Collectorless flotation of talc-magnesite ore with respect to particle size

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Talc-magnesite ore from Hnúšťa-Mútnik deposit in Slovakia was studied for the possibility of talc benefication using collectorless flotation. Ore samples were subjected to contact angle measurement using static sessile drop method showing the difference in hydrophobic talc surface (76.1 ± 5.7°) over hydrophilic magnesite (43.2 ± 6.2°). Comminution and classification of the sample led to accumulation of talc in finer size classes. Empirical model applied to results of laboratory froth flotation kinetics of six individual size classes indicate that optimum particle size for collectorless flotation of the ore is about 115 - 120 µm. Fine particles under 36 - 38 µm showed increased recovery to froth product by entrainment with the effect strongly noticeable for magnesite particles.

Keywords: *froth flotation, collectorless flotation, flotation kinetics, mineral processing, MATLAB*

Introduction

Talc is a hydrous magnesium silicate mineral with the chemical formula $Mg_3Si_4O_{10}(OH)_2$. It is the softest known mineral with the hardness of 1 on Mohs scale and has density of 2.78 (Anthony et al. 2001). Natural floatability of talc is closely related to its three-layer sheet crystal structure. Positive charge of magnesium layer is compensated from two sides by silicon tetrahedra layers creating the lack of surface polarization responsible for its natural hydrophobicity (Fuerstenau et al. 2007). When talc is ground two different surfaces are formed: faces (basal planes) and edges. Hydrophobic faces are formed when weak Van der Waals forces holding together individual sheets are disrupted. These consist of inert $-Si-O-Si-$ groups. Hydrophilic nature of the edges is caused by $SiOH$ and $MgOH$ groups that are formed when ionic bonds inside the three-layered structure are ruptured (Gomes a Oliveira 1991; Morris et al. 2002). Recent studies have shown that grinding/milling method of talc mineral significantly affects particle characteristics such as elongation, roundness and roughness, wetting and consequently also floatability of talc mineral (Yekeler et al. 2004; Terada and Yonemochi 2004; Kursun and Ulusoy 2006; Yin et al. 2012; Mierczynska-Vasilev and Beattie 2013). These findings correspond with postulates that flotation performance and electrochemical properties of talc ore depend on the ratio of hydrophobic/hydrophilic surfaces (Yehia and Al-Wakeel 2000). Investigation of particle size effect of pure talc samples on collectorless column flotation kinetics by Yekeler and Sonmez (1997) led to conclusion, that flotation rate constants decrease with increasing particle size and highest recoveries were achieved for fine particles under 38 μm.

Magnesite is a magnesium carbonate, with the chemical formula $MgCO_3$. It has hardness of 3.5 - 4.5 and the density of 3.0 (Anthony et al. 2001). Magnesite is a polar salt-type mineral that is naturally hydrophilic and therefore performs badly when floated without using a suitable collector (Chen and Tao 2004).

Different methods and approaches were investigated by researchers worldwide for processing of talc-carbonate ores with different proportions of talc, magnesite and dolomite. Yehia and Al-Wakeel (2000) successfully carried out microflotation and flotation tests showing that collectorless flotation is suitable for benefication of Egyptian talc-carbonate ore sample with satisfactory concentrate grade and yield for industrial uses. Concentrate with 93.5 % talc content at 70 % recovery was prepared from ore containing 47.5 % talc using one cleaning stage with regrinding. Laboratory froth flotation tests carried out by Derco and Németh (2002) showed that it is possible to obtain quality talc for electroceramics and pharmaceutical industry from low quality carbonatictalcose rocks of Kokava and Sinec deposits in Slovakia when a set of flotation reagents is used. Investigation of flotation performance of various particle size classes of talc-magnesite ore from Hnúšťa-Mútnik deposit in Slovakia by Zeleňák and Škvarla (2001) in the presence of a collector revealed an adverse effect of fine particle classes when decrease of separation efficiency was observed in the presence of -40 μm particles. Hojamberdiev et al. (2010) on the other hand promotes gravitational methods of separation for talc-magnesite ore from Zinelbulak deposit in Uzbekistan over flotation in the terms of

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economics. Grindability tests revealed that talc and magnetite are completely separated when ground together for 10-12 minutes with talc concentrated in the finer size class (-100 μm) and magnesite in the coarser size class. Possibility of partial concentration of talc by comminution was also confirmed by Ahmed et al. (2011).

Although talc and talc-carbonate ores have been previously well investigated by researchers worldwide in terms of surface characteristics, flotation kinetics, collectorless flotation and size dependent flotation kinetics as well, this article presents results of experiments aimed to evaluate the possibility of collectorless flotation of talc from talc-carbonate ore with respect to particle size. Evaluation of the possibility was done in several steps. Dominant minerals found in ore - talc and magnesite were characterized by contact angle measurements using static sessile drop method as an evaluation of their surface characteristics. Flotation experiments of six size fractions at four combinations of air flow rate and stirring speed were evaluated as a function of mineral recovery on flotation time - flotation kinetics.

Methods

Sample

In experimental section of this work, lump talc-magnesite ore sample from Hnúšťa-Mútnik deposit with 48 % average talc content was used. XRD (X-Ray Diffraction) spectra of the ore shown in Fig. 1 revealed the presence of two minerals - talc $Mg_3Si_4O_{10}(OH)_2$ with β-monoclinic lattice and magnesite with magnesium partially substituted by iron $(FeMg)CO_3$. XPS (X-Ray Photoelectron Spectroscopy) spectra of the ore shown in Fig. 2 revealed minor impurities in form of calcium.

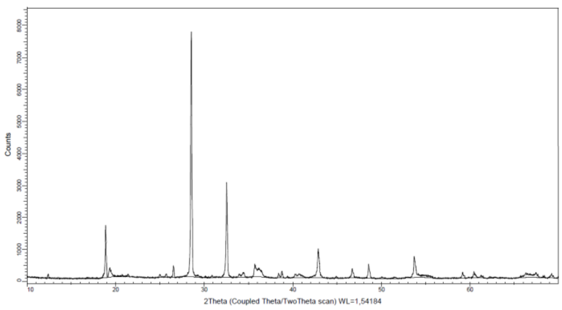

Fig. 1. XRD spectra of talc-magnesite ore used in this study.

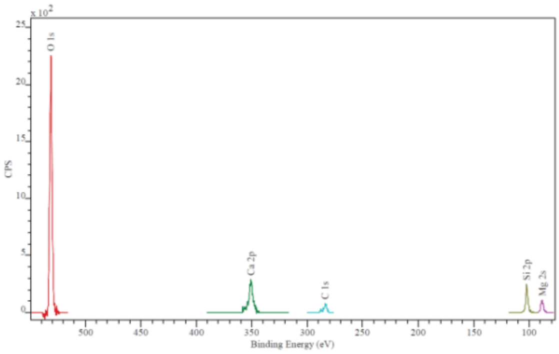

Fig. 2. XPS spectra of talc-magnesite ore used in this study.

Contact angle measurement

Contact angle was measured by static sessile drop method using EasyDrop goniometer by KRÜSS, which uses drop shape analysis of digitalized video frames. Microsyringe was used to place drop of distilled water with volume in the range of 3-5 μl onto mineral surface. Both left and right side contact angle of at least 25 water drops on each mineral was measured. Measurement was done at 22°C.

Sample comminution

Talc-magnesite lump ore: 10 - 100 mm was ground in two stages using cone crusher in first stage with output d_{80} = 11 mm. Second stage consisted of crushing of the output from first stage using roller crusher with output d_{80} = 1.8 mm. Final stage of comminution was milling using friction mill where the output was continuously dry sieved to achieve 100 % of material under 315 μm. Individual size classes used in this study were prepared by dry sieving method in Fritsch Analysette 3 vibratory sieve shaker.

Modified Hallimond tube flotation

Flotation experiments in modified Hallimond tube (microflotation) were done in Hallimond tube with 35 mm diameter and sintered disc at bottom. Air inlet of the tube was plugged to regulated electrical air pump. Digitally adjustable magnetic stirrer was placed under the bottom of the tube. Feed consisted of 5 g of the material. Slurry was stirred for 2 minutes at 250 rpm after the addition of collector. After the conditioning period the air pump with preset air flow was started. Material was floated for 2 minutes after which both air pump and stirrer were stopped. Products were then collected, filtered, dried at 60°C and analyzed for loss on ignition (LOI).

Froth flotation

Flotation experiments were carried out in automated laboratory froth flotation machine using 3 l mechanical Denver flotation cell. Slurry contained 300 $g.l^{-1}$ solids. Five size fractions: 0 - 63 μm, 63 - 90 μm, 90 - 125 μm, 125 - 180 μm and 180 - 250 μm were floated at four different combinations of air flow and agitation speed: 1200 rpm / 4 $l.min^{-1}$, 1200 rpm / 6 $l.min^{-1}$, 1600 rpm / 4 $l.min^{-1}$ and 1600 rpm / 6 $l.min^{-1}$. Slurry pH was not adjusted. Slurry temperature ranged from 18 - 20 °C. The only flotation reagent used was polyethylene glycol (PEG) frother with concentration 100 $g.t^{-1}$ of solid. Slurry was conditioned for 3 minutes before the air introduction. Withdrawal of froth product was done partially at six different cumulative flotation times: 30, 60, 120, 240, 360 and 600 s. Individual froth products were separately filtered, dried and representative samples were analyzed for LOI.

Results and discussion

Wetting characteristics

The difference in wetting characteristics of talc and magnesite from the ore is evident on the result of contact angle measurement. Contact angle measured on ore samples is 76.1 ± 5.7° for talc and 43.2 ± 6.2° for magnesite. Number of authors investigated contact angle of water drop on talc surface using static sessile drop method, where the results lies in the broad range of 50.0° - 83.2° (Schrader and Yariv 1990; Yildirim 2001; Douillard et al. 2002; Castro Lobato 2004). Contact angles calculated from measurement of wicking on powdered samples using capillary rise method ranged between 69.7 ± 5.3 and 86.7 ± 0.3° (Yildirim 2001; Beattie et al. 2006; Casanova et al. 2007). Hydrophilic nature of magnesite surface is also confirmed by contact angle measurements of several authors (Kmeť 1992; Gonzalez and Moreira 1991; Gence 2006). Contact angles are not only dependent on surface chemistry, but also on interplay of various nonidealities of the surface (Škvarla 2003) and these values can thus only be used for comparison of the two mineral surfaces prepared using the same procedure.

Comminution

Because of significant difference in hardness of magnesite over talc, talc cumulates in fines when grinding and milling talc-magnesite ore as stated by Hojamberdiev et al. (2010); Ahmed et al. (2011). Results of chemical analysis of talc-magnesite ground ore size fractions are presented in Tab. 1.

Tab. 1. Chemical composition of individual size fractions used in this study

Fraction [μm]	SiO_2 [%]	MgO [%]	CaO [%]	Fe_2O_3 [%]	Al_2O_3 [%]	LOI [%]
0 - 63	34.9	25.8	0	0.9	0.7	20.2
63 - 90	27.7	32.2	0	1.5	0.5	23.1
90 - 125	19.9	35.5	0	1.6	0.3	26.2
125 - 180	17.2	37.5	0	1.8	0.2	31.3
180 - 250	16.7	38.7	2.2	1.7	0.2	35.0
250 - 315	11.3	41.1	2.2	1.9	0.1	37.7

Loss on ignition was chosen as characteristics for concentrate grade. Pure talc samples from investigated deposit are characterized by loss on ignition about 5 %. Loss on ignition of carbonates such as calcite, magnesite and dolomite exceeds 44.0 % particularly that of magnesite with about 52.2 % based on its chemical composition. Difference in particle size distribution of individual minerals after two stages of grinding and milling to -315 μm was significant and is demonstrated using Rosin-Rammler diagram shown as Fig. 3.

Finest investigated fraction -63 μm contained over 66 % wt. of talc, while fraction 250 - 315 μm contained over 70 % wt. of magnesite.

Flotation in modified Hallimond tube

Although primary intent of the research was investigation of the possibility of obtaining clean talc from talc-magnesite ore using collectorless froth flotation, effect of collector use and dosage was investigated in modified Hallimond tube. Cationic collector Kamisol D-50 was chosen as it was previously successfully used to increase talc recovery from talc-carbonate ore by Zeleňák and Škvarla (2001). Experimental plan contained three variables; five size fractions (63-90 μm, 90-125 μm, 125-180 μm, 180-250 μm, 250-315 μm), five collector dosages (0 $g.t^{-1}$, 50 $g.t^{-1}$, 100 $g.t^{-1}$, 200 $g.t^{-1}$, 400 $g.t^{-1}$) and two air flows (35 ml/min, 50 ml/min). Results are presented in terms of talc recovery as Fig. 4 and Fig. 5.

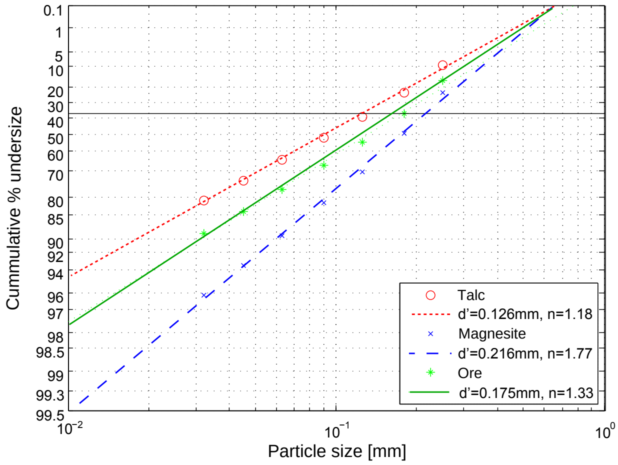

Fig. 3. Rosin-Rammler diagram of particle size distributions of talc, magnesite and talc-magnesite ore after two grinding stages and milling.

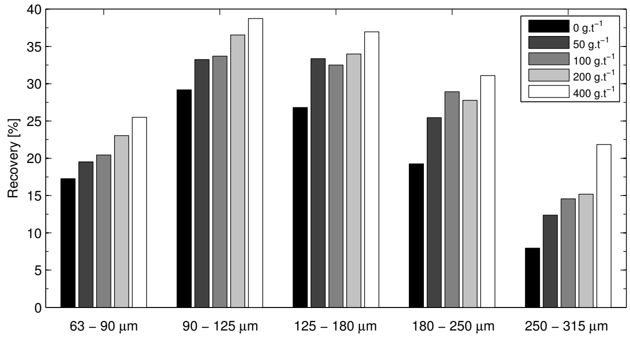

Fig. 4. Recovery of talc at different collector dosages after two minutes of flotation in modified Hallimond tube. Airflow 35 ml/min.

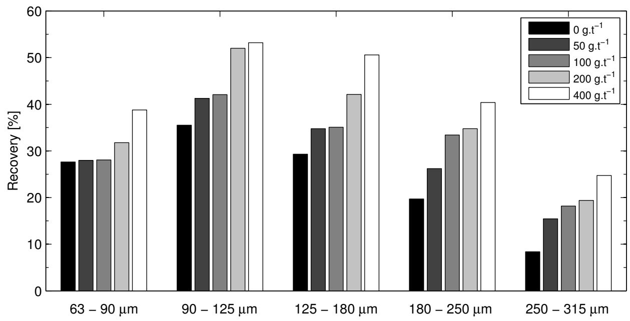

Fig. 5. Recovery of talc at different collector dosages after two minutes of flotation in modified Hallimond tube. Airflow 50 ml/min.

MANOVA (Multivariate Analysis Of Variance) test proved statistically significant effect (p leq 0.05) of all three factors on recovery of talc to concentrate. Effect of factors decreased in order: granularity, air rate and collector dosage. Test also revealed statistically significant effect of granularity and collector dosage on concentrate grade with higher effect of granularity.

Highest recoveries at given flotation time were reported for 90-125 μm a 125-180 μm size fractions. In terms of recovery, coarser, as well as finer size classes performed worse. These results are in agreement with studies on effect of particle size on mineral flotation performance (Santana et al. 2008; Abkhoshk et al. 2010; Rahman et al. 2012; Muganda et al. 2012) that report existence of optimum floatable particle size. In case of talc the decrease in fine particle recovery can be partially accounted to increased proportion of hydrophilic edges over hydrophobic faces in finer size classes. Mineral particle collection efficiency as presented by Derjaguin includes consecutive sub-processes - efficiencies of collision, attachment and stability. Trends in these efficiencies with particle diameter as shown i.e.

in Duan et al. (2003) clearly explain the decrease in flotation rate constant and consecutive recovery to concentrate for fine and coarse particles. Decrease in fine particles recovery is due to lower probability of air bubble - particle collision (Duan et al. 2003; Koh and Schwarz 2003; Liu and Schwarz 2009). Chipfunhu et al. (2012) suggests that kinetic energy of fine particles is insufficient to break the air bubble - mineral particle barrier. Maximum floating particle size is a function of contact angle, particle density and hydrodynamic conditions in cell with minor effect of bubble diameter (Nguyen 2003; Kowalczuk et al. 2011).

Easiest floating particle size for given hydrodynamic conditions and material is in the range of approx. 100-150 μm. Particle size also affected quality of products - concentrate grade. Loss on ignition of concentrate, regardless of collector dosage, increased with increasing average size class: 8.2 ± 0.2 %, 9.6 ± 0.4 %, 9.5 ± 0.4 %, 10.4 ± 0.3 % and 10.8 ± 0.7 %. In terms of talc content it is decrease from 93.3 ± 0.4 % to 87.1 ± 1.6 %. This increase in loss on ignition - decrease in concentrate grade is caused by low degree of liberation of coarse particles. Strong effect of mineral liberation on flotation performance is confirmed by many articles concerning flotation plant optimization by comminution circuit manipulation (Fernandes and Peres 1999; Sosa-Blanco et al. 2000; Lastra 2007; Al-Wakeel et al. 2008). Decrease in concentrate grade with increasing collector dosage was less significant although it is a demonstration of selectivity drop at high collector dosages.

Froth flotation

Recovery of talc and magnesite to froth product was significantly affected by particle size (Fig. 6 and Fig. 7). Highest talc recoveries were achieved for 63 - 90 μm and 90 - 125 μm size fractions. Optimum floatable size is lower value when compared to optimum floatable size in modified Hallimond tube. This is caused by difference in hydrodynamic conditions in Hallimond tube that were with mixing at 250 *rpm* closer to plug flow, while in mechanical cell there was high degree of turbulence, especially at 1600 rpm. Recovery of talc to froth product from 180 - 250 μm size fraction at any combination of stirring speed and air flow did not exceed 20 % and is therefore not suitable for collectorless froth flotation. Grinding to -150 μm is advised for froth flotation of talc-magnesite ore in mechanical cells.

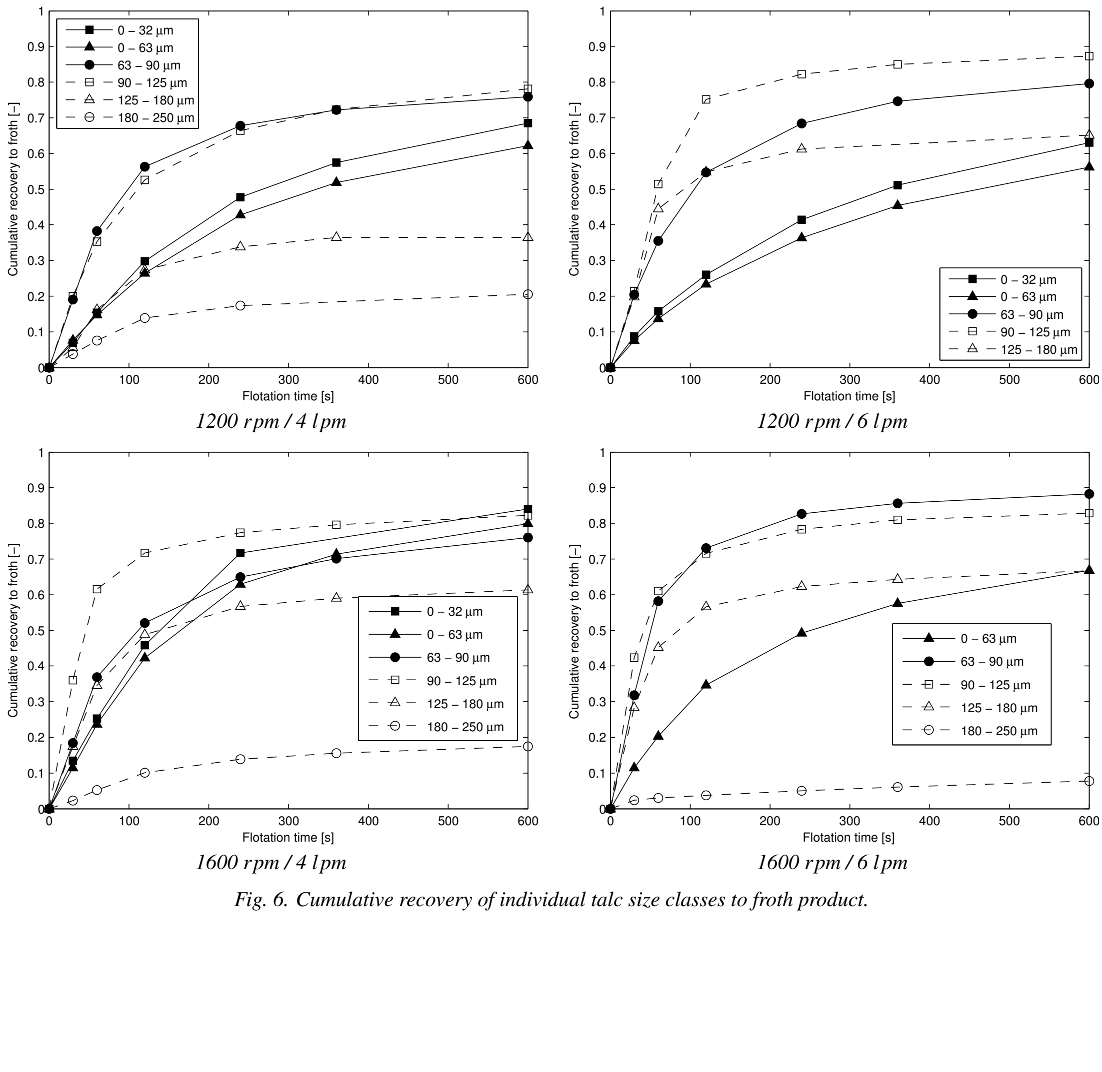

Fig. 6. Cumulative recovery of individual talc size classes to froth product.

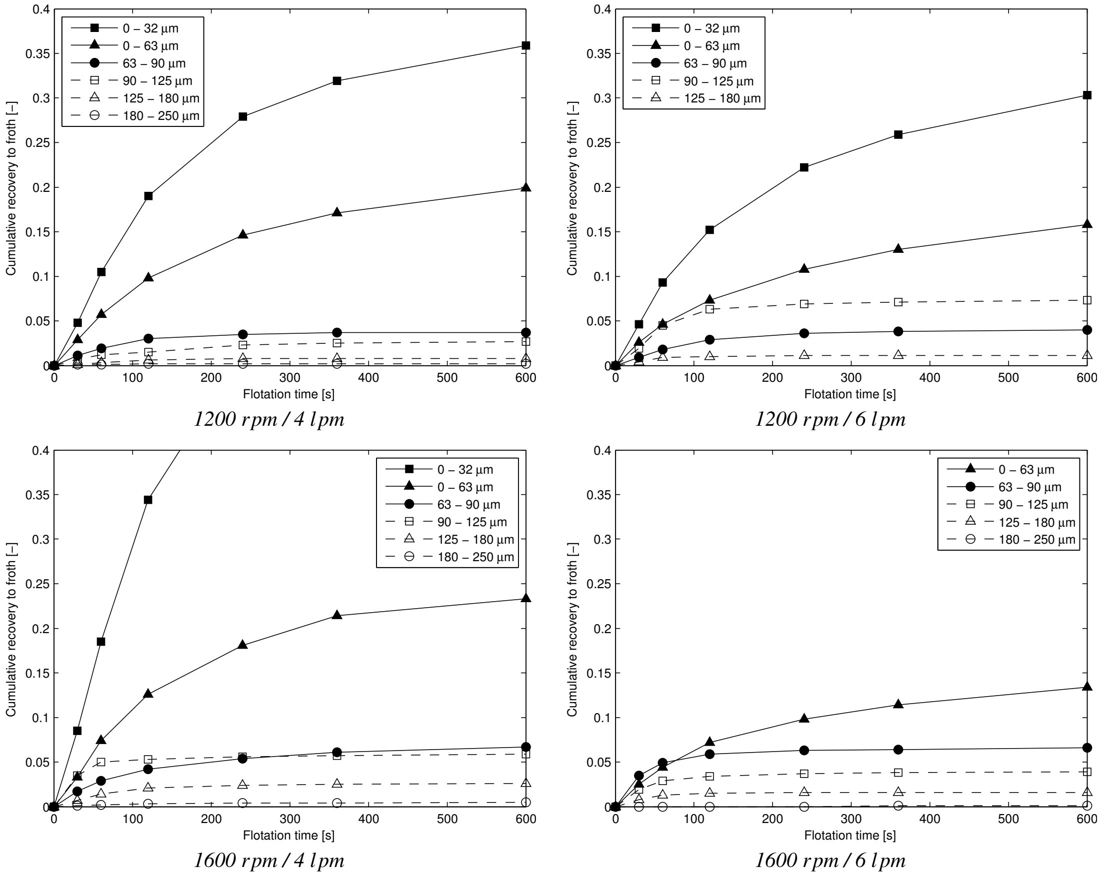

Fig. 7. Cumulative recovery of individual magnesite size classes to froth product.

Notable difference between the results of flotation in Hallimond tube and in mechanical cell was in concentrate grade. There was decrease in concentrate grade with increasing particle size in Hallimond tube due to decrease in liberation. Highest concentrate grades from froth flotation in mechanical cell were achieved for coarse fraction (180 - 250 μm). In turbulent conditions only purest talc particles of coarse size classes were able to attach to air bubbles and form a stable connection causing low recoveries when floating particles over 180 μm at high concentrate grades. LOI of - 63 μm size class in all cases exceeded 11 % (86.7 % talc content). LOI of coarser size classes was about 6.8 $\pm$ 0.9 % (96 $\pm$ 2 % talc content).

Conclusion

Talc is naturally hydrophobic mineral that is frequently occurring with naturally hydrophilic carbonate minerals such as magnesite, calcite and dolomite. Such a combination of wetting characteristics in an ore is well suited for benefication using flotation process. Although floatability of talc can be further increased using collectors (Ahmed et al. 2007; Derco and Németh 2002; Zeleňák and Škvarla 2001) or by pH adjustment (Ahmed et al. 2007) and floatability of magnesite depressed (Gawel et al. 1997; Santana and Peres 2001), collectorless froth flotation using frother as the only chemical as possible benefication process was investigated.

Surface characteristics were measured by contact angle measurements using static sessile drop method of distilled water on lump surfaces. Contact angle measured on talc from Hnúšťa-Mútnik (Slovakia) deposit was 76.1 $\pm$ 5.7°. Magnesite from this deposit has contact angle 43.2 $\pm$ 6.2°. This difference in contact angles of studied minerals is high as expected for naturally hydrophobic talc surface and polar magnesite surface.

Significant difference in hardness of talc and magnesite leads to accumulation of talc in fine size classes when grinding and milling talc-magnesite ore. Talc content in -63 μm size class increased from average 48 % talc to over 66 % wt., while magnesite content in 250 - 315 μm was over 70 % wt.

Results of frothless flotation in modified Hallimond tube using various concentrations of cationic collector proved that the rate at which talc particles float can be further increased. Portion of recovered material after two minutes of flotation with collector was higher than portion of material recovered without collector addition and rose with increasing collector dosage. Collector addition negatively affected flotation selectivity resulting in decreasing concentrate grade with increasing collector dosage. Increase in air flow rate resulted in higher recoveries at given flotation time without significant effect on concentrate grade. Highest influence on both recovery of talc to concentrate and concentrate grade had granularity of material. Highest recoveries were recorded for 90 - 125 μm size fractions with decrease in recoveries towards both finer and coarser fractions at all combinations of collector dosages and air flow rates. Concentrate grade decreased with increasing average particle size which can be attributed to low degree of liberation.

Froth flotation tests aimed at evaluating the possibility of collectorless talc flotation from talc-magnesite ore validated significant effect of particle size on recoveries of individual minerals. Highest recoveries of talc to froth product were achieved for 63 - 90 μm and 90 - 125 μm size fractions depending on hydrodynamic conditions in flotation cell given by combination of stirring speed and air flow rate. +150 μm talc particles float with low recoveries as only purest talc particles were able to attach to air bubbles and form a stable connection. Particles over 150 μm were not sufficiently liberated as indicated also by modified Hallimond tube tests. Highest magnesite recoveries were recorded in finest size fraction -63 μm because of strong entrainment effect that negatively affected concentrate grade.

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