Prosimy cytować jako: Inż. Ap. Chem. 2015, 54, 2, 46-47

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Impact of the ultrasonic preconditioning onto sedimentation process

Introduction

In the mining industry, as a result of mechanical extraction of minerals, fine grain size below 0.5 mm is often encountered. The solid matter is recovered by means of chemical (coagulation, flocculation etc.) and physical (sedimentation, filtration etc.) treatment. In order to increase efficiency of the separation process new, economical and effective solutions are researched. One of them is application of the ultrasounds. The goal of this work is to investigate the impact of the ultrasonic preconditioning of a model sludge onto sedimentation process. Moreover, the possibility of thermosensitive polymer application as stabilization/destabilization agent is assessed.

Experimental

The process of ultrasonic conditioning of the calcium carbonate model slurry was carried out using an ultrasonic stirrer *Sonics Vibra-Cell VCX-130*. Ultrasonic probe was immersed in a 40 [cm^3] slurry of the concentration of 220 [g/dm^3], the amplitude of ultrasonic wave was set to 114 microns and sonication time to 10 [minutes]. The average sonication power was equal to 20 [W] [*Sancewicz, 2014*]. After the sonication the slurry was diluted using Reverse Osmosis water.

In the flocculation experiments the solids content was equal to 12 [g/dm^3]. The suspension was mixed in a thermostated tank of volume equal to 740 [cm^3] (tank diameter was equal to the liquid height) by means of mechanical propeller (400 RPM, diameter equal to 0.038 [m]). As a destabilization agent a thermosensitive flocculant was used. It is characterized precisely in [*Lemanowicz et al., 2014*] (codename DADMAC1). In all experiments the polymer dose equal to 1 mg/g was used. The suspension was heated from the room temperature to 313 [K]. The particle size distribution was measured at different stages of the process.

When the particle mean diameter achieved the steady state at 313 [K] the sedimentation process began. Sedimentation tests were carried out in the 100 [cm^3] thermostated cylinders in order to maintain a constant temperature throughout the process. Thermostated cylinders were placed in a specially prepared laboratory stand, which allowed the study of the process of sedimentation by using digital image analysis.

Results

As it should be expected the ultrasonic conditioning of the slurry reduced the size of the particles (Fig. 1). During sonication, intense cavitation phenomenon took place within the sample. In the result the mean size of the sonicated chalk was 15% smaller comparing to the unmodified one. What is interesting, the volume fraction of the smallest particles were similar. The reduction of the mean particle size was at the cost of the breakage of the largest particles present in the suspension.

Surprisingly, the difference in the Particle Size Distribution (PSD) of the primal suspension practically had no influence on the flocculation process. The PSDs obtained for both unmodified and sonicated chalk at 40[°C] at the steady state are presented in Fig. 2. In both cases the *Sauter* diameter was equal approximately to 6.3 [μm]. In the presented experiments the aggregation phenomenon run on the basis of the hydrophobic interactions between polymer covered surfaces. This mechanism is discussed among the others in [*Imiela, 2014*].

These results indicate that ultrasonic conditioning of slurry does not enhance the flocculation process. On the other hand, the identical final result of aggregation, despite some difference in the initial PSD, may suggest that the process enhancement only recompenses the initial difference in size.

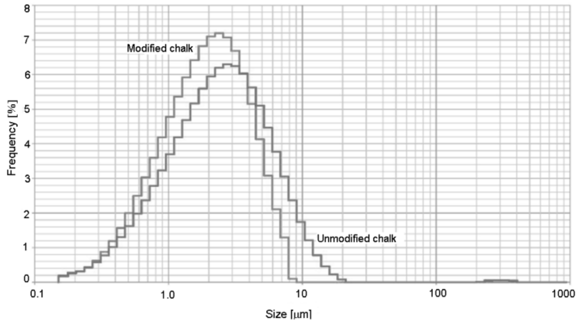

Fig. 1 Differences between a sonicated chalk and unmodified chalk

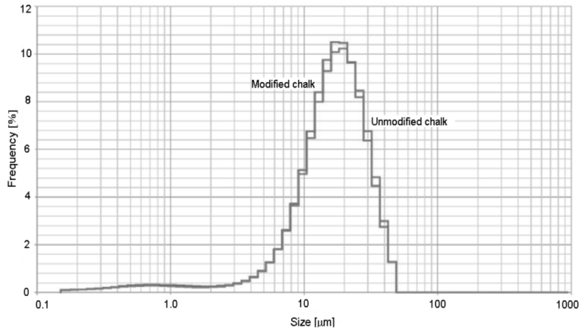

Fig. 2 Differences between a sonicated suspension and unmodified with flocculant

This phenomenon need further investigation.

The sedimentation tests were carried out in the same way as it was proposed by *Sakohara et al.* [*2013*] for thermosensitive polymers. The measurements were done for:

a) Suspension modified and unmodified at 25[°C],
b) Suspension modified and unmodified at 25[°C] with a flocculant in an amount of 1 [mg/g]
c) Suspension modified and unmodified at 40[°C],
d) Suspension modified and unmodified at 40[°C] with a flocculant in an amount of 1 [mg/g].

The sedimentation process was investigated by means of digital image analysis. For this purpose, the image registration process was done using digital camera at a resolution of 1920x1080 pixels and 29 frames per second. The preparation of the pictures required two main operations. In the first step the chosen pictures were extracted from video sequence to .jpg files. In the next step the region of sedimentation was cropped from the pictures. The main program was created in *Matlab* environment. The assumption of the program was to process each of picture to extract two regions: region of sedimentation and region of clarified liquid. Next the percentage of each region was calculated.

This process was carried out according to the following steps:

- loading pictures with sedimentation regions
- converting picture to the grayscale
- filtering with *Gauss* filter to remove noise
- images binarization
- morphological operation of erosion
- morphological operation of dilation
- saving the result images of sedimentation in grayscale and in binary scale.

Fig. 3 and 4 present the fraction of sediment which is defined as the ratio of sedimentation region (the number of white pixels) to the whole investigated region (the total number of pixels).

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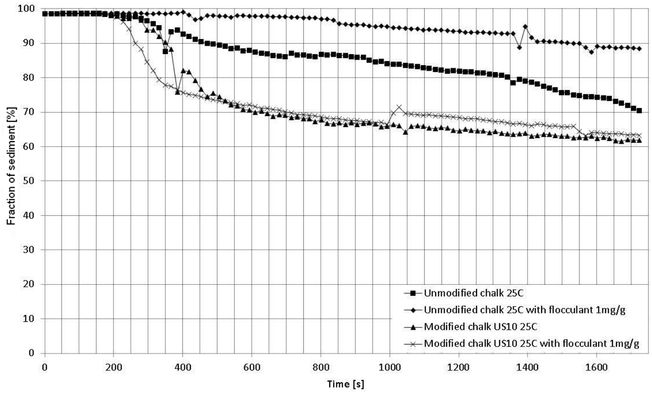

Fig. 3 Sedimentation test in 25^{o}C

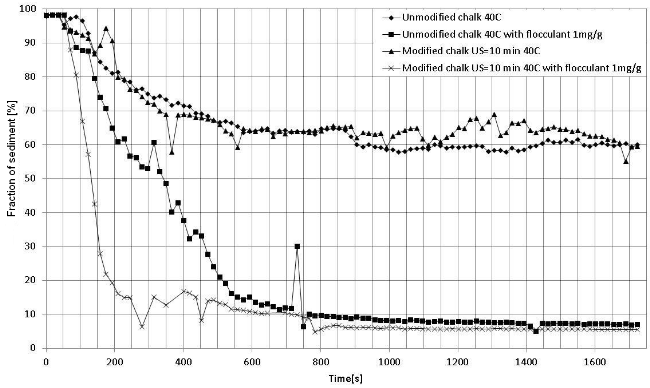

Fig. 4 Sedimentation test in 40^{o}C

Fig. 3 proves that the ultrasonic conditioning increased the rate of sedimentation of chalk without the addition of flocculant. At this point this fact was hard to explain – the presence of smaller particles should result in the opposite effect. On the other hand, it is worth to notice that at the temperature below the Lower Critical Solution Temperature the polymer addition raised the stability of the unmodified suspension. On the other hand, in the case of sonicated slurry the presence of polymer had no impact on the sedimentation. This situation changed drastically, when the temperature of the system was equal to 40[^{o}C], i.e. at the point when the thermosensitive polymer was active (Fig. 4). The addition of flocculant resulted in creation of large aggregates and therefore it led to faster sedimentation. What is more important the sonication of slurry increased the rate of this process, even though the particle size was the same. Simultaneously, the conditioning had no impact on the suspension without the addition of polymer. Another interesting observation was that the water above the border of turbidity in the case of the conditioned suspension was very clear comparing to the unmodified chalk.

There are a few explanations for the observed phenomena. Firstly, the presence of air filling the pores of calcium carbonate particles could influenced the sedimentation process. Ultrasonic conditioning of suspension in the first phase results in degassing of system. Therefore it could decrease the buoyancy of particles. In order to verify this theory a sample suspension was boiled (instead of ultrasonic conditioning) for a few minutes. Unfortunately, the liquid above the sediment was still turbid comparing to the conditioned system. Thus it was decided to analyze the particle porosity using nitrogen adsorption technique. The measurement was made at the Academy of Sciences in Gliwice. Fig. 5 shows that the both samples, i.e unmodified and sonicated one, were identical in terms of porosity.

The next step of presented research was the analysis of the shape of solid particles. For that reason two samples were prepared and dried. Next, they were photographed using Scanning Electron Microscope. In Fig. 6 one may notice that unmodified chalk particles had sharp edges and irregular shape whereas sonicated particles were more smooth and rounded. In our opinion that is the main reason of

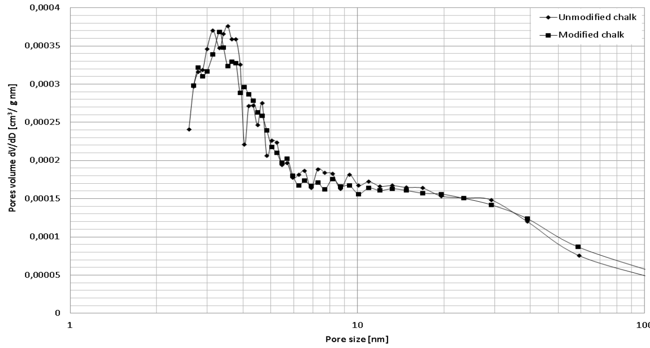

Fig. 5 Pores volume distribution

the difference between sedimentation of both suspension. The primal solid particles generated higher hydrodynamic resistance, therefore their sedimentation rate was lower comparing to sedimentation of more regular sonicated particles. The cavitation phenomenon simply polished the particles.

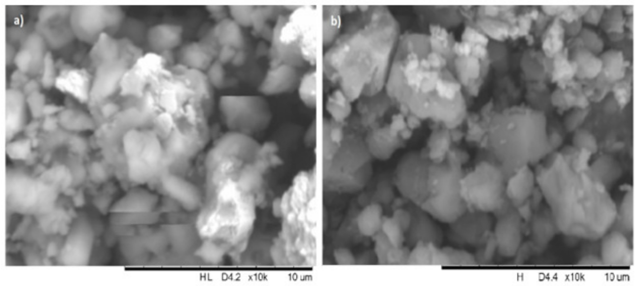

Fig. 6 Scanning Electron Microscope photographs:
a) Unmodified chalk, b) Modified chalk

Conclusions

In the presented research an impact of the ultrasonic preconditioning of a model suspension of calcium carbonate solid particles in Reverse Osmosis water on its sedimentation process was discussed. In experiments a thermosensitive polymer was used as the destabilizing agent. It was proved that the ultrasonic conditioning may be beneficial for the solid liquid separation processes although it decreases the size of particles. The cavitation phenomenon modifies the shape of particles enhancing their movement in liquid. Simultaneously the porosity of the particles was unchanged.

Another important conclusion is that the application of thermosensitive polymers proved to be successful in controlling the stability of a suspension. At low temperatures the sedimentation time was longer comparing to primal suspension whereas at higher temperature it was significantly shorter. What is more important this feature is fully controllable and reversible [*Imiela, 2014*]. The presented results may find application in different kinds of mineral processing, especially in the cases in which process water could be recycled.

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