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Flotation of Fine Gold Particles by the Assistance of Coal-Oil Agglomerates

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This paper describes the use of coal-oil agglomerates in flotation to increase the gold recovery from an ore containing fine gold particles. The effects of operating parameters on gold flotation recovery such as oil type, particle size of agglomerating material, agglomerate/ore and oil/ore ratios were investigated. The studies showed that petroleum oils are more effective than vegetable oils in oil agglomeration of Kozlu coal and coal-oil assisted gold flotation. Gold recovery can be increased using a higher amount of agglomerates in the process; however, gold grade of the flotation concentrates is reduced significantly. The use of bridging oil at high concentrations in the agglomeration process provides high-grade gold concentrates, but lower recoveries. The utilization of coarser coal particles in the coal-oil agglomeration stage leads to higher selectivity and recovery values for gold particles.

Keywords agglomeration; coal; flotation; gold; oil

INTRODUCTION

The problem of recovering fine particles and their behavior in flotation systems have always been an interesting subject (1–9). Several alternative processes, such as particle aggregation, flocculation (10–14), carrier flotation (15,16), oil assisted separation (17–23), have been suggested by various researchers. In oil-assisted separation processes, an immiscible oil is generally used. The function of the oil is to assist the attachment of hydrophobic particles to the bubbles (emulsion flotation), or to provide higher flotation recoveries for fine particles by agglomerate flotation. Oil may also be used to increase the size of the fine particles by agglomeration to allow separation by screening such as oil agglomeration widely used for fine coal beneficiation. The major factors affecting agglomeration of coal with oil include wettability of the coal, the type and amount of the bridging oil and type and intensity of the conditioning. It is often assumed that oily droplets at the coal/water interface spread onto the coal surface and form a thin film (23). However, the deposition of smaller oily droplets can also be observed on the particles (17). Polat et al. (17) summarized particle aggregation in coal flotation from different aspects (Table 1).

The use of coal and oil agglomerates in gold processing is relatively new. Coal-oil-gold agglomeration (CGA) method was first patented by BP Australia Ltd. in 1984 (24,25). The method involves agglomeration of highly hydrophobic coal and gold particles by using an immiscible liquid. The mechanism which lies behind the separation process is the difference in the hydrophobicity or oleophilicity of the particles in the suspension. Coal and gold particles are highly hydrophobic/oleophilic whereas the gangue minerals are generally hydrophilic. The process is most suitable for treating free or liberated gold containing alluvial or free milling ores, gravity concentrates, and tailings (26,27). Different applications for the production of coal-oil-gold agglomerates were proposed by various researchers (28,29). It was found that the gold content of the agglomerates increases with the number of recycles in a linear fashion (26,30,31,34). Wu et al. (31) and Calvez et al. (32) studied the gold adhesion to agglomerates by examining the surface of the coal-oil-gold agglomerates. Wu et al. (33) investigated the kinetics of the CGA process and proposed that the use of smaller agglomerates provides greater collision rates while larger agglomerates give slightly larger attachment probability. Calvez et al. (32) carried out studies on the effect of the amount of bridging liquid and agglomeration material on coal-oil gold agglomeration by changing agglomerate/ore ratio between 0.15 and 1.05. They used oil/coal ratios between 0.25 and 0.43 to produce the agglomerates.

Numerous researchers have conducted CGA tests on different ore types including tailings of different gold recovery processes (35–43). Various semi-commercial and continuous pilot trial tests were conducted about gold recovery by CGA method (25,36,37,44). Bouwer (45) worked on a South Africa coal, with ethyl oleate as the agglomerating agent. Moses and Petersen (46) found that increased stirring speeds increase the gold recovery. Akar

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TABLE 1
Relationship between coal particles and oil droplets without surfactant addition as a function of coal rank and oil concentration (17)

Agent	Rank	Particle aggregation	Recovery	Selectivity
No Surfactant	high	small agglomerates	High	moderate
Low oil	low	no agglomerates	very low	Low
No Surfactant	high	large agglomerates (entrapment)	very high	Low
High oil	low	small agglomerates	Moderate	moderate

et al. (47), Akcil (48), Akcil and Akar (49) conducted some studies on Izmir-Arapdagi epithermal deposit containing 11 g/t gold using different oils (soybean oil, olive oil, kerosene) at four different coal/graphite ratios. Kilinc and Akar (50) also studied the effect of olive oil and graphite on gold recovery from the same ore sample. The best condition resulted in a grade of 51.5 g/t Au and a recovery of 75.2% Au. Akcil et al. (52) conducted an extensive review about the importance of CGA in the processing of the gold ores and summarized the recent research. Our previous work (51) demonstrated that using the artificial gold ore samples the process is very effective for recovering gold particles in a wide size range up to 300 μm particle size and feed grades up to 20 g/t Au without considerable recovery losses. The study reported here is concerned with coal-oil assisted gold flotation (51), which can be defined as a modified coal-oil-gold agglomeration. On the coal-oil-gold agglomeration method, oil and coal particles are used at high consumptions rates to produce coal-oil-gold agglomerates for the recovery of gold. The present study considers assisting gold flotation with micro agglomerates produced by using small amount of oil and coal to help gold recovery. The separation mainly takes place based on two mechanisms: conventional flotation of gold particles and flotation of coal-oil-gold aggregates (Fig. 1).

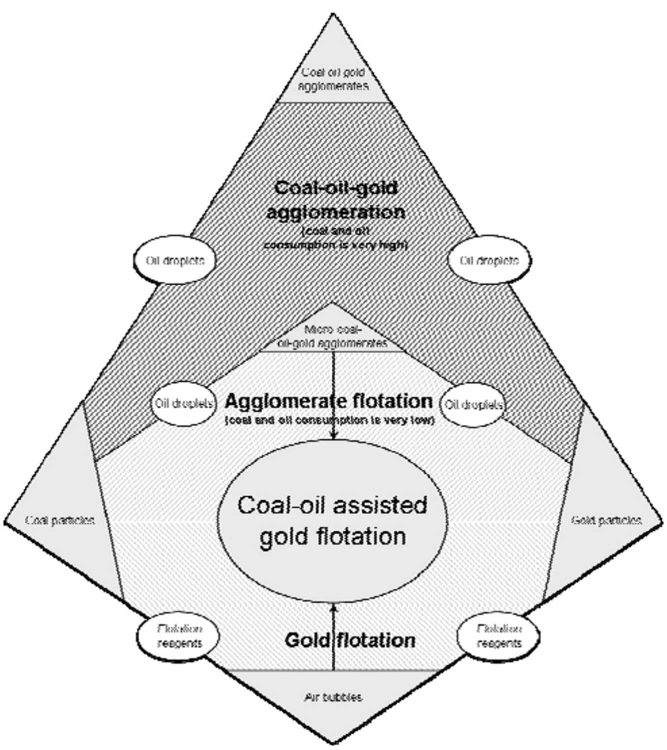

FIG. 1. Schematic representation of coal-oil assisted gold flotation process.

EXPERIMENTAL

Materials

Coal sample was a high-volatile, type A bituminous coal and was received from Kozlu Region of Zonguldak in Turkey. The samples were subjected to characterization studies and the results are presented in Table 2. Coal samples were ground below $d_{80} = 150\,\mu m$ by a hammer mill for experiments.

Gold ore sample was taken from an epithermal gold-silver deposit located in the Western Anatolia. Total sulphide content of the ore is low (<2%) and is dominated by pyrite (Table 3). Gold occurs as fine (<0.05 mm) grains of electrum or in native form, precipitated between interstices of quartz crystals (53). Gold grade of the sample is about 9 g/t Au and about 70% of the gold content is below

TABLE 2
Analysis of Kozlu-Zonguldak coal sample

Proximate analysis	Air dried basis (%)
Moisture	0.95
Ash	13.07
Volatile Matter	27.33
Fixed Carbon	57.75
Total Sulfur	0.90
Calorific Value (Kcal/kg)	
Net	6992
Gross	7224

TABLE 3
Analysis of the gold ore sample

Element	
Au	8.77 g/t
Ag	11.02 g/t
Cu	26.98 g/t
Zn	295.31 g/t
Pb	253.43 g/t
Mn	39.29 g/t
Fe	7102 g/t
Mg	361.46 g/t
Co	9.04 g/t
Cr	111.02 g/t
SiO_2	95.23%
S	0.42%

25 µm. The samples were ground below 60 µm (d80) by a ball mill for experiments (Fig. 2).

Different crude vegetable oils and petroleum oils were used in this study. Viscosity measurements of the oils were carried out with a Brookfield DV-III programmable rheometer. Surface tensions were measured in Kruss-Digital Tensiometer K 10ST. A goniometer/microscope setup (Rame Hart NRL contact angle goniometer model-100) was used for sessile oil drop contact angle measurements in water. The results of the characterization studies of the oil samples were given in Table 4.

Methods

Preparation of the Coal Samples for Contact Angle Measurements

The lumps of coal were broken into small pieces (2.5 cm × 2.5 cm × 1.5 cm) and were placed in a polyester matrix (d = 2 cm). After the polyester matrix was hardened, it was polished using a series of abrasive papers (grits from 60 to 1200), 0.5 µm alumina powders and a fibrous cloth. Abrasive papers and alumina powders were also helped to flatten and smooth the coal surface. Polished samples were stored in vacuumed and sealed plastic bags. The samples were freshly polished again on fibrous cloth before taking contact angle measurements.

Contact Angle Measurements

Contact angle measurements were performed in a square glass container. The container was filled with double-distilled water before the coal sample was placed on a support with the flat surface facing water downward. A micro-droplet of different oils was generated with a U-shaped needle using a microsyringe and placed on a specific portion of the polished coal surface from below. The temperature remained at about 20°C in all measurements.

Coal-Oil Agglomeration Tests

An IKA-Eurostar (Power control-visc) stirrer with a turbine impeller (four bladed) and a four baffled agglomeration cell were used for emulsification. A standard

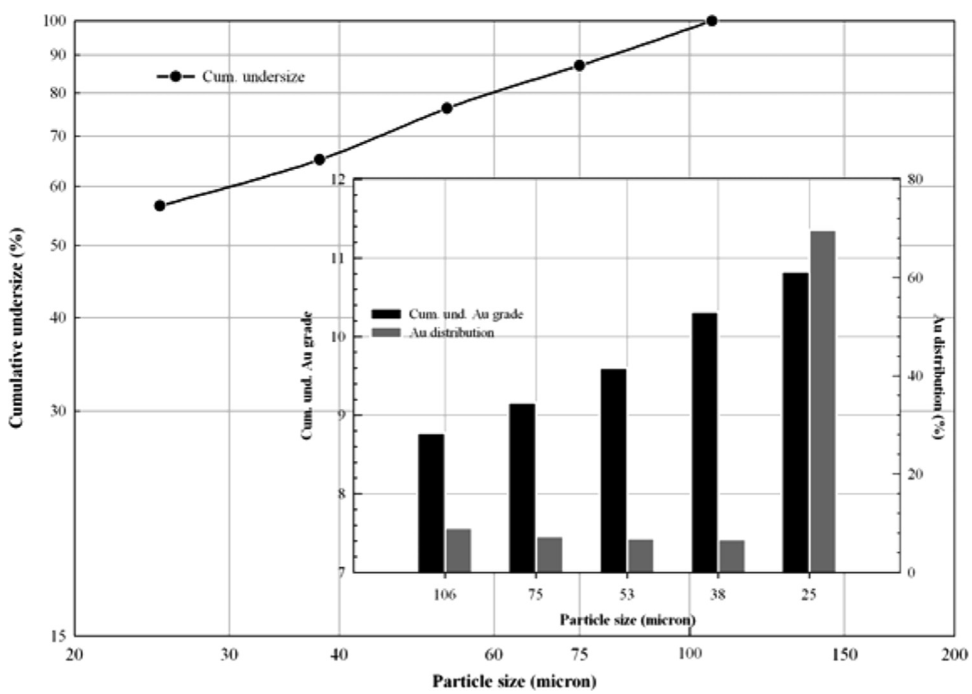

FIG. 2. Bergama-Ovacik gold ore sample particle size and Au distribution curves.

TABLE 4
Physical properties of the oils

Oils Name	Density d (g/cm^3)	Viscosity η (cP)	Interfacial tension $\gamma_{oil/water}$ (dyne/cm)	Contact angle θ_{oil} (°)
Corn oil	0.946	49.5	4.0	56
Cotton oil	0.919	62.0	7.0	56
Olive oil	0.947	68.5	13.2	53
Sunflower oil	0.930	55.0	16.8	52
Diesel oil	0.846	4.16	19.7	45
Kerosene	0.811	1.47	29.9	37

agglomeration procedure was applied in all tests. Oil was added to the emulsifying cell and agitated at 2000 rpm for 5 minutes. Coal/oil ratio was kept constant at 4%. Coal particles (10% by weight) were added to the oil-water emulsion and agitated at 2000 rpm for another 3 minutes. Agitation speed was decreased to 1300 rpm and agglomeration was completed after 2 minutes. The slurry was screened from a 300 μm sieve to separate the agglomerates produced.

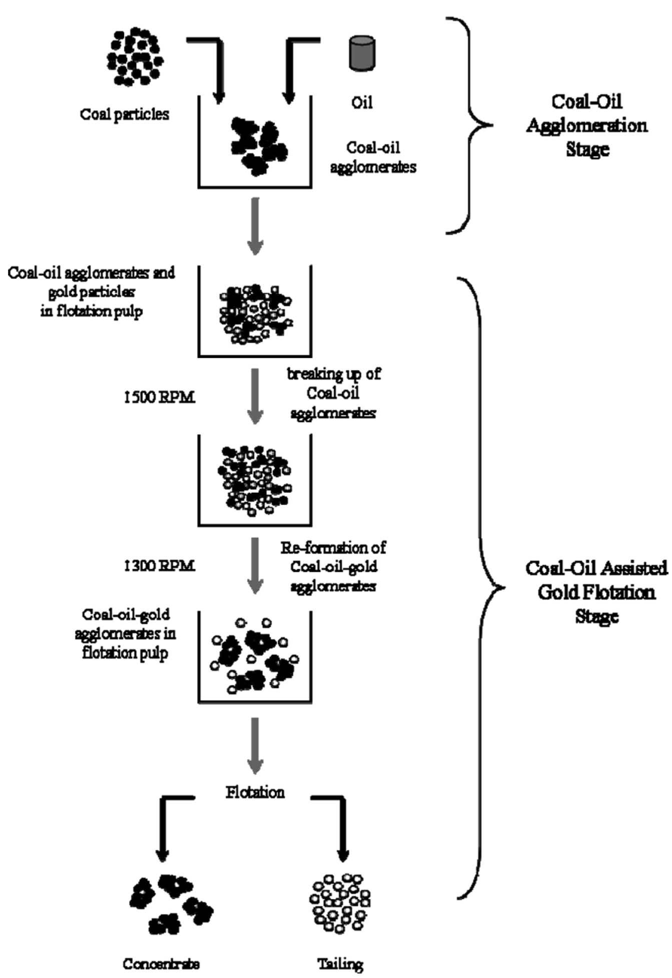

FIG. 3. Schematic representation of the study.

Coal-Oil Assisted Gold Flotation

Outokumpu flotation machine with speed and aeration rate control was used for coal-oil assisted gold flotation. A standard procedure was followed in the flotation tests which were carried out at a solid/liquid ratio of 20%. Flotation reagents (400 g/t Na_2SiO_3, 50 g/t KAX, 50 g/t, Aerofloat 208, and 50 g/t Aerophine 3418A) were added to the pulp which was conditioned for ten minutes at 1500 RPM stirring speed. Previously agglomerated coal particles were added to the gold bearing pulp and agitated at 1500 rpm for 10 minutes. Stirring speed was decreased to 1300 RPM and pulp was further conditioned for 5 minutes for coal-oil-gold agglomerate re-formation. Coal-oil-gold agglomerates obtained were separated from the pulp by flotation (Fig. 3).

Analysis of the Agglomerates

The floated and collected agglomerates were burned in a furnace at 900°C for the removal of carbonaceous and volatile matter. The remaining material was a mixture of ash and gold particles. This mixture was treated with aqua regia to dissolve the gold. The gold assay was determined using an Anayltik JenaAG novAA 330 Atomic Absorption Spectrometer.

RESULTS AND DISCUSSION

The Effect of the Oil Type on Coal-Oil Agglomeration

The analysis revealed that the vegetable oils have similar properties. The specific gravity values of the oils are between 0.919 g/cm^3 and 0.947 cm^3. Petroleum oils are slightly lighter than vegetable oils, with specific gravities in the range 0.846 cm^3 and 0.811 cm^3. Viscosity values of the oils are varied between 49.5 cP and 68.5 cP for crude vegetable oils. Petroleum oils have very low viscosity values (4.16 cP and 1.47 cP, Table 4). It is claimed that more viscous and heavier oils are less efficient agglomerants (23). Interfacial tension values of the petroleum oils are higher than vegetable oils. Lower interfacial tensions of liquids generally provide better conditions for emulsification, due to decreased interfacial energies.

Free energy changes indicating the probability of coal-oil attachment (ΔG_{attach}) and the spreading of oil over the coal particles (ΔG_{spread}), were determined using the data obtained from the contact angle and surface tension measurements. The negative values of ΔG_{attach} in the Table 5 imply that particle-oil attachment is likely to take place (23). ΔG_{spread} is always a positive number, suggesting that the spreading of the oil into a thin film at the

TABLE 5
Free energy changes of the oils for spreading and attachment processes

Oils name	$S = -\gamma_{ow} \cdot (\cos\theta_{oil} + 1)$	$\Delta G_{attach} = -W_{ows} = \gamma_{ow} \cdot (1 + \cos\theta_{oil})$
Corn oil	1.76	−6.24
Cotton oil	3.09	−10.91
Olive oil	5.26	−21.14
Sunflower oil	6.46	−27.14
Diesel oil	5.77	−33.63
Kerosene	6.02	−53.78

coal/water interface is thermodynamically unlikely (23). In practice, however, the remaining oil on the coal surface in the form of droplets after the attachment process may still cause agglomeration. The calculated free energy changes of ΔG_{spread} and ΔG_{attach} are very close for vegetable oils. Sunflower and olive oil possess higher interfacial tension values resulting in better performance in coal-oil agglomeration process. Petroleum oils have lower viscosity values and higher interfacial tension. Their calculated free energy changes for attachment process are much lower than vegetable oils.

Coal-oil agglomeration tests were conducted by using 4% of coal/oil ratio and $-140\,\mu m$ (d_{80}) coal particles. Produced agglomerates were screened from a 300 μm sieve to separate them from the slurry. Yield percentage and EI (efficiency index) terms were used to determine the success of the coal-oil agglomeration process (Fig. 4). As expected, the oils having high interfacial tension values provided better agglomeration performance.

After the coal-oil agglomeration tests were completed, different oils at 0.25% oil/ore ratio were used to determine the best agglomerating oil for the coal-oil assisted gold flotation. An agglomerate/ore ratio of 12.5% was kept constant during these tests, and coal particles ground below 140 μm (d_{80}) were used as agglomerating material. The results are given in Figs. 4 and 5.

The efficiency index, yield, and gold recovery of the coal-oil assisted gold flotation are plotted against oil/water interfacial tension and shown in Fig. 4. As can be seen there is a relationship between these three variables and interfacial tension—oils having higher interfacial tension are more effective in aggregation and flotation processes.

Olive and sunflower oils were determined to be the best crude vegetable oils, resulting in concentrates of 152 g/t Au and 165 g/t Au respectively, for coal-oil assisted gold flotation. The gold recoveries were also very similar (75% and 76%). Owing to the above-mentioned properties, petroleum oils give better results both in coal-oil agglomeration and coal-oil assisted gold flotation processes. A concentrate assaying 109 g/t Au was obtained with 82% gold recovery by using kerosene. Diesel oil was more selective than kerosene, the concentrate gold grade was determined as 152 g/t with a recovery of 78%.

The Effect of Oil/Ore Ratio

The effect of changing the amount of oil in the coal-oil agglomeration stage on gold recovery was investigated in

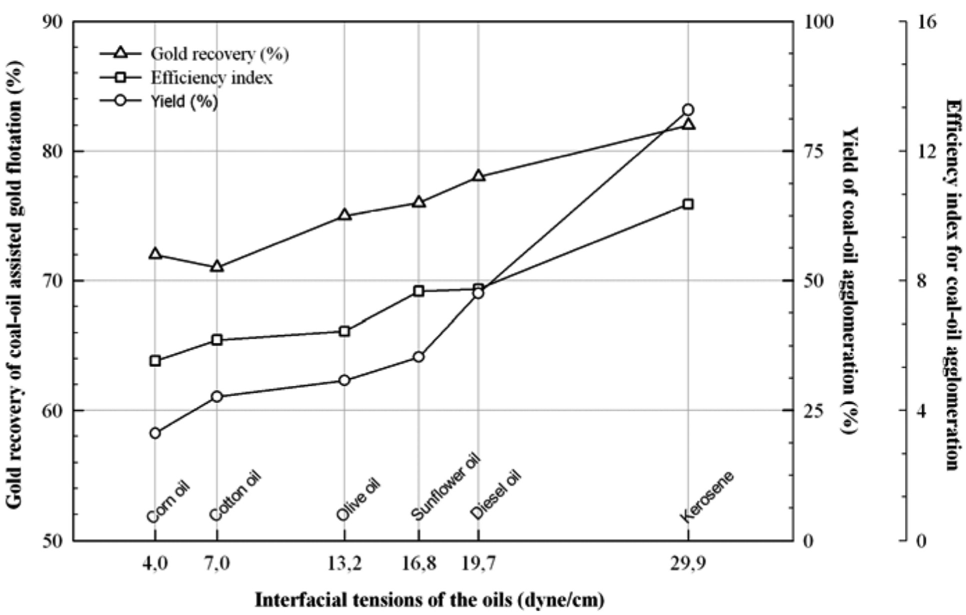

FIG. 4. The effect of the use of different oils on the gold recovery.

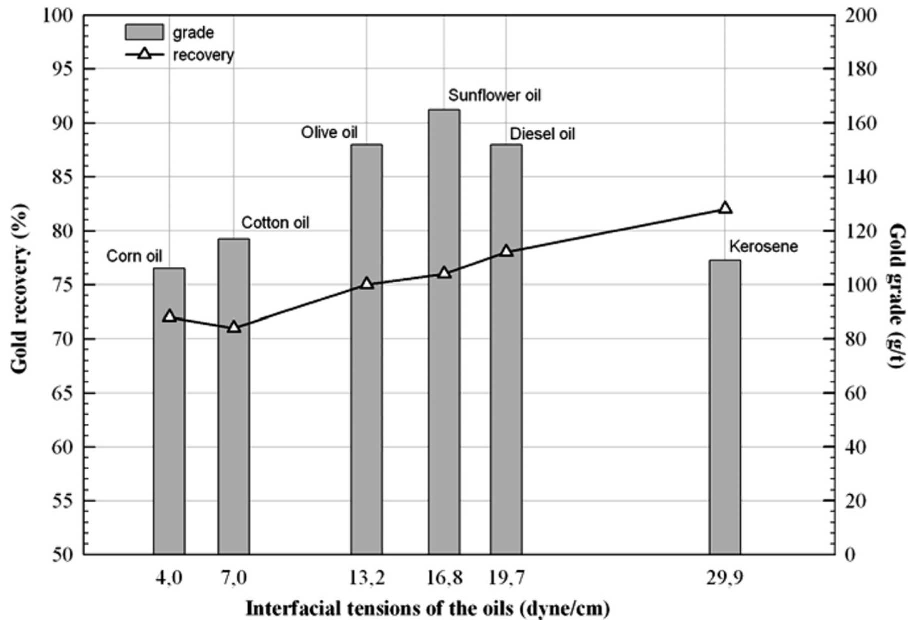

FIG. 5. The relationship between the interfacial tension of the oils and gold recovery-efficiency index.

this set of experiments. The amount of coal was kept constant at 6% of the gold ore. Tests were carried out using different amount of diesel oil in each experiment. Coal particles ground below 140 µm (d_{80}) were used as agglomerating material. The results are given in Fig. 6.

The formation of the agglomerates can be varied from discrete lens-shaped rings to spherical agglomerates according to the amount of available oil in the agglomeration system (20). Micro agglomerates were generated at low oil concentrations. The specific surface area of these micro agglomerates increases, resulting in agglomerates recovering more gold particles. On the other hand, vulnerability against gangue particle penetration increases eventually, and the larger agglomerate surface area causes fine gangue entrainment into the agglomerates.

According to the mathematical model developed by Petela (54) to calculate the final size of the coal-oil agglomerates, the diameter of agglomerates increases

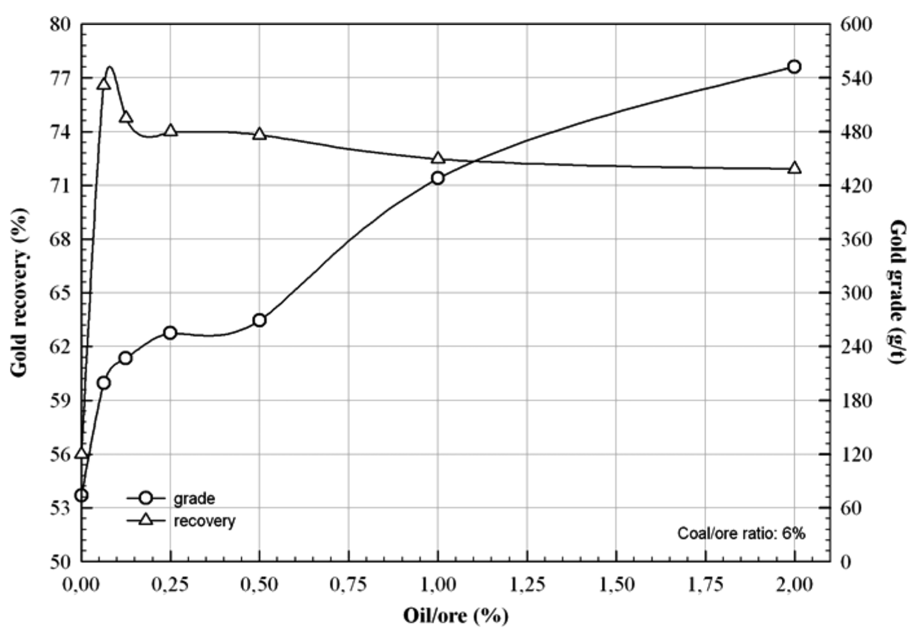

FIG. 6. The effect of oil-ore ratio on the gold recovery.

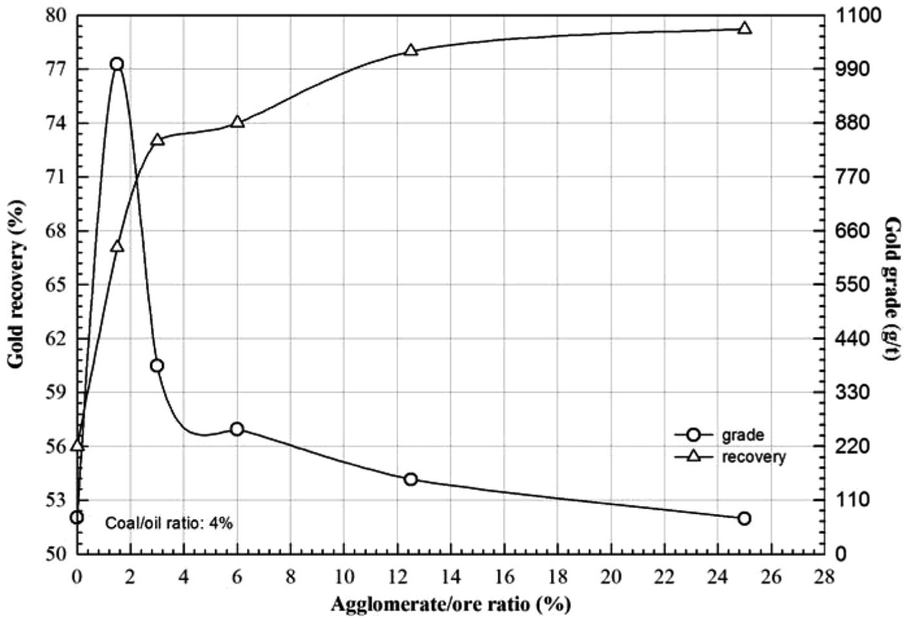

FIG. 7. The effect of agglomerate-ore ratio on the gold recovery.

continuously as the amount of oil is raised. Similarly using higher amount of oil in the coal-oil agglomeration has not produced fresh agglomerates in our tests; the size and integrity of the agglomerates increased, shape of the agglomerates has become more spherical. The structure was more compacted, and penetration into the agglomerates was more difficult for gangue particles as well as some fine gold particles during the flotation process. As a result, slightly lower gold recoveries with high concentrate grades were obtained. Overall results showed that the addition of coal-oil agglomerates into the flotation cell improved the gold grade and recoveries of the gold concentrate which were determined as 76 g/t and 56% respectively for conventional gold flotation (Fig. 6).

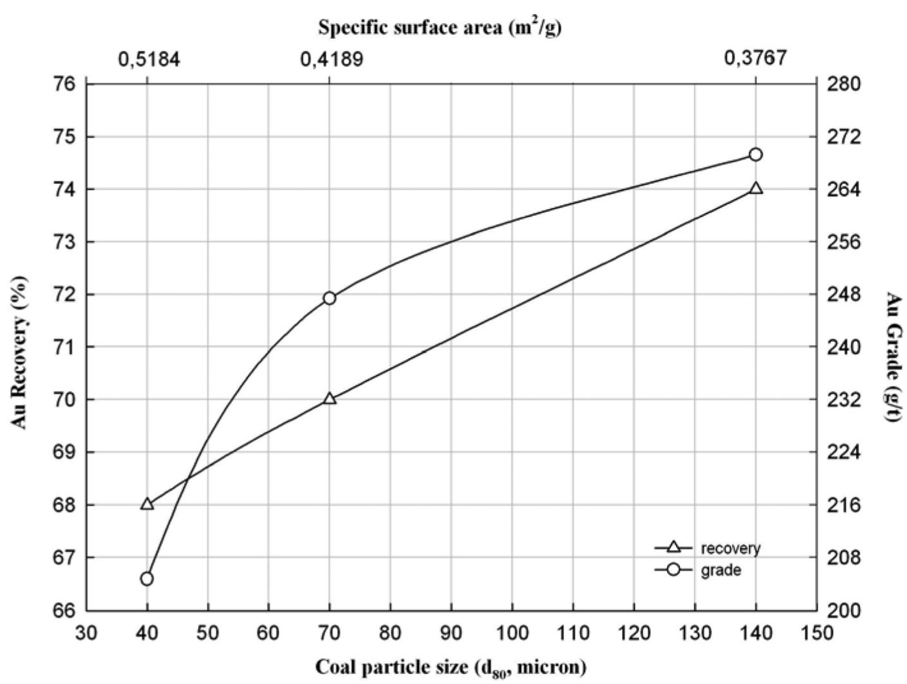

FIG. 8. The effect of coal particle size-specific surface area on gold recovery.

The Effect of Agglomerate/Ore Ratio

Coal-oil agglomerates were produced using coal particles ground below 140 μm (d_{80}) and diesel oil. Coal/oil ratio was kept constant at 4%. The effect of changing the amount of agglomerates on the grade and the recovery of gold concentrates was investigated. The results of these experiments are presented in Fig. 7. The results demonstrate that higher gold recoveries can be observed with higher agglomerate/ore ratios due to the availability of larger agglomerate surface area for gold particle penetration. Accordingly, sensitivity of the process against the gangue particles is decreased, resulting in lower grades of gold concentrates. A concentrate assaying 72 g/t Au with 79% gold recovery was produced using the highest agglomerate/ore ratio, while a concentrate assaying 76 g/t Au with 56% recovery was obtained by using conventional flotation.

The Effect Coal Particle Size

Coal samples were ground into different size fractions prior to experiments to investigate the effect of coal particle size on the process efficiency. 0.25% oil/ore and 6% agglomerate/ore ratios were kept constant during the tests. Figure 8 shows the effect of coal particle size on gold recovery and grade values of the flotation concentrates. Both recovery and concentrate grade increase with coal particle size.

The size of the agglomeration material in oil agglomeration is one of the important parameters determining the amount of oil on each particle. Since the consumption of oil was kept constant in the tests, the amount of oil carried by each particle was lower for finer materials due to the increased specific surface area. Coarser particles carry a higher amount of oil on their surface. For this reason, re-formation of coal-oil-gold agglomerates from coarser coal particles at mild agitation conditions is more pronounced in the flotation cell resulting increased grade and recoveries.

CONCLUSIONS

Our study demonstrates that petroleum oils are more effective than vegetable oils in both coal-oil agglomeration and coal-oil assisted gold flotation processes. However, low cost and environmental friendly characteristics of the crude vegetable oils make them potential bridging liquids for assisting gold flotation.

Increasing the amount of oil used in coal-oil agglomeration decreased the number of the agglomerates in the experiments, more compact and spherical agglomerates were produced. As a result, gold recovery decreased slightly but the gold grade of the flotation concentrates increased when these agglomerates employed in gold flotation. Gold grade of the flotation concentrates tends to decrease when the amount of agglomerates employed in gold flotation increases. Conversely, the use of higher agglomerate amounts in gold flotation enhances the gold recovery by providing a larger surface area for gold particle penetration. The higher amount of oil on coarser particles helps coal-oil-gold agglomerate reformation in the flotation cell, resulting in increased grade and recovery.

Finally, the process is found to be effective for recovering gold from an ore containing very fine gold particles (about 70% of the gold content is below 25 μm) with considerable grade and recovery values.

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