



Relationship between properties of oil/water emulsion and agglomeration of carbonate minerals

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ABSTRACT

In this study, a modification of oil agglomeration processes of dolomite and magnesite particles has been proposed. In this method the emulsion of kerosene with dodecylammonium hydrochloride (DDAHL) was added to the minerals suspension. The experimental results have demonstrated that the agglomeration was governed by the properties of oil/water interface such as zeta potential and interfacial tension ($\gamma_{o/w}$). Addition of cationic surfactant to the emulsion changed the zeta potential value from negative to positive, which resulted in enhanced the adhesion of the oil droplets to negatively charged particles, according to the DLVO theory. The presence of cationic surfactant has an effect on the size of the agglomerate. At the high DDAHL concentration (21 mg/g_{solid}) the recovery and diameter of agglomerates slightly decreased, and finally a paste was formed. It results from decrease of the value of $\gamma_{o/w}$ leading to the formation of weaker liquid bridges between solid particles. This explanation was supported by AFM measurements of adhesion forces.

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1. Introduction

It has been known that during mineral rock processing, a large amount of slime is produced [1]. However, the slime fraction often contains significant quantities of valuable minerals. Therefore, an economically feasible process of minerals recovery from the slime is of great importance [2,3]. A method of recovering these particles from suspension is to increase their size by selective flocculation and then aggregate flotation. Another way may be a selective oil agglomeration of fine particles [2]. Oil agglomeration is a size enlargement method that facilitates the separation operations of solid processing (filtration, flotation, sedimentation). In this process, hydrophobic particles are agglomerated in an aqueous medium by bridging liquid which is immiscible with water and wets the particle surface, whereas, the hydrophilic particles remain in the suspension [4]. The agglomerates of sufficient size can be easily separated from the medium by flotation, sedimentation or screening. The main advantages making this method interesting for industrial applications (e.g. pharmaceuticals, pigments, and pesticides) are: high selectivity, a possibility of fine particles aggregation (below 5 μm) and simple equipment [5–7]. However, the relatively high cost of oil makes this particular method impossi-

ble to use in industry on a large scale. It has been shown, that the quantity of bridging liquid required for sufficient agglomeration of calcite, barite, and coal was 180 kg/tonne, 300 kg/tonne and 128 kg/tonne, respectively [1,8,9]. Therefore, extensive studies on a lowering of oil amount required during the process have carried out for many years [10,11]. From a thermodynamical point of view, the oil agglomeration process follows a decrease of interfacial tension, and it should require a lower consumption of oil, comparing to, e.g. flotation. This can be achieved by introducing the additional components adsorbing onto oil/water interface and changing its properties, such as surfactant or *n*-alcohols, which adsorbs onto [10–12]. A modification of oil agglomeration process proposed in this work, is based on introduction of oil/water emulsion with cationic surfactant to the hydrophilic particles suspended in sodium oleate solution, in a form of emulsion with cationic surfactant. In such a system, the mixture of anionic–cationic surfactants could be expected to exist. The oil/water emulsion behaviour in the presence of ionic surfactants has been described quite well in literature [13,14]. It has been known that the mixture of surfactants has a significantly higher surface activity than its individual components [15]. Thus, the application of anionic–cationic surfactants mixture in oil agglomeration can enhance the efficiency of the agglomeration process.

The aim of this work is to investigate the influence of electrochemical properties of oil/water interface on oil agglomeration process recovery and on the agglomerates size, in the presence of the mixture of anionic and cationic surfactants.

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2. Methods and materials

The experiments were carried out on dolomite and magnesite, brought from a dolomite quarry 'Kletno' and from a magnesite mine 'Grochow' (Lower Silesia, Poland), respectively. The mineral samples were ground and classified. The $-45\ \mu\text{m}$ class was used in the oil agglomeration experiments. The particle size distribution measurements were carried out by using a Mastersizer2000 laser diffractometer, equipped with a HydroMu dispersion unit (Malvern Instruments), and showed that the particle diameter was about $17\ \mu\text{m}$ (dolomite) and $6\ \mu\text{m}$ (magnesite MG). The densities of these materials were $2.77\ \text{g/cm}^3$ and $2.60\ \text{g/cm}^3$ for dolomite and magnesite, respectively. The X-ray diffraction analysis of dolomite showed the insignificant amounts of the following contaminants: quartz and calcite. It has also shown that the magnesite sample (MG) was a polymineral system containing traces of minerals like talc, clinocllore, tremolite, and quartz. The BET specific surface area was determined with FlowSorbII 2300 (Micromeritics) and was found to be 1.92 and $15.4\ \text{m}^2/\text{g}$ for dolomite and magnesite MG, respectively.

Magnesite samples A, B, C, D were produced by a mixing of magnesite fraction $125\text{--}250\ \mu\text{m}$ with a magnesite MG in following ratios: 5, 10, 15, 20, 40% (w/w). The particle size distribution measurements were carried out with Mastersizer2000 laser diffractometer, equipped with a HydroMu dispersion unit (Malvern Instruments), and showed that the particle diameters of samples A, B, C, D were approximately 6.7 , 7.8 , 8.4 and $11.1\ \mu\text{m}$, respectively.

Sodium oleate (SOI) was purchased from J.T. Baker Chemical Co. Dodecylammonium hydrochloride (DDAHCl) was purchased from POCh. All reagents were used without further purification.

The oil agglomeration experiments were carried out in a plastic baker using an overhead stirrer with the marine type impeller (IKA yellow line, OST basic). In each experiment, $5\ \text{g}$ of mineral powder was mixed with $100\ \text{ml}$ of anionic surfactant solution (NaOl) and pH of the suspension was adjusted to appropriate value in the range of $\text{pH}\ 7\text{--}12$. After $24\ \text{h}$, $100\ \text{ml}$ of freshly prepared oil/water emulsion was poured into suspension and the resulting mixture was mixed for $10\ \text{min}$ at $2000\ \text{rpm}$. Emulsion was prepared by mixing kerosene (0.1 and $0.4\ \text{ml/g}_{\text{solid}}$ for dolomite and magnesite, respectively) with cationic surfactant (DDAHCl) for $30\ \text{s}$ using an ultrasonic homogenizer (HD 2070, Bandelin). After the agglomeration process had been completed, agglomerates were screened on a $125\ \mu\text{m}$ sieve and dried in the oven. Each experiment was repeated five times and the results were averaged.

The interfacial tension of oil/water system was determined with K100 tensiometer (Krüss), as a function of DDAHCl concentration. The results were averaged over four independent sets of measurements.

Electrophoretic mobility of particles and droplets was measured with a Zeta Plus apparatus (Brookhaven) and the results were averaged over three independent sets of measurements. pH of the suspension was measured with a Precision Digital pH-meter OP-208/1 and adjusted with $0.1\ \text{M}$ HCl or NaOH, if necessary.

Emulsion stability measurements have been performed with a Turbiscan Lab apparatus (Formulation). Results were analysed using Turbisoft ver. 1.13 software. This apparatus can detect and measure small changes in the emulsion behaviour. The instrument collects transmission (T) and backscattering (BS) light. The backscattered intensity percentage (BS%) vs. time led directly to the photon length value as a function of time, which gives information about the size of the droplet emulsion [16]. Samples were prepared in the following manner: $20\ \text{ml}$ of the emulsion, prepared according to the previously described method, were placed into a cylindrical glass cell, equilibrated for $2\ \text{h}$ and measured in the Turbiscan. The sample without surfactant was measured as a control.

To determine the adhesion force between a single particle and a liquid bridge in the presence of anionic–cationic surfactants mix-

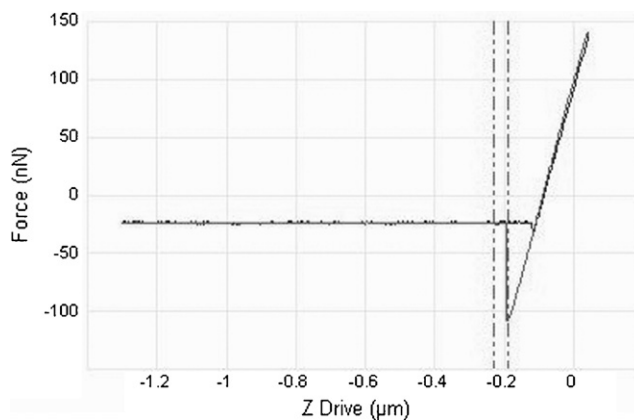


Fig. 1. Force vs. separation curve for the dolomite particle ($14.6\ \text{mg/g}_{\text{solid}}$) – surface covered with the layer of emulsion ($3.5\ \text{mg/g}$ of DDAHCl and $16\ \text{ppm}$ of NaOl) system.

ture, a method employing atomic force microscopy (AFM) was used. AFM cantilevers (spring constant $\sim 1\ \text{N/m}$) were modified by attaching a solid particle. The surface of the particle was modified with sodium oleate ($14.6\ \text{mg/g}_{\text{solid}}$). The layer of emulsion containing both surfactants and kerosene was deposited on a hydrophobic surface (teflon plate). Subsequently, the particle was brought into contact with the emulsion layer in order to create a liquid bridge and the force curves were recorded. For every sample, the measurement was performed in a few spots in order to eliminate the influence of local inhomogeneities. At every spot a series of $20\text{--}25$ curves were recorded in order to provide a sufficient amount of data for statistical analysis. Afterwards, the force of adhesion was calculated and the results are summarized on the graph presented in Section 3.5. The measurements were conducted in air at room temperature ($25\ ^\circ\text{C}$ and $43\% \text{RH}$). Fig. 1 shows an example of the force curve obtained at $3.50\ \text{mg/g}_{\text{solid}}$ of DDAHCl concentration.

All experiments were carried out at room temperature, i.e. $25\ ^\circ\text{C}$.

3. Results and discussion

3.1. Adsorption of sodium oleate (NaOl) on magnesite and dolomite

Data reported in literature showed that the sodium oleate (NaOl) is an appropriate reagent for a surface modification of salt-type minerals [2,17–19]. Upon contact with carbonate minerals at high pH values, sodium oleate precipitates as magnesium oleate or calcium oleate. The chemisorption mechanism on carbonate mineral surfaces has been discussed earlier by Gong et al. [20], Martínez-Luévanos et al. [21], and Sis and Chander [22]. Therefore, all experiments were done approximately at $\text{pH}\ 10$, and the amount of NaOl used was 12.2 and $14.6\ \text{mg/g}$ of magnesite and dolomite, respectively. According to reported data [17,23], this particular amount of sodium oleate in suspension led to the formation of a monolayer of surfactant molecules onto particle surface increasing its hydrophobicity.

3.2. Influence of cationic surfactant (DDAHCl) on oil agglomeration

The recovery of agglomerates and the agglomerates' diameters are shown in Figs. 2 and 3 as functions of DDAHCl concentration for dolomite and magnesite, respectively. The recovery of agglomeration was estimated as the ratio of the weight of obtained agglomerates to the weight of the feed sample. The results show that an initial increase in DDAHCl concentration in the system at a constant NaOl concentration (12.4 , $14.6\ \text{mg/g}_{\text{solid}}$ for magne-

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