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SELECTIVE SEPARATION OF FINE PARTICLES BY A NEW FLOTATION APPROACH

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ABSTRACT

Fine particles often create problems in flotation applications. In this article a new laboratory flotation system for the selective separation of small particles was designed and tested. The device contains an active counter current sedimentation that should prevent entrainment of the fine hydrophilic particles. The cell was used to selectively float fine particles in the size range 2–25 μm. To create small bubbles dissolved air was used.

The study is linked to the problems that fine particles cause by remediation of soils and sediments. Therefore, small silica and small-oxidized carbon black (MT-OX) particles were used as model system. Three different frothers, sodium dodecylsulfate (SDS), Aerofroth, and Montanol were applied to obtain a stable froth.

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*Corresponding author.

The results showed that the equipment works excellent to separate the fine MT-OX particles from the small silica particles. Especially with Aerofroth as frother, the Grade of the flotation experiments was extremely high (98.1%). The MT-OX Recovery was best with SDS (74.6%). The new flotation design provides a promising method for the remediation of contaminated sediments and soils. Next to that it offers an interesting option to separate fine particles and powders in other industrial applications.

INTRODUCTION

Flotation is a separation technique based on the capture of particles by bubbles and their collection in a froth layer. It has already been used in the mining industries for more than 100 years (1). Nowadays, it has also found its way in many other applications like drinking- and waste water treatment (2,3), plastic separation (4), soil and sediment remediation (5–12), and de-inking of paper (13,14).

Capture of particle and bubble can only occur when they undergo a close encounter, which is governed by the hydrodynamics in the reactor. When the particle approaches the bubble within the range of attractive surface forces, film drainage, film rupture and contact line movement occur; and a particle–bubble union is formed. However, at the bottom of the rising bubble the particle may become dislodged from the bubble if the detachment forces exceed the surface forces. Therefore the capture efficiency of a bubble and a particle may be defined as the product of the collision efficiency, the attachment efficiency, and the stability efficiency (15,16). An analysis of these factors (17–20) and flotation praxis (21–24) indicates that the presence of small particles or fines often causes problems in flotation. Fines in flotation are mostly not only colloidal particles but are defined as all particles below 10 μm (18). They are of importance to entrapment, entrainment, excessive adsorption of reagents, and froth stabilization, and these factors may lead to a reduction in the flotation efficiency and/or selectivity (18,25).

Entrapment of fine hydrophilic particles (gangue) by hydrophobic particles or bubble–particle aggregates is a main cause for high gangue recoveries (25). Hydraulic entrainment of particles in the liquid film between air bubbles in the froth becomes prominent when large quantities of fine particles are treated (26). Entrainment increases with water recovery and high slurry density (19,27).

To optimize the flotation of fine particles, small air bubbles and a low air flow rate are desired (25,28). Small bubbles do always give higher attachment efficiencies than the larger bubbles. It has been shown that the increase in attachment efficiency, caused by a decreasing bubble size is more pronounced for smaller particles than for larger particles (16). Therefore it is always more effective to utilize small air bubbles for the flotation of small particles.

An elegant way to create small bubbles is the use of dissolved air. Dissolved air flotation (DAF) is the process whereby small bubbles are precipitated when water, supersaturated with air under high pressure, is released under atmospheric conditions. It is mainly used in the field of drinking- and wastewater treatment (3,29,30). The micro-bubbles formed by the DAF have a diameter generally between 10 and 120 μm and a reasonable estimate of the average bubble diameter is 40 μm (30).

In this research a newly designed flotation device is tested for its ability to selectively float fine particles. Dissolved air is used as the bubble source. In general, our research is linked to the remediation of contaminated soil and sediment. For the remediation of the smallest particle fractions, there is no appropriate classification technique available. Flotation appears to be a promising alternative for the separation of soot type particles, containing polycyclic aromatic hydrocarbons (PAH), from the sand particles (9). Therefore, a model system that simulates a contaminated sludge composed of very small soot/coal–tar particles and small sand particles is composed. In this model system oxidized carbon black particles represent the soot/coal–tar particles and pure silica of the sandy fraction. The carbon black sample and silica sample are both in the particle size range 2–25 μm. Because coals only often need a frother to float (31,32) no collector is added. Three different types of frothers are tested and their influence on the grade and recovery is calculated.

EXPERIMENTAL

Materials

Reagents

Three frothers were tested: Aerofroth 76a, Montanol 350a, and sodium dodecylsulfate.

Sodium dodecylsulfate (SDS) was obtained from Aldrich.

Aerofroth 76a, a commercial mixture of C4–C7 alcohols, was obtained from Cytec Industries B.V. (The Netherlands).

Montanol 350, a mixture of mainly higher alcohols and esters with additives, was obtained from Aventis (formally Hoechst A.G. (Germany)). Montanol was emulsified with Eumulgin ET 10, a commercial ethoxylated fatty alcohol obtained from Henkel. The emulsion was obtained by intensively blending 5 mL Montanol in 41 mL demineralized water with 4 mL of a 0.1 g/L Eumulgin solution.

Flotation experiments were carried out with tap water. The hardness of the tap water based on the total Mg^{2+} and Ca^{2+} concentration was around 0.8 mmol/L.

Particles and Slurries

Oxidized carbon black particles denoted as MT-OX were obtained by oxidation of Sterling MT carbon black obtained from Cabot (USA). Prior to the oxidation, the particles were cleaned by extraction in toluene. The oxidation was carried out with 100 g carbon black in 800 mL 2 M HNO_3 for 6 hr at 75°C. With the oxidation step acidic surface groups are formed (about 20 μmol/g). Oxidation facilitates the dispersion in aqueous solutions. The degradation products of the oxidation were removed by extensive rinsing with subsequently demineralized water, 1% Na_2CO_3 solution and again demineralized water. The thus obtained suspension is washed with 0.1 M HCl to exchange the Na^+ counterions for H^+ and washed with demineralized water again. The final MT-OX is dried and stored in a closed container.

Before use, the MT-OX is redispersed in water. In order to break down larger particle aggregates into small aggregates, 5 g of MT-OX were mixed in a beaker with 500 mL of tap water and ultrasonificated for 15 min using an Elam Transonic T700 (35 kHz). The ultrasonified MT-OX particle aggregates had a diameter of around 4 μm. A microscope image of the particles is shown in Fig. 1.

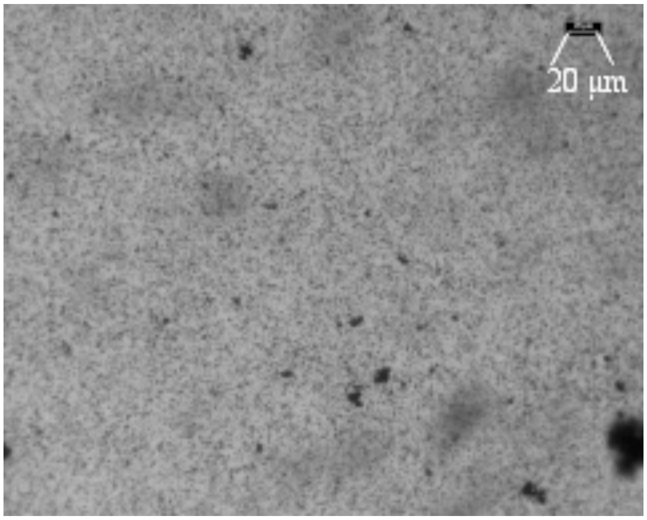

Figure 1. MT-OX particles.

Although the treatment was successful, the presence of some large particle aggregates in the ultrasonified MT-OX could not be avoided.

Silica particles were obtained from Aldrich (code name: Silica gel 28,8500-0). The size range of the silica particles as quoted by Aldrich is 2–25 μm. Figure 2 presents a microscope image of the particles. Dispersions were prepared by simply mixing the silica with water.

Slurries. Three slurries were tested, MT-OX, silica, and MT-OX/silica. The MT-OX slurry contained, 5 g of the ultrasonified MT-OX, the silica slurry contained 25 g silica and the MT-OX/silica slurry contained 25 g silica plus 5 g ultrasonified MT-OX in 500 mL water.

Methods

Flotation Device

The flotation equipment used in this study is a large scale (total volume 2.6 L) modified Hallimond tube, see Fig. 3. Two modifications are incorporated. The tilted zone is extended in length to allow a better settlement of entrapped and entraint particles (9). Such a tilted zone placed after the mixing zone is commonly used in water treatment plants (29). In the tilted tube, the turbulence will decrease because all the rising bubbles float against the inside of the upper wall. Next to this, the settled

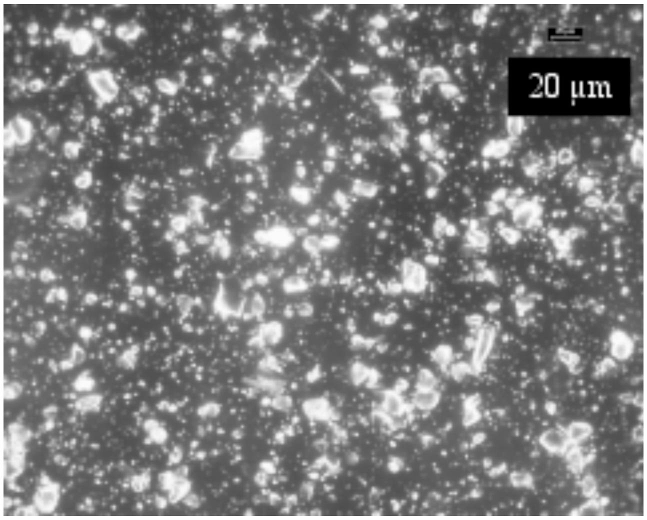

Figure 2. Silica particles.

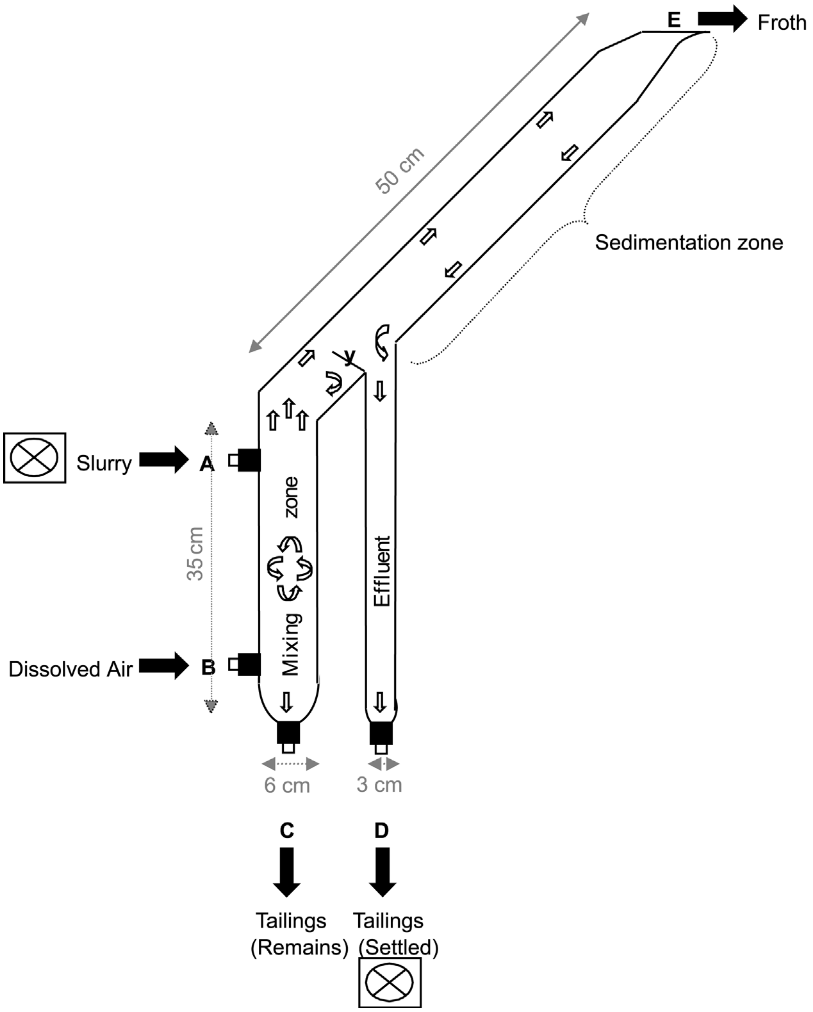

Figure 3. New flotation device.

particles are drained away through an additional compartment as in the Rubio cell (33). Baffle Y prevents the settled particles from falling back into the mixing zone of the reactor, so that there is no accumulation of fine hydrophilic particles in the mixing compartment. The separation of the mixing compartment and sedimentation compartment also creates an active counter current sedimentation flow which improves the settling velocity of the hydrophilic particles.

Dissolved Air Solution

Water (with or without frother) was saturated with air in a saturation tank (5 L) at a pressure of 8 bar. It was released to atmospheric pressure in the flotation cell through an adjustable nozzle (obtained from Hoke), creating small bubbles of 10–100 μm.

Flotation Procedure

Prior and during flotation the particle slurry in the glass beaker is stirred with a magnetic stirrer. Before the slurry was fed into the mixing compartment at point A (30 mL/min) the flotation cell was filled with water. The DA flow was started at point B (90 mL/min), 2 min prior to the sludge flow. During the flotation, the effluent was removed as "Froth" at point E or as "Settled" at point D. The valve at C remained closed till the end of the experiment. The flow at point D was set equal to the flow of A plus the DA flow at B so that the water level at point E was constant. Each minute the froth was manually removed from the top by placing a spoon just under the water table and lifting the froth into a beaker. If no froth occurred, a very small overflow (~5 mL/min) was created, but the particles floating at the top were also removed manually, once a minute. During the experiment, the wall of the influent beaker was rinsed regularly to remove the particles attached to the glass. When the influent was almost finished, water was added to remove also the last particles from the beaker. In total 250 mL water was added for rinsing. When the influent flow was finished, the DA flow was continued for 10 min whereafter the experiment was stopped. After the experiment, the slurry in the mixing part was collected as "Remains". The slurry in the sedimentation tube was added to the "Settled" fraction. In this way three fractions were collected, Froth, Settled, and Remains.

Analyses

All the samples were dried at 95°C to determine the dry matter content. Subsequently the organic matter content was determined by oxidation of the dry matter samples in a oven at 550°C for 24 hr.

Grade and Recovery

The organic content of the samples reflects mainly the MT-OX content. For the precise calculation of both the amount of MT-OX and silica in each sample, the organic contents of silica (2.6%) and MT-OX (99.5%) have been taken into account. With the known amounts of MT-OX and silica in each of the fractions the grade and recovery have been calculated as:

$$\text{Grade} = \frac{\text{Amount of MT-OX in froth (g)}}{\text{Dry matter in froth (g)}} \times 100\% \tag{1}$$

$$\text{Recovery} = \frac{\text{Amount of MT-OX in froth (g)}}{\text{Amount of MT}-\text{OX in Settled, Froth, and Remains (g)}} \times 100\% \tag{2}$$

RESULTS AND DISCUSSION

Frother Addition

Screening tests were done to find the minimum concentration of frother that is necessary to create small bubbles and a stable froth. To this aim, different frother amounts were tested. In preliminary tests frother was added to either the influent slurry or to the water in the saturation tank before air saturation.

The screening tests showed that addition of frother to the DA solution was much more efficient than addition to the particle slurry. With the addition of frother via the DA solution, lower frother amounts were sufficient to create appropriate air bubbles and stable foam. Also the formation of macro bubbles (30,34) is substantially reduced by this way of frother addition.

Minimum frother concentrations of the DA solution at which the formation of small air bubbles and a stable froth was achieved are presented in Table 1. In further experiments frother was always added with the DA.

Flotation Tests of Pure Samples

The first flotation experiments were performed on the pure silica and MT-OX samples. The entrainment of the small particles was measured and the influence of frother on the particle floatability was estimated.

Table 1. Minimum Frother Concentrations to Obtain a Stable Foam and Small Bubbles

Frother	Sodium Dodecylsulfate	Aerofroth 76a	Montanol 350
Concentration	0.028 g/L DA	0.017 mL/L DA	0.008 mL/L DA

Table 2. Mass Distribution over the Three Fractions of Silica

	Remains (%)	Settled (%)	Froth (%)
No frother	16	80	4
SDS (0.028 g/L DA)	17	81	2
Aerofroth (0.017 mL/L DA)	16	82	1
Montanol (0.008 mL/L DA)	18	81	1

Results for silica are shown in Table 2 and those for MT-OX in Table 3. The results show the distribution of mass over the three fractions. A certain amount of material could not be removed from the influent beaker or stayed in the influent tubes. Therefore, the total particle mass of the three fractions was always smaller than the particle mass in the original influent. The percentages given in Tables 2 and 3 are thus based on the total amount in the three fractions.

Without the addition of frother, the froth layer was very thin and the bubbles showed little stability and collapsed mostly at point E. In this case a small overflow was maintained to be able to collect the particles from the top.

The results of the silica samples show that the amount of silica that remained in the mixing compartment (Remains) was around 17% for all the experiments. The amount of silica that was collected in the Froth was very low. Without frother 4% was collected in the Froth, with SDS, Montanol or Aerofroth only 1–2%. The amount of silica that is carried out of the mixing compartment but settled in the sedimentation zone is larger (~80%) and not effected by the frother. As silica should not end up in the froth, the latter result shows the importance of the active sedimentation zone in the flotation equipment. The present device minimizes the entrainment.

The results of the MT-OX samples show that in the presence of frother the amount of MT-OX in the Remains was around 16%. When no frother was added the amount of MT-OX in the Remains equals 37%. This high value is reflected in the relatively low values for the Froth and the Settled fraction. Comparison of the

Table 3. Mass Distribution over the Three Fractions of MT-OX

	Remains (%)	Settled (%)	Froth (%)
No frother	37	55	8
SDS (0.028 g/L DA)	15	49	36
Aerofroth (0.017 mL/L DA)	16	71	14
Montanol (0.008 mL/L DA)	17	72	11

37% with the 16% indicates that the particle–bubble attachment efficiency is improved. Hence, the frothers also act as collectors.

The amount of MT-OX in the froth is very modest. This can be due to the hydrophilic groups present at the surface of MT-OX. Also the flotation device, which is constructed to minimize the entrainment, will be responsible for the low amount of MT-OX in the Froth. The experiment with SDS shows that 36% of the mass is in the Froth, this is four times more than in the absence of SDS. For the other two frothers the amount of mass in the froth is much less (11–14%). The high amount of mass in the Froth in the case of SDS leads to a relatively low amount (49%) of settled material. For Aerofroth and Montanol the amount of settled material is 71–72%. As the "Remains" are the same for the three frothers the results indicate that with SDS more stable particle/bubble complexes are formed than with Aerofroth and Montanol.

Due to the fact that MT-OX is not highly hydrophobic the particles will settle in the sedimentation zone unless an effective collector is present.

Flotation Tests of Mixed Samples

To test the performance of the new flotation device, the particles were mixed and the separation efficiency was calculated by the Grade and Recovery. The results of the flotation tests are shown in Table 4 and in Figs. 4 and 5.

The dry matter percentages given in Table 4 are again based on the total dry matter in the three fractions. The MT-OX percentages are based on the amount of MT-OX in relation to the total amount of silica and MT-OX in that fraction. The amount of MT-OX and silica is calculated from the organic matter content of that fraction as explained in the methods. The values in the table are averages of duplicate experiments.

The results in Table 4 shows that the average amount of material in the Remains was 14.7% for SDS, 19.5% for Aerofroth, and 17.3% for Montanol. These values are slightly higher than those of the pure samples. The MT-OX content of the Remains is 3.6% for SDS and around 5% for the experiments with Aerofroth and Montanol.

The relatively high dry matter percentage in the Remains of Aerofroth is reflected in the lower dry matter percentage of the Settled fraction (70.9%) compared to the amounts in the Settled Montanol (78.7%) and SDS (75.5%).

For SDS the lowest MT-OX contents are found in the Remains (3.6%) and Settled (3.3%) fractions. This corresponds with the better flotation of pure MT-OX in the presence of SDS.

The dry matter percentage of the Froth fraction in the presence of Montanol (4.0%) is lower that in the presence of Aerofroth (9.6%) and SDS (9.8%). Moreover, the MT-OX content in the Froth is for Montanol relatively low. The

Table 4. Dry Matter and MT-OX Percentage in Each of the MT-OX/Silica Samples

	Remains (%)		Settled (%)		Froth (%)	
	Dry Matter	MT-OX	Dry Matter	MT-OX	Dry Matter	MT-OX
SDS (0.028 g/L DA)	14.7	3.6	75.5	3.3	9.8	92.0
Aerofroth (0.017 mL/L DA)	19.5	5.3	70.9	7.6	9.6	98.1
Montanol (0.008 mL/L DA)	17.3	5.0	78.7	9.4	4.0	81.8

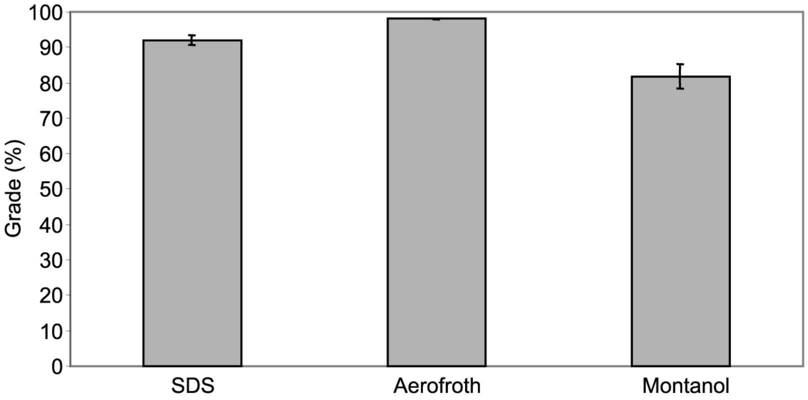

Figure 4. Grade in the presence of SDS, Aerofroth and Montanol.

MT-OX values of the Froth fractions stand for the selectivity of the flotation process and are also presented as Grade in Fig. 4. The values of the duplicates are shown as error bars.

Figure 4 clearly shows that the grade is high in all the experiments. With Aerofroth, the grade was almost maximum showing 98%. The grade with SDS is only slightly less but at 92% is still very high. The grade with Montanol is substantially lower (82%) and as mentioned before the dry matter percentage is low.

The calculated Recovery is presented in Fig. 5, the figures show the averages, with the values of the duplicates presented by the error bars.

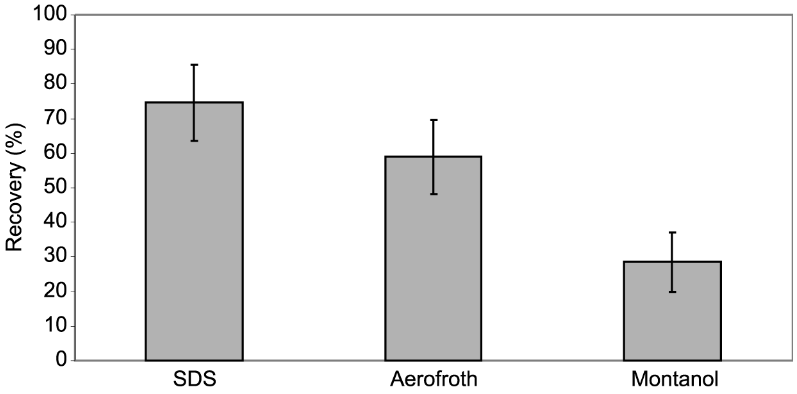

Figure 5. Recovery in the presence of SDS, Aerofroth and Montanol.

The Recovery of the device is lower than the grade. As expected from the previous discussion the best Recovery (74.6%) is obtained with SDS. With Montanol only 28.5% of the MT-OX could be floated. The relatively poor results with Montanol are probably caused by the low particle–bubble stability and the low stability of the froth. With Montanol as frother a thin froth layer with a coarse structure and a high water content was created. The froth was unable to hold sufficient amount of particles.

The Recovery with Aerofroth (58.9%) was, as expected from the pure sample experiments, lower than when SDS (74.6%) was used. Apart from the stability of the particle/bubble complex, the lower recovery of Aerofroth compared to SDS can also be explained by looking at the structure of both froths. The structure of the SDS froth was thicker and more stable than in the case of Aerofroth. Therefore, more particles were recovered.

A striking difference is noticed regarding the MT-OX Recovery with the pure (11–36%) and mixed (28.5–74.6%) samples. Apparently, the higher particle density in the mixed samples results in a better flotation efficiency. As most particles are initially carried from the mixing to the sedimentation zone, a different settling behavior of the MT-OX particles must exist. Most probably the breaking and reformation of MT-OX particle/bubble complexes in the flotation sedimentation zone is affected by the overall particle concentration.

CONCLUSION

By adding frother to the DA solution, instead of to the particle slurry lower frother amounts are sufficient to create small bubbles and good froth stability.

Small silica and MT-OX particles are well suited to test selective flotation of fine particles.

The good Recovery and a very high Grade, show that the new reactor is an excellent device to selectively float fine particles. With an appropriate frother the small MT-OX particles could be separated from the small hydrophilic silica particles. The best recovery (74.6%) was obtained with SDS. The most selective separation was achieved with Aerofroth. The grade of 98.1% showed that entrainment and entrapment of silica was negligible, even for the very small particles used.

In relation to the remediation of contaminated soil and sediments it may be expected that also the finest fractions can be cleaned from soot or coal tar particles. Next to the application in the remediation of contaminated soil, the flotation device with a sedimentation zone and a prevention that the settled particles return to the mixing zone, is probably a promising tool to separate fine particles and powders in other (industrial) applications.

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