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Investigation of the frother effect in two and three phases systems on bubble size, surface tension, recovery and grade in chalcopyrite flotation

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Abstract: In this study, the effect of frother was investigated in two and three phases in the systems of the flotation. While the two-phase system consisted of liquid and gas, the three-phase systems contained a chalcopyrite ore. The study of three-phase systems was performed with the ore on a laboratory and plant scale. Effect of the amount and type of the frothers, their mixtures, and pH were examined depending on the bubble size, grade of the concentrate, and the recovery of chalcopyrite flotation. The results showed that as the amount of frothers increased, there was a reduction in the bubble size in all experiments. Additionally, the frother mixtures gave a positive effect on the chalcopyrite flotation. One of the most important purposes of flotation frothers shrinks the air bubble. As can be understood from the tests this time reduction of the frothers bubble size has a positive effect on the flotation. Likewise, it increases the foam stable value. It is observed from this study that increasing the amount of frothers decreases the surface tension and bubble size at different pH.

Keywords: chalcopyrite, bubble size, surface tension, recovery, grade

1. Introduction

Frothers are heteropolar surface-active compounds containing a polar group and a hydrocarbon radical, capable of adsorbing in an air-water interface. Frothers play several roles in the flotation process (Drzymala et al., 2018). In the literature, there are several different classifications of frothers depending on their properties and behaviour in solution (Klimpel and Hansen, 1988; Laskowski, 2003; Melo and Laskowski, 2006; Bulatovic, 2007; Khoshdast and Sam, 2011; Drzymala et al., 2018).

The most widely used commercial frothers include natural oils such as terpineol (as in pine oil) and cresols, C5–C8 aliphatic alcohols, polypropylene glycols and their alkyl ethers (commonly referred to as polyglycol frothers), mixed ethers, aldehydes, and ketone co-products of oxo alcohol production, and alkoxyalkanes such as TEB (tri-ethyl-butane), Cappuccitti and Finch (2008). Polyglycols are the strongest surface-active frothers utilized. Examples of these include polypropylene glycol methyl ethers and the Dowfroth (DOW) range of frothers which include common flotation frothers such as Dowfroth 200, 250, and 1012. Glycol methyl ethers are low viscosity liquids that are completely or partially soluble in water. Alcohol frothers are mixtures of alcohols containing 5–8 carbon atoms. The most common frothers from this group are methyl isobutyl carbinol (MIBC), and 2-ethyl hexanol. Aliphatic alcohol frothers are used as mixtures of different carbon lengths and as a mixture of hydrocarbon oils, Bulatovic (2007). It is known from the literature that the efficiency of flotation depends on the use of a frother to control the bubble size (Finch et al., 2006; Bulatovic, 2007; Gupta et al., 2007; Khoshdast and Sam, 2011; Karakashev et al., 2020, Guven et al., 2020; Karakashev et al., 2021; Batjargal et al., 2021). The effect of frothers on bubble size has been investigated in detail (Cho and Laskowski, 2002a; Laskowski, 2003). In addition, for each type of ore treated by flotation, there is likely to be an optimum bubble size distribution that will produce the optimum recovery at the highest flotation rate (Dobby and Finch,

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1991; Batjargal et al., 2021). Thus, controlling the generation of bubbles to achieve an optimum size range in a flotation cell is highly attractive since it may improve the recovery of the flotation process by optimizing the collection of particles, i.e., via better size-by-size flotation (Grau and Laskowski, 2006). It is also known that there are some studies in the literature on the effects of frother blends on flotation (Ngoroma et al., 2013; Elmahdy and Finch, 2013; Dey et al., 2014; McFadzean et al., 2016; Bayram et al., 2018). It is observed that frother blends can be more effective than single frothers in achieving the best technical and economic advantage (Tan et al., 2016).

These are mainly heteropolar organic reagents that provide power to air bubbles to transport the collected hydrophobic particles as foam products. It is known that the surface tension of the solution decreases due to the heteropolar nature of the reagent when the frother is added to water. As the surface tension of the solution decreases, more stable foams are produced and bubble coalescence in the pulp phase is prevented (Klimpel and Hansen, 1988).

When the surface area of a dense phase increases, the physical or chemical bonds that hold the atoms together are broken, since no atom or molecule can separate itself from neighbouring atoms the system does a job during the increase of the surface area. The resistance of the atoms against the expansion of the phase boundaries causes the newly formed surface to shrink as much as possible. Thus, when the mobility between atoms or molecules increases, that is, from solid to liquid, it becomes easier for the substance to shrink its surface. The reversible work done by the unit increase of the interface between two phases is called surface tension (Atak, 1982).

As mentioned above, many studies are available for the characterization of frothers in terms of different parameters such as bubble size, concentration, etc., to our knowledge only a few studies have been reported for the fundamental mechanism besides controlling the bubble size which is not directly related to surface tension decrease (Sweet et al., 1997; Machion et al., 1997; Aldrich and Feng, 2000; Grau and Laskowski, 2006). A common explanation is the prevention of coalescence (Laskowski, 2003) but this mechanism of frother action relevant to bubble generation (as opposed to froth formation) remains obscure (Finch et al., 2006). The role of frother is not so clear and is to provide finer bubbles, stabilize the froth, and enhance bubble-particle attachment (Leja and Schulman, 1954; Leja, 1956/57)

In this context, this study aimed to reveal the effects of frother type, amount, mixtures, pH, and surface tension on bubble size, the recovery, and grade of flotation products by utilizing measurements at both laboratory and plant scales.

2. Materials and methods

2.1. Materials

The ore sample used in this study was obtained from Siirt-Madenköy, Turkey. The chemical analysis of the sample is presented in Table 1. As a result of mineralogical analyses of the sample, which are the basis for experimental studies, pyrite mineralization containing chalcopyrite magnetite and sphalerite was determined in the ore composition. Pyrite was observed as the main component in polishing sections, both free and as a locked particle with all minerals in the ore. The other common mineral was chalcopyrite, usually, chalcopyrite phases locked with cataclastic pyrite structure, and chalcopyrite was seen to fill in the fractures of pyrite as a matrix material as well as it showed a colloidal structure with pyrite. Figure 1 shows an example of the polishing section of the ore.

Table 1. Chemical analysis of Siirt-Madenköy ore sample

Element	(%)
Cu	1.183
Pb	0.036
Zn	0.187
Fe	42.71
S	39.67
Al	0.500
Ca	0.290
Mg	0.910

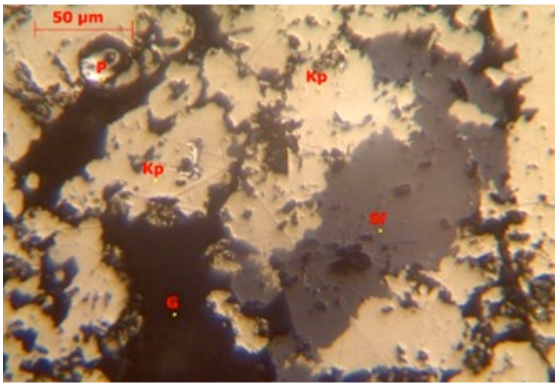

Fig. 1. In the polishing section of the sample, small pyrite (P) and sphalerite (Sf) occurrences in scattered white, which appear white in the body of chalcopyrite (Kp) predominant coarse, dark-colored cracks are filled with gang (G) minerals

Siirt-Madenköy copper plant ore content is around 1.5% Cu and a concentrate has been produced with 20%Cu by selective flotation. 1.670.000 tons of ore are processed annually and 100.000 tons of copper concentrate is obtained. In the Siirt-Madenköy copper plant, Aerophine 3418 A (collector), MIBC, (frother) sodium silicate (depressant), and lime (pH modifier) are used as the flotation reagents. The effect of four different frothers such as MIBC, Pine Oil, AF 70, and Dowfroth 250 was investigated in this study. Table 2 shows frothers tested in this study.

Table 2. Frothers used in this study

Common Name	**Purity**	**Chemical Formula**	**Molecular weight (g/mol)**
MIBC	Technical	$(CH_3)_2CHCH_2CHOHCH_3$	102.18
AF 70	Technical	$(CH_3)_2CHCH_2CHOHCH_3$	102.00
Pine Oil	Technical	$C_{10}H_{16}$	136.24
Dowfroth 250	Technical	$CH_3(C_3H_6O)_4OH$	264.37

2.2. Methods

In this study, some studies were carried out to measure bubble sizes during the flotation experiments (three-phase system study) and without any solid phase (two-phase systems). The experiments were performed at natural pH in a two-phase system, the flotation experiments were applied at pH 12 in three-phase systems. In order to examine the effect of the bubble size on copper grade and recovery, the studies were conducted on both laboratory and plant scales.

Flotation experiments in the laboratory scale were carried out in a Denver flotation machine with a constant speed at 1300 rpm. In each experiment, the sample was conditioned in a 2 dm^3 cell. Then, Aerophine 3418 A was used as the collector for two stages adding (25+25 g/Mg) in every experiment, and four types of frothers were used at two stages with different amounts as 12.5+12.5, 20+20, 25+25 g/Mg. Finally, the flotation products were collected for 5 min at pH 12, and chalcopyrite was separated from pyrite and gang minerals, selectively. In the Siirt-Madenköy copper plant, 20 m^3 Outotec tank cells have been used in the rougher circuit while 10 m^3 Outotec tank cells in cleaner circuits. All tests performed in the laboratory and plant scale were investigated on rougher circuits of chalcopyrite separation. The Image J analysis program has recently found wide fields of study in various sciences (Uslu and Tafakhori, 2013). During the experiments conducted on the laboratory scale, the photos of the bubbles were taken from the top of the flotation cell. The bubble sizes from these photos were measured using the Image J analysis measurement program Approximately 30 bubbles were measured for each condition. For these experiments, the experimental setup and procedure defined in a recent publication were used in the literature (Guven et al., 2020; Batjargal et al., 2021).

For example, first, a scale is placed on the flotation cell, which is measured, and then average bubble size is measured by taking measurements from the same height with a camera and evaluating them in the Image J program. Fig. 2 shows the bubble size measurement method, and Fig. 3 shows the images from the measurements with different frother types.

Also, the bubble size measurements were made by the same method by taking a photograph in Siirt-Madenköy copper plant cells. The bubble size was measured selective- rougher circuit in the plant to be compatible with laboratory measurements. In the literature, the measurements of surface and interface tensions are carried out by static rupture and dynamic methods. In this study, the surface tensions of different types of frothers were measured by the Du-Noüy ring method using the Krüss K6 tensiometer (KRÜSS GmbH, Hamburg, Germany). As seen in Fig. 3, after the platinum-iridium ring was completely immersed in the liquid it was pulled upwards until it reached the maximum force at the point where the liquid and the ring were separated from each other was found by reading from the scale this value was determined as 72.5 mN/m for pure water.

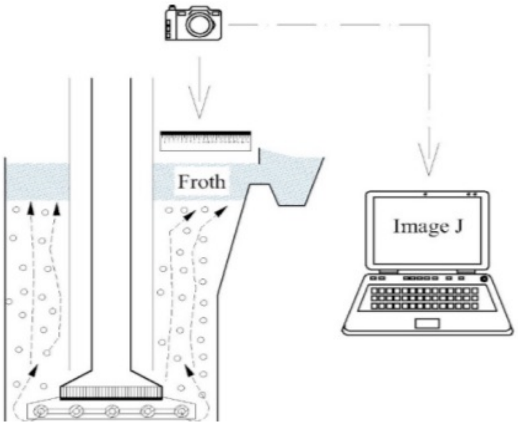

Fig. 2. Schematic presentation of bubble-size measurement units

Fig. 3. Images from measurements with different frother types

3. Results and discussion

3.1. Measurements in two-phase systems (Effect of frothers amount, type, and mixtures)

In these experiments, the amount of frothers and the effect of frothers type on the bubble size were examined. The results are given in Table 3 and shown in Figs. 5, 6, and 7. In two-phase experiments,

Fig. 4. Krüss K6 tensiometer

when the amount of frothers increases, the bubble size decreases in all frothers reagents. In two phases experiments showed that Dowfroth 250 produced the smallest bubble size as shown in Fig. 5.

Also, the effects of frother mixtures were investigated in two-phase systems. Binary mixtures were prepared so that the frothers were half and half in a total dosage of 50 g/Mg. The usages of frothers for single and binary mixtures in the same total amount of frothers are shown in Figures 6 and 7. It can be seen that the smallest bubble size was obtained by the mixture of Dowfroth 250 +MIBC. Based on the results obtained from mixing frothers reagents in equal amounts, the MIBC and Dowfroth 250 mix were used to produce a smaller bubble size than they were alone.

Table 3. Effect of frothers amount types and mixtures on bubble size in two-phase systems

Amount of Frother (g/Mg)	**Frother Type**	**Bubble Size (cm)**
0		0.262
30	MIBC	0.225
	AF 70	0.254
	Pine Oil	0.232
	Dowfroth 250	0.134
50	MIBC	0.201
	AF 70	0.226
	Pine Oil	0.205
	Dowfroth 250	0.111
70	MIBC	0.196
	AF 70	0.213
	Pine Oil	0.199
	Dowfroth 250	0.100
25+25	MIBC+ AF70	0.213
	MIBC+ Pine Oil	0.198
	AF 70+ Pine Oil	0.216
	Dowfroth 250+MIBC	0.104

3.2. Measurement of three-phase systems (Effect of frothers amount, type, and mixtures of three-phase systems)

The results were obtained in three-phase systems in which the amounts, types, and mixtures of frothers were investigated with the ore on a laboratory scale. In the plant scale, only MIBC amounts were examined. The results are given in Table 4, and Fig. 8 shows the results of frother type and amount effect on the bubble size and the grade of copper concentrate. Additionally, Fig. 9 shows the results of

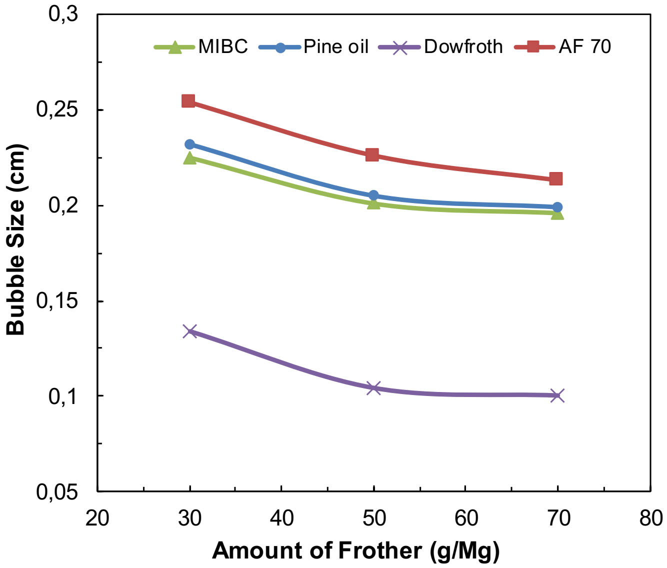

Fig. 5. Effect of amount of frothers amounts on bubble size for two-phase systems

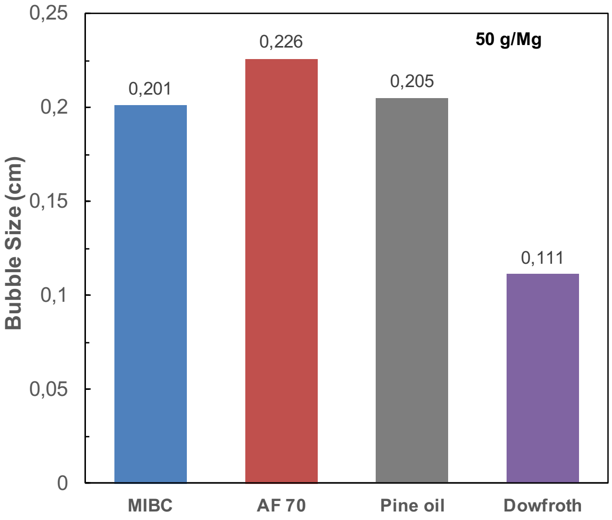

Fig. 6. Effect of single frothers at 50 g/Mg on bubble size for two-phase systems

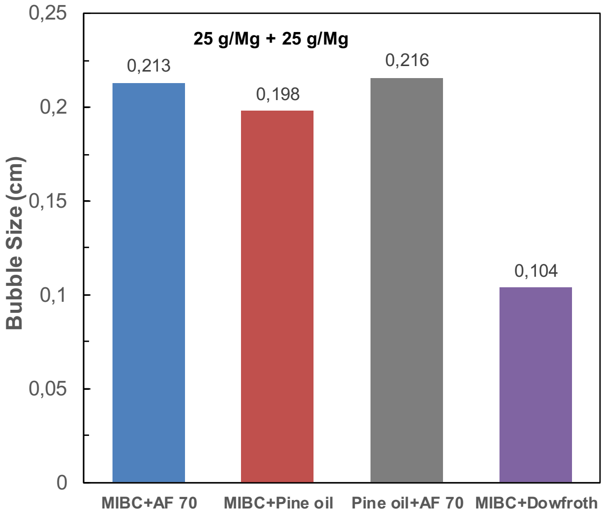

Fig. 7. Effect of binary frothers mixtures at 25 + 25 g/Mg on bubble size for two-phase systems

Table 4. Effect of amount and type of frothers on bubble size concentrate grade, and recovery in three-phase systems

Experimental Medium	Frothers	Amount (g/Mg)	Bubble Size (cm)	Concentrate Grade (%Cu)	Concentrate Recovery (%)
Laboratory	MIBC	25	0.480	4.52	88.07
	AF 70		0.445	3.94	87.27
	Pine Oil		0.427	3.59	86.76
	Dowfroth		0.859	7.89	85.62
	MIBC	40	0.386	4.26	90.87
	AF 70		0.394	3.66	89.40
	Pine Oil		0.379	3.41	88.99
	Dowfroth		0.664	7.18	87.15
	MIBC	50	0.341	4.07	91.72
	AF 70		0.366	3.48	90.17
	Pine Oil		0.326	3.28	89.85
	Dowfroth		0.543	6.63	88.04
	MIBC + AF 70	15+15	0.397	4.17	90.28
	MIBC + Pine Oil	15+15	0.372	4.01	89.71
	Pine Oil + AF 70	15+15	0.425	3.82	88.74
	Dowfroth + MIBC	15+15	0.785	5.93	84.61
In Plant	MIBC	25	0.695	15.12	87.37
		40	0.590	13.25	88.79
		50	0.454	11.83	90.35

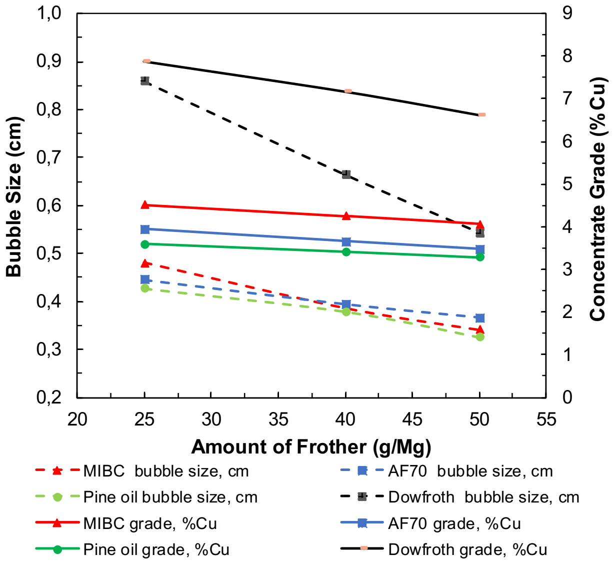

Fig. 8. Effect of amount and types of frothers on bubble size and concentrate grade in three phases system in laboratory scale

frother type and amount effect on the bubble size and the recovery of the copper concentrate.

The results showed that when the amount of frother increases, the bubble size decreases for all frothers. The same situation was observed on the plant scale using MIBC. The general trend for all investigated frothers is fairly similar, the bubble size decreases with the increasing frother amount at a particular concentration, the bubble size levels off (Gupta et al., 2007). As seen in Fig. 8, Dowfroth 250 has the largest bubble size at all dosages comparing the other frothers, unlike Pine Oil. When Dowfroth 250 was used, the highest copper grades were obtained, while these grades were the lowest with Pine

Oil. It can be concluded from these results that there is a direct proportion between bubble size and concentrate grade. The experiments conducted using MIBC at different amounts in the plant scale also confirmed this result. As also shown in Fig. 9, the diameter of the bubble size decreases, and the recovery increases depending on the amount and type of the frothers. The high recovery values were achieved with MIBC in all frothers concentrations. The lowest recovery was obtained with Dowfroth 250.

The effect of frother mixtures on the concentrate grade and bubble size in the laboratory scale is shown in Fig. 10 where the highest concentrate grade was achieved by the mixture of MIBC+Dowfroth 250. The lowest concentrate grade value was obtained with the mixture of Pine Oil+ AF 70. Figure 11 shows the results for the effect of frother mixtures at 30 g/Mg as a ratio of 1:1 (15 + 15 g/Mg) on bubble size and recovery in the laboratory scale experiments. The results showed that the best recovery was achieved using MIBC+AF 70. The lowest recovery was obtained using MIBC+ Dowfroth 250.

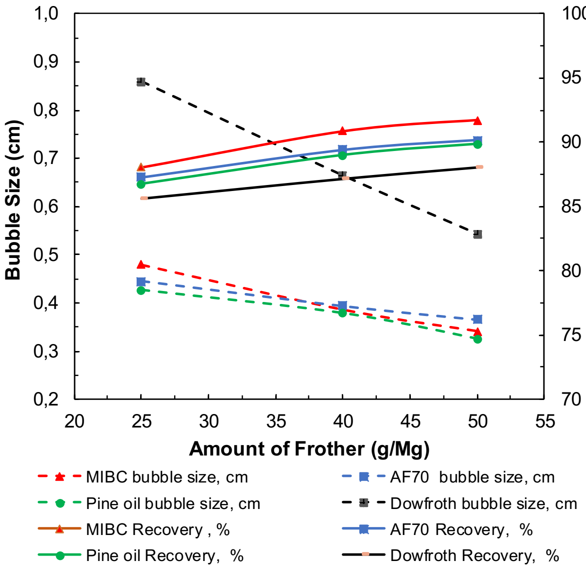

Fig. 9. Effect of amount of and types of frothers on bubble size and flotation recovery in three phases system in laboratory scale

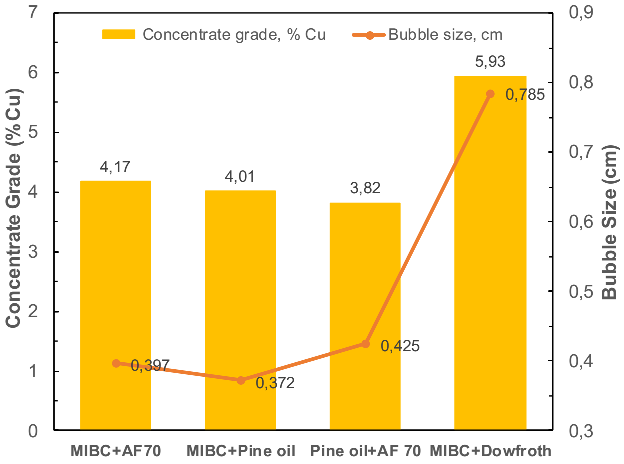

Fig. 10. Effect of frother mixtures on bubble size and concentrate grade

3.3. Effect of pH

The effect of pH on bubble size, foam height, copper grade, and recovery was investigated in laboratory and plant scales using 3418 A (50 g/Mg) as collector and MIBC (25 g/Mg) as the frother. The results for the effect of pH in the laboratory and plant scales are given in Table 5. Figs. 12 and 13 show the effect of pH on bubble size, concentrate grade, and recovery in laboratory and plant scale experiments. Accordingly, as the pH increases, the diameter of the bubble size decreases in both plant and laboratory conditions. According to the results given in Table 5, it was observed that the foam height increased as the pH increased. It was also found that the copper grade and recovery increased as pH increased. The increase in the recovery of rough concentrates was due to pH to increased Cu grade by depressing pyrite at high pH. The increasing recovery also caused bubble sizes to decrease.

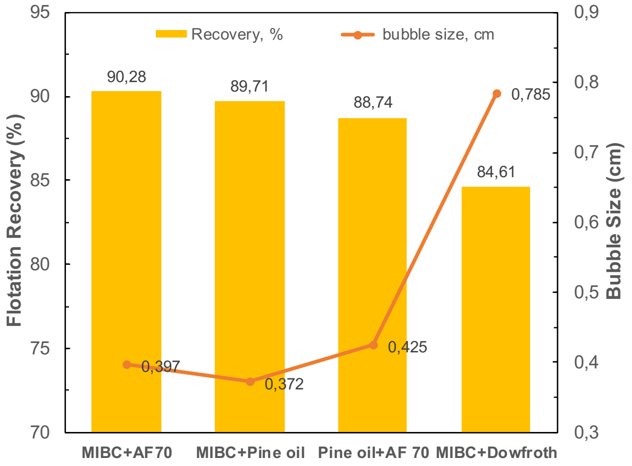

Fig. 11. Effect of frother mixtures on bubble size and concentrate recovery

Table 5. Effect of different pH on bubble size, concentrate grade, and recovery

Experimental Medium	pH	Bubble Size (cm)	Foam Height (cm)	Concentrate Grade (%Cu)	Concentrate Recovery (%)
In Laboratory	10.5	0.869	2.0	3.51	73.30
	11.0	0.690	2.1	3.87	76.37
	11.5	0.535	2.3	4.37	81.07
	12.0	0.432	2.6	4.78	85.68
In Plant	10.5	0.845	18.0	13.44	74.57
	11.0	0.734	21.0	15.33	80.46
	11.5	0.600	25.0	16.47	83.15
	12.0	0.449	29.0	17.65	86.69

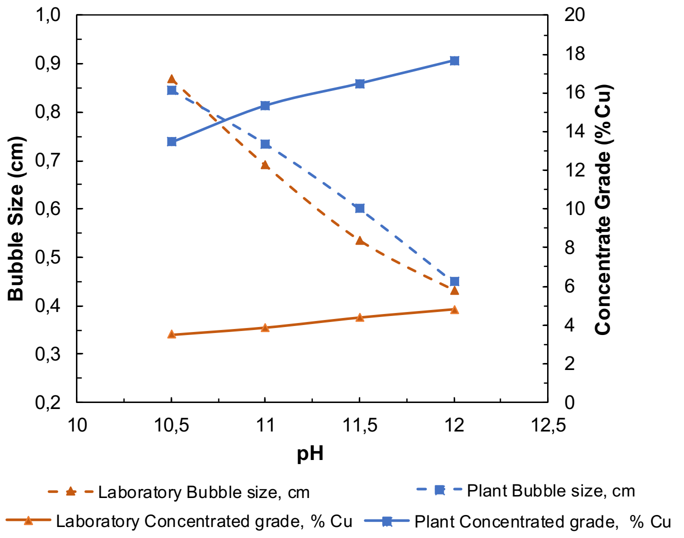

Fig. 12. Effect of pH on bubble size and concentrate grade for laboratory and plant scale experiments

As seen from Figs. 12 and 13 that both measurements were conducted in the laboratory and the plant, and the bubble size decreases due to an increase in pH. In contrast, the copper grade and recovery increased both in the plant and in the laboratory experiments since the sample from Siirt Madenköy is studied at high pH in the plant environment and selective flotation is performed, pyrite is depressed at high pH, the concentrated grade is much higher than the flotation performed in the laboratory scale, the bubble sizes in both scales are close to each other.

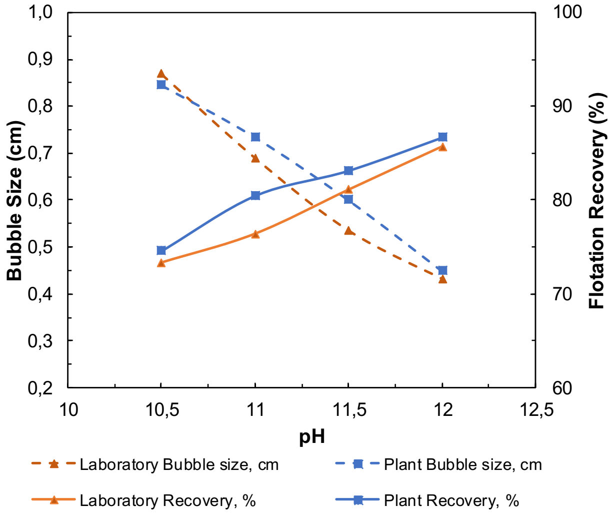

Fig. 13. Effect of pH on bubble size and flotation recovery for laboratory and plant scale experiments

3.4. Effect of surface tension

In these experiments, the effects of different frothers on surface tension were investigated. In the measurements, frothers types, amount, and their effect on surface tension at different pH were examined as presented in Table 6. Figures 14 (a), (b), and (c) show the effect of frothers at 25, 50, and 75 g/Mg amounts at natural pH, and Figs. 15 (a), (b), and (c) at pH 12. And, Table 7 shows the properties of frothers used in the surface tension measurements.

It is clearly observed that increasing the amount of frothers decreases the surface tension and bubble size at natural pH. Additionally, increasing the amount of frothers decreases the surface tension and bubble size at pH 12. It is understood that both bubble size and surface tension are lower than the values at natural pH in the measurements made at pH 12. In the remaining four frothers except for Pine oil, the surface tension decreases with the increasing the PH. The surface tension of Pine oil increases as the pH increases. The biggest difference seen in the surface tension change due to pH change was obtained for MIBC, while the least change was for Dowfroth 250.

Table 6. Effect of different frothers, pH on bubble size, and surface tension

Amount of Frother (g/Mg)	Frothers	Natural pH Surface Tension (mN/m)	Bubble Size (cm)	pH:12 Surface Tension (mN/m)	Bubble Size (cm)
25	MIBC	72	0.225	64	0.114
	F 549	64	0.133	61	0.117
	Pineoil	59	0.232	64	0.137
	Dowfroth 250	60	0.134	60	0.145
50	MIBC	70	0.201	63	0.098
	F 549	65	0.120	59	0.102
	Pineoil	54	0.205	63	0.108
	Dowfroth 250	58	0.104	55	0.104
75	MIBC	69	0.196	63	0.080
	F 549	64	0.113	58	0.097
	Pineoil	49	0.199	60	0.101
	Dowfroth 250	55	0.100	56	0.099

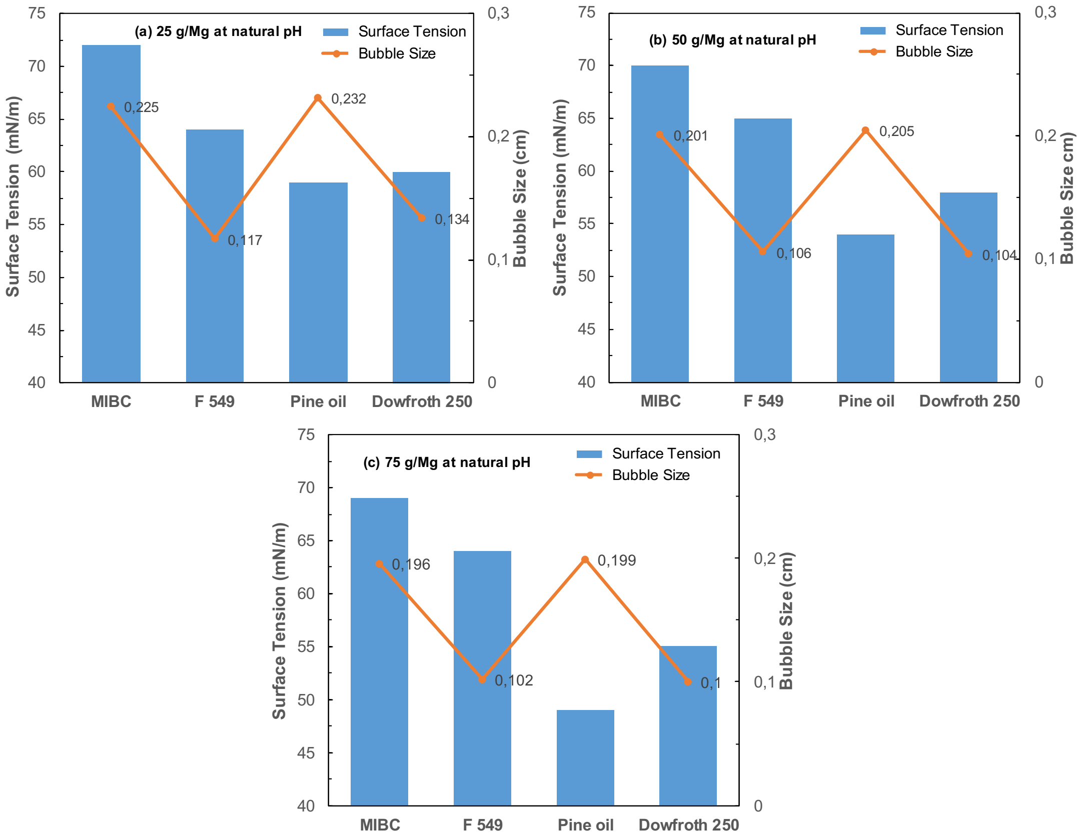

Fig. 14. Effect of type of frothers at natural pH on bubble size and surface tension at (a) 25 g/Mg (b) 50 g/Mg, and (c) 75 g/Mg amounts

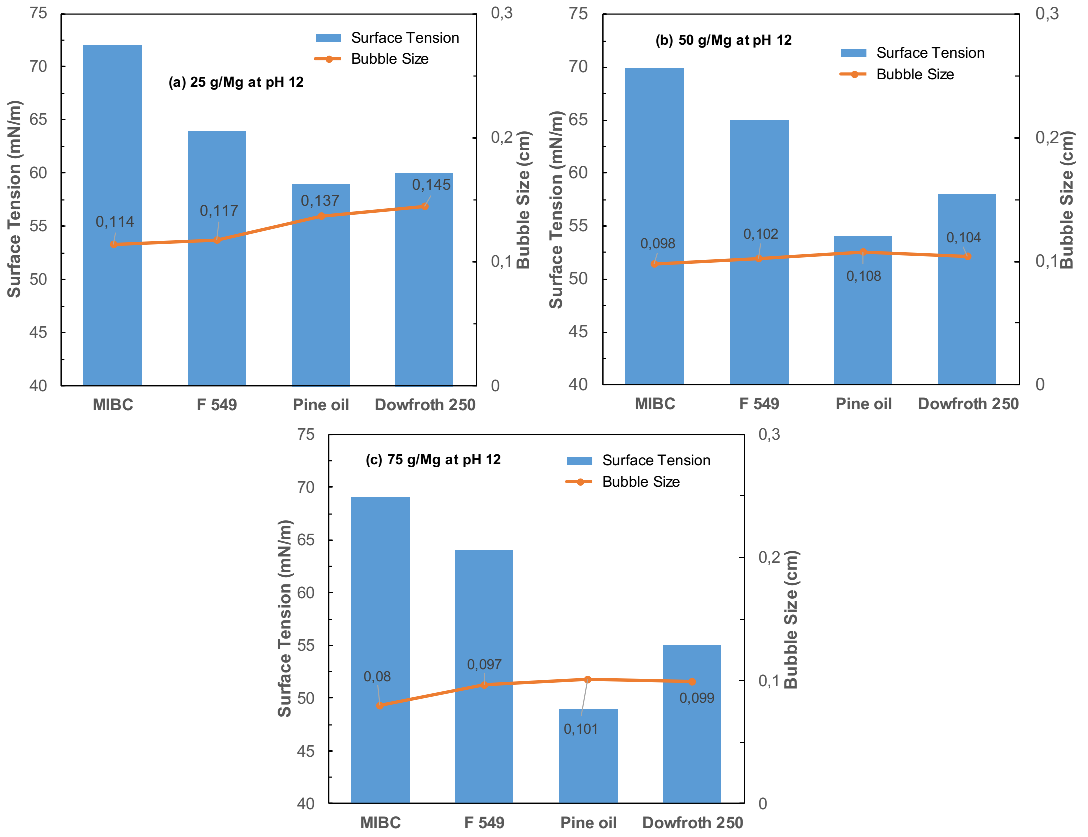

Fig. 15. Effect of type of frothers at pH 12 on bubble size and surface tension at (a) 25 g/Mg (b) 50 g/Mg, and (c) 75 g/Mg amounts

Table 7. Types and properties of frothers used in surface tension measurements

Pine Oil	Turpentine Type
F-549	Glycol
Dowfroth 250	Polyglycol
MIBC	Alcohol

4. Conclusions

In this study, the effect of frothers was investigated in two and three phases systems. In the flotation investigation, two-phase and three-phase systems were defined at the laboratory and plant scale, and it was found that the bubble size changed as a function of the frother amount. All experiments showed that as the amount of frothers increased the size of the bubble decreased. The effect of frothers was also investigated on surface tension. In the measurements frothers types, amount, and their effect on surface tension at different pH values. It was observed that increasing the amount of frother decreased the surface tension and bubble size both natural pH and pH 12.

According to the laboratory and plant studies, it was determined that the copper grade of concentrate decreased and recovery increased due to the smaller bubble sizes for all frothers and their mixtures. When the laboratory and plant results of the frothers were compared, it was determined that similar results were obtained. Finally, it is observed that there are some differences between the basic research studies (two phases systems) and the three phases systems which must be investigated in detail.

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