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Interaction of applying ultrasonic irradiation in Gilsonite flotation

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ABSTRACT

Flotation is a type of mineral separation process that occurs in a water-mineral slurry. The surfaces of the selected minerals are rendered hydrophobic through conditioning with specific reagents. The material used in this study was gilsonite. After the preparation of the samples, two series of flotation experiments on gilsonite, one with irradiation and one without were designed using the central composite design method. Finally, it was concluded that the ultrasonic method achieved a higher desired recovery rate and a lower ash percentage in the concentrate compared to the conventional method in the rougher stage of flotation.

Keywords: *Gilsonite, Flotation, Rougher, Ultrasonic irradiation.*

1. Introduction

Gilsonite is a crucial and widely used mineraloid with applications across various industries (Lozano-Periera et al., 2023). Due to its chemical composition, naturally occurring gilsonite consists of four fractions and is characterized by a high carbon content (>84%) and a minimal sulfur content (≪0.3%). This material exhibits properties similar to hydrocarbons, including saturated compounds, asphaltenes, resins, and aromatics. Its diverse applications encompass energy production, road paving, inks and paints, oil well drilling, gilsocarbon for nuclear reactors, additives for tire rubber, use in petroleum emulsions, metal smelting, and filters for cyanide ions and toluene retention, among others (Lozano-Periera et al., 2023).

Bahrami et al. conducted experiments in two flotation stages, rougher and cleaner, utilizing different reagents: oil as collector with MIBC* as frother and gasoline as collector with pine oil as frother (Bahrami et al., 2019). They also performed tests without any collector and frother to assess the impact of the reagent regime on the kinetic order and flotation rate of a gilsonite sample. The results indicated that the kinetics for the tests using oil and MIBC, as well as those conducted without any collector and frother, followed first-order reactions, contrasting with the kinetics observed in the tests using gasoline and pine oil (Bahrami et al., 2019). The results indicated that all experiments aligned closely with the respective models. The kinetic constants (k) during the rougher stage were determined to be 0.1548 (s^{-1}), 0.2300 (s^{-1}), and 0.2163 (s^{-1}) for oil – MIBC, gasoline – pine oil, and tests conducted without any collector or frother, respectively (Bahrami et al., 2019). In the cleaner stage, these values were 0.0450 (s^{-1}), 0.1589 (s^{-1}), and 0.0284 (s^{-1}), respectively (Bahrami et al. 2019). Additionally, the relationship between k, maximum combustible recovery (R_∞), and particle size was investigated. The findings revealed that R_∞ and k were highest with coarse particle sizes of (−250 + 106) mm during the rougher flotation process and (−850 + 500) mm in the cleaner flotation process (Bahrami et al., 2019).

Bahrami et al. conducted flotation tests using various reagents, including collectors (gas oil, kerosene, and pine oil), frother (MIBC), and depressants (sodium silicate, tannic acid, sulfuric acid, and sodium cyanide) at different dosages (Bahrami et al., 2021). The findings indicated that using kerosene as the collector, MIBC as the frother, and the mixture of sodium silicate, tannic acid, sulfuric acid, and sodium cyanide as the depressant yielded the most favorable outcomes in the gilsonite flotation during the rougher stage (Bahrami et al., 2021).

Doodran and colleagues conducted flotation tests by examining four factors: the dosage of collector, frother, and depressant, as well as the solid-to-liquid ratio across three different levels (Doodran et al., 2020). The aim was to reduce ash content and enhance the recovery of gilsonite. These tests were structured using the Taguchi method, facilitated by Design-Expert software (Doodran et al., 2020). Our findings revealed that the lowest ash content of 5.2% was achieved under conditions that included 200 g/t of gasoil as the collector, 100 g/t of MIBC as the frother, 300 g/t of sodium silicate as the depressant, and a pulp density corresponding to a 5% solid-to-liquid weight ratio (Doodran et al., 2020).

Bahrami and his team conducted flotation tests during both the rougher and cleaner stages to ascertain the kinetic order and flotation rate of a gilsonite sample (Bahrami et al., 2019). The experiments utilized combinations of oil–MIBC and gas oil–pine oil, with one test performed without any collector and frother (Bahrami et al., 2019). Based on the results, the relationship between the flotation rate constant, maximum combustible recovery, and particle size indicated that the highest flotation combustible recovery and flotation rate occurred within the size range of -250 + 106 μm in both the rougher and cleaner stages (Bahrami et al., 2019). It was observed that the combustible recovery and

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flotation rate were higher during the rougher flotation process compared to the cleaner stage (Bahrami et al., 2019).

Kazemi and colleagues conducted flotation experiments on gilsonite samples from Kermanshah province to determine the flotation kinetics, the effect of particle size , and to examine the relationship between flotation rate constant, maximum recovery, and particle size (Kazemi et al., 2018). For this purpose, flotation tests were performed using a cleaner flotation process with a collector of kerosene and MIBC frother, a diesel collector and pine oil frother, and one test without any collector or frother (Kazemi et al., 2018). The results indicated that the maximum recovery and flotation constant in the flotation experiments conducted with gilsonite, without the use of a collector and frother, is associated with particle sizes of -850, +500 µm. In the cleaner tests, the highest k value and recovery were found in the experiment using diesel collector and pine oil frother, with particle sizes of -850, +500 µm (Kazemi et al., 2018).

Bahrami and colleagues conducted flotation experiments to determine the kinetic order and flotation rate of a gilsonite sample, performing tests in both the rougher and cleaner stages (Bahrami et al., 2020). The experiments utilized combinations of oil-MIBC and gasoil-pine oil, along with one test without any collector or frother (Bahrami et al., 2020). Additionally, the relationship between maximum combustible recovery and particle size was analyzed. The results indicated that the highest flotation combustible recovery and flotation rate were achieved at an intermediate particle size in both the rougher and cleaner flotation processes (Bahrami et al., 2020). Moreover, the combustible recovery and flotation rate were greater during the rougher flotation process compared to cleaner stage (Bahrami et al., 2020).

A key challenge in utilizing raw gilsonite is the reduction of detrimental impurities. Several methods exist for removing these impurities, with flotation being the most important technique for very fine materials. However, despite its widespread use, flotation often fails to reduce impurities (ash) to the desired level. Therefore, this paper investigateed the application of ultrasonic irradiation. In flotation technique, hydrophobic particles attach to air bubbles introduced into the pulp and are lifted to the froth layer above the slurry, thereby separating them from the hydrophilic particles (Wills and Finch, 2016). To achieve better concentration and recovery, flotation operations can be conducted in one or multiple stages. When flotation is performed in a single stage, it is referred to as "rougher" flotation. In rougher operations, the primary focus is on the recovery parameter, while in the flotation of gilsonite, the undesirable parameter is the ash content. A low ash percentage combined with high recovery indicates an ideal rougher test. Another technique used in mineral processing is ultrasound. In this technique, ultrasonic waves are extensively utilized for cleaning and preparation of mineral surfaces through physical, chemical, and physico-chemical processes, significantly enhancing recovery rates. These waves improve the efficiency of reagents and improve recovery by cleaning particle surfaces (Ebrahimi and Karamoozian, 2020). It is proposed to use ultrasonic waves as a pre-treatment method for the flotation of coal with high ash content (Ebrahimi and Karamoozian, 2020). Also, in the ultrasonic radiation technique, which is carried out using ultrasonic waves, these waves act as a secondary collector on ore particles, enhancing collector attachment (Ebrahimi and Karamoozian, 2020). The flotation of gilsonite using the ultrasonic radioactivity method is a novel approach that has not been previously applied, making the present investigation innovative in this field.

2. Material and methods

2.1. Gilsonite samples

The sample of gilsonite prepared in this research was obtained from the Gilaneh mine located in Ilam province in western Iran. Its precise coordinates are as follows: (33.877778°N, 46.076944°E). The gilsonite sample used in this research consists of uncrushed and unclassified feed contained gilsonite powder. This sample was sieved into twelve different size fractions to achieve better results for subsequent tests. The aim of this work was to identify a portion of the initial feed that had the lowest percentage of ash. After conducting a test and measuring the ash content, it was determined that the section (-710, +500) µm matched similar specifications, with an ash content measured at 54.74%. Following this stage, the entire feed was crushed using a laboratory roller crusher, resulting in two different size fractions. To separate these two sizes, the ASTM [†] sieve analysis method was employed, with dimensions as follows: (+500) µm and (-500) µm. In the final stage of the experiment, the ash content of these sizes was again examined and evaluated to obtain more accurate results and to gain a better understanding of the characteristics of this sample. In Table 1 and Figure 1, analysis for screening of gilsonite samples and feed size distribution chart is shown, respectively. Table 2 shows the XRF table for analyzing chemical combinations for the detection of tailing minerals present in gilsonite smaples. As observed in this table, the concentrations of the chemical compounds CaO and SO_3 exhibit a high percentage abundance in the composition of these gilsonite samples, indicating that the mineral present in this sample is, as tailings, either the mineral anhydrite or gypsum. Also Figure 2 shows the XRD plot for determining frequency of each chemical element in samples. XRD analysis of the gilsonite waste revealed two chemical phases: calcium sulfate and calcium oxide. However, since the peaks corresponding to calcium sulfate showed a better match (with a scale factor of 0.723) to the main peaks of the sample than those of calcium oxide (with a scale factor of 0.045), the chemical composition of the mineral of gilsonite tailing is determined to be gypsum.

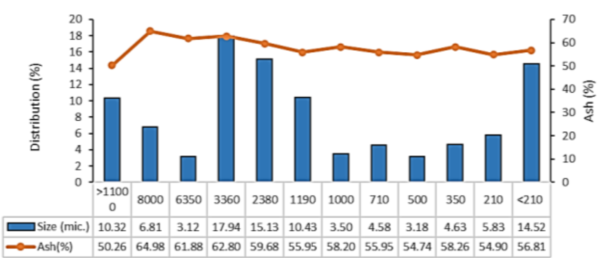


Fig 1. Feed size distribution and ash content plot.

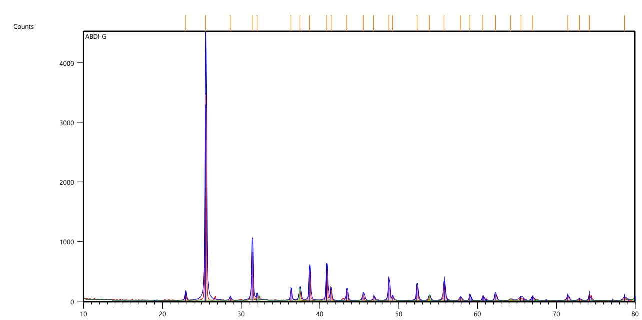


Fig 2. XRD plot for gilsonite samples.

2.1.2. Reagents

The chemical reagents used in these flotation experiments are gasoil and MIBC, where gasoil is utilized as the collector and MIBC as the frother.

[†] American Society for Testing and Materials

Table 1. Analysis table for screening gilsonite samples.

Screen Size (µm)	Residual Weight on Screen (g)	Frequency Percentage (%)	Cumulative Percentage on Screen (%)	Cumulative Percentage Passing from Screen (%)
181864	84.68	10.32	10.32	89.68
131064	55.86	6.81	17.13	82.87
35560	25.63	3.12	20.25	79.75
3360	147.21	17.94	38.20	61.80
2380	124.14	15.13	53.33	46.67
1190	85.53	10.43	63.75	36.25
1000	28.74	3.50	67.26	32.74
710	37.57	4.58	71.84	28.16
500	26.11	3.18	75.02	24.98
350	38.02	4.63	79.65	20.35
210	47.80	5.83	85.48	14.52
<210	119.12	14.52	100.00	0.00
Total	820.41			

2.1.2. Flotation Experiments

The flotation experiments conducted in this study utilized a one-step ore flotation method known as "rougher." To perform the experiments, Denver-type mechanical flotation machines were employed. Regular rougher experiments were carried out using the 2-liter cell, while ultrasonic rougher experiments were conducted with the 1-liter cell. An ultrasonic bath manufactured by Elma was used for irradiation. Several parameters influence the efficiency of the flotation process, and optimizing conditions can enhance the flotation rate. Additionally, the following fixed parameters were established:

- Selected feed size: (-500) µm
- Solid content: 5 (%);
- Collector type: Gasoil;
- Frother type: MIBC;
- Temperature: room temperature (25°C);
- pH: neutral (7);
- Rotor speed: 1000 RPM;
- Ultrasonic device power: 100 W;
- Preparation time: 8 minutes;
- Initial feed mass: 100 g (In regular rougher) and 52.63 g (In ultrasonic Rougher).

Previous research has shown that reducing the solids content increases recovery; therefore, the minimum possible solids content (5%) was considered in this study (Murhula et al., 2022). Given the number and levels of parameters considered for the flotation experiments, a central composite design technique and response surface methodology were selected, resulting in 20 experiments. Experiment design is shown in Table 3, where (R1) is the percentage of gilsonite recovery in the regular rougher method and (R2) is the percentage of gilsonite recovery in the ultrasonic method.

Table 3. Experiment design for gilsonite rougher.

		Factor 1	Factor 2	Response 1	Response 2
Std	Run	A: Collector	B: Frother	R1	R2
		g/t	g/t	%	%
1	9	50	50	87.38	93.12
2	3	200	50	85.47	94.54
3	7	50	200	90.34	86.83
4	2	200	200	86.41	83.15
5	5	19	125	84.29	85.59
6	8	231	125	82.86	86.74
7	6	125	19	93.97	96.47
8	1	125	231	80.14	86.69
9	4	125	125	81.80	87.94
10	10	125	125	82.67	87.41

2.1.3. Equations

Equation (1) was used to calculate gilsonite recovery. (Wills and Finch, 2016).

$$R = \left(1 - \frac{c \times (f-t)}{f \times (c-t)}\right) \times 100 \qquad (1)$$

In this case, the grade of the concentrate (c) is the same as the ash percentage of the concentrate, the grade of the tailings (t) is the same as the ash percentage of the tailings and the grade of the feed (f) is the ash percentage of the initial feed.

2.1.4. Process Machine

In these experiments, the process indicator is the DENVER mechanical flotation cell, which consists of three components: an air valve, a stirring blade, and a speed adjustment dial for the flotation stirring. Figure 3 shows shows the configuration of the setup system.

Fig 3. The setup system for flotation.

2.1.5. Results and discussion

Figures 4 & 5 show the graph of changes in the recovery of a regular gilsonite rougher and ultrasonic rougher with respect to the effect and interaction of the variables of collector concentration and foaming agent concentration, respectively. The range of changes in the concentrations of collector and frother agent parameters was between 50 g/ton and 200 g/ton. In Figure 5, by increasing the value of parameter (A) to the optimal point and also decreasing parameter (B) to a certain optimal point, a better recovery rate can be obtained. In Figure 6, by relatively decreasing the values of parameter (A) and increasing the values of parameter (B) to the maximum point, the optimal point can be reached. Tables 4 and 5 present (ANOVA) tables for (R1) and (R2) responses. In Table 4, the Model F-value of 32.01 indicates that the model is statistically significant, with only a 3.06% probability that an F-value of this magnitude could arise from random noise. P-values below 0.0500 suggest that the corresponding model terms are significant; in this scenario, B, B^2, and A^2B qualify as significant terms. Values exceeding 0.1 indicate that the associated model terms lack significance. If there are multiple insignificant model terms—excluding those necessary to maintain the hierarchical structure, considering model reduction may enhance the overall model performance. The lack of Fit F-value of 2.72 suggests that the lack of fit is not significant when assessed against pure error, with a 34.69% likelihood of obtaining such a large lack of Fit F-value due to noise. A non-significant lack of fit is favorable; our objective is to ensure that the model fits well. In Table 5, the model F-value of 29.52 indicates that the model is statistically significant, with only a 0.30% likelihood that such a large F-value could arise from random variation. P-values below 0.0500 signify that the model terms are significant; in this instance, the terms B and B^2 are deemed significant. Conversely, values exceeding 0.1000 suggest that the model terms are not significant. Should there be multiple insignificant model terms, excluding those necessary to maintain hierarchy, reducing the model could enhance its performance. The lack of Fit F-value of 10.22 suggests that the lack of Fit is not significant when compared to pure error, with a 22.50% chance that such a lack of Fit F-value could occur due to random noise. Tables 6 and 7 show suggested models for "R1" and "R2" responses. To find the appropriate model for the desired response, a model is deemed acceptable if it has higher values of R^2 and adjusted R^2.

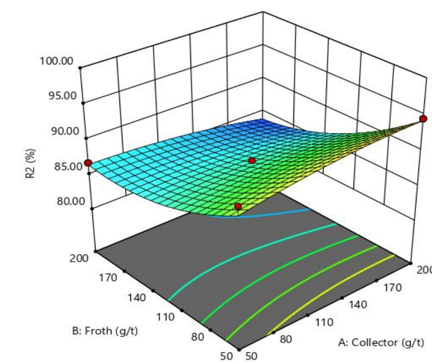


Fig 5. 3D surface plot of ultrasonic rougher flotation tests.

Table 4. ANOVA for reduced quartic model of "R1" response.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	157.13	7	22.45	32.01	0.0306	significant
A-collector	1.03	1	1.03	1.47	0.3488	
B-Froth	95.74	1	95.74	136.52	0.0072	
A^2	1.8	1	1.8	2.57	0.2502	
B^2	23.25	1	23.25	33.16	0.0289	
A^2B	68.85	1	68.85	98.18	0.01	
AB^2	1.82	1	1.82	2.6	0.2484	
A^2B^2	8.69	1	8.69	12.38	0.0721	
Residual	1.4	2	0.7013			
Lack of Fit	1.03	1	1.03	2.72	0.3469	not significant
Pure Error	0.3769	1	0.3769			
Cor Total	158.54	9				

Table 5. ANOVA for reduced quartic model of "R2" response.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	163.5	5	32.7	29.52	0.003	significant
A-Collector	0.0491	1	0.0491	0.0443	0.8436	
B-Froth	124.18	1	124.18	112.11	0.0005	
AB	6.52	1	6.52	5.89	0.0723	
A^2	1.75	1	1.75	1.58	0.2773	
B^2	19.92	1	19.92	17.98	0.0133	
Residual	4.43	4	1.11			
Lack of Fit	4.29	3	1.43	10.22	0.225	not significant
Pure Error	0.1399	1	0.1399			
Cor Total	167.93	9				

Table 6. Suggested model summary statisyics for "R1" response.

Source	Std. Dev.	R^2	Adjusted R^2	Predicted R^2	PRESS	
Linear	4.14	0.2426	0.0262	-0.6345	259.14	Suggested
2FI	4.45	0.249	-0.1264	-1.8261	448.05	
Quadratic	4.46	0.4971	-0.1316	-2.5689	565.81	
Cubic	2.13	0.9428	0.7428	-2.5157	557.37	Aliased

Table 7. Suggested model summary statisyics for “R2” response.

Source	Std. Dev.	R^2	Adjusted R^2	Predicted R^2	PRESS	
Linear	2.5	0.7398	0.6655	0.4037	100.13	
2FI	2.49	0.7786	0.668	0.4235	96.81	
Quadratic	**1.05**	**0.9736**	**0.9406**	**0.815**	**31.07**	**Suggested**
Cubic	0.5972	0.9958	0.9809	0.7781	37.26	Aliased

Figure 6 Perturbation Diagrams illustrate the values of R1 and R2 corresponding to the optimal values of parameters A and B.

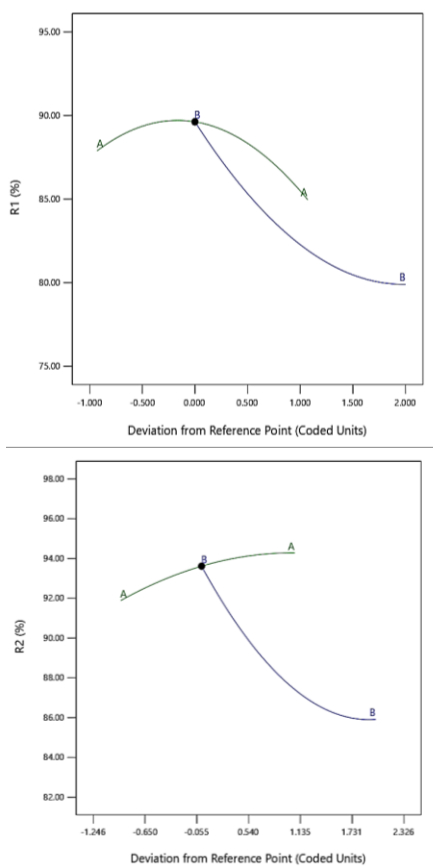
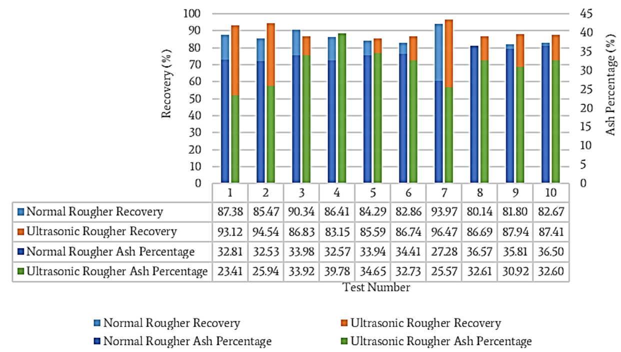


Fig 6. Perturbation plot for R1 and R2.

Figure 7 The comparison of the recovery percentage and ash percentage from tests conducted on both normal and ultrasonic rougher sections. According to this chart, the test number 7 was the best, exhibiting both optimal recovery (highest recovery) and desirable ash percentage (lowest ash percentage) in both sections.



3. Conclusions

The examination and analysis of the findings and results are as follows:

1) In the normal rougher, the best recovery percentage and ideal concentrate ash percentage were obtained, 93.97% and 27.28% and in ultrasonic, 96.47% and 25.57%, respectively.

2) In the ultrasonic rougher, the effect of ultrasonic waves caused a much lower ash percentage to be obtained even in cases where the desired recovery was not obtained.

3) By the application of ultrasonic waves, the desired concentrate ash recovery and percentage can be achieved at different dosages of collector and frother.

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