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## Investigating the effects of ultrasonic energy on the flotation behavior of pyrite and galena minerals

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**Abstract:** Although pyrite is one of the more abundant minerals of the earth crust, it has low economic value. When it reports to the concentrate during flotation along with the valuable minerals, it decreases the grade of the valuable minerals and leads to an increase in smelting costs. Numerous modifications have been suggested in the literature to increase the selective recovery of pyrite containing base metal-sulfide ores. The use of ultrasonic applications is one such method. In this study, the effect of the ultrasonic application on the flotation behavior of galena and pyrite mineral was investigated through systematic Hallimond Tube experiments. In the initial phase of the experiments, the optimum flotation conditions (particle size, pH, amount of air, and amount of reagent) were determined for the two minerals. Subsequent experiments were carried out under these optimums to distinguish the effect of the ultrasonic application. The influence of how the ultrasonic application was applied (i.e. before and during the conditioning stage or before the re-flotation of the concentrate) was also studied. It was observed that the ultrasonic application had a strong activating influence if it was administered before or during the conditioning stage. The effect was similar to whether the minerals were floated individually or from their mixtures. However, when it was applied to a flotation concentrate before re-flotation, it selectively displayed a depressant action for the pyrite to the extent that no depressants were needed. The results conclusively showed that the ultrasonic application could drastically improve the selectivity of the complex ores.

**Keywords:** galena, pyrite, ultrasonic energy, flotation, Hallimond tube

### 1. Introduction

In sulfide ores which are significant sources of base metals (galena, chalcopyrite, sphalerite, etc.), pyrite is usually considered to be an unwanted gangue mineral (Chandra and Gerson, 2010; Huang et al., 2013; Moslemi and Gharabaghi, 2017; Ozun et al., 2019). In sulfide-ore mining, pyrite is removed from valuable minerals by flotation (Li et al., 2012; Taguta et al., 2018). As pyrite decreases the purity of concentrates obtained especially from the flotation of valuable minerals such as galena, sphalerite, and chalcopyrite, it needs to be cleaned carefully (Mu et al., 2016). Therefore, pyrite is separated from valuable minerals by depression using various depressants at high pH values (Bicak et al., 2007). Significant amounts of fine pyrite particles may be found related to ore types in the concentrates of galena, chalcopyrite, and especially sphalerite (Cao et al., 2018). To obtain high-purity concentrates, there is a need for various depressants, complicated flotation set-ups, and high-performance flotation devices (Vasanthakumar et al., 2017; Jia et al., 2019).

Ultrasonic application is used in the various areas of industry. In the mining sector, it is usually used in surface cleaning processes (Zhao et al., 2007; Videla et al., 2016). Ultrasonic application refers to sound waves that vibrate at a higher frequency than 20 kHz (Farmer et al., 2000; Chen et al., 2020). The prevalently used frequency range of ultrasonic application energy is about 20–100 kHz (Donskoi et al., 2012; Kursun, 2014; Ghadyani et al., 2018). It is proven that ultrasonic application shows considerable

effects on the mineral surfaces such as cleaning, degradation, reduction, oxidation, activation, etc. It is also used for reactive emulsification (Gungoren et al., 2017).

The ultrasonic application creates micro-bubbles in the pulp with sizes of around 100  $\mu\text{m}$  (Gungoren et al., 2019). These bubbles cavitate on the mineral surface, creating pressures and temperatures in the order of 100–5000 atmosphere (Pa) and 1000–5000°C (Zhou et al., 2009). In the mineral pulp, this phenomenon forms liquid jets with the speeds in the order of 100 m/sec. These jets can remove surface coatings from minerals, and even eliminate oxidation (Kang et al., 2009, Cao et al., 2017). Therefore, the use of the ultrasonic application has been investigated by several studies for improving flotation efficiency before flotation (Aldrich and Feng, 1999; Celik et al., 2002) or during flotation (Ozkan and Kuyumcu, 2006, Cilek and Ozgen, 2009, Ozkan et al., 2012; Gungoren et al. 2017; Ghadyani et al., 2018; Ozun et al., 2019). Nevertheless, a systematic study on the effects of ultrasonic application, especially for mineral mixtures, will be an important contribution to the literature.

In this study, the effects of ultrasonic application on the flotation behavior of galena and pyrite minerals, separately and together from a mixture, were investigated by using a Hallimond Tube. The study focused on different modes of ultrasonic application: Before and during the conditioning stage as well as its application to the floated particles before re-flotation. Flotation products were also examined by Fourier-transform infrared spectroscopy (FTIR) analyses.

## 2. Materials and methods

### 2.1. Materials

Galena and pyrite samples obtained from the Black Sea region in Turkey were used for the flotation experiments. Representative photographs of the samples are shown in Fig. 1. The mineral compositions of the samples were determined by an X-Ray Diffractometer (Rigaku; Model Miniflex II). Chemical analyses of the samples were carried out by an Atomic Absorption Spectrometer (Perkin Elmer; Model PinAAcle 500). The results of the XRD and AAS analyses showed that pyrite and galena samples had 98% and 99% purity, respectively.

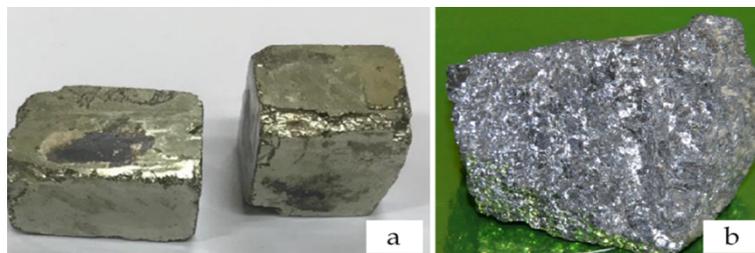


Fig. 1. Pictures of (a) pyrite and (b) galena samples used in this study

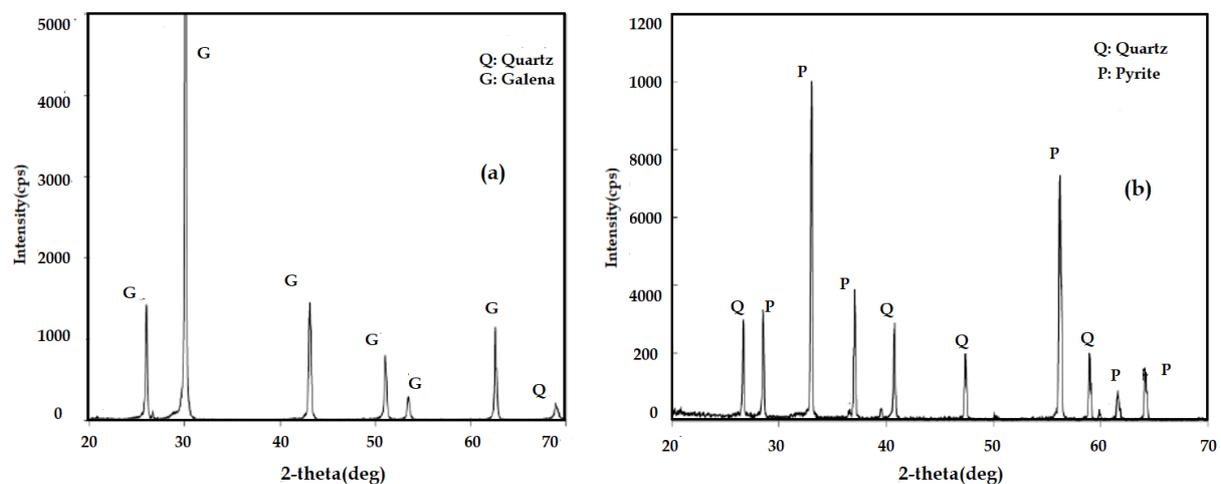


Fig. 2. XRD analysis results of (a) pyrite and (b) galena samples

The changes in the level of collector coverage on the mineral surfaces before and after flotation experiments were investigated using FTIR (Thermo; Model Nicolet Nexus 470).

FTIR is one of the best techniques to determine whether or not there is a change in the chemical composition of surface atoms and/or the collector molecules through the vibration of their bonds by the absorption of FTIR light ( $400\text{-}4000\text{ cm}^{-1}$ ) wavenumbers (Jiao et al., 2019).

## 2.2. Micro-flotation experiments

The micro-flotation experiments were carried using the Hallimond Tube. The set-up seen in Fig. 3 was used for the flotation experiments. An ultrasonic homogenizer with a frequency range of 20 kHz (Maoan-Model UH-250 XO 800 S) was the source for the ultrasonic application (Figs. 4 and 5).

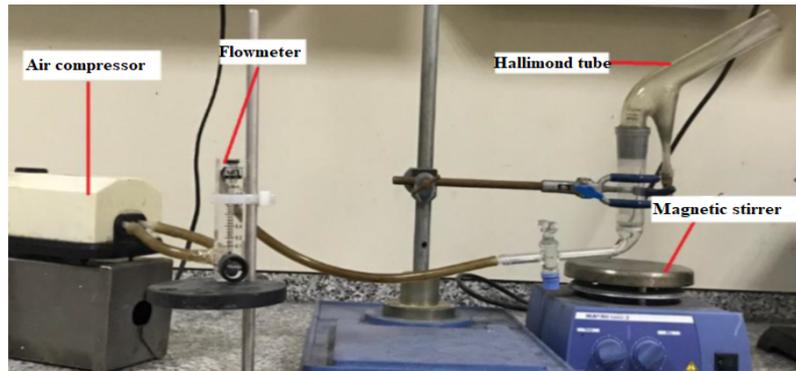


Fig. 3. Experimental set-up for the flotation experiments



Fig. 4. XO-800S Ultrasonic homogenizer



Fig. 5. Ultrasonic energy probe and sample

The general conditions applicable to all flotation experiments were as follows: Potassium Amyl Xanthate (KAX) was used as a collector.  $\text{H}_2\text{SO}_4$  and  $\text{NaOH}$  were used for pH adjustment. Preliminary experiments showed that the best size range for the flotation of these minerals in a Hallimond Tube which was particle size interval "150-75  $\mu\text{m}$ ". A total of 1 g sample was used in each experiment throughout the test work. (Drzymala, 1999; Osasere, 2000; Ansari and Pawlik, 2007). To simulate the actual flotation conditions, the flotation experiments were carried out with tap water.

The general methodology applicable to all flotation experiments was as follows: The conditioning of the particles with a proper amount of KAX was carried out following the pH adjustment to the desired level in a 0.1 dm<sup>3</sup> tap water for 2 min using 1 g sample. The conditioned suspension was then transferred to the Hallimond Tube for flotation. The airflow rate was between 0.1-0.5 dm<sup>3</sup>/min.

While the complete floating of the galena particle size was completing in 1 min, the floating of the pyrite particle size was completed in 5 min. For this reason, the duration for the whole flotation was chosen as 5 min. The float fractions were filtered through a band filter paper (Reeve angel model, type 305 and size 12.5 cm) and dried in 105°C in an oven. The recovery was determined from the float and feed fraction weights if the flotation was carried out in separate galena or pyrite feeds or by microscopic counting if the flotation was carried out from a galena-pyrite mixture. The recovery was used directly as flotation recovery since the pyrite and galena particles were nearly pure.

### **2.3. Determining the optimum conditions for the flotation of pyrite and galena**

Micro-flotation experiments were carried out separately with pyrite and galena at a range of pH, amount of KAX, and amount of air to determine the optimum flotation conditions for both minerals. The ranges tested were 250-500 g/Mg KAX and pH 5-8 for pyrite (Bıcak et al. 2007) and 50-250 g/Mg KAX and pH 7-9 for galena (Wilss, 2007). Ultrasonic application was not used in this section.

### **2.4. Determining the effect of ultrasonic application for pyrite and galena flotations**

The effect of ultrasonic application on the flotation recovery was studied separately with both the pyrite and galena particles. The influence of ultrasonic application on selectivity and recovery was studied in detail under various modes.

In the first set of experiments, the ultrasonic application was applied to the particles before conditioning with the collector (following the pH adjustment). The durations for the ultrasonic application were 5, 10, 15, 30, 60, and 120 sec. The pulp was conditioned for 2 min with KAX after ultrasonic application, and the suspension was transferred to the Hallimond Tube for flotation for 5 min.

In the second set, the particles were subjected to the ultrasonic application for the same durations but this time in the presence of KAX. The total conditioning time was the time for the ultrasonic application plus 2 min. These two modes intended to determine the influence of the application thorough cleaning of the surface from the secondary particles or any other contaminants such as oxide coating for better collector adsorption.

For the third set, the particles were floated for 5 min following the pH adjustment and 2 min of KAX conditioning under the optimum conditions. The float fraction was re-dispersed in 100 cm<sup>3</sup> distilled water and subjected to ultrasonic application for the same duration (5, 10, 15, 30, 60, and 120 sec). The suspension was transferred to the Hallimond Tube and re-floated for an additional 5 min. The purpose of this mode was to distinguish the effect of the application on the tenacity of the collector coverage on pyrite and galena surfaces.

### **2.5. Determining the effect of ultrasonic application for the flotation of pyrite and galena from mixtures**

In this section, the flotation experiments were repeated; but this time with 50-50 pyrite-galena mixtures (by mass) to compare the degree to which the application affects the mineral surfaces when they are floated from a mixture. In the experiments with the individual samples, the ultrasonic application displayed a depressant effect on pyrite whereas there was no difference in the galena response. Since this could be a way of selectively separating the two minerals, the flotation experiments were performed that the same effect could be observed when the two minerals were present as a mixture. Only two modes of ultrasonic application, before conditioning and products floating after the flotation, were used in this section. In the experiments with the individual samples; as the ultrasonic application was observed the same effect before conditioning and during to conditioning. In this section preferred before conditioning. The flotation experiments were carried out under the optimum conditions obtained for

the galena (pH of 6, 500 g/Mg KAX and 0.1 dm<sup>3</sup>/min the amount of air ). The flotation recovery of each mineral was determined by microscopic counting.

### 3. Results and discussion

#### 3.1. Determining of optimum conditions for the flotation of pyrite and galena

Tables 1 and 2, and Fig. 6 show the results of the flotation experiments carried out with pyrite and galena minerals separately in the absence of ultrasonic application under a range of conditions. Figure 6(a) shows the optimum pH was determined to be 6 and 8 for pyrite and galena, respectively. The amount of KAX (pyrite 500 g/Mg and galena 250 g/Mg) and the amount of air for both minerals 0.1 dm<sup>3</sup>/min were used for Fig.6(a). Figure 6(b) shows that the flotation recovery increased with the amount of the collector for both minerals. pH and the amount of air for both minerals 0.1 dm<sup>3</sup>/min was used for Fig.6(b). On the other hand, the recovery decreased with increasing airflow rate as seen in Fig. 6(c). Amount of KAX (pyrite 500g/Mg and galena: 250 g/Mg) and pH (pyrite 6 and galena 8 was used for Fig.6(c). According to the results the optimum flotation conditions and respective recoveries were determined as follows:

- Pyrite: 500 g/Mg KAX, pH=6, and 0.1 dm<sup>3</sup>/min airflow rate → 81% recovery
- Galena: 250 g/Mg KAX, pH=8, and 0.1 dm<sup>3</sup>/min airflow rate → 84% recovery

Table 1. Results of the experiment to determine the optimum flotation conditions of the pyrite mineral

Solid Amount (g)	Air Amount (dm <sup>3</sup> /min)	pH	Collector Amount (g/Mg)	Recovery (%)
1	0.1	6	100	15
1	0.1	6	200	16
1	0.1	6	300	22
1	0.1	6	400	76
1	0.1	6	500	81
1	0.1	5	500	24
1	0.1	5.5	500	51
1	0.1	6.5	500	73
1	0.1	7	500	36
1	0.5	6	500	45
1	0.2	6	500	57
1	0.3	6	500	49
1	0.4	6	500	48

Table 2. Results of the experiment to determine the optimum flotation conditions of the galena mineral

Solid Amount (g)	Air Amount (dm <sup>3</sup> /min)	pH	Collector Amount (g/Mg)	Recovery (%)
1	0.1	8	50	46
1	0.1	8	100	57
1	0.1	8	150	82
1	0.1	8	200	83
1	0.1	8	250	84
1	0.1	7	250	71
1	0.1	7.5	250	77
1	0.1	8.5	250	80
1	0.1	9	250	72
1	0.5	8	250	51
1	0.2	8	250	79
1	0.3	8	250	78
1	0.4	8	250	58

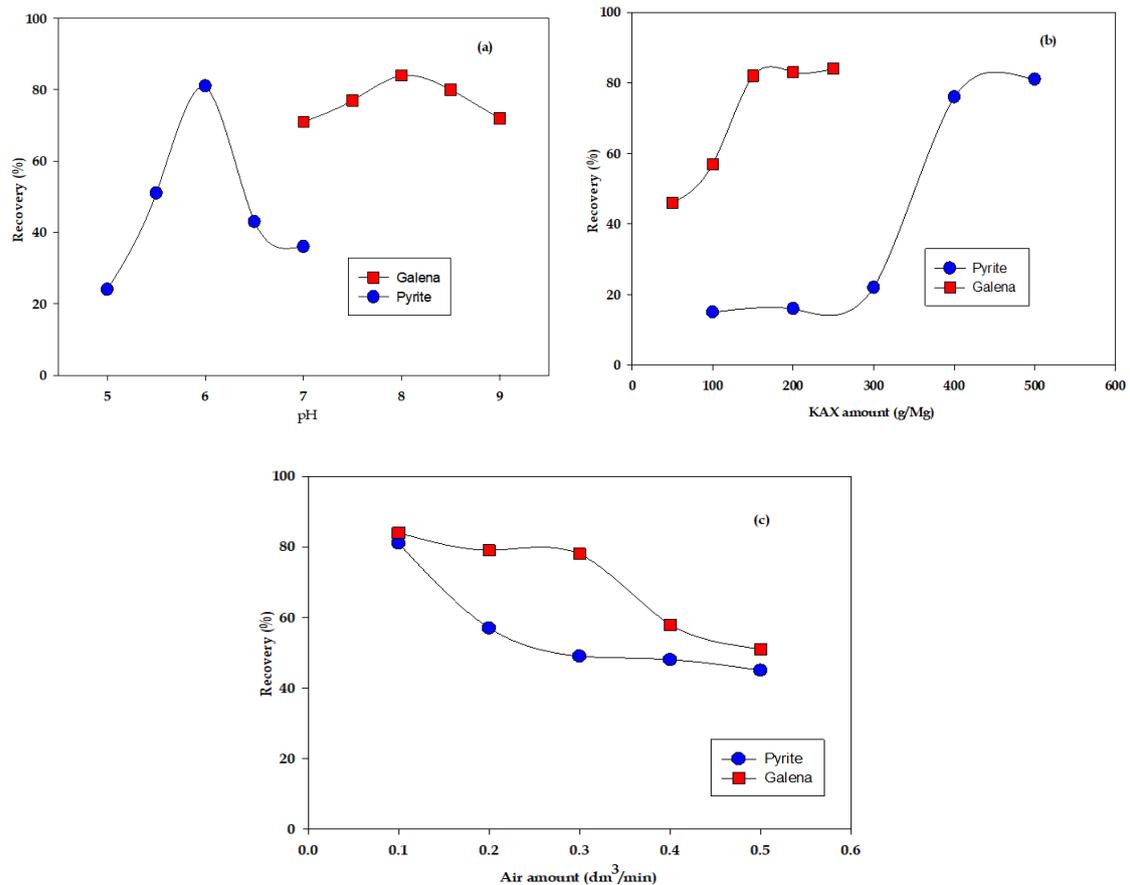


Fig. 6. Effect of (a) pH (b) collector, and (c) air amount on flotation recoveries of pyrite and galena

### 3.2. Effect of ultrasonic application for separate pyrite and galena flotations

The collective results of the experiments are given in Fig. 7 for both minerals. It can be seen from Fig. 7 that the flotation recovery increased slightly for both minerals with the application time when the particles were subjected to the ultrasonic application before and during conditioning. This is most probably due to the progressive removal of the secondary particles or oxide layers from the particle surfaces by the ultrasonic application leading to a better collector adsorption. This observation is in line with the previous studies where the ultrasonic application was found to result in higher recoveries at better selectivity when applied before and during flotation (Aldrich and Feng, 1999; Celik et al., 2002; Ozkan, 2006; Ozkan and Kuyumcu., 2006; Cilek and Ozgen, 2009; Kang et al.; 2009; Ozkan et al., 2012; Gungoren et al., 2017; Cao et al., 2017; Ghadyani et al., 2018; Ozun et al., 2019; Gungoren et al., 2019; Chen et al., 2020).

When the ultrasonic application was applied to the floated particles followed by the re-flotation of these particles, the flotation behavior of the two minerals separated very sharply. While the pyrite recovery decreased towards 15% immediately, the galena recovery remained over 70% even for long durations of the ultrasonic application (Fig. 7).

A sharp decline in the pyrite recovery with the application of ultrasound suggested that the collector coverage on the pyrite surfaces was less strongly attached compared to galena; the ultrasonic application was sufficient for removing the collector coverage from the pyrite surfaces, leading to a relatively more hydrophilic mineral surface. It is known that ferric-dixanthogen,  $\text{Fe}(\text{AX})_2$ , is responsible for the flotation of pyrite (Gaudin, 1976). FTIR spectra of ferric xanthate, dixanthogen, and pyrite conditioned with xanthate indicated that dixanthogen was the only species that can be determined on the pyrite surface (Bulut and Atak, 2002). Adsorption tests showed that the highest level of xanthate adsorption was in the acidic range, yet higher adsorption densities were detected in neutral solutions compared to alkaline conditions. This also explains the floatability behavior of the pyrite particles at around pH 6 in Fig. 6(a). That the solubility of ferric dixanthogen on the pyrite surface ( $\text{Fe}(\text{AX})_2 = 1.3 \times 10^{-}$

$5 \text{ mol/dm}^3$ ) is many orders of magnitude higher than the solubility of lead dioxanthogen ( $\text{Pb}(\text{AX})_2 = 6.8 \times 10^{-17} \text{ mol/dm}^3$ ) on the pyrite surface is in favor of this argument.

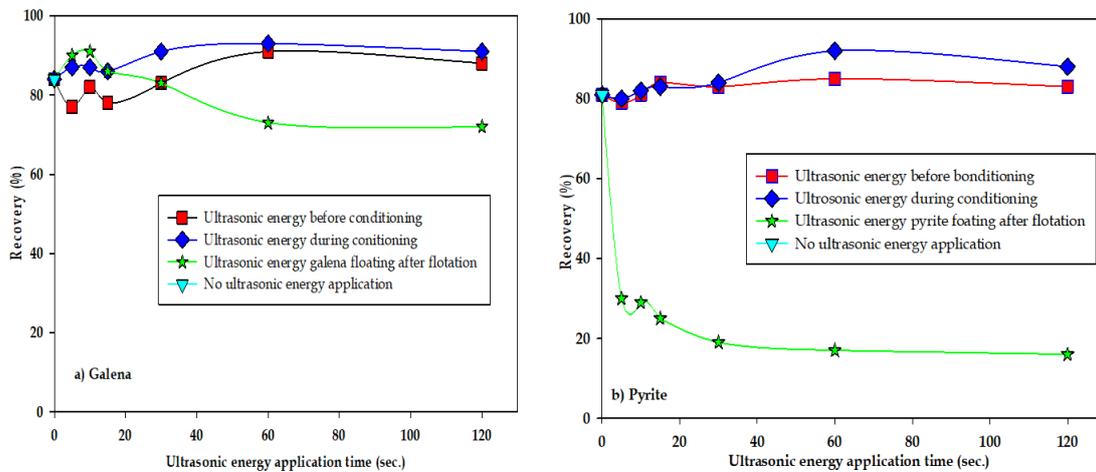


Fig. 7. Effect of ultrasonic conditioning on flotation recovery of (a) galena and (b) pyrite

### 3.3. Effect of ultrasonic application for the flotation of galena and pyrite from mixtures

The experiments for the previous section were repeated with the mixtures of galena and pyrite. The pH of the flotation experiment was kept at 6. The results of these experiments are shown in Fig. 8. It can be seen from Fig. 8 that when the ultrasonic application was applied to the pyrite/galena mixture before the conditioning stage (Fig. 8(a)), both minerals initially displayed increased recoveries (from 80% to 95%). The recovery remained over 80% with extended application of the ultrasonic energy; which meant that there was practically no selectivity in separating the two minerals. It seems that when the ultrasonic application was applied before the conditioning, both minerals benefited similarly from its action; most probably cleaning of the surface from the secondary particles or oxide layers, both of which reduced the effectiveness of the collector adsorption.

When the ultrasonic application was applied to the flotation products followed by re-flotation, two minerals could be separated sharply from each other (Fig. 8(b)). The recovery for the pyrite was quickly dropped below 20% while the recovery of the galena was close to 80% even for extended times of ultrasonic application. According to these results, it is obvious that the two minerals are differently affected by the ultrasonic application even when they are present in a mixture, and could be selectively separated from each other. This observation is extremely important since it may pave the way for much better selectivity in the flotation of pyritic complex ores, especially in the cleaner circuits, when the ultrasonic application is used in conjunction with other depressant recipes.

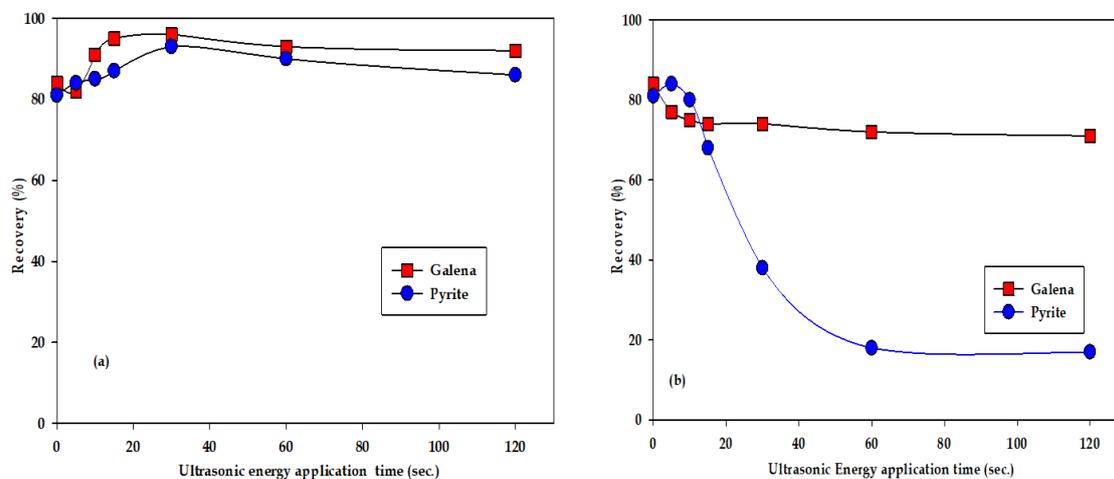


Fig. 8. Flotation recovery of galena and pyrite in (a: before conditioning b: product floating after flotation)

### 3.4. FTIR analyses

The infrared spectra of the floated particles are shown in Fig. 9 for different modes of ultrasonic application. The xanthate presence should be detected on pyrite and galena around 1098-1196  $\text{cm}^{-1}$  as a C-O-C stretching band (Hosseini et al., 2007). Figures 9(a) and (b) clearly show the xanthate presence in the 1098-1196  $\text{cm}^{-1}$  region when the ultrasound was applied before and during the conditioning step, indicating a better surfactant coverage for both minerals. When the ultrasonic energy is applied to float products, this band seems to be preserved on the galena surface whereas it is absent on the pyrite surface.

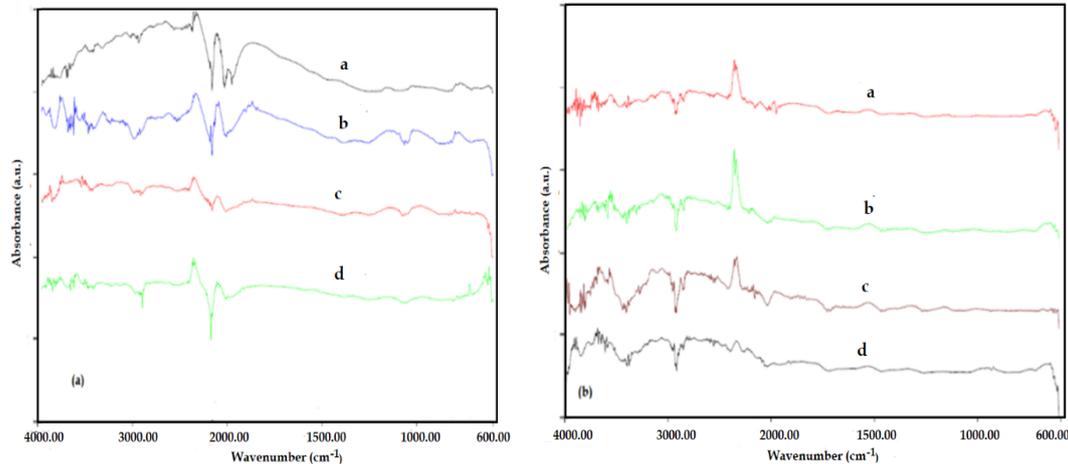


Fig. 9. FTIR analyses for (a) galena and (b) pyrite: (a) Before conditioning (b) During to conditioning (c) No ultrasonic energy application (d) products floating after flotation

### 4. Conclusions

According to the results obtained from this study, the following can be concluded:

- i. The ultrasonic application was observed to increase the flotation recovery of the pure galena and pyrite particles when it was applied before or during the conditioning stage. It showed an activating effect of the mineral surfaces most probably due to the removal of secondary particles or oxidic layer which hamper surfactant adsorption. The optimum time for the application was quite fast, around 60 sec for both cases.
- ii. When it was applied to the float products before re-flotation, the effect of the ultrasonic application on the two minerals was drastically different. While the recovery remained still high for galena even for extended application duration (around 80%), it displayed a swift decrease (below 20%) for the pyrite after the first 10 sec.
- iii. The effect was similar if the two minerals were floated separately or together from their mixtures.
- iv. These results suggest that the ultrasonic application may be employed to increase the recovery in the rougher stage through its action of cleaning the mineral surfaces. By proper tuning, it can also be employed as a pyrite depressant in the cleaner stages.

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