho_L / \mu_L^2$

### Subscripts

$G$	gas
$L$	liquid

**Table 1. Physical-properties for pure liquids at T=20 °C**

	$\rho$ (Kg/m <sup>3</sup> )10 <sup>3</sup>	$\mu$ CP	$\sigma$ dyn/cm	$v_L$ cm <sup>2</sup> /sec
Water	0.998	1.002	72.86	1.004
Methanol	0.791	0.584	22.61	0.738
Ethanol	0.789	1.200	22.27	1.520
Glycerol	1.261	1.005	6304	0.796
Acetic-Acid	1.049	1.219	27.6	1.162
NaCL	2.165	1.295	72	0.598
CMC	1.008	K=0.012 ps.s <sup>n</sup> n=0.8	73	1.23

The solution of CMC (carboxy methyl cellulose) shows non Newtonian, pseudo plastic behavior, which can be described by the power law of Ostwald and deweale:

$$\tau = K \dot{\gamma}^n$$

Where:-

K: Ostwald factor (consistency index)

n: flow behavior index

$\dot{\gamma}$ : shear rate 1/sec

T: shear stress

$$\mu_{\text{eff}} = \tau^{\frac{1}{n-1}}$$

where  $\mu_{\text{eff}}$ : effective liquid phase viscosity Pa.s

$$Y = 5000 Vg \text{ [22]}$$

Where Vg: gas velocity m/sec.

**Table2. Physical properties for mixtures used with various concentration at T=20°C.**

	$\rho$ (kg/m <sup>3</sup> )10 <sup>3</sup>	$\mu$ CP	$\sigma$ dyn/cm	$v_L$ cm <sup>2</sup> /sec
Water-Methanol 10%	0.9815	0.795	22.63	0.8226
Water-Ethanol 10%	0.981	0.910	22.64	0.9400
Water-Acetic acid 10%	1.026	0.916	22.225	0.8932
Water-NaCL 10%	1.0216	0.9247	48.375	0.9051
Water-glycerol 50%	1.126	6.00	64	0.8905
Water-CMC 2%	1.009	K=1.320 Pas <sup>n</sup> n=0.5	69	0.09051

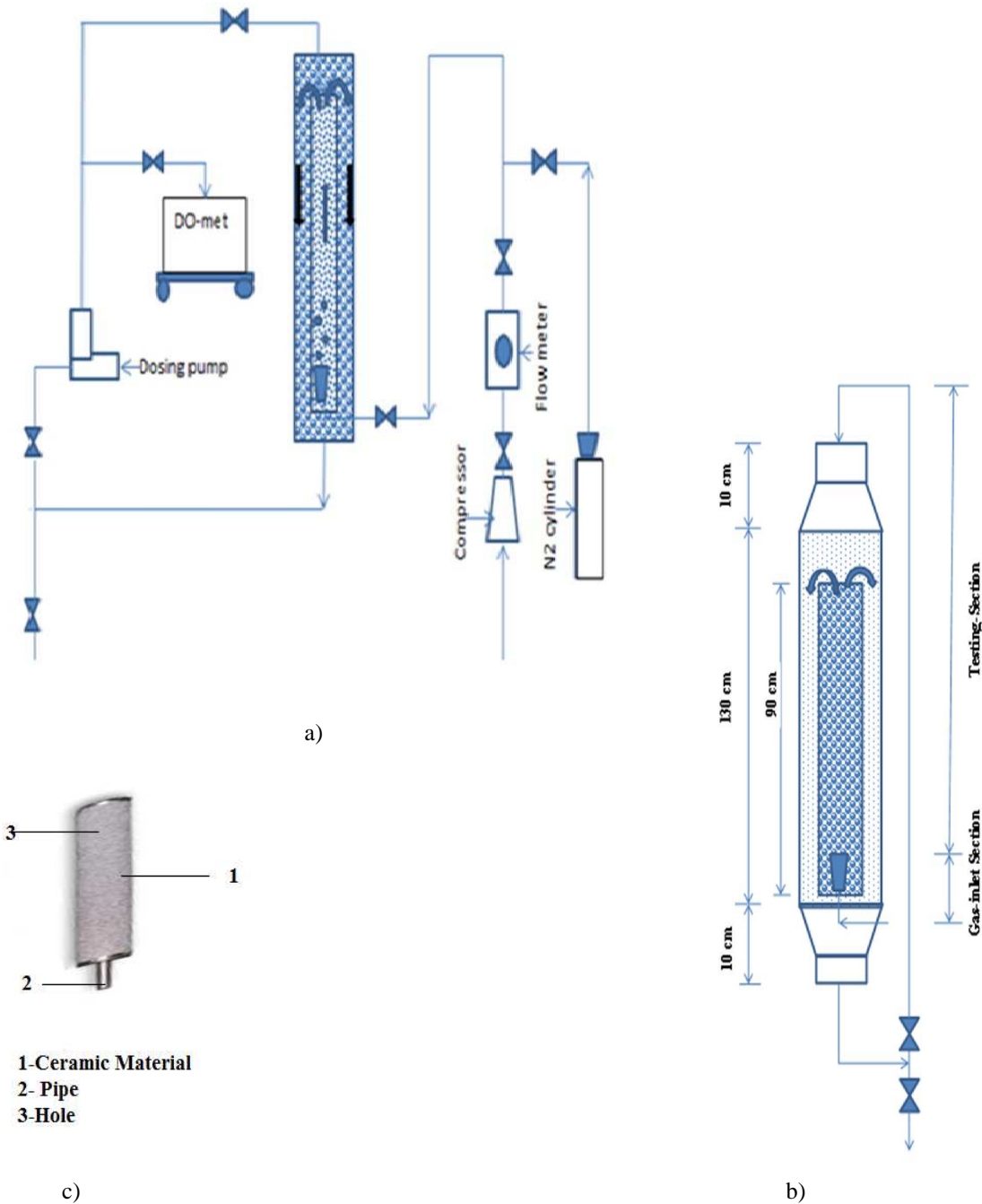


Fig.1.(a) Experimental apparatus; (b)column; (c) gas distributor.

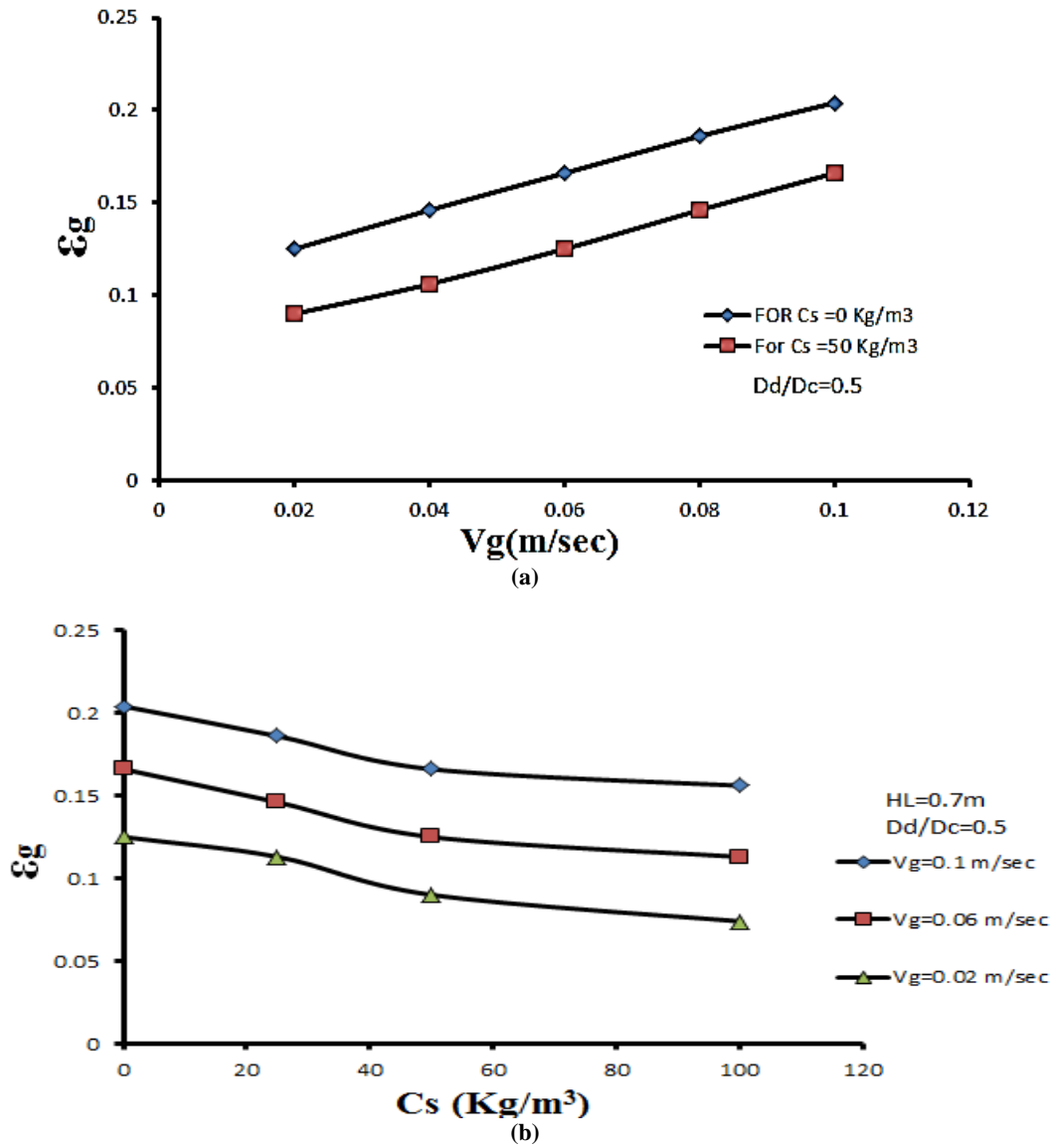


Fig.2.(a) Gas hold-up versus gas velocity for water system; (b) Gas hold up versus solid concentration for water system for various gas velocities.



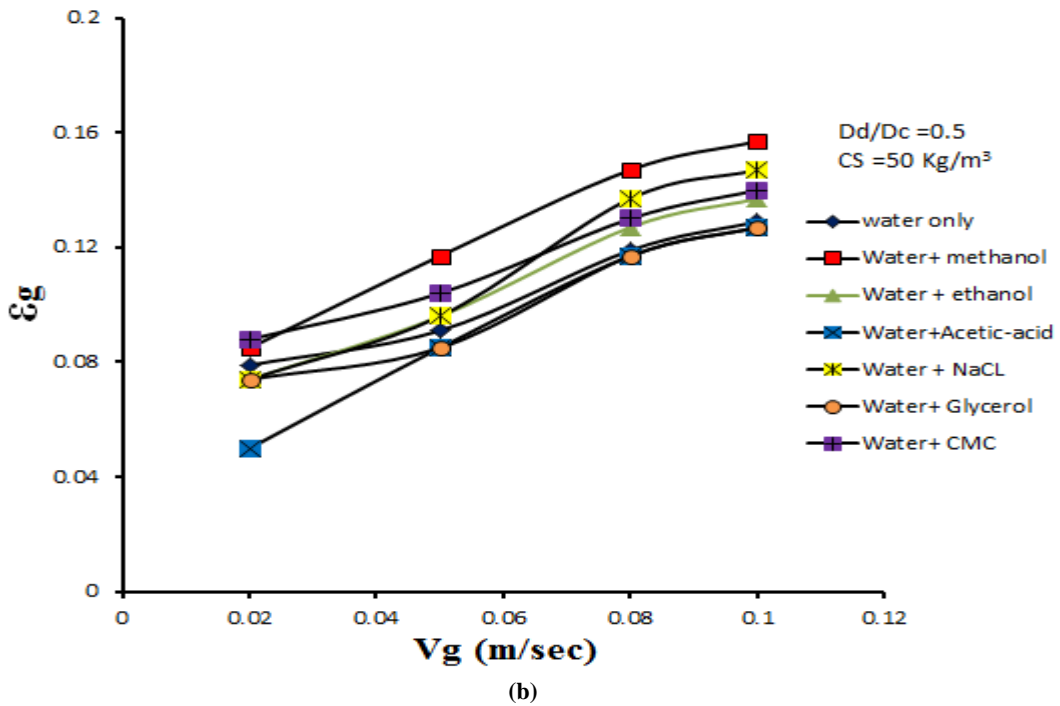
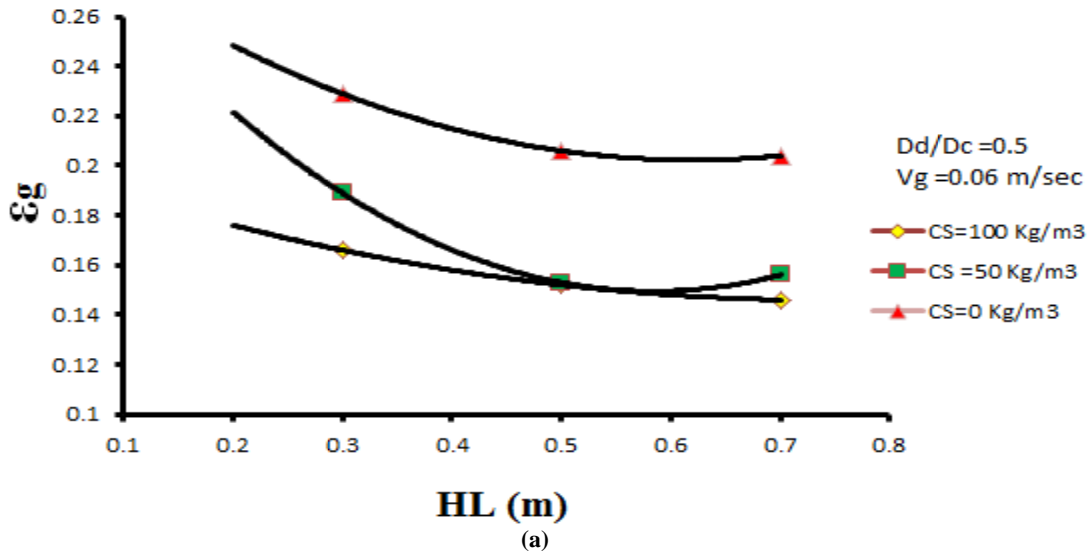


Fig. 3.(a) Gas hold-up versus liquid height for water system for various solid concentrations; (b) Gas hold up versus gas velocity for different liquid phase system.

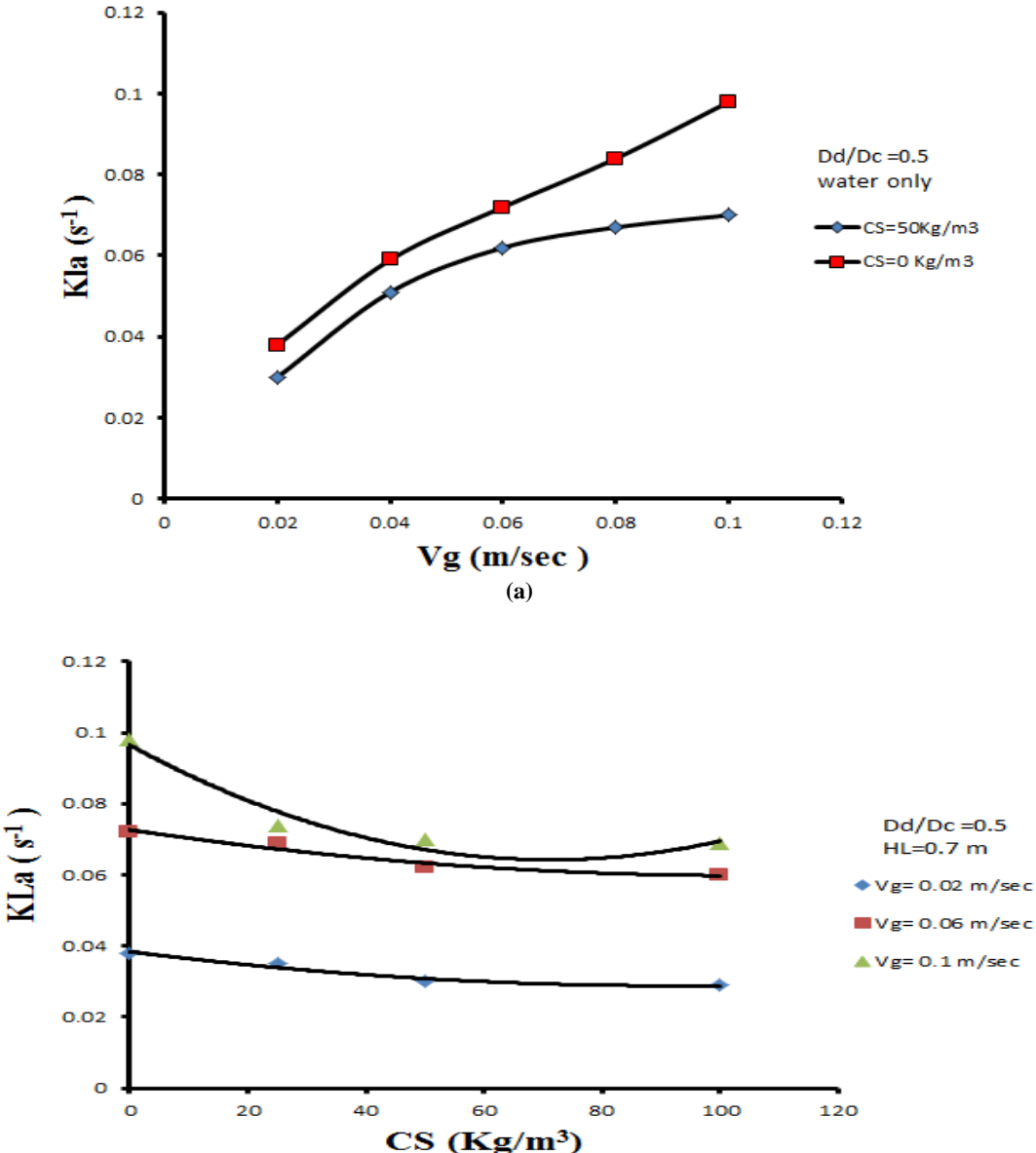


Fig.4.(a) Mass transfer coefficient versus gas velocity for water systems; (b) Mass transfer coefficient versus solid concentration for water system for various gas velocities.

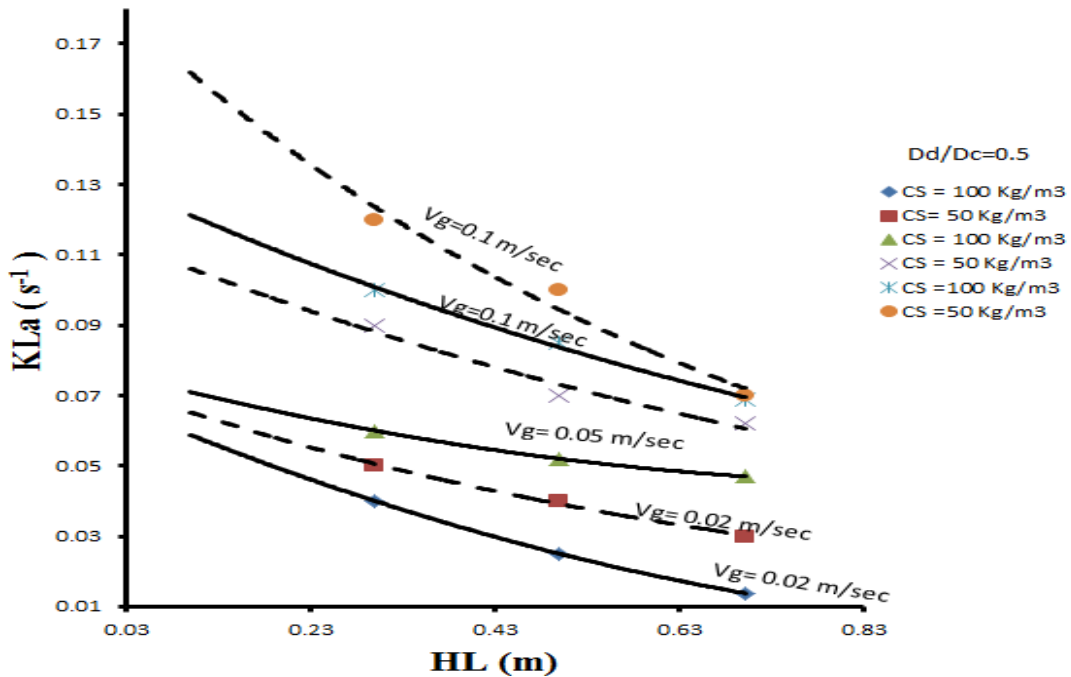
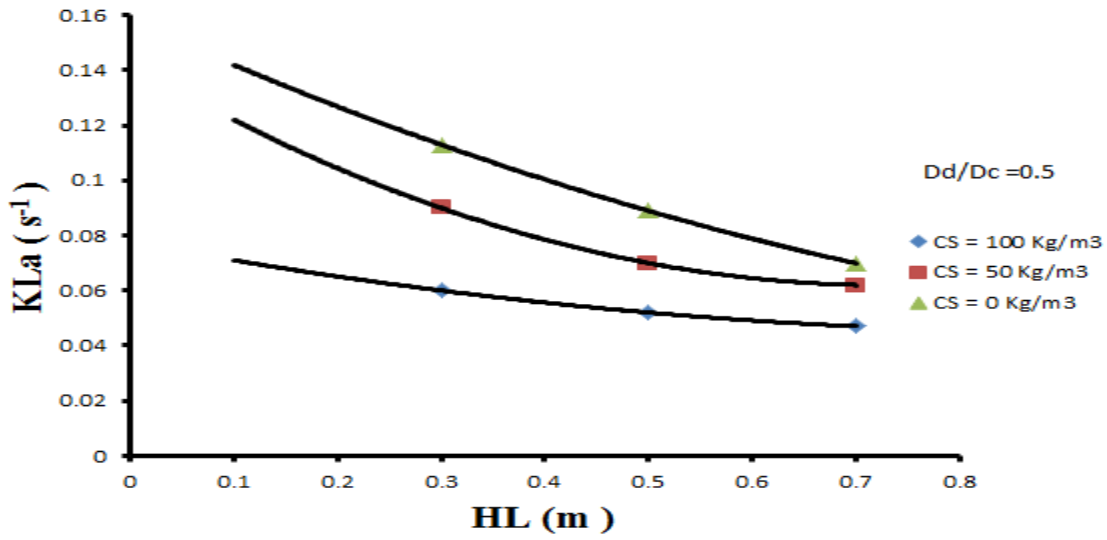


Fig. 5.(a) Mass transfer coefficient versus static liquid height for various solid concentrations at 0.06 m/sec; (b) Mass transfer coefficient versus static liquid height for various solid concentrations and for various gas velocities.

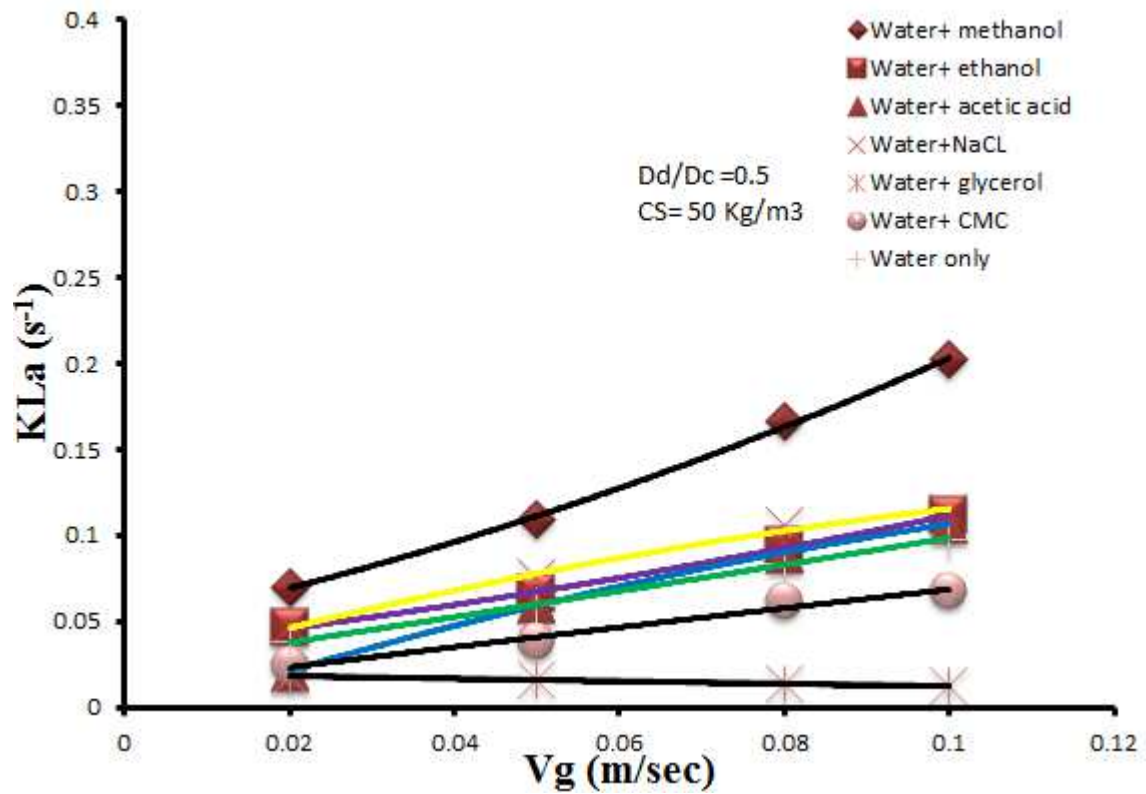


Fig. 6. Mass transfer coefficient versus gas velocity for different liquid phase system.