

## Integration of JKMSI and Oxidation Index to Determine Flotation Behaviour of Sulphide Ores

### *Sülfürlü Cevherlerde Flotasyon Davranımını Belirlemek İçin JKMSI ve Oksidasyon İndeksinin Entegrasyonu*

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#### ABSTRACT

This paper compares the use of the JKMSI technique and the Oxidation Index methodology to evaluate the change in flotation performance of Cu-Zn complex sulphide ore samples treated with hydrogen peroxide to generate artificially different levels of oxidation and thus ore floatability. The objective of the work is to compare the consistence of the two techniques to measure the variability of ore floatability to get a quick response for the estimation of flotation performance. The results showed that JKMSI can be incorporated in the current Oxidation Index methodology and both techniques have the potential to replace the standard batch flotation tests under certain conditions for geometallurgical ore characterisation programmes. This test uses much less material and much faster than a standard batch scale flotation test. However, both techniques is still have some limitations and further work is required for their improvement.

**Keywords:** JKMSI, oxidation index, copper recovery, sulphide minerals, floatability.

#### ÖZ

*Bu çalışma, hidrojen peroksitle muamele edilerek yapay olarak farklı derecelerde oksitlenmiş ve dolayısıyla farklı yüzebilirliğe sahip olan Cu-Zn kompleks sülfürlü cevherinin flotasyon performansındaki değişimi değerlendirmek için JKMSI tekniğini ve Oksidasyon İndeksi metodunun kullanımını karşılaştırmaktadır. Çalışmanın amacı, cevher yüzebilirliğindeki değişkenliğin flotasyon performansı üzerindeki etkisinin hızlı bir şekilde tahmin edilebilmesi için iki tekniğin hassasiyetini karşılaştırmaktır. Sonuçlar, JKMSI'nin mevcut Oksidasyon İndeksi metoduna dahil edilebildiğini ve her iki tekniğin de, jeometalürjik cevher karakterizasyon programları için belirli koşullar altında standart flotasyon testlerini değiştirme potansiyeline sahip olduğunu göstermektedir. Bu test, standart bir toplu ölçek flotasyon testinden çok daha az malzeme kullanımı ve çok daha hızlı olma gibi avantajları beraberinde getirmektedir. Bununla birlikte, her iki teknikte de hala bazı sınırlamalar bulunmakta olup bunları geliştirmek için daha fazla çalışma gerekmektedir.*

**Anahtar Kelimeler:** JKMSI, oksidasyon indeksi, bakır verimi, sülfürlü mineraller, yüzebilirlik.

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## INTRODUCTION

Surface oxidation of sulphide ores is known to affect floatability and recovery during processing. Surface oxidation may vary due to the extent of the weathering process during mining, stockpiling, crushing, milling and flotation. A methodology based on EDTA extraction technique has been developed to measure the surface oxidation of sulfide ores. A calibration curve, namely "Oxidation Index", is derived to determine the relationship between degree of surface oxidation and batch flotation performance. Then, the 'Oxidation Index' of different ore samples can be measured and their floatability can be determined by using a calibration curve (Bicak et al, 2011). The author also has a national patent (TR 2013 15100B) about this index and methodology.

The JKMSI device and methodology has been developed from the original EMDEE Microflot device invented by Michael Chudacek (Chudacek, 1990, Chudacek and Fichera, 1991). According to Bradshaw (2011), the aim of using JKMSI is to develop a test that is robust and reproducible, that can be performed on many samples for statistical robustness. This would provide the basis to identify domains or samples requiring more rigorous or precise metallurgical test work, such as batch or pilot scale tests. A key aspect to this test is the representativity of the sample being tested to the ore domain being evaluated (Lotter, 1995). The sample mass of 10 g is significantly less than the 1 kg or more that is typically used for batch flotation tests. However, for both techniques, to ensure reliable results, test sample should be obtained using the appropriate subsampling methods according to the rules of the Safety Line (Gy, 1979).

The EMDEE Microflot floatability test was developed to yield information about mineral flotation behaviour on very small samples by exposing the tested system to environmental conditions simulating those prevalent in industrial flotation cells. (Chudacek, 1991). According to Chudacek (1991), the flotation task in hand can be pre-optimised by the EMDEE Microflot method so that only a few batch cell or pilot plant studies need to be conducted to confirm the optimal process. The main advantages of the EMDEE Microflot method are its small sample requirement and its high reproducibility through precise control of experimental conditions. This precise control can be achieved by using of a small volume

of pulp in a fully closed system for the flotation step and a highly reproducible agitation performance of the EMDEE Microflot agitator.

Therefore, an alternative method to batch flotation tests to characterize ore floatability known as the JK Mineral Separation Indicator (JKMSI) has been developed to determine the floatability of selected size fractions of ores (Bradshaw 2011). JKMSI has a high performance agitator and fully closed system. This test uses much less material and is much faster than a standard batch scale flotation test.

Flotation of sulphide ores is strongly affected by chemical parameters such as pH, Eh, dissolved ions and surface oxidation (Newell et.al, 2006). A low degree of surface oxidation may enhance the flotation of sulphide minerals by forming surface coatings of metal deficient sulphides (Buckley and Walker, 1988), elemental sulphur (Trahar, 1984) or polysulphides (Luttrell and Yoon, 1984). However, extensive surface oxidation generally reduces flotation recovery and selectivity (Senior and Trahar, 1991; Smart 1991, Newell at.al., 2006) by coating the surface of valuable and gangue minerals.

In literature there have been many techniques used to determine the degree of surface oxidation of sulphide minerals such as; Eh measurement (Baker et al., 1991), the dissolved oxygen demand test (Houot and Duhammet, 1990), EDTA extraction (Rumball and Richmond, 1996; Kant et.al., 1994), XPS and Auger surface analysis (Smart, 1991), optical mineralogy, infrared spectroscopy, flotation, contact angle and zeta potential measurements. These techniques have various practical limitations and developing a practical methodology to measure the surface oxidation of a sulphide ore in flotation plants is sought to predict the flotation behaviour of different ore types.

Ethylenediaminetetraacetic acid, widely abbreviated as EDTA, is a polyamino carboxylic acid and a colourless, water-soluble solid acetate. EDTA has the ability to dissolve and form a complex with the oxidation products of sulphide minerals; it however does not react with metal sulphides (Shannon and Trahar 1986; Shannon et.al., 1994; Grano et al. 1988; Rumball and Richmond 1996). Hence, the EDTA extraction technique is considered as the most successful method to determine the oxidation degree of ores in plant scale measurements (Bicak et.al, 2008).

There has been extensive research reported in the literature investigating the effects of surface oxidation

on flotation performance. However, some of these techniques are qualitative or at best semi-quantitative and do not give a quantitative index representing the degree of oxidation of a sulphide ore. A quantitative method to derive a relationship between the extent of oxidation and flotation performance of a given ore, namely "Oxidation Index (OI)" methodology, has been developed to determine the oxidation degree of an ore in units of percentage (%) (Bicak, 2011a, Ekmekci and Bicak., 2011). Calculation of oxidation index for a copper mineral is given in Equation (1).

$$OI(\%) = \frac{Es\ Cu - Es\ Cu_{min}}{Es\ Cu_{max} - Es\ Cu_{min}} * 100 \quad (\text{Eq. 1})$$

where; Es Cu: EDTA extractable copper of an ore sample with an unknown degree of oxidation, Es Cu<sub>min</sub>: EDTA extractable copper of an unoxidized ore sample and Es Cu<sub>max</sub>: EDTA extractable copper of a heavily oxidized sample (Eq. 1)

The units of these parameters are expressed as Ext. Cu metal (mg) /Cu in ore (g).

Based on this methodology, a calibration curve has been derived for Çayeli Copper Zinc Complex Sulphide Ore which is located on the Black Sea coast of Turkey to demonstrate the relationship between the degree of surface oxidation and flotation performance (Bicak 2011b). The aim was to predict the variation in floatability of different types of ore from Çayeli Cu-Zn complex sulfide ore deposit. This curve is also used in this work to evaluate and compare the results obtained from JKMSI and EDTA extraction tests.

Ore floatability to characterize ore variability is usually determined by batch scale flotation tests, which requires significant quantities of ore, particularly due to the requirement of a milling curve in advance of the flotation tests. This makes the test time consuming and is relatively costly. Therefore, the development of alternative methods is in the interest of many researchers.

In this study, the Oxidation Index methodology and JKMSI were tested and compared by using two different samples of sulphide ores obtained from the Çayeli deposit. The focus of the work was on the consistence of the two techniques to measure the variability of ore floatability.

## EXPERIMENTAL

### Materials

Two different ore samples from Çayeli Cu-Zn complex sulphide ore deposit were used in this study. Chemical and mineralogical analyses of these samples are given in Table 1 and 2, respectively. The samples coded as BS and YO, have different mineralogy, oxidation and flotation behaviour (Bicak, 2011).

In Çayeli ore, chalcopyrite, pyrite and sphalerite are the main sulphide minerals. In addition to these minerals, bornite, galena and chalcocite are found in variable proportions. As seen in Table 1 and 2, BS contains a higher amount of chalcopyrite and also bornite. The main copper mineral of YO is chalcopyrite.

### EDTA Test Procedure

The ore samples were ground dry to obtain 63-65% of the milled product passing 38 µm for EDTA and JKMSI tests. An ore sample of 10 grams was used in the tests. The pH of the EDTA solution was adjusted to 7.5 with a solution of NaOH. Then the ore sample was introduced to the EDTA solution and stirred vigorously in 200 ml of 3 % EDTA solution by weight for 30 minutes. The slurry was filtered through a 0.45 µm filter and chemical analysis of both filtrate and solids were performed for Cu, Zn, Fe by using Atomic Absorption Spectroscopy (AAS) and Inductively Coupled Plasma Spectroscopy (ICP).

To obtain different degrees of oxidation, the ore samples were treated in solutions containing a range of hydrogen peroxide concentrations from 0 to 100%. After treatment with hydrogen peroxide, EDTA extraction tests were conducted to determine the degree of oxidation. The degree of oxidation is expressed on the basis of the metal and the ore, namely Es and Em (Rumball and Richmond 1996).

Es given in Equation 2, represents the oxidation on metal basis and EsCu represents the oxidation of the copper minerals in the ore.

$$Es_{(Cu,Zn,Fe,Pb)} = \frac{\text{Oxidised metal}_{(Cu,Zn,Fe,Pb)} \text{ (mg)}}{\text{Metal in the ore}_{(Cu,Zn,Fe,Pb)} \text{ (gr)}} \text{ (Eq.2)}$$

### JKMSI Test Procedure

The ore was prepared by grinding to the equivalent of the EDTA test size (P65: 38 µm). For each test

Table 1. Chemical composition of the ore samples used in the experiments.  
 Çizelge 1. Deneyde Kullanılan cevher numunelerinin kimyasal bileşimi.

Sample Code	Cu %	Fe %	Pb %	Zn %
BS	6.62	31.2	0.07	2.01
YO	2.45	32.2	0.39	4.13

Table 2. Mineralogical analyses of the ore samples used in the experiments.  
 Çizelge 2. Deneylerde kullanılan cevher numunelerinin mineralojik analizleri.

Minerals (%)	BS	YO
Chalcopyrite	21.92	12.15
Sphalerite	4.05	9.76
Pyrite and Others	68.55	77.06
Chalcocite	0.08	0.02
Bornite	5.38	1.01

representative samples of 10 g of feed material were obtained by careful microriffiling and subsampling. The sample was added to 100ml of water and prepared by sonification. The reagents were added ( $1.4 \times 10^{-4}$  M 3418A as collector and  $1.4 \times 10^{-4}$  M H27 as frother), and the test was commenced. The tailings and concentrate were removed, filtered, dried, weighed and assayed.

## RESULTS AND DISCUSSION

### Effect of Surface Oxidation

The ore samples were oxidised artificially by using hydrogen peroxide solutions to change the surface properties and hence, the flotation behaviour of the sulphide minerals in the ore. The variation in the surface oxidation of the copper minerals was determined by the Oxidation Index methodology and flotation behaviour by JKMSI. The results obtained from these tests were then compared with the Oxidation Index calibration curve of Çayeli ore, which represents the variation in Cu recovery as a function of the Oxidation Index (OI). The Cu recovery values given in the calibration curve were from batch scale flotation tests performed using the artificially oxidized Çayeli ore samples (Bicak, 2011).

In the standard JKMSI procedure, the ultrasonic bath of solids before JKMSI was generally applied to clean the surface of the minerals from slimes and colloidal species and to create fresh surfaces to simulate a grinding effect prior to flotation. However, surface cleaning could also remove some of the oxidised materials which affect the surface properties and flotation behaviour of the minerals. Therefore, the effect of the ultrasonic bath was investigated by using the same samples with and without ultrasonic conditioning for JKMSI. Table 3 shows the JKMSI and EDTA test results after ultrasonic conditioning for a range of oxidation degrees achieved with different concentrations of peroxide from 1% to 75%. The EDTA tests were performed to determine the oxidation degree by means of Es and flotation performance of the same samples was determined by the JKMSI test.

According to Table 3, Es Cu of the samples increased in parallel to peroxide concentration, going up to 50%. In these experiments, after ultrasonic conditioning the solution was decanted to obtain the required pulp density for the EDTA extraction test, which is anticipated to remove some of the surface oxidation products. The fluctuations in the results could be attributed to ultrasonic bath treatment. Therefore, the experiments were repeated in the absence of

Table 3. JKMSI and EDTA results in the case of surface cleaning with ultrasonic conditioning for BS sample.  
Çizelge 3. BS numunesi için ultrasonik banyo ile yüzey temizleme aşamasından sonra JKMSI ve EDTA sonuçları.

BS (Peroxide Concentration %)	Cu Recovery, % (From MSI tests)	Es Cu, mg/g (From EDTA tests)
0	11.532	10.737
1	27.392	53.728
10	18.427	46.062
25	26.364	40.900
50	31.058	27.597
75	25.876	24.300

ultrasonic bath under the same conditions to examine the effect of ultrasonication on the surface oxidation of the minerals.

Table 4 and 5 shows the JKMSI and EDTA results for BS and YO in the absence of ultrasonic conditioning. In these experiments, 5 grams of ore sample was used for JKMSI tests and the copper recovery values obtained were almost doubled as compared to the results obtained using 10 grams of ore after ultrasonic conditioning. This can be attributed to the increase in the bubble carrying capacity and the stability of the froth layer when less amount of floatable particles present in the cell. The Çayeli ores are rich in sulphide minerals and using 5 grams of sample seems more convenient to maximize the bubble carrying capacity and the froth stability.

As seen in Table 4, Cu Rec (%) obtained from JKMSI in the absence of ultrasonic conditioning was much higher as compared to the ore after ultrasonic treatment. Other conditions were studied without ultrasonic treatment since the ore floatability was not affected negatively. Table 4 shows that as peroxide concentration is increased from 0% to 25%, Es Cu increased due to the oxidation effect of peroxide solution. But, increasing the peroxide concentration from 25% to 100% decreases Es Cu, which may be attributed to the surface cleaning effect of peroxide at higher concentrations (Zhang et.al., 2017). Nonetheless, the copper recovery values obtained from JKMSI tests seem to be in correlation with EDTA tests, irrespective of peroxide concentration. But, the highest recovery value was about 51% for the sample having Es Cu value of 16.39 mg/g, which was the sample without peroxide treatment. This indicates that the sample used in the tests was already

oxidised to some extent, probably during sample preparation.

The results of YO ore are given in Table 5. The oxidation behaviour of YO was found to be similar to that of BS. Treatment of the ore with peroxide solution changed the surface oxidation and flotation recovery to some extent. The ore sample without peroxide treatment was also found to be oxidised and the maximum copper recovery was only 56.02%.

Table 4 and 5 shows the average results and standard deviations for BS and YO. The highest standard deviation observed for JKMSI is 4.64% in the case of 25% Peroxide for BS and for Es Cu (1.08 mg/g) in the case of 1% Peroxide for BS. The results showed that reproducibility of EDTA tests was in the acceptable range. However, standard deviation of the JKMSI tests was higher for some of the conditions. This was mainly due to the practical difficulties in collecting of the products from the instrument.

### Effect of Sulphidisation

As mentioned above, the ore could be oxidised already due to sample preparation and unfavorable storage conditions during transportation. If this is the case, it should be possible to increase the recovery and decrease the oxidation degree by sulphidisation of the ore using  $\text{Na}_2\text{S}$ . Therefore, samples from BS ore were sulphidized at three different concentrations to observe the influence of sulphidisation on surface oxidation and flotation recovery.

Figure 1 shows the effect of sulphidisation of BS ore. The sulphidisation was performed by using  $1.5 \times 10^{-3}$  M  $\text{Na}_2\text{S}$ , which reduced the redox potential of the

Table 4. JKMSI and EDTA tests results for BS sample in the absence of ultrasonic bath.

Çizelge 4. BS numunesi için ultrasonik banyonun olmadığı koşullarda JKMSI and EDTA test sonuçları.

BS Peroxide Concentration (%)	AVERAGE		STANDARD DEVIATION	
	Cu Recovery (%)	Es Cu (mg/g)	Cu Recovery (%)	Es Cu (mg/g)
0	50.94	16.39	0.63	0.67
1	46.27	21.80	4.12	1.08
10	38.05	39.69	4.20	0.85
25	34.18	24.40	4.64	0.50
50	44.76	15.85	0.17	0.13
100	46.61	10.24	3.76	0.53

Table 5. JKMSI and EDTA test results in the absence of ultrasonic bath for YO sample.

Çizelge 5. YO numunesi için ultrasonik banyo olmadığında JKMSI ve EDTA test sonuçları.

YO Peroxide Concentration (%)	AVERAGE		STANDARD DEVIATION	
	Cu Recovery (%)	Es Cu (mg/g)	Cu Recovery (%)	Es Cu (mg/g)
0	56.02	18.89	2.52	0.61
1	46.85	20.70	1.07	0.11
10	42.91	26.54	0.25	0.10
25	51.49	18.50	4.21	0.42
100	49.73	11.34	3.81	0.49

pulp to -250 mV (according to ORP electrode Ag/AgCl). The results showed that EsCu decreased from 16.39 mg/g to 14.03 mg/g after sulphidisation, indicating partial surface regeneration. In parallel to EsCu results, Cu recovery increased from 50.94% to 62.70% with surface cleaning by sulphidisation.

#### COMPARISON OF JKMSI AND OXIDATION INDEX

The calibration curve for Çayeli ore was derived by comparison of the EsCu values and flotation recovery of the artificially oxidised ore samples. The recovery values were obtained from kinetic batch flotation tests as performed to determine floatability of minerals in an ore. Therefore, in the calibration curve the relationship between surface oxidation and copper recoveries from batch flotation tests are represented. In this way, comparison of the recoveries

from JKMSI, batch flotation tests and surface oxidation could be illustrated in Figures 2 and 3.

The test results were plotted in the same graph as a function of EsCu (Figure 2) and OI (Oxidation Index- Figure 3). In addition to JKMSI results, the copper recovery values from batch flotation tests of unoxidised BS and YO samples are also included.

Figure 2 shows that the copper recovery values obtained from JKMSI fit to the calibration curve. Most of the data are accumulated in the EsCu range up to 70 mg/g. Figure 2 shows that the recovery values of JKMSI tests are generally lower than the calibration curve. This could be attributed to the drop back of the particles due to low stability of the froth phase. Despite this, the results of the tests performed under different conditions from two different ore types were found to be in good agreement with the calibration curve.

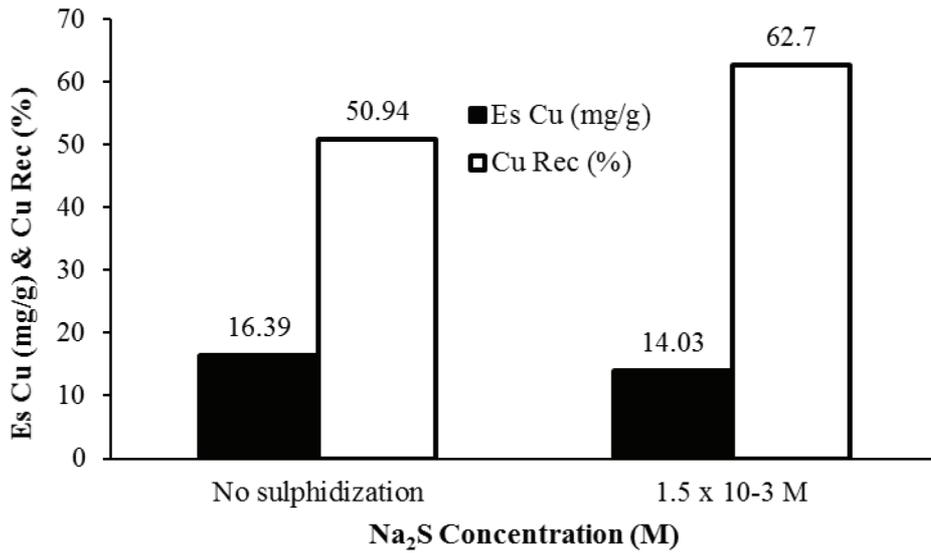


Figure 1. Sulphidization of BS ore: Relationship between Es Cu and Cu Rec in the absence and presence of Na<sub>2</sub>S.  
 Şekil 1. BS cevherinin sülfürlenmesi: Na<sub>2</sub>S varlığında ve yokluğunda Es Cu ve Cu Rec verimi.

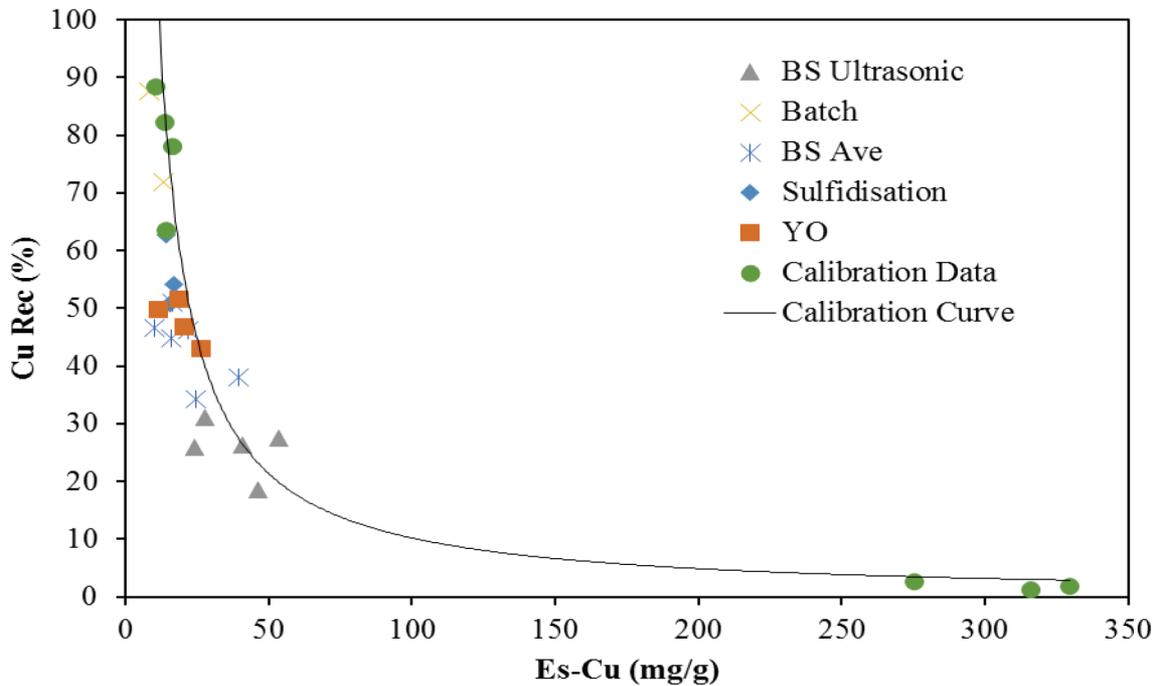


Figure 2. Comparison of the results as a function of EsCu.  
 Şekil 2. Sonuçların Es Cu'nun fonksiyonu olarak kıyaslanması.

As mentioned above, surface oxidation of sulphide minerals is expressed quantitatively with the oxidation index methodology. EsCu values of the samples used in JKMSI are converted to OI (%) using the calibration curve derived for Çayeli ore. The

relationship between copper recovery and OI is illustrated in Figure 3 for all the tests performed in this work. The data were less scattered and closer to the calibration curve with OI. The recovery values obtained from JKMSI were in correlation with the degree

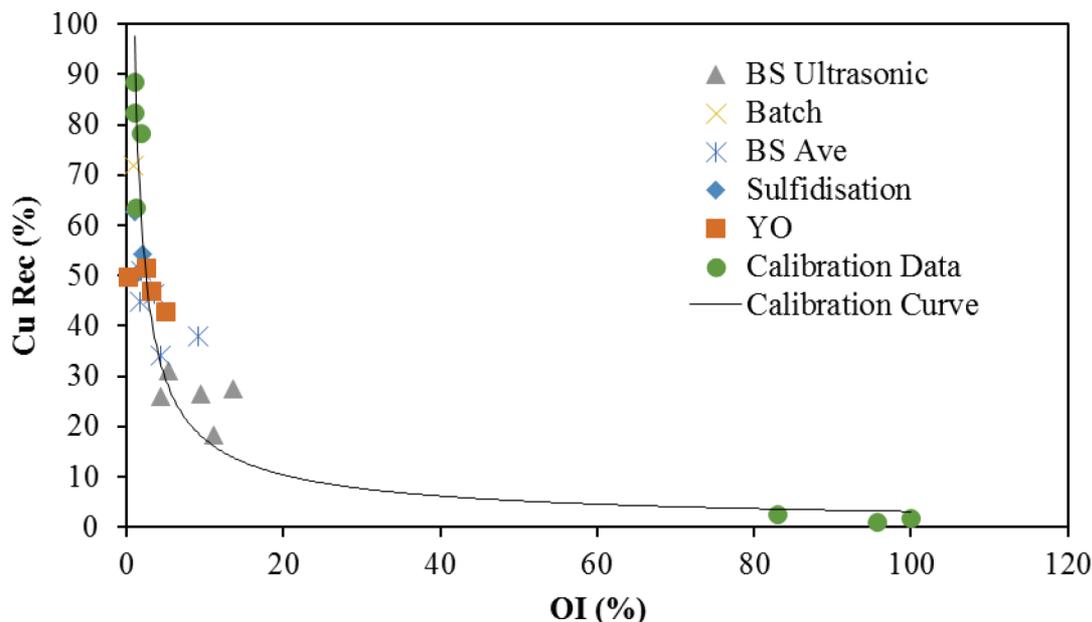


Figure 3. Comparison of the results as a function of Oxidation Index.

Figure 3. Sonuçların oksidasyon indeksinin bir fonksiyonu olarak karşılaştırılması.

of oxidation calculated from “Oxidation Index” and also with the recovery values from batch flotation tests. The results showed that JKMSI can be incorporated into the Oxidation Index methodology. Both techniques have the potential to replace the standard batch flotation tests under certain conditions for geometallurgical ore characterisation programmes.

## CONCLUSIONS

The variability of ore floatability is usually determined by batch scale flotation tests, which require significant quantities of ore. Such tests are time consuming and costly to do on a regular basis. Therefore, alternative techniques are developed to determine floatability. In this work, two techniques, JK Mineral Separability Indicator (JKMSI) and Oxidation Index (OI) methodology were evaluated and compared.

The results showed that it was possible to predict floatability of copper minerals in two different ore types from Çayeli ore by using both JKMSI and Oxidation Index. However, the standard deviation of the recovery values of the JKMSI tests was higher than Oxidation Index probably due to the drop back of the particles from the froth phase and some practical difficulties in collecting the products from the tube. Despite this, there was a good correlation between the Oxidation Index calibration curve of Çayeli ore,

which was developed by Bıçak (2011) and results of JKMSI tests.

It is concluded that JKMSI can be incorporated into the current ‘Oxidation Index’ methodology. This would enhance the commercial potential viability of the methodology and provide a tool for applying to geometallurgical ore characterisation programmes. However, both techniques have still some limitations and further work on different type of ores is required for their improvement.

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