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Effect of nanobubbles on flotation of El-Maghara coal

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ABSTRACT

To extensively explore the advantage of using nanobubbles in the El-Maghara coal flotation process, the effect of nanobubbles on both column and mechanical flotation was investigated under different operating parameters such as diesel oil collector dosage, MIBC frother concentration, superficial feed velocity, superficial air velocity, and superficial wash water velocity in column flotation; besides the slurry flow rate through nanobubbles generator into a 25-liter mechanical flotation cell. The representative coal flotation feed acquired from the El-Maghara deposit located in Sinai, Egypt with chemical characterization using proximate analysis containing 25.27% mineral matter forming ash during coal combustion and with particle size distribution measurement using laser particle size analyzer is 57 μ m d₉₀. Also, the flotation kinetic experiments were done to show the influence of nanobubbles on the flotation time required to obtain high-quality coal products with high combustible recovery. Nanobubbles enhanced the flotation performance and kinetics by up to 24% combustible recovery based on the operation parameters reducing flotation time from 4 to 2.25 min for 80% combustible recovery.

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Introduction

Coal is a heterogeneous, non-crystalline, complex, and macromolecule compound containing mineral matter. It is the most abundant fossil fuel available accounting for 55% of the electricity production worldwide (Mostafa et al. 2017). It mainly contributed to the economic growth of many countries as a main source of energy (Ramudzwagi, Tshiongo-Makgwe, and Nheta 2020). Coal has many other applications in a variety of fields such as steel industries, polymers, dyes, aluminum refining, advanced carbon nanotubes, carbon electrodes, carbon fibers, etc (Mostafa et al. 2017). However, run-of-mine coal usually contains a high amount of mineral matter, so cleaning coal prior to its utilization is essential to achieve clean products that have high values and to minimize environmental impacts due to impurities (Mostafa et al. 2017). In coal flotation, collectors and frothers are utilized to minimize the mineral matter content in the final product. Diesel oil or kerosene is used as a collector to increase the hydrophobicity of combustible materials, whereas aliphatic alcohol, for example, methyl isobutyl carbinol (MIBC) or glycol is utilized as frother (Kumar and Kumar 2018).

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Nanobubble technology has shown a significant enhancement to the separation performance in froth flotation that exploits the difference in surface hydrophobicity of different particles (Ahmadi et al. 2014; Ahmed 2013a; Calgaroto, Azevedo, and Rubio 2015; Calgaroto, Wilberg, and Rubio 2014; Ma et al. 2022; Sobhy and Tao 2013a, 2013b; Sobhy, Wu, and Tao 2021; Tao 2022; Tao and Sobhy 2019; Tao, Wu, and Sobhy 2021; Zhou et al. 1997). In froth flotation, particle aggregation by capillary effect (Knüpfer, Ditscherlein, and Peuker 2017) and hydrophobization by increasing the contact angle are enhanced by the presence of nanobubbles, which in consequence maximizes the recovery of valuable minerals. In contrast, it was found that the flotation with nanobubble alone was not attained, which was expected because of the low lifting power of the nanobubbles and their insignificant buoyancy in water (Calgaroto, Wilberg, and Rubio 2014). Furthermore, nanobubbles are adsorbed and/or nucleated on the surface of hydrophobic particles and serve as nuclei for regular flotation bubbles attachment (Azevedo et al. 2016; Calgaroto, Azevedo, and Rubio 2015) which carry the hydrophobic particles to the product stream. Thus, combining nanobubble technology with conventional flotation is considered an innovative and promising technique (Calgaroto, Wilberg, and Rubio 2014; Ma et al. 2022; Sobhy and Tao 2013b; 2013b; Tao and Sobhy 2019).

When nanobubbles were integrated into mechanical flotation cells, fine and ultrafine quartz recovery improved by 20-30% as a result of increasing quartz contact angle and agglomeration of ultrafine quartz (Calgaroto, Azevedo, and Rubio 2015), and fine/ ultrafine chalcopyrite recovery enhanced by 16-21% (Ahmadi et al. 2014). While integrating nanobubble into column flotation of fine coal particles enhanced the coal combustible recovery by 50% (Ahmed 2013a; Sobhy and Tao 2013a); and reduced collector, frother, and air consumptions (Ahmed 2013a; Ma, Tao, and Tao 2019). Recent studies showed that nanobubbles significantly improved the mineral flotation efficiency, kinetic, and enrichment ratio; and minimized chemical reagent consumption (Ahmed 2013a; Calgaroto, Azevedo, and Rubio 2015). Nanobubbles formed on hydrophobic coal particle surfaces remain attached while those on a hydrophilic particle are detached, which is a selective process that enhances flotation separation efficiency. Thus, in the presence of nanobubbles, hydrophobic particles have a higher collision probability with regular flotation bubbles, a higher attachment probability, and a lower detachment probability, resulting in a greater flotation rate constant and flotation recovery (Ahmed 2013a; Ding et al. 2020). Nanobubbles formed selectively on the particle surface coalesce with the macroscopic air bubbles, enhancing the pinning action of the three-phase contact line and advancing contact angle, and in consequence improve the capillary force between bubbles and particles (Ding et al. 2020).

Some of the flotation studies of El-Maghara coal were done previously. For example, the flotation kinetics of El-Maghara coal samples were investigated using various statistical procedures and models at different conditions such as collector dosages, frother concentrations, and agitation speeds (Ahmed 2013b). The recovery of fine coal particles by flotation from the tailings stream produced from the El-Maghara coal-washing plant was investigated reaching 80% in addition to unitizing the residual clay in producing building bricks (Ramadan, Saleh, and Moharam 2017).

Furthermore, due to the importance of coal as a source of energy, the main objective of this study was to produce high-quality coal from El-Maghara coal samples and to investigate the effects of nanobubble technology on the individual operation parameters

of column flotation. Also, nanobubbles technology was integrated into a mechanical flotation system to illustrate the effect of nanobubbles on mechanical flotation performance. The performance was estimated by measuring ash percentage and combustible recovery.

Material and Methods

Coal Sample

A 50 kg of representation coal sample was acquired from the El-Maghara coal mine located in Sinai, Egypt. At the laboratory, the sample was primarily crushed using a jaw crusher, secondarily crushed using a roll crusher, and ground using a dry ball mill to reach the liberation size of finer than 75 microns. Then the ground sample was immediately mixed thoroughly and homogenized, divided into small portions, and sealed in plastic bags for later usage.

2.2. Flotation reagents

Pure chemical reagents such as methyl isobutyl carbinol (MIBC) as a frother and diesel oil as a collector were purchased from Sigma-Aldrich.

2.3. Sample characterization

A representative sample was subjected to a detailed characterization study of proximate and size analysis.

Proximate analysis was conducted using a standard methodology of the ground coal sample to determine the moisture, fixed carbon, volatile matter, and ash percentages which were 1.5%, 51.03%, 22.20%, and 25.27% respectively.

The laser particle size analyzer was conducted to quantify the size distribution of the ground coal sample. From Fig. 1, the average d_{95} , d_{90} , and d_{50} were 70 µm, 57 µm, and 24 µm respectively.

Flotation Release Analysis

The flotation release analysis is a method used to attain the best possible separation efficiency achievable by any froth flotation process which is analogous to the gravitybased washability analysis. The release analysis was carried out in a conventional 1.5-liter flotation cell and its data was used as a baseline for efficiency evaluation of the nanobubble flotation technology on the flotation process of coal. As illustrated in Fig. 2, the first stage of the release analysis separates the hydrophobic material away from the hydrophilic material by performing multiple cleaning steps with the original feed. The second stage has the goal of separating particles into fractions of different degrees of surface hydrophobicity by controlling air flow rate and impeller rotation speed under starvation reagent conditions.



Figure 1. Size distribution of coal sample using laser particle size analyzer.



Figure 2. Flotation release analysis method of coal sample.

Flotation Kinetic Test

Kinetic flotation tests were conducted in a conventional 1.5-liter flotation cell in the presence and absence of nanobubbles and the kinetic flotation rates were calculated to show the effect of nanobubbles on flotation rate constants. The 5% solids coal slurry was conditioned with diesel oil and MIBC, and with flotation, the products were collected after 15, 30, 45, 60, 120, 240, and 480 s. The products were measured for their weight and ash percentage from which the recovery was calculated.



Figure 3. Flotation column integrated with nanobubbles technology.

Column Flotation Test

The column flotation experiments were investigated using the 5 cm diameter flotation column integrated with a nanobubbles generator to show the usefulness of the nanobubble-enhanced flotation process on coal cleaning shown in Fig. 3.

Lab nanobubble flotation column made of Plexiglas with 2.4 m adjustable height was featured with nanobubbles generator venturi tube and microbubbles generator static mixer. The typical lengths of collection and froth zones used in the tests were 210 cm and 30 cm, respectively. With a diameter of 5.08 cm, the length-to-diameter ratio of the column was around 41:1, which provided near plug-flow conditions.

Wash water was installed in the froth zone at a depth of 1/3 of the froth zone height below the overflow edge. Frother was fed into the feed stream while the air was inserted into the slurry prior to the static mixer and venturi tube. Feed slurry entered the column in the upper pulp zone, 45 cm below the overflow edge. After being fed into the column, coal particles collected by rising bubbles ascend to the top. Those that settle to the bottom of the column is pumped through the static mixer and the venturi tube to have more chances for recovery. The slurry jet comes out of the neck of the Venturi cavitation tube at a speed of 6 to 10 m/s causing hydrodynamic cavitation in the slurry jet enters the column tangentially at the bottom.

The total recycling flow rate through the static mixer and venturi tube is 12 L/min, which splits at a two-way connector into the static mixer and cavitation tube with a splitting ratio of 4:6 respectively.

A microprocessor (Series 2600 Love Controls) receives signals from a pressure transducer located at the bottom of the column. The signal adjusts a Miniflex pinch valve that controls the underflow flow rate and the desired froth level.

Before each test, the feed slurry was conditioned for 5 min with diesel oil used as a collector to enhance the hydrophobicity of coal particle surfaces. Conditioning was conducted in a sump that was equipped with a mixer and four baffles placed vertically and separated by an equal distance along the circumference of the sump. The slurry was fed from a feed tank, which utilized a recirculating line to ensure suspension of all solids, to the flotation column by a peristaltic pump at a pre-determined rate.

Unless otherwise specified, all column flotation tests will be performed under the following conditions: Superficial feed rate: 0.85 cm/s.; superficial gas flow rate: 1.25 cm/s.; superficial wash water rate: 0.064 cm/s; diesel oil: 0.2 kg/t; MIBC frother: 15 ppm; froth depth: 30 cm; and feed slurry solids concentration: 5%.

A period of time equivalent to three particle retention times was allowed to achieve steady-state conditions. After reaching the steady state, samples of feed, product, and tailing streams were collected simultaneously. These samples were filtered, dried, weighed, and analyzed for ash percentage. Flotation performance was evaluated in terms of combustible recovery.

Major process parameters were examined individually to investigate their effects on flotation recovery and concentrate ash percentage with and without the venturi tube. They included collector dosage, frother concentration, superficial feed velocity, superficial air velocity, and superficial wash water velocity.

Mechanical Flotation Test

To show the benefits of retrofitting existing mechanical flotation by adding a nanobubble generator device, a flotation experiment was performed with a 25-liter mechanical flotation cell according to the setup shown in Fig. 4.



Figure 4. Conventional flotation system of coal slurry integrated with nanobubble technology.

The examined major operating variable was the flow rate from 0 to 12 L/min to the nanobubbles (NBs) generator of 3 mm inner throat diameter and 12 mm inner pipe diameter. During the experiment, the slurry was conditioned with 0.2 kg/t diesel oil for 15 min, and it circulated from and back to the conditioning tank with a flow rate of 12 L/min. At different flow rates through the nanobubbles generators, a portion of the slurry of approximately 1.5 L/min was fed to the flotation cell as a continuous flotation process. A period of time equivalent to three particle retention times was allowed to achieve steady-state conditions. After reaching the steady state, samples of feed, product, and tailing streams were collected simultaneously. These samples were filtered, dried, weighed, and analyzed for ash percentages. Flotation performance was evaluated in terms of combustible recovery. In addition, the mechanical flotation performance data were compared to the release analysis data to show the influence of the nanobubbles on the flotation performance.

Results and Discussion

Flotation Release Analysis

The release analysis method is used to characterize the coal response to the flotation process. The release analysis results shown in Fig. 5 indicate the ultimate separation performance of the El-Maghara coal sample that can be obtained by froth flotation. For example, for 6% ash coal product, the maximum recovery was 79% with 85.4 ash rejection.

Effect of Nanobubble on Flotation Kinetic Rate

Flotation rate experiments show how fast the product reports to the concentrate stream as shown in Fig. 6. The kinetics of the flotation in the presence of nanobubbles was faster than that in the absence of nanobubbles by 44.27%. With nanobubbles, the flotation rate constant increased from 0.3897 min^{-1} to 0.5621 min^{-1} in the first 2 min then from 0.1443 min^{-1} to 0.2035 min^{-1} in the following 6 min. This was attributed to the enhanced collision and attachment probabilities and the minimized detachment probability of the hydrophobic



Figure 5. Flotation release analysis of tested El-Maghara coal sample.



Figure 6. Flotation rate tests in the presence and absence of nanobubbles.



Figure 7. Ash percentage and combustible recovery as a function of flotation time in the presence and absence of nanobubbles.

coal particles to the regular flotation bubbles (Tao and Sobhy 2019) as well as increasing the froth stability in the presence of nanobubbles (Sobhy and Tao 2018).

For example, as shown in Fig. 7, 80% combustible recovery was obtained in 2.25 min in the presence of nanobubbles, and 4 min in the absence of nanobubbles with keeping the product quality at a value of less than 10% ash. Thus, nanobubbles enhance the production rate and in consequence the device capacity and production rate.

Effect of Nanobubbles on Coal Column Flotation

The column flotation was used to investigate the different parameters in the presence and absence of nanobubbles to show the significant influence of nanobubbles on the flotation



Figure 8. Effect of collector dosage in the presence and absence of nanobubbles.

performance as indicated in Figs. 8, Figs. 9, 10, Fig. 11, and Fig. 12. The major process parameters were collector dosage, frother concentration, superficial feed velocity, superficial air velocity, and superficial wash water velocity. In most cases, there was an increase in the combustible recovery by up to 25% with maintaining the product ash percentage at the same value or with a slightly higher value. The collector dosage had a positive influence on the combustible recovery by 24.22% and 20.35% in the presence and absence of nanobubbles respectively, and nanobubbles had an additional positive impact by 12% with keeping the product ash percentage at about 10% (Fig. 8).

The frother is mainly used to strengthen the stability of the air bubbles by reducing the liquid surface tension. The concentration of frother required for flotation is varying from one mineral to another especially if the collector possesses some frothing action besides its collecting action. Frother concentration had a significant positive impact on the combustible recovery with a slight increase in the concentrate ash



Figure 9. Effect of frother concentration in the presence and absence of nanobubbles.



Figure 10. Effect of superficial feed velocity in the presence and absence of nanobubbles.



Figure 11. Effect of superficial air velocity in the presence and absence of nanobubbles.

percentages as shown in Fig. 9. Increasing the frother concentration from 15 ppm to 60 ppm increased the combustible recovery from 54.42% and 60.36% to 77.83% and 86.16% in the absence and presence of nanobubbles respectively. In addition, the presence of nanobubbles enhanced the combustible recovery by up to 20%. Whereas, the product ash percentage was higher by less than 10% by increasing the frother concentration from 15 ppm to 60 ppm (Fig. 9).

The influence of the slurry feed rate on the flotation system was negative. Increasing the feed rate from 0.2 cm/s to 1.2 cm/s significantly reduced the combustible recovery from 58.37% and 79.90% to 44.45% and 48.16% as well as the product quality that was slightly reduced by increasing the ash percentages from 8.8% and 9.2% to 9.9% and 9.7% in the absence and presence of nanobubbles respectively (Fig. 10).

Figure 11 shows that the product ash percentage and combustible recovery were enlarged by increasing the air flow rate. Furthermore, the increase in the airflow rate



Figure 12. Effect of superficial wash water velocity in the presence and absence of nanobubbles.

from 0.4 cm/s to 1.6 cm/s increased the combustible recovery from 78.69% and 60.80% to 91.37% and 71.76% in the presence and absence of nanobubbles respectively. Thus, nanobubbles enhanced the combustible recovery by up to 23%. This increase in concentrate combustible recovery and inconsequence the concentrate flowrate was at the expense of an increase in the product ash percentage from 9.2% and 8.8% to 11% and 10.3% in the presence and absence of nanobubbles respectively.

The wash water is usually used to clean the froth zone. Thus, it had a positive impact on the concentrate quality by reducing the ash percentages by 2.7% and 2.3% in the presence and absence of nanobubbles respectively. This caused a negative influence on the combustible recovery by 29% and 12% in the presence and absence of nanobubbles respectively (Fig. 12).



Figure 13. Effect of flow rate through nanobubbles generator on mechanical flotation performance.



Figure 14. Comparing mechanical flotation experiments in the presence and absence of NBs with release analysis curve.

Effect of Nanobubbles on Coal Mechanical Flotation

Mechanical flotation experiments performed with a 25-liter cell showed the benefits of adding nanobubbles to the system. The slurry flow rate to the nanobubbles generator was the major examined operating parameter, and the results are shown in Fig. 13. Figure 13 indicates that with nanobubbles, a high-quality product of equal to or less than 9% ash was produced at a higher combustible recovery by 10-15%. It was demonstrated that increasing the slurry flow rate into the nanobubbles generator reduces the nanobubbles size (Zhang et al. 2020). Besides, flotation kinetic rate experiments showed that the product reports to the concentrate stream were faster in the presence of nanobubbles.

The flotation performance of mechanical flotation data compared to the release analysis data shown in Fig. 14 confirms that nanobubbles enhance the flotation performance where in the presence of nanobubbles, the points are closer to the release analysis curve than that in the absence of nanobubbles.

Conclusion

Based on the above results of column flotation, diesel oil collector, MIBC frother concentration, and superficial air velocity positively impacted combustible recovery by up to 24%, 26%, and 13%; whereas nanobubbles showed an additional improvement by up to 12%, 20, and 23% respectively. In contrast, superficial feed velocity and superficial wash water velocity negatively influenced the combustible recovery by 32% and 29% respectively, but again nanobubbles enhanced the recovery by 24% and 23% respectively. Besides, nanobubbles maintained the product ash at approximately the same value where in all cases the ash percentage was reduced from 25.27% (feed grade) to less than 11.5% ash in the concentrate. In addition, nanobubble significantly enhanced the flotation kinetics by 44.26% by increasing the differential flotation rate between valuable and gangue minerals which reduced the flotation time from 4 to 2.25 min to produce 10% ash with 80% recovery. Integrating nanobubbles into a 25-liter mechanical flotation cell by using different slurry flow rates through a nanobubbles generator significantly enhanced the combustible recovery by 10– 15% with maintaining the ash percentage at a value less than 9% in the concentrates.

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