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## ON-LINE PHASE HOLDUP MEASUREMENT AND ANALYSIS IN FLOTATION COLUMNS

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A thesis submitted to the Faculty of Graduate Studies and Research in partial fulfillment of the requirements for the degree of Doctor of Philosophy. ċ

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February, 1994

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In the name of God, the Compassionate, the Merciful

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To the memory of MOHAMMAD HOSSEIN FAHMIDEH, symbol of resistance against aggression

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### **ABSTRAC**<sup>T</sup>

A technique for on-line simultaneous determination of gas *and* solids holdup, based on a combination of conductivity and pressure difference measurements, was developed and tested in a laboratory flotation column (10 cm in diameter and 447 cm in height). The actual holdup was determined by an isolating technique.

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It was found that the shape of dispersed particles could significantly affect the conductivity of the dispersion. For flake-shaped particles, this resulted in unacceptable estimates of solids holdup in solid-water systems when the classical models for a spherical dispersed phase (e.g., Maxwell (1892) and Bruggeman (1934)) were used. Fricke's model (1924), which accounts for particle shape, was found suitable.

A critical ratio of dispersed to continuous phase conductivity, CCR, was introduced beyond which the dispersion conductivity is insensitive to the dispersed phase conductivity. Two equations to approximate CCR for any given dispersion were proposed.

The effect of concentration, size and type of solid particles on gas holdup was investigated. The presence of solids significantly decreased the gas holdup, by up to 40% relative. Possible mechanisms to explain the effect of solid particles were evaluated based on bubble coalescence, slurry density/viscosity changes, radial profiles and wake structure effects. It was experimentally shown that bubble coalescence was not responsible for the gas holdup reduction. It was proposed that the effect of solids on reducing gas holdup is a combination of an increase in the rise velocity of bubbles due to stabilization of the bubble wake and a change in the gas holdup profile from flat to saddle-shaped.



RÉSUMÉ

## RÉSUMÉ

Une technique de détermination en continu de la fraction gazeuse et solide d'une pulpe, basée sur une mesure de conductivité et différence de pression, a été développée et vérifiée dans une colonne de flottation de laboratoire (de 10 cm de diamètre et 447 cm de hauteur). Les fractions ont été mesurées de façon précise en isolant la section centrale de la colonne.

La forme des particules dispersées peut modifier la conductivité électrique de la dispersion de façon appréciable. Par conséquent, les modèles classiques de Maxwell (1892) et Bruggeman (1934), développés pour des particules sphériques, ne permettent pas d'estimer la fraction solide de façon acceptable lorsque celle-ci est formée de particules de forme très lamellaire. Par contre, le modèle de Fricke (1924), qui prend en considération la forme des particules, est tout à fait adéquat.

La thèse propose le concept d'un rapport critique de la conductivité de la phase dispersée sur celle de la phase continue, CCR, au-dessus duquel la conductivité de la dispersion est insensible aux variations de la conductivité de la phase dispersée, et présente deux équations qui permettent de l'estimer.

L'effet de la concentration, de la taille et du type de particules solides a été étudié. La phase solide diminue la fraction gazeuse, dans certain cas de 40% (relatif). Nous avons analysé les mécanismes qui pourraient expliquer ce phénomène, soit la coalescence des bulles, un changement de la densité et viscosité de la pulpe, les profils gazeux axiaux et la structure du sillage des bulles. Nous avons démontré de façon expérimentale que la coalescence des bulles n'était pas en cause. La thèse propose que l'effet des solides est une combinaison de deux facteurs, soit une augmentation de la vitesse ascendante des bulles causée par la stabilisation de leur sillage et une déformation des profils gazeux axiaux d'une forme plane à une forme de selle.

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# **CHAPTER 1**

### **INTRODUCTION**

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## **CHAPTER 1**

### **INTRODUCTION**

#### Abstract

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The development and principal characteristics of column flotation are briefly described. The definition of phase holdup and the significance of gas holdup are addressed. The objectives of the study are presented, with a description of the thesis structure.

#### **1.1 General Introduction**

The flotation column, now often called the 'conventional' column, was first introduced in 1916 by Gahl (1916), but it was not successful until major changes in concept and design were made by Wheeler (1966) and Boutin and Wheeler (1967). Industrial applications and fundamental studies on the flotation column did not receive much attention in the western world until 1980 when the first commercial flotation column was installed at Les Mines Gaspé (Québec, Canada). However, large-scale flotation columns had been used in China since 1961 (Hu and Liu, 1988). Since 1980, numerous studies have been reported throughout the world (Wheeler, 1988; Finch and Dobby, 1990a). Publication of one book (Finch and Dobby, 1990b) and organization of two international conferences (Sastry, 1988; Agar et al., 1991) in the last few years

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demonstrate a fast growing interest in column flotation.

General modifications of the conventional flotation column have been made in recent years, e.g., the Packed column (Yang, 1988), the Jameson cell (Jameson, 1988) and the Microbubble column now called 'Microcell' (Yoon et al., 1987; Adel et al., 1991; Yoon, 1993). These reflect the results of worldwide research in the application of column to complex systems.

The present work is mainly concerned with the conventional column and the word 'column' refers to this type.

### **1.2 Column Characteristics**

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A flotation column is shown schematically in Figure 1.1. Commercial column flotation units are typically 9-15 m in height and 0.5-3.0 m in diameter. The crosssection of the column may be either square or circular: circular cross-section columns are preferred for the home-made units. The size of column is specified by either the side of a square column or the diameter of a circular column (Finch and Dobby, 1990b).

The column consists of two distinct zones: the collection zone, which is also known as the slurry, pulp or recovery zone,





and the froth or cleaning zone. These two zones are separated by an interface which defines the froth depth or interface level. The mineral slurry is introduced to the collection zone below the interface. Solid particles are contacted countercurrently with

#### CHAPTER 1 INTRODUCTION

a bubble swarm formed by the bubble generators (spargers) located near the bottom of the column. Hydrophobic particles collide with and attach to the bubbles and are carried to the froth zone. Noncollected hydrophilic and less hydrophobic particles settle and are ultimately discharged from the bottom of the column. In the froth zone, wash water is added near the top of the froth to lessen the hydraulic entrainment of fine hydrophilic particles into the concentrate (Dobby and Finch, 1985; Yianatos, 1987; Kaya and Laplante, 1989). It is common to maintain a net downward flow of water through the froth; this is called a positive bias.

The efficiency of flotation columns for fine particle flotation has been established in various plants around the world (Espinosa-Gomez et al., 1988). It has been reported that it may achieve upgrading in a single stage comparable to that in several stages of mechanical flotation machines, often with improved recoveries (Cienski and Coffin, 1981; Amelunxen and Redfearn, 1985; Egan et al., 1988). The main reason for this superior performance is attributed to the rejection of hydrophilic (gangue) particles through the wash water action (Yianatos et al., 1987).

#### **1.3 Phase Holdup Definition**

Gas introduced into a column through a sparger is dispersed as small bubbles which move upwards due to buoyancy. Gas holdup  $\epsilon_g$  is defined as the volumetric fraction of gas in the system and is usually expressed as a volume percentage. Similarly,  $\epsilon_s$  and  $\epsilon_l$  are solids and liquid holdup, respectively.

### **1.4** Significance of Gas Holdup in Column Flotation

The performance of flotation columns is significantly affected by the flow regime and gas holdup. The particle collection rate, which controls recovery, is determined by bubble size and gas rate. These factors in turn affect gas holdup. Since gas holdup reflects the prevailing bubble size and rise velocity, any system property which changes the bubble size or rise velocity affects gas holdup. Thus, measurement of holdup provides data for fundamental modelling and in-plant would permit diagnosis and eventually new control strategies to be designed. Moreover, gas holdup not only affects the collection kinetics, but also determines the phase residence time. This shows the direct influence gas holdup has on the metallurgy of the process.

### **1.5** Objectives of the Study

1. To develop a reliable technique to measure on-line gas and solids holdup simultaneously in flotation columns.

2. To evaluate the effect of solids on gas holdup.

3. To propose and test the mechanism(s) of the effect of solids on gas holdup.

#### **1.6** Structure of the Thesis

<u>.</u> . The thesis consists of seven chapters. Although the chapters are conceptually linked, they have been structured independently. The stand-alone structure of the chapters enables the reader to locate and study the topic of interest easily without need to refer to other chapters. There is, as a consequence, some overlap in the content of the chapters, but it is felt that this drawback is outweighed by the convenience of having all the relevant information together. As indicated at appropriate places some of the chapters are versions of papers published in journals.

Chapter 1 introduces the development and characteristics of the flotation column along with the definition and importance of phase holdups. The objectives and the structure of the thesis are presented.

Chapter 2 reviews models of the conductivity of dispersions. The effect of

#### CHAPTER 1 INTRODUCTION

dispersed phase shape on the conductivity of the dispersions is discussed. A critical conductivity ratio, CCR, is introduced, which is the value of the ratio of conductivity of the dispersed and continuous phases above which the dispersion conductivity is insensitive to the dispersed phase conductivity. The practical implication of the CCR on the estimation of holdup and conductivity of the dispersed phase is discussed.

Chapter 3 describes the on-line phase holdup estimation in two-phase systems using methods based on conductivity and pressure difference measurements. Fundamental concepts underlying the conductivity technique and the effect of relevant parameters, (e.g., temperature and cell constant) on the phase holdup estimation are discussed.

Chapter 4 is devoted to the estimation of holdup of flake-shaped particles in solidwater systems using conductivity. The estimates for mica and graphite in water slurries are compared with the actual volume fraction of solids as determined by an isolating technique.

Chapter 5 presents the technique and results of on-line simultaneous estimation of gas and solids holdup in a laboratory flotation column. The effect of solids concentration, solids density and distance between the pressure transducers on the relative accuracy of the estimates is addressed.

Chapter 6 discusses the effect of solid particles on gas holdup in flotation columns. The effect of concentration, size and type of solid particles is investigated. Possible mechanisms to explain the effect are evaluated based on bubble coalescence, slurry density/viscosity effects, radial gas holdup and flow profiles, and bubble wake structure.

Chapter 7 presents overall conclusions, claims for original research, and recommendations for future work.

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# **CHAPTER 2**<sup>\*</sup>

### **ELECTRICAL CONDUCTIVITY OF DISPERSIONS**

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## Nomenclature

Α	constant (Equation (2.9))
a,b,c	half axes of ellipsoids, cm
В	constant (Equation (2.10))
e	radius of spherical dispersed phase, cm
f	volumetric fraction of dispersed phase
Н	temperature gradient, °C/cm
i	current density, A/cm <sup>2</sup>
K <sub>m</sub>	conductivity ratio ( $\kappa_{\rm m}/\kappa_{\rm c}$ )
K <sub>d</sub>	conductivity ratio $(\kappa_d/\kappa_c)$
L <sub>e</sub>	matrix thermal conductivity, W/cm °C
L <sub>d</sub>	dispersed phase thermal conductivity, W/cm °C
М	effective conductivity of continuum, mS/cm
n	number of spheres (Equation (2.16))
Р	volume of added dispersed phase (Equation (2.20)), cm <sup>3</sup>
R	regression coefficient
г	radius (spherical coordinates), cm
Т	temperature, °C
t <sub>e</sub>	matrix temperature, °C
t <sub>d</sub>	dispersed phase temperature, °C
x	term in Equation (2.30)
у	radius of sphere (Equation (2.16)), cm
W	term in Equation (2.28)

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## **Greek Symbols**

α	angle (spherical coordinates), degree
β	term describing shape effect (Equation 2.23)
θ	angle (spherical coordinates), degree
κ	conductivity, mS/cm
κ <sub>c</sub>	conductivity of continuous phase, mS/cm
κ <sub>d</sub>	conductivity of dispersed phase mS/cm
κ <sub>m</sub>	conductivity of dispersion, mS/cm
φ	parameter in Equation 2.27

## Subscripts

с	continuous phase
d	dispersed phase
m	dispersion (mixture)

## **CHAPTER 2**

### ELECTRICAL CONDUCTIVITY OF DISPERSIONS

#### Abstract

Measurement of the electrical conductivity of dispersions has been used in mineral processing systems, for example, to estimate level and gas holdup (in flotation cells) and as a substitute for pH measurement for  $pH \ge 12$ . The models of dispersion conductivity which are of potential interest in these systems are reviewed. Special attention is given to models which consider volume concentration, shape and size distribution of the dispersed phase, namely the models of Maxwell, Bruggeman and Fricke. The limitations of each model are addressed considering the basic assumptions in their derivation.

It is shown that the shape of the dispersed phase affects the conductivity of dispersions if the aspect ratio is less than about 0.4; this effect is more pronounced when the dispersed phase is more conductive than the continuous phase.

A critical conductivity ratio, CCR, is introduced which is the value of the ratio of conductivity of the dispersed and continuous phases above which the dispersion conductivity is insensitive to the dispersed phase conductivity. The practical implications of the CCR on the estimation of holdup and conductivity of the dispersed phase are discussed. Two equations to approximate the CCR for any given dispersion are proposed.

### 2.1 Introduction

Unlike some properties of mixtures which can be predicted simply from averaging the properties of the pure phases, electrical conductivity exhibits a complex relationship with that of the pure phases. There have been a number of studies over the last 100 years (e.g. Maxwell, 1892; Rayleigh, 1892; Fricke, 1924; Bruggeman, 1935; Meredith and Tobias, 1962; Weissberg, 1963; Keller, 1963; Buyevich, 1974; Yianatos et al., 1985; Churchill, 1986). Measurement of the conductivity of dispersions has several potential applications of interest in mineral processing systems.

One immediate application is a method for obtaining the volume concentration of the dispersed phase (when the conductivity of the dispersed and continuous phases is known). This approach has been applied on several occasions (Lee et al., 1974; Turner, 1976; Zrymiak and Hill, 1986; Naser-El-Din et al., 1987; Uribe-Salas et al., 1993; Banisi et al., 1994) and can be considered a standard technique (Fan, 1989).

An investigation of the conductivity of a dispersion may serve as a means of estimating the conductivity and shape of the dispersed phase in the suspension (Chiew and Glandt, 1983; Furuuchi et al., 1988).

### **2.2** Theoretical Considerations

The propagation of electrical energy is a linear function of the difference in potential. This may be represented by Ohm's law which states that the current flowing in any part of a system should be a linear function of the potential gradient. This may be expressed by:

$$\mathbf{i} = -\kappa \nabla \mathbf{V} \tag{2.1}$$

where  $\nabla V$  for rectangular coordinates is:

$$\nabla V = \frac{\partial V}{\partial x} + \frac{\partial V}{\partial y} + \frac{\partial V}{\partial z}$$
(2.2)

i is the current density,  $\kappa$  is the conductivity, and  $\nabla V$  is the potential gradient at any point.

According to the law of conservation of current, the net resultant of the current entering and leaving is equal to zero, i.e.:

$$\nabla \mathbf{i} = \mathbf{0} \tag{2.3}$$

Substituting Equation 2.2 in Equation 2.1 and assuming an electrically homogenous medium (i.e.,  $\kappa$  is constant), the divergence of the gradient of the potential is zero, i.e.,:

$$\nabla \cdot \nabla \mathbf{V} = \nabla^2 \mathbf{V} = \mathbf{0}. \tag{2.4}$$

This mathematical formulation is known as the Laplace equation.

The physical consequences of the above imply that the lines of flow describing the path of the electric charge show no discontinuities --i.e. the flow is 'smooth'-- and that at all points within the system, including the boundaries, the lines of flow are orthogonal to equipotential surfaces. The streamlines converge in regions of high conductivity and diverge where the conductivity is low. Quantitatively, then, it is anticipated that a simple volumetric averaging of the conductivities of individual phases would not lead to the correct estimate of the conductivity of multiphase mixtures (Stratton, 1941; Meredith and Tobias, 1961).

### 2.3 Conductivity Models

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Theoretical and experimental investigations have been performed on the conductivity of dispersions. A comprehensive review of these studies can be found elsewhere (Meredith and Tobias, 1962; Churchill, 1986). These investigations can be
categorized in four groups:

- 1- classical solutions
- 2- ordered arrangements of dispersed phase
- 3- approximations involving no empirical parameters
- 4- relations involving empirical parameters

The focus here is on the classical solutions, particularly those relevant to mineral processing systems. Table 2.1 presents the major classical solutions with their corresponding physical considerations.

Name	Application
Maxwell (1892)	dilute dispersions of uniform spheres (f<0.2)
Rayleigh (1892)	uniformly sized spheres in a simple cubic array
Wagner (1914)	very dilute (f < 0.10) dispersions of spheres
Fricke (1924)	dispersions of spheroids
Bruggeman (1935)	wide size distribution of dispersed phase
Meredith and Tobias (1960)	dispersions of uniform spheres in a cubic lattice
Weissberg (1963)	dispersions of uniform or nonuniform spheres
Keller and Sachs (1964)	dispersions of infinitely conducting cylinders in a square array
Buyevich (1974)	dispersions of non-uniform spheres
Yianatos et al., (1985)	non-conductive dispersed phase: one model for dilute, and second one for high dispersed phase concentration (froths)

Table 2.1: Conductivity models (classical solutions) for dispersions.

Figure 2.1 compares the predicted ratio of dispersion to continuous phase conductivity ( $K_m$ ) as a function of volume fraction (f) of a non-conductive ( $\kappa_d$ =0) dispersed phase using the different models. It is apparent that for dilute dispersions (f<0.15) the values are in close agreement; however, they start to deviate for concentrated dispersions.



Figure 2.1: Comparison of conductivity models.

#### CHAPTER 2 ELECTRICAL CONDUCTIVITY OF DISPERSIONS

This chapter is principally concerned with the effect of the volume fraction, size distribution, shape and conductivity of the dispersed phase on dispersion conductivity. The three models due to Maxwell (1982), Bruggeman (1935) and Fricke (1924, 1925a, 1925b, 1953) cover these parameters (Table 2.1).

### 2.3.1 The Maxwell Model

The simplest two-phase dispersion consists of spherical particles of conductivity  $\kappa_d$  imbedded in a medium of conductivity  $\kappa_c$ . Maxwell considered a single sphere in a continuum where the field was unidirectional and linear at great distances from the sphere. By solving the Laplace equation for the potential in spherical coordinates both inside and around the sphere, and using the principle of continuity, Maxwell obtained an expression describing the variation of the potential in the continuous phase due to the presence of this single sphere. Subsequently, the conductance of dilute dispersions was obtained by regarding the dispersion itself as a sphere, and setting the effect of the dispersion on the potential in the continuous phase outside of the spherical boundary equal to that obtained for the case in which the sphere consists of a single phase only (Figure 2.2). Using this technique Maxwell obtained:

$$K_{m} = \frac{K_{d} + 2 - 2f(1 - K_{d})}{K_{d} + 2 + f(1 - K_{d})}$$
(2.5)



Figure 2.2: Schematic presentation of the Maxwell model.

The assumptions made in the derivation mean that this solution is rigorously valid only for dilute dispersions (<20%) where the fields surrounding each sphere do not perturb each other to any appreciable extent. However, in practice it has been found that conductivity data may be accurately represented by the Maxwell model for random dispersions of spheres when the volume fraction of the dispersed phase is up to about 0.5 (Marchese et al., 1992). The Maxwell model has been successfully used for gas and non-conductive solids (provided they are near-spherical) holdup estimation in a laboratory flotation column (Uribe-Salas et al., 1993; Banisi et al., 1994).

# 2.3.1.1 The Maxwell Model: Thermal Conductivity of Composites Analogy

A clear understanding of the Maxwell model seems necessary for evaluating the potential for its use in different systems: it is important to appreciate the limitations of the model which may help provide a physical foundation for more accurate interpretation of the measurements. Since the electrical approach to the derivation of the Maxwell model is mathematically complex, the thermal analogy will be used from which the Maxwell model may be derived more readily (Carslaw and Jaeger, 1959; Hasselman and Johnson, 1987).

A physical picture of heat conduction can be derived from the concept of electron drift; good conductors of heat are also good conductors of electricity. Since the conduction of electricity is postulated on the theory of free electron drift, it appears rational to ascribe heat conduction primarily to the mobility of free or valence electrons (Schneider, 1957; Ozisik, 1985).

The general equation for three-dimensional steady-state conduction without heat sources in rectangular coordinates is (Laplace equation):

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$$\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} = 0$$
(2.6)

where T is the temperature. Equation 2.6 is valid for electric conduction provided that temperature (T) is replaced by the electrical potential (E) (Holman, 1981). In spherical coordinates where (Figure 2.3):

 $x = r \sin\theta \cos\alpha$  $y = r \sin\theta \sin\alpha$  $z = r \cos\theta$ 

:



Figure 2.3: Spherical coordinate system  $(r, \alpha, \theta)$ .

the general equation at steady-state eventually becomes (Schneider, 1955):

$$\frac{1}{r}\frac{\partial^2}{\partial r^2}(rT) + \frac{1}{r^2\sin\theta}\frac{\partial}{\partial\theta}\left(\sin\theta\frac{\partial T}{\partial\theta}\right) + \frac{1}{r^2\sin^2\theta}\frac{\partial^2 T}{\partial\alpha^2} = 0$$
(2.7)

For a temperature field which has "azimuthal symmetry"  $\frac{\partial^2 T}{\partial \alpha^2} = 0$ , i.e., temperature varies only with a change in  $\theta$ , the equation reduces to:

$$\frac{1}{r}\frac{\partial^2}{\partial r^2}(rT) + \frac{1}{r^2\sin\theta}\frac{\partial}{\partial\theta}\left(\sin\theta\frac{\partial T}{\partial\theta}\right) = 0$$
(2.8)

It is assumed that particles of the dispersed phase with thermal conductivity  $L_d$  are embedded in a matrix with thermal conductivity  $L_e$ . The volume fraction of the dispersed phase is assumed to be sufficiently dilute that interactions between the local temperature fields of neighbouring particles are absent. To comply with the Maxwell model assumptions, the interface barrier resistance is assumed to be negligible. The

temperature  $t_d$  within the spherical dispersed phase of radius 'e' and the temperature  $t_c$  in the surrounding matrix are assumed to be of the general form (Carslaw and Jaeger, 1959):

$$t_d = rA\cos\theta \tag{2.9}$$

$$t_{c} = Hr\cos\theta + \frac{B}{r^{2}}\cos\theta \qquad (2.10)$$

where H is the temperature gradient at large distances away from the dispersions, A and B are constants to be solved and  $\theta$  and r are spherical coordinates,  $\theta$  being the angle between the radius vector r and the temperature gradient. These two suggested forms satisfy the Laplace equation in spherical coordinates (Equation 2.8). At r=e the following boundary conditions can be applied to Equations 2.9 and 2.10:

$$\mathbf{L}_{d}\left(\frac{\partial \mathbf{t}_{d}}{\partial \mathbf{r}}\right) = \mathbf{L}_{c}\left(\frac{\partial \mathbf{t}_{c}}{\partial \mathbf{r}}\right)$$
(2.11)

$$\mathbf{t}_{\mathrm{d}} = \mathbf{t}_{\mathrm{c}} \tag{2.12}$$

This states that the heat-transfer rate at r=e from the sphere to its surroundings and vice versa are the same. Solving for A and B and substitution in Equations 2.9 and 2.10, yields:

$$t_{d} = Hr\cos\theta \frac{3L_{c}}{2L_{c} + L_{d}}$$
(2.13)

$$t_{c} = Hr\cos\theta + \frac{1}{r^{2}}\cos\theta \frac{He^{3}(L_{c} - L_{d})}{2L_{c} + L_{d}}$$
(2.14)

#### CHAPTER 2 ELECTRICAL CONDUCTIVITY OF DISPERSIONS

The derivation of the thermal conductivity of the composite relies on assessing the cumulative effect on  $t_c$  of n spheres of radius 'e' within a large sphere of radius y, which is considered to exhibit the thermal conductivity  $L_m$  (Hasselman and Johnson, 1987). In terms of radius e, Equation 2.14 becomes:

$$t_{c} = Hr\cos\theta + \frac{1}{r^{2}}\cos\theta \frac{Hne^{3}(L_{c} - L_{d})}{2L_{c} + L_{d}}$$
(2.15)

This equation in terms of the sphere of radius y and thermal conductivity  $L_m$  is equal to:

$$t_{c} = Hr\cos\theta + \frac{1}{r^{2}}\cos\theta \frac{Hy^{3}(L_{c} - L_{m})}{2L_{c} + L_{m}}$$
(2.16)

It is apparent that  $ne^3 = fy^3$  where f is the volume fraction of the dispersed phase. From Equations 2.15 and 2.16:

$$\frac{(L_{c} - L_{m})}{2L_{c} + L_{m}} = \frac{f(L_{c} - L_{d})}{2L_{c} + L_{d}}$$
(2.17)

Replacing thermal conductivity (L) with electrical conductivity ( $\kappa$ ) and rearranging yields:

$$K_{m} = \frac{K_{d} + 2 - 2f(1 - K_{d})}{K_{d} + 2 + f(1 - K_{d})}$$
(2.18)

Equation 2.18 is identical to the Maxwell equation for the conductivity of dispersions with a dilute spherical dispersed phase.

## 2.3.2 The Bruggeman Model

To extend the Maxwell model to systems with random dispersions of spherical particles with a wide size-range, Bruggeman (1935) proposed a model which accounts for the effect of neighbouring particles (Figure 2.4). He assumed that if a relatively

large spherical particle is added to a dispersion containing smaller much particles, the disturbance of the field around the large sphere due to the small spheres may be considered negligible. On this basis, the conductivity of such a system may be evaluated by considering the surroundings of the large sphere as a continuum having a conductivity M. Since as far as the large sphere is concerned the system is dilute, the Maxwell equation may be applied. The Maxwell equation can be written as:



$$\frac{\kappa_{\rm m} - \kappa_{\rm c}}{\kappa_{\rm m} + 2\kappa_{\rm c}} = f \frac{\kappa_{\rm d} - \kappa_{\rm c}}{\kappa_{\rm d} + 2\kappa_{\rm c}}$$
(2.19)

The change in M with the volume fraction of the dispersed phase may then be expressed in differential form:

$$\frac{dM}{3M} = \frac{dP}{1+P} \left( \frac{\kappa_d - M}{\kappa_d + 2M} \right)$$
(2.20)

where dM is equal to  $\kappa_m - \kappa_c$ , P is the volume of the added particles and M is substituted for  $\kappa_c$ . Integrating with the boundary conditions  $M = \kappa_c$  as P = 0 and at the upper limit  $M = \kappa_m$  as  $P = \frac{f}{1 - f}$  gives (see Appendix 2.1)<sup>†</sup>:

$$\int_{0}^{\frac{f}{1-f}} \frac{1}{1+P} dP = \int_{\kappa_{c}}^{\kappa_{m}} \frac{\kappa_{d} + 2M}{3M(\kappa_{d} - M)} dM$$
(2.21)

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<sup>&</sup>lt;sup>†</sup>The integration limits in the original paper are incorrect, although the resulting solution is correct.

$$\ln \frac{1}{(1-f)} = \int_{\kappa_{c}}^{\kappa_{m}} \frac{\kappa_{d}}{3M(\kappa_{d}-M)} dM + \int_{\kappa_{c}}^{\kappa_{m}} \frac{2M}{3M(\kappa_{d}-M)} dM$$
(2.22)

which yields:

$$1 - f = \frac{\kappa_m - \kappa_d}{\kappa_c - \kappa_d} \sqrt[3]{\frac{\kappa_c}{\kappa_m}}$$
(2.23)

For a non-conductive dispersed phase ( $\kappa_d = 0$ ) Equation 2.23 reduces to:

$$\mathbf{K}_{\rm m} = (1 - f)^{3/2} \tag{2.24}$$

The model has been used to estimate the dispersed phase holdup and conductivity of dispersions (De La Rue and Tobias, 1959; Landauer, 1978). It is best applied to a wide size range and low concentration of dispersed phase, otherwise the physical conditions necessary to justify Bruggeman's approximation are not satisfied.

### 2.3.3 The Fricke Model

In his mathematical treatment, Fricke (1924, 1954) considered the general case of a suspension of homogeneous ellipsoids. The interaction of the suspended particles was taken into account by adding to the original field the mean value of the forces due to the charges on the suspended particles throughout the whole dispersion. Assuming ellipsoids with half axes a, b, and c in the suspension, for the case where  $a \neq b = c$  (i.e. oblate spheroid), Fricke derived the following equation:

$$\left(\frac{\kappa_{\rm m} - \kappa_{\rm c}}{\kappa_{\rm d} - \kappa_{\rm m}}\right) \left(\frac{\kappa_{\rm d}}{\kappa_{\rm c}} - 1\right) = \frac{\beta f}{1 - f}$$
(2.25)

where

$$\beta = \frac{1}{3} \left[ \frac{2}{1 + \left(\frac{\kappa_d}{\kappa_c} - 1\right) \frac{1}{2} W} + \frac{1}{1 + \left(\frac{\kappa_d}{\kappa_c} - 1\right) (1 - W)} \right] \left(\frac{\kappa_d}{\kappa_c} - 1\right)$$
(2.26)

and

.

$$W(a < b) = \frac{\left(\varphi - \frac{1}{2}\sin 2\varphi\right)}{\sin^3 \varphi} \cos \varphi , \quad \cos \varphi = \frac{a}{b}$$
(2.27)

$$W(a>b) = \frac{1}{\sin^2 \varphi'} - \frac{1}{2} \frac{\cos^2 \varphi'}{\sin^3 \varphi'} \log \left( \frac{1 + \sin \varphi'}{1 - \sin \varphi'} \right), \quad \cos \varphi' = \frac{b}{a}$$
(2.28)

The effect of the geometry of the dispersed phase is fully characterized by  $\beta$  (Equation 2.25). Figure 2.5 illustrates the variation of  $\beta$  for various ratios of the conductivity of the dispersed and continuous phases. It is apparent that the effect of the shape of the dispersed phase on  $\beta$  is generally more pronounced when the dispersed phase is more conductive than the continuous phase. Fricke's equation can be rearranged to obtain one analogous to that of Maxwell:

$$\frac{\left(\frac{\kappa_{\rm m}}{\kappa_{\rm c}}\right) - 1}{\left(\frac{\kappa_{\rm m}}{\kappa_{\rm c}}\right) + x} = f \frac{\left(\frac{\kappa_{\rm d}}{\kappa_{\rm c}}\right) - 1}{\left(\frac{\kappa_{\rm d}}{\kappa_{\rm c}}\right) + x}$$
(2.29)



Figure 2.5: Variation of  $\beta$  for various ratios of conductivity of dispersed and continuous phases ( $a \neq b = c$ ).

where

$$\mathbf{x} = -\frac{\left(\frac{\kappa_{d}}{\kappa_{c}} - 1\right) - \left(\frac{\kappa_{d}}{\kappa_{c}}\right)\boldsymbol{\beta}}{\left(\frac{\kappa_{d}}{\kappa_{c}} - 1\right) - \boldsymbol{\beta}}$$
(2.30)

For spherical particles (a=b=c), x=2 and Fricke's equation reduces to Maxwell's.

Figure 2.6 illustrates the effect of shape on the ratio of dispersion conductivity to dispersed phase conductivity ( $K_m$ ) estimated using Fricke's model for a dispersed phase fraction of 0.15. The effect on  $K_m$  is significant when a/b <0.40 for the  $\kappa_d/\kappa_c > 1$  case and when a/b <0.25 for the  $\kappa_d/\kappa_c < 1$  case.

As an illustration, if perfect spheres are assumed for a dispersed phase where it actually has a shape factor of 0.40, the error in the holdup estimation is about 1.3% and 2.9% for non-conductive and conductive dispersed phases, respectively. It may be concluded that except for a "flaky" dispersed phase (such as mica or graphite) the effect of the shape of the dispersed phase is not significant.

# 2.4 Critical Conductivity Ratio, CCR $\kappa_d/\kappa_c > 1$ case:

Figure 2.7 illustrates the sensitivity of the dispersion conductivity (using Maxwell's model) to a change in the dispersed phase conductivity for various phase holdups (f). It is apparent that there is a limit beyond which any further increase in the conductivity of the dispersed phase does not result in a noticeable change in the conductivity of the dispersion. This limiting value of dispersed phase conductivity relative to the continuous phase conductivity will be referred to as the critical conductivity ratio (CCR). The CCR is a function of the volume fraction of the dispersed phase.

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Figure 2.6: Effect of shape of dispersed phase on conductivity ratio using Fricke's model (Equation 2.25).



Figure 2.7: Sensitivity of dispersion conductivity (using Maxwell's model) to variation in conductivity of dispersed phase  $(\frac{\kappa_a}{\kappa_c} > 1)$ .

#### CHAPTER 2 ELECTRICAL CONDUCTIVITY OF DISPERSIONS

To try to define this limitation, the values of  $K_m$  and  $K_d$  were calculated using the Maxwell model for different volume fractions of the dispersed phase. The CCR was obtained using a numerical method in which  $K_d$  was increased in steps of 5 up to a value where a further increase did not affect  $K_m$  ( $\Delta K_m/K_m < 0.002$  for a given  $\Delta K_d$ ). An exponential curve was fitted to these CCR values (R = 0.998) (Figure 2.7): this equation approximates the CCR for a given  $K_m$ .

The CCR for any given dispersion depends only upon the volume fraction of the dispersed phase. To characterize the nature of the relationship between the CCR and volume fraction of the dispersed phase (applying the Maxwell model and a numerical method), the CCR as a function of phase holdup was also computed (Figure 2.8). It is apparent that the relationship between the CCR and f is linear (R=0.998). This simple function provides the CCR for any given f and vice versa. For example, the CCR for volume fractions of 0.2 and 0.4 is 36.8 and 72.6, respectively.

### $\kappa_{\rm d}/\kappa_{\rm c} < 1$ case:

For further analysis of the sensitivity of the dispersion conductivity to the conductivity of dispersed phase Figure 2.9 was prepared. When  $\kappa_d/\kappa_c$  varies from 2 to 4, a significant change in the conductivity of the dispersion is observed; however, a change from 40 to 1000 only becomes notable at higher holdups. In the case of  $\kappa_d/\kappa_c < 1$ , when  $\kappa_d/\kappa_c$  changes from 0.025 to 0.001 (i.e., the reciprocal of the 40 and 1000 values just considered), there is no difference in the conductivity of the dispersion at any holdup. In other words,  $\kappa_d/\kappa_c \sim 0.025$  represents the CCR for the  $\kappa_d/\kappa_c < 1$  situation.

#### **Implications:**

The principal implications are that, beyond the CCR. measurement of dispersion conductivity cannot be used to estimate dispersed phase conductivity, but it is an advantage in holdup estimation, since there is no need for accurate knowledge of the dispersed phase conductivity.

There is little experimental data to illustrate these implications. One such set is due to Meredith and Tobias (1961) who conducted a series of experiments on emulsions (in the range 0 < f < 0.5 and  $0.172 < \kappa_d/\kappa_c < 101$ ) to investigate the accuracy of predicted



Figure 2.8: Computed CCR for any given phase holdup using Maxwell's model.

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Figure 2.9: Response of dispersion conductivity to change in dispersed phase conductivity (using Maxwell's model).

. . dispersion conductivity. The emulsions were prepared from water and various oils of different  $\kappa_d$  with propylene carbonate as a stabilizer. For the various dispersed phases, the authors studied the accuracy of different models in estimation of the conductivity of the dispersions: they concluded that their modified version of the Maxwell model was accurate. A set of data generated based on their value of  $\kappa_d/\kappa_c = 101$  is plotted (Figure 2.10) along with the predicted results for other values of  $\kappa_d/\kappa_c$  (50, 500 and 1000). It is apparent that there is no significant difference in prediction among the various values for the conductivity ratio. This stems from the fact that all the conductivity ratios are beyond the CCR which makes the method insensitive to changes in the conductivity of the dispersed phase. In view of the above considerations, Meredith and Tobias (1961) could have obtained the same results with an arbitrary choice of  $\kappa_d/\kappa_c$  (provided  $\kappa_d/\kappa_c > CCR$ ). It appears that they did not notice that  $\kappa_d$  does not affect  $\kappa_m$  above a certain value (i.e.,  $\kappa_d/\kappa_c > CCR$ ).

#### CCR and Fricke's Model:

The concept of a critical conductivity ratio (CCR) remains valid when the dispersed phase is not spherical; Fricke's model was used to investigate the sensitivity to a change in the conductivity of the dispersed phase (Figure 2.11). For illustration purposes the dispersed phase shape factor (a/b) was taken as 0.20. It is apparent the result is similar to that using Maxwell's model (Figure 2.9).

### 2.5 Conclusions

1- Derivation of the Maxwell model using the thermal conductivity of composites analogy re-emphasizes that the model theoretically is only valid for dilute (f < 0.20) dispersions.

2- Following the mathematical derivation reveals that the Bruggeman model is invalid when the size distribution of the dispersed phase becomes too narrow.

3- The shape of the dispersed phase can be important in determining dispersion conductivity: from Fricke's model, the effect is important when the ratio of thickness



Figure 2.10: Response of dispersion conductivity to change in dispersed phase conductivity above CCR (based on original work of Meredith and Tobias: Maxwell's model was used).



Figure 2.11: Response of dispersion conductivity to change in dispersed phase conductivity for non-spherical dispersed phase (using Fricke's model).

4- There is a critical value of  $\kappa_d/\kappa_c$  for any dispersion beyond which the conductivity of the dispersion is not sensitive to changes in  $\kappa_d/\kappa_c$ : this is referred to here as a critical conductivity ratio, CCR. This implies, for example, that the use of dispersion conductivity to estimate the conductivity of the dispersed phase will result in erroneous conclusions if the CCR is not considered.

5- To approximate the CCR for the  $\kappa_d/\kappa_c > 1$  case, an equation based upon the dispersed phase and dispersion conductivity is proposed. For any given dispersion the CCR is a function of the dispersed phase holdup (f). It was found that relationship between the CCR and f is linear. This function provides a second approach to estimating the CCR. For the  $\kappa_d/\kappa_c < 1$  case the CCR for any given dispersed phase holdup was approximately 0.025.

6- The insensitivity of the conductivity method to the change in the conductivity of the dispersed phase beyond the CCR facilitates phase holdup estimation using the conductivity method. Since, beyond the CCR, the phase holdup is independent of the conductivity of the dispersed phase, in such systems only an approximate value of  $\kappa_d/\kappa_c$  is sufficient to estimate the phase holdup.

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# Appendix 2.1

# Integral limits of Bruggeman's model

Let P<sup>f</sup> be the total volume of the added dispersed phase then:

$$\mathbf{f} = \frac{\mathbf{P}^{f}}{\mathbf{P}^{f} + 1} \tag{2.31}$$

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$$\mathbf{P}^{f} = \frac{\mathbf{f}}{1 - \mathbf{f}} \tag{2.32}$$

When P=0 then  $M = \kappa_c$  and when  $P = f/(1-f) = P^f$  then  $M = \kappa_m$ . The integral limits then are P=0 and P= f/1-f.

# **CHAPTER 3**\*

# ON-LINE PHASE HOLDUP ESTIMATION IN TWO-PHASE SYSTEMS

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# Nomenclature

А	cross-sectional area, cm <sup>2</sup>
A <sub>c</sub>	cross-sectional area of the column, cm <sup>2</sup>
A,	effective cross-sectional area at holdup $\epsilon$ , cm <sup>2</sup>
С	solids reporting to concentrate, g/s
C <sub>p</sub>	specific heat of water, J g <sup>-1</sup> (°C) <sup>-1</sup>
D	column diameter, m
Е	electric intensity, V/cm
F	cell constant, cm
f	volume fraction of dispersed phase
g	gravitational acceleration, cm/s <sup>2</sup>
h	gas-free liquid height, cm
1	current, A
J <sub>g</sub>	superficial gas velocity, cm/s
J	current density, A/cm <sup>2</sup>
K <sub>d</sub>	ratio of conductivity of dispersion and continuous phase $(\kappa_d/\kappa_c)$
K <sub>m</sub>	ratio of conductivity of dispersed phase and continuous phase
	$(\kappa_{\rm m}/\kappa_{\rm c})$
	length, cm
L	distance between the pressure transducers, cm
L,	effective length between electrodes, cm
L <sub>u</sub>	unit length, m
m	mass of water, g
P <sub>A</sub>	pressure at point A, g/s <sup>2</sup> /cm
P <sub>B</sub>	pressure at point B, g/s <sup>2</sup> /cm
q	thermal convection coefficient, $J m^{-2} s^{-1}$
Q	total heat, J
R	resistance, Ohm

# CHAPTER 3 ON-LINE PHASE HOLDUP ESTIMATION IN TWO-PHASE SYSTEMS ... 3-iv

r	fractional drop back
t	time, s
Т	temperature, °C
V	potential difference, V

# **Greek Symbols**

α	coefficient of variation of temperature (Equation 3.26), $^{\circ}C^{-1}$
ΔΤ	temperature difference, °C
γ	relative conductivity, dimensionless
γκ	relative conductance, dimensionless
ε <sub>d</sub>	dispersed phase holdup
$\epsilon_{\rm g}$	gas holdup
$\epsilon_1$	liquid holdup
€ <sub>s</sub>	solids holdup
κ	electrical conductivity, mS/cm
κ <sub>c</sub>	conductivity of continuous phase, mS/cm
ĸ	conductivity of dispersed phase, mS/cm
κ <sub>i-g</sub>	conductivity of liquid-gas system, mS/cm
K <sub>s-i</sub>	conductivity of solid-liquid system, mS/cm
K <sub>s-l-g</sub>	conductivity of solid-liquid-gas system, mS/cm
ρ	resistivity, Ohm-cm
$ ho_{ m b}$	bubble-particle aggregate density, g/cm <sup>3</sup>
$ ho_{ m g}$	density of gas, g/cm <sup>3</sup>
$ ho_1$	density of water, g/cm <sup>3</sup>
$ ho_{ m s}$	density of solid, g/cm <sup>3</sup>
$ ho_{ m sl}$	density of slurry, g/cm <sup>3</sup>
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# **CHAPTER 3**

# ON-LINE PHASE HOLDUP ESTIMATION IN TWO-PHASE SYSTEMS

### Abstract

The available methods of holdup estimation in two-phase systems are reviewed. Special attention is given to two methods, one based on conductivity and the second based on pressure difference measurements.

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These two methods were used to estimate phase holdup in two phase systems (solid-water and gas-water) in a laboratory flotation column (10 cm in diameter and 447 cm in height). Fundamental concepts underlying the conductivity technique and the effect of relevant parameters, e.g., temperature and cell constant on the phase holdup estimation, are discussed. Holdup estimates from both methods were in good agreement with the actual holdup values as determined by an isolating technique. Provided that the necessary precautions (in particular, calibration of the pressure transducers) are taken, holdup estimates using the pressure difference and conductivity-based techniques are comparable.

### 3.1 Introduction

The relative magnitude of the phase holdups (i.e., volumetric fraction of each phase) affects the flow regime and performance of a reactor (e.g., flotation column). This has provided the prime stimulus for investigations which have been conducted in two- (solid-liquid or gas-liquid) and three- (solid-liquid-gas) phase systems (e.g., Fan, 1989). Although there exist several techniques for holdup determination particularly in two-phase systems, on-line holdup estimation methods are scarce.

Since a combination of the techniques used in the two-phase systems is planned for phase holdup estimation in the three-phase flotation system, it is desirable to understand the basics and to evaluate the techniques first in two-phase systems. In this chapter, the techniques are reviewed and the effect of various parameters on the accuracy of the estimates is discussed.

# 3.2 Gas Holdup: Measurement Techniques

Gas holdup can be measured in various ways. Some of these techniques have been described by Finch and Dobby (1990). Based on the type of gas holdup measurements, these techniques can be divided in two groups:

a) Techniques which measure the gas holdup for the whole vessel, often called 'overall' gas holdup, such as the bed expansion and the pressure difference techniques (Figures 3.1(a) and 3.1(b)); and

b) Techniques which measure the gas holdup over a given section of the vessel, often called 'local' gas holdup, such as the conductivity-based and  $\gamma$ -ray methods (Figure 3.1(c)).



Figure 3.1: Techniques of gas holdup measurement.

### 3.2.1 Overall Gas Holdup Measurement

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The Bed Expansion Technique: this technique provides the overall gas holdup by measuring the bed expansion ( $\Delta$ h) due to introduction of the gas and the gas-free liquid height (h):

$$\epsilon_{g} = \frac{\Delta h}{\Delta h + h}.$$
(3.1)

This technique has limited applications in flotation columns because detection of the interface between liquid and gas in the presence of froth is difficult. However, this technique is commonly used in chemical engineering (Fan, 1989). This approach is mostly suited for batch systems when gas rate fluctuation is not significant.

The Pressure Difference Method: referring to Figure 3.1(b), the pressure difference between two points (B and A) is given by (assuming wall friction is negligible):

$$\frac{\mathbf{P}_{B} - \mathbf{P}_{A}}{L} = g(\rho_{g}\epsilon_{g} + \rho_{1}\epsilon_{1} + \rho_{s}\epsilon_{s})$$
(3.2)

Where:

 $\rho_i$ : density of i (gas, liquid, solid)

 $\epsilon_i$ : holdup of i

In the case of solid-water systems where  $\epsilon_g = 0$ , Equation 3.2 reduces to:

$$\frac{\mathbf{P}_{\mathrm{B}} - \mathbf{P}_{\mathrm{A}}}{\mathrm{L}} = g(\rho_{1}\epsilon_{1} + \rho_{\mathrm{s}}\epsilon_{\mathrm{s}}). \tag{3.3}$$

For the gas-water systems where  $\epsilon_s = 0$  and  $\rho_g = 0$ , the pressure difference is simply given by:

$$\frac{\mathbf{P}_{\mathrm{B}} - \mathbf{P}_{\mathrm{A}}}{L} = \rho_{\mathrm{I}} \epsilon_{\mathrm{I}} g \tag{3.4}$$

hence, gas holdup is equal to:

$$\epsilon_{g} = 1 - \frac{P_{B} - P_{A}}{L \rho_{1} g}.$$
(3.5)

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Other Approaches: in the presence of hydrophobic particles the gas holdup can be expressed in terms of the slurry and bubble-particle aggregate densities ( $\rho_{sl}$  and  $\rho_{b}$ ):

$$\epsilon_{g} = \frac{\rho_{sl}g L - (P_{B} - P_{A})}{(\rho_{sl} - \rho_{b})gL}.$$
(3.6)

To obtain the gas holdup from this approach,  $\rho_{sl}$  and  $\rho_b$  must be known. Since the measurement of bubble-particle aggregate density is not a trivial task, in practice it is assumed that the bubbles are lightly loaded ( $\rho_b \approx 0$ ) (Uribe-Salas, 1991). In recent work, Yianatos and Levy (1989) proposed a method to estimate the average density of bubble-particles aggregates. Using the concept of drop back, which is the fraction of solids

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entering the froth that is rejected back into the collection zone (Falutsu and Dobby, 1989), they defined:

$$\rho_{\rm b} = \frac{C}{(1-r)J_{\rm g}A_{\rm c}} \tag{3.7}$$

where C is the solids flowrate reporting to concentrate, r the drop back fraction,  $J_g$  the superficial gas velocity, and  $A_c$  the column cross-sectional area. However, calculation of the drop back remains a challenge.

Vasalos et al. (1977, 1982) proposed a technique to measure the overall gas holdup. They used a radioactive gaseous tracer and measured the residence time distribution of the gas phase from which they obtained the mean gas holdup (and other information on gas mixing).

#### 3.2.2 Local Gas Holdup Measurement

These techniques are intended to measure gas holdup over the section detected by the sensors. Techniques based on electrical conductivity are very common (Serizawa et al., 1975; Fan, 1989; Uribe-Salas et al., 1991).

Punctual electrical resistivity probes have been used to measure local gas holdup, bubble size and bubble velocity (Nassos, 1963; Burgess and Calderbank, 1975; Castillejos, 1986; Fukuma et al., 1987).

The use of isokinetic sampling methods for local gas holdup measurements have been reported by Serizawa et al. (1975) and Chen et al. (1983). To determine the local holdup by this method, a sample of the dispersion is collected at the point of sampling.

An optical fibre probe has also been used to measure gas holdup in a liquid-gas

#### CHAPTER 3 ON-LINF PHASE HOLDUP ESTIMATION IN TWO-PHASE SYSTEMS ... 3-6

system (Wachi et al. 1987). This method is based upon the distinct difference between the refractive index of gas and liquid. Phase detection measurement is carried out at the surface of the probe tip. This surface is shaped to reflect incoming light internally when it is surrounded by gas and to refract light when surrounded by liquid (Lee et al., 1984). The time fraction of the reflected light will then provide the local holdup (Vince et al., 1982; Ishida and Tanaka, 1982; Hu et al., 1985; Lee and De Lasa, 1986; De Lasa and Lee, 1987).

For liquid-gas systems, methods based on the attenuation of  $\beta$ -rays (Nassos, 1963) and  $\gamma$ -rays (Lee and Worthington, 1974; Lockett and Kirkpatrick, 1975) have been proposed. These methods utilize a radioactive source and a detector. The intensity of the attenuated beam passing through the liquid-gas system is a function of the volumetric fraction of gas (Uribe-Salas, 1991).

Other techniques based on ultrasonic pulse transmission (Stravs and Von Stockar, 1985) and scattering of a laser beam (Soto, 1989) are used in liquid-gas systems to calculate interfacial surface area which in conjunction with bubble diameter determines the gas holdup.

Unlike most methods, the conductivity-based methods do not require calibration. This feature makes these methods very attractive for industrial applications. Since in this study conductivity measurements were used extensively, a brief review of the concept and the method of measurement is desirable.

### **3.3 Electrical Conductivity: Fundamentals**

### 3.3.1 Definition

Electrical conductivity is a measure of the ability of a material to conduct an electrical current. Two types of conductors are common: electronic (metallic) and

#### CHAPTER 3 ON-LINE PHASE HOLDUP ESTIMATION IN TWO-PHASE SYSTEMS ... 3-7

electrolytic. Electronic conduction involves valence electrons, which are relatively free to move from atom to atom. Electrolytic or ionic conduction occurs mainly with salts in the solid, liquid, or dissolved state and involves the migration of ions rather than electrons (Miller et al., 1988). In other words, in electrolytic conductors the carriers of electrical energy are charged particles of atomic or molecular size, and a transfer of matter takes place, whereas in metallic conductors, no matter is transferred, and current flow involves electrons only.

Compared to chemical elements and compounds, metal alloys, minerals and electrolytes are electrically complex and their conductivities depend upon the chemical composition and the physical microstructure.

### 3.3.2 Basic Concepts: Electrical Resistivity and Conductivity

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The current density J in a conductor depends on the electric intensity E, and on the nature of the conductor. The dependence of J on E can be quite complex, but for some materials, especially metals and electrolytes, it can be represented quite well by a direct proportionality. For such materials the ratio of E to J is constant which is termed resistivity:

$$\rho = \frac{E}{J}.$$
 (3.8)

The greater the resistivity, the greater the field needed to establish a given current density, or the smaller the current density for a given field.

The discovery that  $\rho$  is a constant for a metallic conductor at constant temperature was made by G.S. Ohm and is called Ohm's law (Sears et al., 1979). A material obeying Ohm's law is called an ohmic conductor or a linear conductor.

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It is often difficult to measure E and J directly, and it is useful to put Equation 3.8 in a form involving readily measured quantities such as total current and potential difference. Figure 3.2 illustrates a conductor with uniform cross-section area A and length *l*. Assuming a constant current density over a cross section, and a uniform



electrical field along the length of the conductor, the total current I is given by:

$$\mathbf{I} = \mathbf{J}\mathbf{A} \tag{3.9}$$

and the potential difference V between the ends is:

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$$V = E1.$$
 (3.10)

Solving these equations for J and E yields:

$$V = \rho \frac{1}{A} I. \tag{3.11}$$

Thus the total current is proportional to the potential difference. The quantity  $\rho l/A$  for a particular specimen is called its resistance R. The SI unit of resistance is the Ohm ( $\Omega$ ); hence, the unit of resistivity is Ohm meter ( $\Omega$ m).

Conductivity ( $\kappa$ ) is defined as the reciprocal of the resistivity

$$\kappa = \frac{1}{\rho}.$$
 (3.12)

Electrical conductivity may be thought of as the conductance (reciprocal of resistance) of a cube of 1 cm edge, assuming the current to be perpendicular to opposite faces of the cube (Braunstein and Robbins, 1971). The unit  $\Omega^{-1}$ , the unit of conductance, in the SI system is called the siemens (S):  $1 \text{ S} \equiv 1 \Omega^{-1}$ . Thus, the SI unit of conductivity is S m<sup>-1</sup>.

Electrical conductivity has various equivalent terms: conductivity (Rayleigh, 1892; Gilmont and Walton, 1956; Wagner, 1962; Atkins, 1982; Levine, 1988; Uribe-Salas 1991), specific conductance (Condon, 1967; Andrews, 1970; Barrow, 1973), and specific conductivity (Kasper, 1940; Adamson, 1979). In the present work, the term conductivity and the Greek letter kappa,  $\kappa$ , and the cgs unit S/cm (or the submultiple mS/cm) will be used.

# 3.3.3 Measurement of Conductivity

Several phenomena occur in aqueous solutions under a potential field that do not occur in metallic conductors. These necessitate a specific operational definition for conductivity in aqueous solutions. Ions in solution are surrounded by a sphere of oppositely charged ions and water. When a potential field is imposed on a solution, the migration of the central ion deforms the cosphere of water and oppositely charged ions. This effect may be viewed as a drag on the migrating ion. Both cations and anions experience similar effects, but in opposite directions. Under a constant potential field, the cations and anions accumulate at the electrodes until a solution potential is achieved which balances the applied potential. This phenomenon is called polarization. Polarization could result in termination of transfer of charge in solution, hence an absence of electrical current. The use of alternating potential polarity (alternating current) prevents polarization and reduces the drag effect on the ions. Primarily for these reasons, all standard devices that measure conductivity use alternating current, commonly with a frequency of 1 kHz (Miller, et al., 1988).

Using the cell shown in Figure 3.3, the resistance of the electrolyte is determined by applying Ohm's law (V=IR). Once R is known, the conductivity  $(1/\rho)$  can be calculated from:

$$\kappa = \frac{1}{AR}.$$
(3.13)

Since 1/R is the conductance (K), Equation 3.13 then becomes:

$$\mathbf{K} = \kappa \frac{\mathbf{A}}{\mathbf{l}}.$$

A/l is called the cell constant which is defined by the geometry of the cell. Xu (1991) has studied the effect of cell geometry on the cell constant.

It is not always convenient or practical to design a measurement cell to exact dimensions and a cubic shape; therefore, a cell constant (F) relating the measured conductivity to the conductivity of an accepted standard is determined for a cell. The cell constant is readily determined from a resistance measurement on a Wheatstone bridge or other device, using:

$$\kappa_{\rm m} = F \kappa_{\rm s} \tag{3.15}$$

Figure 3.3: One form of an ideal electrode for conductivity measurement.

where  $\kappa_m$  and  $\kappa_s$  are the measured and known conductivity of the standard, respectively (Daniels and Alberty, 1967). The primary standard for determining cell constants is mercury, but because mercury has a high conductivity, aqueous potassium chloride solutions are commonly used as secondary standards (Harned and Owen, 1964). These methods are suited for small cells.

## 3.3.4 Electrical Conductivity of Two-Phase Systems

The electrical conductivity of two-phase dispersions (a continuous phase plus one dispersed phase) has been referred to as 'effective conductivity' (Hashin, 1968; Neale and Nader, 1973; Fan et al., 1985; Uribe-Salas, 1991), 'apparent conductivity' (Turner, 1976) or simply 'conductivity' (Linneweber and Blass, 1983). In the present study, to distinguish the conductivity measured for a multiphase system from that for a single phase, the term effective conductivity or conductivity with appropriate subindices indicating the type of system will be used. For example,  $\kappa_{g-l-s}$  refers to the conductivity of a gas-liquid-solid system.

Numerous experimental and theoretical studies have been conducted concerning the problem of evaluating the effective conductivity of dispersions. The classical solution was derived by Maxwell (1892):

$$K_{m} = \frac{K_{d} + 2 - 2f(1 - K_{d})}{K_{d} + 2 + f(1 - K_{d})}$$
(3.16)

where

 $K_{\rm m} = \kappa_{\rm m}/\kappa_{\rm c}$   $K_{\rm d} = \kappa_{\rm d}/\kappa_{\rm c}$   $\kappa_{\rm m} : \text{effective conductivity of dispersion}$   $\kappa_{\rm c} : \text{conductivity of continuous phase}$   $\kappa_{\rm d} : \text{conductivity of dispersed phase}$ 

f : volume fraction of dispersed phase.

Hashin (1968) has reported an expression equivalent to Maxwell's equation for the electrical and thermal conductivity properties of solid heterogeneous media. An excellent agreement between experimental data and Maxwell's equation was obtained by Neale and Nader (1973) for glass spheres (less than 0.22 mm diameter) in an aqueous

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solution. Maxwell's model has also been successfully used by Turner (1976) for measuring holdup in liquid-fluidized beds of spheres: a range of solid particle diameters (0.15-1.0 mm) and conductivities  $(0-0.03 \text{ S cm}^{-1})$  were used.

In the case of a non-conductive dispersed phase ( $\kappa_d = 0$ ) Maxwell's model reduces to:

$$K_{\rm m} = \frac{1 - f}{1 + 0.5f} \tag{3.17}$$

rearranging yields:

$$f = \frac{1 - K_m}{1 + 0.5 K_m}.$$
 (3.18)

In gas-water systems, where the conductivity of gas compared to that of water can be considered to be zero, gas holdup may be determined using Equation 3.18. Xu (1991) and Marchese (1991) have reported excellent agreement between the experimentally measured gas holdup and the values estimated using Equation 3.18.

Since Maxwell's work was published, several attempts have been made to represent the conductivity of dispersions of random-sized particles. One such relation due to Bruggeman (1935) has been applied repeatedly (De La Rue and Tobias (1959); Naser-El-Din et al., (1987); Uribe-Salas (1991); Marchese et al. (1991)):

$$f = 1 - \frac{K_m - K_d}{K_m^{1/3} (1 - K_d)}$$
(3.19)

where:

$$K_{\rm m} = \kappa_{\rm m}/\kappa_{\rm c}$$
$$K_{\rm d} = \kappa_{\rm d}/\kappa_{\rm c}$$

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 $\kappa_{\rm m}$  : effective conductivity of dispersion

 $\kappa_{\rm c}$ : conductivity of continuous phase

 $\kappa_d$ : conductivity of dispersed phase

f : volume fraction of dispersed phase.

The physical picture corresponding to this model requires an infinite range of particle size within the dispersion (Figure 3.4). Bruggeman used the first two terms of the Taylor expansion of Maxwell's model to evaluate in successive steps the effective conductivities obtained after the addition of each particle. Hence, from the point of view of each newly added particle, the existing suspension must be regarded as very dilute, otherwise the use of Maxwell's equation would not be justified. However,

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some researchers (Naser-El-Din et al., 1987; Uribe-Salas, 1991) have failed to notice this and stated that Bruggeman's equation is valid for any concentration. For a non-conductive dispersed phase ( $\kappa_d$ =0), Equation 3.19 becomes:

$$f = 1 - K_m^{2/3}.$$
 (3.20)

De La Rue and Tobias (1959) reported good agreement of data with Equation 3.20 on suspensions which consisted of nonconducting spheres that varied in size by three orders of magnitude. However, they found that when narrow size ranges were used the data fell between the curves representing Maxwell's and Bruggeman's models. In an attempt to take into account the interaction of fields around particles of the dispersed phase, the authors proposed a new equation based on Maxwell's equation, namely:

$$\kappa_{1-s} = \kappa_1 (1-\epsilon_s)^m \tag{3.21}$$

where m = 1.35 to 1.56 depending on the 'irregularity' and size distribution of particles.

Buyevich (1974) derived an equation when  $\kappa_d/\kappa_c \ll 1$ :

$$\frac{\kappa_{l-s}}{\kappa_l} = \frac{24 - 17\epsilon_d}{24 + 19\epsilon_d}.$$
(3.22)

This equation was originally derived for evaluation of the effective thermal conductivity of granular materials. Equation 3.22 takes into account the effects of paired interaction between two dispersed particles and the size distribution of the dispersed particles. Kato et. al (1981) found that for fluidized beds of uniform glass beads of 0.42, 0.66, 1.2, and 2.2 mm, Equation 3.22 fitted the data closely. A similar behaviour was reported by Tang and Fan (1987) and Naser-El-Din et al. (1987). For a gas-liquid system, the Maxwell and Buyevich equations predicted very accurately the relationship between the effective conductivity and the gas volume fraction up to 0.13 and above 0.25, respectively (Tang and Fan, 1987).

For gas-water systems, assuming the conductivity of the dispersion was still the one of the continuous phase, Yianatos et al. (1985) derived a geometrical model to relate the cell constant with gas holdup. Based on their model, for the collection zone of a flotation column, gas holdup is estimated by:

$$\epsilon_{g} = \frac{1 - \gamma_{K}}{1 + 0.55 \gamma_{K}} \tag{3.23}$$

where  $\gamma_{\rm K}$  is the relative conductance (K<sub>1-g</sub>/K<sub>1</sub>). This model assumes that the conductivity used for the aqueous solution and for the dispersion<sup>3</sup> is the same, but the cell constant changes from A/L to A<sub>e</sub>/L<sub>e</sub> where:

$$A_{\epsilon} = A(1 - \epsilon_{g}) \tag{3.24}$$

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and

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$$\mathbf{L}_{\epsilon} = \mathbf{L}(1 + 0.55\epsilon_{\mathrm{g}}). \tag{3.25}$$

This approach oversimplifies the dependence of cell constant on the gas fraction and also does not address the possible effect of cell geometry on the measurements (Xu, 1991).

# 3.3.5 Applicability of the Available Techniques for On-Line Phase Holdup Estimation in Flotation Columns

The hydrodynamics of bubble column reactors has been the subject of many investigations in chemical engineering. Some of this work was presented in the previous sections: a more complete review can be found elsewhere (Fan, 1989). However, the results of those studies are not readily applicable to flotation columns. This stems from differences between bubble and flotation columns, two of which are addressed below:

1) Bubble size in a flotation column is much smaller (0.5-2 mm). This severely limits the use of gas holdup and bubble size measurement techniques which have been developed for the bubble columns. Fan (1989) states that new techniques must be developed which can detect bubbles with a size smaller than 1 mm. He also mentions that these probes must rely on techniques which do not intercept or pierce the bubble.

2) Gas flowrate in flotation columns is relatively low (0.5-3 cm/s) compared to bubble columns (2-20 cm/s) (Sada et al., 1986).

# **3.4** Experimental Section

# 3.4.1 Apparatus

The flotation column used in this work (Figure 3.5) was made of Plexiglas, 4.47 m in height and 10.18 cm in diameter. Bubbles were generated with a porous ( $\approx 10 \ \mu m$ holes) stainless steel sparger at the bottom of the column. The middle 0.94 m section of the column was chosen for the holdup estimation in order to limit disturbances due to the feed and gas injection. Two pressure transducers (0-5 psi, Series 440 X - Omega) were installed to measure the pressure at the top and the bottom of the section. Two grid electrodes (Uribe-Salas, 1991) were placed close to the pressure transducers from a cell to measure the conductivity. The electrodes were placed as close as possible to the pressure transducers to make the working section the same for both measurements. Two air-actuated ball valves with an internal diameter of 10 cm were used to isolate the middle (sampling) section. The required pressure to operate the valves through a solenoid valve was 827 kPa (120 psi) which resulted in a response time of 250 ms. The feed was introduced to the column from the top section and the underflow was recycled to the reservoir. To measure the conductivity of the clear water, a conductivity cell was installed in the feed line between the feed inlet to the column and the feed pump (Figure 3.5, item 3). A Masterflex pump (720-33) was used to pump the feed to the column. In solid-water systems, a second pump was used in the underflow line to circulate the solid-water mixture. A gas flowmeter (TYLAN Model FM-380) and MIC 2000 controller were used to measure and display the air flowrate.

Estimation of solids holdup using conductivity-based methods requires two measurements: the conductivity of the clear water and the conductivity of the solid-water mixture. Simultaneous measurement of the two conductivities is desirable. Two approaches to act as a check were taken for the on-line measurement of the conductivity of the clear water.

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Figure 3.5: Laboratory flotation column set-up.

In the first approach, a Plexiglas tube, 1.5 cm in diameter and 1.2 m in height, was added to the feed line before it enters the column (Figure 3.5, item 6). In this way, a portion of the feed was directed to the tube (added section). Two grid electrodes were also installed in the added section, 20 cm above the feed line, to measure the conductivity of clear water. Since water in the added section was not in motion, particles settled before reaching the electrodes. As a result, the volume between the electrodes filled with clear water. This was particularly apparent for the coarse particles ( $90\% +75 \mu m$ ) used in the present study. For fine particles this approach may not be appropriate.

In the second approach, two grid electrodes inside the column, 20 cm above the feed inlet, were installed (Figure 3.5, item 6).

# 3.4.2 Materials

The gas and liquid phases were air and tap water, respectively. Calcite, which is a non-conductive mineral, from Steep Rock Resources Inc. (Ontario) was used as the solid phase. To limit the wear on the valves, a soft material like calcite was preferred. The density of the calcite was  $2.72\pm0.01$  g/cm<sup>3</sup> as determined with a gas pycnometer. Table 3.1 shows its size distribution.

Table 3.1: Size distribution of the calcite.

Size (µm)	+300	+212	+150	+106	+75	+ 53	-53
Weight (%)	5.15	15.70	26.03	27.26	15.82	7.31	2.73

# 3.4.3 Data Acquisition System

A computer data acquisition system (Figure 3.6) was used extensively in the present study. The data acquisition system consisted of the following:



Figure 3.6: Data acquisition system.

- a microcomputer (286, IBM compatible, 1MB of memory),
- a 24-channel relay board (Metrabyte, model ERB-24),
- an I/O interface board to control the relay board (Metrabyte, model PI-12),
- an A/D convertor interface board (Metrabyte model DAS-8PGA),
- a conductivity meter (Tacussel, model CD-810)
- two pressure transducers (Omega, PX 440), and

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- two power supplies (U24Y100) to activate the pressure transducers.

The pressure transducer range was 0-5 psi (0-34.5 kPa) and the output signal was a current between 0 to 4 mA. By adding a 50 KOhm resistor to the circuit, current output of the pressure signal was converted to a 0.2-1 volt signal which was suitable for connection to the A/D board. The output of the conductivity meter was between 0 to 5 volts. A simple resistive divider was used to convert the output of the conductivity meter to 0-1 V. The idea was to use the whole range of the A/D board in order to increase the resolution of the conversion.

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Programs in GWBASIC were developed for each task. The flow chart of the data-acquisition program is given in Figure 3.7 (see also data acquisition program in Appendix 3.1). Since the response time of the conductivity meter was about 3 seconds, the time interval between the samples was usually selected as 3 seconds or more. To increase the statistical validity of the results, more than 15 readings for each point were taken. During the experiment each measurement of the conductivity and pressure was displayed on the monitor. This provided a means of detecting any abnormality in the system, e.g., a sudden change in the pressure reading could be traced, for example, to a variation in the feed flowrate. Moreover, it provided a clear picture of the effects of the different variables on the measured parameters. For each set of measurements the average, standard deviation and relative error were calculated. All collected data and the results of the gas holdup estimation from the various models were saved in a file.



# Figure 3.7: Flowsheet of data acquisition program (gas-water system, batch operation).

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# 3.4.4 Procedure

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; ;ł To generate small bubbles, the desired amount of frother was added to the reservoir and mixed for a few minutes. The column was filled with water and gas was then introduced. To test for steady-state conditions, the conductivity of four consecutive time intervals was measured and compared through a program in the data acquisition system. When the difference between them was equal or smaller than an expected error (see Section 3.5.1.10), the conductivity and pressure measurements of the gas-water system were made. Otherwise, this routine was repeated till steady-state was reached.

The number of samples (readings) and the time interval between samples were specified at the beginning of each experiment. All experiments were performed by taking 10-15 readings with time intervals of 3 seconds. During conductivity measurements, to avoid possible electrical interference from the pressure transducers a a relay board (Metrabyte, ERB-24) was used to switch off the power supply to the pressure transducers while measuring the conductivity. The reciprocal precaution was taken when making pressure measurements.

The average values of the pressure difference and conductivity measurements were used to estimate the gas holdup.

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Upon completion of the conductivity and pressure measurements, the valves were simultaneously closed to isolate the section. By closing the valves, the column was separated into three sections. By opening each valve separately, independent discharge of the contents from the three sections via the underflow port was possible. The sample of the middle section provided the actual gas holdup measurement.

Determination of the Volume between the Valves in the Middle Section: in order to determine the volume between the valves, the column was filled with water and then

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the valves were activated. The air trapped inside the valves was released by opening and closing the valves before making any measurements. The water between the valves was collected and weighed; the weight was converted to volume by assuming the density of water was 1000 kg/m<sup>3</sup> (at 25 °C). Due to the importance of this basic measurement, it was repeated forty times.

Actual Gas and Solids Holdup Measurement: the measurement of the actual gas and solids holdup was carried out by a method which is often called the "isolating technique" (Uribe-Salas, 1991; Marchese, 1991). This technique makes use of two ball valves which can be closed simultaneously by a solenoid valve. The actual gas holdup was determined by weighing the sample and comparing it with the volume between the valves. In the solid-water system, the sample was weighed, filtered and dried to determine the actual percent solids. This actual measure of the gas and solids holdup provided a standard to evaluate the phase holdup estimated from pressure difference and conductivity-based methods.

- **3.5 Results and Discussion**
- 3.5.1 Gas-Water

# **3.5.1.1** Calibration of the Pressure Transducers

One of the difficulties associated with the pressure transducers is calibration. In other words, at a zero head pressure the output of the pressure transducers usually is not zero (or, in this case, 4 mA).

In order to test the calibration of the pressure transducers a set of experiments was run. At the beginning of each experiment, the column was discharged and the pressure was measured. The pressure transducers used in the present study were gauge

transducers, i.e., they gave the difference between system and atmospheric pressure. It was expected that the output of the pressure transducers in the empty column would be zero. However, due to imperfections and sensitivity (0-5 psi), the output was 0.965 kPa (0.14 psi) for the bottom and 1.517 kPa (0.22 psi) for the top pressure transducer. The column was then filled with different levels of water and pressure measurements were made. In each test, the pressure of a single point was measured 10-15 times and the average value was assigned as the pressure at that point (Figure 3.8a). Another test was performed on a different day to validate the results (Figure 3.8b). It was observed that the slope of the lines from the two experiments was essentially identical but the intercept was different. This indicated that the pressure measurements were reproducible except for a bias.

A potential problem was that the bias was time dependent. For example, the initial pressure for the top pressure transducer in one case was 0.972 kPa (0.141 psi) and in another case was 1.400 kPa (0.203 psi). This variation of 0.428 kPa (0.062 psi) in the pressure is equivalent to approximately 5% gas holdup. This implies that an absolute error of 5% in gas holdup determination may originate solely due to the pressure transducers being used.

Figure 3.9 shows the pressure variation for zero head pressure for the period of 4 days. The variation does not seem to have any specific trend. The figure clearly demonstrates the importance of calibration of the pressure transducers. Although the direction of the change in both pressure transducers is the same, the magnitude is different. The changes in the absolute pressure can be attributed to changes in the local atmospheric pressure. (This was verified by monitoring the change in local atmospheric pressure over the corresponding days.) The calibration problem seems to be an inherent difficulty with pressure transducers.

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# Figure 3.8: Calibration of the pressure transducers.

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# 3.5.1.2 Zero Balance Circuit

As was mentioned earlier, the current output of the pressure transducers for the zero head was expected to be 4 mA. However, as was shown earlier (Figure 3.8 (a and b)), the 'zero' changed which consequently made the conversion of the analog to digital signals not only difficult but also inaccurate. This inaccuracy originated from the fact that there was no single digital number which could be assigned to the zero of the instrument. In order to have a constant zero for the pressure transducers (i.e., 4 mA current), the connectors were replaced with zero balance circuit connectors. These connectors have the facility to externally set the output signal to zero by using adjustment screws. This feature made the calibration procedure easy and the analog to digital conversion accurate.

# 3.5.1.3 The Effect of the Zero Balance Circuit on the Gas Holdup Estimation

A set of experiments was performed to compare the gas holdup estimation with and without the zero balance circuit connectors. Figure 3.10 demonstrates the results. Two different frother concentrations (5 and 25 ppm) were used and a wide range of gas holdup was covered (3 to 25%). For the whole range, the gas holdup estimation was more accurate using the zero balance circuit. Experiments were carried out on two different days and in both cases calibration was done at the beginning of the day. Based on these results, the zero balance circuit was permanently installed in the system.

# 3.5.1.4 Period of Calibration

Since the calibration procedure imposes a pause in the operation, it is essential to investigate the effect of the calibration period on the final results. Four experiments were performed to determine the effect of calibration period. In the first experiment, the pressure transducers were calibrated before making all measurements. This was repeated

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Figure 3.9: Variation of pressure with zero head with time.





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for all measurements ranging from 4 to 30% gas holdup. For the second experiment, the calibration was carried out once at the beginning of the day. The third experiment was run one day after calibration. Finally, the fourth experiment was performed two days after calibration. Figure 3.11 presents the results. It is clear that by increasing the lapse since calibration the estimated gas holdup values deviate more from the measured gas holdup values. However, the difference between the results of calibrating each test (which is quite laborious) and one calibration per day does not appear to be significant. This suggests that one calibration per day is sufficient. In the cases where high accuracy measurements are not required, the calibration period can be extended.

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# **3.5.1.5** The Effect of Temperature on Conductivity

The conductivity of an aqueous solution is determined by the concentration, charge, temperature and mobility of the dissolved ions. Temperature affects the viscosity of the fluid and thus the mobility of ions in solution. Moreover, the size of the associated cosphere of water and oppositely charged ions around each ion and the concentration expressed in volume units are also affected by temperature (Miller et al., 1988).

The conductivity of water increases with temperature 2 to 3 percent per degree Celsius above 0 °C (Hem, 1970). As the temperature increases above 4 °C, water expands in volume about 0.025 percent per degree. Because quantities of solute in solution remain the same, in the absence of other effects, expansion results in a small decrease in concentration expressed in per unit volume which should cause a corresponding small decrease in conductivity. Clearly, volume changes alone cannot account for the change in conductivity with temperature. The viscosity of water decreases with increasing temperature by about 2 percent per degree Celsius (Weast, 1976), thus decreasing the resistance to flow, and tending to increase conductivity. The direction and magnitude of this effect approaches the observed effect on conductivity. As temperature increases, solvation of ions (association with water molecules) decreases

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Figure 3.11: Effect of calibration period on gas holdup estimation.

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and the size of the associated cosphere decreases thus increasing ion mobility and conductivity (Daniels and Alberty, 1967). Rosenthal and Kidder (1969) give a range of 0.5 to 3 percent for the increase in conductivity per degree Celsius, but this range was determined on industrial solutions (Miller et al., 1988).

For on-line gas or solids holdup estimation simultaneous measurement of the conductivity of the water and water-gas system is required. Since the conductivity of the water in the solid-water system, unlike the gas-water system, is measured in the added section which has a diameter equal to one-tenth of the diameter of the column, a temperature effect was anticipated. It should be noted that the water was generally colder than the body of the column. This temperature difference changes the measured conductivity which in turn results in a biased holdup estimation. Hence, it is important to correct the holdup estimation results for any change in temperature.

Conductivity of electrolytes increases with increasing temperature. Glasstone (1942) proposed the following relationship:

$$\kappa = \kappa_{25^{\circ}C} [1 + \alpha_{25^{\circ}C} (t - 25^{\circ}C)]$$
(3.26)

where  $\kappa$  and  $\kappa_{25^{\circ}C}$  are the conductivities (mS/cm) of the electrolyte at the temperature t and 25°C, respectively, and  $\alpha_{25^{\circ}C}$  is the coefficient of variation of the conductivity per degree Celsius with respect to the conductivity at 25°C. Glasstone (1942) and Uribe-Salas (1991) have reported temperature coefficients (at 25°C) of 0.020 and 0.019;

The extent of the effect of temperature on conductivity in the experimental set up was investigated. The column was filled with water and the conductivity of the water was measured in the column and in the added section. The time between samples was 3 seconds and the measurement lasted for 11 minutes. Figure 3.12 illustrates the results. The conductivity of the water in the column did not change significantly, whereas it

increased in the added section with time. To alleviate the heat transfer from the room to the added section, it was insulated (see Appendix 3.2 for the analysis of heat transfer in the added section and in the column). To evaluate the effect of the insulation, the test was repeated. Results are given in Figure 3.13. The rate of change of conductivity in the added section decreased in comparison to the previous test, while the conductivity of the water in the column remained unchanged. These results suggested the permanent use of the insulation for the added section.

To monitor the temperature in the column and in the added section while measuring conductivity, two thermistors were installed, one in the middle of the sampling section inside the column and the other in the added section between the two electrodes (Figure 3.5, item 4). The calibration curve for the two thermistors was derived off-line (see Appendix 3.3). The temperature was varied between 10 to 25°C corresponding to the range encountered in this study: this limited range provided accurate temperature measurements. Figure 3.14 presents the temperature of the water in the middle of the column and in the added section. It should be noted that the temperature of the water in the feed tank was constant. Hence, any change in the temperature originated from different temperatures in the two sections.

The effect of temperature on the measured conductivity in the cell at the top of the column, in the added section and in the middle of the column, was determined. The column was filled with water and the conductivity of the water was measured in the three mentioned sections. Figure 3.15 depicts the results. The conductivity of the water increased for the three sections; however, the increase was pronounced only in the added section. The measured conductivity at the top of the column was in good agreement with that measured in the column (Figure 3.15). Due to its smaller diameter, the added section experienced considerable changes in temperature and consequently, in measured conductivities. This suggested that the conductivity without temperature compensation.

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Figure 3.12: Variation of conductivity in the column and in the added section.



Figure 3.13: Effect of the insulation on measured conductivity in the added section.



Figure 3.14: Temperature variation in the middle of the column and in the added section.



Figure 3.15: Conductivity variation in the three sections of the column.

Considering that the experiments did not take more than 8 minutes, this assumption appeared legitimate.

# **3.5.1.6** Effect of Gas Injection on Temperature

In the batch experiments where the conductivity of the water and gas-water systems is measured in the same column one shortly after another, any change in temperature can be attributed to the introduction of gas. In order to investigate the effect of gas injection on the temperature of the water, a set of experiments was performed. The column was filled with water and the temperature was measured every 3 seconds for 2.5 minutes. While temperature was being measured, gas was introduced in the column. Figure 3.16 presents the results of the measurements. The temperature decreased upon introduction of gas which was cooler than the water; however, the magnitude of the change  $(0.10-0.15 \, ^\circ C)$  was not significant. Hence, the effect of gas injection on temperature, and in turn on holdup estimation, can be neglected.

# 3.5.1.7 Variation of Cell Constant

To investigate the variation of cell constant over a wide range of conductivities, two conductivity cells, one in the feed line and another one in the added section, were installed. Since it was also intended to study the effect of cell constant on the conductivity measurements, the cell constant of the new cells was designed to be different from the old cells. The new cells were placed close to the old cells to create the possibility of using a combination of electrodes for the conductivity measurements. For instance, the first electrode of the old cell and the last electrode of the new cell were used to obtain a cell with a small cell constant (one tenth of the cell constant of the cell inside the column, Figure 3.17).

The cell constants of the three cells in the feed line were 0.55, 0.18 and 0.11

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Figure 3.16: Effect of gas injection on temperature.





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cm<sup>2</sup>/cm, respectively. The added section cells had the same cell constants. The column was filled with water and operated continuously. The conductivity of water was decreased by lowering the temperature (by adding ice to the reservoir) and increased by adding potassium chloride. The conductivity of the water was measured consecutively in seven cells in the following order: one cell in the column, three cells in the feed line and three cells in the added section. For each point ten measurements were made and the average value was designated as the conductivity.

Results of the measurements are shown in Figures 3.18 and 3.19. A wide range of conductivity from 0.16 mS/cm to 2.3 mS/cm was covered. The conductivity measured inside the column was compared to that measured in the feed line and in the added section. It was found that upon increasing the conductivity, the two conductivity measurements tended to deviate. This was the case for all cells regardless of difference in the cell constants. However, for conductivities in the range of 0.20 to 0.50 mS/cm no deviations were observed in the measured conductivities at the various locations. This was probably because this range was close to the conductivity of water on which the cells were calibrated. In the present study, the range of conductivity encountered was well within the same range which ensures accurate conductivity measurements. Nevertheless, precautions should be taken where large changes in the conductivity are expected.

# 3.5.1.8 Gas Holdup Estimation

Batch Operation: the objective of the experiments was to evaluate the two methods of gas holdup estimation against the isolating technique which gave the actual measure of gas holdup.

Three sets of experiments with frother concentration of 5, 15 and 25 ppm (Dowfroth 250C) were run. To vary gas holdup, the superficial gas flow rate was varied between 0.5 and 3 cm/s. High superficial gas rates were used when the frother



Figure 3.19: Conductivity measured in the added section vs. measured inside the column.

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# CHAPTER 3 ON-LINE PHASE HOLDUP ESTIMATION IN TWO-PHASE SYSTEMS ... 3-38 concentration was low.

Figures 3.20, 3.21 and 3.22 show that the conductivity-based method gave slightly better estimates of gas holdup than the pressure difference method (without the zero balance circuit). In general, however, the estimated gas holdup from the two methods was in good agreement with the actual gas holdup.

In the conductivity method, for gas holdup less than 20%, the Maxwell model was better than that of Bruggeman, giving a relative error of 3.24% as opposed to 4.26%. This may originate from the fact that below 20% gas holdup the distance between the bubbles was larger than their diameter which ensured that there was no interaction between the fields around the bubbles, this complying with one of the restrictions of the Maxwell model (Meredith and Tobias, 1960, 1961; see Chapter 2). This was more evident for low frother concentrations where bubbles were neither uniform nor small. When frother concentration was 25 ppm, the prediction of the gas holdup by Maxwell's model did not deteriorate even at high gas holdup (26%)(Figure 3.21).

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The Bruggeman model, originally proposed for systems with a random dispersed phase size, predicted the gas holdup accurately at low frother concentrations and high gas rates. Referring to Figures 3.20 and 3.21, at low frother concentrations (5 ppm and 15 ppm) the gas holdup estimation from the Bruggeman model was better than the Maxwell model at high gas rates (high gas holdup). However, at the high frother concentration (25 ppm), where bubbles size was more uniform, Maxwell's model estimation was preferable even at high gas rates (Figure 3.22).

Figures 3.23, 3.24 and 3.25 depict the gas holdup estimates using the pressure transducers when they were equipped with the zero balance circuits. Gas holdup estimates were more accurate compared with the zero balance circuits.



Figure 3.20: Gas holdup measurement from conductivity and pressure (5 ppm frother).



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Figure 3.22: Gas holdup measurement from conductivity and pressure (25 ppm frother).

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Figure 3.24: Gas holdup estimation using zero balance circuit (15 ppm frother).

Continuous Operation: the results of the gas holdup estimation in the continuous operation are illustrated in Figure 3.26. The pressure measurements were made when the pressure transducer was equipped with the zero balance circuit. The gas holdup estimation from the pressure difference and conductivity methods was now equally accurate.

# 3.5.1.9 Error Propagation Analysis

It is important to determine the effect of error in the measurement of conductivity and pressure on the estimated gas holdup. The prime concern is to find the distribution of the estimated gas holdup and to verify that it is within an acceptable range. The functions which relate measured variables and gas holdup are known (Maxwell's and Bruggeman's models and the pressure difference relationship). The assumption is that the measured variables are accurate. This is reasonable because the instruments were calibrated at regular intervals.

The variance of the estimated gas holdup is calculated by expanding each function as a Taylor series about the mean. If only the zero and first order terms of the expansion are considered and the covariances between the variables are assumed to be zero, the variance of the three approaches will be as follows:

1- The Maxwell Model

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Maxwell equation for gas holdup estimation is:

$$\epsilon_{\rm g} = \frac{\kappa_{\rm l} - \kappa_{\rm l-g}}{\kappa_{\rm l} + 0.5 \,\kappa_{\rm l-g}} \tag{3.27}$$

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where  $\kappa_l$  is the average conductivity of the clear water and  $\kappa_{l,g}$  is the average conductivity of the gas-water system. In view of the above considerations, the variance



Figure 3.25: Gas holdup estimation using zero balance circuit (25 ppm frother).

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Figure 3.26: On-line gas holdup measurement (15 ppm frother).

 $(S^2)$  is equal to:

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$$\mathbf{S}_{\boldsymbol{\epsilon}_{g}}^{2} = \left(\frac{\partial \boldsymbol{\epsilon}_{g}}{\partial \boldsymbol{\kappa}_{l-g}}\right)^{2} \mathbf{S}_{\boldsymbol{\kappa}_{l-g}}^{2} + \left(\frac{\partial \boldsymbol{\epsilon}_{g}}{\partial \boldsymbol{\kappa}_{l}}\right)^{2} \mathbf{S}_{\boldsymbol{\kappa}_{l}}^{2}$$
(3.28)

which gives:

$$S_{\epsilon_{g}}^{2} = \left(\frac{-1.5 \kappa_{l}}{(\kappa_{l} + 0.5 \kappa_{l-g})^{2}}\right)^{2} S_{\kappa_{l-g}}^{2} + \left(\frac{1.5 \kappa_{l-g}}{(\kappa_{l} + 0.5 \kappa_{l-g})^{2}}\right)^{2} S_{\kappa_{l}}^{2}.$$
(3.29)

An example of the results for 25 ppm frother is shown in Table 3.2.

Table 3.2: A typical gas holdup estimation result from the Maxwell model for 25 ppm frother.

κ <sub>i</sub> (mS/cm)	S (mS/cm)	κ <sub>l∙g</sub> (mS/cm)	S (mS/cm)	estimated	S (%)	e (%) actual
0.3008	0.0010	0.2327	0.0012	16.33	0.37	16.03

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# 2- The Bruggeman Model

The Bruggeman equation for gas holdup estimation is:

$$\epsilon_{g} = 1 - \left(\frac{\kappa_{l-g}}{\kappa_{l}}\right)^{2/3}$$
(3.30)

The variance of the estimated gas holdup is:

$$\mathbf{S}_{\epsilon_{g}}^{2} \doteq \left(\frac{\partial \epsilon_{g}}{\partial_{\kappa_{l-g}}}\right)^{2} \mathbf{S}_{\kappa_{l-g}}^{2} + \left(\frac{\partial \epsilon_{g}}{\partial_{\kappa_{l}}}\right)^{2} \mathbf{S}_{\kappa_{l}}^{2}$$
(3.31)

which yields:

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$$S_{\epsilon_{g}}^{2} = \left[ -\frac{2}{3} \left( \frac{\kappa_{l-g}}{\kappa_{l}} \right)^{-\frac{1}{3}} \frac{1}{\kappa_{l}} \right]^{2} S_{\kappa_{l-g}}^{2} + \left[ -\frac{2}{3} \left( \frac{\kappa_{l-g}}{\kappa_{l}} \right)^{-\frac{1}{3}} \frac{\kappa_{l-g}}{\kappa_{l}^{2}} \right]^{2} S_{\kappa_{l}}^{2}.$$
(3.32)

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An example of the results for 25 ppm is presented in Table 3.3.

Table 3.3: A typical gas holdup estimation result from the Bruggeman model for25 ppm frother.

κ <sub>t</sub> (mS/cm)	S (mS/cm)	κ <sub>ig</sub> (mS/cm)	S (mS/cm)	ς <sub>z</sub> (%) estimated	S (%)	$\epsilon_{g}$ (%) actual
0.3008	0.0010	0.2327	0.0012	15.73	0.34	16.03

3- The Pressure Difference Method

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Gas holdup is estimated using the following equation:

$$\epsilon_{\rm g} = 1 - \frac{\Delta P}{\rho_{\rm I} g L} \tag{3.33}$$

where  $\Delta P$  is the pressure difference, g the acceleration due to gravity,  $\rho_1$  the density of water and L is the distance between the pressure transducers. The variance is equal to:

$$S_{\epsilon_g}^2 = \left(-\frac{1}{\rho_1 g L}\right)^2 S_{\Delta P}^2.$$
(3.34)

Table 3.4 presents an example of the results obtained for an experiment with 25 ppm frother.

 Table 3.4: An example of the gas holdup estimation from the pressure difference method for 25 ppm frother.

∆P (Pa)	S (Pa)	L (cm)	S (cm)	$\epsilon_{i}$ (%) estimated	S (%)	$\epsilon_{g}(\%)$ actual
6551.3	55.2	81.9	0.01	16.96	0.69	16.03

### **3.5.1.10** Expected Error of the A/D Convertor

In the data acquisition system an A/D convertor was used to convert the analog voltage signals to the integer (digital) numbers. The maximum size of the integer number, and thus the resolution of the conversion, depends on the integer number of bits handled by the convertor. The range of the integer I is given by  $I_{range} = 2^N$ , where N is the number of bits. The A/D convertor was a 12-bit type with a selectable analog input range. Thus, the maximum available integer range for the convertor was 4096. The expected error of the conductivity and pressure measurements are discussed below.

The Conductivity Measurements: for these measurements, the 0-5 mS range of the conductivity meter was used. The output of the conductivity measurements after signal conditioning was between 0-1 volt, which was one of the available ranges in the A/D board. This provided the maximum utilization of the A/D board, and hence a resolution R which was equal to:

$$R = \frac{5}{4096} = 0.0012 \text{ mS.}$$
(3.35)

Thus the expected error is  $\pm \frac{1}{2}R = \pm 0.0006$  mS. This magnitude of error translates to an error of 0.3% in the gas holdup estimation when the actual gas holdup is about 18%. This introduces a relative error of 1.7% which will be added to the other sources of error. This error which originates from the A/D conversion is often called "expected error" (Ray, 1981).

The Pressure Measurements: the pressure transducer output after conditioning was between 0.2-1 volt. The same A/D board input range (0-1 V) was used. Since 0 pressure had an output of 0.2 V, the whole range of the integer number was not accessible. The maximum integer range was 3277 (4096-819) which led to a lower resolution than in the case with conductivity. The pressure transducer's range was

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between 0-5 psi and the resolution was equal to:

$$R = \frac{5}{3277} = 0.0015 \text{ psi} (\text{or } 10.34 \text{ Pa}). \tag{3.36}$$

Thus the expected error of  $\pm \frac{1}{2}R=5.17$  Pa (0.0008 psi). This translates to an error of 0.06% in the gas holdup estimation when the gas holdup is approximately 18%. The expected error from the pressure difference method is negligible compared to the conductivity method.

#### 3.5.1.11 Relative Error Analysis

The relative error of the gas holdup estimation for the three methods was calculated. The relative error is defined as:

Figure 3.27, 3.28 and 3.29 depict the relative error for the three set of experiments (frother concentrations of 5, 15 and 25 ppm). The relative error of the Maxwell model estimate increased when the gas holdup increased. For instance, for the 15 ppm frother, the relative error increased from 1% to 4% when gas holdup increased from 3% to 24%. This trend was also the case for 5 ppm and 25 ppm frother. This probably stems from the fact that by increasing gas rate with a constant frother concentration, bubbles lose their uniform size which in turn violates an assumption of the model. As expected, the relative error of the gas holdup estimation using the pressure difference method decreased when the gas rate increased. This can be shown from the basic consideration of the gas holdup estimation. Recall,

$$\epsilon_{g} = 1 - \frac{\Delta P}{L \rho_{1} g}$$
(3.38)



Figure 3.27: Relative error of gas holdup estimation from pressure and conductivity (5 ppm frother).



Figure 3.28: Relative error of gas holdup estimation from pressure and conductivity (15 ppm frother).

By considering error propagation, it is evident that with a constant error on the pressure measurements the relative error on gas holdup estimation decreases as gas holdup increases. Figure 3.30 illustrates the relative error assuming a constant error of 55.16 Pa (0.008 psi) on the pressure measurements. The trend agrees well with that of the experimental relative errors.

The Bruggeman model had rather interesting results. For the low frother concentration (5 ppm), where bubble size was not uniform, the relative error decreased when gas rate increased. With a moderate frother concentration (15 ppm), the relative error was almost constant for different gas rates. The moderate frother concentration provided a fairly broad bubble size distribution which fulfilled the original requirement of the model. With the higher frother concentration (25 ppm), the relative error increased when gas flowrate increased. This increase in the relative error probably reflects the presence of bubbles with a fairly uniform size where Bruggeman's model fails to predict gas holdup accurately. This suggests that the Bruggeman model could have a potential use in qualitative evaluation of bubble size distribution. In other words, any inaccuracy in gas holdup estimation by the Bruggeman model could indicate a narrow bubble size distribution and vice versa. This needs to be verified by measuring the bubble size distribution.

## 3.5.2 Solid-Water System

### **3.5.2.1** Variation of the Pressure Measurements

The initial pressure measurements in the continuous operation demonstrated a high standard deviation compared to that of the batch operation. Table 3.5 illustrates some of the results.



Figure 3.29: Relative error of gas holdup estimation from pressure and conductivity (25 ppm frother).



Figure 3.30: Relative error of gas holdup estimation from pressure assuming a constant error on pressure measurements.

Continuou	IS	Batch		
Pressure Difference (Pa)	S (Pa)	Pressure Difference (Pa)	S (Pa)	
9087.1	2922.6	7195.3	33.8	
9283.6	1508.5	6962.2	49.0	
8865.1	1419.5	6619.6	59.3	
8787.2	1151.4	6372.7	40.7	

Table 3.5: The standard deviations of the pressure difference measurements for continuous and batch operations.

The pressure measurements were made every 3 seconds. Because of the large variation of the pressure measurements in continuous operation, the holdup estimation was inaccurate, e.g., the standard deviations could be higher than the actual pressure differences. In order to give more detail on the pressure measurements, the sampling time was reduced from 3 seconds to 0.05 seconds. Two experiments were conducted in continuous operation: one in the water only system and another one in the solid-water system. Figures 3.31 and 3.32 present the pressure measurements for the top and the bottom pressure transducers and the pressure difference (for the water-solid system only). The variation in the pressure measurements for both the gas-water and solid-water systems was wide. This eliminated the idea that these dramatic changes were due to the introduction of solids. The constant pressure disturbances in the continuous operation suggested a common source of disturbance which did not exist in the batch operation. It was found that the pump in the underflow line was the source of the pressure disturbances. To verify this, during operation (gas-water system) the underflow pump was turned off and the pressure measurements were repeated. Figure 3.33 depicts the results: the pressure measurements returned to the disturbance-free signals. However, continuous operation of solid-water systems without a pump was not possible because of plugging. To reduce the pressure disturbances the distance between the pump and the underflow discharge was extended. Figure 3.34 illustrates the pressure measurements in the solid-water system: the pressure signals have significantly improved compared to the previous tests (Figures 3.31, 3.32). Nevertheless, the complete removal of these

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Figure 3.32: Pressure measurement variation in watersolid system (pump at the tailing port).



Figure 3.34: Pressure measurement variation in watersolid system (free tailings drainage).

disturbances in the continuous operation proved difficult.

## 3.5.2.2 Effect of Gas Content on the Pressure Disturbances

Figure 3.35 presents the pressure measurements for a gas holdup of 10%. The results showed a pronounced dampening by air bubbles which gave near-to-ideal pressure signals. A second test was carried out with 5% gas holdup. The results are presented in Figure 3.36. The results indicated that the dampening effect of air bubbles decreased when gas content decreased. Since in practice gas holdup varies between 5-25%, this virtually eliminates problems due to pressure disturbances.

## 3.5.2.3 Solids Holdup Estimation Results

Figures 3.37 and 3.38 present the results of two tests with solids. The first test was performed when the pressure disturbances were still present in the system. The solids holdup estimation from conductivity agreed well with the measured values, whereas, the estimated values from the pressure were very inaccurate. Because of the pressure disturbances, the solids holdup estimates were randomly distributed.

Unlike gas-water systems where the performance of the Maxwell and Bruggeman models was different for the various gas holdup ranges, in solids-water systems both methods performed generally quite well. This may be because for air bubbles shape depends upon the frother concentration and gas flowrate while for solid particles shape is invariant.

In the second test, the pressure disturbances had been reduced (Figure 3.38). A significant improvement in the accuracy of the solids holdup estimations compared to the previous test indicated the importance of removing pressure disturbances. The accuracy of the solids holdup estimation from conductivity was similar to the previous test.



Figure 3.35: Effect of gas content on dampening of pressure disturbances (10% gas).

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Figure 3.36: Effect of gas content on dampening of pressure disturbances (5% gas).





The solids concentration range of 5-17% by volume used in the experiments covered the normal industrial range. For instance, the 17% solids by volume is an equivalent of 46.24% by weight for solids with a specific gravity of 4.2 which is commonly encountered in grinding circuits, and of course, in thickeners. This assured the validity of the models for the prediction of holdup over a wide range.

The reproducibility of results was verified by repeating some of the experimental points. As shown in Figure 3.38 the repeat test results were very close. The accuracy of both approaches, the pressure difference and conductivity-based methods, was good with estimated values within  $\pm 1\%$  of the actual values.

## 3.6 Conclusions

1) On-line measurement of the gas holdup in the gas-water system using the pressure difference and conductivity-based methods was in good agreement with the actual values from the isolating system. For off-line gas holdup measurement, agreement between the actual and estimated gas holdup values was excellent.

2) Solids holdup in the solid-water system over a wide range (5 to 17%) was estimated on-line in a laboratory column. Two independent approaches based on conductivity and pressure difference were taken. The results of both were verified by comparison with the actual solids holdup obtained by the isolating system. The accuracy of the solids holdup estimation was high (maximum absolute error of 2%); however, the conductivity-based methods gave more accurate results than the pressure difference method.

3) Pressure disturbances caused by pumps can significantly degrade the accuracy of the holdup measurements using the pressure difference method. Dampening of the pressure disturbances is required for accurate holdup estimation.

4) Frequent (every day) re-calibration of pressure transducers was found to be essential for accurate holdup estimation.

5) Temperature affects the conductivity of clear water (about 2% per degree Celsius). Since on-line phase holdup estimation requires simultaneous measurement and comparison of two conductivity signals (that of the dispersion and the continuous phase), any change in temperature of the systems could result in biased conductivity measurements. Recording of the temperature while measuring the conductivity was found essential.

6) Cell constant does vary if the range of conductivities is large. This necessitates re-calibration of the cells if large changes in conductivity occur.

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# Appendix 3.1

## **Computer Data Acquisition Program**

10 CLEAR:CLS	
20 KEY OFF	
30 PRINT"	
40 PRINT"	
50 PRINT"	
60 PRINT"	**************************************
70 PRINT"	***************************************
SO PRINT"	**************************************
	1 wo-1 hase bystems
110 PRINT	
170 PRINT"	
130 PRINT"	
140 PRINT	
150 PRINT"	Rv: S. Banisi "
160 PRINT"	Lift O. Dumin
170 PRINT"	McGill University June 1991 "
180 PRINT"	
190 PRINT"	
200 PRINT"	
210 PRINT"	
220 PRINT"	
230 PRINT"	
240 PRINT"	
250 INPUT"Press	any key to continue".A\$
260 CLEAR:CLS	
close THI	E GAS VALVE TO MEASURE THE CONDUCTIVITY OF WATER
070 0175 8 11202	0.1700
270 UUT &H303,	, <b>&amp;</b> H8U
260 UUI &H300,	,δ
290 PRINT	AR LIGHT CONDUCTIVITY MEASUREMENT
210 DDINT"	AR LIQUID CONDUCTIVITIT MEASUREMENT
JIU EKINI ENTED E	WREDIMENTAL CONDITIONS
	AFERIMENTAL CONDITIONS
320 INPUT "NUM	MBER OF SAMPLES="; N
330 DIM COND(	500), P1(500), P2(500),NO(500)
340 INPUT "TIM	E BETWEEN READINGS, SEC="; T
350 INPUT "CON	<b>ND. METER RANGE mS=";R</b>
360 PRINT "	n
370 J=1:'counter	
380 GOTO 410	
INITIALI	ZING THE A/D BOARD AND THE RELAY
390 IF FL%=0 T	HEN RETURN

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400 PRINT "ERROR IN DAS8:";MD%;" ";FL%:END 410 DEF SEG=&H3000 420 BLOAD "DAS8.BIN",0 430 DAS8=0 440 BASADR%=&H310 450 OUT &H303, &H80 460 OUT &H300,9:TRC#=TIMER+T 470 T2#=TIMER 480 IF (T2#-TRC#)> = 0 THEN 500 490 GOTO 470 A/D CONVERSION

```
500 MD% =0:FL% =0:CALL DAS8(MD%, BASADR%, FL%):GOSUB 390
510 MD% =1:LT%(0)=3:LT%(1)=3:FL%=0:CALL DAS8(MD%, LT%(0), FL%):GOSUB 390
520 MD% =19: VR% =9:FL% =0:CALL DAS8(MD%, VR%, FL%):GOSUB 390
530 MD% =4:D% =0:FL% =0:CALL DAS8(MD%, D%, FL%):GOSUB 390
540 COND(J)=(D%/2048)*R
550 PRINT "Conductivity (mS)=" COND(J)
CHECK THE NUMBER OF MEASUREMENTS
```

```
560 J=J+1 : IF J>N THEN 620

570 OUT &H300,8

580 TRC#=TIMER+T

590 T2#=TIMER

600 IF (T2#-TRC#)> = 0 THEN 460

610 GOTO 590

CALCULATE THE AVERAGE AND STANDARD DEVIATION
```

```
620 FOR J=1 TO N: FA=COND(J)+FA

630 NEXT J

640 KL=FA/N

650 FOR J=1 TO N:QA=QA+(COND(J)-KL)^2

660 NEXT J

670 KLS=(QA/(N-1))^.5

680 PRINT "

690 PRINT "

700 PRINT "AVG. Conductivity (mS)=" KL

710 PRINT "AVG. Conductivity (mS)=" KLS: RE0=KLS*100/KL

720 PRINT "Conductivity, S.D. (mS)=" KLS: RE0=KLS*100/KL

720 PRINT "Relative Error (%)=" RE0

730 PRINT "

0PEN THE GAS VALVE
```

740 OUT &H303,&H80 750 OUT &H300,0 760 TRC#=TIMER+60 770 T2#=TIMER 780 IF (T2#-TRC#)> = 0 THEN 800 790 GOTO 770 OPEN AND CLOSE THE BALL VALVES TO RELEASE THE ENTRAPED AIR

800 OUT &H300,16

```
810 TRC#=TIMER+3
820 T2#=TIMER
830 IF (T2#-TRC#)> = 0 THEN 850
840 GOTO 820
850 OUT &H300.0
860 TRC#=TIMER+3
870 T2#=TIMER
880 IF (T2#-TRC#)> = 0 THEN 900
890 GOTO 870
       MEASURE THE CONDUCTIVITY OF THE GAS-WATER SYSTEM
900 OUT &H300.1:TRC#=TIMER+T
910 T2#=TIMER
920 IF (T2#-TRC#)> = 0 THEN 940
930 GOTO 910
940 MD%=0:FL%=0:CALL DAS8(MD%, BASADR%, FL%):GOSUB 390
950 MD%=1:LT%(0)=3:LT%(1)=3:FL%=0:CALL DAS8(MD%,LT%(0), FL%):GOSUB 390
960 MD% = 19: VR% = 9:FL% = 0:CALL DAS8(MD%, VR%, FL%):GOSUB 390
970 MD%=4:D%=0:FL%=0:CALL DAS8(MD%, D%, FL%):GOSUB 390
980 COND(J) = (D\%/2048)*R
990 PRINT
1000 PRINT "
       DETERMINE THE STEADY-STATE SITUATION
1010 PRINT "Conductivity 1 (mS)=" COND(J)
1020 PRINT "Conductivity 2 (mS)=" COND(J-1)
1030 PRINT "Conductivity 3 (mS) = " COND(J-2)
1040 PRINT "Conductivity 4 (mS) = " COND(J-3)
1050 IF (ABS(COND(J)-COND(J-1)) < = .0024) AND (ABS(COND(J-2)-COND(J)) < = .0024) THEN
GOTO 1080
1060 J = J + 1
1070 GOTO 900
1080 \text{ IF}(ABS(COND(J-3)-COND(J-1)) < = .0024) \text{ AND}(ABS(COND(J-2)-COND(J-1)) < = .0024) \text{ THEN}
GOTO 1100
1090 GOTO 1060
1100 PRINT "
1110 PRINT "
1120 PRINT" The system has reached steady state"
1130 PRINT"
1140 PRINT "
1150 J = 1
       MEASURE THE CONDUCTIVITY OF THE GAS-WATER SYSTEM
1160 OUT &H300, 1:TRC#=TIMER+T
1170 T2#=TIMER
1180 IF (T2\#-TRC\#) > = 0 THEN 1200
1190 GOTO 1170
1200 MD%=0:FL%=0:CALL DAS8(MD%, BASADR%, FL%):GOSUB 390
1210 MD%=1:LT%(0)=3:LT%(1)=3:FL%=0:CALL DAS8(MD%,LT%(0), FL%):GOSUB 390
1220 MD%=19: VR%=9:FL%=0:CALL DAS8(MD%, VR%, FL%):GOSUB 390
1230 MD%=4:D%=0:FL%=0:CALL DAS8(MD%, D%, FL%):GOSUB 390
```

1240 COND(J) = (D%/2048) R1250 PRINT "Conductivity (mS) =" COND(J) MEASURE THE BOTTOM PRESSURE 1260 OUT &H300, 2:TRC#=TIMER+T 1270 T2#=TIMER 1280 IF (T2#-TRC#) > = 0 THEN 13001290 GOTO 1270 1300 MD%=1:LT%(0)=1:LT%(1)=1:FL%=0:CALL DAS8(MD%,LT%(0), FL%):GOSUB 390 1310 MD% = 19:D%(0) = 11;FL% = 0:CALL DAS8(MD%,D%(0),FL%):GOSUB 390 1320 MD%=4:D%(0)=0:FL%=0 1330 CALL DAS8(MD%,D%,FL%):GOSUB 390 1340 P1(J) = 5-5\*(4096-D%)/(4096-819)1350 PRINT "Bottom Pressure (psi)=" P1(J) MEASURE THE TOP PRESSURE 1360 OUT &H300, 4:TRC#=TIMER+2 1370 T2#=TIMER 1380 IF (T2#-TRC#)> = 0 THEN 1400 1390 GOTO 1370 1400 MD%=1:LT%(0)=2:LT%(1)=2:FL%=0:CALL DAS8(MD%,LT%(0), FL%):GOSUB 390 1410 MD% = 19:D%(0) = 11:FL% = 0:CALL DAS8(MD%,D%(0),FL%):GOSUB 390 1420 MD% = 4:D%(0) = 0:FL% = 01430 CALL DAS8(MD%, D%, FL%): GOSUB 390 1440 P2(J) = 5-5\*(4096-D%)/(4096-819)1450 PRINT "Top Pressure (psi)=" P2(J) 1460 PRINT " 1470 NO(J) = JCHECK FOR THE NUMBER OF MEASUREMENTS 1480 J=J+1: IF J>N THEN 1500 1490 GOTO 1160 SIMULTANEOUSLY CLOSE THE GAS VALVE AND THE BALL VALVES 1500 OUT &H303,&H80 1510 OUT &H300.24 CALCULATE THE AVERAGE AND STANDARD DEVIATION 1520 FOR J=1 TO N: F=COND(J)+F1530 PT1 = P1(J) + PT11540 PT2 = P2(J) + PT21550 NEXT J 1560 AV = F/N:AP1 = (PT1/N) :AP2 = (PT2/N)1570 FOR J=1 TO N:0=0+(COND(J)-AV)^2  $1580 M = M + (P1(J)-AP1)^{2}:MT = MT + (P2(J)-AP2)^{2}$ 1590 NEXT J  $1600 \text{ SD1} = (Q/(N-1))^{.5}$  $1610 \text{ SD2} = (M/(N-1))^{.5}$  $1620 \text{ SD3} = (MT/(N-1))^{.5}$ 1630 EG1 = 100\*(KL-AV)/(AV\*.5+KL)1640 PD = API - AP2

```
1650 EG2=100*(1- (PD/1.155503))
1660 EG3 = 100*(1-((AV/KL)^{(2/3)}))
16705DC2=(((2/3)*((AV)^(2/3))/(KL^(5/3)))^2)*(SD1^2)+(((2/3)/(KL^(2/3)*(AV^(1/3))))^2)*(KLS^2))
1680 \text{ SDGC2} = \text{SDC2}^{-5}
1690 \text{ SDC} = (((.5*AV + AV)/((.5*AV + KL)^2))^2)*(KLS^2) + (((.5*KL + KL)/((.5*AV + KL)^2))^2)
*(SD1^2)
1700 SDGC=SDC^.5
1710 \text{ SDD} = ((\text{SD2})^2 + (\text{SD3})^2 + .0002)^{-5}
1720 \text{ SDG} = (.7466*(\text{SDD}^2)) + .0003*(\text{PD}^2)
1730 \text{ SDGP} = \text{SDG}^{-5}
1740 PRINT "
1750 PRINT "
1760 PRINT "AVG. Top Pressure (psi)=" AP2
1770 RE1=SD3*100/AP2
1780 PRINT "Relative Error (%)=" RE1
1790 PRINT "Top Pressure, S.D. (psi)=" SD3
1800 PRINT "
1810 PRINT "AVG. Bottom Pressure (psi=)" AP1
1820 RE2=SD2*100/AP1
1830 PRINT "Relative Error (%)=" RE2
1840 PRINT "Bottom Pressure, S.D. (psi)=" SD2
1850 PRINT "
1860 PRINT "Pressure Drop (psi)=" PD
1870 PRINT "Pressure Drop, S.D. (psi)=" SDD
1880 RE3=SDD*100/PD
1890 PRINT "Relative Error (%)=" RE3
1900 PRINT "
1910 PRINT "AVG. Conductivity (mS)=" AV
1920 PRINT "Conductivity, S.D. (mS)=" SD1: RE4=SD1*100/AV
1930 PRINT "Relative Error (%)=" RE4
1940 PRINT "
1950 PRINT "Number of Measurements = " N
1960 PRINT "
1970 LI$ = "LIQUID CONDUCTIVITY ="
1980 MA$="GAS HOLDUP (MAXWELL)="
1990 BR$="GAS HOLDUP (BRUGGEMANN)="
2000 P$="GAS HOLDUP (PRESSURE)="
2010 PRINT MA$,EG1
2020 PRINT "S.D.", SDGC
2030 PRINT BR$,EG3
2040 PRINT "S.D.", SDGC2
2050 PRINT P$,EG2
2060 PRINT "S.D.", SDGP
2070 B$="TEST SPECIFICATIONS:"
2080 V= "GAS RATE (L/M)="
2090 W= "FROTHER CONC. (PPM) = "
2100 SA$="NUMBER OF SAMPLES="
2110 PRINT "
        SAVE THE DATA IN A FILE
```

2120 INPUT "lotus file name"; F\$:F\$=F\$+".prn"

2130 INPUT "Test Specifications";L\$ 2140 INPUT "Gas Rate";K\$ 2150 INPUT "Frother Conc.";M\$ 2160 OPEN F\$ FOR OUTPUT AS#1 2170 PRINT #1,F\$ 2180 PRINT " 2190 PRINT #1,B\$,SPC(1)L\$ 2200 PRINT #1,V\$,SPC(1)K\$ 2210 PRINT #1,W\$,SPC(1)M\$ 2220 PRINT #1,SA\$,N 2230 PRINT " 2240 X = STRING\$(2,45) 2250 PRINT #1, SPC(18) X\$"Cond."X\$, SPC(4) X\$"PB"X\$, SPC(2) X\$"PT"X\$,X\$"PD"X\$ 2260 PRINT #1,SPC(18)X\$"(mS)"X\$,SPC(3)X\$"(psi)"X\$,SPC(1) X\$"(psi)"X\$,X\$"(psi)"X\$ 2270 FOR J=1 TO N 2280 PRINT #1,NO(J),USING "##############";COND(J),P1(J),P2(J),P1(J)-P2(J) 2290 NEXT J 2300 X\$=STRING\$(2,45) 2310 PRINT #1, SPC(18) X\$"Cond. "X\$, SPC(4) X\$"PB"X\$, SPC(2) X\$"PT"X\$,X\$"PD"X\$ 2330 PRINT #1,"S.D.",USING "#############";SD1,SD2,SD3,SDD 2350 PRINT #1,LI\$,KL 1 2360 PRINT #1, "S.D.", KLS 2370 PRINT #1, MA\$, EG1 2380 PRINT #1,"S.D.",SDGC 2390 PRINT #1, BR\$,EG3 2400 PRINT #1, "S.D.", SDGC2 2410 PRINT #1, P\$,EG2 2420 PRINT #1, "S.D.", SDGP 2430 CLOSE #1: END

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### Heat Transfer in the Column and Added Section

The thermal convection coefficient (q) for the two sections is the same. In other words, the amount of heat per unit area per unit time which transfers from the outside to both sections is equal. In the present study, the temperature of the water usually was lower than the outside temperature (Figure 3.39). If heat loss due to the walls of the two sections is assumed to be negligible, the total amount of heat which transfers from the outside to the water will be equal to:

$$Q_{1} = qA_{1}t = q\pi D_{1}Lt = m_{1}C_{p}\Delta T_{1}.$$
(3.39)

Similarly for the added section:

$$Q_{2} = qA_{2}t = q\pi D_{2}Lt = m_{2}C_{p}\Delta T_{2}$$
(3.40)

where:

~

Q:	total neat
A:	surface area
t:	time
D:	column diameter
C <sub>p</sub> :	specific heat of water

4 + 4 - 1 1 - - 4

ΔT: temperature difference

L: unit length

m: mass of water

(subindices 1 and 2 refer to the column and added section, respectively).



Figure 3.39: Heat transfer in the column and added section.

Substituting for  $m_1$  and  $m_2$  yields:

$$q \pi D_1 Lt = \rho \pi \frac{D_1^2}{4} LC_p \Delta T_1$$
 (3.41)

and

$$q\pi D_2 Lt = \rho \pi \frac{D_2^2}{4} LC_p \Delta T_2$$
 (3.42)

where  $\rho$  is the density of water. Dividing Equation 3.41 by Equation 3.42 gives:

$$\frac{T_{12} - T_1}{T_{22} - T_1} = \frac{D_2}{D_1}$$
(3.43)

where:

ŝ

T1:	initial temperature of water
T <sub>12</sub> :	temperature of water in the column
T <sub>22</sub> :	temperature of water in the added section.

Since  $D_1 \ge D2$ , substituting and rearranging give:

$$T_{22} > T_{12}$$
 (3.44)

This clearly explains the higher conductivity in the added section.

## Appendix 3.3

## **Calibration of the Thermistors**



Figure 3.40: Calibration of the thermistors.

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# **CHAPTER 4**<sup>\*</sup>

# DETERMINATION OF HOLDUP OF FLAKE-SHAPED PARTICLES IN SOLID-WATER SYSTEMS USING CONDUCTIVITY

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<sup>\*</sup>A version of this chapter is to be published in *International Journal of Mineral Processing* (accepted, October 1993).

# Nomenclature

a, b, c	three half axes of an ellipsoid
f	solids holdup
S	standard deviation, %
SS	sum of squares
<b>W</b>	parameter in Equation 4.2
β	parameter defining particle shape (Equation 4.1)
€s	solids holdup, %
Kc	conductivity of continuous phase, mS/cm
ĸ <sub>d</sub>	conductivity of dispersed phase, mS/cm
κ <sub>m</sub>	conductivity of dispersion, mS/cm
arphi	parameter in Equation 4.3



## **CHAPTER 4**

# DETERMINATION OF HOLDUP OF FLAKE-SHAPED PARTICLES IN SOLID-WATER SYSTEMS USING CONDUCTIVITY

### Abstract

The volume fraction (holdup) of mica and graphite in water slurries has been determined in a continuously operating column (10 cm in diameter and 447 cm in height), using an electrical conductivity-based method. The results were checked against those given by a pressure method and the actual volume fraction of solids determined by an isolating technique.

The shape of the dispersed particles significantly affected the conductivity of the dispersion. Neither Maxwell's (1892) nor Bruggeman's (1935) model yielded acceptable estimates of holdup. Fricke's (1924) model, which takes the shape of the particles into account, adequately predicted solids holdup (5-12% v/v in this case). The required shape factor for Fricke's model was obtained by SEM (scanning electron microscope) analysis of several particles. In the case of graphite, an estimation of particle conductivity was required which was obtained by back-calculation.

## 4.1 Introduction

The electrical conductivity method has been used to estimate the volume fraction of dispersed phase (or holdup) in solid-water dispersions (e.g., Meredith and Tobias, 1962; Naser-El-Din et al., 1987; Marchese et al., 1992; Uribe-Salas, et al., 1992). Most studies have considered spherical, or near-to-spherical particles for which the Maxwell (1892) or Bruggeman (1935) model is adequate (Banisi et al., 1993). The holdup of flake-shaped materials is examined in this chapter, as represented by mica and graphite, the latter providing the added complication of being a conductor. It is shown that the Maxwell and Bruggeman models cannot be employed but the model of Fricke (1925a, 1925b, 1953, 1954) which accounts for shape is valid and permits an accurate estimation of solids holdup. The study was prompted by the potential use of conductivity in on-line holdup estimation in flotation systems.

## 4.2 Theory

Fricke's dispersion conductivity model takes into account the effect of particle shape. Shape is described by the three half axes a, b and c of an ellipsoid. The model for an arbitrary orientation of ellipsoids to the field direction is:

$$\left(\frac{\kappa_{\rm m} - \kappa_{\rm c}}{\kappa_{\rm d} - \kappa_{\rm m}}\right) \left(\frac{\kappa_{\rm d}}{\kappa_{\rm c}} - 1\right) = \frac{\beta f}{1 - f}$$
(4.1)

where

$$\beta = \frac{1}{3} \left[ \frac{2}{1 + \left(\frac{\kappa_{d}}{\kappa_{c}} - 1\right) \frac{1}{2} W} + \frac{1}{1 + \left(\frac{\kappa_{d}}{\kappa_{c}} - 1\right) (1 - W)} \right] \left(\frac{\kappa_{d}}{\kappa_{c}} - 1\right)$$
(4.2)

and

$$W(a < b) = \frac{\left(\varphi - \frac{1}{2}\sin 2\varphi\right)}{\sin^3 \varphi} \cos \varphi , \ \cos \varphi = \frac{a}{b}$$
(4.3)

- $\kappa_{\rm m}$  : conductivity of dispersion
- $\kappa_{c}$  : conductivity of continuous phase (liquid)
- $\kappa_d$  : conductivity of dispersed phase (solid)
- f : volume fraction of dispersed phase.

Knowing a/b,  $\kappa_d$ ,  $\kappa_m$  and  $\kappa_c$  the holdup can be estimated using Equation 4.1. As an independent check on the holdup estimated by conductivity, holdup was also estimated by a pressure difference technique (Fan, 1989) and by direct measurement using the isolating technique (Uribe-Salas et al., 1993).

## 4.3 Experimental Section

#### 4.3.1 Apparatus and Procedure

The apparatus (essentially a flotation column) used in this work (Figure 4.1) was made of Plexiglas, 4.47 m in height and 10 cm in diameter. The middle 0.93 m section was chosen for the holdup estimation in order to limit disturbances due to feed and gas injection. Two pressure transducers were installed to measure the pressure at the top and the bottom of the section. Two grid electrodes, shown elsewhere to be suited to measuring dispersion conductivity (Uribe-Salas et al., 1993), were placed close to the pressure transducers. All pressure and conductivity signals were collected by a data acquisition system. Two air-actuated ball valves with an internal diameter of 10 cm were used to isolate the section. The required pressure to operate the valves was 827 kPa which resulted in a response time of 250 ms. The feed was introduced to the column near the top and the underflow was recycled via a reservoir. Masterflex pumps (model



Figure 4.1: Laboratory column set-up.

720-33) were used to feed the column and a stirrer was used in the reservoir to mix the slurry and keep the solids in suspension.

Estimation of solids holdup using conductivity-based models requires two conductivity measurements, that of the clear (solids free) water and the solid-water mixture. Simultaneous measurement of the two conductivities is desirable. Two approaches were taken for the on-line measurement of the conductivity of the clear water.

In the first approach, a Plexiglas tube, 1.5 cm in diameter and 1.2 m in height (above the top of the column), was put in the feed line (item 6, Figure 4.1). In this way, a portion of the feed was directed to the tube. Two grid electrodes were installed in this tube (the bottom one 20 cm above the feed line). Since water in the tube was not in motion, particles settled out before reaching the electrodes (this was particularly evident for the coarse particles (96% +75  $\mu$ m) used in the present study). As a result, the conductivity of clear water was measured.

In the second approach, two grid electrodes inside the column, the bottom one 20 cm above the feed inlet, were installed (Figure 4.1). It was found that the conductivity of the clear water was the same in both approaches.

The pressure sensors proved susceptible to noise, a problem traced to the underflow pump. It was virtually eliminated by increasing pump speed, introducing a constriction downstream and increasing the frequency and number of pressure measurements (60 readings over 3 minutes). Pressure and conductivity over four consecutive time intervals were compared using an algorithm in the data acquisition system. When the variations were equal or close to the expected error (0.004 kPa for pressure and 0.008 mS/cm for conductivity), it was assumed steady-state had been reached. Between 10 and 15 readings of pressure and conductivity were taken for each point. The maximum time between each of the measurements was 3 seconds.

The valves were then closed simultaneously to isolate the sample between the electrodes which was removed, weighed, filtered and dried to determine the actual volume fraction of solids (i.e., holdup).

#### 4.3.2 Data Acquisition

The data acquisition system consisted of the following:

- a microcomputer (286, IBM compatible, 1MB of memory),
- a 24-channel relay board (Metrabyte, model ERB-24),
- an I/O interface board to control the relay board (Metrabyte, model PI-12),
- an A/D convertor interface board (Metrabyte model DAS-8PGA),
- a conductivity meter (Tacussel, model CD-810),
- two pressure transducers (Omega, PX 440), and
- two power supplies (U24Y100) to activate the pressure transducers.

The pressure transducer range was 0-34.5 kPa (0-5 psi) and the output signal was a 0 to 4 mA current. Programs in GWBASIC were developed for each task. Since the response time of the conductivity meter was about 3 seconds, the time interval between samples was usually selected as 3 seconds or more. During the experiment each measurement of the conductivity and pressure was displayed on a monitor. This provided a convenient means of detecting any disturbance to the system, e.g., a sudden change in the pressure reading could be traced, for example, to a variation in the feed flowrate. Moreover, it provided a clear picture of the effects of the different variables on the measured parameters. For each set of measurements the average, standard deviation and relative error were calculated.
## 4.1.3 Mica and Graphite Characteristics

Mica from Suzorite Mica Products Inc. (Québec) and Graphite from Stratmin Graphite Inc. (Mont-Laurier, Québec) were used. Their size distribution is shown in Table 4.1, showing that 95% was between 425  $\mu$ m and 106  $\mu$ m. The density of mica and graphite was 2.83±0.01 g/cm<sup>3</sup> and 2.34 ±0.01 g/cm<sup>3</sup>, respectively, as determined with a gas pycnometer.

Table 4.1: Size distribution of mica and graphite.

	Size (µm)	425/300	+212	+150	+106	+75	+ 53	+38	-38
Mica	Weight (%)	0.00	19.35	40.59	30.61	5.77	1.72	0.92	1.00
Graphite	Weight (%)	11.13	38.65	37.90	6.57	2.03	1.67	0.94	1.11

The Fricke model requires a shape factor, taken as the geometric aspect ratio (thickness to diameter ratio or a/b) of the particles. To estimate the shape factor of the mica and graphite flakes, well dispersed samples were analyzed with a scanning electron microscope (SEM JEOL 840). Some of the flakes were positioned vertically on the sample mount to measure their thickness (Figures 4.2 and 4.3). The thickness to diameter ratio for both mica and graphite was about 1 to 20 (a/b = 0.05).

The conductivity of graphite is required in Fricke's model ( $\kappa_d$  for mica is ~0). This measurement requires a special experimental set-up (Klein, 1966; Kelly, 1981; Deprez and McLachlan, 1988). In the present case, the conductivity was back-calculated from Fricke's model using the directly measured graphite holdup.



Figure 4.2a: Top-view of a mica flake (SEM micrograph).



Figure 4.2b: Edge-view of a mica flake (SEM micrograph).



Figure 4.3a: Top-view of a graphite flake (SEM micrograph).



Figure 4.3b: Edge-view of a graphite flake (SEM micrograph).

## 4.4 RESULTS AND DISCUSSION

### 4.4.1 Mica Holdup Estimation

Figure 4.4 shows the mica holdup estimation from the conductivity and the pressure difference methods; both agree well with the actual holdup obtained using the isolating technique. Table 4.2 presents typical results with their corresponding standard deviations.

Conductivity		Pressure Dif	Actual	
Estimated e, (%)	S (%)	Estimated $\epsilon_{s}$ (%)	S (%)	ε, (%)
3.23	0.48	3.85	0.69	3.41
5.59	0.51	5.34	1.12	4.96
6.95	0.55	7.42	1.18	7.12
7.86	0.59	8.29	3.33	8.49
8.18	0.60	9.69	1.65	9.30
10.02	0.67	10.53	1.91	10.23

 
 Table 4.2: Typical mica holdup estimation from conductivity and pressure difference methods and their respective standard deviations.

To illustrate the effect of the shape on the mica holdup estimation, Maxwell's and Bruggeman's models were also used to estimate the holdup (Figure 4.5). Holdup values from both models compare poorly with actual holdups (about 160% error). Unlike Fricke's model, the Maxwell and Bruggeman models assume spherical particles: this observation suggests that close attention must be paid to particle shape if it is flaky.

The sensitivity of the holdup estimation to the shape factor (a/b) was examined by re-calculating for two other cases: a/b=0.03 and a/b=0.07 (Figure 4.6). Unlike the a/b=0.05 case, the other two deviated significantly from the actual holdup. This implies that Fricke's model may be of a potential interest to determine particle shape (a/b).

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Figure 4.4: Mica holdup estimation using conductivity and pressure difference methods.



Figure 4.5: Mica holdup estimation using conductivity method.

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Figure 4.6: Sensitivity of mica holdup estimation to shape factor (a/b) using Fricke's model.

## 4.4.2 Graphite Conductivity by Back-Calculation

The first requirement is a value for graphite conductivity. Conductivity was estimated by back-calculation using Equation 4.1, knowing f, a/b,  $\kappa_c$  and  $\kappa_m$ . The average value from eight tests over a range of 1-9% solids v/v was 10.82±1.81 mS/cm. Reported values of graphite conductivity range from 0.70 to 50 mS/cm for loose powders (Kelly, 1981) to 90,000±9,000 mS/cm to 1,130,000±110,000 mS/cm for compacted powders (Deprez and McLachlan, 1988). The estimated value here may be considered as related to randomly oriented graphite particles. The wide variation in published values makes verification of graphite's conductivity difficult.

### 4.4.3 Graphite Holdup Estimation

The estimated graphite holdup using the conductivity (with Fricke's model) and pressure methods vs. the actual holdup is shown in Figure 4.7. Table 4.3 shows typical estimated graphite holdups from the conductivity and the pressure difference methods with their respective standard deviations. The Maxwell and Bruggeman models again failed to describe the system (Figure 4.8); for example, back-calculating  $\kappa_d$  using these models gave *negative* values.

Conductivity		Pressure Di	Actual	
Estimated e, (%)	S (%)	Estimated $\epsilon$ , (%)	S (%)	ε, (%)
2.90	0.31	4.10	0.78	3.48
4.01	0.39	5.01	1.89	4.57
5.40	0.23	6.35	0.41	5.72
7.17	0.32	7.24	2.29	7.46
8.82	0.67	9.21	2.00	10.20
10.85	0.75	11.75	1.60	12.27

 Table 4.3: Typical graphite holdup estimation from conductivity and pressure difference methods and their respective standard deviations.



Figure 4.7: Graphite holdup estimation using conductivity and pressure difference methods.

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Figure 4.8: Graphite holdup estimation using conductivity method.

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In the case of graphite both shape (a/b) and conductivity are important variables. Figure 4.9 shows the sensitivity of holdup estimation to a/b with the corresponding backcalculated value of graphite conductivity. There appears to be less sensitivity to a/b compared to the mica case (Figure 4.6) but this is because the back-calculated  $\kappa_d$  accounts for some of the "error". The possibility exists for simultaneous estimation of shape and the conductivity of flake-shaped particles relevant to their orientation in water dispersions.

Figure 10 presents the role of a/b in a different manner. The minimum in SS  $(\Sigma(f_{actual} - f_{estimated})^2)$  corresponds to a/b ~0.05 which could be taken as the best estimate of a/b; however, over the range 0.02 < a/b < 0.07 all model fits could be considered acceptable.

#### 4.5 CONCLUSIONS

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1- To estimate the holdup of mica and graphite flakes in water dispersions using conductivity, Fricke's model, which accounts for particle shape, was found suitable. The Maxwell and Bruggeman models (the classical ones for a spherical dispersed phase) were in error by up to 160%.

2- The use of dispersion conductivity to estimate particle shape when the particle conductivity is known (as is the case with mica where  $\kappa_d=0$ ) is suggested.

3- When particle conductivity is unknown, as with graphite, simultaneous estimation of shape and particle conductivity may be possible from measurement of dispersion conductivity. The estimated particle conductivity will correspond to that orientation the particles adopt in suspension, which may have a practical use.



Figure 4.9: Sensitivity of graphite holdup estimation to shape factor (a/b) using Fricke's model.

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Figure 4.10: Effect of shape factor on back-calculated graphite conductivity and sum of squares (SS).

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# **CHAPTER 5**<sup>\*</sup>

# **ON-LINE GAS AND SOLIDS HOLDUP ESTIMATION** IN SOLID-LIQUID-GAS SYSTEMS

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<sup>\*</sup>A version of this chapter is to be published in *Minerals Engineering* (accepted, February 1994).

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## Nomenclature

g	acceleration due to gravity, m s <sup>-2</sup>
L	volume, liter
L <sub>e</sub>	distance between pressure transducers, m
P <sub>A</sub>	pressure at point A, kPa
P <sub>B</sub>	pressure at point B, kPa
S	standard deviation
ε <sub>l</sub>	liquid holdup
€ <sub>s</sub>	solids holdup
$\epsilon_{\mathrm{g}}$	gas holdup
κ <sub>si</sub>	conductivity of slurry, mS/cm
κ <sub>sl-g</sub>	conductivity of slurry-gas mixture, mS/cm
$ ho_{l}$	density of liquid, kg/m <sup>3</sup>
$ ho_{s}$	density of solid, kg/m <sup>3</sup>
$ ho_{ m g}$	density of gas, kg/m <sup>3</sup>

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## **CHAPTER 5**

# ON-LINE GAS AND SOLIDS HOLDUP ESTIMATION IN SOLID-LIQUID-GAS SYSTEMS

## Abstract

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A technique for on-line simultaneous estimation of gas and solids holdup (volume fraction), based on a combination of conductivity and pressure difference measurements, is proposed and tested in a laboratory flotation column (10 cm in diameter and 447 cm in height). Fundamental concepts underlying this technique and the effect of relevant parameters are discussed. In the studied range, 3-30% v/v gas holdup and 2-18% v/v solids holdup, experimental results showed good agreement between the estimated and actual holdup values (as determined by an isolating technique). The relative accuracy of the solids holdup estimate increased when solids density or solids concentration increased. It was shown that for a given system there is a minimum distance between the pressure sensors to achieve a given relative accuracy of the holdup estimate.

## 5.1 Introduction

Flotation is an example of a three-phase (solid-liquid-gas) system. The relative magnitude of the phase holdups (i.e., volumetric fraction of each phase) in part governs the flow regime and metallurgical performance. Consequently, measurement of the three phase holdups would provide data for fundamental modelling and in-plant would permit diagnosis and eventually new control strategies to be designed. Measurement in batch systems is reasonably straightforward (Fan, 1989) but flotation is a continuous process and no on-line technique to date has been demonstrated.

The problem can be stated quite succinctly: to determine the three holdups simultaneously three equations are required. One obvious equation is the overall or global volume balance, i.e., the sum of all holdups is unity. The remaining two, in principle, can come from signals dependent on the holdups, provided the appropriate sensor can be constructed and appropriate model relating the signal to the holdups can be developed.

Some of the possible sensors are summarized in Table 5.1. Two of those, based on conductivity and pressure, appear to be the most promising. They are essentially standard techniques for liquid-gas systems (Fan, 1989) and both have been used in at least solids-liquid systems (see Chapter 3). Uribe-Salas et al. (1993) have shown that the conductivity technique is applicable to gas holdup measurement in the three-phase systems typical of flotation, i.e., bubbles less than about 2-3 mm stabilized by frother and solids less than 200-300  $\mu$ m. The pressure technique has the dual virtue of using widely available sensors and having a simple model relating signal to holdup.

In this chapter it is demonstrated that reliable estimates of the three phase holdups, simultaneously and on-line, can be made by combining the global volume balance equation with equations based on conductivity and pressure signals. Since the

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method involves combining measurements, an error analysis is included to determine confidence levels and provide a guide to the application of the procedure.

Method	References	Comments
Conductivity	Serizawa, et al., 1975; Fan, 1989; Uribe- Salas et al., 1993	liquid-gas; solid-liquid; solid- liquid-gas
Resistivity	Burgess and Calderbank, 1975; Fukuma et al., 1987; Otero and Fuente, 1991	gas holdup; bubble velocity and size
Optical Fibre	Vince et al., 1982; Ishida and Tanaka, 1982; Hu et al., 1985; Lee and De Lasa, 1986; Wachi et al., 1987	liquid-gas
Attenuation of $\beta$ (or $\gamma$ )-rays	Nassos, 1963; Lee and Worthington, 1974; Lockett and Kirkpatrick, 1975	liquid-gas
Laser Scattering	Soto,1989	liquid-gas
Ultrasonics	Stravs and Von Stockar, 1985	liquid-gas
Pressure	Fan, 1989	liquid-gas; solid-liquid

#### Table 5.1: Sensors used to measure phase holdup.

## 5.2 Theoretical Considerations

## 5.2.1 Global Volume Balance Equation

This equation is simply:

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$$\epsilon_{\rm s} + \epsilon_{\rm l} + \epsilon_{\rm g} = 1$$

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where  $\epsilon_i$  is holdup of i (solid, liquid, gas).

## (5.1)

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## 5.2.2 Conductivity Equation

Uribe-Salas et al. (1993) developed the appropriate cell and showed that Maxwell's two-phase model (Maxwell, 1892) could be modified for three-phase systems to give gas holdup by treating the solid-liquid (slurry) as a continuous phase:

$$\epsilon_{g} = \frac{1 - \frac{\kappa_{sl-g}}{\kappa_{sl}}}{1 + 0.5 \frac{\kappa_{sl-g}}{\kappa_{sl}}}$$
(5.2)

where:

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 $\kappa_{sl-g}$ : conductivity of slurry and gas mixture (mS/cm)  $\kappa_{sl}$ : conductivity of slurry (mS/cm)  $\epsilon_{g}$ : gas holdup.

To estimate  $\epsilon_g$ , therefore, requires two conductivity measurements, that of the slurry alone and that of the slurry-gas mixture (or dispersion).

## 5.2.3 Pressure Equation

The pressure difference between two vertically spaced points (A and B), is given by (assuming dynamic component of pressure is negligible):

$$\frac{\mathbf{P}_{\mathrm{B}} - \mathbf{P}_{\mathrm{A}}}{\mathrm{L}} = g(\rho_{\mathrm{g}}\epsilon_{\mathrm{g}} + \rho_{\mathrm{I}}\epsilon_{\mathrm{I}} + \rho_{\mathrm{s}}\epsilon_{\mathrm{s}})$$
(5.3)

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where:

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 $\rho_i$ : density of i (gas, liquid, solid) (g/cm<sup>3</sup>)

 $\epsilon_i$ : holdup of i

g: acceleration due to gravity  $(cm/s^2)$ 

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L: vertical distance between two points (cm).

Since  $\rho_g \ll \rho_s, \rho_i$ , it is assumed that  $\rho_g = 0$  and substituting  $\epsilon_i$  for  $\epsilon_s$  and  $\epsilon_g$  using Equation 5.1, Equation 5.3 can be re-arranged to give  $\epsilon_s$ :

$$\epsilon_{\rm s} = \frac{\frac{\Delta P}{L} - g \rho_1 (1 - \epsilon_{\rm g})}{g (\rho_{\rm s} - \rho_{\rm l})}$$
(5.4)

where  $\Delta P = P_B - P_A$ .

### 5.2.4 The Method

The estimation of the gas and solids holdup is accomplished in two steps: Step 1- Gas holdup is estimated from the conductivity measurements using Equation 5.2; Step 2- Solids holdup is estimated from the pressure measurements using Equation 5.4 after substituting the estimate of  $\epsilon_{g}$  from Equation 5.2.

#### 5.2.5 Error Analysis

Since the method involves error propagation in estimating gas, and in particular solids holdup, its impact on the confidence interval of the estimates needs to be considered. The variance of the estimated phase holdup is calculated by expanding each function as a Taylor series about the mean. If only the zero and first order terms of the expansion are considered and the covariances between the variables are assumed to be zero, the variance ( $S^2$ ) of the gas holdup estimation using Equation 5.2 is equal to:

$$\mathbf{S}_{\epsilon_{g}}^{2} \approx \left(\frac{\partial \epsilon_{g}}{\partial \kappa_{sl-g}}\right)^{2} \mathbf{S}_{\kappa_{sl-g}}^{2} + \left(\frac{\partial \epsilon_{g}}{\partial \kappa_{sl}}\right)^{2} \mathbf{S}_{\kappa_{sl}}^{2}$$
(5.5)

which gives:

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$$S_{\epsilon_{g}}^{2} \approx \left(\frac{-1.5 \kappa_{sl}}{(\kappa_{sl} + 0.5 \kappa_{sl-g})^{2}}\right)^{2} S_{\kappa_{sl-g}}^{2} + \left(\frac{1.5 \kappa_{sl-g}}{(\kappa_{sl} + 0.5 \kappa_{sl-g})^{2}}\right)^{2} S_{\kappa_{sl}}^{2}.$$
 (5.6)

For the solids holdup estimation from Equation 5.4, the variance is:

$$\mathbf{S}_{\epsilon_{s}}^{2} \approx \left(\frac{\partial \epsilon_{s}}{\partial \Delta \mathbf{P}}\right)^{2} \mathbf{S}_{\Delta \mathbf{P}}^{2} + \left(\frac{\partial \epsilon_{s}}{\partial \epsilon_{g}}\right)^{2} \mathbf{S}_{\epsilon_{g}}^{2}$$
(5.7)

which yields:

$$S_{\epsilon_s}^2 \approx \left(\frac{1}{gL(\rho_s - \rho_l)}\right)^2 S_{\Delta P}^2 + \left(\frac{\rho_l}{(\rho_s - \rho_l)}\right)^2 S_{\epsilon_g}^2.$$
(5.8)

## 5.3 Experimental Section

### 5.3.1 Apparatus

The flotation device used in this work was a column (Figure 5.1) made of Plexiglas, 4.47 m in height and 10.18 cm in diameter. Bubbles were generated with a porous ( $-10 \mu$ m holes) stainless steel sparger at the bottom of the column. The middle 0.94 m section was chosen for the holdup estimation in order to limit disturbances due to feed and gas injection. Two air-actuated valves were used to isolate the middle (sampling) section. The required pressure to operate the valves was 827 kPa which resulted in a response time of 250 ms. A calibrated Tylan gas flowmeter (model FM-380) and MIC 2000 controller were used to measure and display the air flowrate.

Conductivity cells of two stainless steel grid electrodes, shown elsewhere to be suited to measuring dispersion conductivity (Chapter 3; Turner, 1976; Marchese et al., 1992; Uribe-Salas et al., 1993), were placed in the feed line and in the middle section of the column. The cell in the column was 10.18 cm x 74.00 cm; the one in the feed



Figure 5.1: Laboratory flotation column set-up.

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line was 1.29 cm x 1.19 cm. Two pressure transducers (0-5 psi, Series 440X- Omega) were installed to measure the pressure at the top and the bottom of the sampling section. The operation of the valves, gas injection, pressure and conductivity measurements were controlled by computer. All conductivity signals were collected by a data acquisition system which consisted of a microcomputer (IBM compatible, 1MB of memory), a 24-channel relay board (Metrabyte, model ERB-24), an I/O interface board to control the relay board (Metrabyte, model PIO-12), an A/D converter interface board (Metrabyte, model DAS-8GPA) and a conductivity meter (Tacussel, model CD-810). Masterflex pumps (model 720-33) were used to feed the column from a 60 L baffled feed tank equipped with a stirrer (Lightnin-Type TPR).

## 5.3.2 Materials

The gas and liquid phases were air and tap water, respectively. Three types of solid particles were used: calcite from Steep Rock Resources Inc. (Ontario), silica from Indusmin Inc. (Québec) and coal from QIT-Fer et Titane (Québec). Their size distributions are shown in Table 5.2.

The density of calcite, silica and coal was  $2.72 \pm 0.01$  g/cm<sup>3</sup>,  $2.64 \pm 0.01$  g/cm<sup>3</sup>, and  $1.79 \pm 0.07$  g/cm<sup>3</sup>, respectively, as determined with a gas pycnometer. In all tests 25 ppm of frother (Dowfroth 250 C) was used unless otherwise specified.

Size (µm)		Weight (%)							
	+150	+106	+75	+53	+38	+25	-25		
Calcite	0	0	2.00	3.35	20.42	25.34	48.89		
Silica	0	0	3.34	11.79	24.19	10.73	49.95		
Coal	0	1.25	5.00	9.86	15.84	20.59	47.55		

Table 5.2: Size distribution of calcite, silica and coal.

#### 5.3.3 Procedure

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Phase holdup estimation from conductivity in continuous operation requires online measurement of the dispersion (slurry-air in this case) and continuous phase (slurry) conductivity. In the present set-up, the slurry-air conductivity was measured in the column and that of the slurry alone in the feed line (Figure 5.1, item 3). Steady-state operation was tested by measuring and comparing the conductivity of the system over four consecutive time intervals. When the difference between them was equal or close to the expected error (0.004 mS/cm based on A/D board resolution), it was assumed that steady-state had been reached and the pressure and conductivity measurements were taken. The gas holdup was determined following the method described, i.e.,  $\epsilon_g$  was obtained from Equation 5.2, and the solids holdup was obtained from Equation 5.4 (see Appendix 5.1 for a specimen calculation).

Upon completion of the conductivity and pressure measurements, the valves were simultaneously closed to isolate the section. To determine the actual phase holdup of the sample isolated between the valves, a specially designed vessel was used. The vessel was constructed by joining a 5 cm-diameter graduated cylinder (2.65 L) with a tapered tank (5.58 L); the total volume was equal to the volume of the sampling section (i.e., the volume between the two valves, 8.23 L). The volume and the weight of the sample were measured in the vessel and knowing the density of the phases, the holdup of each phase was calculated and taken as the actual value. The sample was returned to the reservoir after the holdup determination. This method of actual holdup measurement not only allowed continuous operation of the column without interruption but also preserved a constant solids concentration during the experiment.

During conductivity measurements, to avoid possible electrical interference from the pressure transducers a relay board (Metrabyte, ERB-24) was used to switch off the power supply to the pressure transducers while measuring the conductivity. The

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reciprocal precaution was taken when making pressure measurements. All experiments were performed by taking 10-15 readings with a time interval of 3 seconds. For each set of measurements the average, standard deviation and relative error were calculated.

## 5.4 **Results and Discussion**

#### 5.4.1 Phase Holdup Estimation

Gas holdup determination for the silica-gas-water system is presented in Figure 5.2. For the studied range (4-26% v/v), the estimated values were in good agreement with the actual gas holdup. The average absolute standard deviation was  $\pm 0.25\%$  v/v. The accuracy of the gas holdup estimation (R=0.9715) confirmed the assumption that the slurry phase can be treated as a single phase (i.e., continuous phase) in Maxwell's model. Figure 5.3 shows the silica holdup estimation. The estimated values were in a reasonable agreement (R=0.9410) with the actual solids holdup. The agreement is not as good as in the gas holdup case because solids holdup estimates require both conductivity and pressure measurements, resulting in error propagation. Typical results of the gas and solids holdup estimation and their corresponding error (standard deviation, S) using Equations 5.2, 5.4, 5.6 and 5.8 are presented in Table 5.3.

Conductivi	ty (mS/cm)	Gas Ho	oldup (%_v/v)	Pressu	re (Pa)	Solids II	oldup (% v/v)
κ <sub>sl</sub> ±S	κ <sub>uls</sub> ±S	Actual	Estimated	$P_{B} \pm S$	P <sub>A</sub> ±S	Actual	Estimated*
0.2454±0.0020	0.2192±0.0002	10.02	9.62±0.50	21486±24	13674±30	3.97	4.11±0.42
0.2662±0.0003	$0.2065 \pm 0.0003$	16.35	16.16±0.11	24253±22	15375±15	17.65	16.03±0.22
$0.2401 \pm 0.0001$	$0.1645 \pm 0.0004$	22.65	23.45±0.14	19576±75	12417±75	8.58	7.55±0.82

Table 5.3: Typical conductivity and pressure measurements with actual and estimated phase holdups.

\* L=81.9 cm, g=980.66 cm/s<sup>2</sup>

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Figure 5.2: Gas holdup estimation in a silica-gas-water system.

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Figure 5.3: Solids holdup estimation in a silica-gas-water system.

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Further examination of the technique was performed on a calcite-gas-water system. Table 5.4 presents typical results of phase holdup estimation with the corresponding standard deviation. As in the case of silica, the estimated holdups agreed well with the actual values (for gas holdup estimation R=0.9629 and for solid holdup estimation R=0.8415).

Test	Actual Gas	Estimated Gas	Actual Solids	Estimated Solids
No.	Holdup (%v/v)	Holdup (%v/v)	Holdup (%v/v)	Holdup (%v/v)
1	10.34	11.55±0.17	16.32	14.93±0.51
2	13.12	12.68±0.25	9.71	8.23±0.58
3	15.24	16.75±0.18	16.37	$14.74 \pm 0.40$
4	19.98	$20.41 \pm 0.33$	7.51	8.96±0.39
5	24.93	26.86±0.16	3.56	4.48±0.53

Table 5.4: Typical holdup estimation in the calcite-gas-water system.

It appears that for similar gas holdup values (see Tests 1 and 2), the estimation of solids holdup is relatively more accurate at higher solids concentrations.

To test the technique for the case of hydrophobic particles, coal was chosen. The results of gas holdup and solids holdup estimation are shown in Figures 5.4 and 5.5, respectively. For the range 5-30% v/v gas holdup, the estimated values were in good agreement (R=0.9593) with the actual gas holdup. Referring to Figure 5.5, the estimation of the coal holdup (R=0.6488) for the studied range (2-10% v/v), however, was not as accurate as in the silica and calcite cases.



Figure 5.4: Gas holdup estimation in a coal-gas-water system.



Figure 5.5: Solids holdup estimation in a coal-gas-water system.

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#### 5.4.2 Error Analysis

Gas Holdup: the relative error on gas holdup will decrease as gas holdup increases. Two examples are shown in Figure 5.6, one related to the conductivities here  $(\kappa_{sl} \approx 0.24, S = \pm 0.0004 \text{ mS/cm})$ , and a second from experience with an absolute conductivity about 10 times higher. Both show a similar trend. Taking a relative error of 5% as the maximum desirable, the minimum gas holdup for which the technique is acceptable is about 5%.

Solids Holdup: Figure 5.7a illustrates the effect of the relative error of the gas holdup on the relative error of the solids holdup estimation. To illustrate, for a constant gas holdup (12% v/v) and solids holdup (10% v/v), the effect of the density of the solid particles on the relative error on the solids holdup estimation was calculated (Figure 5.7a). It was assumed in these calculations that there is no error on the pressure measurements and only the error on the conductivity measurements caused the error on the gas holdup estimation decreases with increasing solids density. For example, for a 20% relative error on the gas holdup estimation is 13%, whereas, for a solids density of 3.2 g/cm<sup>3</sup> the error is reduced to 7%. An error in gas holdup estimation translates to a certain magnitude of pressure difference. Since the corresponding volume of solid equal to this pressure difference is larger for lower density solids, the error on the solids holdup estimation is correspondingly higher. This explains the higher error observed in the coal holdup estimation compared to the calcute and silica cases.

The effect of the solids concentration on the solids holdup estimation for a given error on the gas holdup estimation is presented in Figure 5.7b. At a constant gas holdup (12% v/v) and solids density, for a given error on the gas holdup estimation, the error on solids holdup estimation decreases with increasing solids holdup. For example, for



Figure 5.6: Relative error on gas holdup estimation vs. gas holdup.

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Relative Error on Gas Holdup Estimation (%)

Figure 5.7b: Effect of solids concentration on solids holdup estimation.

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a 20% relative error on the gas holdup estimation, the relative error on solids holdup estimation decreases from 18 to 6% when the solids holdup increases from 5 to 15% v/v.

Since the absolute error on the pressure measurements is approximately constant ( $\approx \pm 60$  Pa), the relative error decreases as the absolute pressure increases and correspondingly so does the relative error on solids holdup. Thus, any system parameter (e.g., solids concentration, solids density and distance between the pressure sensors) which increases the absolute pressure difference will reduce the relative error. Figure 5.8 presents the relative error on the solids holdup estimation vs. solids holdup, including the effect of solids density and the distance between the pressure sensors. An average absolute standard deviation of the gas holdup estimation (0.4%, relevant to conditions here) and of the pressure measurements (62 Pa) was used in the calculation.

Increasing the density of the solids decreases the relative error: the poorer estimates of holdup for coal compared to silica and calcite illustrate this effect. The relative error increases with decreasing distance between the pressure sensors: a distance as small as 20 cm should clearly be avoided. In the current situation the distance (L=81.9 cm) preserved acceptable accuracy over the tested range. Again taking 5% relative error as acceptable, the minimum solids holdup even for solids of density 2.66 g/cm<sup>3</sup> is about 8% for the present set-up. For any operation, a "master" graph like Figure 5.8 could serve as a useful tool for determining the spacing of the pressure sensors and understanding the level of accuracy of the estimates.

## 5.5 Concluding Remarks

The results have clearly shown that simultaneous on-line estimation of gas and solids holdup in a flotation system is possible. Laboratory conditions were used in the evaluation and this raises two points with regard to eventual transfer of the method to the

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Figure 5.8: Effect of solids density and the distance between the pressure sensors on the solids holdup estimation.

plant: the level of accuracy is higher in the laboratory, and the technique of measuring slurry conductivity in the feed line is probably not applicable in the plant. In the absence of plant measurement accuracy data the best that can be said regarding the expected minimum reliable holdup estimate is that it will be greater than 5% but probably less than 10% for both gas and solids.

The measurement of slurry conductivity in the absence of gas poses a significant challenge in an industrial unit especially if it is to be accomplished close to where the . slurry-air conductivity is to be measured (which is the desired situation to avoid a possible variation in the slurry conductivity between the two locations). A recently described cell (Gomez et al., 1993) may provide the solution to this problem and is the subject of current testwork.

### 5.6 Conclusions

1) A technique for on-line simultaneous estimation of gas and solids holdup was proposed and tested in a laboratory flotation column. The approach is based on a combination of conductivity and pressure difference measurements.

2) Estimates of gas (3-30% v/v) and solids (2-18% v/v) holdup using this technique were in good agreement with actual values.

3) The technique is recommended for gas holdup  $\geq 5\%$  v/v and, for the present set-up, for solids holdup  $\geq 8\%$  v/v.

4) The relative accuracy of the solids holdup estimation increased with increasing solids density and solids concentration.

5) There is a minimum distance between the pressure sensors for any operation below which the accuracy of the solids holdup estimation is significantly reduced.

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## Appendix 5.1

#### **Specimen Calculation**

• Phase Holdup Estimation from the Signals

The following measurements were made at a given set of conditions:

$$a_{sl} = 0.2279 \text{ mS/cm}$$

 $\kappa_{\rm sl-g} = 0.1805 \, {\rm mS/cm}$ 

 $P_{A} = 215.04 \text{ kPa}$ 

,

 $P_{\rm B} = 136.95 \, \rm kPa$ 

Step 1. Gas holdup from Equation 5.2. Substitution in Equation 5.2 gives the gas holdup  $\epsilon_{g} = 14.91 \%$ .

Step 2. Solids holdup from Equation 5.4. Substituting in Equation 5.4, where L=80.9 cm, g=980.66 cm/s<sup>2</sup>, $\rho_1$ =1 g/cm<sup>3</sup> and  $\rho_s$ =2.72 g/cm<sup>3</sup> (calcite), results in solids and liquid fractional holdups of 0.08033 and 0.7706, respectively.

• Phase Holdup Determination by the Isolating Technique

For the following measured volume and weight of the sample isolated by the valves:

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W = 8.011 Kg

the volume of gas was obtained by subtracting the measured volume from the total volume between the valves (8.230 L). Thus, the gas holdup is 0.1535 (=1.263/8.230). The calcite holdup (0.0737) was calculated from:

$$\epsilon_{s} = \frac{W - \rho_{1} V}{(\rho_{s} - \rho_{1}) V} (1 - \epsilon_{g})$$
(5.9)

## CHAPTER 6

### EFFECT OF SOLID PARTICLES ON GAS HOLDUP IN FLOTATION COLUMNS . .

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## Nomenclature

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Α	column cross-sectional area, cm <sup>2</sup>
Со	distribution parameter
d <sub>b</sub>	bubble diameter, cm
d <sub>c</sub>	column diameter, cm
g	acceleration due to gravity, cm/s <sup>2</sup>
Н	constant in Equation 6.23
J <sub>e</sub>	local mixture superficial velocity at the centre of column, cm/s
J <sub>i</sub>	local superficial velocity of the i <sup>th</sup> phase, cm/s
Ко	terminal velocity coefficient
m S	parameter in Equation 6.12
r	radial variable, cm
R	column radius, cm
Re <sub>b</sub>	bubble Reynolds number
Re <sub>s</sub>	bubble Reynolds number in a swarm
S	constant in Equation 6.22
$\mathbf{U}_{sb}$	local slip velocity of gas phase with respect to the continuous phase,
<i>2</i>	cm/s
Ut	single bubble terminal velocity, cm/s
v <sub>i</sub>	local slip velocity of i (gas, liquid, or solid)
< >	value averaged over the cross-sectional area
€j	holdup (or volume fraction) of i <sup>th</sup> phase
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6 <sub>gc</sub>	local gas holdup at the centre of column
$\Delta  ho$	density difference between two phases, g/cm <sup>3</sup>
$\mu_{f}$ .	viscosity of fluid (Equation 6.16), poise
$\mu_1$	viscosity of liquid (Equation 6.24), poise
$\mu_{s}$	viscosity of suspension, poise

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## **CHAPTER 6**

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## EFFECT OF SOLID PARTICLES ON GAS HOLDUP IN FLOTATION COLUMNS

#### Abstract

Gas holdup (volume fraction of gas) is a key parameter for the scale-up, design and performance evaluation of flotation columns. Gas holdup in gas-water and solid-gaswater systems was determined in a continuously operating column (10 cm in diameter and 447 cm in height) by an isolating technique and a conductivity method. The effect of concentration (0-15% v/v), size (95% -53  $\mu$ m, 90% +75  $\mu$ m) and type (hydrophilic (quartz, calcite), hydrophobic (coal)) of solid particles on gas holdup was investigated.

The presence of solids significantly *decreased* the gas holdup, by up to 40% relative. The gas holdup decrease increased with increasing solids concentration and decreasing particle size and hydrophilic particles had more effect than hydrophobic ones.

Possible mechanisms to explain the effect of solid particles were evaluated based on bubble coalescence, slurry density/viscosity changes, radial profiles, and wake structure effects. It was experimentally shown that bubble coalescence due to the presence of solids was not responsible for the gas holdup reduction. Using the drift flux model, it was shown that the changes in the density and viscosity of the water due to the presence of solids could not account for the observed reduction in the gas holdup. From a drift flux test, it was found that single bubble rise velocity in the water-gas system was lower than in the slurry-gas system which could account for the reduced gas holdup. In the 'radial profile' mechanism, assuming an originally flat gas holdup profile in the water-gas system, it was shown that if the introduction of solids caused circulation near the wall this could result in a lower average gas holdup. In the proposed 'wake structure' mechanism it was postulated that wake stability is increased in the presence of solids due to increased viscosity. This in turn increases the probability of in-line bubble-bubble interaction where the wake velocity of the leading bubble is superimposed on the rise velocity of the trailing bubble. The resulting higher rise velocity of the bubbles would reduce the gas holdup.

It is proposed that the effect of solids on reducing gas holdup is a combination of an increase in the rise velocity of bubbles due to stabilization of the bubble wake and a change in the gas holdup profile from flat to non-uniform.

## 6.I. Measurement

### 6.I.1 Introduction

A considerable body of data used to describe the hydrodynamic behaviour of flotation columns has been derived from two-phase (gas-water) studies. The implicit assumption is that the presence of solids does not affect the behaviour substantially. For example, gas holdup or volume fraction of gas, which is one of the most fundamental factors governing the hydrodynamics, is often assumed to be the same for both two- and three-phase systems (operated under otherwise identical conditions). As a result, the same gas flowrate vs. gas holdup relationship is used for both systems. This assumption may subject the current scale-up and design approaches to errors of unknown

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consequence(s). One example will serve to illustrate.

In flotation columns, there is a maximum gas throughput for a given operation beyond which the performance significantly deteriorates (Finch and Dobby, 1990). This maximum gas flowrate is frequently approximated as the point of deviation from linearity of the gas holdup vs. gas flowrate relationship (Xu et al., 1991; Tsuchiya et al., 1992). In column flotation operations, this maximum in gas throughput is usually respected with gas flowrates lower than this critical value. If the presence of solids has any effect on the maximum gas throughput, then the investigation of this possibility is of prime practical importance.

The study of the effect of solids on gas holdup could also serve as a step toward a better understanding of the complex hydrodynamics of flotation columns. For example, this may facilitate the interpretation of radial and axial gas holdup profiles in slurry-gas flotation systems to extend the original work on gas-water systems (Uribe-Salas et al., 1992; Xu et al., 1992).

As a general observation, it is important to evaluate the performance of flotation columns in operation; this task would be neither comprehensive nor accurate without taking the effect of solids into account.

#### 6.I.1.1 Previous Work

In three-phase reactor design, the investigation of the effect of solids on gas holdup has been the focus of many studies (see Table 6.1). The results of these studies are often contradictory (e.g., Ostergaard et al., 1966; Vasalos et al., 1980; Kara et al., 1982; Dayan and Zalmanovich, 1982; Koide et al., 1984; Sada et al., 1986; Dharwadkar et al., 1987; Fukuma, et al., 1987; Bukur et al., 1990; Khare et al., 1990; O'Connor et al., 1990; Mills, 1992). Referring to Table 6.1, no definite trend in the effect of solid

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Gas holdup in Solid-Gas-Water Compared to Gas-water	Particle Size (µm)	% Solids (v/v)	Den- sity (g/ml)	Column Dimensions (\$\phi x H) (cm)	Gas Flow- rate (cm/s)	Investigators & Comments	
Lower	<5	2.6-7.7	5.1	50 x 300	2-12	Bukur et al., (1990)	
Lower	10	3	3.18	9.5 x 100	0-8	Quiker et al., (1984) higher the %solids lower the gas holdup	
Lower	20-44	4.6-2.7	2.7	5 x 300	2-12	Bukur et al., (1990) continuous operation	
Lower	45-63	2.2	2.5	7.5∙x 300	1-6	Clark, (1990) high temperature and pressure	
Lower .	53	20-40	2.5	12.5 x 12.5	2.5	Joosten et al., (1977) higher the %solids lower the gas holdup	
Lower	63-125	4	2.52	12.2 x 196	2-6	Kato et al., (1972)	
Lower	75-250	1-8	2.5	10 x 230	2-12	Koide et al., (1984)	
Lower	<140	6.6	2.7	12.5 x 152	0-6	Ying et al., (1980) no effect of further increase in %solids	
Lower	100-150	small	2.55	10 x 200	>4	Imafuku et al., (1968) no effect of further increase in %solids	
Lower (slightly)	44265	1.6-6	2.6	7.5 x 265	0.06-2	Shah et al., (1983) little effect of %solids	
Lower	118-1300	10-30	2.5	7 x 42.5	2-9	Matsumoto et al., (1989) dependent on %solids not particle size	
Lower	1000-2000	1-5	1.2	7.7 x 128	0-4	Tang et al., (1989)	
Lower	2500	0.9-2.7	1.15	15.2 x 122	0-5	Fan et al., (1984) higher the %solids lower the gas holdup	
Lower	280	2.8-8.6	2.96	10 x 300	0-2	Ostergaard et al., (1966)	
Higher	10<	0.4	2.24	7.8 x 150	2-15	Sada et al., (1986) dependent on particle size	
Higher	100<	0.6	4.0	20 x 280	2-22	Khare et al., (1990) dependent on particle size &%solids	
Higher	813	0.08-0.2	1.2	15.2 x 200	1-15	Jamilalahmadi et al., (1991) opposite effect for non-wettable particles	
Higher	928	14	2.7	10 x 150	0.5-2	Viswanathan ct al., (1964)	
Higher	6000	40-55	2.5	65x 2.5x 210	0-11	Armstrong et al., (1975) opposite effect for non-wettable particles	
Higher	2200-4650	1-12	2.5	15 x 270	2-13	Hidaka et al., (1992)	
Higher	1000-6000	30-60	2.3	16x 2.5x 243	1-6	Kim et al., (1975)	

Table 6.1: Major studies on the effect of solids on gas holdup in three-phase reactors.



particles on gas holdup emerges; both increasing and decreasing effects have been reported. Some consensus emerges, however: for very fine particles (<10  $\mu$ m) with a low solids concentration (<0.6% v/v) and very large particles (>2000  $\mu$ m) with a high solids concentration (>10% v/v), the trend is an increase in the gas holdup (e.g., Sada et al., 1986; Hidaka et al., 1992); for the intermediate particle size (10  $\mu$ m-2000  $\mu$ m) and moderate solids concentration (>3% v/v), the effect is a decrease in the gas holdup (e.g., Ostergaard, 1971, 1978; Ying et al., 1980; Clark, 1990).

To account for an increase in gas holdup due to the presence of solids, mechanisms have been proposed for very fine, very large and intermediate size particles. It is believed that fine particles hinder the coalescence of bubbles, resulting in smaller bubbles with lower rise velocities, and hence a higher gas holdup (Sada et al., 1986). Bubble splitting because of the impact of particles is known to be responsible for the increase in the gas holdup in the case of large particles (Armstrong et al., 1975; Kim et al., 1977; Peterson et al., 1987). A coalescence-promoting effect is the commonly proposed mechanism for the decrease in gas holdup sometimes observed with intermediate size particles (Joosten et al., 1977).

A generally accepted mechanism of the effect of solids on gas holdup is not available. Part of the problem is a lack of direct evidence for a change in bubble size and hence velocity- which lies at the heart of the proposed mechanisms. One of the major obstacles to obtaining bubble size/velocity is the opacity of three-phase systems which makes visual inspection difficult.

Because of the unique features of flotation columns, the results of the above studies cannot directly be applied. Some of the major operating parameters of flotation columns as used in the mineral processing industry, which distinguish them from other bubble column systems, are as follows:

- small bubbles (<3 mm) maintained by the presence of frother

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- low gas flowrates ( <2-3 cm/s)
- countercurrent and continuous operation
- intermediate solids concentration (10-15% v/v)
- small particle size (<100  $\mu$ m).

#### 6.I.1.2 Objectives of the Study

The objectives of the present study are:

- to evaluate and clarify the effect of solid particles (i.e., concentration, size and type) on gas holdup in flotation columns
- to propose and test mechanisms regarding the effect of solids.

## 6.I.2 Experimental Section

### 6.I.2.1 Apparatus

The flotation column used in this work (Figure 6.1) was made of Plexiglas, 4.47 m in height and 10.18 cm in diameter. The bubble generation was through a porous ( $\sim 10 \ \mu$ m diameter holes) cylindrical (37 cm x 68 cm) stainless steel sparger at the bottom of the column. The middle 0.94 m section was chosen for the holdup estimation in order to limit disturbances due to feed and gas injection. Two air-actuated valves were used to isolate the middle (sampling) section. The required pressure to operate the valves was 827 kPa which resulted in a response time of 250 ms. This way of taking samples, often called the isolating technique, measures the phase holdup directly and accurately. A calibrated Tylan gas flowmeter (model FM-380) and MIC 2000 controller were used to measure and display the air flowrate.



Figure 6.1: Laboratory column flotation for phase holdup estimation.

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#### 6.I.2.2 Materials

The gas and liquid phases were air and tap water, respectively. Three types of solid particles were used: calcite in two size classes from Steep Rock Resources Inc. (Ontario), silica from Indusmin Inc. (Québec) and coal from QIT-Fer et Titane (Québec). Their size distributions are shown in Table 6.2.

	Weight (%)									
Size (µm)	+150	+106	+75	+ 53	+38	+25	-25			
Calcite 20	0	0	2.00	3.35	20.42	25.34	48.89			
Silica	0	0	3.34	11.79	24.19	10.73	49.95			
Coal	0	1.25	5.00	9.86	15.84	20.59	47.55			
Size (µm)	+300	+212	+150	+106	+75	+53	-53			
Calcite 75	5.15	15.70	26.03	27.26	15.82	7.31	2.73			

Table 6.2: Size distribution of calcite 20, silica, coal, and calcite 75.

The density of calcite, silica and coal was  $2.72 \pm 0.01$  g/cm<sup>3</sup>,  $2.64 \pm 0.01$  g/cm<sup>3</sup>, and  $1.79 \pm 0.07$  g/cm<sup>3</sup>, respectively, as determined with a gas pycnometer. In all tests 25 ppm of frother (Dowfroth 250 C) was used, unless otherwise specified.

#### 6.I.2.3 Procedure

 $\sum_{i=1}^{n}$ 

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The effect of solid particle concentration, size, and type on gas holdup was evaluated by comparing the gas holdup in the gas-water and solid-gas-water systems under the same operating parameters (i.e., gas and feed flowrates). The steady-state condition was tested by measuring and comparing the conductivity of the system in four consecutive time intervals through a program in the data acquisition system. When the difference between them was equal or smaller than the expected error (0.004 mS/cm), it was assumed that steady-state had been reached and the valves were simultaneously

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closed. To determine the actual gas holdup of the sample, isolated between the valves, a specially designed vessel was used. The vessel was constructed by joining a 5 cmdiameter graduated cylinder (2.65 L) to a tapered tank (5.58 L); the total volume was equal to the volume of the sampling section (i.e., the volume between the two valves, 8.23 L). The volume and the weight of the sample were measured with the vessel and knowing the density of the phases, the holdup of each phase was calculated. The sample was returned to the reservoir after the holdup determination. This method of actual holdup measurement not only allowed continuous operation of the column without interruption but also provided a constant concentration of the solids during the experiment.

The size distribution of the first calcite sample (95% -53  $\mu$ m) and gas flowrate were chosen to be within the range of interest in many industrial flotation column installations (Finch and Dobby, 1990). For each solid concentration (5-17% v/v), the gas flowrate was varied (0-2 cm/s) and gas holdup was measured. The feed flowrate was kept constant at 0.6 cm/s in all tests. All experiments were performed at room temperature and two thermistors were used to monitor the temperature of the middle and added sections.

Due to plugging, the performance of the sparger decreased significantly after introducing solids. This was confirmed by comparing the gas holdup values in the gaswater system before and after the solid-gas-water tests. It was found that an absolute decrease of more than 3% in gas holdup occurred. The presence of fast-moving large bubbles, observed visually, was responsible for the gas holdup reduction. To resolve this problem, it was decided to perform the gas-water system tests after the solid-gas-water tests thus eliminating any decrease in the gas holdup originating from plugging. The tests in the solid-gas-water system were performed twice, one from low to high gas flowrates and vice versa, and the average of the two values is reported as the gas holdup. The sparger was acid ( $\approx 1\%$  HCl) washed in an ultrasonic bath after each set

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of tests.

There was no detectable effect related to a change in the concentration of frother during a 48-hour period, as determined by measuring and comparing the gas holdup values in the gas-water system at the beginning and end of this period. This was done to insure that the measured changes during the study did not originate from any variation in the frother concentration.

Possible reductions in frother concentration due to adsorption by solid particles were examined. This was carried out by taking water from the solid-gas-water tests and comparing the gas holdup values with those for freshly prepared water/frother solutions.

## 6.I.3 Results and Discussion

#### 6.I.3.1 Effect of Solids Concentration

The results of the gas holdup determination in the gas-water and solid-gas-water systems are presented in Figure 6.2. The feed flowrate was 0.6 cm/s and calcite concentration was 15% v/v (32% w/w). The gas holdup in the solid-gas-water system is significantly lower than the corresponding gas-water system (Figure 6.2). The expected linear gas holdup vs. gas flowrate relationship is evident for both systems. Since all gas-water experiments were performed after the solid-gas-water experiments, the actual gas holdup in the gas-water system might be even higher - as some plugging of the sparger may have occurred. The isolating technique provided an accurate ( $\pm 0.3\%$  absolute error) measure of the phase holdup (i.e., gas and solids). The measurements were repeatable within  $\pm 0.7\%$  with higher reproducibility at low gas flowrates.

To investigate the effect of solids concentration on gas holdup, three different concentrations (5%, 10%, and 15% v/v) of calcite (95% -53  $\mu$ m) were used. Figure 6.3

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Figure 6.2: Gas holdup estimation in the gas-water and calcite-gas-water systems (tests done in replicate)



Figure 6.3: Effect of solids concentration on gas holdup.

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shows the results: the gas holdup decreases as the solids concentration increases. To illustrate, increasing solids concentration from 5 to 15% v/v increased the reduction in the gas holdup from about 8 to 32% relative to that in the gas-water only case.

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# 6.I.3.2 Effect of Particle Size

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To investigate the effect of particle size on the gas holdup, calcite in two size classes (labelled "20" and "75" in Table 6.2) was tested. For the purpose of comparison, with each series of tests an experiment without solids was also performed. The results are illustrated in Figure 6.4: decreasing the particle size decreases the gas holdup.

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#### 6.I.3.3 Effect of Particle Surface Charge

In tap water (pH~7.5) the surface of calcite (point of zero charge=pH 8; Kelly and Spottiswood, 1989) and air bubbles are positively and negatively charged, respectively (Yoon and Yordan, 1986). To explore the possible effect of electrostatic interaction on bubble coalescence, and consequently on gas holdup, two approaches were taken. First, the pH of the water was changed to 11.7 by adding lime to make the surface of calcite negatively charged. Second, silica particles (point of zero charge=pH 2; Kelly and Spottiswood, 1989), whose surface charge in tap water is negative, were used. The silica concentration and size distribution were 11.5% v/v and 85% passing 53  $\mu$ m, respectively. For both cases, the gas holdup in the slurry-water and gas-water systems was measured. Figures 6.5 and 6.6 show the results of the gas holdup estimation for the calcite-gas-water and silica-gas-water and their corresponding gas-water systems, respectively. The gas holdup in the three-phase system is again lower than the two-phase but the magnitude is independent of the pH (Figure 6.5) or type of solid (Figure 6.6). The results suggest that the particle surface charge does not have any significant effect on the gas holdup in the systems studied. ļ



Figure 6.4: Effect of particle size on gas holdup.

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Figure 6.5: Gas holdup in a calcite-water-gas system at pH=11.7 (tests done in replicate).



Figure 6.6: Gas holdup estimation in the gas-water and silica-gas-water systems (tests done in replicate).

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The effect of hydrophobic particles on the gas holdup was investigated using coal as the solid phase at a concentration of 15% v/v. The results are the two lower sets of data in Figure 6.7. The presence of coal decreases the gas holdup; the reduction (21% relative on average), however, is not as significant as for the hydrophilic particles (calcite and silica- e.g., 34% relative on average for calcite). A decrease in the bubble rise velocity due to attached particles of coal is probably responsible for this reduced effect. A similar observation has also been reported by Tustsumi et al. (1991) and Armstrong et al. (1975) when comparing hydrophilic and hydrophobic particles.

As was mentioned earlier, in order to take into consideration variations caused by sparger plugging and frother adsorption by solids, the gas-water tests were performed after the solid-gas-water tests (after settling out the solids). Unlike the situation with calcite and silica, coal particles adsorbed a significant amount of frother. This was confirmed by the much higher gas holdup values obtained in the gas-water system when fresh frother/water was used (Figure 6.7 upper data set).

## 6.II. Investigation of Mechanisms of Gas Holdup Reduction in Presence of Solids

A reduction in gas holdup upon addition of solids has been established. The possible mechanisms responsible for this effect will be discussed in this section. Since point gas holdup values and bubble size and velocity were not measured and since the visibility of the system in the presence of solids was poor, direct confirmation of the mechanisms proposed in this study was not possible. To help compensate for this, measured values reported in the literature were incorporated in the analysis. The mechanisms considered were:



Figure 6.7: Gas holdup estimation in gas-water and and coal-gas-water sytems.

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- 1) Coalescence promoting effect of solid particles
- 2) Change in the density and viscosity of the slurry
- 3) Radial gas holdup and flow profiles
- 4) Bubble wake effects.

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#### 6.II.1 Coalescence Promoting Effect of Solid Particles

As reviewed in Section 6.1.1.1, when the presence of solid particles reduces gas holdup the most common cause is taken to be bubble coalescence. However, there has been neither experimental verification nor a general explanation of the phenomenon to date, in particular for flotation systems. In this part of the study, an experimental approach to examine the coalescence enhancing effect of solid particles in solid-water-gas systems is presented and tested.

#### 6.II.1.1 Experimental Procedure and Apparatus

A Plexiglas tube, 3.8 cm in diameter and 97 cm in height, was added to the feed line (item 4, Figure 6.8) to measure the conductivity of the clear (solid free) water. Grid electrodes, shown elsewhere to be suited to measuring dispersion conductivity (Turner, 1975; Marchese et al., 1992), were placed in the feed line, in the middle, at the top (15 cm above the feed line), and in the added section (27 cm above the feed line) of the column. The operation of the valves, gas injection, and conductivity measurements was controlled by a computer. All conductivity signals were collected by a data acquisition system which consisted of a microcomputer (IBM compatible, 1 MB of memory), a 24-channel relay board (Metrabyte, model ERB-24), an I/O interface board to control the relay board (Metrabyte, model PIO-12), an A/D converter interface board (Metrabyte, model DAS-8GPA) and a conductivity meter (Tacussel, model CD-810). The programming language was GWBASIC. Masterflex pumps (model 720-33) were used to feed the column and a stirrer was used in the reservoir to mix the slurry and keep the



Figure 6.8: Laboratory flotation column set-up for testing coalescence mechanism.

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solids in suspension.

To verify if solid particles caused a change in bubble size, the gas holdup was simultaneously measured in the sampling section (i.e., in presence of solids) and in the top section (i.e., in the absence of solids). The electrical conductivity method was used for the gas holdup determination. The gas holdup was given by the Maxwell model (Maxwell, 1892):

$$\epsilon_{\rm g} = \frac{1 - \gamma}{1 + 0.5 \gamma} \tag{6.1}$$

where  $\epsilon_g$  is the gas holdup and  $\gamma$  is the dispersion to continuous phase conductivity ratio. In three-phase systems, the dispersion is the solid-gas-water mixture and the continuous phase is the solid-water (slurry) phase. These two conductivity measurements were made with the electrodes in the sampling (middle) section and in the feed line, respectively. In the top section of the column, which was solids free due to the high settling rate of the coarse particles (90% +75  $\mu$ m) used, the conductivity of gas-water and clear water were used to estimate gas holdup. The electrodes in the top section provided the gas-water conductivity and the clear water conductivity was measured in the added section. The assumption that the conductivity of the clear water is the same as the conductivity of the water at the top section was verified by operating the column without gas and comparing the water conductivity at the two positions.

While operating the column in the solid-gas-water mode two distinct systems were established: a three-phase mixture in the middle section (below the feed entrance) and a two-phase mixture in the top section (above the feed entrance). This configuration provided an on-line simultaneous comparison of the gas holdup in these two systems. In addition, while operating the column with gas-water, the gas holdup in the top section was also measured to compare with the values from the solid-gas-water tests. During runs each measurement of the conductivity was displayed on a monitor. This provided a convenient means of detecting any abnormality in the operation, e.g., a sudden change in the conductivity reading having its origin in a variation in the solids concentration. Moreover, it provided a continuous and clear picture of the effects of the different variables on the measured parameters.

The holdup estimation reported is the average of ten measurements with a 3 and second time interval between each where each measurement was the average of ten conductivity readings. For each gas flowrate all conductivity measurements, estimated holdup values and their corresponding standard deviations were saved in a separate file through the data acquisition system.

#### 6.II.1.2 Results and Discussion: Testing of the Coalescence Mechanism

To examine the occurrence of coalescence due to the presence of particles, the gas holdup at the top and the middle sections of the column were compared while operating in the presence of solids in the middle section. In addition, the gas holdup at the top solids-free section of the column was compared with that of the corresponding gas-water system.

It was necessary to establish the nature of the relationship between the gas holdup in the top and middle sections of the column before introducing solids. Consequently, the gas holdup was measured at these two locations, while operating the column with gas-water only (Figure 6.9). The gas holdup is slightly higher in the middle section, particularly at high gas flowrates (>1cm/s). The downward liquid flow at this location is responsible for the higher gas holdup. In fact, the top section is a concurrent system as opposed to the countercurrent system in the middle section.

The results for gas holdup in the top and the middle sections of the column in the



Figure 6.9: Gas holdup at the middle and top sections of the column (gas-water system).

solid-gas-water system as well as in the top section of the gas-water only system under equivalent conditions are presented in Figure 6.10. There are two points to note: the gas holdup in the middle section is lower than in the top section and there is also no significant difference between the gas holdup in the top section in either system. This clearly indicates that coalescence *cannot* be the reason for gas holdup reduction in the presence of solids as the gas holdup returns in the top section to the same value as obtained in the absence of solids. The possibility that coalesced bubbles return to their original size over the short distance between the two cells is too remote to contemplate.

The difference between the gas holdup in the solid-gas-water and gas-water systems is not as high as in the previous tests (Figures 6.6). There are two reasons: first, the particle size is coarser  $(3\% -53 \ \mu m \ vs. 95\% -53 \ \mu m)$  in this case; second, unlike the previous tests, the gas holdup in the top section, which is lower (see Figure 6.9), is compared with the middle section.

### 6.II.2 Change in Density and Viscosity of Liquid

Any increase in bubble velocity will decrease bubble residence time and thus gas holdup. One possible cause could be the increase in density of the liquid due to the presence of solids; the resulting increased buoyancy force will increase the bubble rise velocity. (At this point the increase in liquid viscosity due to solids and the consequent decrease in velocity is ignored.)

To examine the extent of the change in the velocity of bubble which could result in the observed gas holdup reduction, the drift flux model (Dobby et al., 1988; Wallis, 1969; see also Section 6.II.3.2) was used. For a given initial gas holdup and single bubble terminal velocity, the decrease in the gas holdup due to an increase in the bubble velocity was calculated (Figure 6.11). Two initial terminal velocities were considered, 9 cm/s and 10.42 cm/s. Referring to Figure 6.11, an increase of 15% in the rise



Figure 6.10: Gas holdup estimation at the middle and top sections of the column in calcite-gaswater system.

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Figure 6.11: Gas holdup vs. terminal velocity (based on drift flux model).

velocity of bubbles from an initial velocity of 10.42 cm/s resulted in a 21% decrease in the gas holdup.

To examine the extent of the effect of the density of the liquid on the gas holdup, the drift flux model was used to predict the gas holdup. In order to establish the suitability of the model, it was first tested on present data. Figure 6.12 presents the model fit and measured gas holdup values for the water-gas and slurry-gas systems (see Appendix 6.1 for the calculation procedure).

The model was used to estimate the gas holdup for various cases. The results are shown in Figure 6.13. The first case is where no solid particles are present (water-gas system). For the second case, it was assumed that solid particles with a density of 2.71 g/cm<sup>3</sup> and concentration of 10% v/v were added to the gas-water system. Assuming that solids only affect the density of the liquid, the gas holdup was estimated for various gas flowrates. The gas holdup decreased compared to the water-gas system; however, the reduction was significantly less than that actually observed in the presence of solids of this density and concentration (Figure 6.3). This suggested that the change in the density of the *liquid alone cannot* be responsible for the measured gas holdup reduction in the presence of solids.

The analysis gave maximum weight to the effect of density since viscosity was not considered: any increase in viscosity due to the presence of solids will counter the effect of density on bubble velocity. Since fine particles  $(95\% -53 \mu m)$  were used in this study, it is legitimate to take the viscosity change into account as well as density. When both density and viscosity effects were considered (see Appendix 6.2), the estimated gas holdup in the presence of solids was predicted to be even higher than that of the water-gas system suggesting the viscosity effect is predominant (third data set Figure 6.13). This emphasises the above conclusion that the change in the density of the liquid cannot be the mechanism of gas holdup decrease.
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Figure 6.12: Measured and estimated gas holdup values for the water-gas and slurry-gas systems.



Figure 6.12: Measured and estimated gas holdup values for the water-gas and slurry-gas systems.



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Figure 6.13: Effect of density and viscosity on gas holdup.

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# 6.II.3 Radial Gas Holdup and Flow Profiles

# 6.II.3.1 Introduction

A variation in gas holdup across the column diameter due to liquid/gas circulation reduces the average gas holdup (Guy et al., 1986; Lockett and Kirkpatrick, 1975; Hills, 1974; Freedman et al., 1969). Freedman et al. claimed that the decrease was due to a decrease in the residence time of gas bubbles in the column. Xu et al. (1992) confirmed the existence of radial gas holdup profiles in flotation columns. The question posed is whether the addition of solids causes a profile to develop which reduces the gas holdup.

# 6.II.3.2 Theory

The magnitude of the gas holdup depends on relative motion of the dispersed phases (gas bubbles and solid particles) with respect to the continuous phase (Lapids and Elgin, 1957). A one-dimensional theory of this flow has been proposed by assuming that the local slip velocity of the gas bubbles (i.e., velocity relative to the continuous phase) depends only on their concentration and the physical properties of the system (Bhaga, 1970; Wallis, 1969).

The definition of the terms and general correlations which will be used in the analysis are as follows:

v <sub>i</sub>	= local linear velocity of i (gas, liquid or solid)
$U_{sb}$	= local slip velocity of gas phase with respect to the continuous phase
J <sub>i</sub>	= local superficial velocity of the $i^{th}$ phase (upward flow is positive)
ε <sub>i</sub>	= holdup (or volume fraction) of $i^{th}$ phase

$$\mathbf{J}_{\mathbf{i}} = \boldsymbol{\epsilon}_{\mathbf{i}} \mathbf{v}_{\mathbf{i}} \tag{6.2}$$

$$\mathbf{J} = \mathbf{J}_{\mathbf{g}} + \mathbf{J}_{\mathbf{I}} + \mathbf{J}_{\mathbf{s}}$$
(6.3)

$$\epsilon_{g} + \epsilon_{l} + \epsilon_{s} = 1 \tag{6.4}$$

$$\mathbf{U}_{sb} = \mathbf{v}_{g} - \mathbf{v}_{l} \tag{6.5}$$

Combining Equations 6.2 and 6.5 yields:

$$\mathbf{U}_{sb} = \frac{\mathbf{J}_g}{\boldsymbol{\epsilon}_g} - \frac{\mathbf{J}_1}{\boldsymbol{\epsilon}_1}$$
(6.6)

Substituting Equation 6.4 in Equation 6.6 and rearranging, gives:

$$(1 - \epsilon_{\rm s}) \mathbf{J}_{\rm g} = (\mathbf{J}_{\rm g} + \mathbf{J}_{\rm l}) \epsilon_{\rm g} + \mathbf{U}_{\rm sb} \epsilon_{\rm g} \epsilon_{\rm l}$$
(6.7)

In multiphase systems, the data are often expressed as average rather than local values; for example, the gas holdup is usually obtained over a certain height of the column. Thus, Equation 6.7 should be written in terms of the average values. The average value of a quantity Y, over the cross-sectional area is defined by:

$$\langle Y \rangle = \frac{1}{A} \int_{A} Y \, dA$$
 (6.8)

Averaging each term of Equation 6.7:

$$<(1-\epsilon_{\rm s}) J_{\rm g} > = <(J_{\rm g}+J_{\rm l})\epsilon_{\rm g} > + < U_{\rm sb}\epsilon_{\rm g}\epsilon_{\rm l} >$$
(6.9)

Dividing throughout by  $<\epsilon_g>$ , and multiplying and dividing the first term on the righthand side of Equation 6.9 by  $<J_g + J_1>$ :

$$\frac{\langle (1-\epsilon_{s}) J_{g} \rangle}{\langle \epsilon_{g} \rangle} = \operatorname{Co} \langle (J_{g}+J_{l}) \rangle + \frac{\langle U_{sb} \epsilon_{g} \epsilon_{l} \rangle}{\langle \epsilon_{g} \rangle}$$
(6.10)

where Co is the distribution parameter which is defined by:

$$C_{o} = \frac{\langle (J_{g}+J_{i})\epsilon_{g} \rangle}{\langle (J_{g}+J_{i}) \rangle \langle \epsilon_{g} \rangle} = \frac{\left[\frac{1}{A}\int_{A} (J_{g}+J_{i})\epsilon_{g} dA\right]}{\left[\frac{1}{A}\int_{A} (J_{g}+J_{i}) dA\right]\left[\frac{1}{A}\int_{A} \epsilon_{g} dA\right]}$$
(6.11)

The slip velocity is related to a single bubble's terminal rise velocity in an infinite pool,  $U_t$ , and gas holdup. A frequently used relationship is (Richardson and Zaki, 1954):

$$U_{sb} = U_t (1 - \epsilon_g)^{m-1}$$
(6.12)

where m is a function of Reynolds number:

$$\mathbf{m} = \left(4.45 + 18\frac{\mathbf{d}_{b}}{\mathbf{d}_{c}}\right) \mathbf{R} \mathbf{e}_{b}^{-0.1} \qquad 1 < \mathbf{R} \mathbf{e}_{b} < 200 \tag{6.13}$$

$$m = 4.45 \operatorname{Re}_{b}^{-0.1} \quad 200 < \operatorname{Re}_{b} < 500 \quad (6.14)$$

and

$$\operatorname{Re}_{b} = \frac{U_{\iota} \rho_{f} d_{b}}{\mu_{f}}$$
(6.15)

where:

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 $\mu_{\rm f}$  = viscosity of fluid (poise)

 $d_b$  = bubble diameter (cm)

 $d_c = column diameter (cm)$ 

Masliyah (1979) applied the drift-flux concept (Wallis, 1969) to multi-species systems. In a somewhat similar approach, and using the drag coefficient relationship of Schiller and Naumann (1933), bubble terminal velocity in a bubble swarm can be expressed by:

$$U_{t} = \frac{gd_{b}^{2}\Delta\rho}{18\mu_{f}(1+0.15Re_{s}^{0.687})}$$
(6.16)

where

$$\operatorname{Re}_{s} = \frac{\operatorname{d}_{b} U_{sb} \rho_{f}(1 - \epsilon_{g})}{\mu_{f}}$$
(6.17)

 $\Delta \rho$  = density difference between two phases (g/cm<sup>3</sup>)

g = acceleration due to gravity  $(cm/s^2)$ .

For low solids concentration (<10% v/v), it is assumed that <(1- $\epsilon_s$ )  $J_g$ > = <(1- $\epsilon_s$ )> < $J_g$ >. By substituting Equation 6.12 in Equation 6.10, it can be shown that:

$$\frac{\langle (1-\epsilon_s) J_g \rangle}{\langle \epsilon_g \rangle \langle \epsilon_l \rangle \langle (1-\epsilon_g) \rangle^{m-1}} = \operatorname{Co} \frac{\langle (J_g+J_l) \rangle}{\langle \epsilon_l \rangle \langle (1-\epsilon_g) \rangle^{m-1}} + \operatorname{Ko} U_{\iota}$$
(6.18)

where the terminal velocity coefficient, Ko, is defined by:

$$\operatorname{Ko} = \frac{\langle (1 - \epsilon_g)^{m-1} \epsilon_g \epsilon_l \rangle}{\langle (1 - \epsilon_g)^{m-1} \rangle \langle \epsilon_g \rangle \langle \epsilon_l \rangle}$$
(6.19)

For flotation systems, where the particles are small, the slurry can be treated as a pseudo-homogeneous phase (Fan, 1989; Kara et al., 1982; see Section 6.II.4.8), and Equation 6.18 reduces to:

$$\frac{\langle J_g \rangle}{\langle \epsilon_g \rangle \langle (1 - \epsilon_g) \rangle^m} = \operatorname{Co} \frac{\langle (J_g + J_1) \rangle}{\langle (1 - \epsilon_g) \rangle^m} + \operatorname{Ko} U_{\tau}$$
(6.20)

In Equation 6.20 the distribution parameter, Co, takes into account the effect of nonuniform (i.e., non flat) flow and holdup profiles. The terminal velocity coefficient, Ko, is a measure of uniformity of holdup profiles plus the hindering effect of the gas bubbles on bubble rise velocity. In most industrial flotation columns, the profiles are assumed to be flat, i.e., Co=Ko=1 (Dobby et al., 1988), which indicates that the local values are equal to the average values. With this assumption the general Equation 6.20 becomes:

$$U_{t} = \frac{J_{g}}{\epsilon_{g}(1-\epsilon_{g})^{m}} - \frac{J_{g}+J_{1}}{(1-\epsilon_{g})^{m}}$$
(6.21)

To examine the effect of non-uniform flow and holdup profiles (i.e., Co and Ko) on the gas holdup, the gas holdup was calculated from Equation 6.19 for various Co keeping Ko constant for a given set of operating parameters and vice versa. Figures 6.14 and 6.15 present the results: increasing Co and Ko decreases the gas holdup. Thus, assuming a change in the flow profiles from uniform (i.e., flat) in the water-gas system to non-uniform in the presence of solids does reduce the gas holdup and could, therefore, be the mechanism responsible for the observations here.

The objective is to determine Co and Ko experimentally in the water-gas and slurry-gas systems and to propose the type of profiles which could yield the measured gas holdup decrease in the slurry-gas system.

## 6.II.3.3 Experimental Procedure

The flotation column described in Section 6.1.2.1 was used. To determine the single bubble rise velocity,  $U_t$ , the common practice is to measure the gas holdup for various gas flowrates in the column when the liquid velocity is zero, i.e., batch operation. The frother (Dowfroth 250 C) concentration was 25 ppm and the gas flowrate was varied between 0 to 1.8 cm/s. Once the steady-state condition was reached, as determined from stability of the conductivity measurements, the gas holdup was measured by the isolating technique. All gas holdup measurements were repeated three times.

For Co and Ko determination in the water-gas and slurry-gas systems, in order to change the gas holdup without changing bubble size, the gas flowrate was kept constant while the downward liquid flowrate was varied. The liquid (or slurry) flowrate range was 0.3-1.4 cm/s which resulted in a maximum gas holdup of 21% v/v for the water-gas system with a constant gas flowrate of 0.8 cm/s.

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Figure 6.14: Effect of distribution parameter, Co, on gas holdup.



Figure 6.15: Effect of terminal velocity coefficient, Ko, on gas holdup.

### 6.II.3.4 Results and Discussion

### 6.II.3.4.1 Co and Ko Determination

Figure 6.16 shows the gas holdup for various gas flowrates. The slope of the resulting curve at  $\epsilon_g = 0$  is equal to the single bubble rise velocity (see Appendix 6.3). The single bubble rise velocity in the water-gas system was  $8.24 \pm 0.20$  cm/s. For gas flowrates greater than 1 cm/s, which corresponds to a gas holdup of about 15% v/v, there is a slight deviation from linearity which results from the hindering effect of the bubble swarm.

In order to determine Co and Ko, the exponent m of Equation 6.2 had to be calculated. Two approaches were taken:

First approach: The calculation steps involved an iterative routine (Dobby et al., 1988; see Appendix 6.4):

1) estimate m;

- 2) calculate  $U_t$ , Equation 6.20;
- 3) calculate  $d_b$ , Equations 6.15 and 6.16, iterating on  $d_b$ ; and
- 4) calculate m, Equations 6.17 and 6.13, and compare with step 1; iterate on m.

Second approach: The experimental data (gas holdup, feed and gas flowrates) in the water-gas and slurry-gas systems were plotted as  $\frac{\langle J_g \rangle}{\langle \epsilon_g \rangle \langle (1-\epsilon_g) \rangle^m}$  versus  $\frac{\langle (J_1+J_g) \rangle}{\langle (1-\epsilon_g) \rangle^m}$  for values of m varying from 0 to 4 (see Appendix 6.4). A least squares straight line was fitted to the data points and the correlation coefficient, R, was calculated for each value of m. The value of m which corresponded to the highest value of R was chosen. Both approaches gave a value of m of about 3 for the water-gas and slurry-gas systems. The



Figure 6.16: Single bubble rise velocity determination in a batch water-gas system.

value of 3 was then used throughout the analysis.

To obtain Co and Ko, the experimental data were plotted as  $\frac{\langle J_g \rangle}{\langle \epsilon_g \rangle \langle (1-\epsilon_g) \rangle^3}$  versus  $\frac{\langle (J_1+J_g) \rangle}{\langle (1-\epsilon_g) \rangle^3}$  (Figure 6.17). The straight line was fitted (R=0.998) to the water-gas data and the slope (Co) and the intercept (Ko U<sub>0</sub>) were calculated. The Co and Ko U<sub>t</sub> values were  $1.12\pm0.03$  and  $8.02\pm0.04$  cm/s, respectively. From the previously determined U<sub>t</sub>, Ko is, therefore, 0.97. Dobby et al. (1988) found Co=1.24 for a 2.6 cm ×2 m column with frother (Dowfroth 250C) concentration of 10 ppm. Bhaga (1970) reported Ko values close to 1 in 3.7 cm x 132 cm column. The values of Co and Ko obtained for water-gas in this study suggest the existence of flat gas holdup and flow profiles.

For the slurry-gas system Co and Ko U<sub>t</sub> were  $2.21\pm0.19$  and  $11.93\pm0.23$  cm/s (R=0.978), respectively (Figure 6.18), values which differ significantly from those in the water-gas system. This implies that the profile in the slurry-water system must be different from that in the water-gas system. Thus, the determination of profiles with Co and Ko values similar to those experimentally determined for the slurry-gas case was attempted.

It is also important to note that, since the maximum value for Ko is 1, the minimum single bubble rise velocity in the slurry-gas system must be 11.93 cm/s which is an increase of 40% compared to the water-gas system.

# 6.II.3.4.2 Shape of Flow and Gas Holdup Profiles

Of the possible profiles, the parabolic shape, which has been reported (Yu and



Figure 6.17: Distribution parameter and terminal velocity coefficient determination in a water-gas system.



Figure 6.18: Distribution parameter and terminal velocity coefficient determination in a calcite-water-gas system.

Kim, 1988), was examined first. The following flow and gas holdup distribution profiles were considered:

$$\frac{\epsilon_{\rm g}}{\epsilon_{\rm gc}} = 1 - \left(\frac{\rm r}{\rm R}\right)^{\rm S} \tag{6.22}$$

$$\frac{J}{J_{c}} = \left(\frac{r}{R}\right)^{H} - 1$$
 (6.23)

where:

 $\epsilon_{gc}$  =local gas holdup at the centre of column

 $J_e$  =local mixture superficial velocity at the centre of column

r =radial variable

R =column radius

S,H =constants

Figure 6.19 shows various gas holdup profiles. S=1 represents a triangular profile and as S increases, the profile becomes more flat and for S > 30 the profiles could be considered as flat. From the concept of the average gas holdup and flow across the column (Equation 6.7) and using Equations 6.11 and 6.19 Co and Ko can be calculated (see Appendix 6.5). The distribution parameter, Co, for this type of profile, should range from 1.5 for triangular profiles (S=H=1) to 1 for flat profiles ( $S=H=\infty$ ) (Figure 6.20). This suggests that the Co=2.21 obtained for the slurry-gas system cannot originate from parabolic profiles. The decrease in Ko as gas holdup increases (Figure 6.21) reflects hindering effect of bubble swarms (Figure 6.21).

The above result implies that the profiles occurring in the slurry-gas system are more complex than parabolae. To investigate possible values of Co and Ko, combinations of idealized profiles were used (Bhaga, 1970). The proposed profiles are



Figure 6.19: Gas holdup profile for various values of S.

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Figure 6.20: Effect of constants H and S on distribution parameter, Co.



Figure 6.21: Effect of gas holdup and constant S on terminal rise velocity coefficient, Ko.

presented in Figure 6.22. There are four parameters: b indicates the location of change in the gas holdup profile; y is the ratio of local gas holdup to the gas holdup at the centre; a shows the position of the change in the flow and x is the flow (gas plus slurry) at any point across the column relative to flow at the centre. For a given set of parameters, Co and Ko may be computed using Equations 6.8, 6.9 and 6.19. The details of the calculation are reported in Appendix 6.6.

To limit the number of profiles resulting from all the possible combinations of the parameters, a few hydrodynamic constraints were incorporated in the profile search routine. First, it was assumed that a=b. This assumption seems reasonable as any change in the flow should result in a change in the gas holdup profile. Second, the range in values for x and y found in the literature were chosen as the search range for the profiles. Values in the range 0.6 to 0.9 and -0.3 to -0.5 have been reported for y and x, respectively (Clark and Flemmer, 1992; Miyahara et al., 1989; Ulbrecht et al., 1985; Walter and Blanch, 1982; Morooka et al., 1982; Hills, 1974).

It was found that to obtain Co values greater than 1, x had to be negative, which physically indicates the existence of circulation (Figure 6.22). The results of the Co calculations for x=-0.4 and various y values are presented in Figure 6.23. The measured Co value (2.21) in this study lies within the calculated range 1-3. It was also found that at a given position of the flow change, increasing the ratio of local gas holdup to gas holdup at the centre (y) decreases Co. This decrease in Co follows because an increase in y results in a more uniform gas holdup profile and eventually, for a flat profile (y=1), Co is equal to 1. Figure 6.24 shows the results of the effect of circulation on the distribution parameter (Co): an increase in x increases Co. Selecting values of 0.7 and -0.4 for y and x, respectively resulted in a(=b)=0.6 for Co=2.21.

The terminal velocity coefficient, Ko, is presented in Figure 6.25 for various ratios of local gas holdup to gas holdup at the centre (y). As the difference in the gas

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Figure 6.22: Idealized dimensionless gas holdup and flow profiles.



Figure 6.23: Effect of position and magnitude of gas holdup change across the column on distribution parameter.



Figure 6.24: Effect of position and magnitude of circulation across the column on distribution parameter.



Figure 6.25: Effect of gas holdup variation across the column on terminal rise velocity coefficient.

holdup at the centre and the region close to the wall increases, Ko decreases. Since Ko reflects the hindering effect of the bubble swarm on the single bubble rise velocity, it is inversely proportional to the absolute gas holdup; for example, when gas holdup increases from 15 to 27%, Ko decreases from 0.98 to 0.96. For the b=0.6 and y=0.7 case (obtained from fitting Co), Ko is between 1 and 0.98 for the range in gas holdup encountered in this study (0-30%). The Ko and b relationship for various gas holdups is shown in Figure 6.26. As expected, the variation of Ko was not significant in the studied range of parameters in this work. The values close to 1 for Ko confirm the higher bubble rise velocity (U<sub>1</sub> =11.9 cm/s) in the slurry-gas system compared to the water-gas system (8.0 cm/s).

Figure 6.27 illustrates the possible profiles based on the above calculations. The profile is similar to the profiles which have been reported in bubble and flotation columns by previous investigators (Ityokumbul et al., 1994; Deng et al., 1994; Xu et al., 1992; Bayerlain, 1985; Drew and Lehavey, 1981; Herringe and Davis, 1976; Serizawa et al., 1975).

# 6.II.4 Bubble Wake Effects

The bubble wake plays an important role in the fluid dynamics of multiphase systems. It has been identified as the major contributor to such phenomena as solids mixing, bed contraction, bubble breakup, and coalescence (e.g., Guthrie and Bradshaw, 1973; Dayan and Zalmannovich, 1982; Tang and Fan, 1989; Miyahara et al., 1991). In the following section, the bubble wake will be described to the extent necessary for the analysis of the experimental data in this study.

# 6.II.4.1 Wake Formation Behind a Bubble

When the relative speed between a body and the surrounding medium is



Figure 6.26: Effect of location and magnitude of gas holdup variation across the column on terminal rise velocity coefficient.



# Figure 6.27: Proposed dimensionless gas holdup and flow profiles.

sufficiently low, the flow just outside the body will closely follow the body surface. That is, at sufficiently small Reynolds numbers (defined based on the transverse dimension of the body), Re, the whole contour of the body forms part of a streamline, known as the dividing streamline. As the Re exceeds a certain critical value, the flow starts to separate from the surface of the body. The critical Re depends on the shape and nature of the surface of the body as well as the turbulence level in the surrounding stream. At Reynolds numbers moderately above the critical value, the separated, or free, streamlines branching off from the body contour will later rejoin at some distance behind the body, forming a closed region. This region is often called the wake (Fan et al., 1990). The term "wake" is often used to describe the entire area of non-zero vorticity downstream of a body placed in a uniform fluid flow.

The crucial issue in wake formation is the position of the separation points (Figure 6.28). Boundary layer growth begins at the stagnation point and energy is dissipated in overcoming the resistance caused by shear stresses in the boundary layer. The momentum of fluid particles in the boundary layer is thus considerably less than for those at corresponding positions in the ideal flowfield; the momentum of such particles is further reduced by the unfavourable pressure gradient until at some point they come to rest, accumulate, and are given a rotary motion by the surrounding flow. As the eddy increases in size, flow separation from the object becomes significant and the wake is formed (Vennard and Street, 1975).

Up to a Re of 0.1, the Stokes Equation (i.e., the condition of no significant fluid resistance) applies and the drag coefficient results from frictional effects; this implies that no wake is formed. As the Re is increased to about 10, separation and wake eddies begin to form (Vennard and Street, 1975). It has been reported that the critical bubble size for formation of a wake in water is about 1 mm (Fan and Tsuchiya, 1990). The wake of a bubble in this size range is correspondingly very small and this has made its direct measurement difficult.



Figure 6.28: Flow about a sphere at various Reynolds numbers (after Vennard and Street, 1975).

#### CHAPTER 6 EFFECT OF SOLID PARTICLES ON GAS HOLDUP ...

Fan and Tsuchiya (1990) divided the wake into two regions: primary (near) wake and secondary (far) wake. The near wake is associated with the wake phenomena near the body base such as vortex formation, growth and shedding. The far wake includes the rest of the wake and its structure does not depend strongly on the type of body. Kitano and Fan (1988) further subdivided the near wake region.

## 6.II.4.2 Wake Instability

It is believed that instability is the most important hydrodynamic characteristic of the wake (Fan and Tsuchuiya, 1990). As a bubble rises in a continuous medium, the bubble introduces energy to the medium at a rate equal to the bubble rise velocity times the net buoyancy force acting on the bubble. In a very viscous medium, the energy can be completely dissipated in the medium due to viscosity, causing a rectilinear motion of the bubble. This type of energy dissipation is known as laminar viscous dissipation. In a low viscosity medium, however, the energy generated by the rising bubble may not be absorbed by the viscous dissipation alone (Crabtree and Bridgwater, 1967). Some energy is released through wake shedding known as turbulent dissipation which induces bubble oscillations (secondary motion). The periodic shedding of vortices behind the bubbles is shown to be the cause of secondary motion (Fan and Tsuchuiya, 1990). Edge and Grant (1971) and Miyahara et al. (1988) observed that bubble oscillation starts with the onset of vortex shedding from the wake. Characterization of the secondary motion which has been observed over a wide range of bubble Reynolds numbers is very complicated. The motion is a combination of a "rigid body" type vibration identified by zigzag or spiral trajectories and a "non-rigid" deformation identified by shape expansion or base oscillations (Lindt, 1972; Clift et al., 1978; Bhaga and Weber, 1981).

Experimental work on wake characterization is usually based on visual observation of the flow pattern around the bubble using dye or powder tracers (Bessler and Littman, 1987; Ostergaard, 1965). Using this approach, Yabe and Kunii (1978)

found the wake structure shifts from a growing closed wake to a periodically discharging wake structure while the bubble rises. For 20 mm bubbles rising in viscous solutions (9-72 mPa.s), they found that the periodic shedding occurred when the Reynolds number was about 70 (i.e., critical Re). Wegener and Parlange (1973) and Bhaga and Weber (1981) have reported the critical Reynolds number to be in the range 100-110 for bubbles larger than 18 mm. The onset and frequency of vortex shedding are two factors which are commonly used to characterize wake instability (Lindt and Groot, 1974).

# 6.II.4.3 Wake Size

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The size or capacity of the wake governs the extent of the exchange between the wake phase and the continuous phase. It is then important to identify the boundary between individual wake regions and measure the size of the primary wake. Because of wake instability, exact evaluation of the wake size is not possible. The common practice is to measure the wake size over a time period which includes several cycles of the vortex formation-shedding process, and report time-averaged wake size (Fan and Tsuchiya, 1990). This is carried out by monitoring a rising bubble and its wake with a video camera which moves vertically at the same speed as the bubble (Tsuchiya et al., 1989). Values in the range 20-23 for the primary wake to bubble volume ratio have been reported for bubbles with Reynolds number about 100 (Coppus et al., 1977; Komasawa et al., 1980; Bhaga and Weber, 1981).

## 6.II.4.4 Wake Velocity Profile

Bhaga and Weber (1981) measured the axial velocity profile and the velocity along the horizontal plane containing the central filament of the vortex ring of a bubble with a closed toroidal wake (Bhaga and Weber, 1980, 1981). They used a tracer technique and photography. The measurements were conducted in viscous liquids with Re < 110 where they found closed steady wakes. They obtained a maximum velocity in the wake about 40-50% higher than the bubble terminal rise velocity (34.2 cm/s in their case). This velocity decreased with distance but was still appreciable even 30 diameters downstream.

# 6.II.4.5 In-line Bubble-Bubble Interaction

It has been shown that the bubble wake plays a vital role in capturing non-aligned bubbles, in increasing the rise velocity of trailing bubbles, and in the subsequent possible coalescence (e.g., Narayanan et al., 1974; Otake et al., 1977; Komasawa et al., 1980). Crabtree and Bridgwater (1971) demonstrated that bubbles up to 70 cm apart initially coalesced. They attributed this to enhancement of the rise velocity of the trailing bubble due to the wake velocity of the leading bubble. They also proposed a way of estimating the coalescence rate by incorporating the effect of wake velocity. Tscuhiya et al. (1989) reported a 22% increase, on average, in the rise velocity of trailing bubbles because of the wake velocity of the leading bubbles.

Bhaga and Weber (1980) by measuring the velocity in the wake of a bubble in a viscous liquid, confirmed the general explanation proposed by Crabtree and Bridgwater (1971). They showed that the in-line interaction of two bubbles can be described by a superposition approximation in which the trailing bubble rises at a velocity equal to its terminal velocity plus the wake velocity from the leading bubble. Figure 6.29 presents the results of the in-line interaction of two spherical cap bubbles with closed wakes. The position of the nose of the two bubbles as a function of time and the position of the trailing bubble were it rising in isolation are shown. The additional component of velocity due to the wake is evident as the trailing bubble approaches and eventually catches the leading bubble. The rise velocity of the leading bubble remains constant until coalescence occurs. Elongation of the trailing bubble and the time when the nose of the trailing bubble reaches the rear and then the forward stagnation points of the wake, A and B, respectively, are also shown. At point A, the trailing bubble enters the wake of

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Figure 6.29: Position of bubbles during in-line bubble interaction (data from Bhaga and Weber, 1980)

the leading bubble, where it experiences the extra velocity, causing deformation as shown by the increase in the aspect ratio. Coalescence occurs at point B. Bhaga and Weber (1980) concluded that in a sufficiently tall column a trailing bubble will always catch the leading bubble of equal size.

Nevers and Wu (1971), observing bubble rise velocities up to 30% higher than terminal rise velocities, adopted a different argument. They claimed that when the trailing bubble enters the wake of the leading bubble, its projected area which is exposed to stagnant fluid is decreased by the amount of its projected area which is in the wake of the leading bubble. This area sheltered in the wake of the leading bubble makes no contribution to the drag on the trailing bubble. Thus, as the projected area exposed to stagnant fluid decreases, the velocity relative to stagnant fluid must increase to keep the buoyant and drag forces equal.

The effect of wake can also be traced in the work of Kapellas (1973), Marks (1973), and Coppock et al. (1951) in studies on the effect of bubble injection frequency on bubble terminal velocity. They measured the velocity of bubbles with various injection frequencies (e.g., 42 and 400 bubbles/min), and observed that bubbles with the higher injection frequency have significantly higher terminal velocities. Marks (1973) proposed a model which assumes that the velocity of a bubble is equal to the velocity of a solitary bubble rising in still water plus the velocity of the turbulent wake generated by the bubbles ahead of it.

# 6.II.4.6 Effect of Wake Stability on In-line Bubble-Bubble Interaction

The structure of the wake determines the extent of the in-line bubble-bubble interaction. A closed and steady wake favours this interaction because of a higher probability of alignment and entrance of the trailing bubble in the wake of the leading bubble. Weber and Bhaga (1981) found that the higher the viscosity the more stable the ÷

wake, causing more frequent bubble coalescence. In another study, Bhaga and Weber (1980) showed that at Reynolds numbers above 150 the wake is open and unsteady. As a result, the coalescence sequence was not reproducible. Komasawa et al. (1980) observed that for Reynolds number in the range 90-250 the wake extends a longer distance than the laminar wake, but the approaching velocity is considerably reduced. This implies the existence of a less intensive wake. Otake et al. (1977) concluded that in the case of viscous liquids (e.g., 62 wt% glycerol solution) a wake with a stable vortex is easy to form, hence there is ample time for two bubbles to come close together and for coalescence to take place. Merchuk and Ben-Zvi (1992) found that the interfacial area decreases as the viscosity of liquid increases. In a similar study, Crabtree and Bridgwater (1971) found that in a sugar solution coalescence time is noticeably shorter than in water. They attributed this to the existence of a less rapidly moving wake which in turn puts a delay on the decay of velocity along the leading bubble's axis. Ostergaard (1966) also stated that the rate of bubble coalescence is mainly a result of the variation of bed viscosity. It has been found that the stability of the wake depends upon the Reynolds number: at Reynolds number about 200 the wake starts to shed and shedding frequency increases as Reynolds number increases (Tsushiya et al., 1990).

# 6.II.4.7 Pseudo-Homogeneous Assumption for Slurry

The liquid-solid suspension through which a bubble passes may be viewed as either heterogeneous or pseudo-homogeneous depending on the ratio of the bubble to solid particle diameters (Fan and Tsuchiya, 1990; Batchelor, 1967). If the ratio is small, the bubble interacts with the flow fields of individual particles. As the ratio of bubble to solid particle diameters increases (->15), the bubble rises as if the mixture were a pseudo-homogeneous suspension. In this case, the bed can be assigned an apparent viscosity (Bly and Worden, 1992). It has been observed that the wake structure behind large bubbles in three-phase fluidized beds of small particles was very regular and similar to that observed in viscous liquids (Massimilla et al., 1961; Rigby and Capes, 1970; 3
Henriksen et al., 1974). These observations suggest the solid-water mixture will behave as pseudo-homogeneous in the present case given the size of bubble (1000-2500  $\mu$ m) relative to particle (20-30  $\mu$ m) and thus it can be assigned an apparent viscosity and density (Ostergaard, 1973; Grbavcic et al., 1992).

#### 6.II.4.8 Solids Concentration and Viscosity

Viscosity, which is a measure of the ease of the relative movement of liquid layers with different velocities, increases with concentration of solids in suspension. From a hydrodynamic point of view, the increase in viscosity of a suspension may be looked upon as a consequence of the perturbation of the flow of liquid around the suspended particles. This leads to an increased rate of energy dissipation. The relative viscosity of the suspension is then determined by the increase in energy dissipation because of the presence of the dispersed phase. The viscosity of suspensions is affected by the shape, size and the mass of the suspended particles, the volume occupied by the particles, thermodynamic conditions and particle surface electric charge (Yen, 1968). Among these, concentration and size are often the most important. Generally, the viscous behaviour of a suspension is defined according to the concentration of the suspended phase. Yen (1968) has shown that for particles in mineral processing systems Roscoe's (1952) equation fairly accurately describes the solids concentration - viscosity relationship. The equation relates the volume fraction of dispersed phase ( $\phi$ ), viscosity of liquid ( $\mu_1$ ) and suspension ( $\mu_s$ ) in the following manner:

$$\mu_{\rm s} = \mu_{\rm 1} (1 - \phi)^{-2.5} \tag{6.24}$$

The wide variation of solids concentration (5-15% v/v) in this work therefore had a significant (10-50%) effect on the viscosity of the mixture.

### 6.II.4.9 Wake Stability and Viscosity

The presence of solid particles increases the viscosity of a liquid. This makes the wake of bubbles more stable in the solid-gas-water system compared to the corresponding gas-water system. Since in-line bubble-bubble interaction is more pronounced when the bubble wake is stable, this leads to the argument of a higher rise velocity of trailing bubbles in the solid-gas-water system, and hence a lower gas holdup, compared to the gas-water system.

#### 6.II.4.10 Effect of Bubble Size

The effect of bubble size on the extent of reduction in the gas holdup was investigated. Large bubbles (2-6 mm) were generated, as confirmed by visual observation in the gas-water system, by not adding any frother. Calcite (90% +75  $\mu$ m) with concentration of 10% v/v was used as the solid phase. The results of the gas holdup estimation at the middle and the top sections are presented in Figure 6.30. There is a considerable difference in the gas holdup between the two sections. (The absolute gas holdup is lower (16% v/v vs. 30% v/v for gas flowrate of 2 cm/s) than that for tests where frother was used.) Since the large bubbles have large wakes (Kozanoglu and Levy, 1991), a case can be made that the in-line bubble-bubble interaction is more intense, causing a higher reduction in gas holdup upon introduction of solids. Unlike the case where frother was used, in these water only tests coalescence of bubbles may have played a role in the gas holdup achieved in the presence of solids but clearly could not account for the increase in gas holdup when the solids are no longer present.



Figure 6.30: Gas holdup at the middle and top sections of the column (solid-gas-water system without frother).

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## 6.III. Conclusions

#### a) Measurement

1) The presence of solid particles (5-15% v/v, 95% -53  $\mu$ m) in flotation columns significantly decreased (by up to 40% v/v relative) the gas holdup.

2) Increasing solids concentration from 5 to 15% v/v increased the decrease in the gas holdup from about 8% relative to 32% relative, respectively.

3) The reduction in gas holdup increased when the particle size decreased from 90% +75  $\mu$ m to 95% -53  $\mu$ m.

4) The particle surface charge did not have any significant effect on the gas holdup.

5) The reduction in gas holdup (21% relative) in the presence of hydrophobic particles was not as significant as in the hydrophilic particle case (34% relative for calcite).

#### b) Mechanisms

#### 1) Coalescence:

It was experimentally shown that in flotation columns, the coalescence of bubbles due to the presence of solids was not responsible for the gas holdup reduction: coalescence is largely suppressed by the frothers used in flotation.

#### 2) Density/viscosity:

Using the drift flux model, it was shown that the change in the density and viscosity of

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the liquid due to the presence of solids cannot fully account for the observed reduction in gas holdup.

3) Gas holdup/flow profiles:

(i) To examine a change in the flow and gas holdup profiles as a possible mechanism of the gas holdup reduction in the presence of solid particles, the distribution parameter (Co) and terminal velocity coefficient (Ko) in the water-gas and slurry-gas systems were measured.

(ii) The significant difference between measured Co in the water-gas (1.12) and slurrygas (2.21) systems suggested a change from a flat profile in the water-gas system to a non-flat profile in the presence of solid particles.

(iii) The assumption of parabolic profiles for the slurry-gas system did not result in a Co value close to the measured one, suggesting the profile was more complex.

(iv) A method was proposed to investigate complex profiles based upon combinations of idealized profiles, an approach first used by Bhaga (1970). The application of the method resulted in a series of possible profiles with a distribution parameter equal to the one measured (2.21).

(v) It was shown that in the slurry-gas system, circulation close to the wall  $(r/R \ge 0.6)$  would decrease the gas holdup in that region which in turn would lead to a lower average gas holdup compared to the water-gas system where the profiles are flat. The type of gas holdup profile which was proposed was similar to the saddle shape profile reported in the literature.

(vi) It was found that the single bubble rise velocity in the water-gas system (8.0 cm/s)

is lower than in the slurry-gas system (11.9 cm/s). This suggests that an increase in the rise velocity of bubbles in the presence of solids contributes to the reduction in the gas holdup.

4) Bubble wake:

It is postulated that an increase in the viscosity of slurry due to the presence of solid particles stabilizes the bubble wake. This in turn increases the probability of in-line bubble-bubble interaction where the wake velocity of the leading bubble is superimposed on the rise velocity of the trailing bubbles. The resulting increased rise velocity of the trailing bubbles contributes to the measured increase in rise velocity of bubbles (see (vi)) and thus contributes to the lower gas holdup in the solid-gas-water system.

#### c) Postulated Mechanism

The decrease in gas holdup upon addition of solids is attributed to an increase in bubble velocity due to wake stabilization and a change in radial gas holdup profile from flat to saddle-shaped.

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## Appendix 6.1

#### Gas Holdup Estimation from the Drift Flux Model

Finch and Dobby (1990) proposed an empirical equation relating bubble size  $(d_b)$  to gas flowrate  $(J_g)$  for a porous sparger:

$$d_b = C J_g^{0.25} \tag{6.25}$$

where the constant C depends principally on frother concentration.

The following steps were taken to estimate C for a set of tests:

- 1) assume a value for C;
- 2) calculate  $d_b$  for a given  $J_g$  (Equation 6.25);
- 3) calculate  $U_{sb}$  from Equation 6.6;
- 4) calculate  $U_1$  using Equation 6.12;
- 5) calculate  $\text{Re}_{s}$  from Equation 6.17;
- 6) calculate  $U_{t}2$  from Equation 6.16;
- 7) calculate  $(U_1 U_1 2)^2$ ;
- 8) repeat steps 1-7 for all experimental points (i.e.,  $J_g$  and  $\epsilon_g$ );
- 9) minimize  $\sum (U_1 U_2)^2$  using C as a variable.

Table 6.3 presents a specimen calculation of constant C.

J <sub>g</sub> (cm/s)	$\epsilon_{g}$ measured	d* <sub>b</sub> (mm)	U <sub>sb</sub> (cm/s)	U <sub>t</sub> 1 (cm/s)	Re,	U,2 (cm/s)	$(U_1 - U_1 - U_1$
0.4	0.0587	0.62	7.45	8.38	43.25	6.91	2.161
0.6	0.0882	0.68	7.46	8.93	46.42	8.20	0.541
0.8	0.1145	0.73	7.66	9.72	49.76	9.16	0.307
1.0	0.1530	0.78	7.24	10.01	47.57	10.46	0.200
1.2	0.1766	0.81	7.52	10.99	50.27	11.17	0.031
1.4	0.2078	0.84	7.49	11.80	50.07	12.08	0.078
1.6	0.2454	0.87	7.32	12.67	48.13	13.16	0.244
1.8	0.2800	0.90	7.26	13.78	46.95	14.12	0.117

Table 6.3: A specimen calculation of constant C.

\*  $J_1=0.6 \text{ cm/s}, \rho_f=1 \text{ g/cm}^3, \mu_f=0.01 \text{ g/cm.s}, m=2.95$ 

From the minimization (step 9), a value of 0.78 was obtained for constant C.

Once constant C had been calculated, the following steps were taken to estimate the gas holdup from the drift flux model:

1) assume a value for  $\epsilon_{\rm g}$ ;

2) calculate  $U_{sb}1$  for a given  $J_g$  (Equation 6.6);

3) calculate  $d_b$  from Equation 6.25;

4) calculate  $\text{Re}_{s}$  from Equation 6.17 (linked with step 5);

5) calculate  $U_t$  using Equation 6.16;

6) calculate  $U_{sb}$ 2 from Equation 6.12;

7) calculate  $(U_{sb}1-U_{sb}2)^2$ ;

8) minimize  $(U_{sb}1-U_{sb}2)^2$  using  $\epsilon_g$  as a variable;

9) repeat steps 1-8 for all measured  $J_g$  values.

Table 6.4 presents a specimen calculation for the gas holdup estimation using the drift flux model.

J <sub>k</sub> (cm/s)	$\epsilon_{\rm g}$ estimated	U <sub>sb</sub> 1 (cm/s)	d <sub>b</sub> (mm)	Re,	U <sub>t</sub> (cm/s)	U <sub>sb</sub> 2 (cm/s)	$\frac{(U_{sb}1-U_{sb}2)^2}{(cm/s)^2}$
0.4	0.0689	6.52	0.62	37.03	7.41	6.45	0.00
0.6	0.0946	7.06	0.68	43.14	8.48	6.98	0.00
0.8	0.1208	7.39	0.73	47.14	9.40	7.31	0.00
1.0	0.1468	7.60	0.78	49.73	10.25	7.52	0.00
1.2	0.1739	7.72	0.81	51.15	11.07	7.63	0.00
1.4	0.2026	7.77	0.84	51.54	11.92	7.66	0.01
1.6	0.2341	7.72	0.87	50.89	12.82	7.62	0.01
1.8	0.2713	7.61	0.90	48.91	13.85	7.47	0.02

Table 6.4: A specimen calculation of the gas holdup estimation using the drift flux model.

\*  $J_1=0.6$  cm/s,  $\rho_f=1$  g/cm<sup>3</sup>,  $\mu_f=0.01$  g/cm.s, m=2.95

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## Appendix 6.2

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#### Effect of Density and Viscosity on Gas Holdup

Using the approach explained in Appendix 6.1, the constant C (see Equation 6.25) was calculated based on a set of gas holdup values (Table 6.4, column 2). A value of 0.78 was obtained for C. The density of the slurry was calculated using:

$$\rho_{sl} = \rho_1 \epsilon_1 + \rho_s \epsilon_s \tag{6.26}$$

where  $\rho$  is density and  $\epsilon$  is the holdup. A slurry density of 1.171 g/cm<sup>3</sup> was obtained for calcite ( $\rho_s = 2.71$  g/cm<sup>3</sup>) particles with a fractional concentration ( $\epsilon_s$ ) of 0.1. The slurry viscosity was estimated (0.0131 g/cm.s) from Equation 6.24 and used for the gas holdup estimation. Table 6.4 presents the gas holdup estimates which were obtained using the approach outlined in Appendix 6.1.

	Gas flowrate	Gas holdup	Gas holdup	Gas holdup
	(cm/s)	gas-water system	density effect	density and viscosity effects
		(%)	(%)	(%)
	0.2	4.02	3.76	4.28
	0.4	6.76	6.33	7.16
Ň	0.6	9.33	8.73	9.88
50. 1	0.8	11.86	11.07	12.53
	1.0	14.43	13.42	15.24
	1.2	17.10	15.83	18.11
6 27-20	1.4	19.94	18.33	21.21
	1.6	23.06	20.98	24.63

Table 6.4: Effect of density and viscosity on gas holdup.

## Appendix 6.3

### Single Bubble Rise Velocity Estimation

For a batch system  $(J_1=0)$  Equation 6.20 reduces to:

$$\frac{\langle J_g \rangle}{\langle \epsilon_g \rangle \langle (1 - \epsilon_g) \rangle^m} = \operatorname{Co} \frac{\langle J_g \rangle}{\langle (1 - \epsilon_g) \rangle^m} + U_{\iota} \operatorname{Ko}$$
(6.27)

Rearranging Equation 6.27 gives:

$$\langle \mathbf{J}_{g} \rangle = \frac{\mathrm{Ko}(1 - \epsilon_{g})}{1 - \mathrm{Co} \langle \epsilon_{g} \rangle} \langle \epsilon_{g} \rangle \langle (1 - \epsilon_{g})^{m-1} \rangle \mathbf{U}_{t}$$
(6.28)

Examining the Ko and Co values obtained along with the measured gas holdup values revealed that the first term in the right hand side of the Equation 6.28 is approximately equal to 1. Thus Equation 6.28 becomes:

$$\langle \mathbf{J}_{g} \rangle = \langle \boldsymbol{\epsilon}_{g} \rangle \langle (1 - \boldsymbol{\epsilon}_{g})^{\mathfrak{m}^{-1}} \rangle \mathbf{U}_{\mathfrak{l}}$$
(6.29)

Differentiating Equation 6.29 at  $\epsilon_g = 0$  yields:

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$$\frac{d < J_g >}{d < \epsilon_g >} = U_t \tag{6.30}$$

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## Appendix 6.4

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#### Determination of the Exponent m in Equation 6.12

First approach: Table 6.6 presents results of estimation of the exponent m using Dobby's approach (Dobby et al., 1988) for two gas flowrates in a gas-water system.

Table 6.6: Typical results of estimation of the exponent m.

J <sub>g</sub> (cm/s)	ε <sub>g</sub> (%)	U* <sub>sb</sub> (cm/s)	U, (cm/s)	d <sub>b</sub> (cm/s)	Re,	m	Re <sub>b</sub>
0.4	5.87	7.45	8.43	0.70	49.21	3.04	59.15
1.8	28.0	7.26	13.39	0.87	45.34	2.86	116.08

\*  $J_1=0.6 \text{ cm/s}, \rho_f=1 \text{ g/cm}^3, \mu_f=0.01 \text{ g/cm.s}$ 

The above calculation was repeated for various gas flowrates in the water-gas and slurrygas (5% and 10% solids concentrations) systems (Table 6.7).

	gas-water	system	slurry-ga (10% v/v	slurry-ga (15% v/v	urry-gas system 15% v/v solids)	
J <sub>s</sub> (cm/s)	ε, (%)	m	ε <sub>g</sub> (%)	m	ε <sub>κ</sub> (%)	m
0.2	2.88	3.06	1.35	2.74	1.04	2.64
0.4	<b>'5.87</b>	3.04	3.54	2.83	2.92	2.76
0.6	8.82	3.02	6.17	2.88	5.07	2.80
0.8	11.45	2.98	8.38	2.87	7.49	2.83
1.0	15.30	2.97	11.42	2.88	9.8	2.83
1.2	17.66	2.94	13.61	2.86	11.57	2.81
1.4	20.78	2.91	16.6	2.86	13.57	2.80
1.6	24.54	2.88	19.64	2.85	16.70	2.81
1.8	28.0	2.86	22.82	2.83	19.50	2.80
2.0	31.21	2.83	24.91	2.81	22.00	2.79

Table 6.7: Estimated m values for the water-gas and slurry-gas systems.

The value of m=3 was chosen for both systems for use in Co and Ko determination.

Second approach: A least squares straight line was fitted to the data points of the watergas system for various values of m and the correlation coefficient (R) was calculated (Table 6.8).

Table 6.8: The correlation coefficients for various values of m.

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m	0	1	1.5	2.5	3.0	3.5	4.0
R	0.6836	0.9773	0.9870	0.9959	0.9950	0.9938	0.9925

The value  $m \approx 3$ , which gave one of the highest values of R, was chosen. This value is in agreement with the value obtained from the first approach.

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## Appendix 6.5

### Co and Ko Calculation for Parabolic Type Profiles

Let  $J_g + J_1 = J$  where J is the total local superficial velocity of two phases. Then, Equation 6.11 reduces to:

$$Co = \frac{\langle J \epsilon_g \rangle}{\langle J \rangle \langle \epsilon_g \rangle}$$
(6.31)

From the concept of the average gas holdup and flow across the column (Equation 6.8) and using Equation 6.22, the average gas holdup can be expressed as:

$$\langle \epsilon_{g} \rangle = \frac{1}{A} \int_{A} \epsilon_{gc} \left( 1 - \left(\frac{r}{R}\right)^{s} \right) dA = \frac{2\epsilon_{gc}}{R^{2}} \int_{0}^{R} \left( 1 - \left(\frac{r}{R}\right)^{s} \right) r dr$$
 (6.32)

or

$$\langle \epsilon_{g} \rangle = \frac{S}{S+2} \epsilon_{gc}$$
 (6.33)

Similarly from Equations 6.8 and 6.23, one could obtain:

$$\langle \mathbf{J} \rangle = \frac{-\mathbf{H}}{\mathbf{H}+2} \mathbf{J}_{c}$$
(6.34)

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From Equations 6.8, 6.22 and 6.23, it can be shown that:

$$\langle \mathbf{J}\boldsymbol{\epsilon}_{g} \rangle = \frac{2J_{c}\boldsymbol{\epsilon}_{gc}}{R^{2}} \int_{0}^{R} \left( \left(\frac{\mathbf{r}}{R}\right)^{H} - 1 \right) \left( 1 - \left(\frac{\mathbf{r}}{R}\right)^{S} \right) \mathbf{r} d\mathbf{r}$$
 (6.35)

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Integrating and re-arranging Equation 6.35 gives:

$$< J\epsilon_{g} > = -\frac{HS(4+H+S)}{(H+2)(H+S+2)(S+2)} J_{c}\epsilon_{g}$$
 (6.36)

Substituting Equations 6.33, 6.34 and 6.36 in Equation 6.31 gives:

$$Co = \frac{H + S + 4}{H + S + 2}$$
(6.37)

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For slurry-gas systems where  $\epsilon_i = 1 - \epsilon_g$ , Equation 6.19 becomes:

$$Ko = \frac{\langle (1-\epsilon_g)^2 \epsilon_g \rangle}{\langle (1-\epsilon_g) \rangle^2 \langle \epsilon_g \rangle} = \frac{\langle (1+\epsilon_g^2 - 2\epsilon_g) \epsilon_g \rangle}{(1+\langle \epsilon_g \rangle^2 - 2\langle \epsilon_g \rangle) \langle \epsilon_g \rangle}$$
(6.38)

which can be rearranged to:

$$\operatorname{Ko} = \frac{1 + \frac{\langle \epsilon_{g}^{3} \rangle}{\langle \epsilon_{g} \rangle} - 2 \frac{\langle \epsilon_{g}^{2} \rangle}{\langle \epsilon_{g} \rangle}}{1 + \langle \epsilon_{g} \rangle^{2} - 2 \langle \epsilon_{g} \rangle}$$
(6.39)

Using Equation 6.22, it can be shown that:

$$<\epsilon_{g}^{2}> = \frac{2\epsilon_{gc}^{2}}{R^{2}}\int_{0}^{R} \left(1-(\frac{r}{R})^{S}\right)^{2} r \, dr = \frac{S^{2}}{S^{2}+3S+2}\epsilon_{gc}^{2}$$
 (6.40)

Similarly,

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$$<\epsilon_{g}^{3}> = \frac{2\epsilon_{gc}^{3}}{R^{2}} \int_{0}^{R} \left(1 - \left(\frac{r}{R}\right)^{S}\right)^{3} r dr = \frac{3S^{3}}{3S^{3} + 11S^{2} + 12S + 4} \epsilon_{gc}^{3}$$
 (6.41)

Substituting Equations 6.33, 6.40 and 6.41 in Equation 6.39 gives:

Ko = 
$$\frac{1 - 2\frac{S^2 + 4S + 4}{S^2 + 3S + 2} < \epsilon_g > + \frac{3(S^3 + 6S^2 + 12S + 8)}{3S^3 + 11S^2 + 12S + 4} < \epsilon_g >^2}{1 - 2 < \epsilon_g > + < \epsilon_g >^2}$$
(6.42)

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## Appendix 6.6

### Co and Ko Calculation for the Proposed (Dimensionless) Profiles

From Figure 6.22 and Equation 6.8, it can be shown that:

$$<\epsilon_{g}> = \frac{2\pi\epsilon_{gc}}{A} \left( \int_{0}^{aR} r dr + \int_{aR}^{R} y r dr \right) = \epsilon_{gc} \left( a^{2} + y \left( 1 - a^{2} \right) \right)$$
(4.43)

Similarly,

$$= J_{c}(a^{2}+x(1-a^{2}))$$
 (6.44)

and

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$$\langle J\epsilon_{g} \rangle = \frac{2\pi J_{c}\epsilon_{gc}}{A} \left( \int_{0}^{aR} r dr + \int_{aR}^{R} y x r dr \right) = J_{c}\epsilon_{gc} \left( a^{2} + xy(1 - a^{2}) \right)$$
(6.45)

Using Equations 6.8 and 6.43, the average of the gas holdup to the power two and three across the column is, respectively:

$$<\epsilon_{g}^{2}> = \frac{2\pi\epsilon_{gc}^{2}}{A} \left( \int_{0}^{aR} r \, dr + \int_{aR}^{R} y^{2} r \, dr \right) = \epsilon_{gc}^{2} (a^{2} + y^{2} (1 - a^{2}))$$
(6.46)

and

$$<\epsilon_{g}^{3}> = \epsilon_{gc}^{3}(a^{2}+y^{3}(1-a^{2}))$$
 (6.47)

Substituting Equations 6.43, 6.44 and 6.45 in Equation 6.31 gives:

$$Co = \frac{(a^2 + xy(1 - a^2))}{(a^2 + x(1 - a^2))(a^2 + y(1 - a^2))}$$
(6.48)

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Ko can be calculated by substituting Equations 6.43, 6.46 and 6.47 in Equation 6.39 which gives:

Ko = 
$$\frac{1 - \frac{2(a^2 + y^2(1 - a^2))}{(a^2 + y(1 - a^2))^2} < \epsilon_g > + \frac{a^2 + y^3(1 - a^2)}{(a^2 + y(1 - a^2))^3} < \epsilon_g >^2}{1 + < \epsilon_g >^2 - 2 < \epsilon_g >}$$
(6.49)

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# **CHAPTER 7**

## CONCLUSIONS

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## **CHAPTER 7**

## CONCLUSIONS

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## 7.1 Overall Conclusions

A critical ratio of dispersed to continuous phase conductivity, CCR, was introduced beyond which the dispersion conductivity is insensitive to the dispersed phase conductivity. Two equations to approximate CCR for any given dispersion were proposed.

It was found that the shape of dispersed particles could significantly affect the conductivity of the dispersion. This resulted in unacceptable estimates of solids holdup in solid-water systems when the classical models for a spherical dispersed phase (e.g., Maxwell (1892) and Bruggeman (1934)) were used. Fricke's model (1924), which accounts for particle shape, was found suitable.

A technique for on-line simultaneous determination of gas *and* solids holdup, based on a combination of conductivity and pressure difference measurements, was developed and tested in a laboratory flotation column (10 cm in diameter and 447 cm in height). The results were checked against the actual holdup determined by an isolating

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technique.

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The effect of concentration, size and type of solid particles on gas holdup was investigated. The presence of solids significantly decreased the gas holdup, by up to 40% relative. Possible mechanisms to explain the effect of solid particles were evaluated based on bubble coalescence, slurry density/viscosity changes, radial profiles and wake structure effects. It was experimentally shown that bubble coalescence was not responsible for the gas holdup reduction. It was proposed that the effect of solids on reducing gas holdup is a combination of an increase in the rise velocity of bubbles due to stabilization of the bubble wake and a change in the gas holdup profile from flat to saddle-shaped.

### 7.2 Claims for Original Research

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1) A technique for on-line simultaneous estimation of gas *and* solids holdup for threephase systems by combining pressure and conductivity measurements was developed.

2) The first systematic experimental study of the effect of solid particles (i.e., concentration, size and type) on gas holdup in the inique conditions of column flotation was conducted.

3) A consistent <u>reduction</u> in gas holdup upon addition of solids was demonstrated. A mechanism based on increased bubble rise velocity due to wake effects coupled with a change in gas holdup radial profile upon addition of solids was proposed.

4) Fricke's conductivity model for dispersions of non-spherical particle was adapted to the case of flake-shaped minerals.

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5) A critical conductivity ratio (CCR) for any dispersion was introduced beyond which the conductivity of the dispersion is not sensitive to changes in the relative conductivity of dispersed to continuous phases. Two equations to estimate CCR for any given dispersion were proposed.

## 7.3 Suggestions for Future Research

1) A sensor to measure gas holdup where the conductivity of the slurry and slurry-gas mixture can be measured at the same location needs to be developed for industrial applications.

2) The local (point) radial and axial gas holdup should be investigated in three-phase systems. This is essential for validating the proposed mechanism of effect of solids on gas holdup.

3) Direct measurement of the gas and liquid flows across the column cross-section is needed to study circulation in the column.

4) Tests using a mixture of hydrophilic and hydrophobic particles are recommended. This could contribute to further insight into the effect of solids on gas holdup in practical conditions.

5) The use of Fricke's model to estimate particle shape when the particle conductivity is known is worth examining.

6) Additional work is required to design new conductivity electrodes suitable for industrial applications. For example, ring electrodes flush with the container wall should be examined in detail as the electrodes used in this work "invade" the processing space.

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7) The use of the Bruggeman model, because of its sensitivity to size distribution of the dispersed phase, deserves investigation for evaluating the performance of sparging systems.

8) Experimental confirmation of the predicted insensitivity of dispersion conductivity to a change in the conductivity of the dispersed phase beyond the CCR is recommended.

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