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EFFECTS OF TEMPERATURE DURING ULTRASONIC CONDITIONING IN QUARTZ-AMINE FLOTATION

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Abstract: In this study, the effect of ultrasound on flotation recovery of quartz-amine flotation was investigated in terms of temperature influence. For this purpose, an ultrasonic probe was used for conditioning quartz surfaces in presence of dodecylamine hydrochloride (DAH), and the change in the temperature was recorded. The temperature-controlled ultrasonic conditioning tests were also carried out at various ultrasonic powers (30, 90, and 150 W) to investigate the effect of increasing temperature on the quartz-DAH flotation. The results showed that temperature of the suspension sharply increased from 23 up to 75 °C at the end of 10 min of conditioning at 150 W ultrasonic power. The flotation results for the temperature controlled and uncontrolled samples indicated that the flotation recovery increased from 45 to 65% by 90 W ultrasonic power. However, higher ultrasonic power levels affected the flotation recovery negatively. On the other hand, the ultrasonic application decreased the flotation recovery at all ultrasonic power levels in the temperature-controlled tests. Finally, the shape analysis was also performed for the particles treated with the ultrasound at various ultrasonic powers. As a conclusion, the positive effect of ultrasound on the quartz-amine flotation recovery could be related to temperature increase during conditioning.

Keywords: *Quartz, amine, flotation, ultrasound, temperature effect*

Introduction

Ultrasound is a sound wave, and used in mineral processing alternatively to improve flotation recoveries. While an ultrasonic wave is propagated in a liquid, it increases the ambient temperature up to 5000 K. It is above 20 kHz frequency, above a human perception limit. It needs a material medium to propagate as well as audible sound waves. While it does not show any significant effect in solid and air, it creates several extreme conditions in liquid such as cavitation. While an ultrasonic wave is propagated in a liquid, it creates a negative pressure that can break the bonds in liquid molecules, and hence forms bubbles. After that, these bubbles collapse during compressing a period of the ultrasonic wave. The gas and vapor in a collapsing bubble

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are in a compressed state. Heat generation of cavitation bubbles in compressing and collapsing phases occurs faster than heat transportation. Therefore, the temperature of the immediate vicinity of the bubble increases. Thus, some high temperature areas occur in the liquid called "short-lived hot spots". Although temperatures of these areas are very high, their durations are very short and sizes are very small according to the volume of the entire liquid. Therefore, the generated temperature disperses without doing any measurable change under the environmental conditions. Rayleigh (1917) developed a mathematical model which showed that very high temperature values can be reached in bubble collapses in liquids. Suslick (1989) calculated heating/cooling rate which was more than 1 billion °C/sec. On the other hand, Luque-Garcia and Luque de Castro (2003) reported 10 billion °C/sec.

It is impossible to measure such short-lived temperatures by a physical thermometer due to this measurement difficulty. Suslick (1989) and Suslick et al. (1999) used an alternative method that the theoretical temperatures could be calculated by experimental methods. The temperature of a definite chemical reaction is depended on the inverse logarithm of its rate. Therefore, the temperature can be calculated by observing the rate of a specific reaction. For this purpose, they observed some reaction rates of several specific reactions and calculated the temperature after a collapsing bubble reached up to 5000 °C (Mason, 1997; Alp, 1998; Mason and Lorimer, 2002; Gurpinar, 2007; Gungoren, 2009; Baig and Varma, 2012; Ensminger and Bond, 2012; Ozkan, 2012; Ozkan and Gungoren, 2012; Ozkan et al., 2015).

In literature, there are several studies on the effect of temperature on flotation. O'Connor and Mills (1990) studied the effect of temperature on pyrite flotation. Their results indicated that temperature increase caused the increase in the flotation rate of fast-floating pyrite particles and decrease in the water recovery and bubble sizes in the pulp. Lazarov et al. (1994) used quartz particles and glass ballotini for quartz flotation with amine. The authors reported that the increase of temperature lead to an increase of the flotation recovery of fine glass ballotini particles (40-70 μm). On the other hand, the temperature increase affected the flotation recovery of larger particles negatively. It was also stated that three-phase contact formation was the determining factor for the recovery increase.

Previous researchers carried out various studies on the use of ultrasound for flotation. Breitbach et al. (2003) investigated the effect of ultrasound on adsorption and desorption processes. The authors reported that the heat caused by ultrasonic application promotes desorption. Altun et al. (2009) investigated the enhancement possibilities of flotation of oil shale by ultrasonic treatment, and obtained a concentrate with lower ash contents. Cilek and Ozgen (2009) used ultrasound in the froth phase for various ores, and reported that the use of ultrasound in the froth phase of flotation decreased entrainment and increased selectivity. Ambedkar et al. (2011a, 2011b) studied the ultrasound-assisted process for sulfur and ash removal from coal, and they reported that the use of ultrasound appears to be a promising technique to remove ash and sulfur from coal based on the laboratory experimental studies.

Albijanic et al. (2014) studied the bubble–particle attachment mechanism for the DAH–glass system. The study indicated that maximum flotation recovery occurred at the isoelectric point of particles and bubbles, and hydrophobic force is the driving force of bubble–particle attachment. Barry and Klima (2015) and Barry et al. (2015, 2016) investigated the effect of ultrasonic cavitation on coal, and reported that ultrasonic cavitation reduced the overall size distribution of coal, increased the flotation recovery, decreased the ash value and increased the heating value of coal. Ahmad et al. (2016) developed a simple, accurate, economic and less time consuming ultrasonic washing process for clay minerals. Vasseghian et al. (2016) used low frequency ultrasound in a column flotation to remove ash and pyritic sulfur from bitumen, and reported that ash and pyritic sulfur removal drastically increased through the use of ultrasound. Videla et al. (2016) studied the effect of ultrasonic treatment on copper flotation, and explained the increase in the flotation recovery by facilitating the action of the reagents with the help of the cleaning feature of the cavitation. Gungoren (2016) studied the effect of ultrasonic energy on the particle-bubble interactions in quartz flotation, and reported that the use of ultrasound increased the flotation recovery as a result of increasing the contact angle and decreasing particle-bubble attachment time. No significant change was observed on the particle size, shape, roughness, BET surface area, and DAH adsorption of quartz. Gungoren et al. (2016) studied evaluation of coal preparation plant tailings by ultrasonic flotation and reported higher ash rejection recoveries and separation efficiencies in the experiments with ultrasound.

The aim of the present study was to investigate the effect of temperature during the ultrasonic conditioning in quartz-amine flotation. Within this scope, ambient temperature and pH values were measured during the ultrasonic application. The particle size and shape analyses were also carried out in addition to the micro-flotation tests.

Materials and methods

In this study, analytical grade quartz (particle size of 1.2×0.6 mm) purchased from Carl Roth Company was used for the experiments. The quartz samples were first reduced to 212×150 μm particle size by a close circuit automatic agate mortar. The XRD analysis of the sample indicated that only quartz mineral was the main mineral as seen in Fig. 1. The sample contains ~98% SiO_2, ~1.1% Al_2O_3, ~0.04% TiO_2, and ~0.02% Fe_2O_3 according to the chemical analysis. In literature, the cleaning of quartz samples with acid solutions is a widely used method to remove possible organic contaminants that may be present on the surfaces (Smith and Rajala, 1989; Yoon and Yordan, 1990; Scott and Smith, 1991; Koh and Smith, 2011; Albijanic et al., 2014). In the present study, the samples were cleaned with 2.5% H_2SO_4 by volume and 2.5% NaOH by weight, and then rinsed with de-ionized (*DI*) water in accordance with Koh and Smith (2011). This method has an advantage of using relatively low concentrations of acid and alkali solutions to prevent the quartz surfaces from damage.

In literature, 1-3% solid ratio of quartz was used in micro-flotation tests according to the experimental conditions (Gorman and Smith, 1991; Irannajad et al., 2009; Birinci et al., 2010; Ejtemaei et al., 2012). In this study, 2 g of 212×150 μm quartz samples were first conditioned with $2\cdot10^{-5}$ mol/dm^3 dodecyl amine hydrochloride (*DAH*). The analytical grade *DAH* (99% of $CH_3(CH_2)_{11}NH_2\cdot HCl$) was obtained from Arcos Organics Company. The conditioning processes were carried out at 1% solid ratio using DI water on a magnetic stirrer at 500 rpm for 10 min in the absence and presence of ultrasound. In the ultrasonic experiments, the ultrasound was applied at 20 kHz frequency and at various ultrasonic power levels (30, 90, and 150 W) with an adjustable ultrasonic probe (Bandelin HD 3200) with a magnetic stirrer.

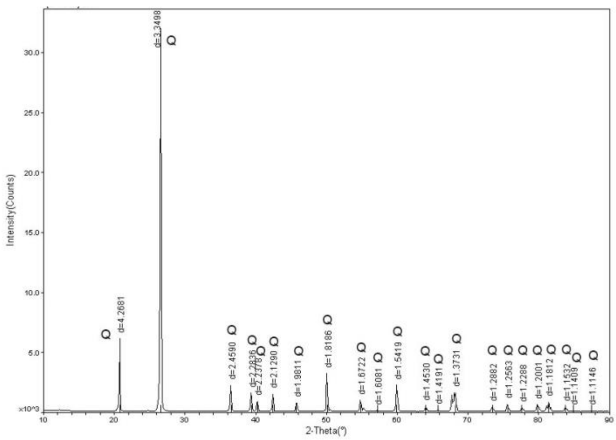

Fig. 1. XRD analysis of quartz sample (Q: Quartz)

No pH adjustments were conducted in the experiments. However, the pH of the suspension was monitored during the ultrasonic conditioning to investigate the effect of ultrasound on the pH. The temperature of the suspension was also measured with a digital thermometer. The conditioning process is given in Fig. 2.

In addition to the investigation of the effect of ultrasound on the suspension temperature, some temperature-controlled ultrasonic conditioning tests were also carried out to eliminate the effect of temperature on the conditioning by keeping the suspension stable at room temperature (23±1 °C). For this purpose, a temperature-controlled water circulator and a water jacketed beaker were employed. The suspension temperature was kept stable at room temperature by adjusting the temperature of the water in the circulator bath for each experiment, and the cooling water was circulated around the suspension. The experimental setup for the temperature-controlled ultrasonic conditioning is seen in Fig. 3.

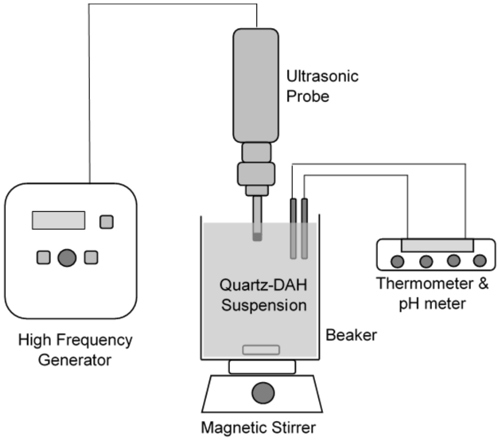

Fig. 2. Conditioning setup in the absence and presence of ultrasound

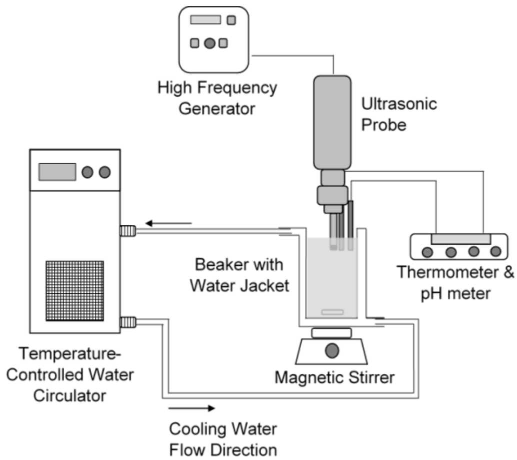

Fig. 3. Temperature-stabilized ultrasonic conditioning setup

After the conditioning processes, the micro-flotation tests were carried out. The suspensions of temperature-uncontrolled ultrasonic conditioning tests were kept for cooling down to the room temperature before the transferring the quartz-DAH suspensions to the micro-flotation cell. Nitrogen (N_2) gas and a micro-flotation cell with 200 cm^3 volume were used in the experiments, and all tests were performed at room temperature. The tests were performed at 50 cm^3/min N_2 flow rate for 2 min. After the micro-flotation tests, float and sink products were dewatered with a filter paper and dried at 105 °C in a drying oven. The micro-flotation recoveries were calculated using the product weights. The micro-flotation experimental setup and experimental conditions is seen in Fig. 4 and Table 1, respectively.

Within the scope of this study, the effect of ultrasound on the particle size and shape factor of quartz were also investigated. The particle size measurements were carried out using CILAS 1090 laser particle size analyzer for the quartz samples before and after ultrasonic application at 30, 90, and 150 W ultrasonic powers.

In a similar way, the shape factor analyses were performed for the samples before and after ultrasonic application at 30, 90, and 150 W ultrasonic powers. In addition, the analysis of the sample after temperature-controlled ultrasonic application at the highest ultrasonic power (150 W) was also carried out. The particle shape factors were calculated using Leica QWin image analyzing software.

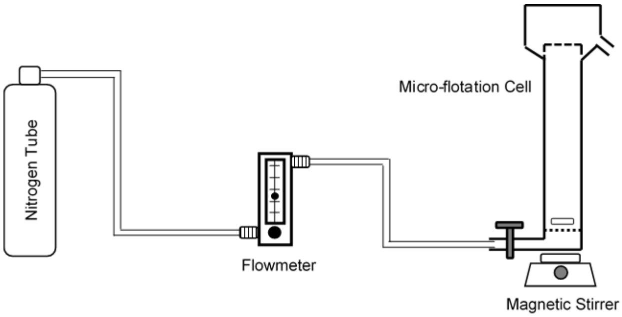

Fig. 4. Micro-flotation experimental setup

Table 1. Micro-flotation experimental setup

Parameters	Conditions
Cell colume	200 cm^3
Water	DI Water
Mineral mass	2 g
Particle size	212×150 µm
Solid ratio	1%
Conditioning stirring speed	~500 rpm
Gas (N_2) flow rate	50 cm^3/min
pH	Natural (6-6,5)
Surfactant, DAH	2×10^{-5} mol/dm^3 DAH
Conditioning time	10 min
Flotation time	2 min

Results and discussion

The change in the suspension temperature with respect to the ultrasonic application time is given in Fig. 5. As seen in Fig. 5, the suspension temperature increased with the ultrasonic power and time. The temperature increased to 31.9, 60.5, and 75.1 °C from room temperature (23.0 °C) at 30, 90, and 150 W ultrasonic powers, respectively, in 200 cm^3 suspension volume at the end of 10 min.

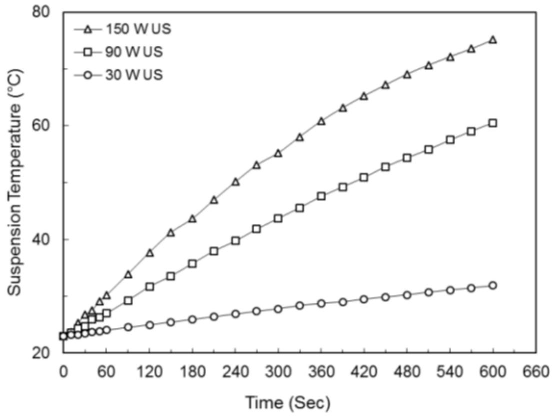

Fig. 5. Suspension temperature and emitted energy during ultrasonic conditioning

The pH measurements during the ultrasonic application are shown in Fig. 6. Although the natural pH of quartz was around 6, the measured suspension pH started around 5 because of the stirring factor. According to Fig. 6, it is seen that ultrasound showed no significant effect at 30 W. It is also interesting that the pH of the suspension slightly increased from 5 to 6 at 90 and 150 W. As known from literature, it can be attributed to the decomposition of the water molecule to free $[H^+]$ and $[OH^-]$ radicals during cavitation (Suslick, 1989; Kang et al., 2009). However, there is a need to conduct more research to understand this phenomenon. On the other hand, it is assumed that this pH change is not very important for amine flotation of quartz because the zeta potential of quartz is at negative values above around pH 2.

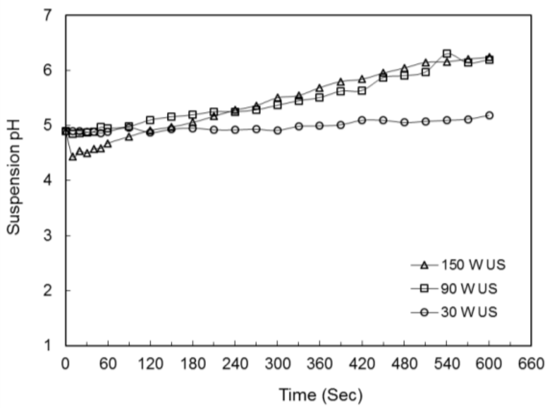

Fig. 6. Suspension pH during the ultrasonic conditioning

The comparison of the micro-flotation recoveries of quartz after the temperature-controlled and temperature-uncontrolled ultrasonic conditioning with respect to the suspension temperature is given in Fig. 7. As seen from Fig. 7, the micro-flotation recovery was 45.45% without the ultrasonic application. However, the micro-flotation recovery increased to 63.64 and 65.57% at 30 and 90 W, respectively, with the temperature-uncontrolled ultrasonic conditioning tests. On the other hand, it decreased to 37.50% at 150 W.

At the same time, when the temperature was kept stable in the temperature-controlled tests at 23 °C, it decreased to 42.13, 15.38 and 14.87% at 30, 90, and 150 W, respectively. These results indicated that the turbulent medium caused by ultrasound affected the DAH-quartz adsorption negatively.

The increase in the flotation recoveries can be attributed to the increase of the activity of DAH molecules with temperature. On the other hand, decrease in the flotation recoveries can be caused of the extremely turbulent medium during the ultrasonic application due to the inefficient adsorption of the surfactant on the particles.

The fact of obtaining lower flotation recoveries at higher ultrasonic powers in temperature-controlled tests than the tests without ultrasound, support these hypotheses, because of increasing turbidity with ultrasonic power. Meanwhile, the DAH molecules with increased activity at high temperatures could be absorbed on the quartz surface in spite of the turbulent medium until 150 W ultrasonic power. The turbulent at 150 W was too strong for adsorption. However, these hypotheses must be proven by additional research to understand the mechanism behind this.

The particle size measurements of the quartz samples before and after 10 min of ultrasonic application at various ultrasonic powers (30, 90, and 150 W) are seen in Fig. 8. It is seen from Fig. 8 that the d_{80} and d_{50} particle sizes of the samples are around 212 and 118 µm respectively, and the ultrasound shows no significant effect on the particle size of quartz at any ultrasonic power.

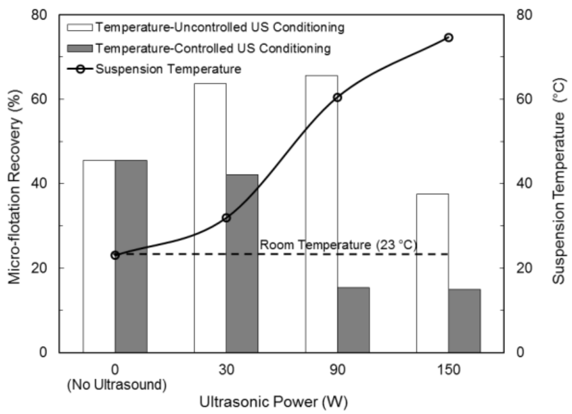

Fig. 7. Suspension temperature in conditioning and micro-flotation recoveries of quartz after temperature-controlled and temperature-uncontrolled ultrasonic conditioning

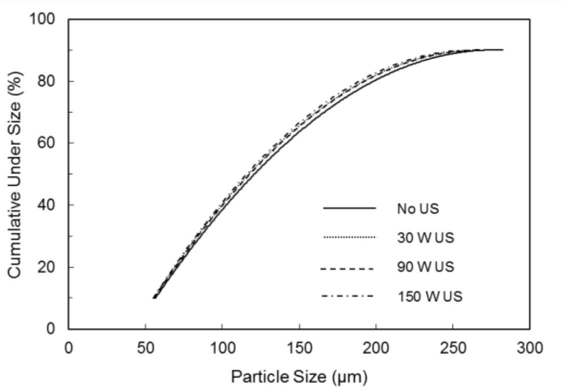

Fig. 8. Particle size measurements of the quartz samples before and after 10 min of ultrasonic application at various ultrasonic powers (30, 90, and 150 W)

The particle shape analysis for the samples before and after the ultrasonic application at various ultrasonic powers (30, 90, and 150 W) is seen in Fig. 9. Meanwhile, the comparison of particle shape factors of the samples before the ultrasonic application, after the temperature-controlled and temperature-uncontrolled ultrasonic application at 150 W is given in Fig. 10. These tests were conducted at 150 W ultrasonic power to investigate the effect of ultrasound at the maximum power. As seen from Figs. 9 and 10, the ultrasound did not make any remarkable changes in the particle shape factors of quartz particles. Therefore, it was though that there was no need for the tests for lower ultrasonic powers in temperature-controlled and uncontrolled.

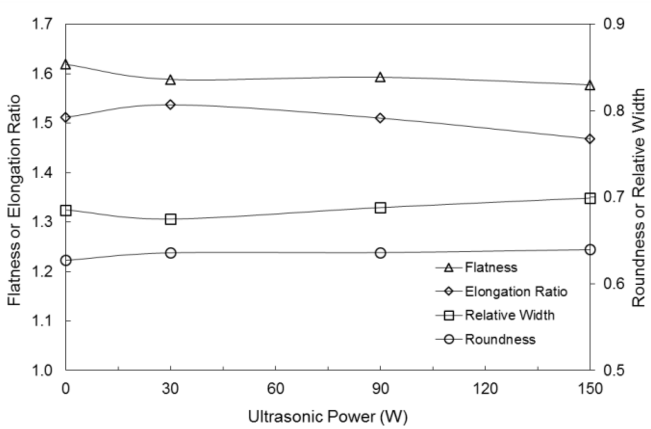

Fig. 9. Particle shape analyses of samples before and after ultrasonic application at various ultrasonic powers (30, 90, and 150 W)

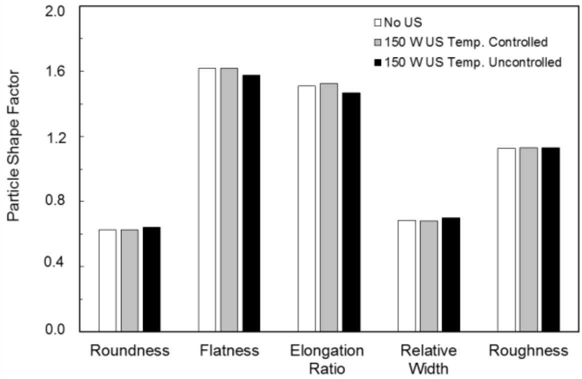

Fig. 10. Particle shape analyses of samples before the ultrasonic application, after temperature-controlled, and temperature-uncontrolled ultrasonic application at 150 W

Conclusions

In this study, the role of temperature during ultrasonic conditioning in quartz-amine flotation was investigated with temperature and pH measurements, the micro-flotation tests, particle size and shape analyses.

The temperature and pH results showed that the suspension temperature in the conditioning increased with the ultrasonic power and application time. The temperature increased linearly, then the increment slowed down with respect to the ultrasonic power and application time.

The micro-flotation test results showed that the micro-flotation recoveries of the samples after the temperature-uncontrolled conditioning were higher than that after the conditioning without the ultrasound application. However, the micro-flotation recoveries significantly decreased when the temperature was kept at room temperature during the ultrasonic conditioning. Thus, the increase in the micro-flotation recovery after the ultrasonic application seems to depend on the temperature increase in the conditioning.

Overall, it can be concluded that the effect of ultrasound on the pH of quartz can be neglected, and the increase in the temperature increase may cause an improvement in the activity of amine molecules in the flotation system.

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