Original Article

Effect of different reagent regime on the kinetic model and recovery in gilsonite flotation

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ABSTRACT

Gilsonite is a natural fossil resource, similar to an oil asphalt high in asphaltenes. To determine the effect of reagent regime on the kinetic order and rate of flotation for a gilsonite sample, experiments were carried out in both rougher and cleaner flotation process. Experiments were conducted using different combinations of reagent: oil – MIBC; gasoline – pine oil; and one test without any collector and frother. According to results, kinetic in the test performed using the oil – MIBC and without any collector and frother were found to be first-order unlike the kinetic in the test conducted using the gasoline – pine oil. Five kinetic models were applied to the modeling of data from the flotation tests by using MATrix LABoratory software. The results show that all experiments are highly in compliance with all models. The kinetic constants (k) in rougher stage were calculated as 0.1548 (s⁻¹), 0.2300 (s⁻¹) and 0.2163 (s⁻¹) for oil – MIBC, gasoline – pine oil, and test without any collector and frother, respectively. These amounts in the cleaner stage were 0.0450 (s⁻¹), 0.1589 (s⁻¹) and 0.0284 (s⁻¹), respectively. The relationship between k, maximum combustible recovery (R∞) and particle size was also studied. The results showed that the R∞ and k were obtained with a coarse particle size of (~250 + 106) μm in the rougher and (~850 + 500) μm in cleaner flotation processes.

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1. Introduction

Froth flotation is a common mineral processing method to selectively separate hydrophobic valuable minerals from hydrophilic minerals. Specific reagents are added to the slurry prior to the flotation process to increase the differences in surface properties of the desired and gangue minerals, allowing better separation in terms of selectivity and recovery. The efficiency in this process is determined by the grade and recovery of the valuable minerals. In practical flotation operations, the reagent dosage, froth level, airflow rate, and pH of the slurry are manipulated to ensure an expected concentrate grade [1-4].

Flotation kinetic models dominate almost all the flotation conditions regardless of the ore type and characteristics as well as flotation cell configurations. In addition, applying the
Table 1 – Flotation kinetic models used in this study.

<table>
<thead>
<tr>
<th>No.</th>
<th>Models</th>
<th>$F(k)$</th>
<th>Kinetic parameters</th>
<th>$R$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Classical first-order model [9]</td>
<td>$\delta (k - k_{aw})$</td>
<td>$k_{aw}$-average rate constant</td>
<td>$R_\infty \left( \frac{1}{1 - e^{-kt}} \right)$</td>
</tr>
<tr>
<td>2</td>
<td>First-order model with rectangular distribution of floatability [10]</td>
<td>$\frac{1}{k_{max}} \cdot 0 \leq k \leq k_{max}$</td>
<td>$k_{max}$-fastest floating rate constant</td>
<td>$R_\infty \left( \frac{1}{1 - e^{-kt}} \right)$</td>
</tr>
<tr>
<td>3</td>
<td>Fully mixed reactor model [11]</td>
<td>$\frac{kt}{1 + e^{-kt}}$, $k_{aw} = \frac{1}{e^{kt}}$</td>
<td>$k_{aw}$- average rate constant</td>
<td>$R_\infty \left( \frac{1}{1 - e^{-kt}} \right)$</td>
</tr>
<tr>
<td>4</td>
<td>Second-order kinetic model [12]</td>
<td>$\frac{kt^{1/2}}{t_{pp}}$, $k_{aw} = \frac{1}{e^{kt^{1/2}}}$</td>
<td>$k_{aw}$- average rate constant</td>
<td>$R_\infty \left( \frac{1}{1 - e^{-kt^{1/2}}} \right)$</td>
</tr>
<tr>
<td>5</td>
<td>Second-order model with rectangular distribution of floatability [13]</td>
<td>$\frac{1}{c^2}$ for $0 \leq k \leq c$, $0 for c \leq k \leq \infty$</td>
<td>$c$- average rate constant</td>
<td>$R_\infty \left( \frac{1}{1 - \frac{c^2}{t}} \ln (1 + k) \right)$</td>
</tr>
</tbody>
</table>

The equation above was suggested by Arbiter (1951), where $c$ is the solids concentration, $t$ is time, $k$ is the flotation rate constant, and $n$ is the kinetic order [8]. Some of the conventional kinetic models are presented in Table 1.

The classical first-order kinetic model is comparatively a better model and can be utilized to optimize the flotation process. It is also applicable to both batch and continuous flotation processes with high confidence level [14]. The first-order models can be used to describe most mineral flotation processes, while there is also evidence that the non-integral-order equation is capable of representing the kinetic characteristics of the batch flotation process [15]. Albijanic et al. (2015) focused on the potential application of kinetic models to variable pulp chemical conditions. The flotation tests were performed at a wide range of chemical conditions. The results showed that fully mixed reactor model (No. 3 in Table 1) and second-order kinetic model (No. 4 in Table 1) predicted flotation recovery slightly better than the first-order model with Dirac delta function (No. 1 in Table 1) and rectangular model (No. 2 in Table 1) for both fixed and variable pulp chemical conditions [16]. However, a kinetic model relating the operating variables (reagent dosage, interface level, airflow rate, and slurry pH) to flotation performance (concentrate grade and recovery) in an actual flotation plant has been difficult to develop due to the lack of efficient on-line measurement instruments.

Gilsonite deposits are often found in oil-producing countries. USA, Canada, Iran (holds about 15% of the gilsonite reserves), Iraq, Russia, Venezuela, China, Australia, Mexico, and the Philippines are the main locations of gilsonite reserves [24]. The world’s annual consumption of gilsonite is more than 90 Mt and has different applications such as [18,25]:

- 60–70% is used for road paving;
- 10–20% is used in waterproofing and anticorrosive materials; and
- 10–20% is used in other industrial applications (e.g. as an additive in oil drilling fluids, as both pigment and binding agent in paints, enamels, and inks).

Gilsonite is a natural fossil resource, similar to a petroleum asphalt high in asphaltenes [17]. It can be in liquid or solid form, possibly originating from protein transformations and biodegradations. Solid gilsonite is known for its high content...
Asphaltenes, usually derived from either coal or petroleum vacuum distillation residues, are widespread, and a class of compounds that are characterized by their solubility, i.e. insoluble in n-heptane and soluble in toluene [18]. Fig. 1 shows the schematic model of the gilsonite structure.

Gilsonite asphaltenes are similar to other petroleum source asphaltenes, having a polar core and they tend to interact and form aggregates or macromolecules not observed in simple liquids [18]. Gilsonite occurs as a highly diverse mixture of molecules of various size and structure. The model
structure shown here represents the high molecular weight fraction, while smaller fragments are also common [17]. Mostly, gilsonite deposits are used without any processing operations in various industries according to their performance grade (PG). Nowadays, processing studies on gilsonite are important due to a reduction in the number of high-grade reserves, and consequently, there is a necessity for the extraction and concentration of low-grade gilsonite resources. Most of the previous studies about gilsonite have looked at the structure and applications of it, and less attention has been paid to its processing [17–22]. Alkyl chain content derived from IR spectra have been quantitatively related to insolubility [23]. In addition, given the natural properties of gilsonite particles (their hydrophobicity) and their associated waste materials (extractive waste), flotation is one of the methods for processing fine gilsonite particles. Due to the different structure of gilsonite and other minerals (the presence of organic and inorganic materials in its composition), different surface properties of gilsonite particles and associated waste minerals and the effect of operating conditions on particles floatability, determination of the kinetic parameters of gilsonite flotation is important.

The present work is part of a comprehensive study on the processing and selecting the best location for a gilsonite processing plant (in Kermanshah province-Iran).

In line with this research work and concerning the flotation process for gilsonite, our previous publications have covered the following aspects:

- The order of kinetic models, rate constant distribution, and maximum combustible recovery in gilsonite flotation [26]; and
- The difference in recovery and kinetics of various size fractions of gilsonite in rougher and cleaner flotation processes [27].

The present study is carried out in order to determine the effect of reagent regime on kinetic parameters of gilsonite flotation. The efficiency of the gilsonite flotation process is different from other minerals due to its different structure and the presence of organic and inorganic materials in its composition. In this research, with the aim of determining the optimal operating conditions of the flotation, the effect of operational parameters on kinetic parameters of the flotation process was investigated. In addition, the difference in flotation rates of various size fractions of gilsonite between rougher and cleaner flotation processes was studied. Five flotation kinetic models were applied in fitting the flotation process data from the rougher and cleaner flotation tests. MATLAB software was used to estimate the relationship between the flotation rate constant (k), the maximum combustible recovery ($R_{\infty}$), and the particle size based on the nonlinear least-square optimization method.

2. Materials and methods

2.1. Materials

The natural bitumen deposits of Iran lie along the main Zagros fault with a SE-NW direction. These deposits are found in the Folded Zagros belt, which is 200–250 km wide (Fig. 2). The sample used in this study was obtained from Geraveh mine of the Kermanshah province, Iran (Located on the border with Iraq) (Fig. 2). Shale, Sulfur and non-sulfur particles were detected as impurities (or gangue) within the sample using optical microscopic analysis (Fig. 3); silica, shale and silt particles were dispersed in the bitumen background and small cracks were filled with calcite as a secondary mineral. The main gangue materials in the gilsonite sample were carbonate (calcite and dolomite) and shale compounds, marl,^2^ sulfates like gypsum, fine silica, and opaque^3^ minerals.

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^1^ Roman and infrared spectroscopy

^2^ A kind of soil is calcium carbonate, which has several types of minerals and aragonite

^3^ Lacking the ability to transmit light through a subject, which in mineral terms describes those that cannot be seen through and will not let any light through
Table 2 – Typical properties and elemental compositions of the gilsonite sample.

<table>
<thead>
<tr>
<th>Test method</th>
<th>Result</th>
<th>Test</th>
</tr>
</thead>
<tbody>
<tr>
<td>ASTM-D3173(^a)</td>
<td>≤3</td>
<td>Moisture content, wt.%</td>
</tr>
<tr>
<td>ASTM-D3172(^b)</td>
<td>29</td>
<td>Fixed carbon, wt.%</td>
</tr>
<tr>
<td>ASTM-D3289(^c)</td>
<td>1.11</td>
<td>Density 25°C</td>
</tr>
<tr>
<td>–</td>
<td>Black</td>
<td>Color in mass</td>
</tr>
<tr>
<td>–</td>
<td>Brown</td>
<td>Color in streak powder</td>
</tr>
<tr>
<td>Element Analysis</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ASTM-D5291(^d)</td>
<td>74.00</td>
<td>Carbon, wt.%</td>
</tr>
<tr>
<td>ASTM-D5291</td>
<td>7.10</td>
<td>Hydrogen, wt.%</td>
</tr>
<tr>
<td>ASTM-D5291</td>
<td>0.67</td>
<td>Nitrogen, wt.%</td>
</tr>
<tr>
<td>ASTM-D5291</td>
<td>3.10</td>
<td>Oxygen, wt.%</td>
</tr>
<tr>
<td>LECO (s, Analyzer)</td>
<td>4.00</td>
<td>Sulfur, wt.%</td>
</tr>
</tbody>
</table>

\(^a\) Standard test method for moisture in the analysis sample of coal and coke.
\(^b\) Standard practice for proximate analysis of coal and coke.
\(^c\) Standard test method for density of semi-solid and solid asphalt materials (nickel crucible method).
\(^d\) Standard test methods for instrumental determination of carbon, hydrogen, and nitrogen in petroleum products and lubricants (LECO analyzer).

Table 3 – Results of the nonlinear regression of the rougher data using kinetic models (solid of concentration 10%).

<table>
<thead>
<tr>
<th>Models no.</th>
<th>Test with oil collector and MIBC frother</th>
<th>Test gasoline collector and pine oil frother</th>
<th>Test in the absence of collector and frother</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(R_m (%)) k (s(^{-1}) R(^2))</td>
<td>(R_m (%)\ k (s(^{-1}) R(^2))</td>
<td>(R_m (%)\ k (s(^{-1}) R(^2))</td>
</tr>
<tr>
<td>1</td>
<td>78.32 0.0451 0.997</td>
<td>88.03 0.0554 0.999</td>
<td>78.85 0.0563 0.984</td>
</tr>
<tr>
<td>2</td>
<td>85.12 0.1013 0.999</td>
<td>94.60 0.1310 0.999</td>
<td>84.92 0.1316 0.999</td>
</tr>
<tr>
<td>3</td>
<td>85.25 0.1536 0.999</td>
<td>96.91 11.10 0.999</td>
<td>87.52 11.50 0.998</td>
</tr>
<tr>
<td>4</td>
<td>85.25 0.0007 0.999</td>
<td>96.91 0.0901 0.999</td>
<td>87.52 0.0010 0.998</td>
</tr>
<tr>
<td>5</td>
<td>93.06 0.1548 0.998</td>
<td>100 0.2300 0.999</td>
<td>91.52 0.2163 0.999</td>
</tr>
</tbody>
</table>

Table 4 – Results of the nonlinear regression of the cleaner data using kinetic models (solid of concentration 10%).

<table>
<thead>
<tr>
<th>Models no.</th>
<th>Test with oil collector and MIBC frother</th>
<th>Test gasoline collector and pine oil frother</th>
<th>Test in the absence of collector and frother</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(R_m (%)) k (s(^{-1}) R(^2))</td>
<td>(R_m (%)\ k (s(^{-1}) R(^2))</td>
<td>(R_m (%)\ k (s(^{-1}) R(^2))</td>
</tr>
<tr>
<td>1</td>
<td>85.01 0.0221 0.996</td>
<td>100.20 0.0454 0.982</td>
<td>78.74 0.0154 0.991</td>
</tr>
<tr>
<td>2</td>
<td>97.09 0.0422 0.999</td>
<td>109.09 0.1004 0.986</td>
<td>91.61 0.0284 0.999</td>
</tr>
<tr>
<td>3</td>
<td>105.96 0.4215 0.986</td>
<td>112.57 15.15 0.988</td>
<td>103.26 70.84 0.997</td>
</tr>
<tr>
<td>4</td>
<td>105.96 0.0002 0.986</td>
<td>112.57 0.0660 0.987</td>
<td>103.26 0.0001 0.997</td>
</tr>
<tr>
<td>5</td>
<td>117.49 0.0450 0.999</td>
<td>118.40 0.1589 0.984</td>
<td>117.25 0.0266 0.999</td>
</tr>
</tbody>
</table>

Table 2 shows the chemical characteristics of the gilsonite sample. Moisture, fixed carbon, and density measured by a standard test method for the analysis sample of coal and coke. LECO analyzer (CS844) was used to measure the concentration of elements in the gilsonite sample. LECO analysis is a reliable method for determining the concentration of elements within an organic and metallic sample, including Carbon, Hydrogen, Nitrogen, Oxygen, and Sulfur. LECO supplies a variety of high purity accelerators for use in induction furnace carbon and sulfur determinators. The LECO system requires only a nominal sample weight, and analysis involves oxidizing the sample material using an induction furnace. During the combustion process, carbon is converted to CO\(_2\) and measured with an infrared detector.

Organic and inorganic waste materials with gilsonite are called ash (similar definition of ash about coal). The presence of ash causes a reduction in the thermal value of gilsonite, pollution of the environment due to SO\(_2\) and H\(_2\)S (sulfur) emissions and generally decreases the quality of gilsonite for various industrial applications. In order to determine the amount of ash in a gilsonite sample, 3–4 g of powdered gilsonite particles were burnt in a furnace at 700 °C for 90 min. The initial ash content of the sample was 35%. Results of the sieve analysis is showed in Fig. 4.

2.2. Flotation experiments

All flotation reagents were of analytical grade and utilized without any further purification. Oil and gasoline were used as collectors and MIBC\(^4\) and pine oil as frothers. Rougher tests were conducted using three reagent combinations: oil – MIBC; gasoline – pine oil; and without collector and frother. The rougher tests were performed using two solids concentrations of 10% and 25% by weight. Moreover, the concentrate obtained in rougher stage was subjected to a cleaner stage.

\(^4\) Methyl Isobutyl Carbinol
Rougher experiments were conducted on the sample with size fraction of $-850 \mu m$, using a 4.5 L Denver D12 flotation cell with 1800 rpm agitation speed. To perform flotation tests, after preparing the pulp with a defined solid ratio, the collector was added to the cell and mixed for 2 min; afterward, frother was also added and mixed for 30 sec. The flotation process proceeded after opening the air valve and froth was collected for 200 sec.

In order to compare the kinetic parameters of the various size fractions between the rougher and cleaner stages, the operational parameters were the same in both stages. The cleaner feed was the rougher concentrate (with pulp density of 10% solids by weight). Collector and frother were not added in the cleaner stage.

The final products of all experiments (both the rougher and the cleaner stages) were divided into 6 products according to the collection periods: (0–20), (20–40), (40–60), (60–80), (80–120), and (120–200) s (Fig. 5). Furthermore, to determine the kinetic of flotation for each size fraction, each product was screened into five narrow size fractions: 850, 500, 250, 106, and 75 $\mu m$, and all the fractions were weighed and analyzed for their ash content. After obtaining ash content, flotation recovery ($%R$) was calculated according to the Eq. 2.

$$%R = \frac{W_c (100 - A_c)}{W_f (100 - A_f)}$$

where, $W_c$ is the concentrate weight, $A_c$ is the ash content of the concentrate and $W_f$ and $A_f$ are the feed weight and the ash content of the feed, respectively.
Matrix Laboratory (MATLAB) software (Version 8.3) was used to simulate the flotation rate constant (k), maximum combustible recovery ($R_\infty$), and the correlation coefficient ($R^2$) based on the nonlinear least-square optimization method. Nonlinear least squares optimization has been widely used in the non-linear regression, curve fitting and optimization of nonlinear model parameters.

Also, the cumulative combustible recoveries of various size fractions after (0–20), (20–40), (40–60), (60–80), (80–120), and (120–200) s of flotation time were fitted using the six kinetic models. The narrow size fractions included (−850 + 500), (−500 + 250), (−250 + 106), (−106 + 75), and (−75) μm.

3. Results and discussions

3.1. Kinetics orders, models and parameters of rougher and cleaner tests

There are two ways for specifying the flotation kinetic: (1) obtaining the graph of linear kinetic equations and (2) investigating the dependence of the obtained concentration on the initial concentration value [28]. The recovery diagrams of the rougher experiments using different combinations of solid concentration, collectors and frothers are presented in Fig. 6. According to these graphs, the time required for 50% recovery in the oil – MIBC experiments for both 10 and 25 percent of
solids concentration was 20 sec. It is worth mentioning that 50% recovery for both 10 and 25 percent solids concentration in the absence of collector and frother was obtained in 20 sec. Half-time (the time for 50% recovery of combustible material) in the gasoline – pine oil test at 25% solids was 25 sec, and in the 10% solids not be achieved. Considering the results, the oil – MIBC test and test in the absence of collector and frother were found to follow first-order kinetics, unlike test performed using the gasoline – pine oil. In the next section, the kinetic parameters of the galsonite flotation are derived.

As shown in Fig. 6, the recovery in gasoline – pine oil test with a pulp density of 10% solids by weight, has reached up to 63.05% within 20 sec of the processing time. This amount of recovery is 13% higher than that in the oil – MIBC test and 9% higher than that in the test with no collector and frother addition. According to Li et al. [18] with respect to FITR analysis, the contrast interface of the darker region and the brighter region corresponds to possible phase boundaries between the polycrystalline structures and the amorphous materials or some structures at a different zone axis [18]. Considering the space between galsonite layers, it can be concluded that its structure is similar to a graphite sheet. However, galsonite contains steric distortions due to peripheral alkyl groups on the stacked polycyclic aromatics, or some other type of small clusters of fused rings, such as porphyrins, which are known to self-assemble and form stacked structures [17]. In froth flotation, graphite ore is treated with hydrocarbon oil to enhance the hydrophobicity and in turn recovery. The effect of gasoline and pine oil on flotation performance was discussed earlier and appropriate results have been obtained [29].

Therefore 63% of the recovery in the gasoline – pine oil test can be attributed to the proper effect of gasoline collectors on the flotation of galsonite particles. As presented in Fig. 6, it is clear that recovery of over 90% is obtained in less than 50 sec in the oil – MIBC test and test with no collector and frother addition, in the pulp density of 25% solids by weight. While in similar conditions, this amount of recovery in gasoline – pine oil test is achieved in 200 sec. Considering the significant difference in residence time and kinetic, the appropriate reagent regime could be a key factor in unit design and development for the flotation process. The results of the experiments at the rougher were fitted to the kinetic models (listed in Table 1) and the associated parameters were calculated. Fig. 7 indicates that the results of the rougher flotation experiments are highly in compliance with all models. Similar findings were also reported by other researchers for coal flotation [30,31]. The calculated parameters are presented in Table 3.

According to Table 3, in the flotation test with gasoline – pine oil, values of $R_{\infty}$ and $k$ in all models, are more than that in the other tests. Gasoline is frequently used as a collector for coal, graphite, and material with similar structure floatability. Application of gasoline can reduce reagents consumption; increase the recovery of flotation and flotation rate. It can also improve the flotation selectivity of coal slime [32–34]. In addition, the gasoline, which is with the addition of hexyl alcohol in preparation, is of the strongest stability and the highest
flotation recovery of graphite and artificial mixing ore [35]. Some researchers [36,37] have found that the smaller gasoline droplets tend to increase gold particle collision speed, improve the flotation rate and enlarge adsorption heat quantity of oil on the coal surface.

In rougher experiments using the oil – MIBC combination, the maximum kinetic constant (except model 3) is 0.1548 (s\(^{-1}\)), the retention time is 200 sec and the infinite recovery (\(R_{\infty}\)) is 93.06%. These parameters were 0.2163 (s\(^{-1}\)), and 91.52%, respectively for the test performed in the absence of collector and frother. In the test using gasoline – pine oil combination, the kinetic constant (in the best match with the second-order rectangular distribution model) is 0.2300 (s\(^{-1}\)) and the combustible recovery is 100%. The ash contents of the flotation concentrates for oil – MIBC, gasoline – pine oil and the test with no reagents were 24.56%, 25.55%, and 26.41%, respectively. Therefore, to obtain a gilsonite product with lower ash content, the combination of oil – MIBC is proposed.

Fig. 8 illustrates the fitness of the kinetic models for the recovery-time data of the cleaner experiments. The obtained curves indicate that the kinetic data for all tests at the cleaner stage also is highly in compliance with all of the models (\(R^2 > 0.98\)). The values of the kinetic parameters for each kinetic model are presented in Table 4.

According to Table 4, it can be observed that all of the \(R_{\infty}\) values of models 3, 4 and 5 were greater than 100% in the cleaner flotation process. However, the maximum combustible recovery obtained from the flotation test was more than 100%, which was a theoretical value, and it is physically meaningless. In theory, the maximum \(R_{\infty}\) value calculated by kinetic models was also 100% at most. Therefore, we tentatively suggest that fitting the results obtained from the cleaner flotation process to models 3, 4 and 5 were considered to be unreasonable. The results may be attributed to the low convergence speeds of these models, i.e., the time for obtaining the maximum yield (\(R_{\infty}\) value) in the fitting curves of these models was much greater than that in the rougher tests. Also, the fitted values for three of the models with \(R_{\infty} > 100\%\) might have raised because mathematical parameter fitting techniques can provide a mathematical solution without any connection with physical realities.

Comparison of Tables 3 and 4 indicates that the combustible recoveries (\(R_{\infty}\)) of all models in the cleaner flotation process were bigger than those in the rougher flotation process. It should be pointed out that the combustible recoveries in the cleaner flotation test were calculated on the basis of the gilsonite in the rougher concentrate (the feed to the cleaner), while those in rougher flotation process were calculated on the basis of the feed of the raw gilsonite. The reason for the combustible recoveries in the cleaner flotation process being greater than those in the rougher flotation process might be related to the difference in floatability of the feed used in the rougher and cleaner flotation tests. A large number of difficult-to-float particles were rejected to the tailings in the rougher flotation, and the floatability of the feed in the cleaner flotation was better than that in the rougher flotation since the feed in the rougher flotation was raw gilsonite.

![Fig. 11](image)

**Fig. 11** – Effect of particle size on recovery in the cleaner flotation. (A) Oil – MIBC, (B) gasoline – pine oil, and (C) absence of collector and frother.

### 3.2 Effect of particle size on the flotation kinetics

The recovery-time graphs of the flotation experiments using different size fractions are depicted in Fig. 9. As time passes, recovery increases and then approaches to a constant value. The highest recovery is achieved in particle size fraction of (–250 + 106) μm. This indicates that the maximum recovery is obtained for the particles with a medium-size fraction. Similar results were reported for coal flotation [31,38,39].

Flotation is performed in three sub processes including particle-bubble collision, attachment and stable transport of the particle-bubble aggregate [31,39,40]. On the other hand, the separation efficiency between the valuable mineral and
fully liberated and dispersed gangue depended on the degree of entrainment. Unlike real flotation, which is chemically selective to the mineral surface properties, both gangue and valuable minerals similarly can be recovered by entrainment and entrapment. The entrainment of hydrophilic gangue is controlled by four factors, including the recovery rate of water [41], slurry viscosity [42], particle mass and size [43] and shape. Clearly, the particle size is one of the most important factors that affect flotation efficiency.

Particles within size fraction of \((-250 + 106) \mu m\) has the most recovery, due to not having the weight and size for the entrainment of fine particles. In smaller size fractions, the presence of fine particles and free clay, create a coating on the surface of the particles and reduce the flotation recovery. In case of the coarser particles, the dissemination of fine clay particles within gilsonite grains and ash-bearing component such as dolomite and shale, decreases the overall recovery.

Gilsonite recovery at size fraction of \((-250 + 106) \mu m\) and in the presence of various reagent regimes are presented in Fig. 10. Regarding the figure in the whole flotation time, the most recovery in this fraction is related to the gasoline-pine oil flotation test. Using this collector, the gangue consisting of quartz, which is hydrophilic, will ultimately report to the froth phase. Thus, with the increase in the grade of the final concentrate the recovery increased compared to other cases.

Fig. 11 shows the recovery-time curves of the cleaner flotation stage using different particle size fractions. Overall, the variation of the flotation recovery for different size fractions in the cleaner stage for the test with oil – MIBC is similar to the rougher stage. The two highest recoveries are in the \((-250 + 106) \mu m\) and the \((-500 + 250) \mu m\) fraction. This range accounts for 49.05% of the feed. Based on the microscopic studies presented in Fig. 12, the minimum amount of impurities within gilsonite grains, is observed in this size fraction \((-500 + 250 \text{ and } -250 + 106 \mu m)\). In coarse particle, the presence of impurities in the structure of the gilsonite particles is observed.

In case of the test with gasoline – pine oil, the maximum combustible recovery in the cleaner flotation process is obtained from particles in the size fractions of \((-850 + 500) \mu m\) and \((-500 + 250) \mu m\), respectively. Given that particle fracture is also an effective parameter on recovery, it can be said that in the fractions of \((-850 + 500) \mu m\), gilsonite particles have the spherical fracture and the materials in this particle size show the best separation performance.

The occurrence of angular and sharp surfaces in particles causes the accumulation of electric charge at these points. As a result, it reduces the particle hydrophobicity. Therefore, flat spherical shapes are the most appropriate form of the mineral particles in flotation process. Thus, attachment of them to the air bubbles is stronger and the recovery will be higher. The results of conducted test in the absence of collector and frother is similar to the gasoline – pine oil test.

4. Conclusion

In this investigation, the difference in kinetic flotation parameters of the flotation process of gilsonite using various reagent regime was studied. Several flotation experiments were performed on a gilsonite sample with different types of collector and frother, and values of pulp density. According to results, it was concluded that rougher flotation using the oil – MIBC combination and without any collector and frother correlate with the first-order kinetic model. However, the flotation test of gilsonite using gasoline-pine oil (collector-frother) correlate with another model (second-order). Statistical analysis showed that the results of all experiments are highly in compliance with all models \((R^2 > 0.99)\). The maximum kinetic constants \((k)\) (except for model 3) in the rougher stage were calculated as 0.1548 (s\(^{-1}\)), 0.2300 (s\(^{-1}\)) and 0.2163 (s\(^{-1}\)) for oil - MIBC, gasoline – pine oil, and test without any collector and frother, respectively. These parameters in cleaner stage were 0.0450 (s\(^{-1}\)), 0.1589 (s\(^{-1}\)) and 0.0284 (s\(^{-1}\)), respectively. In all of the kinetic models, the maximum recoveries were related to the gasoline-pine oil experiments. In addition, regarding the recovery values and ash contents of the concentrates, rougher and cleaner stages using oil as collector and MIBC as frother are proposed for the gilsonite flotation. Furthermore, the influence of particle size on the recovery of the rougher and cleaner tests was investigated. In the rougher experiments, the highest recoveries were associated with the \((-250 + 106) \mu m\) and the \((-500 + 250) \mu m\) size fractions. The variation of the flotation recovery in the cleaner stage, for the test with oil – MIBC is similar to the rougher stage. In case of the test with gasoline – pine oil, the maximum com-
bustible recovery in the cleaner stage related to the particles in the size fractions of \((850 + 500) \mu m\) and \((-500 + 250) \mu m\), respectively. The results of the conducted test in the absence of collector and frother is similar to the gasoline-pine oil test.

REFERENCES


