Evaluation of Dissolved Air Flotation for the Treatment of Mineral Slimes

By

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ABSTRACT

The recovery of BaSO₄ mineral slimes (-10 microns) from silica using dispersed and dissolved air flotation, was studied. Experiments were performed in a modified commercial batch flotation machine. The influence of flotation variables such as collector concentration, concentration of silica depressant, pH, aeration and mineral purity on flotation parameters were also examined.

Experimental evidence suggests that dissolved air flotation results in enhanced BaSO₄ recovery over that normally achieved using dispersed air flotation for a limited range of experimental conditions. This range is characterized by poor BaSO₄ flotation conditions such as low pH or collector concentration. The flotation of BaSO₄ was found to be strongly influenced by variables which regulate the surface chemistry of the mineral, such as collector concentration, pH, silica depressant and mineral purity. It was also speculated that mechanical transfer or gangue into the froth may be a critical factor in the upgrading of barite ores.
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TABLE OF CONTENTS

1 INTRODUCTION
   1.1 Treatment Difficulties ........................................... 3
   1.2 Survey of New Slime Treatment Techniques
      1.2.1 Piggy Back Flotation ........................................ 5
      1.2.2 Electrophoresis ............................................. 6
      1.2.3 Magnetic and Electrostatic Separation ..................... 7
      1.2.4 Liquid-Liquid Extraction ................................... 7
      1.2.5 Selective Flocculation ..................................... 8

2 DISSOLVED AIR FLOTATION ........................................... 11
   2.1 Mechanism and Factors Influencing Dissolved Air Flotation
      2.1.1 Factors Influencing the Size and Rate of Precipitation of Air Bubbles
      2.1.2 Factors Influencing the Attachment of Precipitating Air to the Mineral Surface
      2.1.3 Mineral Surface Activation by Precipitation of Fine Bubbles
   2.2 Summary of a Previous Investigation in Dissolved Air Flotation of Mineral Slimes .. 35

3 EXPERIMENTAL APPROACH ........................................... 47
   3.1 Apparatus ......................................................... 48
   3.2 Experimental Procedure ......................................... 51
4 RESULTS AND DISCUSSION

4.1 Introduction

4.2 D.A.F.

4.3 Collector Concentration

4.4 Depressant

4.5 Purity of BaSO₄

4.6 pH

4.7 Aeration

4.8 Celestite Flotation

5 CONCLUSIONS

References

Nomenclature

A1 DEVELOPMENT OF EQUATION TO PREDICT VOLUME OF AIR PRECIPITATING FROM SOLUTION

A2 EXPERIMENTAL DETAILS

A2.a Experimental Procedure

A2.a.1 Mineral Preparation

A2.a.2 Pre-Flotation Procedure

A2.a.3 Flotation Procedure

A2.a.4 Analysis of Flotation Products

A2.b Equipment Description

A3 EXPERIMENTAL RESULTS

A4 STATISTICAL ANALYSIS

A4.a Estimate of Variance

A4.b Hypothesis Tests
<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>A4.b.1 Randomized Block Test</td>
<td>169</td>
</tr>
<tr>
<td>A4.b.2 Hypothesis Test for Comparing Two</td>
<td>171</td>
</tr>
<tr>
<td>Population Means</td>
<td></td>
</tr>
<tr>
<td>A4.b.3 Hypothesis Test for Comparison of</td>
<td>173</td>
</tr>
<tr>
<td>an Experimental Value to an Expected Value</td>
<td></td>
</tr>
<tr>
<td>A4.c Factorial Design Analysis</td>
<td>174</td>
</tr>
<tr>
<td>A4.c.1 2^2 Factorial Design</td>
<td>175</td>
</tr>
<tr>
<td>A4.c.2 2^3 Factorial Design</td>
<td>176</td>
</tr>
<tr>
<td>A4.c.3 Determination of Significant Effects</td>
<td>176</td>
</tr>
<tr>
<td>A5 SAMPLE CALCULATION OF FLOTATION RESULTS</td>
<td>195</td>
</tr>
<tr>
<td>A5.a Determination of Maximum Volume of</td>
<td>196</td>
</tr>
<tr>
<td>Dissolved Air Available for Precipitation</td>
<td></td>
</tr>
<tr>
<td>A5.b Determination of Solid Weight to</td>
<td>196</td>
</tr>
<tr>
<td>Slurry Volume Ratio</td>
<td></td>
</tr>
<tr>
<td>A5.c Determination of Weight % Floated</td>
<td>198</td>
</tr>
<tr>
<td>A5.d Determination of % BaSO_4 Recovered</td>
<td>199</td>
</tr>
<tr>
<td>A5.e Determination of the Upgrading Factor</td>
<td>200</td>
</tr>
<tr>
<td>A5.f Determination of the % Separation</td>
<td>201</td>
</tr>
<tr>
<td>Efficiency</td>
<td></td>
</tr>
<tr>
<td>A6 PARTICLE SIZE DETERMINATION</td>
<td>203</td>
</tr>
<tr>
<td>A6.a Procedure</td>
<td>202</td>
</tr>
<tr>
<td>A6.b Particle Size Distribution</td>
<td>205</td>
</tr>
</tbody>
</table>
LIST OF TABLES

Table  Title Page

1   Klassen's Comparative Results of Flotation Experiments 44
2   Klassen's Size Analysis of Flotation Products of Barite Slimes 46
3   Random Block Analysis of the Observed Differences between D.A.F. and Dispersed Air Flotation 61
4   Mineral Solubility Data 71
5   Comparison of Klassen's Results and Current Experimental Observations for the Flotation of Barite Slimes 96
6   Magnitude of Effects of Flotation Variables Relative to D.A.F. 99

A2-1   Equipment Specification and Suppliers 123
A4-1   Individual and Pooled Variance Estimates for Each Flotation Parameter 180
A4-2   Observed Differences between D.A.F. and Dispersed Air Flotation 181
A4-3   Calculated T Values for the Effect of Dissolved Air Concentration on Flotation Parameters 182
A4-4   Calculated T Values for the Difference between D.A.F. and Dispersed Air Flotation 183
A4-5   Calculated T Values for the Effect of Collector Concentration on Flotation Parameters 184
<table>
<thead>
<tr>
<th>Table</th>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>A4-6</td>
<td>Calculated T Values for the Effect of Depressant on Flotation Parameters</td>
<td>185</td>
</tr>
<tr>
<td>A4-7</td>
<td>Calculated T Values for Effects of Purity of BaSO₄ on Flotation Parameters</td>
<td>186</td>
</tr>
<tr>
<td>A4-8</td>
<td>Calculated T Values for the Difference in Flotation Parameters for the Flotation of Celestite and Barite from Silica</td>
<td>187</td>
</tr>
<tr>
<td>A4-9</td>
<td>Calculated T Values for the Effect of pH on Flotation Parameters</td>
<td>188</td>
</tr>
<tr>
<td>A4-10</td>
<td>Calculated T Values for the Effect of Aeration (RPM) on Flotation Parameters</td>
<td>189</td>
</tr>
<tr>
<td>A4-11</td>
<td>Calculated T Values for the Difference between Klassen's Results and Current Experimental Observations for the Flotation of Barite Slimes</td>
<td>190</td>
</tr>
<tr>
<td>A4-12</td>
<td>Effects of Collector Concentration, Depressant and D.A.F.</td>
<td>191</td>
</tr>
<tr>
<td>A4-13</td>
<td>Effects of BaSO₄ Purity, Depressant and D.A.F. on Flotation Parameters</td>
<td>192</td>
</tr>
<tr>
<td>A4-14</td>
<td>Effects of Celestite Flotation, pH and D.A.F. on Flotation Parameters</td>
<td>193</td>
</tr>
<tr>
<td>A4-15</td>
<td>Effects of Aeration and D.A.F. on Flotation Parameters</td>
<td>194</td>
</tr>
<tr>
<td>A6-1</td>
<td>Particle Size Distribution</td>
<td>206</td>
</tr>
</tbody>
</table>
LIST OF FIGURES

<table>
<thead>
<tr>
<th>Figure</th>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>The Various Aspects of the Slimes Problem with Particular Reference to Flotation</td>
<td>4</td>
</tr>
<tr>
<td>2</td>
<td>Effect of Frother Concentration on the Precipitation of Gases from Solution</td>
<td>14</td>
</tr>
<tr>
<td>3</td>
<td>Effect of Saturation of the Solution and of Frother on the Average Size of Gas Bubbles Precipitating from Solution</td>
<td>16</td>
</tr>
<tr>
<td>4</td>
<td>Total Surface Area of Gas Bubbles Precipitating from 1 L of Solution at Different Pressures</td>
<td>19</td>
</tr>
<tr>
<td>5</td>
<td>Volume of Gases Precipitating from Solution at Different Pressures</td>
<td>21</td>
</tr>
<tr>
<td>6</td>
<td>Variation of $W_1/W_2$ with Contact Angle</td>
<td>27</td>
</tr>
<tr>
<td>7</td>
<td>Coalescence and Direct Attachment of a Bubble to a Solid Surface</td>
<td>31</td>
</tr>
<tr>
<td>8</td>
<td>Activation of Flotation by Precipitation of Gas Bubbles from Solution</td>
<td>33</td>
</tr>
<tr>
<td>9</td>
<td>Flotation of a Mixture of Fluorite and Quartz of Size less than 10 micron with Different Additions of Sodium Silicate</td>
<td>37</td>
</tr>
<tr>
<td>10</td>
<td>Effect of Pulp Density on the Flotation of a Mixture of Fluorite and Quartz of a Size less than 10 microns</td>
<td>39</td>
</tr>
<tr>
<td>Figure</td>
<td>Title</td>
<td>Page</td>
</tr>
<tr>
<td>--------</td>
<td>----------------------------------------------------------------------</td>
<td>------</td>
</tr>
<tr>
<td>11</td>
<td>Flotation of Barite and Quartz of a Size less than 10 microns with Different Additions of Sodium Silicate</td>
<td>41</td>
</tr>
<tr>
<td>12</td>
<td>Flotation Cell and Pressure Chamber</td>
<td>49</td>
</tr>
<tr>
<td>13</td>
<td>Batch Dissolved/Dispersed Air Flotation Apparatus</td>
<td>50</td>
</tr>
<tr>
<td>14</td>
<td>Distribution of Differences in Weight % Floated between D.A.F. and Dispersed Air Flotation</td>
<td>56</td>
</tr>
<tr>
<td>15</td>
<td>Distribution of Differences in % BaSO₄ Recovery between D.A.F. and Dispersed Air Flotation</td>
<td>57</td>
</tr>
<tr>
<td>16</td>
<td>Differences in Upgrading Factor between D.A.F. and Dispersed Air Flotation</td>
<td>58</td>
</tr>
<tr>
<td>17</td>
<td>Distribution of Differences in Separation Efficiency between D.A.F. and Dispersed Air Flotation</td>
<td>59</td>
</tr>
<tr>
<td>18</td>
<td>Effect of Collector Concentration on the Weight % Floated</td>
<td>64</td>
</tr>
<tr>
<td>19</td>
<td>Effect of Collector Concentration on % BaSO₄ Recovery</td>
<td>65</td>
</tr>
<tr>
<td>20</td>
<td>Effect on Collector Concentration on the Upgrading Factor</td>
<td>66</td>
</tr>
<tr>
<td>21</td>
<td>Effect of Collector Concentration on % Separation Efficiency</td>
<td>67</td>
</tr>
<tr>
<td>22</td>
<td>Effect of pH on Weight % Floated</td>
<td>79</td>
</tr>
<tr>
<td>23</td>
<td>Effect of pH on % BaSO₄ Recovery</td>
<td>80</td>
</tr>
<tr>
<td>24</td>
<td>Effect of pH on the Upgrading Factor</td>
<td>81</td>
</tr>
<tr>
<td>Figure</td>
<td>Title</td>
<td>Page</td>
</tr>
<tr>
<td>--------</td>
<td>---------------------------------------------------</td>
<td>------</td>
</tr>
<tr>
<td>25</td>
<td>Effect of pH on % Separation Efficiency</td>
<td>82</td>
</tr>
<tr>
<td>26</td>
<td>Variation of Air Flowrate with Impeller Speed</td>
<td>85</td>
</tr>
<tr>
<td>27</td>
<td>Effect of Aeration on the Weight &amp; Floated</td>
<td>86</td>
</tr>
<tr>
<td>28</td>
<td>Effect of Aeration on % BaSO₄ Recovery</td>
<td>87</td>
</tr>
<tr>
<td>29</td>
<td>Effect of Aeration on the Upgrading Factor</td>
<td>88</td>
</tr>
<tr>
<td>30</td>
<td>Effect of Aeration on the % Separation Efficiency</td>
<td>89</td>
</tr>
<tr>
<td>A2-1</td>
<td>Dissolved/Dispersed Air Flotation Flowsheet</td>
<td>127</td>
</tr>
<tr>
<td>A6-1</td>
<td>Silica Particle Size Distribution</td>
<td>207</td>
</tr>
<tr>
<td>A6-2</td>
<td>Barite Particle Size Distribution</td>
<td>208</td>
</tr>
<tr>
<td>A6-3</td>
<td>BaSO₄ Particle Size Distribution</td>
<td>209</td>
</tr>
</tbody>
</table>
INTRODUCTION
CHAPTER 1

INTRODUCTION

Mineral slimes is a derogatory term used to indicate the nuisance characteristics of fine mineral particles. An acceptable general definition describes slimes as "the fraction of ore that is too fine to be commercially exploited by the conventional processes developed for coarser size fractions" [1].

The upper size limit used to define slimes varies depending on the ore being treated (i.e. an upper size limit of 3 μm for Cassiterite [2,3] to an upper size limit of 50 μm for fluorite [4] has been observed) and actual treatment process (i.e. upper size limits for galena have been industrially observed to be between 7 μm [5] and 20 μm [6]).

Primary slimes are those caused by the weathering and decomposition of certain rock components. Secondary slimes are produced as a result of comminution of ore.

Presently many large ore bodies are not being exploited since the liberation size of the desired mineral is below 30 μm [7]. Furthermore, often conventional mineral processes reject slimes which may contain an appreciable mineral content because of treatment difficulties.

However, with the continued exhaustion of natural resources it is important that problems in recovering slimes be resolved.
1.1 Treatment Difficulties

The problems encountered in treating slimes may be attributed to their small mass, high surface area and high surface energy. Several aspects of the slime problem with particular reference to flotation are summarized in Figure 1.

One of the difficulties in the conventional flotation of slimes is in establishing solid-air attachment. Since the probability of a collision between a bubble and a particle is a function of the square of the particle diameter [8] the fine size of slimes make their recovery by conventional flotation more difficult than for larger particles. Even upon collision the small mass and thus lower momentum of the particle may not be sufficient to overcome the disjoining pressure opposing the thinning of the film between the particle and the bubble.

The high surface energy of slimes may cause increased solubility and hydration along with rapid surface reactions which may leave the mineral unsuitable for collector adsorption. This factor combined with coating of the particle by unwanted gangue may increase its hydrophilicity and thus reduce the chance of successful air-solid attachment.

Another problem in treating slimes is factors which tend to reduce the grade of the floated material by the recovery of unwanted gangue in the froth. Gangue may be recovered due to entrainment or due to heterocoagulation with the desired mineral. (This is the reverse of the slime coating problem.)
FIGURE 1: The various aspects of the slimes problem with particular reference to flotation. Ref. (19).
Increased dissolution of the desired material may result in the adsorption of ions on gangue and thus activating the gangue for collector adsorption.

The increased surface area of slimes requires that higher reagent quantities be used which as a result tends to reduce the selectivity of the collector.

Problems also arise in the normal operation of flotation units due to high pulp viscosity and greater froth rigidity caused by slimes. As a consequence of some of the above problems there has been an effort in recent years to develop new techniques for treating slimes.

1.2 Survey of New Slime Treatment Techniques

In the recent years several new methods have been investigated to handle mineral slimes. These methods have been reviewed by Kitchener [7] and by Collins and Read [1]. A brief summary condensed from these reviews is provided in the following sections.

1.2.1 Piggy Back Flotation

The principle here is that a carrier mineral of fine but flotable size is used to recover ultra fines. Little is known of the mechanism although it is one of agglomeration.

Samygin et al. [9] postulated that the rate of adhesion of ultra fine particles to larger particles is $10^3$ to $10^4$ times higher than the cohesion rate between ultra fine particles in turbulent flow.
Collisions occur by an inertia mechanism followed by a diffusion mechanism. Collision efficiency is mainly determined by the hydrophobic characteristics of the colliding particles.

The disadvantages of piggy back flotation are that a high reagent dosage is required and that a method of separating the slime from the carrier particle is required. This technique does, however, provide a means of extending the normal flotation range.

1.2.2 Electrophoresis

Electrophoresis might at first glance appear to be an attractive method of separating slimes since the electrophoretic mobility depends on the zeta potential of the mineral, but, within certain limits, it is independent of the particle size. Since high potential gradients cannot be used in an aqueous medium (due to electrolysis) only very low particle velocities can be obtained. Flow patterns in the device used must be carefully controlled to avoid the electrophoretic motion from being masked by hydrodynamic noise in the system. It is doubtful as to whether this restriction could be met in plant operation.

Electrophoretic separation in non-aqueous solutions may be possible where fields of 5000 V/cm are possible [1]. Such forces should disrupt heterocoagulation. Important factors requiring further investigation are the effect of solids content and solvent re-use.
1.2.3 Magnetic and Electrostatic Separation

Magnetic flocculation of slimes can be induced although the separation of floca requires low fluid flow.

Under these conditions the separation is non-selective and as a consequence is finding some commercial usage in treating waste water effluent [10].

The addition of small quantities of ferromagnetic or strongly paramagnetic fines will tend to bridge magnetic particles when passed through a magnetic field. Non-magnetic particles are also carried down however.

One process to remove magnetic fines is to pass the slurry through a bed of iron turnings in the presence of a strong magnetic field. Particles are drawn to the intense regions of a non-uniform magnetic field. After collection the external field is removed and the particles are washed out of the bed [11].

The extension of dry electrical processes to particle sizes as low as 5 μm may be possible using high tension separators [12]. With high frequency pulsing corona discharge, finer particles are charged before the coarser particles are fully charged.

They thus reach the earthed cell before being blocked out by larger non-conductive particles.

1.2.4 Liquid-Liquid Extraction

The high surface areas in oil/water systems generated by emulsions makes the extraction of minerals from an aqueous phase into
an oil phase attractive.

The engulfment of the mineral species is dependent on the interfacial energies, $\gamma_{SO}$, $\gamma_{SW}$ and $\gamma_{OW}$.

Surfactants can be utilized to control the hydrophobicity of the mineral and stabilize the emulsion. Thus by selective conditioning a favourable mineral/oil/water contact angle allows the mineral to be partially engulfed at the oil/water interface. The transient emulsion floats to the top where it is separated and broken with the oil being recycled. Serious difficulties may be anticipated in breaking surfactant stabilized emulsions.

Another variation of the capillarity principle is to apply a small amount of oil to a thick pulp. Mixing the pulp in a revolving drum allows the more oleophilic particles to be wetted by the oil forming strong adhesive clots which can be retained on a screen. This process has been developed by Puddington and co-workers under the name "spherical agglomeration" [13].

1.2.5 Selective Flocculation

The principle of selective flocculation is to get particles of one species to form aggregates while the other species remains dispersed. Conventional sedimentation techniques are used to separate the flocs.

Three methods may be used to promote selective flocculation:

1) Electrolytic Coagulation - This involves the suppression of the double layer of the desired mineral through use of an
indifferent electrolyte.

2) Hydrophobic Bonding - Minerals conditioned with surface-active agents are attracted to each other as a result of the hydrophobic coating.

3) Polymer Bridging - Particles are bridged together by long chain high molecular weight polyelectrolytes. This mechanism forms the strongest aggregates.

Electrolytic coagulation and hydrophobic bonding are limited in scope because interparticle forces which depend on particle size are London-Van der Waal forces.

The aggregates are thus weak and are not easily extracted from the slurry. The use of polymers result in stronger flocs, however the chemical mechanism of adsorption is not sufficiently understood.

Detailed investigations are required to establish useful polymers for preferential adsorption. This will probably require the introduction of complexing or chelating groups onto the polymer.

Systems which will probably be favourable must have at least one well-dispersed component, a small fraction of +30 um particles and have significant differences in surface properties.

One major obstacle in selective flocculation is to discourage unwanted gangue from becoming entrapped in the floc matrix and thereby reducing the grade recovered.

Kitchener [14] who has had some experimental success in separating the galena/quartz and galena/calcite systems, has emphasized the need for more specific flocculants suggesting that
functional groups from successful flotation collectors (i.e. xanthates) be incorporated into the polymer flocculants.

One advantage of selective flocculation over conventional flotation is the elimination of the need to successfully contact mineral bubbles to solids. Selective flocculation still requires specific and discriminating adsorption of the flocculant and thus assumes that the surface properties of small particles do not deviate substantially from those of larger particles.

Using this same premise, Dissolved Air Flotation (D.A.F.) may provide an alternative solution to the treatment of slimes. In D.A.F. the nucleation of the air bubble on the particle directly eliminates problems in air/solid contact.
DISSOLVED AIR FLOTATION
CHAPTER 2

DISSOLVED AIR FLOTATION

D.A.F. is a commonly used process for separating solids from liquids. It is used in wastewater treatment to remove biological solids [15], in the pulp and paper industry [15], in coal processing [16] and for recovering oils in water in the refining industry [17].

In D.A.F. air is produced by a sudden reduction of the pressure imposed on the solution. The solution thus becomes supersaturated with respect to dissolved gases (notably air) and equilibrates through the precipitation of gases out of solution.

In the case where flocculants are used as commonly found in wastewater treatment Vrablik [15] has defined three mechanisms of bubble attachment resulting in flotation. These are:

a) Adhesion of gas bubble to the solid by
   i) collision between bubble and dispersed phase,
   ii) precipitation of bubbles onto the dispersed phase.

b) the entrapment of rising bubbles in the floc matrix

c) the adsorption of gas bubbles in the forming floc matrix.

If the adhesion of gas bubbles to the solid can be made the predominant mechanism then D.A.F. could be used apart or in conjunction with conventional dispersed air flotation to enhance slime treatment.
2.1 Mechanism and Factors Influencing Dissolved Air Flotation

The role of factors such as surface tension on the size and amount of precipitation of air bubbles, attachment to solids, mineral surface activation and consequently their effects on D.A.F. are examined in the following sections.

2.1.1 Factors Influencing the Size and Rate of Precipitation of Air Bubbles

Several factors affecting the formation of bubbles from precipitation of air from solution were studied by Klassen and Meshcheriakov [18,19].

One aspect of their research focussed on the effect of surfactant on bubble formation. Using pine oil as a surfactant they observed that the amount of air actually precipitated from solution was greatly enhanced by the addition of a surfactant. They also observed that the amount of air precipitated approached the theoretical limit based on Henry's Law. This is illustrated in Figure 2. The role of surfactant in this effect is to reduce the air/liquid surface tension and thus requires less energy be utilized in forming bubbles.

The average bubble size formed was also found to be smaller in solutions with surfactants present than in solutions containing only water. This is shown in Figure 3.

As a consequence of the smaller bubble diameters the total overall air/liquid surface area is larger. As shown in Figure 4 the total surface area is 2 to 3 times larger with surfactant present in
FIGURE 2  Effect of Frother Concentration on the Precipitation of Gases from Solution

1) 18.0 kPa
2) 14.7 kPa
3) 11.3 kPa
4) 8.0 kPa
5) 4.7 kPa
FIGURE 2

Quantity of gases precipitating from aqueous solutions, \%

Total gas volume dissolved in the water, \%

Concentration of pine oil mg/l.

0 20 40 60 80 100 120 140 160 180 200

Graph showing the relationship between the concentration of pine oil and the quantity of gases precipitating from aqueous solutions. The graph includes five curves, each labeled with a number (1 to 5). The y-axis represents the percentage of gases precipitated, while the x-axis represents the concentration of pine oil in mg/l.
FIGURE 3  Effect of Saturation of the Solution and of Frother on the Average Size of Gas Bubbles Precipitating from Solution

1) Concentration of Pine Oil = 100mg/L
2) Concentration of Pine Oil = 0mg/L
FIGURE 3

Bubble diameter, mm

Vacuum, mm Hg

Absolute Pressure, kPa
solution. The phenomena caused by the surfactant is again due to the lowering of the surface tension which allows smaller air bubbles to become stabilized.

Klassen and Meshcheriakov [18] also studied the effect of supersaturation by controlling the amount of vacuum imposed on the solution. By increasing the degree of supersaturation they found that not only did the amount of air precipitating from solution increase and approach the theoretical limits but also that the bubble size also increased. The increase in bubble size was found to be most significant in pure water solutions as shown in Figure 3. The increase in the amount of air precipitated may be explained by the greater deviation from equilibrium accompanying an increase in supersaturation. This larger driving force results in more air precipitating from solution. The non-linear increase in bubble size as a result of the decrease in pressure (as shown in Figure 5) is due to the inverse relationship between volume and pressure for gases.

Other factors which were also found to govern the precipitation of air from solution include the initial concentration of gases in solution, the degree of their dispersion and the intensity of agitation.

For D.A.F. it is desirable to have a large amount of air precipitate from solution to transport solids out of the bulk solution to the surface. The size of the air bubble is also important since it is advantageous to have a large surface area for solid/air attachment while also maintaining a large enough air bubble.
FIGURE 4  Total Surface Area of Gas Bubbles Precipitating from 1L of Solution at Different Pressures 19

1) Concentration of Pine Oil = 100g/tonne
2) Concentration of Pine Oil = 0g/tonne
FIGURE 4

Total surface area of bubbles, $10^3$ cm$^2$

Vacuum, mm Hg

Absolute Pressure, kPa
FIGURE 5  Volume of Gases Precipitating
from Solution at Different Pressures

1) Contained in Original Solution
2) Theoretical Precipitation
3) Observed Precipitation at Pine Oil Concentration = 100mg/L
4) Observed Precipitation at Pine Oil Concentration = 30mg/L
5) Observed Precipitation at Pine Oil Concentration = 15mg/L
6) Observed Precipitation at Pine Oil Concentration = 0mg/L
FIGURE 5

Volume of gases precipitating from 1 litre of aqueous solutions, mL.

Vacuum, mm Hg

Absolute Pressure, kPa
to supply enough buoyancy to carry the mineral out of solution.

2.1.2 Factors Influencing the Attachment of Precipitating Air to the Mineral Surface

The precipitation of air bubbles on particles is important since it may lead to the direct flotation of the particle itself or act as an activator for large bubble attachment.

The mineral pulp may become supersaturated with respect to air by reducing the imposed pressure. When the molecules of gas diffuse to the solid interface across the external layer of the solids hydrated film, the time required to reach the solid surface is dependent on the degree of hydration of the external film. That is, the greater the hydration of the surface, the longer the time required for diffusion of air to the solid surface.

This resistance by the hydrated layer is due to an increased distance for diffusion and also to the fact that the oriented dipoles of bonded water lose their solubilizing properties [19].

In circumstances of incomplete wettability of the solid surface by water, precipitating air bubbles can more readily displace water from the mineral surface than separate water molecules from each other. Consequently, air bubbles preferentially form on the solid surface as opposed to forming in the bulk solution. Klassen [19] showed this by a thermodynamic analysis of the work involved in bubble formation.

The work expended in the formation and growth of an air bubble
can be approximated by the work to form a gas/liquid interface:

\[ W_a = 4\pi r^2 \gamma_{lg} / g \]  

(1)

where

\( r \) = bubble radius

\( \gamma_{lg} / g \) = liquid-gas surface tension

A more thorough analysis would include work to form a film, \( W_b \), and work to fill the bubble with vapour, \( W_c \), where:

\[ W_b = \frac{4}{3} \pi r^3 p \]  

(2)

\[ W_c = -\frac{4}{3} \pi r^3 p_v \]  

(3)

where

\( p \) = pressure in an air bubble

\( p_v \) = vapour pressure of the liquid.

Combining these terms gives the total work of formation of an air bubble as:

\[ W_1 = 4\pi r^2 [\gamma_{lg}/g - \frac{r}{3} (p_v - p)] \]  

(4)

This process can be assumed to be isothermal. On the other hand, the work to form a bubble on a solid surface is completely different. For such nucleation it can be assumed that at the initial moment of formation, the bubble possesses a form of an ideal spherical segment. The small size and high capillary internal pressure results in a constant curvature of the bubble. The elasticity of the films surrounding the bubble counteracts the deforming forces trying to sever the bubble from the solid. At
initiation, an equilibrium contact angle is formed and there is no hysteresis effect until the bubble grows, causing the three-phase interface to move.

Since the precipitating bubbles are small, the attached solid surface may be considered to be much larger than that of the bubbles. Furthermore, if it is assumed that the work is only due to changes in free energy, then the work of precipitation of an air bubble on a solid surface is:

\[ W_2 = S_2 \frac{\gamma_{L/g}}{g} + S_1(\gamma_{S/g} - \gamma_{S/L}) \]  

(5)

where

- \( S_1 \) = area of surface attachment
- \( S_2 \) = area of surface of spherical segment

If \( S_2 \) is expressed in terms of the height of the spherical segment and \( \gamma_{S/g} - \gamma_{L/g} \) is expressed in terms of \( \gamma_{L/g} \) and contact angle, \( \theta \), then

\[ W_2 = \frac{\gamma_{L/g}}{g} S_1 K \]  

(6)

where

\[ K = 1 + \cos \theta + \cot^2 \frac{\theta}{2} \]  

(7)

From the expression for \( K \) it can be seen that the higher the contact angle, \( \theta \), the less work is required and as a result the better the probability of precipitation of air on the solid surface.

A comparison of the work of precipitation of equal volume bubbles in bulk solution, \( W_1 \), to the work of formation by nucleation onto a solid surface, \( W_2 \), can be approximated by:
\[ \frac{W_1}{W_2} = \frac{1 + \tan^2 \frac{\theta}{2}}{\left(3 \tan \frac{\theta}{2} + 1\right)^{1/3}} \]  

(8)

As shown in Figure 6, more work is required to precipitate air in the bulk solution than on the mineral surface with increasing contact angle. Thus, the nucleation of a bubble on a solid surface is favoured. After nucleation of the air bubble, other factors which influence the survival of the air bubble become important.

In his thermodynamic calculations, Zeldovich (20) determined the probability of the formation of a surviving bubble, \( \lambda \), which does not redissolve due to capillary pressure as:

\[ \lambda = k_1 \exp(-4\pi r^2 \gamma_{l/g} / 3RT) \]  

(9)

where:

- \( r \) = bubble radius
- \( R \) = gas constant
- \( T \) = temperature
- \( k_1 \) = proportionality constant

The minimum bubble size which is stable in water as determined by surface tension and extent of supersaturation is given by (21):

\[ r_{\text{min}} = \frac{2 \gamma_{l/g}}{p - p_1} = \frac{2 \gamma_{l/g}}{k(c - c_1)} \]  

(10)

where:

- \( p - p_1 \) = decrease in pressure under conditions of initial supersaturation of solution by air.
- \( k \) = Henry's law constant
- \( c - c_1 \) = magnitude of supersaturation.
FIGURE 6  Variation of $W_1/W_2$ with contact angle (at constant volume of air precipitating from solution)  Ref. (19)
Substitution of this expression in Zeldovich's equation yields:

\[ \lambda = k_1 \exp[-16 \pi \gamma_{l/g} / 3 RT K^2 (C - C_i)^2] \]  \hspace{1cm} (11)

This shows that the probability of bubble formation is enhanced by lowering the surface tension and increasing the degree of supersaturation.

The time interval for the formation of a surviving bubble is almost instantaneous and has been estimated by Zeldovich (20) to be about \(10^{-12}\) sec. The number of nucleating bubbles, \(N\), forming per unit time, is given by (21)

\[ N = b r_{\min} D (8 \pi \gamma_{l/g} / RT)^{1/2} \]  \hspace{1cm} (12)

where \(D\) = analogous term to diffusion coefficient

\[ b = \text{constant} \]

The growth of the air bubble after reaching the minimum stable radius is favoured by reduced internal pressure, increased interface area, and intensity of mixing.

Klassen and Meshchinakov (19) studied the kinetics of bubble growth on the surface of barite and fluorite and observed good agreement between theory and experimental data. Diffusion of gases into bubbles takes place when the partial pressure of gases in solution exceeds the partial pressure in the bubble. Increased electrolyte in the pulp decreased the concentration of dissolved gases. This results in a decrease in the number of bubbles precipitated but does not affect their size.

A thermodynamic analysis suggests that dissolved air should more readily diffuse into existing bubbles rather than form new ones.
This is contradicted by kinetics, since extremely short time intervals in bubble formation are involved.

The actual distribution of precipitated air is mainly determined by the rate of diffusion of dissolved gases across the hydrated film of the particle and the distance gases must diffuse.

In tests (22) about 80 to 90% of the overall precipitating air has been observed to evolve from the solution on mineral surfaces, with the remainder diffusing into existing bubbles.

When the pulp undergoes a sharp supersaturation and contains an optimum quantity of surfactant (to stabilize bubbles) millions of micro-bubbles may be formed. Although such bubbles activate flotation, bubbles which form on the particle surfaces are more efficiently used.

2.1.3 Mineral Surface Activation by Precipitation of Fine Bubbles

The precipitation of fine bubbles on mineral surfaces may activate the surface and thus enhance flotation or lead to flotation directly.

The fine bubbles improve the probability of larger bubbles penetrating the hydrated layer resulting in successful mineral bubble contact. This activation may be explained by a number of reasons:

i) small bubbles act as spearheads on the mineral surface,

ii) the precipitated bubble surface is always much less hydrated than the mineral surface, as a result the stability of the hydrated film between the large and fine bubble is far lower
than the stability of the film separating the bubble and mineral surface,

iii) the difference in the diameter of the precipitated and colliding bubble results in differences in the capillary pressure which acts as an additional force in the disruption of the water film.

Coalescence attachment of a large air bubble to a mineralized surface bearing a precipitated bubble can be shown to be thermodynamically more favourable than for the case of direct attachment of an air bubble to an unactivated mineral surface. Figure 7 represents coalescence and direct attachment and the surfaces involved.

A thermodynamic comparison of these mechanisms is subject to much uncertainty since the system is far from equilibrium especially in the case of coalescence attachment due to hysteresis phenomena in the spreading of attachment area from $S_1$ to $S$. Under identical conditions, attachment is more likely for the case with the greater decrease in the free energy of the system.

The free energy of the system for direct attachment, $\Delta G_b$, is expressed by:

$$\Delta G_b = \gamma_{l/g} (S_5 - S_6 - S_7 \cos \theta)$$

(13)

For coalescence attachment the free energy, $\Delta G_a$, is represented by:

$$\Delta G_a = \gamma_{l/g} (S_3 + S_2 - S_4) + S(\gamma_{S/l} - \gamma_{S/g}) + S_1 (\gamma_{S/g} - \gamma_{S/l})$$

(14)

Upon introduction of the contact angle, $\theta$, and re-arrangement equation (13) becomes:
Figure 7 Coalescence and direct attachment of a bubble to a solid surface

(a) attachment through coalescence;
(b) direct attachment
Ref. (19)
\[ \Delta G_a = \gamma L/S \left( S_3 + S_2 - S_4 - S \cos \theta + S_1 \cos \theta \right) \]  

(15)

For the case of attachment of equal sized bubbles, the volumes are as follows:

\[ V_2 = V_5 = V_4 \quad \text{and} \quad V_3 = V_1 + V_2 \]  

(16)

then \( \Delta G_a \) is greater than \( \Delta G_b \) if \( S_3 = S_5 \) and \( r_8^3 = r_7^3 - r_6^3 \)  

(17)

Regrouping gives:

\[ \frac{\Delta G_a}{\Delta G_b} = \frac{S_3 - (S_4 - S_2) - (S - S_1) \cos \theta}{S_3 - S_6 - S_7 \cos \theta} \]  

(18)

However, since

\[ S - S_1 = \pi (r_7^2 - r_6^2) < S_7 = \pi (r_7^3 - r_6^3)^{2/3} \]  

(19)

and

\[ S_4 - S_2 = \pi \left( 2 + \cot^2 \frac{\theta}{2} \right) (r_7^2 - r_6^2) < S_6 = \pi \left( 1 + \cot^2 \frac{\theta}{2} \right) \]  

(20)

\[ (r_7^3 - r_6^3)^{2/3} \]

then

\[ \frac{\Delta G_a}{\Delta G_b} > 1 \]  

(21)

Thus, the decrease in the free energy of the system is greater for coalescence attachment as opposed to direct attachment. Consequently, the precipitation of fine air bubbles leads to the activation of the mineral for flotation.

The activation of flotation due to fine bubbles present on the mineral surface has been demonstrated by Klassen (23) in the following experiment.

Using a pneumatic flotation machine fluorite was floated with
FIGURE 8  Activation of Flotation by Precipitation of Gas Bubbles from Solution

1) Flotation solely by bubbles precipitating from solution

2) Flotation solely by bubbles produced by mechanical means

3) Combined flotation by air precipitated from solution and by bubbles produced by mechanical means.
oleic acid using three mechanisms of pulp aeration. Air bubbles were produced either by precipitation from solution, sparging air through a porous membrane or a combination of the two. The flotation rate was found to be much higher in the third case as illustrated in Figure 8.

Dzienisiewicz and Pryor (24), while studying the attachment of bubbles to fixed mineral surfaces, discovered that smaller bubbles attached to mineral surfaces more readily than larger bubbles and that the presence of fine bubbles greatly improved the attachment of larger bubbles. From this they concluded that optimum flotation should consist of two aeration stages. In the first, the pulp would be treated with fine bubbles which should activate the mineral surface. During the second stage larger bubbles are introduced which coalesce with existing activating bubbles and float the mineral particles.

2.2 Summary of a Previous Investigation in Dissolved Air Flotation of Mineral Slimes

Using the principles of D.A.F. Klassen and Meshcheriakov (18,19) undertook a comparative study of the flotation of fine (≤10 μm) particles of fluorite/quartz and barite/quartz systems using dispersed and dissolved air.

Their experimental apparatus allowed a continuous flotation procedure (continuous feed and float and tails removal) plus both dissolved and dispersed air flotation tests in the same flotation
cell.

Sodium oleate was used as a collector and sodium silicate was used as a depressant against quartz flotation. Experimental results reported by Klassen and Mescheriakov are shown in Figure 9, 10 and 11.

The term, α, is defined (18) as the concentration of minerals capable of undergoing flotation in the original mixture (or grade of valuable mineral component in the feed).

All figures show that dissolved air flotation results in an improved recovery and grade (content) when compared to dispersed air flotation. Regrettably, some key data are missing from these analyses. The error bands and reproducibility were not indicated for the experiments; the details of conditions, such as amount of collector, pH of operation, quality of water, were not specified.

The recovery of fluorite was found to decrease with an increase of sodium silicate as shown in Figures 9 and 10. The grade however increased with increased dosage of sodium silicate.

Sodium silicate acts as a flotation depressant for siliceous gangue, and it may also prevent the precipitation of calcium soaps. The results shown on Figures 9 and 10 suggest that sodium silicate interacts with both quartz and fluorite however the interaction with quartz is more pronounced. This is illustrated by the reduction in fluorite recovery with increased sodium silicate dosage along with an increase in the grade of fluorite. The reduction in fluorite recovery indicates sodium silicate depresses fluorite. However, the
FIGURE 9  Flotation of a Mixture of Fluorite and Quartz of Size less than 10 micron with Different Additions of Sodium Silicate 19

1) Vacuum Flotation
2) Dispersed Air Flotation
FIGURE 9

Addition of sodium silicate, g/ton

Recovery \( \varepsilon \), \%  
Content \( \beta \), \%  

\( \alpha = 25\% \)

Recovery

Content
FIGURE 10  Effect of Pulp Density on the Flotation of a Mixture of Fluorite and Quartz of a Size less than 10 microns

1) Vacuum Flotation
2) Dispersed Air Flotation
FIGURE 11  Flotation of Barite and Quartz of a Size less than 10 microns with Different Additions of Sodium Silicate

1) Vacuum Flotation
2) Dispersed Air Flotation
FIGURE 11

![Graph showing recovery and content as a function of solids content in the pulp.]

- Recovery (line 1)
- Content (line 2)

\( \alpha = 25\% \)

Content of solids in the pulp, \( \% \)

Recovery \( \epsilon, \% \)

Content \( \beta, \% \)
increase in fluorite grade indicates silica is depressed to a greater degree by sodium silicate.

At higher concentrations of sodium silicate, the grade of the concentrate increases more rapidly for D.A.F. than for dispersed air flotation. This suggests that air precipitated from solution overcomes more easily the energy barrier of the hydrated film caused by sodium silicate.

For barite flotation, from Figure 11 it can be seen that recovery varies linearly with pulp density with the recovery obtained from D.A.F. being 10 to 15% higher than dispersed air flotation. The grade of the concentrate however appears to reach an optimum at 15% pulp density.

Experimental results obtained under optimum conditions are given in Table 1. As shown in the previous Figures, the recovery using D.A.F. is superior compared to the recovery obtained by dispersed air flotation.

One interesting point is the loss in selectivity due to increased supersaturation. Concentrate grades of about 10% higher compared to those obtained using dispersed air flotation were found when the imposed pressure was reduced by 89.3 kPa. However if the imposed pressure was reduced further by 96.0 kPa concentrate grades from D.A.F. were approximately the same as the grade found for dispersed air flotation. At the higher applied vacuum however recoveries were about 8% higher than observed at the lower applied vacuum and about 20% higher than the recoveries observed using
Table 1: Klassen's Comparative Results of Flotation Experiments

<table>
<thead>
<tr>
<th>Reagent Addition (kg/tonne)</th>
<th>Sodium Oleate</th>
<th>Sodium Silicate</th>
<th>Pressure (kPa)</th>
<th>Weighted Floated W/W %</th>
<th>Content BaSO$_4$ W/W %</th>
<th>Recovery BaSO$_4$ W/W %</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.75</td>
<td>1.70</td>
<td></td>
<td>101.3</td>
<td>18.5</td>
<td>54.4</td>
<td>30.6</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>12.0</td>
<td>24.3</td>
<td>63.6</td>
<td>45.7</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>5.3</td>
<td>32.6</td>
<td>53.6</td>
<td>52.9</td>
</tr>
<tr>
<td>5.00</td>
<td>1.70</td>
<td></td>
<td>101.3</td>
<td>28.6</td>
<td>51.3</td>
<td>44.2</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>12.0</td>
<td>30.4</td>
<td>61.5</td>
<td>56.4</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>5.3</td>
<td>39.9</td>
<td>52.2</td>
<td>63.1</td>
</tr>
</tbody>
</table>

1) Reference (19)

2) pH = unknown
dispersed air flotation. This phenomena is similar to the effect of increasing the collector concentration as also shown in Table 1. Increasing the collector concentration from 3.75 kg/tonne to 5.0 kg/tonne resulted in little change in concentrate grade but increased the actual recovery observed.

Table 2 shows a size distribution of the flotation products. The performance of dissolved and dispersed air techniques are about equal in the 5 to 10 μm size range. The recovery of BaSO₄ in the -5 μm size range in the froth using D.A.F. is almost double that using dispersed air flotation. Therefore the superiority of dissolved air over dispersed air flotation in recovering slimes we can infer to be due to the ability of D.A.F. to float particles in the -5 μm range. At such a fine size the probability of bubble-solid contact is low in dispersed air flotation and thus recovery of the fine slimes is poor.
Table 2: Klassen's Size Analysis of Flotation Products of Barite Slimes

<table>
<thead>
<tr>
<th>Flotation Products</th>
<th>Normal Flotation (101.3 kPa)</th>
<th>Vacuum Flotation (&lt; 101.3 kPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weight W/W %</td>
<td>Content BaSO₄ W/W %</td>
</tr>
<tr>
<td>Float</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-10 + 5</td>
<td>14.5</td>
<td>65.4</td>
</tr>
<tr>
<td>-5 + 0</td>
<td>11.5</td>
<td>46.4</td>
</tr>
<tr>
<td>Total</td>
<td>26.0</td>
<td>57.0</td>
</tr>
<tr>
<td>Tails</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-10 + 5</td>
<td>30.1</td>
<td>29.0</td>
</tr>
<tr>
<td>-5 + 0</td>
<td>43.9</td>
<td>22.2</td>
</tr>
<tr>
<td>Total</td>
<td>74.0</td>
<td>24.9</td>
</tr>
<tr>
<td>Feed</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-10 + 0</td>
<td>100.0</td>
<td>33.3</td>
</tr>
</tbody>
</table>
CHAPTER 3
EXPERIMENTAL APPROACH

The experimental approach considers the apparatus and the procedure.

3.1 Apparatus
To investigate D.A.F. as a method of treating mineral slimes it was necessary that a batch, commercial dispersed air flotation machine be modified to allow the flotation cell to operate under different pressures.

This objective was achieved by enclosing the flotation cell, impeller and part of the standpipe within a pressure chamber as shown in Figure 12.

The chamber pressure could be increased above ambient pressure using air from an air cylinder or the chamber pressure could be reduced using a vacuum pump. Air was drawn directly from the chamber for dispersed air flotation thus allowing flotation experiments to be carried out at any pressure. The pressure chamber also acted as a collecting pan for the floated product. Make-up water could be added to the flotation cell from an external reservoir.

The overflowing froth could be washed down with water from a series of nozzles located above the front lip of the flotation cell.

A mercury manometer was used to measure the difference in
FIGURE 12  Flotation Cell and Pressure Chamber
FIGURE 13  Batch Dissolved/Dispersed

Air Flotation Apparatus
pressure between the cell and the ambient atmosphere.

A picture showing the flotation machine, pressure chamber and control panel is shown in Figure 13.

3.2 Experimental Procedure

Prior to actual flotation experiment, the desired quantities of feed material (barite and quartz), collector (sodium oleate) and depressant (sodium meta silicate) were weighed and added to the flotation cell. A pre-measured volume of distilled water was then added and the pH was adjusted to the desired value using 1.0 M NaOH or 1.0 M H$_2$SO$_4$ solutions.

The pressure chamber would then be sealed and the feed mixture was conditioned for 30 minutes. Air from an air cylinder could be used to pressurize the slurry to about 200 kPa if desired or the pressure could be left equal to ambient pressure.

After conditioning a dispersed air flotation test could be performed by simply opening the air line joining the standpipe and pressure chamber.

A D.A.F. test could be carried out by first venting any pressure in excess of atmospheric pressure and then evacuating the air in the pressure chamber with a vacuum pump. To allow only D.A.F. the impeller action of the flotation machine would be shut off (for a period of time referred to as deadtime). A combination of dissolved and dispersed air flotation could also be used by opening an the air line from the pressure chamber to the standpipe while evacuating the
chamber with the vacuum pump.

During flotation a set volume of makeup water would be added and any froth buildup at the front of the flotation cell would be washed away.

Flotation was allowed to continue until froth was no longer observed to overflow. At this point the flotation machine would be shut off, the pressure in the chamber equilibrated with the atmosphere and the float and tails collected separately into pre-weighted beakers.

The float and tails were then dried in an oven at about 150°C for 72 hours. The solids were then weighed and samples prepared for barium composition analysis by either neutron activation or by X-ray fluorescence.
RESULTS AND DISCUSSION
4.1 Introduction

A set of 33 experiments were performed to study the behaviour of D.A.F. as compared to dispersed air flotation.

The experiments were carried out under a variety of flotation conditions to ascertain not only where D.A.F. was effective but also to determine whether any enhancement in flotation using D.A.F. was significant compared to other influencing factors such as collector concentration and pH.

The results of each flotation test were characterized in terms of the weight % floated, WF; % BaSO₄ recovered, R; upgrading factor, UF; and the % separation efficiency, SE. WF describes the actual physical amount floated while UF, the ratio of the grade of the float to the grade of the initial feed, describes the degree of concentrating of the desired species. The other two factors, R and SE, are determined from WF and UF and are useful in assessing the merit of the actual flotation product.

The following sections will discuss the observed effects of D.A.F. and other factors on the flotation of BaSO₄ from quartz.

4.2 D.A.F.

To study the difference between D.A.F. and dispersed air
flotation we focus on the results of experiments where all experimental conditions were held constant except the mechanism used to introduce air into the slurry.

From test BPV-4 it was found that barite could be floated using only dissolved air with results similar to dispersed air flotation. Most D.A.F. tests however consisted of two stages. In the first stage only dissolved air was used. When the height of the froth remains stationary the second stage is commenced using dispersed air to carry over the existing froth and to float any activated minerals.

Using a random block statistical analysis on the observed results of all the experiments it was found that there was no significant overall improvement using D.A.F. compared to dispersed air flotation. The results of the statistical analysis are shown in Table 3 (see Appendix A4-2 for more detail). As illustrated in Table 3 the overall observed differences between the flotation results obtained for D.A.F. and dispersed air flotation were found to be statistically insignificant for all parameters. A histogram of the distribution of the difference between the weight % floated obtained using D.A.F. and dispersed air flotation is shown in Figure 14. Histograms for the differences in the % BaSO₄ recovered, upgrading factor and separation efficiency obtained in a similar manner are shown in Figures 15, 16, 17. As can be seen in these histograms the differences in the parameters are centered about zero indicating that no overall improvement was obtained by using D.A.F. in place of dispersed air flotation.
Figure 14: Distribution of differences in weight % floated between D.A.F. and dispersed air flotation.
Figure 15: Distribution of differences in % BASO$_4$ recovery between D.A.F. and dispersed air flotation.
FIGURE 16 DIFFERENCES IN UPGRADING FACTOR BETWEEN D.A.F. AND DISPERSED AIR FLOTATION

FREQUENCY, %

DIFFERENCE IN UPGRADING FACTOR BETWEEN D.A.F. AND DISPERSED AIR FLOTATION
FIGURE 17 DISTRIBUTION OF DIFFERENCES IN SEPARATION EFFICIENCY BETWEEN D.A.F. AND DISPERSED AIR FLOTATION
Furthermore it was found by using hypothesis tests that increasing the degree of supersaturation (or the available amount of dissolved air) made no significant difference in the observed parameters from flotation experiments. Results of this analysis in a similar form to Table 3 are given in Appendix A4-3.

An estimate of the experimental variance obtained from replicates was used for hypothesis tests on the differences observed between the two flotation mechanisms for individual sets of experiments. It was found that there was a statistical difference for seven sets of experiments (see Appendix A4-4 for the results of analysis). The difference observed in two sets, series 10 and 11, may be attributed to another factor which will be discussed later.

An improvement in the recovery of barite using D.A.F. was observed for low collector concentrations in the absence of depressant at pH = 4.0 and pH = 7.0. This improvement was mainly observed in the weight % floated. Under similar conditions except at pH = 10 D.A.F. was found to reduce the upgrading factor and separation efficiency of SrSO₄. A reduction in the same parameters using D.A.F. was also found for the flotation of BaSO₄ at the 90% level of confidence.

These changes due to D.A.F. may be rationalized in terms of the activating effect of D.A.F. The conditions which D.A.F. was found to improve flotation are also the least favourable conditions for flotation of barite that were studied. Under these conditions there may not have been an ample amount of oleate ions available to
Table 3: Random Block Analysis of the Observed Differences between D.A.F. and Dispersed Air Flotation, $d^*$

<table>
<thead>
<tr>
<th></th>
<th>Flotation Parameters, $y$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weight Floated W/W %</td>
</tr>
<tr>
<td>$d$</td>
<td>1.90</td>
</tr>
<tr>
<td>Var($d$)</td>
<td>86.12</td>
</tr>
<tr>
<td>Var($d$)</td>
<td>4.78</td>
</tr>
<tr>
<td>Calculated T value</td>
<td>0.87</td>
</tr>
<tr>
<td>Significant at 95% Confidence Level</td>
<td>No</td>
</tr>
</tbody>
</table>

1) $t = 2.110$ at 95% confidence level on 17 d.f., Ref. (25).

$d^* = y_{D.A.F.} - y_{Dispersed}$
sufficiently adsorb onto the barite surface. Thus a portion of the barite may not have been hydrophobic enough to successfully attach itself to an air bubble after a collision. However the mineral may have been hydrophobic enough to allow dissolved air to precipitate onto its surface and thus activate it for further flotation.

Similarly at pH = 10 quartz may become suitable for oleate ion adsorption if a polyvalent cation is available (26) as was the case due to the large amount of $S_\text{r}SO_4$ present in the ore. The activated quartz would compete with the barite for the adsorption of oleate ions and as a result may become slightly hydrophobic. This would enhance the amount of dissolved air successfully precipitating onto quartz and thus activating it for flotation. This increased flotation of quartz would reduce the upgrading factor of BaSO$_4$ as was observed.

A factorial design analysis of the results given in Appendices A4-12 to A4-15 confirms the previously discussed observations that D.A.F. did not significantly effect the flotation of BaSO$_4$ statistically relative to other factors except for case of low collector concentration and no depressant at different values of pH.

The fact that D.A.F. does not improve the flotation of BaSO$_4$ except under adverse conditions suggests that the poor flotation of slimes is not due to a low probability of mineral/bubble collision but instead the result of insufficient treatment of the mineral surface for flotation.
4.3 Collector Concentration

A total of 13 experiments were performed to study the effect of the concentration of the collector using three concentration levels of 1.0, 3.0 and 5.0 kg of sodium oleate/tonne of ore. The upper limit of 5 kg/tonne was chosen in accordance with Klassen's work (19) and the lower level of 1 kg/tonne was based on the suggested (27) upper limit for barite flotation using sodium oleate by conventional methods. Sodium oleate also acted as a frother in these experiments due to its excellent frothing ability.

Since the collector is the key reagent in making the mineral surfaces hydrophobic it is important that an ample amount be present to ensure a sufficient change in the hydrophobicity of the mineral. An excessive amount of collector is undesirable since it may lead to the activation of undesired gangue or result in excessive concentration of surfactant at the air bubble interface making strong mineral attachment difficult.

Larger collector concentrations are needed for fine slimes than for larger particles since there is a significant increase in the surface area for smaller particles. Also due to excessive grinding, the mineral surface may have more high energy surface sites for collector adsorption.

The experimental results suggest that the collector concentration was a significant factor. In general, an increase in all flotation parameters was observed with an increase of collector concentration. Figures 18–21 show the improvement observed in the
FIGURE 18  EFFECT OF COLLECTOR CONCENTRATION ON THE WEIGHT % FLOATED

COLLECTOR CONCENTRATION, KG SODIUM OLEATE/TONNE OF ORE

WEIGHT % FLOATED

DISPERSED AIR FLOTATION
D.A.F.
FIGURE 19  EFFECT OF COLLECTOR CONCENTRATION ON
% BASO₄ RECOVERY

COLLECTOR CONCENTRATION, KG SODIUM OLEATE/TONNE OF ORE

% BASO₄ RECOVERY

- - - - - - - - - - - DISPERSED AIR FLOTATION
D.A.F.
FIGURE 20  EFFECT OF COLLECTOR CONCENTRATION ON THE UPRGRADING FACTOR

COLLECTOR CONCENTRATION, KG SODIUM OLEATE/TONNE OF ORE

UPGRADING FACTOR

- DISPERSED AIR FLOTATION (D.A.F.)
Figure 21. Effect of Collector Concentration on % Separation Efficiency.

Collector Concentration, kg sodium oleate/tonne of ore.

- Dispersed Air Flotation (D.A.F.)
- D.A.F.
flotation of \( \text{BaSO}_4 \) due to collector concentration in the presence of depressant. Figure 18 shows that for an increase of collector concentration from 1.0 to 3.0 kg/tonne the resulting improvement is mainly due to an increase in the weight % floated. An increase of the collector concentration from 3.0 to 5.0 kg/tonne also results in an improvement of the upgrading factor. This behaviour may be rationalized by the following mechanism. At low collector concentrations it may be assumed that the oleate ions adsorb on minerals with the higher energy sites. Increasing the oleate concentration extends the number of mineral particles undergoing collector adsorption and thus activated for flotation. Up to the mid collector concentration level of 3 kg/tonne these high energy adsorption sites may have been predominantly due to grinding and may not reflect the true nature of the mineral surface. As the collector concentration increases however oleate ions adsorb onto the lower energy sites which are characteristic of barite. Similar adsorption sites for quartz are less numerous. Thus the amount of the collector adsorbed on barite increases relative to the amount adsorbed onto quartz. Consequently the probability that a collision with an air bubble will result in successful attachment increases for barite at a greater rate than that for quartz. The result is reflected in the improvement of the upgrading factor as observed in experiments.

A factorial design analysis given in Appendix A4-12 shows the effect of a collector concentration to be statistically significant.
in improving all the measured flotation parameters. When compared with the effect of D.A.F., the effect of collector concentration is found to be larger by a factor of over 3 times for the upgrading factor and up to about 30 times larger for weight % floated.

The fact that collector concentrations far in excess of those that are normally used for barite flotation make a significant improvement further suggest the importance of proper treatment of the mineral surface in recovering slimes.

4.4 Depressant

Sodium Meta Silicate produced by the Nichols Chemical Co. (99.98% pure) was used in 13 of the experiments to depress the flotation of silica. In these experiments 1.7 kg of sodium silicate/tonne of ore was added to correspond to the same dosages used by Klassen (19).

One role of sodium silicate is to disperse siliceous and iron oxide slimes. The dispersed slimes become strongly hydrated. Consequently, the probability for flotation is reduced (19) because of the lower probability of mineral/bubble collision and lower adhesion forces between the mineral and the bubble.

Sodium silicate also minimizes the precipitation of calcium soaps in hard water by forming complexes with the dissolved cations (19). The precipitation of calcium soaps is undesirable since it reduces the effective collector concentration.

The depressant may also adsorb on the desired mineral and reduce
its flotability.

The general overall expected behaviour of the depressant would be a lower weight % floated due to deactivation of silica, lower valuable mineral recoveries due to some deactivation of the desired mineral but improved upgrading factors since the silica is more strongly depressed than the valuable ore. In cases where soap precipitation is a problem the addition of depressant may have the effect of improving the weight % floated, % \( \text{BaSO}_4 \) recovered and upgrading factors as a result of an increase in the effective collector concentration.

In the experimental study the presence of depressant was found to improve the weight % floated and % \( \text{BaSO}_4 \) recovered for the impure barite ore containing celestite (\( \text{SrSO}_4 \)) using dispersed air flotation. Statistical analyses shown in Appendix A4-6 however show that there was no statistical difference using depressant in the flotation of pure \( \text{BaSO}_4 \) powder. This indicates that the presence of the \( \text{Sr} \) impurity is an apparent problem in the flotation of barite.

Solubility data shown in Table 4 shows that \( \text{BaSO}_4 \) is relatively insoluble in water however \( \text{SrSO}_4 \) is 40 to 50 times more soluble. As a consequence the \( \text{SrSO}_4 \) may have dissolved to an appreciable extent and \( \text{Sr} \) cations may be precipitating with sodium oleate and thus effectively reducing the collector concentration. This is the same problem found in using hard water due to calcium but the magnitude of the problem is reduced due to the lower solubility of \( \text{SrSO}_4 \).

The fact that depressant was ineffective at the 95% confidence
Table 4: Mineral Solubility Data

<table>
<thead>
<tr>
<th>Compound</th>
<th>Solubility (g/mL)</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Cold</td>
<td>Hot</td>
</tr>
<tr>
<td>BaSO$_4$</td>
<td>0.000222 (18)</td>
<td>0.000336 (50)</td>
</tr>
<tr>
<td>SrSO$_4$</td>
<td>0.0113 (0)</td>
<td>0.014 (30)</td>
</tr>
<tr>
<td>CaSO$_4$</td>
<td>0.209 (30)</td>
<td>0.030 (50)</td>
</tr>
</tbody>
</table>

1) Reference temperature in °C given in brackets.

2) Reference (28).
level for the flotation of pure BaSO\(_4\) is probably due to the fact that no strontium was present to reduce the collector concentration. At the 90\% confidence level however a significant difference using pure BaSO\(_4\) and dispersed air is found. This may be due to dissolved Ba precipitating out with the collector. However the degree of reduction in the collector concentration would not be as great as with the case where Sr is present due to the lower solubility of BaSO\(_4\). Since the degree of the effect of oleate precipitation is not as great, the improvement in using a depressant can only be realized at a lower level of confidence.

The fact that the effect of depressant is not realized at higher collector concentrations indicates that the reduction due to oleate precipitation is not as significant as the increase in collector concentration. The observation that the improvement in using the depressant is not as large or statistically significant when D.A.F. is used instead of dispersed air flotation suggests that the dissolved air compensates for the reduced collector concentration by activating poorly treated mineral surfaces.

A factorial design analysis shown in Appendix A4-12 and A4-13 shows that the effect of depressant is significant in improving the weight % of floated and the % barite recovery. The magnitude of the depressant effect was also found to be several times larger than the effect of D.A.F. but not as great as the effects of concentration or purity of BaSO\(_4\). The larger effect of the purity of BaSO\(_4\) may be indicative of either an insufficient amount of sodium silicate to
handle all the dissolved Sr or due to favourable mineral surface properties of the pure BaSO$_4$ powder relative to the properties of the ground barite ore.

4.5 Purity of BaSO$_4$

Two different sources of BaSO$_4$ were used in the flotation experiments.

For 8 of the experiments BaSO$_4$ powder (99.98% pure) obtained from J.T. Baker Chemical Co. was used. White barite ore (containing approximately 38% BaSO$_4$ and 62% SrSO$_4$) from Madoc, Ontario was used in the rest of the experiments.

Experiments were performed at pH = 7 and a collector concentration of 1.0 kg/tonne of ore with and without depressant.

The presence of SrSO$_4$ in the ore can have a negative effect on the flotation of BaSO$_4$ as discussed in the section on the effect of depressant.

Divalent Sr cations can precipitate with the sodium oleate and thus reduce the effective collector concentration. The Sr cations may also adsorb onto the quartz surface and thus activate the quartz for flotation. These factors can be expected to hinder the flotation of barite.

The difference in the procedure used to produce the fine particles may also influence the flotation behaviour. The pure BaSO$_4$ powder was produced by precipitation and as a result may have significantly different surface properties compared to the freshly
ground barite ore. The crushed barite ore can be expected to have a higher surface energy as a result of comminution and may result in an increased collector requirement. The particle size of the BaSO₄ mineral from the two different sources also differs considerably. Using transmission electron microscopy the geometric number average particle diameter for the ground barite ore from Madoc was found to be 0.22 μm while the pure BaSO₄ powder had a geometric number average particle diameter of 0.298 μm. However as shown in Appendix A6 the geometric standard deviation for the ground barite ore is about 2.71 while for the pure BaSO₄ powder the geometric standard deviation was 1.53. From the Kapteyn relationships (shown in Appendix A6) the geometric surface weighted average particle size for the pure BaSO₄ powder would be 0.43 μm while the geometric surface weighted average particle diameter for the barite ore is 1.59 μm. Calculation of the specific surface area assuming similar shape factors indicates that the specific surface area of the BaSO₄ powder is about 3.7 times larger than the specific surface area for barite ore. As a result of the larger surface area per unit mass the pure BaSO₄ powder is capable of adsorbing more collector than the impure barite ore.

Results from experimental studies indicate that pure BaSO₄ powder floated better than the barite ore regardless of the flotation mechanism or whether depressant was used. Statistical tests shown in Appendix A4-7, illustrated that the improvement in flotation using pure BaSO₄ powder was mainly observed in the improvement of the BaSO₄ recovered with some tests showing an improvement in the weight
A factorial design analysis of the effects of the purity of BaSO$_4$, depressant and D.A.F. (see Appendix A4-13) illustrates that the effect of the purity of BaSO$_4$ is several times larger than the effects of either depressant or D.A.F. From the statistical analysis it was found that the purity of BaSO$_4$ had a significant effect in improving the weight % floated, % BaSO$_4$ recovered and the % separation efficiency.

This apparent improvement may be attributed to the effect of divalent Sr cations reducing the effective collector concentration by precipitation in the flotation of the impure barite ore. This is illustrated by the effect of depressant in improving the flotation of the impure barite ore while having no statistical effect on the flotation of pure BaSO$_4$ powder. Results of the statistical analysis are found in Appendix A4-7.

It was also found that D.A.F. made a significant improvement in the flotation of impure barite ore but made no difference in the flotation of pure BaSO$_4$ powder in the absence of depressant (see Appendix A4-4). The improvement in the flotation of impure barite ore using D.A.F. has been attributed earlier to the poor mineral surface conditioning. The poor conditioning was stated to be due to the lack of sufficient collector resulting from Sr precipitation of oleate ions. The pure BaSO$_4$ powder does not suffer from the problem introduced by Sr and consequently the system has more available collector to adsorb onto the mineral. Therefore there is a lower probability that the
mineral is improperly conditioned and as a result the activation of poorly conditioned mineral particles by D.A.F. is not observed in the flotation of pure BaSO₄ powder.

The observations of the effect of the purity of BaSO₄ on flotation further suggest that the surface chemistry factor in the flotation of slimes is more significant than the mechanical aspect of mineral/bubble collision.

4.6 pH

Flotation experiments were performed at slurry pH values of 4.0, 7.0 and 10.0 to investigate the effect of the hydrogen ion concentration on flotation.

Hydrogen and hydroxyl ions may affect flotation by influencing the surface coverage of the collector by either competing with the collector through parallel adsorption onto the mineral or by displacing the collector from the mineral surface. Hydrogen and hydroxyl ions may also change the active form of the collector, the electrical properties of the mineral surface and the ionic composition of the pulp.

The pH may influence the hydrolysis of the surfactant. For sodium oleate the hydrolysis may be expressed by:

\[ C_{17}H_{33}COO^- + Na^+ + H_2O \rightleftharpoons C_{17}H_{33}COOH + Na^+ + OH^- \]

From the above equilibrium equation it can be seen that by Le Chatelier's principle an increase in the pH will shift the equilibrium to the left and thus produce more oleate ions in
solution. This is desirable since the oleate ion is the active form of the collector which adsorbs onto the mineral surface. At a significantly high value of pH however the hydroxyl ion may compete with the oleate ion for adsorption sites on the mineral.

The pH of the slurry may also affect the strength of the froth produced. Since sodium oleate is also used as a frothing agent the predominant form of oleate in the slurry effects the froth. Under alkaline conditions when the oleate ion form is favoured its presence as a charged entity in the froth results in a stable froth. Acidic conditions which favour the oleic acid form reduces the charge on the froth surface and thus reduces the froth's stability. This can result in a rapid breakdown of the froth and consequently cause any entrained or captured minerals to be released back into the slurry.

Hydrogen and hydroxyl ions may also act as the potential determining ions for the mineral surface. The electrokinetic potential of quartz becomes increasingly negative with an increase in pH with a zero point charge at a pH value below 4.0 (19). BaSO₄ has a positive electrokinetic potential which decreases with pH to a zero point charge at about 11.8 (19). The difference in sign of the electrokinetic potential allows an anionic surfactant such as sodium oleate to adsorb selectively onto BaSO₄.

One problem which may arise at higher values for pH, is the possible activation of quartz by polyvalent cations such as Ca²⁺ or Sr²⁺. The adsorption of these cations onto the quartz surface results in an overall positive surface charge. This allows quartz to
compete with BaSO₄ for collector adsorption. As a consequence the flotation process becomes less selective and thus the upgrading factor is reduced.

Experimental results for the effect of pH on flotation are shown in Figures 22 to 25. The improvement in weight % floated and BaSO₄ recovery with increasing pH may be attributed to corresponding increase in oleate ion concentration. Qualitatively more excessive and stable frothing was observed at pH = 10 and very weak and anemic frothing was observed at pH = 4 as expected.

One interesting observation is the apparent interaction between pH and D.A.F. which was found to be statistically significant in effecting the % BaSO₄ recovery, upgrading factor and separation efficiency as shown in Appendix A4-14.

At low values of pH D.A.F. resulted in superior selectivity while at higher values of pH D.A.F. caused a reduction in selectivity. This effect may be rationalized in terms of the activating effect of D.A.F. At lower pH values oleate adsorption onto silica is expected to be insignificant; however oleate adsorption on to BaSO₄ is anticipated although limited by the reduction in the oleate ion concentration. Since there are less oleate ions available for adsorption less mineral is expected to float as observed since the BaSO₄ is only weakly hydrophobic. Consequently a mineral bubble collision may not result in successful attachment. Slight activation of the BaSO₄ mineral may allow dissolved air to selectively precipitate onto the mineral, and thus further activate or float.
FIGURE 22  EFFECT OF PH ON WEIGHT % FLOATED

- DISPERSED AIR FLOTATION
- D.A.F.
Figure 23: Effect of pH on % BaSO₄ Recovery

% BaSO₄ Recovery

PH

Dispersed Air Flotation
D.A.F.
FIGURE 24  EFFECT OF PH ON THE UPGRADE FACTOR

![Graph showing the effect of pH on the upgrading factor. The graph plots PH on the x-axis and upgrading factor on the y-axis. Two lines are shown: one for dispersed air flotation (D.A.F.) indicated by solid line, and another for dispersed air flotation (D.A.F.) indicated by dotted line. The graph indicates a decrease in upgrading factor as PH increases from 4.0 to 7.0, followed by an increase as PH further increases.]
FIGURE 25  EFFECT OF pH ON % SEPARATION EFFICIENCY

% SEPARATION EFFICIENCY

PH

DISPERSED AIR FLOTATION
D.A.F.
BaSO₄. The result is a general improvement in the flotation results using D.A.F. compared to dispersed air flotation.

At high pH values (pH = 10) a similar situation exists except that under these conditions it is quartz which is weakly activated. The increased oleate ion concentration in the alkaline pulp ensures BaSO₄ is sufficiently activated and thus floats with a high selectivity using dispersed air flotation.

The quartz however may become slightly activated due to Sr ion adsorption but not sufficiently enough to adsorb a significant amount of collector to result in successful flotation using dispersed air. The quartz may become hydrophobic enough to allow dissolved air to precipitate onto its surface during D.A.F. This further activation may lead to quartz flotation. This enhanced quartz flotation resulting from D.A.F. reduces the selectivity as illustrated by the lower upgrading factor experimentally observed.

Experimental results for flotation tests at pH = 7.0 have already been discussed in the section on D.A.F.

From a factorial analysis the magnitude of the pH effect is several times larger than the D.A.F. effect on most parameters except for the upgrading factor. The pH/D.A.F. interaction effect is larger than either pH or D.A.F. effects on influencing the upgrading factor.

4.7 Aeration

To investigate the influence of air volume flowrate the rotational speed (RPM) of the impeller was varied for four of the
experiments. The air volume flowrate as a function of the impeller RPM using pure water for the slurry is given in Figure 26 for different liquid volumes in the cell.

The air volume flowrate at the lower impeller RPM was between 50 to 88% of the flowrate observed at higher impeller RPM depending on the liquid volume in the cell.

Increasing the impeller RPM, apart from introducing more air into the cell, may also result in smaller bubbles due to the increased agitation. The increase in the number of air bubbles will improve the probability of mineral/bubble collision. Also the reduced bubble size may result in an increase of the residence time of the air in the pulp.

The experimental results are shown on Figures 27 to 30. The enhanced performance of dispersed air flotation at low impeller RPM and at higher RPM using DAF are not characteristic of the anticipated behaviour based on previous experiments. Quantitatively these two experiments differed from the rest by having upgrading factors larger than normally observed. Qualitatively both these experiments had unexpected frothing during the conditioning period.

Eliminating the two abnormal experiments as not being due to the effect of different impeller RPM the remaining results show no significant response to the impeller RPM. The only possible exception is the apparent improvement in the upgrading factor at the higher impeller RPM.

Statistically no change in any of the flotation factors in the
FIGURE 26 VARIATION OF AIR FLOW RATE WITH IMPELLER SPEED

- □ CELL VOLUME OF 0.8L
- ■ CELL VOLUME OF 1.0L
- ○ CELL VOLUME OF 1.2L
FIGURE 27  EFFECT OF AERATION (RPM) ON THE
WEIGHT % FLOATED

WEIGHT % FLOATED

IMPELLER SPEED, RPM

---

- D.A.F.
- DISPERSED AIR FLOTATION
FIGURE 28  EFFECT OF AERATION (RPM) ON % BASSO₄ RECOVERY

- D.I.G.  (DISPERSED AIR FLOTATION)
- D.A.F.  (D.A.F.)

% BASSO₄ RECOVERY

IMPELLER SPEED, RPM
FIGURE 29  EFFECT OF AERATION (RPM) ON THE UPGRADE FACTOR

UPGRADING FACTOR

IMPELLER SPEED, RPM

--- DISPERSED AIR FLOTATION
--- D.A.F.
FIGURE 30  EFFECT OF AERATION (RPM) ON THE % SEPARATION EFFICIENCY

% SEPARATION EFFICIENCY

IMPELLER SPEED, RPM

---

---

DISPERSED AIR FLOTA TION
D.A.F.
remaining runs were found to be significant at the 5% level of significance. However, the improved upgrading factor at the higher RPM was found to be significant at the 10% level of significance. This enhanced selectivity may be due to increased aeration or due to demineralization caused by increased agitation.

The greater agitation increases the degree of turbulence resulting in larger separation forces. Larger particles are the first to be separated due to their larger mass (19). Since from particle size analysis (see Appendix A.6) geometric number average particle size of silica is more than 2.5 times that of barite, silica will be removed from air bubbles selectively. This could explain the increased selectivity observed at the higher impeller RPM.

A factorial design analysis given in Appendix A.4-15 shows that the effect of RPM and D.A.F. to be both statistically insignificant. The D.A.F./RPM interaction effect was found to be highly significant.

This second order interaction term corresponds directly to the effect of frothing during conditioning. This slow aeration during conditioning was a phenomena which could neither be controlled or predicted and was probably due to a small leak in one of the seals or fittings.

This phenomenon was observed in 9 out of 33 experiments. For three of the experiments using pure BaSO₄ the effect of frothing was not found to be significant. Similarly for two of the experiments using barite ore the degree of frothing was small and the observed flotation results were within the expected range based on other
experiments. However, for four of the experiments frothing was observed early in the conditioning period. The froth grew slowly but steadily and by the end of conditioning there was a substantial overflow of froth from the cell. These experiments resulted in significantly larger upgrading factors which were between 138% to 188% larger than the overall average upgrading factor observed for non-frothing experiments. The observed weight % floated for two of the experiments were lower than anticipated and slightly higher for the other two. The resulting separation efficiencies were about twice the anticipated value. The ratio of the weight of solids to the volume of the float product was also larger than normally observed in other runs.

Typically an average of about 0.18 g of solids per mL of froth was observed. However the average ratio, for runs when frothing was found to be a significant factor, was 0.33 g of solids per mL of froth.

One possible explanation of this improvement in flotation due to frothing may be a result of slow but prolonged aeration of the pulp causing the air bubbles to successfully attach and carry over only the most hydrophobic minerals which would be predominantly BaSO₄. As a consequence the froth contains a high grade of BaSO₄.

Another plausible explanation may be the reduction in mechanical transfer of solids into the froth by water entrained with the air bubbles. During the normal flotation experiments water entrained into the froth may not have sufficient time to drain back into the
slurry before being carried over. As a result any solids entrained with the water, but not legitimately attached to an air bubble are also carried over. The fact that the particles are so small increases the probability that they are entrained and do not have a chance to drain back into the slurry. In cases where frothing during conditioning was observed however the froth formation was usually slow and as a result more time was required before the froth overflowed out of the cell. This increased residence time may have allowed a sufficient amount of water and solids to drain back into the slurry before being carried over. Since less entrained gangue is present in the float product the upgrading factor is higher. Also since less water is carried over a higher ratio of solids to float product volume is observed. For the two cases where frothing during conditioning did not seem to improve the flotation of impure barite ore the actual degree of frothing was found to be small and to occur late in the conditioning period. Consequently the effect of frothing was too little and too late to be significant. For the flotation of pure $\text{BaSO}_4$ powder when frothing during conditioning occurred it was observed to be extensive and similar to the frothing normally observed for dispersed air flotation. The resulting solid weight to float product volume ratios were typical of the normal runs. This suggests that the degree of frothing was too great to allow a sufficient time for entrained water to drain back into the slurry.

It thus appears that small mineral particles provide a mechanical problem in flotation due to mechanical transfer of solids
into the froth by entrained water. This problem may be controlled by ensuring a sufficient residence time for the froth above the cell to allow adequate drainage of entrained water.

4.8 Celestite Flotation

Experimental results from four of the flotation tests were analyzed for Sr concentration to determine whether there was any significant difference in the performance of celestite flotation compared to barite flotation.

Both Sr and Ba are members of the alkaline earth metals and as a result have similar chemical properties. In conventional flotation both minerals use the same collector and modifying reagents.

Differences in the rate and degree of flotation between the two different minerals may exist due to different charge to size ratio affecting the collector adsorption. However, the magnitude of these differences may not be significant. Statistical analyses of the test results shown in Appendices A4-8 and A4-14 indicate that celestite and barite floated with the same overall performance. The effect of the different minerals on the flotation parameters were negligible suggesting that the flotation system had no preference for either mineral.

Although it was concluded earlier that Sr had a negative effect on barite flotation it apparently has an equally negative effect on its own flotation.
4.9 Summary

In the preceding sections it has been observed that D.A.F. results in improved BaSO₄ flotation only under certain experimental conditions. These experimental conditions correspond to situations where the mineral was only weakly activated for flotation. For example improvements were found at low collector concentrations, in the absence of a silica depressant and at low values of pH for the impure barite ore. At high pH values D.A.F. was found to improve the flotation of weakly activated quartz.

In all the cases where D.A.F. was found to make a significant change in the flotation of barite the results were explained in terms of D.A.F.'s effect in activating a weakly hydrophobic mineral surface. Furthermore other factors such as collector concentration, pH and mineral purity were found to have a more significant effect on the flotation of BaSO₄. These factors are also concerned with altering the chemistry of the mineral surface. It thus appears that the major problem in the flotation of barite slimes is the proper treatment of the mineral surface rather than Klassen's suggestion that the problem is due to poor mineral/bubble contact.

Earlier it had been stated that Klassen attributed the observed improvements using D.A.F. due to flotation of particles smaller than 5 μm.

It was reasoned that these particles were too small to be efficiently floated by conventional dispersed air methods due to the low probability of air/mineral contact. Klassen reports using a
particle size distribution containing 65% less than 5 μm (by sedimentation). In this experimental study 56% of the impure barite ore was less than 5 μm and over 99% of the pure BaSO₄ powder was less than 5 μm (based on mass weighting).

Then according to Klassen's theory there should be some improvement in the flotation of impure barite ore and an even larger improvement in flotation of pure BaSO₄ powder using D.A.F. No improvement however was observed in the flotation of the pure BaSO₄ and only slight improvement was found for the impure barite ore. Furthermore by Klassen's reasoning the pure BaSO₄ powder should have floated poorly using dispersed air flotation due to the fine size; however, it was found that the BaSO₄ powder actually floated better than the larger sized impure barite ore. Direct comparison with Klassen's results is difficult due to lack of information of his experimental conditions and the difference in actual flotation procedure. An attempt has been made as shown in Table 5. As can be readily seen there is a significant difference between the observed weight % floated and % BaSO₄ recovered. Klassen's lower values may be explained by the fact that he used a continuous experimental design and thus probably used shorter residence times. The upgrading factors reported by Klassen although higher are not statistically different than the ones observed in this study.

His slightly higher values may be due to the fact that he may have only collected the initially richer froth. In this study flotation occurred to depletion which would include the poorer quality
Table 5: Comparison of Klassen’s \(^{19}\) Results and Current Experimental Observations for the Flotation of Barite Slimes

<table>
<thead>
<tr>
<th>Flotation Parameter</th>
<th>Experiment No.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Klassen (^{19})</td>
</tr>
<tr>
<td>Volume of Precipitating Air (mL/L)</td>
<td>0.0</td>
</tr>
<tr>
<td>Weight Floated W/W %</td>
<td>28.6</td>
</tr>
<tr>
<td>Recovery BaSO(_4) W/W</td>
<td>44.2</td>
</tr>
<tr>
<td>Upgrading Factor W/W</td>
<td>1.55</td>
</tr>
<tr>
<td>Separation Efficiency W/W</td>
<td>15.77</td>
</tr>
</tbody>
</table>

1) Klassen’s results based on Table 1.

2) Collector concentration: 5.0 kg sodium oleate/tonne.

3) Depressant concentration 1.7 kg sodium silicate/tonne.

* Included to indicate trends.
froth in the later time stages. The fact that the upgrading factors are similar suggest that the ore floated by Klassen was similar to the one studied.

It is perhaps possible that the improved differences observed by Klassen using D.A.F. for particles less than 5 \( \mu \text{m} \) is due to the inability of the fine particles to adsorb a sufficient amount of collector to ensure that the majority of the mineral/bubble collisions resulted in successful adhesion. In D.A.F. however precipitating air may activate these weakly hydrophobic particles. The fact that Klassen observes a decrease in the upgrading factor but an improvement in weight % floated as the degree of supersaturation is increased further suggests that D.A.F. affects the surface properties more significantly than the mechanics of air/mineral collision.

The one mechanical problem in floating slimes is speculated to be the entrainment of unwanted gangue by water into the froth. This is based on the improved flotation observed when slow frothing during conditioning occurs. The most dramatic improvements were observed when this phenomena occurred which allowed a sufficient amount of time for the entrained gangue to drain back into the slurry.

In general however apart from this phenomena it was observed that factors which influenced the surface chemistry of the mineral were more predominant in influencing the flotation results. In retrospect the effect of D.A.F. on the flotation results was observed to be small relative to these factors.
To compare the relative magnitude of each effect they were normalized by dividing each one by the D.A.F. effect as shown in Table 6. The effects of aeration (RPM) and the actual mineral floated (SrSO₄ vs. BaSO₄) are generally not as large as the effect of D.A.F. The collector concentration and purity of the mineral are found to have the most significant effect on barite flotation. The mechanical transfer or frothing effect (measured by D.A.F./RPM interaction effect) was the next most significant effect followed by pH and silica depressent both of which were greater than the D.A.F. effect.

As a result of the narrow limits in which D.A.F. is useful and the small improvement it makes compared to dispersed air/flotation it appears that D.A.F. is not the key to the slime problem. The solution to this problem based on observations made from this study appear to lie in the proper conditioning of the mineral surface and in the elimination of mechanical transfer of gangue into the froth.

It should be noted that curves on flotation graphs are not correlation lines, but are included only so that the reader can see the trends. The mid points on each graph are duplicates with replicates so close as to be covered by the symbol.
Table 6: Magnitude of Effects of Flotation Variables Relative to D.A.F.

<table>
<thead>
<tr>
<th></th>
<th>Flotation Parameters, y</th>
<th>Weight Floated W/W %</th>
<th>Recovery BaSO₄ W/W %</th>
<th>Upgrading Factor W/W</th>
<th>Separation Efficiency W/W %</th>
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</thead>
<tbody>
<tr>
<td>D.A.F.</td>
<td></td>
<td>1.0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Collector Concentration</td>
<td></td>
<td>29.4</td>
<td>13.7</td>
<td>3.4</td>
<td>5.5</td>
</tr>
<tr>
<td>Purity of BaSO₄</td>
<td></td>
<td>5.4</td>
<td>12.5</td>
<td>4.0</td>
<td>15.6</td>
</tr>
<tr>
<td>RPM/D.A.F.*</td>
<td></td>
<td>1.9</td>
<td>10.4</td>
<td>2.9</td>
<td>10.8</td>
</tr>
<tr>
<td>pH</td>
<td></td>
<td>3.1</td>
<td>9.4</td>
<td>0.7</td>
<td>4.5</td>
</tr>
<tr>
<td>Depressant</td>
<td></td>
<td>9.1</td>
<td>5.1</td>
<td>0.6</td>
<td>3.0</td>
</tr>
<tr>
<td>RPM</td>
<td></td>
<td>0.9</td>
<td>1.7</td>
<td>0.4</td>
<td>0.3</td>
</tr>
</tbody>
</table>

* RPM/D.A.F. interaction effect corresponds to frothing during conditioning.
CONCLUSIONS
CHAPTER 5

CONCLUSIONS

Conclusions arrived at as a result of this study are now presented.

1) At a collector concentration of 1.0 kg of sodium oleate/tonne of ore it was found that $\text{BaSO}_4$ could be selectively floated from silica using only air precipitated from solution at pH 7.0.

2) Whereas Klassen claimed that D.A.F. yielded better $\text{BaSO}_4$ recovery and selectivity than observed for dispersed air flotation over a wide range of experimental conditions, our results indicate that improvements were limited to only a narrow range of conditions. Klassen's conditions included a range of sodium oleate concentrations of 3.75 kg to 5.0 kg/tonne of ore, a range of solids content in the pulp of 10 to 30% and a range of the grade of $\text{BaSO}_4$ in the feed of 25 to 33%. The pH of the slurry and the concentration of sodium meta silicate were not completely reported.

Our experimentally observed improvements were restricted to sodium oleate concentrations of 1.0 kg/tonne, approximately 13% solids content in the pulp, in the absence of depressant and at a slurry pH of 4.0 and 7.0. Improvements were only observed when an impure barite ore containing Sr was used. Improvements were observed to be predominantly in the weight % floated.
At pH = 10.0, D.A.F. produced significantly poorer results than dispersed air flotation with a reduction in the upgrading factor and separation efficiency.

Where the effect of D.A.F. was found to be significant its influence was attributed to its ability to activate poorly conditioned mineral surfaces.

Klassen attributed improvements to an increase in the probability of mineral/bubble contact.

3) Whereas Klassen found that the degree of supersaturation of the slurry by air influences the weight % floated and upgrading factor, our results show no significant influence can be attributed to this factor.

4) An increase in the concentration of sodium oleate, as the collector (in the range of 1 to 5 kg/tonne of ore), showed a significant increase in the flotation parameters. It was hypothesized that the observed improvements were due to the improved mineral surface conditioning. Increases in collector concentration were also concluded to be more influential in improving BaSO$_4$ flotation than the use of D.A.F.

5) The use of 1.7 kg of sodium meta silicate/tonne of ore, as a silica depressant, showed no significant effect on any of the flotation parameters for the separation of pure BaSO$_4$ from silica. However, the depressant was found to yield a significant increase in the weight % floated and % BaSO$_4$ recovered in the flotation of barite ore containing approximately 60% celestite.
Based on these observations, it is hypothesized that the sodium silicate prevented the precipitation of the collector by the soluble Sr originating from the celestite. The effect of the depressant for the flotation of the impure barite ore was found to be larger than the effect of D.A.F.

6) The flotation parameters for the beneficiation of pure BaSO$_4$ were found to be significantly larger than were observed for the impure barite ore. This was attributed to the negative effect of Sr on the collector concentration.

7) The weight % floated and % BaSO$_4$ recovered was found to increase with increasing pH. The interaction effect between pH and D.A.F. was found to be very significant in influencing the upgrading factor.

At pH = 4.0 an increase in the upgrading factor using D.A.F. was found while at pH = 10.0 a decrease was observed relative to results obtained using dispersed air flotation. It is speculated that at pH = 10 the adsorption of Sr onto silica results in a weak activation of silica for flotation. At pH = 4.0 probably the resulting lower oleate ion concentration only weakly activates the barite. The interaction effect of pH and D.A.F. arises from the ability of D.A.F. to further activate these poorly conditioned minerals.

8) The degree of aeration as controlled by the impeller speed (RPM) was found to be a statistically insignificant factor. On the other hand, the interaction effect of impeller speed and D.A.F.
was highly significant in improving the upgrading factor and separation efficiency. This interaction effect corresponds to the physically observed phenomena of frothing during conditioning. It is hypothesized that due to the slower rate of frothing observed during conditioning relative to normal flotation any entrained water and gangue in the froth had a better opportunity to drain back into the slurry. This hypothesis was supported by the observation that less water was found in the float product for these runs.

9) The flotation of celestite from silica was not found to be significantly different than the flotation of barite from silica under the same experimental conditions.

10) A comparison of the observed results of this study to those of Klassen's under similar experimental conditions (5.0 kg of sodium oleate and 1.7 kg of sodium meta silicate/tonne of ore) were found to be significantly different except for the upgrading factor. These differences were attributed to the different experimental procedure used by Klassen. Our experimental observations were made using a batch flotation scheme with flotation occurring until the froth was depleted. Klassen used a continuous flotation scheme which would allow control over the quantity of ore floated.

11) As a result of a relative ranking of effects, all factors which influence the surface chemistry of the mineral (collector concentration, pH, depressant and purity of BaSO₄) were found to
be more significant in improving flotation than the effect of D.A.F. The observed phenomena of frothing during conditioning was also found to have a larger effect than D.A.F.

12) The geometric number average particle diameter for silica was 0.59 μm with a geometric standard deviation of 2.80. This was obtained from the standard grinding procedure used in this research.

13) The geometric number average particle diameter for the impure barite ore was 0.22 μm with a geometric standard deviation of 2.71. This was obtained by the standard grinding procedure used in this research.

14) The geometric number average particle diameter for the pure BaSO₄ was 0.30 μm with geometric standard deviation of 1.53. This is for a sample as purchased from the J.T. Baker Chemical Co.
REFERENCES


22. Wrobel, S., Mine and Quarry, p. 313 (1952), As cited in Ref. 19, p. 122.


NOMENCLATURE
NOMENCLATURE

- \( b \) = estimated value of the co-efficient of least squares equation
- \( c \) = concentration of dissolved air in water
- \( D_p \) = particle diameter
- \( \langle D_p \rangle_G \) = geometric number average particle diameter
- \( G \langle D_p \rangle_{32} \) = geometric surface weighted average particle diameter
- \( G \langle D_p \rangle_{43} \) = geometric mass weighted average particle diameter
- \( d \) = difference between response variables
- \( \Delta G_a \) = change in free energy for coalescence attachment of bubbles
- \( \Delta G_b \) = change in free energy for direct attachment of bubble to solid
- \( GF \) = grade of \( BaSO_4 \) in float
- \( GI \) = grade of \( BaSO_4 \) in feed
- \( GT \) = grade of \( BaSO_4 \) in tails
- \( H \) = Henry's law constant
- \( N \) = number of nucleating bubbles per unit time; concentration of dissolved air in solution
- \( P_1 \) = conditioning pressure
- \( P_f \) = flotation pressure
- \( P \) = pressure
- \( P_v \) = vapour pressure
- \( R \) = gas constant
- \( r \) = radius
\( S_1 = \) surface area of attachment
\( S_2 = \) surface area of spherical segment
\( SE = \) \% BaSO₄ separation efficiency
\( s = \) standard deviation
\( s_p = \) pooled standard deviation
\( T = \) temperature; calculated hypothesis test value
\( \text{UF} = \) BaSO₄ upgrading factor
\( \Delta V = \) maximum volume of precipitating air per litre of solution
\( \text{VI} = \) volume of feed
\( \text{VT} = \) volume of tails
\( W_a = \) work to form gas/liquid interface
\( W_b = \) work to form a film
\( W_c = \) work to fill a bubble with vapour
\( W_1 = \) total work to form a bubble
\( W_2 = \) work to precipitate bubble on a solid surface
\( \text{WF} = \) weight of solids in float
\( \text{WI} = \) weight of solids in feed
\( \text{WT} = \) weight of solids in tails
\( X = \) independent variable
\( x = \) mole fraction of air in water
\( Y = \) response variable
\( y = \) response variable
\( \bar{y} = \) average response variable
\( \alpha = \) grade of valuable mineral in feed
\( \beta = \) co-efficient of least squares equation
\( \gamma_{L/G} \) = liquid/gas interfacial energy
\( \gamma_{OW} \) = oil/water interfacial energy
\( \gamma_{SO} \) = solid/oil interfacial energy
\( \gamma_{SW} \) = solid/water interfacial energy
\( \lambda \) = probability of formation of a surviving bubble
\( \theta \) = contact angle
\( \sigma_G \) = geometric standard deviation
APPENDIX 1  DEVELOPMENT OF EQUATION TO PREDICT
VOLUME OF AIR PRECIPITATING FROM SOLUTION
APPENDIX A1 Development of Equation to Predict Volume of Air Precipitating from Solution

In order to predict the maximum volume of air capable of precipitating from solution as a result of a reduction of the imposed pressure it was assumed that the solubility of air in water could be described by Henry's Law:

\[ P_{\text{air}} = Hx \]  

where

- \( P_{\text{air}} \) = imposed air pressure
- \( H \) = Hennry's Law Constant
- \( x \) = mole fraction of air in water

The reported\(^{29}\) value for Henry's Law constant for air dissolved in water at 20\(^\circ\)C is 6.726 \times 10^6 kPa. Then the ratio of the number of moles of air to water is given by

\[ \frac{\text{Moles of air}}{\text{Moles of water}} = \frac{x}{1-x} = \frac{P/H}{1-P/H} \]  

However since the maximum imposed pressure is about 200 kPa then \( P/H \ll 1 \). Equation A2 can be simplified to

\[ \frac{\text{Moles of air}}{\text{Moles of water}} = \frac{P}{H} \]  

For this range of pressures the error introduced by this assumption is less than 0.01%. Then if the M.W. of H\(_2\)O is 18.02 and the density
of water at 20°C is about 0.997 kg/L then equation A3 can be expressed in terms of moles of air per litre of solution. This equation expressed as a sole function of pressure becomes:

\[ N \left( \frac{\text{mole of air}}{L \text{ of water}} \right) = (8.292 \times 10^{-6} \frac{\text{mole of air}}{L \cdot \text{kPa}}) P(\text{kPa}) \quad A4 \]

Then the number of moles of air precipitated, \( \Delta N \), as a result of changing the imposed pressure from \( P_1 \) to \( P_f \) is given by

\[ \Delta N \left( \frac{\text{mole of air}}{L \text{ of water}} \right) = (8.292 \times 10^{-6} \frac{\text{mole of air}}{L \cdot \text{kPa}}) (P_1 - P_f) \quad A5 \]

The above equation assumes that the solution is saturated with air before and after changing the imposed pressure. If it is further assumed that the ideal gas law is valid, changes in volume due to the saturation of air with water vapour is negligible and that pressure differences resulting from the hydrostatic head are small then equation A5 may be rewritten in terms of the volume of air precipitating from 1 L of solution.

Using the ideal gas law the volume of gas released is

\[ \Delta V = \frac{\Delta N \cdot RT}{P_f} \quad A6 \]

Then if

\[ R = 8.311.7 \frac{\text{mL} \cdot \text{kPa}}{\text{gmol} \cdot \text{K}} \]

and substituting A5 into A6 at 20°C (293K) the expression becomes

\[ \Delta V = 20.19 \left( \frac{\text{mL of air}}{L \text{ of water}} \right) \left( \frac{P_1 - P_f}{P_f} \right) \quad A7 \]

The above equation was used to calculate the theoretical amount of air precipitating from solution during D.A.F. tests.
APPENDIX 2  EXPERIMENTAL DETAILS
APPENDIX A2  Experimental Details

A detailed description of the experimental procedures and equipment is provided in the following sections.

A2.a  Experimental Procedure

A detailed discussion of procedures used in this experiment is provided in the following sections.

A2.a.1  Mineral Preparation

White barite ore from Madoc, Ontario was obtained from the Arbour Scientific Co. Silica sand was obtained courtesy of Dofasco Ltd., Hamilton, Ontario.

The barite ore and silica sand were both wet ground (approximately 1:1 weight ratio of solids to distilled, de-ionized water) in a porcelain grinding jar with a Burundum grinding media. Mineral samples were ground until 100% by weight passed through a -325 mesh (-44 μm) sieve. For ease of storage the samples were dried at 70°C. Mineral samples underwent further dry grinding for a period of about 24 hours.

To ensure purity of the silica sand the mineral was contacted with 10% HCl for several hours then rinsed with distilled, de-ionized water until the pH of the solution was between 6.5 and 7.0. No special precautions were taken to purify the barite ore.
Several of the experimental tests used pure BaSO₄ powder obtained from the J.T. Baker Chemical Co. No effort was made to further reduce the size or increase the purity of the BaSO₄ powder.

A2.a.2 Pre-Flotation Procedure

The preparation of the slurry for flotation was similar for the different mechanisms of flotation used.

Desired quantities of the solids were pre-weighed and added to the flotation cell located in the pressure chamber. Pre-determined reagent dosages based on the weight of solids being treated were weighed out and dissolved in an aqueous solution. The concentrated reagent solution was added to the flotation cell along with distilled de-ionized water such that the total volume of water added was constant (1.25 L of water). The slurry was agitated and the pH adjusted to the desired value using 1.0 M NaOH or 1.0 M H₂SO₄. Once the desired pH was established the pressure chamber was sealed and the slurry was conditioned for 30 minutes at the same RPM to be used during flotation experiments.

To increase the solubility of air in solution the pressure chamber could be pressurized during the conditioning period for some experiments (BPVT series). This was accomplished by closing all vents and opening the pressurized air valve and the standpipe air valve. Air was introduced at both locations to prevent the slurry from being forced up the standpipe or to prevent pre-mature flotation caused by a pressure gradient between the standpipe and pressure
chamber.

A2. a. 3 Flotation Procedure

The flotation mechanisms used included conventional dispersed air flotation (BDAT series), combined dispersed and dissolved air flotation with the slurry being pre-pressurized during conditioning (BPVT series) and without pre-pressurization of the slurry (BVT series).

The simplest procedure used was for the dispersed air flotation tests. At the end of the 30 minute conditioning period the standpipe air valve was opened allowing air from the chamber to be induced down the standpipe. The froth was allowed to overflow out of the flotation cell into the pressure chamber. Froth forming at the front lip of the flotation cell was continuously washed away using the float spray located directly above the front lip. After 2 minutes, distilled de-ionized make up water was slowly added (usually 250 mL). The flotation was allowed to proceed until froth was no longer observed to overflow out of the cell at which time the flotation machine was turned off. The pressure chamber was then opened and float and tails samples collected.

The procedure used for the D.A.F. experiments was similar to the one described above with the addition of several extra steps. At the end of conditioning the pressure in the pressure chamber is released to the atmosphere by opening the vent. Once the pressure has equilibrated the vent is closed; then the vacuum pump is turned on
and the flotation machine is shut off simultaneously. The flotation machine is left off for a period of 4 to 6 minutes (referred to as the deadtime) during which time any flotation is due directly to dissolved air precipitating from solution. The pressure rises to within 10% of its final value within the first 90 seconds. During the deadtime any high pressure air in the standpipe or make-up water line is released into the pressure chamber. Froth forming at the front lip is continuously washed away by the overhead float spray. After the deadtime has elapsed the flotation machine is turned on and dispersed air flotation is commenced. Dispersed air flotation is used to help carry over any existing froth and to float particles activated by dissolved air. Make-up water (approximately 250 mL) is added shortly after dispersed air flotation begins. Flotation is allowed to continue until no further overflow of froth from the cell is observed at which time the flotation machine and the vacuum pump are turned off. The final pressure in the chamber is recorded and then the vent is opened allowing air into the chamber restoring it to atmospheric pressure. The pressure chamber is then opened and the float and tail products collected.

A2.a.4 Analysis of Flotation Products

After each experiment the float and tails product are collected in pre-weighed 1 L glass beakers. The approximate volume of the tails is noted to be used in determining the solids weight to volume ratio of the float indirectly. The float and tails samples are
allowed to dry in an oven for 3 days at 150°C then placed in a
desciclator for 1 hour prior to weighing.

After weighing samples of both float and tail products are
prepared for analysis of Ba composition by Neutron Activation or by
x-ray fluorescence.

A2.2 Equipment Description

A flowsheet of the equipment used is given in Figure A2.1. Equipment
specification and suppliers are given in Table A2.1.

A commercial batch flotation machine (Denver, Model D-12) and a
250 g (1.71 L) stainless steel flotation cell were used for the
experiments. The flotation cell was modified by trimming all sides
down to the same level to allow the froth to overflow with more ease.
A copper tube (0.635 cm O.D.) was also added as an inlet at the
bottom of the cell. This tube allowed make-up water to be added into
the cell. Water was added from a 0.5 L graduated reservoir, and the
flow was controlled using the make-up water valve (H). Air in this
line could be removed using the make-up water bleed valve (I).

The flotation cell and the impeller of the flotation machine
were enclosed in a pressure chamber. The pressure chamber was built
from a 25.4 cm O.D. (22.9 cm I.D.) by 17.8 cm long acrylic cylinder.
The ends of the cylinder were enclosed by two 27.94 cm diameter, 1.27
cm thick stainless steel plates which were held in place by twelve
0.635 cm diameter threaded rods and nuts. The top plate was machined
to allow six 0.635 cm diameter inlet lines into the chamber. These
<table>
<thead>
<tr>
<th>Equipment</th>
<th>Specifications</th>
<th>Suppliers</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pressure Chamber</td>
<td>See Appendix A2.b for details</td>
<td>Machine Shop, Eng. Bldg., McMaster University, Hamilton, Ontario</td>
</tr>
<tr>
<td>Vacuum Pump</td>
<td>186 W - 1725 PPM</td>
<td>Central Scientific Co., Chicago, Ill., U.S.A.</td>
</tr>
<tr>
<td>Swagelok Fittings and Valves</td>
<td>Type 316 Stainless Steel</td>
<td>Niagara Valve, 102 Parkdale Ave. W., Hamilton, Ontario</td>
</tr>
<tr>
<td>0.625 cm O.D. tubings</td>
<td>Type 316 Stainless Steel</td>
<td>Niagara Valve, Hamilton, Ontario</td>
</tr>
<tr>
<td>Compressed Air</td>
<td>Breathing Quality</td>
<td>Canadian Liquid Air, 850 Burlington St. E., Hamilton, Ontario</td>
</tr>
<tr>
<td>Vacuum Tube</td>
<td>6.4 mm Bore 4.8 mm Wall Thickness</td>
<td>Fisher Scientific Co., 184 Railside Rd., Don Mills, Ontario</td>
</tr>
<tr>
<td>pH Meter</td>
<td>Analog Meter</td>
<td>Fisher Scientific Co., Don Mills, Ontario</td>
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<td>Graduated Cylindrical Funnel</td>
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<td>Fisher Scientific Co., Don Mills, Ontario</td>
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<td>Gas Wash Bottles</td>
<td>250 mL</td>
<td>Fisher Scientific Co., Don Mills, Ontario</td>
</tr>
<tr>
<td>Manometer</td>
<td>u-tube</td>
<td>Fisher Scientific Co., Don Mills, Ontario</td>
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<tr>
<td>Equipment</td>
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<td>Suppliers</td>
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<td>-----------------</td>
<td>----------------</td>
<td>------------------------------------------------</td>
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<tr>
<td>Silica Sand</td>
<td>-200 Mesh</td>
<td>Dofasco Inc., 1330 Burlington St., Hamilton, Ontario</td>
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<tr>
<td>Barite Ore</td>
<td>Crystalline</td>
<td>Arbour Scientific Co., 1820 Mattawa St., Cooksville, Ont.</td>
</tr>
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<td>Barium Sulfate</td>
<td>Reagent Grade</td>
<td>Canlab Chemicals, 80 Jutland Rd., Toronto, Ontario</td>
</tr>
<tr>
<td>Sodium Oleate</td>
<td>Reagent Grade</td>
<td>Canlab Chemicals, Toronto, Ontario</td>
</tr>
<tr>
<td>Sodium Meta Silicate</td>
<td>Reagent Grade</td>
<td>Canlab Chemicals, Toronto, Ontario</td>
</tr>
</tbody>
</table>
inlets were used for:

1) float-spray water
2) make-up water
3) vent line
4) pressure release valve
5) vacuum line
6) pressurized air line

A 10.1 cm diameter hole was also made in the top plate to allow the impeller to be lowered or raised from the pressure chamber. A seal about the standpipe of the flotation machine was accomplished by an acrylic flange which was tightly fitted onto the standpipe. This flange could be bolted to the top plate covering the hole.

To reduce air leakage two O-rings were located between the flange and the standpipe, and one O-ring was situated between the flange and the top plate. Spigots were also machined into both plates to seat the acrylic cylinder. A plastic sealant was used to reduce leaks where the plates and cylinder came in contact. A conical skimmer for the purpose of forcing froth to overflow was attached to the upper flange.

The cell was pressurized by compressed air from cylinders. Air could be introduced directly into the pressure chamber and standpipe was controlled by the pressurized air valve (G). With the pressurized air valve (G) closed the same air line could be used to draw air from the pressure chamber into the standpipe. This allowed conventional dispersed air flotation to occur under any pressure.
A vacuum was obtained using a 186 W mechanical vacuum pump. The vacuum line was located at the same level as the top of the flotation cell to prevent excessive froth buildup in the pressure chamber. Froth removed by this fashion was collected in one of two gas wash bottles placed in series to prevent liquid from reaching the vacuum pump. The final pressure could be controlled by admitting air into the vacuum line through the vacuum bleed valve (D). The pressure could be equilibrated to atmospheric pressure using the vent (J).

A mercury manometer was used to measure the chamber pressure relative to the external ambient pressure.

A 12.7 cm long (0.635 cm diameter) copper tube sealed at both ends with perforations along its length was located above the major overflow lip of the cell. This tube acted as a float spray (M) which physically washed down overflowing froth with water. This was crucial since the cell was close to the chamber wall at this location and as a result froth tended to build up here. The perforations on the float spray (M) were aimed such that wash water would clear the top lip but not enter into the flotation cell. Tap water was used, and the flowrate was adjusted using the float spray control valve.
FIGURE A2-1  Dissolved/Dispersed Air Flotation Flowsheet

A - Float Spray Control Valve
B - Vacuum Control Valve
C - Gas Wash Bottles
D - Vacuum Bleed Valve
E - Standpipe
F - Standpipe Air Valve
G - Pressurized Air Valve
H - Make-Up Water Valve
I - Make-Up Water Bleed Valve
J - Vent
K - Pressure Chamber
L - Flotation Cell
M - Float Spray
N - Pressure Release Valve
O - Impeller
FIGURE A2-1  DISSOLVED/DISPERSED AIR FLOTATION FLOWSHEET

AIR SUPPLY  MAKE-UP WATER  MERCURY MANOMETER  VACUUM PUMP
APPENDIX 3  EXPERIMENTAL RESULTS
EXPERIMENT NO. BDAT-1

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g Barite ore, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 1.0 g Sodium Oleate
Depressant: 0.34 g Sodium Meta Silicate
pH: 6.9 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 101.3 kPa
Initial BaSO₄ Grade: 9.56%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Time - min.; Flotation 8 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 64.60
Overall Mass Balance Error %: 0.0
Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.17 g./mL
Max. Dissolved Air Available for Precipitation: 0.0 mL/L of Solution
% BaSO₄ Grade: Float 13.71; Tails 3.83
% BaSO₄ Mass Balance Error: 6.83
% BaSO₄ Recovered: 89.23 ± 4.83
Upgrading Factor: 1.381 ± 0.075
% Separation Efficiency: 24.63 ± 4.826

COMMENTS
EXPERIMENT NO. BVT-1

DATE: 27/08/81

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 silica
Initial Slurry Volume: 1.25 L
Collector: 1.0 g. Sodium Oleate
Depressant: 0.34 g. Sodium Meta Silicate
pH: 7.0 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 5.8 ± 0.3 kPa
Initial BaSO₄ Grade: 9.56%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Time - min.; Flotation 8 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 55.78 ± 0.18
Overall Mass Balance Error %: -0.25
Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.17 g./mL
Max. Dissolved Air Available for Precipitation: 329.7 mL/L of Solution
%BaSO₄ Grade: Float 14.44; Tails 1.00
%BaSO₄ Mass Balance Error: -11.33
%BaSO₄ Recovered: 89.72 ± 8.01
Upgrading Factor: 1.608 ± 0.138
% Separation Efficiency: 33.95 ± 7.83

COMMENTS

Flotation is faster than observed in BDAT1.
EXPERIMENT NO. BPVT1

DATE: 28/08/81

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica

Initial Slurry Volume: 1.25 L

Collector: 1.0 g Sodium Oleate

Depressant: 0.34 g Sodium Meta Silicate

pH: 7.0 ± 0.1

R.P.M.: 900 ± 10

Pressures: Conditioning 195.4 ± 3 kPa; Flotation 9.9 ± 0.3 kPa

Initial BaSO₄ Grade: 9.56%

Volume of Make-up Water: 0.25 L

Times: Conditioning 34 min.; Dead Time - min.; Flotation 6 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 59.45 ± 0.50

Overall Mass Balance Error %: -0.70

Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.18 g./mL

Max. Dissolved Air Available for Precipitation: 377.4 mL/L of Solution

%BaSO₄ Grade: Float 13.61; Tails 1.27

%BaSO₄ Mass Balance Error: -10.52

%BaSO₄ Recovered: 89.40 ± 7.44

Upgrading Factor: 1.503 ± 0.113

% Separation Efficiency: 29.95 ± 6.95

COMMENTS

pH of float and tails is 6.8.
EXPERIMENT NO. BDAT-2  

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica

Initial Slurry Volume: 1.25 L

Collector: 0.60 g. Sodium Oleate

Depressant: 0.34 g. Sodium Meta Silicate

pH: 7.0 ± 0.1

R.P.M.: 900 ± 10

Pressures: Conditioning 101.3 kPa; Flotation 101.3 kPa

Initial BaSO₄ Grade: 9.56%

Volume of Make-up Water: 0.25 L

Times: Conditioning 30 min.; Dead Time - min.; Flotation 8 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 63.32% ± 0.07

Overall Mass Balance Error %: -0.10

Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.20 g./mL

Max. Dissolved Air Available for Precipitation: 0.0 mL/L of Solution

%BaSO₄ Grade: Float 11.71; Tails 6.19

%BaSO₄ Mass Balance Error: +1.21

%BaSO₄ Recovered: 76.92 ± 0.87

Upgrading Factor: 1.214 ± 0.015

% Separation Efficiency: 13.57 ± 0.94

COMMENTS
EXPERIMENT NO. BVT2

DATE: 04/09/81

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica

Initial Slurry Volume: 1.25 L

Collector: 0.60 g. Sodium Oleate

Depressant: 0.34 g. Sodium Meta Silicate

pH: 7.0 ± 0.1

R.P.M.: 900 ± 10

Pressures: Conditioning 101.3 kPa; Flotation 10.6 ± 0.3 kPa

Initial BaSO₄ Grade: 9.56%

Volume of Make-up Water: 0.26 L

Times: Conditioning 30 min.; Dead Time - min.; Flotation 8 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 62.65 ± 1.91

Overall Mass Balance Error %: -2.70

Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.18 g./mL

Max. Dissolved Air Available for Precipitation: 172.7 mL/L of Solution

% BaSO₄ Grade: Float 12.43; Tails 6.60

% BaSO₄ Mass Balance Error: +4.56

% BaSO₄ Recovered: 77.42 ± 3.22

Upgrading Factor: 1.237 ± 0.089

% Separation Efficiency: 14.78 ± 5.13

COMMENTS
EXPERIMENT NO. BPVT2

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica

Initial Slurry Volume: 1.25 L

Collector: 0.60 g. Sodium Oleate

Depressant: 0.34 g. Sodium Meta Silicate

pH: 7.1 ± 0.1

R.P.M.: 900 ± 10

Pressures: Conditioning 199.8 ± 0.3 kPa; Flotation 15.3 ± 0.3 kPa

Initial BaSO₄ Grade: 9.56%

Volume of Make-up Water: 0.25 L

Times: Conditioning 30 min.; Dead Time – min.; Flotation 6 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 66.23 ± 1.17

Overall Mass Balance Error %: -1.65

Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.18 g./mL

Max. Dissolved Air Available for Precipitation: 242.5 mL/L of Solution

%BaSO₄ Grade: Float 11.44; Tails 5.04

%BaSO₄ Mass Balance Error: -4.37

%BaSO₄ Recovered: 80.44 ± 3.09

Upgrading Factor: 1.214 ± 0.025

% Separation Efficiency: 14.22 ± 1.92

COMMENTS
EXPERIMENT NO. BDAT3

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 0.20 g Sodium Oleate
Depressant: 0.34 g Sodium Meta Silicate
pH: 6.8 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 101.3 kPa
Initial BaSO₄ Grade: 9.56%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Time — min.; Flotation 6 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 45.00 ± 0.28
Overall Mass Balance Error %: −0.40
Solids Wt./Slurry Volume: Feed 0.13 g/mL; Float 0.19 g/mL
Max. Dissolved Air Available for Precipitation: 0.0 mL/L of Solution
%BaSO₄ Grade: Float 11.59; Tails 7.51
%BaSO₄ Mass Balance Error: −2.64
%BaSO₄ Recovered: 55.63 ± 1.87
Upgrading Factor: 1.236 ± 0.034
% Separation Efficiency: 10.63 ± 1.58

COMMENTS

Less frothing observed during this experiment.
EXPERIMENT NO. BVT3

DATE: 10/09/81

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica

Initial Slurry Volume: 1.25 L

Collector: 0.20 g. Sodium Oleate

Depressant: 0.34 g. Sodium Meta Silicate

pH: 6.8 ± 1

R.P.M.: 900 ± 10

Pressures: Conditioning 101.3 kPa; Flotation 12.3 ± 0.3 kPa

Initial \( \text{BaSO}_4 \) Grade: 9.56%

Volume of Make-up Water: 0.25 L

Times: Conditioning 30 min.; Dead Time – min.; Flotation 6 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 42.08 ± 0.46

Overall Mass Balance Error %: -0.65

Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.21 g./mL

Max. Dissolved Air Available for Precipitation: 145.1 mL/L of Solution

% \( \text{BaSO}_4 \) Grade: Float 12.62; Tails 7.49

% \( \text{BaSO}_4 \) Mass Balance Error: 0.24

% \( \text{BaSO}_4 \) Recovered: 54.99 ± 0.17

Upgrading Factor: 1.307 ± 0.018

% Separation Efficiency: 12.92 ± 0.63

COMMENTS
EXPERIMENT NO. BPVT-3

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica

Initial Slurry Volume: 1.25 L

Collector: 0.20 g. Sodium Oleate

Depressant: 0.34 g. Sodium Meta Silicate

pH: 7.0 ± 0.1

R.P.M.: 900 ± 10

Pressures: Conditioning 198.1 ± 0.3 kPa; Flotation 10.6 ± 0.3 kPa

Initial BaSO₄ Grade: 9.56%

Volume of Make-up Water: 0.25 L

Times: Conditioning 30 min.; Dead Time - min.; Flotation 6 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 47.33 ± 0.46

Overall Mass Balance Error %: -0.65

Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.24 g./mL

Max. Dissolved Air Available for Precipitation: 357.1 mL/L of Solution

%BaSO₄ Grade: Float 11.30; Tails 8.05

%BaSO₄ Mass Balance Error: -0.36

%BaSO₄ Recovered: 55.74 ± 0.26

Upgrading Factor: 1.178 ± 0.006

% Separation Efficiency: 8.42 ± 0.19

COMMENTS
EXPERIMENT NO. BDAT4

DATE: 17/09/81

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica

Initial Slurry Volume: 1.25 L

Collector: 0.20 g Sodium Oleate

Depressant: 0 g Sodium Meta Silicate

pH: 6.9 ± 0.1

R.P.M.: 900 ± 10

Pressures: Conditioning 101.3 kPa; Flotation 101.3 kPa

Initial BaSO4 Grade: 9.56%

Volume of Make-up Water: 0.25 L

Times: Conditioning 30 min.; Dead Time - min.; Flotation 8 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 23.00 ± 0.42

Overall Mass Balance Error %: -0.60

Solids Wt./Slurry Volume: Feed 0.13 g./ml; Float 0.36 g./ml

Max. Dissolved Air Available for Precipitation: 0.0 mL/L of Solution

BaSO4 Grade: Float 12.80; Tails 8.39

BaSO4 Mass Balance Error: -2.29

BaSO4 Recovered: 31.54 ± 1.62

Upgrading Factor: 1.371 ± 0.045

% Separation Efficiency: 8.54 ± 1.20

COMMENTS
EXPERIMENT NO. BPVT-4

DATE: 17/09/81

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica

Initial Slurry Volume: 1.25 L

Collector: 0.20 g Sodium Oleate

Depressant: 0.0 g Sodium Meta Silicate

pH: 6.9 ± 0.1

R.P.M.: 900 ± 10

Pressures: Conditioning 199.5 ± 0.3 kPa; Flotation 8.5 ± 0.3 kPa

Initial BaSO₄ Grade: 9.56%

Volume of Make-up Water: 0.25 L

Times: Conditioning 25 min.; Dead Time 9 min.; Flotation 9 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 37.88 ± 1.732

Overall Mass Balance Error %: -2.45

Solids Wt./Slurry Volume: Feed 0.13 g/mL; Float 0.42 g/mL

Max. Dissolved Air Available for Precipitation: 450.1 mL/L of Solution

%BaSO₄ Grade: Float 10.96; Tails 7.99

%BaSO₄ Mass Balance Error: -7.08

%BaSO₄ Recovered: 45.56 ± 5.01

Upgrading Factor: 1.201 ± 0.077

% Separation Efficiency: 7.68 ± 3.28

COMMENTS
EXPERIMENT NO. BDAT5

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 1.0 g. Sodium Oleate
Depressant: 0.0 g. Sodium Meta Silicate
pH: 7.0 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 101.3 kPa
Initial BaSO₄ Grade: 9.56%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min; Dead Time - min.; Flotation 8 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 59.23 ± 0.25
Overall Mass Balance Error %: -0.35
Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.17 g./mL
Max. Dissolved Air Available for Precipitation: 0.0 mL/L of Solution
%BaSO₄ Grade: Float 14.07; Tails 3.73
%BaSO₄ Mass Balance Error: 2.75
%BaSO₄ Recovered: 85.53 ± 1.94
Upgrading Factor: 1.444 ± 0.039
% Separation Efficiency: 26.31 ± 2.19

COMMENTS

Slight frothing observed during the end of the conditioning period.
EXPERIMENT NO. BVT-5

DATE: 18/09/81

EXPERIMENTAL CONDITIONS

Wt. of Feed Used:  50 g barite ore, 150 g silica
Initial Slurry Volume:  1.25 L
Collector:  1.0 g. Sodium Oleate
Depressant:  0.0 g. Sodium Meta Silicate
pH:  7.0 ± 0.1
R.P.M.:  900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 10.6 ± 0.3 kPa
Initial BaSO₄ Grade:  9.56%
Volume of Make-up Water:  0.50 L
Times: Conditioning 30 min.; Dead Time 6 min.; Flotation 8 min.

EXPERIMENTAL RESULTS

Wt. % Floated:  50.10 ± 0.42
Overall Mass Balance Error %:  -0.60
Solids Wt./Slurry Volume:  Feed 0.11 g./mL; Float 0.15 g./mL
Max. Dissolved Air Available for Precipitation:  172.7 mL/L of Solution
%BaSO₄ Grade:  Float 14.62; Tails 2.38
%BaSO₄ Mass Balance Error:  -11.49
%BaSO₄ Recovered:  81.91 ± 8.13
Upgrading Factor:  1.634 ± 0.148
% Separation Efficiency:  31.81 ± 7.70

COMMENTS

Slight frothing observed during the end of the conditioning period.
EXPERIMENT NO. BDAT6

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 0.20 g Sodium Olate
Depressant: 0.0 g Sodium Meta Silicate
pH: 7.1 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 101.3 kPa
Initial BaSO₄ Grade: 9.56%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Time - min.; Flotation 8 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 25.10 ± 0.50
Overall Mass Balance Error %: -0.70
Solids Wt./Slurry Volume: Feed 0.13 g/mL; Float 0.18 g/mL
Max. Dissolved Air Available for Precipitation: 0.0 mL/L of Solution
%BaSO₄ Grade: Float 11.46; Tails 9.25
%BaSO₄ Mass Balance Error: +1.80
%BaSO₄ Recovered: 28.77 ± 1.27
Upgrading Factor: 1.147 ± 0.073
% Separation Efficiency: 3.67 ± 1.77

COMMENTS
EXPERIMENT NO. BVT-6

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 0.20 g. Sodium Oleate
Depressant: 0.0 g. Sodium Meta Silicate
pH: 6.9 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 24.8 ± 0.3 kPa.
Initial BaSO₄ Grade: 9.56%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Time 4 min.; Flotation 12 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 37.53 ± 0.39
Overall Mass Balance Error %: -0.55
Solids Wt./Slurry Volume: Feed 0.13 g/mL; Float 0.25 g/mL
Max. Dissolved Air Available for Precipitation: 62.1 mL/L of Solution
%BaSO₄ Grade: Float 11.45; Tails 8.31
%BaSO₄ Mass Balance Error: -1.32
%BaSO₄ Recovered: 45.27 ± 0.93
Upgrading Factor: 1.206 ± 0.120
% Separation Efficiency: 7.75 ± 0.542

COMMENTS
EXPERIMENT NO. BDAT7

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 1.0 g Sodium Oleate
Depressant: 0.0 g Sodium Meta Silicate
pH: 7.1 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 101.3 kPa
Initial BaSO₄ Grade: 9.56%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Time - min.; Flotation 9 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 35.28 ± 0.18
Overall Mass Balance Error %: -0.25
Solids Wt./Slurry Volume: Feed 0.13 g/mL; Float 0.31 g/mL
Max. Dissolved Air Available for Precipitation: 0.0 mL/L of Solution
%BaSO₄ Grade: Float 25.50; Tails 0.67
%BaSO₄ Mass Balance Error: -1.71
%BaSO₄ Recovered: 94.62 ± 1.21
Upgrading Factor: 2.682 ± 0.210
% Separation Efficiency: 59.34 ± 1.03

COMMENTS

Slow but steady frothing observed during most of the conditioning period.
EXPERIMENT NO. BVT7

DATE: 01/10/81

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 1.0 g. Sodium Oleate
Depressant: 0.0 g. Sodium Meta Silicate
pH: 6.9 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 10.2 ± 0.3 kPa
Initial BaSO₄ Grade: 9.56%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Time 6 min.; Flotation 11 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 32.18 ± 0.18
Overall Mass Balance Error %: -0.25
Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.43 g./mL
Max. Dissolved Air Available for Precipitation: 179.1 mL/L of Solution
%BaSO₄ Grade: Float 25.02; Tails 2.00
%BaSO₄ Mass Balance Error: -1.96
%BaSO₄ Recovered: 84.86 ± 1.38
Upgrading Factor: 2.673 ± 0.028
% Separation Efficiency: 52.69 ± 1.20

COMMENTS

Slow but steady frothing observed during most of the conditioning period.
EXPERIMENT NO. BDAT8

DATE: 07/10/81

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 0.20 g. Sodium Oleate
Depressant: 0.0 g. Sodium Meta Silicate
pH: 10.0 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 101.3 kPa
Initial BaSO₄ Grade: 9.56%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Time - min.; Flotation 9 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 46.48 ± 0.32
Overall Mass Balance Error %: -0.45
Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.23 g./mL
Max. Dissolved Air Available for Precipitation: 0.0 mL/L of Solution
% BaSO₄ Grade: Float 17.62; Tails 2.72
% BaSO₄ Mass Balance Error: +0.41
% BaSO₄ Recovered: 85.04 ± 0.29
Upgrading Factor: 1.830 ± 0.019
% Separation Efficiency: 38.46 ± 0.61

COMMENTS

1) Rich tick froth
2) Rapid Formation
EXPERIMENT NO. BDATBA

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 0.20 g. Sodium Olate
Depressant: 0.0 g. Sodium Meta Silicate
pH: 10.1 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 101.3 kPa
Initial SrSO₄ Grade: 18.2%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Time - min.; Flotation 9 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 46.48 ± 0.32
Overall Mass Balance Error %: -0.45

Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.32 g./mL
Max. Dissolved Air Available for Precipitation: 0.0 mL/L of Solution
SrSO₄ Grade: Float 34.33; Tails 1.82
SrSO₄ Mass Balance Error: -7.40
SrSO₄ Recovered: 90.95 ± 5.25
Upgrading Factor: 1.957 ± 0.100
% Separation Efficiency: 44.48 ± 4.93

COMMENTS
EXPERIMENT NO. BVT8

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 0.20 g. Sodium Oleate
Depressant: 0.0 g. Sodium Meta Silicate
pH: 10.0 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 14.0 ± 0.3 kPa
Initial BaSO₄ Grade: 9.56%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Time 6 min.; Flotation 10 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 58.28 ± 0.46
Overall Mass Balance Error %: -0.65
Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.20 g./mL
Max. Dissolved Air Available for Precipitation: 125.9 mL/L of Solution
%BaSO₄ Grade: Float 13.02; Tails 3.00
%BaSO₄ Mass Balance Error: -8.08
%BaSO₄ Recovered: 82.97 ± 5.72
Upgrading Factor: 1.423 ± 0.087
% Separation Efficiency: 24.69 ± 5.25

COMMENTS
EXPERIMENT NO. BVT8A

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 0.20 g Sodium Oleate
Depressant: 0.0 g Sodium Meta Silicate
pH: 10.0 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 14.0 ± 0.3 kPa
Initial SrSO₄ Grade: 18.20%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Time 6 min.; Flotation 10 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 58.28 ± 0.46
Overall Mass Balance Error %: -0.65
Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.20 g./mL
Max. Dissolved Air Available for Precipitation: 125.9 mL/L of Solution
SrSO₄ Grade: Float 23.45; Tails 5.21
SrSO₄ Mass Balance Error: -13.46
SrSO₄ Recovered: 81.41 ± 9.53
Upgrading Factor: 1.396 ± 0.15
% Separation Efficiency: 23.13 ± 9.07

COMMENTS
EXPERIMENT NO. BDAT9

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 silica
Initial Slurry Volume: 1.25 L
Collector: 0.20 g Sodium Oleate
Depressant: 0.0 g Sodium Meta Silicate
pH: 4.0 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 101.3 kPa
Initial BaSO₄ Grade: 9.56%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Time — min.; Flotation 9 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 12.93 ± 0.18
Overall Mass Balance Error %: -0.25
Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.26 g./mL
Max. Dissolved Air Available for Precipitation: 0.0 mL/L of Solution
%BaSO₄ Grade: Float 11.91; Tails 8.73
%BaSO₄ Mass Balance Error: -4.65
%BaSO₄ Recovered: 18.27 ± 3.29
Upgrading Factor: 1.412 ± 0.235
% Separation Efficiency: 5.35 ± 3.11.

COMMENTS

1) Frothing is less extensive.
2) Froth is very fragile.
EXPERIMENT NO. BDAT9A

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 0.20 g. Sodium Olate
Depressant: 0.0 g. Sodium Meta Silicate
pH: 4.0 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 101.3 kPa
Initial Sr-SO₄ Grade: 18.20%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Time - min.; Flotation 9 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 12.93 ± 0.18
Overall Mass Balance Error %: -0.25
Solids Wi./Slurry Volume: Feed 0.13 g./mL; Float 0.26 g./mL
Max.-Dissolved Air Available for Precipitation: 0.0 mL/L of Solution
Sr-SO₄ Mass Balance Error: -9.38
Sr-SO₄ Recovered: 19.62 ± 6.65
Upgrading Factor: 1.515 ± 0.493
% Separation Efficiency: 6.70 ± 6.47

COMMENTS
EXPERIMENT NO. BVT9

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica

Initial Slurry Volume: 1.25 L

Collector: 0.20 g. Sodium Oleate

Depressant: 0.0 g. Sodium Meta Silicate

pH: 4.0 ± 0.1

R.P.M.: 900 ± 10

Pressures: Conditioning 101.3 kPa; Flotation 11.2 ± 0.3 kPa

Initial $\text{BaSO}_4$ Grade: 9.45%

Volume of Make-up Water: 0.25 L

Times: Conditioning 30 min.; Float Time 6 min.; Flotation 10 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 23.53 ± 0.53

Overall Mass Balance Error %: -0.75

Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.46 g./mL

Max. Dissolved Air Available for Precipitation: 161.1 mL/L of Solution

$\%\text{BaSO}_4$ Grade: Float 15.31; Tails 7.64

$\%\text{BaSO}_4$ Mass Balance Error: -2.11

$\%\text{BaSO}_4$ Recovered: 38.13 ± 1.49

Upgrading Factor: 1.620 ± 0.027

$\%$ Separation Efficiency: 14.61 ± 0.96

COMMENTS
EXPERIMENT NO. BVT9A  DATE: 08/10/81

EXPERIMENTAL CONDITIONS

Wt. of Feed Used:  50 g barite ore, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 0.20 g. Sodium Oleate
Depressant: 0.0 g. Sodium Meta Silicate
pH: 4.0 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 11.2 ± 0.3 kPa
Initial SrSO₄ Grade: 18.20%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Time 6 min.; Flotation 10 or 167? min.

EXPERIMENTAL RESULTS

Wt. % Floated: 25.93 ± 0.35
Overall Mass Balance Error %: -0.75
Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.46 g./mL
Max. Dissolved Air Available for Precipitation: 161.1 mL/L of Solution
SrSO₄ Grade: Float 23.59; Tails 14.04
SrSO₄ Mass Balance Error: -11.26
SrSO₄ Recovered: 35.65 ± 7.98
Upgrading Factor: 1.512 ± 0.31
% Separation Efficiency: 12.13 ± 7.45

COMMENTS
EXPERIMENT NO. BDAT-10

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 0.20 g. Sodium Oleate
Depressant: 0.0 g. Sodium Meta Silicate
pH: 7.0 ± 0.1
R.P.M.: 800 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 101.3 kPa
Initial BaSO₄ Grade: 9.56%
Volume of Make-up Water: 0.25 L
Times; Conditioning 30 min.; Dead Time - min.; Flotation 10 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 43.50 ± 0.42
Overall Mass Balance Error %: -0.60
Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.37 g./mL
Max. Dissolved Air Available for Precipitation: 0.0 mL/L of Solution
%BaSO₄ Grade: Float 18.79; Tails 1.15
%BaSO₄ Mass Balance Error: -8.33
%BaSO₄ Recovered: 89.07 ± 5.89
Upgrading Factor: 2.047 ± 0.115
% Separation Efficiency: 45.57 ± 5.47

COMMENTS

Slow frothing observed during most of the conditioning period.
EXPERIMENT NO. BVT10

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 0.20 g. Sodium Oleate
Depressant: 0.0 g. Sodium Meta Silicate
pH: 7.0 ± 0.1
R.P.M.: 800 ± 10
Pressures: Conditioning 103.1 kPa; Flotation 14.0 ± 0.3 kPa
Initial BaSO₄ Grade: 9.56%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Time 5 min.; Flotation 10 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 37.68 ± 0.53
Overall Mass Balance Error %: -0.75
Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.22 g./mL
Max. Dissolved Air Available for Precipitation: 125.9 mL/L of Solution
%BaSO₄ Grade: Float 12.65; Tails 7.39
%BaSO₄ Mass Balance Error: -2.76
%BaSO₄ Recovered: 50.73 ± 1.95
Upgrading Factor: 1.346 ± 0.033
% Separation Efficiency: 13.06 ± 1.42

COMMENTS
EXPERIMENT NO. BDAT11

DATE: 15/10/81

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica

Initial Slurry Volume: 1.25 L

Collector: 0.20 g. Sodium Oleate

Depressant: 0.0 g. Sodium Meta Silicate

pH: 7.0 ± 0.1

R.P.M.: 1000 ± 10

Pressures: Conditioning 101.3 kPa; Flotation 101.3 kPa

Initial BaSO₄ Grade: 9.56%

Volume of Make-up Water: 0.25 L

Times: Conditioning 30 min.; Dead Time - min.; Flotation 14 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 25.75 ± 0.21

Overall Mass Balance Error %: -0.30

Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float - g./mL

Max. Dissolved Air Available for Precipitation: 0.0 mL/L of Solution

%BaSO₄ Grade: Float 15.19; Tails 7.59

%BaSO₄ Mass Balance Error: -1.29

%BaSO₄ Recovered: 39.73 ± 0.91

Upgrading Factor: 1.605 ± 0.023

% Separation Efficiency: 14.98 ± 0.70

COMMENTS

Flotation is longer than normal.
EXPERIMENT NO. BVT11                  DATE: 16/10/81

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g barite ore, 150 g silica

Initial Slurry Volume: 1.25 L

Collector: 0.20 g. Sodium Olate

Depressant: 0.0 g. Sodium Meta Silicate

pH: 7.0 ± 0.1

R.P.M.: 1000 ± 10

Pressures: Conditioning 101.3 kPa; Flotation 11.9 ± 0.3 kPa

Initial BaSO₄ Grade: 9.46%

Volume of Make-up Water: 0.26 L

Times: Conditioning 30 min.; Dead Time 5 min.; Flotation 11 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 44.30 ± 0.71

Overall Mass Balance Error %: -1.0

Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.29 g./mL

Max. Dissolved Air Available for Precipitation: 150.8 mL/L of Solution

% BaSO₄ Grade: Float 18.21; Tails 1.89

% BaSO₄ Mass Balance Error: -5.66

% BaSO₄ Recovered: 86.26 ± 4.00

Upgrading Factor: 1.947 ± 0.059

% Separation Efficiency: 41.96 ± 3.29

COMMENTS

Slow but steady frothing observed during conditioning.
EXPERIMENT NO. BDAT12

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g BaSO₄ powder, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 0.20 g. Sodium Oleate
Depressant: 0.0 g. Sodium Meta Silicate
pH: 7.0 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 101.3 kPa
Initial BaSO₄ Grade: 25.0%
Volume of Make-up Water: 0.275 L
Times: Conditioning 30 min.; Dead Time - min.; Flotation 10 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 45.15 ± 0.21
Overall Mass Balance Error %: -0.30
Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.19 g./mL
Max. Dissolved Air Available for Precipitation: 0.0 mL/L of Solution
%BaSO₄ Grade: Float 40.55; Tails 17.58
%BaSO₄ Mass Balance Error: +11.46
%BaSO₄ Recovered: 67.26 ± 8.10
Upgrading Factor: 1.490 ± 0.186
% Separation Efficiency: 22.11 ± 8.31

COMMENTS

Slight frothing during the end of the conditioning period.
EXPERIMENT NO. BVT-12

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g BaSO$_4$ Powder, 150 g silica

Initial Slurry Volume: 1.25 L

Collector: 0.20 g. Sodium Oleate

Depressant: 0.0 g. Sodium Meta Silicate

pH: 7.0 ± 0.1

R.P.M.: 900 ± 10

Pressures: Conditioning 101.3 kPa; Flotation 11.2 ± 0.3 kPa

Initial BaSO$_4$ Grade: 25.0%

Volume of Make-up Water: 0.25 L

Times: Conditioning 30 min.; Dead Time 5 min.; Flotation 8 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 54.03 ± 0.39

Overall Mass Balance Error %: -0.55

Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.22 g./mL

Max. Dissolved Air Available for Precipitation: 161.1 mL/L of Solution

% BaSO$_4$ Grade: Float 35.39; Tails 17.09

% BaSO$_4$ Mass Balance Error: +7.33

% BaSO$_4$ Recovered: 72.42 ± 5.18

Upgrading Factor: 1.341 ± 0.106

% Separation Efficiency: 18.40 ± 5.57

COMMENTS

Slight frothing observed during the end of the conditioning period.
EXPERIMENT NO. BDAT-13

DATE: 22/10/81

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g BaSO₄ powder, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 0.20 g Sodium Oleate
Depressant: 0.0 g Sodium Meta Silicate
pH: 7.0 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 101.3 kPa
Initial BaSO₄ Grade: 25.0%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Time - min.; Flotation 8 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 58.20 ± 0.28
Overall Mass Balance Error %: -0.40
Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.18 g./mL
Max. Dissolved Air Available for Precipitation: 0.0 mL/L of Solution
%BaSO₄ Grade: Float 30.37; Tails 15.31
%BaSO₄ Mass Balance Error: -4.07
%BaSO₄ Recovered: 72.49 ± 2.88
Upgrading Factor: 1.245 ± 0.043
% Separation Efficiency: 14.29 ± 2.59

COMMENTS

Slight frothing observed during the conditioning period.
EXPERIMENT NO. BVT-13

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g BaSO₄ powder, 150 g silica
Initial Slurry Volume: 1.25 L
Collector: 0.20 g. Sodium Oleate
Depressant: 0.0 g. Sodium Meta Silicate
pH: 7.0 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 11.9 ± 0.3 kPa
Initial BaSO₄ Grade: 25.0%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Time 5 min.; Flotation 8 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 46.45 ± 0.50
Overall Mass Balance Error %: -0.70
Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.18 g./mL
Max. Dissolved Air Available for Precipitation: 150.8 mL/L of Solution
%BaSO₄ Grade: Float 37.68; Tails 21.78
%BaSO₄ Mass Balance Error: +15.80
%BaSO₄ Recovered: 61.57 ± 11.19
Upgrading Factor: 1.327 ± 0.255
% Separation Efficiency: 15.12 ± 11.69

COMMENTS
EXPERIMENT NO. BDAT14

DATE: 28/10/81

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g BaSO₄ powder, 150 g silica

Initial Slurry Volume: 1.25 L

Collector: 0.20 g Sodium Oleate

Depressant: 0.34 g Sodium Meta Silicate

pH: 7.0 ± 0.1

R.P.H.: 900 ± 10

Pressures: Conditioning 101.3 kPa; Flotation 101.3 kPa

Initial BaSO₄ Grade: 25.0%

Volume of Make-up Water: 0.25 L

Times: Conditioning 30 min.; Dead Time - min.; Flotation 10 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 59.40 ± 0.57

Overall Mass Balance Error %: -0.80

Solids Wt./Slurry Volume: Feed 0.13 g/mL; Float 0.20 g/mL

Max. Dissolved Air Available for Precipitation: 0.0 mL/L of Solution

%BaSO₄ Grade: Float 29.42; Tails 10.05

%BaSO₄ Mass Balance Error: -14.17

%BaSO₄ Recovered: 76.64 ± 10.19

Upgrading Factor: 1.289 ± 0.159

% Separation Efficiency: 17.24 ± 9.62

COMMENTS

Excessive frothing during the conditioning period which is similar to regular flotation.
EXPERIMENT NO. BVT14

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g BaSO₄ powder, 50 g silica
Initial Slurry Volume: 1.25 L
Collector: 0.20 g. Sodium Oleate
Depressant: 0.34 g. Sodium Meta Silicate
pH: 7.0 ± 0.1
R.P.M.: 900 ± 10
Pressures: Conditioning 101.3 kPa; Flotation 11.9 ± 0.3 kPa
Initial BaSO₄ Grade: 25.0%
Volume of Make-up Water: 0.25 L
Times: Conditioning 30 min.; Dead Page 5 min.; Flotation 10 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 53.80 ± 1.13
Overall Mass Balance Error %: -1.60
Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.17 g./mL
Max. Dissolved Air Available for Precipitation: 150.8 mL/L of Solution
%BaSO₄ Grade: Float 32.33; Tails 11.79
%BaSO₄ Mass Balance Error: -9.48
%BaSO₄ Recovered: 73.56 ± 7.11
Upgrading Factor: 1.366 ± 0.103
% Separation Efficiency: 19.76 ± 5.98

COMMENTS
EXPERIMENT NO. BDAT15

DATE: 29/10/81

EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g BaSO₄ powder, 150 g silica

Initial Slurry Volume: 1.25 L

Collector: 0.13 g Sodium Oleate

Depressant: 0.20 g Sodium Meta Silicate

pH: 7.0 ± 0.1

R.P.M.: 900 ± 10

Pressures: Conditioning 101.3 kPa; Flotation 101.3 kPa

Initial BaSO₄ Grade: 25.0%

Volume of Make-up Water: 0.25 L

Times: Conditioning 30 min.; Dead Time - min.; Flotation 10 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 51.15

Overall Mass Balance Error %: 0.0

Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.19 g./mL

Max. Dissolved Air Available for Precipitation: 0.0 mL/L of Solution

%BaSO₄ Grade: Float 37.85; Tails 9.63

%BaSO₄ Mass Balance Error: -3.74

%BaSO₄ Recovered: 79.31 ± 2.65

Upgrading Factor: 1.551 ± 0.052

% Separation Efficiency: 28.16 ± 2.65

COMMENTS
EXPERIMENTAL CONDITIONS

Wt. of Feed Used: 50 g BaSO₄ powder, 150 g silica

Initial Slurry Volume: 1.25 L

Collector: 0.20 g. Sodium Oleate

Depressant: 0.34 g. Sodium Meta Silicate

pH: 7.0 ± 0.1

R.P.M.: 900 ± 10

Pressures: Conditioning 101.3 kPa; Flotation 11.2 ± 0.3 kPa

Initial BaSO₄ Grade: 25.0%

Volume of Make-up Water: 0.25 L

Times: Conditioning 30 min.; Dead Time 5 min.; Flotation 10 min.

EXPERIMENTAL RESULTS

Wt. % Floated: 51.15 ± 0.71

Overall Mass Balance Error %: -1.0

Solids Wt./Slurry Volume: Feed 0.13 g./mL; Float 0.18 g./mL

Max. Dissolved Air Available for Precipitation: 161.1 mL/L of Solution

∥BaSO₄∥ Grade: Float 33.99; Tails 13.75

∥BaSO₄∥ Mass Balance Error: -0.89

∥BaSO₄∥ Recovered: 74.95 ± 0.923

Upgrading Factor: 1.359 ± 0.001

% Separation Efficiency: 19.81 ± 0.22

COMMENTS
APPENDIX 4  STATISTICAL ANALYSIS
APPENDIX A4 Statistical Analysis

An outline of the statistical tests used to analyse the results from flotation experiments is provided in the following sections.

A4.a Estimate of Variance

An estimate of the variance for each parameter was obtained by pooling individual variance estimates from six sets of replicate experiments.

Individual variance estimates for each replicate set is given by:

\[ S^2 = \frac{1}{n-1} \sum_{i=1}^{n} (y_i - \bar{y})^2 \]  \hspace{1cm} A8

where \( n \) = number of replicates

\( y \) = observed parameter value, and

\( \bar{y} \) = arithmetic average of observed parameter values.

The pooled variance was found using:

\[ S_p^2 = \frac{\sum_{j=1}^{R} (n_j - 1) S_j^2}{\sum_{j=1}^{R} (n_j - 1)} \]  \hspace{1cm} A9

where \( R \) = number of replicate sets

\( S_j^2 \) = variance estimate of the \( j \)th replicate set

\( n_j \) = number of observations of \( j \)th replicate set
The number of degrees of freedom is given by:

\[ d.f. = \sum_{j=1}^{R} (n_j - 1) \]  

A10

In determining the pooled variance estimate the assumption is made that all variances are members of the same population.

Using equations A-8 to A-10 the individual and pooled variance estimates for each parameter were calculated as shown in Table A4-1. The estimate of the pooled variance is based on 6 degrees of freedom.

A4-b Hypothesis Tests

Three types of hypothesis tests were employed in the analysis of results. A randomized block test was made to investigate whether D.A.F. was more efficient than dispersed air flotation. Another statistical test was made to determine whether two experimental results were significantly different. A third hypothesis test was made to determine whether an experimentally observed value was significantly different from a hypothesized value. The procedures used along with a sample calculation are illustrated in the following sections.

A4.b.1 Randomized Block Test

This test was used to examine whether the overall observed difference between D.A.F. and dispersed air flotation experiment were significantly from zero.

The difference, \( d_1 \), is defined by
\[ d_i = y_{D.A.F.,i} - y_{D.A.1} \]

where \( y_{D.A.F.,i} \) = observed parameter using D.A.F.,

\( y_{D.A.1} \) = observed parameter using dispersed air flotation.

Then the average difference is

\[ \bar{d} = \frac{1}{n} \sum_{i=1}^{n} d_i \]

Variance estimates are given by:

\[ \text{Var}(d) = \frac{1}{n-1} \sum_{i=1}^{n} (d_i - \bar{d})^2 \]

and

\[ \text{Var}(\bar{d}) = \frac{1}{n} \text{Var}(d) \]

These expressions are used to test the following hypothesis

\[ H_0: \ E(\bar{d}) = 0 \]

\[ H_1: \ E(\bar{d}) \neq 0 \]

The test statistic, \( T \), used is given by:

\[ T = \frac{\bar{d}}{[\text{Var}(\bar{d})]^{1/2}} \]

on \( n-1 \) degrees of freedom.

Then at the 95\% confidence level if \( T > t_{0.025}(n-1) \) then there is statistical evidence to suggest that there is a difference between the two methods.
Example

Calculated values for the difference in the difference in the % Barite recovery are given in Table A4-2. From this table the following values were calculated.

$\bar{d} = 1.88 \quad \text{Var}(d) = 278.81$

$\text{Var}(\bar{d}) = 15.49 \quad n = 18$

Then the test statistic is

$$T = \frac{\bar{d}}{\sqrt{\text{Var}(\bar{d})}}$$

Therefore $T = 0.478$ on 17 degrees of freedom. From t-tables (25) at the 5% level of significance and 17 degrees of freedom $t_{table} = 2.110$

Since $T < 2.110$ then there is no reason to believe that there is a significant difference between the % Barite recovery using either D.A.F. or dispersed air flotation at the 5% level of significance.

A4.b.2 Hypothesis Test for Comparing Two Population Means

This test is used to determine if there is a difference between a parameter from two different experiments. The pooled standard deviation from replicate experiments is used to estimate the variance.

If $y_1$ = parameter from one experiment based on $n_1$ observations and $y_2$ = parameter from another experiment based on $n_2$ observations, then the hypothesis tested is

$$H_0: E(y_1) - E(y_2) = 0$$
\[ H_1: \ E(y_1) - E(y_2) \neq 0 \]

The test statistic, \( T \), is given by
\[
T = \frac{|y_1 - y_2|}{\sqrt{\frac{1}{n_1} + \frac{1}{n_2}}}^{1/2}
\]
on 6 degrees of freedom. Then at the 95\% level of confidence, if \( T > t_{0.025} \) (6) then there is statistical evidence to suggest there is a difference between the two methods.

**Example**

To test whether there is a significant difference in the % BaSO\(_4\) recovered as a result of using sodium meta silicate on pure BaSO\(_4\) powder floated by dispersed air the average results of BDAT 14&15 are compared to BDAT 12&13.

From Appendix A3
\[ \bar{y}_1 = 69.88 \text{ on 2 observations} \]
and
\[ \bar{y}_2 = 77.91 \text{ on 2 observations} \]
Also, the standard deviation, \( S_p \), is \( 3.673 \) on 6 degrees of freedom for % BaSO\(_4\) recovered.

Then the test statistic is
\[
T = \frac{|69.88 - 77.91|}{3.673 \sqrt{\frac{1}{2} + \frac{1}{2}^{1/2}}}
\]
or \( T = 2.186 \) on 6 degrees of freedom.

From \( t \)-tables (25) at the 95\% level of confidence and 6 degrees of freedom is
\( t_{\text{table}} = 2.447 \)

Then since \( T < 2.447 \) there is no statistical difference in using depressant under these experimental conditions at the 95% level of confidence.

A4.6.3 Hypothesis Test for Comparison of an Experimental Value to an Expected Value

This test is to determine whether the measured effects are significantly different than zero and also compare observed values to results obtained by Klassen. The pooled standard deviation is again used as an estimate of variance.

If \( y_1 \) is the experimentally observed value and \( \mu \) is the hypothesized value then the hypothesis tested is:

- \( H_0: \ E(y_1) = \mu \)
- \( H_1: \ E(y_1) \neq \mu \)

The test statistic, \( T \), is given by:

\[
T = \frac{|y_1 - \mu|}{Sp/\sqrt{n}} \quad \text{A17}
\]

on 6 degrees of freedom.

Then if at the 95% confidence level \( T > t_{0.025}(6) \) then there is statistical evidence to suggest that there is a statistically significant difference between the observed value and the hypothesized value.
Example

To test whether the upgrading factor reported by Klassen for dispersed air flotation (given in Table 5) is significantly different from the observed upgrading factor for similar experimental conditions the result of BDAT-7 is used.

Then the required values are:

\[ u = 1.55 \]
\[ y_1 = 1.381 \]
\[ S_p = 0.1222 \text{ on 6 degrees of freedom.} \]

Then the test statistic is

\[ T = \frac{|1.381 - 1.55|}{0.1222/\sqrt{1}} \]

or \[ T = 1.383 \text{ on 6 degrees of freedom.} \] From t-tables (25) at the 95% confidence level on 6 degrees of freedom \[ t_{\text{table}} = 2.447. \] Since \[ T < 2.447 \] there is evidence to suggest that there is no difference between the observed and reported values.

Results of hypothesis tests are given in Tables A4-2 to A4-11.

A4.c Factorial Design Analysis

Factorial design analysis was used to investigate the effects of various experimental variables (i.e. collector concentration) on observed parameter responses (i.e. upgrading factor). The measured effect of each variable was tested to determine whether it was significantly different than zero.

To simplify calculations the experimental variables, \( x \), are scaled such that extreme values are either -1 or 1, i.e.
Two different levels of factorial design were used to study the effects of 2 and 3 variables simultaneously.

A4.c.1 $2^2$ Factorial Design

A $2^2$ factorial design is used to fit the following model:

$$n = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{12} x_1 x_2$$

where $n$ is the expected value of the experimentally observed parameter (i.e., upgrading factor), $x$ is the normalized experimental variables and $\beta_1$ is the effect of variable $x_1$.

To perform this analysis, $4$ experiments are required which include all possible combinations of $x_1$ and $x_2$ at their extreme values.

Using a least-squares fit, the effects may be estimated by

$$\hat{\beta}_1 = \frac{\sum_{u=1}^{4} x_{1u} y_u}{\sum_{u=1}^{4} x_{1u}^2}$$

where $y_u$ is the observed value of $n_u$.

The variance of the effect estimate is given by:

$$\text{Var}(\hat{\beta}_1) = \frac{\text{Var}(y)}{N}$$

$$\frac{\sum_{u=1}^{4} x_{1u}^2}{\sum_{u=1}^{4} x_{1u}}$$

It should be noted that

$$\sum_{u=1}^{4} x_{1u}^2 = 4$$
for this design.

A4.c.2  2³ Factorial Design

A 2³ factorial design is used to fit the following model:

\[ n = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 + \beta_{123} x_1 x_2 x_3 \]  

where \( n \), \( \beta \), and \( x \) are as defined in Section A4.c.1. To perform this analysis 8 experiments are required which include all possible combinations of the extreme values of \( x_1 \), \( x_2 \) and \( x_3 \).

Using a least squares fit the effects may be estimated by:

\[ \beta_1 = \frac{1}{8} \sum_{u=1}^{8} x_{1u} y_u \]  

The variance of the effect estimate is given by:

\[ \text{Var}(\beta_1) = \frac{\text{Var}(y)}{8} \sum_{u=1}^{8} \frac{x_{1u}^2}{x_{1u}} \]  

for this design.

A4.c.3 Determination of Significant Effects

The estimated effects, \( \hat{\beta}_1 \)'s, may be tested to determine whether they are significantly different from zero using the method developed in Appendix A4.b.3.

For this special case the hypothesized value, \( \mu \), is zero. The
test static becomes:

\[ T = \frac{|\hat{B}|}{(\text{Var}(\hat{B})^{1/2})} \]  \hspace{1cm} (A25)

In order for \( \hat{B} \) to be significant at the 95% confidence level on 6 degrees of freedom

\[ T > 2.447 \]

Substituting this inequality into equation A-24 and re-arranging gives

\[ |\hat{B}| > 2.447 (\text{Var}(\hat{B}))^{1/2} \]  \hspace{1cm} (A26)

Where

\[ \text{Var}(\hat{B}) = \frac{\text{Var}(y)}{4} \]

for \( 2^2 \) factorial design and

\[ \text{Var}(\hat{B}) = \frac{\text{Var}(y)}{8} \]

for the \( 2^3 \) factorial design. Estimates of \( \text{Var}(y) \) for different parameters is given in Table A4-1 on 6 degrees of freedom.

Results of \( 2^3 \) factorial analysis are given in Tables A4-12 to A4-14 and for \( 2^2 \) factorial analysis in Table A4-15.

Based on these results the following effects were found to be significant.

a) Weight % Floated: \[ |\hat{B}| > 4.341 \] for \( 2^2 \) factorial
   \[ |\hat{B}| > 6.140 \] for \( 2^2 \) factorial

Significant effects are:

1) collector concentration
2) depressant
3) purity of BaSO₄
4) pH
5) D.A.F. (from A₄-14 only)
6) RPM/D.A.F.

c) % BaSO₄ Recovered: |\hat{b}₄| > 3.178 for 2³ factorial
   |\hat{b}₅| > 4.494 for 2² factorial

   Significant effects are:
   1) collector concentration
   2) depressant
   3) purity of BaSO₄
   4) pH/D.A.F.
   5) pH
   6) RPM/D.A.F.

c) Upgrading Factor: |\hat{b}₃| > 0.106 for 2³ factorial
   |\hat{b}₄| > 0.150 for 2² factorial

   Significant effects are:
   1) collector concentration
   2) pH/D.A.F.
   3) RPM/D.A.F.

   d) % Separation Efficiency: |\hat{b}₅| > 3.66 for 2³ factorial
   |\hat{b}₆| > 5.178 for 2² factorial

   Significant effects are:
1) collector concentration
2) purity of BaSO₄
3) pH
4) pH/D.A.F.
5) RPM/DAF

It should be noted that the RPM/DAF interaction effect corresponds to the effect of frothing during conditioning.
### TABLE A4-1 Individual and Pooled Variance Estimates for Each Flotation Parameter

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Variances</th>
<th>Weight Floated W/W %</th>
<th>Recovery BaSO_4 W/W %</th>
<th>Upgrading Factor W/W</th>
<th>Separation Efficiency W/W %</th>
</tr>
</thead>
<tbody>
<tr>
<td>BDAT 4&amp;6</td>
<td></td>
<td>2.205</td>
<td>3.838</td>
<td>0.02510</td>
<td>11.859</td>
</tr>
<tr>
<td>BVT 4&amp;6</td>
<td></td>
<td>0.061</td>
<td>0.042</td>
<td>0.00001</td>
<td>0.003</td>
</tr>
<tr>
<td>BDAT 12&amp;13</td>
<td></td>
<td>85.156</td>
<td>13.675</td>
<td>0.03000</td>
<td>30.576</td>
</tr>
<tr>
<td>BVT 12&amp;13</td>
<td></td>
<td>28.730</td>
<td>58.860</td>
<td>0.00010</td>
<td>5.399</td>
</tr>
<tr>
<td>BDAT 14&amp;15</td>
<td></td>
<td>34.036</td>
<td>3.565</td>
<td>0.03430</td>
<td>59.623</td>
</tr>
<tr>
<td>BVT 14&amp;15</td>
<td></td>
<td>0.912</td>
<td>0.966</td>
<td>0.00002</td>
<td>0.001</td>
</tr>
<tr>
<td>$s^2_p$</td>
<td></td>
<td>25.183</td>
<td>13.491</td>
<td>0.0149</td>
<td>17.907</td>
</tr>
<tr>
<td>$s^2_p$</td>
<td></td>
<td>5.018</td>
<td>3.673</td>
<td>0.1222</td>
<td>4.232</td>
</tr>
</tbody>
</table>
Table A4-2 Observed Differences between D.A.F. and Dispersed Air Flotation, \( d \)

<table>
<thead>
<tr>
<th>Floatation Parameter Differences, ( d )</th>
<th>Weight Floated W/W %</th>
<th>Recovery BaSO(_4) W/W %</th>
<th>Upgrading Factor W/W</th>
<th>Separation Efficiency W/W %</th>
</tr>
</thead>
<tbody>
<tr>
<td>BVT1/BDAT1</td>
<td>-8.90</td>
<td>0.49</td>
<td>0.227</td>
<td>9.32</td>
</tr>
<tr>
<td>BPVT1/BDAT1</td>
<td>-5.15</td>
<td>0.17</td>
<td>0.112</td>
<td>5.32</td>
</tr>
<tr>
<td>BVT2/BDAT2</td>
<td>-0.70</td>
<td>0.55</td>
<td>0.023</td>
<td>1.21</td>
</tr>
<tr>
<td>BPVT2/BDAT2</td>
<td>2.88</td>
<td>3.57</td>
<td>0.000</td>
<td>0.65</td>
</tr>
<tr>
<td>BVT3/BDAT3</td>
<td>-2.92</td>
<td>-0.64</td>
<td>0.071</td>
<td>2.29</td>
</tr>
<tr>
<td>BPVT3/BDAT3</td>
<td>2.33</td>
<td>0.11</td>
<td>-0.058</td>
<td>-2.21</td>
</tr>
<tr>
<td>BPVT4/BDAT4</td>
<td>14.98</td>
<td>14.02</td>
<td>-0.170</td>
<td>-0.86</td>
</tr>
<tr>
<td>BVT5/BDAT5</td>
<td>-9.13</td>
<td>-8.62</td>
<td>0.190</td>
<td>5.50</td>
</tr>
<tr>
<td>BVT6/BDAT6</td>
<td>12.43</td>
<td>16.50</td>
<td>0.059</td>
<td>4.08</td>
</tr>
<tr>
<td>BVT7/BDAT7</td>
<td>-3.10</td>
<td>-9.76</td>
<td>-0.009</td>
<td>-6.65</td>
</tr>
<tr>
<td>BVT8/BDAT8</td>
<td>11.80</td>
<td>-2.07</td>
<td>-0.407</td>
<td>-13.87</td>
</tr>
<tr>
<td>BVT9/BDAT9</td>
<td>10.60</td>
<td>19.86</td>
<td>0.208</td>
<td>9.26</td>
</tr>
<tr>
<td>BVT10/BDAT10</td>
<td>-5.82</td>
<td>-38.34</td>
<td>-0.701</td>
<td>-32.51</td>
</tr>
<tr>
<td>BVT11/BDAT11</td>
<td>19.55</td>
<td>46.53</td>
<td>0.342</td>
<td>26.98</td>
</tr>
<tr>
<td>BVT12/BDAT12</td>
<td>8.88</td>
<td>5.16</td>
<td>-0.096</td>
<td>-3.71</td>
</tr>
<tr>
<td>BVT13/BDAT13</td>
<td>-11.75</td>
<td>-10.98</td>
<td>-0.149</td>
<td>0.83</td>
</tr>
<tr>
<td>BVT14/BDAT14</td>
<td>-5.60</td>
<td>-3.23</td>
<td>0.077</td>
<td>2.52</td>
</tr>
<tr>
<td>BVT15/BDAT15</td>
<td>4.00</td>
<td>-4.56</td>
<td>-0.192</td>
<td>-8.35</td>
</tr>
</tbody>
</table>

\( d = y_{D.A.F.} - y_{Dispersed Air} \)
Table A4-3: Calculated T-Values for the Effect of Dissolved Air Concentration on Flotation Parameters

<table>
<thead>
<tr>
<th>Experiments</th>
<th>Flotation Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weight Floated W/W %</td>
</tr>
<tr>
<td>BPVT1/BVT1</td>
<td>0.52</td>
</tr>
<tr>
<td>BPVT2/BVT2</td>
<td>0.50</td>
</tr>
<tr>
<td>BPVT3/BVT3</td>
<td>0.74</td>
</tr>
<tr>
<td>BPVT4/BVT6</td>
<td>0.05</td>
</tr>
</tbody>
</table>

1) \( t = 2.447 \) at 95% confidence level on 6 d.f., Ref. (25).
Table A4-4: Calculated T Values for the Difference between D.A.F. and Dispersed Air Flotation

<table>
<thead>
<tr>
<th>Experiments</th>
<th>Flotation Parameters</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weight Floated W/W %</td>
<td>Recovery BaSO₄ W/W %</td>
<td>Upgrading Factor W/W</td>
<td>Separation Efficiency W/W %</td>
</tr>
<tr>
<td>BDAT1/BVT1</td>
<td>1.25</td>
<td>0.09</td>
<td>1.31</td>
<td>1.56</td>
</tr>
<tr>
<td>BDAT1/BPV1</td>
<td>0.73</td>
<td>0.03</td>
<td>0.71</td>
<td>0.89</td>
</tr>
<tr>
<td>BDAT2/BVT2</td>
<td>0.10</td>
<td>0.11</td>
<td>0.13</td>
<td>0.20</td>
</tr>
<tr>
<td>BDAT2/BPV2</td>
<td>0.41</td>
<td>0.69</td>
<td>0.00</td>
<td>0.11</td>
</tr>
<tr>
<td>BDAT3/BVT3</td>
<td>0.41</td>
<td>0.12</td>
<td>0.41</td>
<td>0.38</td>
</tr>
<tr>
<td>BDAT3/BPV3</td>
<td>0.33</td>
<td>0.02</td>
<td>0.34</td>
<td>0.37</td>
</tr>
<tr>
<td>BDAT4/BVT4</td>
<td>2.10</td>
<td>2.70</td>
<td>0.98</td>
<td>0.14</td>
</tr>
<tr>
<td>BDAT5/BVT5</td>
<td>1.29</td>
<td>0.70</td>
<td>1.10</td>
<td>0.92</td>
</tr>
<tr>
<td>BDAT6/BUT6</td>
<td>1.75</td>
<td>3.18</td>
<td>0.34</td>
<td>0.68</td>
</tr>
<tr>
<td>BDAT7/BVT7</td>
<td>0.44</td>
<td>1.88</td>
<td>0.05</td>
<td>1.11</td>
</tr>
<tr>
<td>BDAT8/BVT8</td>
<td>1.66</td>
<td>0.40</td>
<td>2.36</td>
<td>2.32</td>
</tr>
<tr>
<td>BDAT9/BVT9</td>
<td>1.49</td>
<td>3.82</td>
<td>1.20</td>
<td>1.55</td>
</tr>
<tr>
<td>BDAT10/BVT10</td>
<td>0.82</td>
<td>7.38</td>
<td>4.06</td>
<td>5.43</td>
</tr>
<tr>
<td>BDAT11/BVT11</td>
<td>2.75</td>
<td>8.96</td>
<td>1.98</td>
<td>4.51</td>
</tr>
<tr>
<td>BDAT12/BVT12</td>
<td>1.25</td>
<td>0.99</td>
<td>0.56</td>
<td>0.62</td>
</tr>
<tr>
<td>BDAT13/BVT13</td>
<td>1.66</td>
<td>2.11</td>
<td>0.86</td>
<td>0.14</td>
</tr>
<tr>
<td>BDAT14/BVT14</td>
<td>0.79</td>
<td>0.62</td>
<td>0.45</td>
<td>0.42</td>
</tr>
<tr>
<td>BDAT15/BVT15</td>
<td>0.56</td>
<td>0.88</td>
<td>1.11</td>
<td>1.40</td>
</tr>
</tbody>
</table>

Celestite

<p>| | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>BDAT8A/BVT8A</td>
<td>1.66</td>
<td>1.84</td>
<td>3.25</td>
</tr>
<tr>
<td>BDAT9A/BVT9A</td>
<td>1.49</td>
<td>3.09</td>
<td>0.02</td>
</tr>
</tbody>
</table>

1) $t = 2.447$ at 95% confidence level on 6 d.f., Ref. (25).
Table A4-5: Calculated T Values for the Effect of Collector Concentration on Flotation Parameters

<table>
<thead>
<tr>
<th>Experiments</th>
<th>Weight Floated W/W %</th>
<th>Recovery BaSO₄ W/W %</th>
<th>Upgrading Factor W/W</th>
<th>Separation Efficiency W/W %</th>
</tr>
</thead>
<tbody>
<tr>
<td>BDAT1/2</td>
<td>0.18</td>
<td>2.38</td>
<td>0.97</td>
<td>1.85</td>
</tr>
<tr>
<td>BDAT1/3</td>
<td>2.76</td>
<td>6.47</td>
<td>0.84</td>
<td>2.34</td>
</tr>
<tr>
<td>BDAT2/3</td>
<td>2.59</td>
<td>4.09</td>
<td>0.13</td>
<td>0.49</td>
</tr>
<tr>
<td>BVT1/2</td>
<td>0.97</td>
<td>2.37</td>
<td>2.15</td>
<td>3.20</td>
</tr>
<tr>
<td>BVT1/3</td>
<td>1.93</td>
<td>6.69</td>
<td>1.74</td>
<td>3.51</td>
</tr>
<tr>
<td>BVT2/3</td>
<td>2.90</td>
<td>4.32</td>
<td>0.41</td>
<td>0.31</td>
</tr>
<tr>
<td>BPVT1/3</td>
<td>1.71</td>
<td>6.48</td>
<td>1.88</td>
<td>3.60</td>
</tr>
<tr>
<td>BPVT2/3</td>
<td>2.66</td>
<td>4.76</td>
<td>0.21</td>
<td>0.97</td>
</tr>
<tr>
<td>BPVT1/2</td>
<td>0.96</td>
<td>1.72</td>
<td>1.67</td>
<td>2.63</td>
</tr>
<tr>
<td>BDAT4&amp;6/5</td>
<td>5.72</td>
<td>12.31</td>
<td>0.75</td>
<td>3.90</td>
</tr>
<tr>
<td>BDAT4&amp;6/7</td>
<td>1.82</td>
<td>14.33</td>
<td>9.51</td>
<td>10.27</td>
</tr>
<tr>
<td>BVT4&amp;6/5</td>
<td>2.02</td>
<td>8.11</td>
<td>2.87</td>
<td>4.65</td>
</tr>
<tr>
<td>BVT4&amp;6/7</td>
<td>0.90</td>
<td>8.77</td>
<td>9.82</td>
<td>8.68</td>
</tr>
</tbody>
</table>

1) \( t = 2.447 \) at 95% confidence level on 6 d.f., Ref. (25).
**Table A4-6: Calculated T Values for the Effect of Depressant on Flotation Parameters**

<table>
<thead>
<tr>
<th>Experiments</th>
<th>Flotation Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weight Floated W/W %</td>
</tr>
<tr>
<td>BDAT 12&amp;13/14&amp;15</td>
<td>0.72</td>
</tr>
<tr>
<td>BVT 12&amp;13/14&amp;15</td>
<td>0.84</td>
</tr>
<tr>
<td>BDAT 4&amp;6/3</td>
<td>3.41</td>
</tr>
<tr>
<td>BVT 4&amp;6/3</td>
<td>0.71</td>
</tr>
<tr>
<td>BPVT 4&amp;6/3</td>
<td>1.57</td>
</tr>
<tr>
<td>BDAT 1/5</td>
<td>0.76</td>
</tr>
<tr>
<td>BVT 1/5</td>
<td>0.80</td>
</tr>
<tr>
<td>BPVT 1/BVT 5</td>
<td>1.32</td>
</tr>
<tr>
<td>BDAT 1/7</td>
<td>4.13</td>
</tr>
<tr>
<td>BVT 1/7</td>
<td>3.33</td>
</tr>
<tr>
<td>BPVT 1/BVT 7</td>
<td>3.84</td>
</tr>
</tbody>
</table>

1) t = 2.447 at 95% confidence level on 6 d.f., Ref. (25).
Table A4-7: Calculated T Values for Effects of Purity of BaSO₄ on Flotation Parameters

<table>
<thead>
<tr>
<th>Experiments</th>
<th>Weight Floated W/W %</th>
<th>Recovery BaSO₄ W/W %</th>
<th>Upgrading Factor W/W</th>
<th>Separation Efficiency W/W %</th>
</tr>
</thead>
<tbody>
<tr>
<td>BDAT4&amp;6/12&amp;13</td>
<td>5.51</td>
<td>10.81</td>
<td>0.89</td>
<td>2.86</td>
</tr>
<tr>
<td>BVT4&amp;6/12&amp;13</td>
<td>2.50</td>
<td>5.87</td>
<td>1.06</td>
<td>2.14</td>
</tr>
<tr>
<td>BDAT3/14&amp;15</td>
<td>1.67</td>
<td>4.95</td>
<td>1.23</td>
<td>2.33</td>
</tr>
<tr>
<td>BVT3/14&amp;15</td>
<td>2.02</td>
<td>4.23</td>
<td>0.37</td>
<td>1.33</td>
</tr>
<tr>
<td>BPVT3/14&amp;15</td>
<td>1.16</td>
<td>4.06</td>
<td>1.24</td>
<td>2.19</td>
</tr>
</tbody>
</table>

1) $t = 2.447$ at 95% confidence level on 6 d.f., Ref. (25).
Table A4-8: Calculated T Values for the Difference in Flotation Parameters for the Flotation of Celestite and Barite from Silica

<table>
<thead>
<tr>
<th>Experiments</th>
<th>Recovery BaSO₄ W/W %</th>
<th>Upgrading Factor W/W</th>
<th>Separation Efficiency W/W %</th>
</tr>
</thead>
<tbody>
<tr>
<td>BDAT8/8A</td>
<td>1.14</td>
<td>0.73</td>
<td>0.99</td>
</tr>
<tr>
<td>BVT8/8A</td>
<td>0.30</td>
<td>0.16</td>
<td>0.26</td>
</tr>
<tr>
<td>BDAT9/9A</td>
<td>0.26</td>
<td>0.60</td>
<td>0.23</td>
</tr>
<tr>
<td>BVT9/9A</td>
<td>0.48</td>
<td>0.62</td>
<td>0.35</td>
</tr>
</tbody>
</table>

1) \( t = 2.447 \) at 95% confidence level on 6 d.f., Ref. (25).
Table A4.91 Calculated T-Values for the Effect of pH on Flotation Parameters

<table>
<thead>
<tr>
<th>Experiments</th>
<th>Flotation Parameters</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weight Floated W/W %</td>
<td>Recovery BaSO₄ W/W %</td>
<td>Upgrading Factor W/W</td>
<td>Separation Efficiency W/W %</td>
</tr>
<tr>
<td>BDAT4&amp;6/9</td>
<td>1.81</td>
<td>2.64</td>
<td>1.02</td>
<td>0.15</td>
</tr>
<tr>
<td>BVT4&amp;6/9</td>
<td>2.31</td>
<td>1.62</td>
<td>2.78</td>
<td>1.33</td>
</tr>
<tr>
<td>BDAT4&amp;6/8</td>
<td>3.65</td>
<td>12.20</td>
<td>3.82</td>
<td>6.26</td>
</tr>
<tr>
<td>BVT4&amp;6/8</td>
<td>3.35</td>
<td>8.35</td>
<td>1.46</td>
<td>3.27</td>
</tr>
<tr>
<td>BDAT9/8</td>
<td>4.73</td>
<td>12.85</td>
<td>2.79</td>
<td>5.55</td>
</tr>
<tr>
<td>BVT9/8</td>
<td>4.90</td>
<td>8.63</td>
<td>1.23</td>
<td>1.68</td>
</tr>
</tbody>
</table>

1) $t = 2.447$ at 95% confidence level on 6 d.f., Ref. (25).
Table A4-10: Calculated T Values for the Effect of Aeration (RPM) on Flotation Parameters

<table>
<thead>
<tr>
<th>Experiments</th>
<th>Weight Floated W/W %</th>
<th>Recovery BaSO₄ W/W %</th>
<th>Upgrading Factor W/W</th>
<th>Separation Efficiency W/W %</th>
</tr>
</thead>
<tbody>
<tr>
<td>BDA14&amp;6/10</td>
<td>3.16</td>
<td>13.10</td>
<td>5.27</td>
<td>7.61</td>
</tr>
<tr>
<td>BVT4&amp;6/10</td>
<td>0.004</td>
<td>1.18</td>
<td>0.95</td>
<td>1.03</td>
</tr>
<tr>
<td>BDA14&amp;6/11</td>
<td>0.11</td>
<td>2.13</td>
<td>2.31</td>
<td>1.71</td>
</tr>
<tr>
<td>BVT4&amp;6/11</td>
<td>1.07</td>
<td>9.08</td>
<td>4.96</td>
<td>6.61</td>
</tr>
<tr>
<td>BDA10/11</td>
<td>2.64</td>
<td>9.50</td>
<td>2.56</td>
<td>5.11</td>
</tr>
<tr>
<td>BVT10/11</td>
<td>0.93</td>
<td>6.84</td>
<td>3.48</td>
<td>4.83</td>
</tr>
<tr>
<td>BDA10/BVT11</td>
<td>0.11</td>
<td>0.54</td>
<td>0.58</td>
<td>0.60</td>
</tr>
<tr>
<td>BVT10/BDA11</td>
<td>1.28</td>
<td>2.12</td>
<td>1.50</td>
<td>0.32</td>
</tr>
</tbody>
</table>

1) $t = 2.447$ at 95% confidence level on 6 d.f., Ref. (25).

2) Frothing during conditioning observed during BDA10 and BVT-11.
Table A4-11: Calculated T Values for the Differences between Klassen's\textsuperscript{19} Results and Current Experimental Observations for the Flotation of Barite Slimes

<table>
<thead>
<tr>
<th>Experiments</th>
<th>Flotation Parameters</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weight Floated W/W %</td>
<td>Recovery BaSO\textsubscript{4} W/W %</td>
<td>Upgrading Factor W/W</td>
<td>Separation Efficiency W/W %</td>
</tr>
<tr>
<td>BDAT1/Klassen\textsuperscript{19}</td>
<td>7.17</td>
<td>12.25</td>
<td>1.38</td>
<td>2.09</td>
</tr>
<tr>
<td>BVT-1/Klassen</td>
<td>3.17</td>
<td>7.24</td>
<td>0.25</td>
<td>2.54</td>
</tr>
<tr>
<td>BPVT1/Klassen</td>
<td>3.83</td>
<td>7.16</td>
<td>0.63</td>
<td>1.59</td>
</tr>
</tbody>
</table>

1) \( t = 2.447 \) at 95\% confidence level on 6 d.f., Ref. (25).

2) Results based on comparative study given in Table 5.
Table A4-12: Effects of Collector Concentration, Depressant and D.A.F. on Y*

<table>
<thead>
<tr>
<th>Correlating Parameter for the Independent Variable, X</th>
<th>Weight Floated W/W %</th>
<th>Recovery BaSO₄ W/W %</th>
<th>Upgrading Factor W/W</th>
<th>Separation Efficiency W/W %</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \hat{\beta}_0 )</td>
<td>47.88</td>
<td>66.60</td>
<td>1.370</td>
<td>18.73</td>
</tr>
<tr>
<td>( \hat{\beta}_1 )</td>
<td>10.01</td>
<td>19.96</td>
<td>0.134</td>
<td>9.95</td>
</tr>
<tr>
<td>( \hat{\beta}_2 )</td>
<td>5.11</td>
<td>5.85</td>
<td>-0.016</td>
<td>0.74</td>
</tr>
<tr>
<td>( \hat{\beta}_3 )</td>
<td>-0.34</td>
<td>1.46</td>
<td>0.040</td>
<td>1.81</td>
</tr>
<tr>
<td>( \hat{\beta}_{12} )</td>
<td>-1.88</td>
<td>-3.01</td>
<td>-0.020</td>
<td>-1.13</td>
</tr>
<tr>
<td>( \hat{\beta}_{13} )</td>
<td>-3.69</td>
<td>-2.29</td>
<td>0.052</td>
<td>1.40</td>
</tr>
<tr>
<td>( \hat{\beta}_{23} )</td>
<td>-1.48</td>
<td>-1.45</td>
<td>0.006</td>
<td>0.03</td>
</tr>
<tr>
<td>( \hat{\beta}_{123} )</td>
<td>2.01</td>
<td>2.43</td>
<td>-0.010</td>
<td>0.42</td>
</tr>
</tbody>
</table>

\[*Y = \hat{\beta}_0 + \hat{\beta}_1 X_1 + \hat{\beta}_2 X_2 + \hat{\beta}_3 X_3 + \hat{\beta}_{12} X_1 X_2 + \hat{\beta}_{13} X_1 X_3 + \hat{\beta}_{23} X_2 X_3 + \hat{\beta}_{123} X_1 X_2 X_3*

Code: \( X_1 \) = collector concentration
\[= -1 \oplus 1.0 \text{ kg sodium oleate/tonne of ore} \]
\[= 1 \oplus 5.0 \text{ kg sodium oleate/tonne of ore} \]

\( X_2 \) = Depressant Concentration
\[= -1 \oplus 0.0 \text{ kg sodium silicate/tonne of ore} \]
\[= 1 \oplus 1.7 \text{ kg sodium silicate/tonne of ore} \]

\( X_3 \) = D.A.F.
\[= -1 \text{ for Dispersed Air Flotation} \]
\[= 1 \text{ for D.A.F.} \]
Table A4-13: Effects of BaSO₄ Purity, Depressant and D.A.F. on Flotation Parameters, Y

<table>
<thead>
<tr>
<th>Correlating Parameter for the Independent Variable, X</th>
<th>Weight Floated W/W %</th>
<th>Recovery BaSO₄ W/W %</th>
<th>Upgrading Factor W/W</th>
<th>Separation Efficiency W/W %</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \hat{\beta}_0 )</td>
<td>45.39</td>
<td>59.42</td>
<td>1.303</td>
<td>14.07</td>
</tr>
<tr>
<td>( \hat{\beta}_1 )</td>
<td>7.53</td>
<td>12.78</td>
<td>0.068</td>
<td>5.29</td>
</tr>
<tr>
<td>( \hat{\beta}_2 )</td>
<td>4.47</td>
<td>6.31</td>
<td>0.012</td>
<td>1.88</td>
</tr>
<tr>
<td>( \hat{\beta}_3 )</td>
<td>1.39</td>
<td>1.02</td>
<td>-0.017</td>
<td>-0.34</td>
</tr>
<tr>
<td>( \hat{\beta}_{12} )</td>
<td>-2.51</td>
<td>-2.54</td>
<td>0.008</td>
<td>0.01</td>
</tr>
<tr>
<td>( \hat{\beta}_{13} )</td>
<td>-1.95</td>
<td>-2.73</td>
<td>-0.005</td>
<td>-0.75</td>
</tr>
<tr>
<td>( \hat{\beta}_{23} )</td>
<td>-1.66</td>
<td>-2.06</td>
<td>0.005</td>
<td>-0.38</td>
</tr>
<tr>
<td>( \hat{\beta}_{123} )</td>
<td>1.82</td>
<td>1.82</td>
<td>-0.011</td>
<td>0.01</td>
</tr>
</tbody>
</table>

\[ Y = \hat{\beta}_0 + \hat{\beta}_1 X_1 + \hat{\beta}_2 X_2 + \hat{\beta}_3 X_3 + \hat{\beta}_{12} X_1 X_2 + \hat{\beta}_{13} X_1 X_3 + \hat{\beta}_{23} X_2 X_3 + \hat{\beta}_{123} X_1 X_2 X_3 \]

Code:
- \( X_1 \) = BaSO₄ Purity
  - \( = -1 \) for barite ore (with celestite)
  - \( = 1 \) for pure BaSO₄ powder
- \( X_2 \) = depressant
  - \( = -1 \) @ 0 kg sodium silicate/tonne of ore
  - \( = 1 \) @ 1.7 kg sodium silicate/tonne of ore
- \( X_3 \) = D.A.F.
  - \( = -1 \) for dispersed air flotation
  - \( = 1 \) for D.A.F.
Table A4-14: Effects of Celestite Flotation, pH and D.A.F. on Flotation Parameters, Y

<table>
<thead>
<tr>
<th>Correlating Parameter for the Independent Variable, X</th>
<th>Weight Floated W/W %</th>
<th>Recovery BaSO₄ W/W %</th>
<th>Upgrading Factor W/W</th>
<th>Separation Efficiency W/W %</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \beta_0 )</td>
<td>35.31</td>
<td>56.51</td>
<td>1.583</td>
<td>21.21</td>
</tr>
<tr>
<td>( \beta_1 )</td>
<td>0.00</td>
<td>-0.40</td>
<td>-0.012</td>
<td>-0.40</td>
</tr>
<tr>
<td>( \beta_2 )</td>
<td>17.08</td>
<td>28.59</td>
<td>0.068</td>
<td>11.51</td>
</tr>
<tr>
<td>( \beta_3 )</td>
<td>5.60</td>
<td>3.03</td>
<td>-0.095</td>
<td>-2.57</td>
</tr>
<tr>
<td>( \beta_{12} )</td>
<td>0.00</td>
<td>-0.68</td>
<td>-0.013</td>
<td>-0.69</td>
</tr>
<tr>
<td>( \beta_{13} )</td>
<td>0.00</td>
<td>1.41</td>
<td>0.046</td>
<td>1.41</td>
</tr>
<tr>
<td>( \beta_{23} )</td>
<td>0.30</td>
<td>-5.94</td>
<td>-0.147</td>
<td>-6.24</td>
</tr>
<tr>
<td>( \beta_{123} )</td>
<td>0.00</td>
<td>0.45</td>
<td>-0.007</td>
<td>0.46</td>
</tr>
</tbody>
</table>

\( Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{123} X_1 X_2 X_3 \)

**Code:**
- \( X_1 \) = Celestite/Barite Flotation
  - \(-1\) for celestite flotation
  - \(1\) for barite flotation
- \( X_2 \) = pH
  - \(-1\) if \( pH = 4.0 \)
  - \(1\) if \( pH = 10.0 \)
- \( X_3 \) = D.A.F.
  - \(-1\) for dispersed air flotation
  - \(1\) for D.A.F.
Table A4-15: Effects of Aeration (RPM) and D.A.F. on Flotation Parameters, Y

<table>
<thead>
<tr>
<th>Correlating Parameter for the Independent Variable, X</th>
<th>Dependent Variable, Y</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weight Floated W/W %</td>
</tr>
<tr>
<td>( \hat{\beta}_0 )</td>
<td>37.56</td>
</tr>
<tr>
<td>( \hat{\beta}_1 )</td>
<td>-3.03</td>
</tr>
<tr>
<td>( \hat{\beta}_2 )</td>
<td>3.43</td>
</tr>
<tr>
<td>( \hat{\beta}_{12} )</td>
<td>6.34</td>
</tr>
</tbody>
</table>

\[ Y = \hat{\beta}_0 + \hat{\beta}_1 X_1 + \hat{\beta}_2 X_2 + \hat{\beta}_{12} X_1 X_2 \]

Code:  
- \( X_1 = \) aeration  
  - 1 @ 800 RPM  
  - 1 @ 1000 RPM  

- \( X_2 = \) D.A.F.  
  - -1 for dispersed air flotation  
  - 1 for D.A.F.

1) \( \hat{\beta}_{12} \) (RPM/D.A.F.) interaction effect corresponds to frothing during conditioning.
APPENDIX 5  SAMPLE CALCULATION OF FLOTATION RESULTS
APPENDIX A5  Sample Calculation of Flotation Results

For each flotation experiment the following measurements are made:

i) mass of float and tail products
ii) % composition of float and tail products
iii) volume of tails
iv) conditioning and flotation pressures.

These measurements, along with initial weight of solids, slurry composition and slurry volume are used to determine parameters which describe the flotation experiment. These parameters include:

a) weight % floated
b) initial and floated solids weight to slurry volume ratio
c) maximum volume of dissolved air available for precipitation
d) % BaSO_4 recovered in float
e) upgrading factor
f) separation efficiency

The procedure used in determining the above descriptive parameters is illustrated below for experiment #BPVT-3.

For BPVT-3 initial feed slurry properties were:

initial weight of solids, WI = 200 g
initial BaSO_4 grade, GI = 0.0956
initial slurry volume, VI = 1.50 L (including make-up water)

As a result of experimentation the following measurements were
made:

weight of float, \(WF = 94.0\) g

weight of tails, \(WT = 104.7\) g

\(\text{BaSO}_4\) grade of float, \(GF = 0.113\)

\(\text{BaSO}_4\) grade of tails, \(GT = 0.0805\)

volume of tails, \(VT = 1.1\) L

conditioning pressure, \(P_i = 198.3\) kPa

flotation pressure, \(P_f = 10.6\) kPa

A5.a Determination of Maximum Volume of Dissolved Air Available for Precipitation

From the equation derived in Appendix A1 the maximum volume of dissolved air capable of precipitating was found to be:

\[
WV = 20.19 \left( \frac{P_i - P_f}{P_f} \right) \text{ mL of air} \quad \text{L of water}
\]

Substituting for \(P_i = 198.1\) kPa and \(P_f = 10.6\) kPa gives

\[
WV = 20.19 \left( \frac{198.1 - 10.6}{10.6} \right) \text{ mL of air} \quad \text{L of water}
\]

\[
= 357.1 \quad \text{mL of air} \quad \text{L of water}
\]

Thus \(357.1\) mL of air per L of water was capable of precipitating from solution in this experiment.

A5.b Determination of Solid Weight to Slurry Volume Ratio

The initial weight to volume ratio of the feed is given by
\[
\text{initial wt./vol.} = \frac{\text{WI}}{\text{VI}}
\]

\[
\begin{align*}
&= \frac{200 \text{ g}}{1,500 \text{ mL}} \\
&= 0.13 \text{ g/mL}
\end{align*}
\]

The weight of the float was found to be 94.0 g and the volume of the float was estimated by

\[
\text{Vol. of float} = \text{VI} - \text{VT}
\]

\[
= 1,500 \text{ mL} - 1,100 \text{ mL}
\]

\[
= 400 \text{ mL}
\]

Then the weight to volume ratio of the float is given by

\[
\text{floated wt./vol.} = \frac{94.0 \text{ g}}{400 \text{ mL}}
\]

\[
= 0.24 \text{ g/mL}
\]

Thus the final floated weight to volume ratio is 0.24 g/mL. It should be noted that this is only a rough estimate of the true value since volume measurements were subject to error. This parameter was determined only to approximate how much water was entrained in the froth in comparison to other experiments.

A5.c Determination of Weight % Floated

The weight % floated may be calculated based on the observed weight of the float or the tails.

Based on the weight of the tails:

\[
\text{Wt.} \% \text{ floated} = \frac{\text{WF}}{\text{WI}} \times 100\%
\]
\[ = \frac{94.0 \text{ g}}{200 \text{ g}} \times 100\% \]
\[ = 47.00\% \]

Thus, the weight \% floated based on the float is 47.00\%.

Based on the weight of the tails the weight \% floated is given by:

\[ \text{Wt.}\% \text{ floated} = \frac{(\text{WI} - \text{WT})}{\text{WI}} \times 100\% \]
\[ = \frac{(200 \text{ g} - 104.7 \text{ g})}{200 \text{ g}} \times 100\% \]
\[ = 47.65\% \]

Thus the weight \% floated based on the tails is 47.65\%.

The average weight \% floated is 47.33 \pm 0.460\%. The overall mass balance error may be determined from

\[ \text{overall mass balance error} = \frac{(\text{WF} + \text{WT} - \text{WI})}{\text{WI}} \times 100\% \]
\[ = \frac{(94.0 \text{ g} + 104.7 \text{ g} - 200 \text{ g})}{200 \text{ g}} \times 100\% \]
\[ = -0.65\% \]

Therefore the overall mass balance error is -0.65\%.

A\text{5.d} Determination of \% BaSO\text{4} Recovered

The \% BaSO\text{4} recovered may be determined based on the observed values of composition and weight of either the tails or the float.

Based on the observations of the float:

\[ \% \text{ BaSO}_4 \text{ recovered} = \frac{(\text{GF})(\text{WF})}{(\text{GI})(\text{WI})} \times 100\% \]
\[
\frac{(0.113)(94.0 \text{ g})}{(0.0956)(200.0 \text{ g})} \times 100\% = 55.55\%
\]

Therefore 55.55\% of the \( \text{BaSO}_4 \) was recovered based on the float measurements.

Based on the measurements of the tails:

\[
\% \text{BaSO}_4 \text{ recovered} = \frac{[(\text{GI})(\text{WI}) - (\text{GT})(\text{WT})]}{(\text{GI})(\text{WI})} \times 100\%
\]

\[
= \frac{[(0.0956)(200.0)-(0.0805)(104.7\text{ g})]}{(0.0956)(200.0 \text{ g})} \times 100\% = 55.92\%
\]

Therefore 55.92\% of the \( \text{BaSO}_4 \) was recovered based on the tail measurements.

The average \% \( \text{BaSO}_4 \) recovered based on float and tail measurements is 55.74 \pm 0.258\%.

The \% \( \text{BaSO}_4 \) mass balance error can also be determined from:

\[
\% \text{BaSO}_4 \text{ mass balance error} = \frac{[(\text{GT})(\text{WT}) + (\text{GF})(\text{WF}) - (\text{GI})(\text{WI})]}{(\text{GI})(\text{WI})} \times 100\%
\]

\[
= \frac{[(0.0805)(104.7\text{ g}) + (0.113)(94.0\text{ g}) - (0.0956)(200.0\text{ g})]}{(0.0956)(200.0 \text{ g})} \times 100\% = -0.36\%
\]

Therefore the \% \( \text{BaSO}_4 \) mass balance error was 0.36\%.

A5.5 Determination of the Upgrading Factor

The upgrading factor (ratio of grade of \( \text{BaSO}_4 \) in the float to that of the feed) may also be calculated from float or tail measurements.
Based on float product observations:

upgrading factor = \( \frac{GF}{GI} \)

\[ \begin{align*}
&= \frac{0.113}{0.0956} \\
&= 1.182
\end{align*} \]

The upgrading factor based float measurements is 1.182.

Based on tail measurements

\[ \begin{align*}
\text{upgrading factor} &= \frac{(GI)(WI) - (GI)(WT)}{(WI - WT) GI} \\
&= \frac{(0.0956)(200.0 g) - (0.0805)(104.7 g)}{(200 g - 104.7 g) 0.0956} \\
&= 1.174
\end{align*} \]

Therefore the upgrading factor is 1.174 based on the tail measurements. The average upgrading factor is 1.178 ± 0.006.

A5.6 - Determination of the % Separation Efficiency

The % separation efficiency is the difference between the % recovery of the desired component and the % recovery of the undesired component in the float. The % separation efficiency may be determined from:

\[ \% \text{ separation efficiency} = \% \text{ BaSO}_4 \text{ recovered} \left[ 1 - \frac{1}{\text{upgrading factor}} \right] \]

Then based on float measurements
% separation efficiency = 55.55% \left(1 - \frac{1}{1.182}\right) \\
= 8.55\% \\

Therefore the % separation efficiency based on float measurements is 8.55%.

Then based on tail measurements

% separation efficiency = 55.92% \left(1 - \frac{1}{1.174}\right) \\
= 8.29\% \\

Thus based on tail measurements the separation efficiency is 8.29%.

The average % separation efficiency is 8.42 ± 0.188%.
APPENDIX 6 PARTICLE SIZE DETERMINATION
APPENDIX A6  Particle Size Determination

The average particle size for minerals used in the flotation experiments was determined using electron transmission microscopy.

A6.a Procedure

In order to use the transmission electron microscope copper grids containing the mineral sample had to be prepared. It was imperative that the mineral sample be well dispersed on the grids.

To obtain an adequate mineral dispersion approximately 5 g of ore was added to 100 mL of methanol and agitated for one hour. After this time period several drops of this slurry was pipetted into approximately 80 mL of methanol until a slight turbidity in this solution was observed.

To further ensure adequate dispersion the dilute slurry was treated with an ultra-sonic dismembrator for 15 minutes.

One drop of this dilute slurry was placed on copper grid covered with a parlodium film and was allowed to dry. Upon drying a drop of a dilute solution containing 2.0 μm latex spheres was added to each grid.

After being allowed to dry for a second time the grids were then coated with carbon by passing an electrical current through carbon electrodes under a high vacuum.
The grids were then analyzed with an electron transmission microscope with photographs taken. The particles on the photograph were measured and counted using a ZIess particle counter. The latex spheres were used to establish an accurate magnification factor.

A6.6 Particle Size Distribution

The results obtained from the Zeiss particle counter were plotted on log-normal probability paper (Figures A6-1 to A6-3) and found to yield a straight line over most of its length.

From Figures A6-1 to A6-3 the geometric number weighted average particle diameter, \( \langle D_p \rangle \), and the geometric standard deviation, \( \sigma_G \), were estimated. The results are given in Table A6-1. Also included in Table A6-1 are the geometric surface weighted particle diameter, \( G \langle D_p \rangle_{32} \) and mass weighted average particle diameter, \( G \langle D_p \rangle_{43} \).

These diameters are obtained using the Kapteyn's equations given below:

\[
\ln G \langle D_p \rangle_{32} = \ln G \langle D_p \rangle + 2 \ln^2 \sigma_G \\
\ln G \langle D_p \rangle_{43} = \ln G \langle D_p \rangle + 3 \ln^2 \sigma_G
\]

The number of particles counted for each size distribution is also included in Table A6-1.
<table>
<thead>
<tr>
<th></th>
<th>Silica</th>
<th>Barite Ore</th>
<th>BaSO₄ Powder</th>
</tr>
</thead>
<tbody>
<tr>
<td>No. of Particles Counted</td>
<td>1,765</td>
<td>2,014</td>
<td>1,222</td>
</tr>
<tr>
<td>G&lt;sub&gt;Dp&lt;/sub&gt; (μm)</td>
<td>0.59</td>
<td>0.218</td>
<td>0.298</td>
</tr>
<tr>
<td>d&lt;sub&gt;G&lt;/sub&gt;</td>
<td>2.80</td>
<td>2.71</td>
<td>1.53</td>
</tr>
<tr>
<td>G&lt;sub&gt;Dp&lt;/sub&gt;₃₂ (μm)</td>
<td>4.92</td>
<td>1.59</td>
<td>0.43</td>
</tr>
<tr>
<td>G&lt;sub&gt;Dp&lt;/sub&gt;₄₃ (μm)</td>
<td>14.19</td>
<td>4.30</td>
<td>0.51</td>
</tr>
</tbody>
</table>