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PURIFICATION OF FELDSPAR FROM COLORED IMPURITIES USING ORGANIC ACIDS

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Abstract: In this study oxalic, citric, and glycolic acids were used in order to reduce Fe and Ti (colored impurities) from the slimes (-75 μm) feldspar samples using organic acids. The results showed that removal ratios of the colored impurities from the feldspar samples were 67.9% for Fe and 43.75% for Ti using oxalic acid and the agitated leaching (AL) method. The influence of main parameters (temperature, pulp density, leaching time, and acid concentration) were examined by using full the factorial design (2^4) ANOVA-Yates test technique. Next, the removal ratios of Fe% and Ti% in the tests were determined to be 80.44% and 45.39%, respectively. Additionally, the main parameters which were obtained from the best results of AL were optimized for the -500+75 μm feldspar sample. Finally, the microwave-assisted pressure leaching (MAPL) method was apply to determine the effect of pressure on leaching. The obtained results indicated that the optimum removal ratios obtained were 95.74% for Fe and 70.88% for Ti by using the MAPL tests with oxalic acid. Furthermore, the measured whiteness (L) values were observed to be over 90%. This is a suitable purification ratio for the ceramic and glass industry.

Keywords: *chemical extraction, feldspar, organic acids, statistical analysis, microwave*

Introduction

The main feldspar minerals are Na-feldspar (albite), Ca-feldspar (anorthite), and K-feldspar (microcline). Their alkali content determines product quality and price. Feldspars are mostly used in the ceramics, glass making, plastics, rubbers, and paint industry as fillers. The alumina content in feldspar provides durability, hardness, and resistance to chemical corrosion in the glass making process. Additionally, feldspar is added as a flux in ceramic making, and this action results in lower melting temperature of the mixture. Melting fluxes form a vitreous matrix which ties components together in the furnace (Demir et al., 2001; Bayraktar and Cakir, 2002). The presence of iron and titanium in feldspar is not desired, because they change the color of the concentrates. Therefore, the iron content should be limited below certain values, that is 0.10% in glass making and 0.07-0.08% in ceramics. Since the particle size of feldspar concentrate is between 500 μm and 74 μm, the slime particles are discarded to tailings

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in industrial-scale applications (Amarante et al., 1997; Dogu and Arol, 2004). Agitation leaching is a general method applied for removing Fe and Ti from feldspar ores using inorganic acids. This method is usually suitable for achieving a high degree of iron removal but is expensive, has complex operating conditions, and is environmentally hazardous (Mesquita et al., 1996). Meanwhile, organic acids having carboxylic groups are less corrosive than inorganic ones during dissolving of the impurities. Besides being biodegradable, they are efficient reagents and accepted for metal dissolution at slightly acidic pH in comparison to be other extracting solutions, apart from the microwave assisted applications (MAPL) (Cameselle et al., 2003; Del Dacera and Babel, 2006; Jonglertjunya and Rubcumintara, 2013). Recently, there has been a growing interest in procedures using MAPL for aqueous systems. This interest is derived from the advantages of MAPL against conventional heating (Haque, 1999; Suoranta et al., 2015; Pinto and Soares, 2013).

In this research oxalic ($H_2C_2O_4$), citric ($H_8C_6O_7$), and glycolic ($C_6H_{12}O_7$) acids were used for bleaching of feldspar ores in order to reduce their iron and titanium contents. The leaching parameters were examined by the 2^4 factorial design (DOE) using the Yates method for the statistically agitation leaching tests. Finally, the MAPL experiments were applied under the best conditions of the AL tests by using optimum parameters.

Experimental

Materials and procedures

The feldspar sample used in this study was obtained from the Cam-Is Mining Company (Turkey) where flotation and magnetic separation were applied as conventional methods. The chemical analysis of the sample was performed using lithium tetraborate fusion method (HCl digestion) and an atomic absorption spectrophotometer (AAS) analysis. The results are presented in Table 1.

Table 1. Composition of the sample

Contents	SiO_2	Al_2O_3	Na_2O	CaO	MgO	K_2O	P_2O_5	Fe_2O_3	TiO_2	LOI
Grade (%)	68.70	18.00	9.90	0.66	0.47	0.38	0.17	0.25	0.24	0.11

The iron(III) oxide (Fe_2O_3), titanium dioxide (TiO_2) contents and cumulative passing by weight of the fractions of the sample are shown in Fig. 1. Besides, the chemical analysis of the -75 μm in size sample was determined to be: 0.580% Fe_2O_3, 0.520% TiO_2, and 0.013% LOI while -500+75 μm had 0.310% Fe_2O_3, 0.270% TiO_2, and 0.490% LOI. According to the XRD results, the sample mainly consists of albite ($NaAlSi_3O_8$), quartz (SiO_2), muscovite ($KAl_2(AlSi_3O_{10})(F,OH)_2$), and tourmaline ($(Na,Ca)(Mg,Li,Al,Fe^{2+},Fe^{3+})_3 (Al,Mg)_6(BO_3)_3Si_6O_{18}(OH,O,F)_4$) minerals (Fig. 2). All tests were conducted with a magnetic stirring-hot plate in a beaker (250 cm^3), and the temperature was controlled by a thermometer. After the leaching process was finished, the solid-liquid separation was applied, and the iron-titanium removal ratios were determine by the AAS for each test. Reagents from Sigma Aldrich and distilled water were used in the experiments. After the comminution (-500 μm), the sample was divided into two size fractions (-500+75 and -75 μm), which were used for the AL. After this stage, the oversized sample (-500+75 μm) was used for the optimization tests.

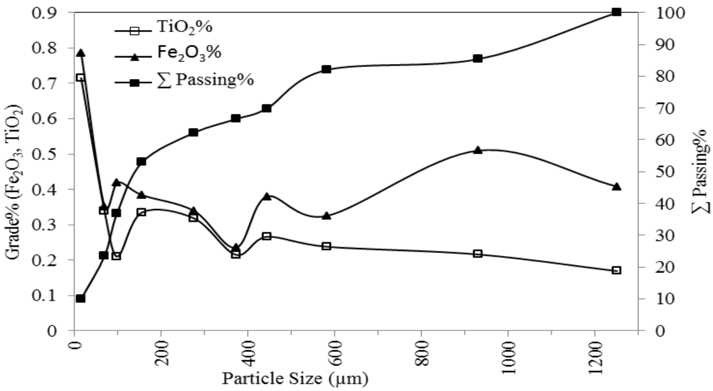

Fig. 1. Fe_2O_3-TiO_2 contents and ∑ passing of the sample

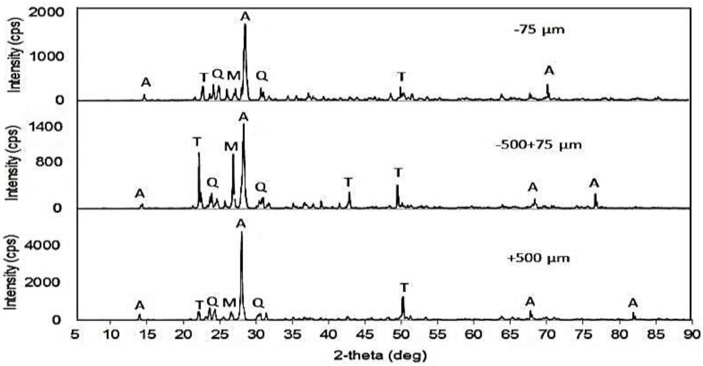

Fig. 2. XRD patterns of samples: albite: A, quartz: Q, tourmaline: T, muscovite: M

The MAPL tests were applied for the -500+75 μm sample. The tests were carried out with a CEM Mars 6 microwave oven, which has temperature (°C), time (min), and pressure (MPa) adjusting options. The device was equipped with 200 cm^3 vessels made of PFA. Then, the Konica Minolta CM-5 colorimeter was used for color measurements of the solid products of the MAPL tests. A statistical planning of the tests was used when it was desired to learn the basic and synergic consequences of the used experimental parameters (Montgomery, 1991; Naik et al., 2005). Furthermore, the influence of a variable was established by alteration the output created by changing the level of the factor. Two factors were accepted as interactions when the influence of a factor is based on the level of another one. In the first stage, four variables were taken into account, and the 2^4 full factorial unrepeated tests were run to reveal their main and synergetic effects. The Yates algorithm was applied in this research in order to label each experiment. For example, treatment *ABCD* was the experimental route in which variables *A* (acid concentrate: mol/dm^3), *B* (solid/liquid ratio: wt%), *C* (time, min), and *D* (temperature, ^{o}C) were fixed at their peak level or test *AB* was the experiment at which variable *C* and *D* were at their low level when the variables *A* and *B* were fixed at their high level. A set of 16 tests for each of the acids were arranged according to the calculation of $N=2^n$, in which *N* was the test number, and *n* was the factor of variables. Table 2 presents the calculated values with the corresponding experimental results obtained from 16 tests for the Fe extraction by using oxalic acid (three replicated) which constituted the central point of the DOE, determined by arithmetic means of the highest and lowest levels. The same tests were performed for the Fe and Ti removal for other organic acids. The higher and lower levels were denoted as '+' and '−', respectively. Three experiments were conducted at the middle level to predict the standard deviation, and error in reference to principles of the design of the experiments where *Y*=response (Fe% extraction), X_1=acid concentrate, X_2= solid/liquid ratio, X_3=time, X_4=temperature. The ANOVA experimental parameter levels were chosen as follows: solid/liquid ratio 10-30% (w/v), leaching time 30-120 min, oxalic, glycolic and citric acid concentrations 0.5-3.0 mol/dm^3, temperature, 20-70 ^{o}C.

Results and discussion

The Yates technique test results using experimental design to determine parametrical effects of impurities dissolution (Fe, Ti with oxalic acid) are given in Tables 2 to 4. The *F* values for the 9th column with *AB*, *AC*, *ABC*, *AD*, *BD*, *ABD*, *ACD*, and *ABCD* combinations obtained by the performed leaching experiments and according to the Yates design for Fe removal were lower than the *F* table values given in the 10th column for oxalic acid in Table 2. In this regard, it was determined that these combinations were ineffective. The highest Fe removal was obtained for the *AD* combination (67.90%) by oxalic acid leaching, *ACD* combination (41.53%) by the citric acid leaching, and *CD* combination (34.61%) by the glycolic acid leaching while the best Ti dissolution was recorded at for *ACD* combination (55.54%) by the oxalic acid leaching, *ACD* combination (40.07%) by the citric acid leaching, and *ACD* combination (37.07%) by the glycolic acid leaching. Short descriptions of DOE (2^4) for the Fe% and Ti% removals for all organic acids are presented in Tables 3 and 4, respectively. It was found that oxalic acid showed a better effect than other organic acids for the purification of feldspar. Chemical reactions for oxalic acid (1), citric acid (2), and glycolic acid (3) are, respectively:

$$C_2H_2O_{4(s)} + H_2O \rightarrow H_3O + C_2HO_4^- \quad (1)$$

$$C_6H_8O_{7(s)} + H_2O \rightarrow H_3O + C_2H_7O_7^- \quad (2)$$

$$C_6H_{12}O_{7(s)} + H_2O \rightarrow H_3O + C_2H_{11}O_7^- \,. \quad (3)$$

Iron and titanium, as undesired cations for each acid, were dissolved according the formula:

$$Fe_2O_3 + TiO_2 + 4H_3O^+ \rightarrow TiO^{2+} + Fe^{3+} + 6H_2O \,. \quad (4)$$

The regression equations, involving the applied organic acids and given by Eqs. 5 and 6 for oxalic, citric and glycolic acids, determine the removal ratios of the colored impurities:

$$y = 32.56 + 3.03X_1 + - 3.14X_2 - 14.07X_3 + 5.99X_2X_3 + 1.59X_4 - 6.42X_3X_4 - 2.20X_2X_3X_4 \quad (5)$$

$$y = 22.91 + 1.50X_1 - 0.88X_2 + 1.78X_3 + 1.52X_1X_3 - 0.69X_1X_2X_3 + 5.29X_4 + 1.89X_1X_4 - 1.55X_2X_4 - 0.34X_1X_2X_4 + 2.03X_3X_4 - 1.37X_2X_3X_4 + 0.29X_1X_2X_3X_4 \quad (6)$$

$$y = 21.16 + 1.39X_1 + 1.43X_1X_2 + 1.38X_3 - 0.70X_1X_3 - 0.55X_2X_3 + 0.70X_1X_2X_3 + 3.33X_4 - 0.76X_2X_4 - 0.58X_1X_2X_4 + 1.37X_3X_4 - 0.63X_1X_3X_4 - 1.92X_2X_3X_4 + 0.95X_1X_2X_3X_4. \quad (7)$$

The experiments iron removal ratios have high correlations with the calculated values (Fig. 3). High coefficient of determination (R^2) proved that the data fit the created equations well. The results of the Yates technique (DOE) for the AL tests showed that oxalic acid was the best alternative among the three organic acids. Therefore, the optimization tests were completed using oxalic acid to reach a maximum effectiveness.

Optimization tests for agitated leaching

The highest removal ratio of Fe was obtained in the *AD* test combination as shown Table 2, which was taken into consideration for the optimization tests as a base level (Z_{JO}). The optimization steps for each test were calculated according to Table 5 using the steepest ascend method, depending on X_1, X_2, X_3, and X_4 variables. The selection of b_J steps for each variable were completed as follows: molar concentration of oxalic acid 0.1 mol/dm^3, leaching time 3 min, and temperature 3 ^{o}C. Meanwhile solid wt% rate was –1.0 as a decreasing step in order to extract more Fe and Ti (Table 6).

Table 2. Oxalic acid leaching experimental results for Fe% removal ratio

Yates Order	Fe (%)	Analysis of Yates				$(IV)^2/16$	*DF*	*F* Calculated	F	Decision	X_1	X_2	X_3	X_4	*Y*
		I	II	III	IV										
1	46.09	97.34	154.43	247.66	520.92	16959.61	1	95.789	4.49	*E*	–1	–1	–1	–1	
a	51.25	57.09	93.23	273.25	48.48	146.92	1	24.715	4.49	*E*	1	–1	–1	–1	52.98
b	27.78	33.95	218.56	19.67	–50.39	158.68	1	26.693	4.49	*E*	–1	1	–1	–1	24.23
ab	29.31	59.28	54.69	28.81	–4.91	1.51	1	0.253	4.49	*NE*	1	1	–1	–1	30.29
c	14.50	125.74	6.70	–14.92	–225.08	3166.19	1	532.646	4.49	*E*	–1	–1	1	–1	15.23
ac	19.45	92.82	12.97	–35.47	4.53	1.28	1	0.215	4.49	*NE*	1	–1	1	–1	21.23
bc	25.63	28.62	15.28	–0.55	95.95	575.43	1	96.804	4.49	*E*	–1	1	1	–1	25.33
abc	33.65	26.07	13.54	–4.37	12.00	9.00	1	1.514	4.49	*NE*	1	1	1	–1	31.39
d	57.84	5.16	–40.25	–61.20	25.59	40.93	1	6.885	4.49	*E*	–1	–1	–1	1	58.56
ad	67.90	1.54	25.33	–163.87	9.14	5.22	1	0.878	4.49	*NE*	1	–1	–1	1	64.62
bd	43.80	4.94	–32.92	6.27	–20.55	26.40	1	4.441	4.49	*NE*	–1	1	–1	1	44.67
abd	49.02	8.03	–2.55	–1.74	–3.82	0.91	1	0.153	4.49	*NE*	1	1	–1	1	50.73
cd	11.04	10.05	–3.63	65.58	–102.67	658.86	1	110.839	4.49	*E*	–1	–1	1	1	9.99
acd	17.58	5.22	3.08	30.37	–8.01	4.01	1	0.673	4.49	*NE*	1	–1	1	1	16.06
bcd	9.53	6.54	–4.83	6.71	–35.22	77.51	1	13.039	4.49	*E*	–1	1	1	1	11.29
abcd	16.54	7.00	0.46	5.29	–1.41	0.12	1	0.021	4.49	*NE*	1	1	1	1	17.35

E: effective, *NE*: ineffective, *DF*: Degrees of freedom, *Y*: Calculated Fe content,
Mean: 44.80 (for triple tests).

Table 3. Short descriptions of DOE (2^4) for Fe removal ratio

Reagent	Mean (%)	St. Dev. (%)	Min.	Median	Max.
Oxalic	32.70	17.91	9.53	28.55	67.90
Citric	22.91	7.25	13.62	21.59	41.53
Gluconic	21.36	4.86	14.13	20.31	31.61

Table 4. Short descriptions of DOE (2^4) for Ti removal ratio

Reagent	Mean (%)	St. Dev. (%)	Min.	Median	Max.
Oxalic	26.63	15.46	9.60	24.57	55.54
Citric	22.05	13.14	8.02	19.85	40.07
Gluconic	19.78	11.21	7.71	18.05	37.07

The optimum test condition was determined to be 4% solid/liquid ratio, 1.1 mol/dm^3 oxalic acid concentration, 88 ^{o}C and 48 min in Test 7 of the optimization for the agitational leaching. The highest removal value was 80.44% for Fe. It was expected that tests 8 and 9 would provide a higher removal ratio for lower solid wt% at higher temperatures, time, and acid concentrations. However, the desired ratios could not be achieved because the dissolved iron and titanium ions passed to the solid phase in the supersaturated oxalic acid in the leach solution due to water evaporation (Table 5).

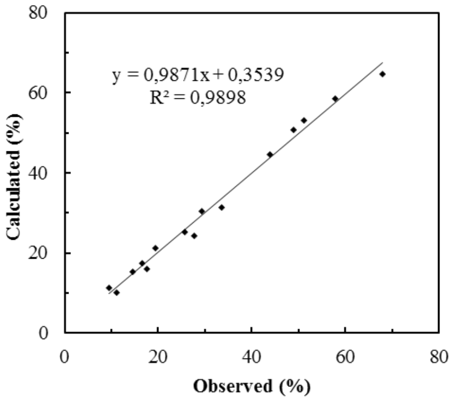

Fig. 3. Observed versus calculated Fe removal ratios with oxalic acid

Table 5. Optimization test conditions for removal ratios of Fe and Ti of AL

Incremental change	Molarity (mol/dm^3)	Solid (wt%)	Time (min)	Temperature (^oC)
Optimum level, Z_{JO}	0.5	10	30	70
Increment, ΔZ_J	1.25	10	45	25
Coefficient, b_J	3.03	-3.15	-14.07	1.60
$\Delta Z_J \, x \, b_J$	3.79	-31.50	-633.15	40.0
Normal Step	0.1	-1	3	3

Table 6. Results of optimization tests for AL using oxalic acid

Test Order	Solid (wt%)	Molarity (mol/dm^3)	Time (min)	Temp. (^oC)	(%)	
					Fe	Ti
1	10	0.5	30	70	32.22	18.10
2	9	0.6	33	73	32.89	22.23
3	8	0.7	36	76	31.24	29.77
4	7	0.8	39	79	44.76	35.60
5	6	0.9	42	82	49.38	38.07
6	5	1.0	45	85	60.47	41.42
7	4	1.1	48	88	80.44	45.39
8	3	1.2	51	91	65.48	45.09
9	2	1.3	54	94	68.21	42.47

Microwave-assisted pressure leaching tests (MAPL)

All the tests were conducted under the same conditions of 4% solid/liquid ratio, 1.1 mol/dm^3 oxalic acid concentration and 45 min time. The effects of pressure on the iron and titanium dissolutions were observed by gradual increase of temperature. The iron and titanium dissolutions were calculated from the leach solutions by AAS after the solid/liquid separation. The temperatures were 100, 130, 160, 190, 220, and 250 ^{o}C, respectively. The total leaching time was 45 min as a fixed value. The MAPL experiments were shown in Fig. 4.

The results showed that the iron and titanium removal ratios were higher than the conventional leaching in MAPL stage because of high pressure. The best removal ratio was obtained in test 3. The iron and titanium removal ratios were almost the same for tests 4, 5, and 6 (Table 7).

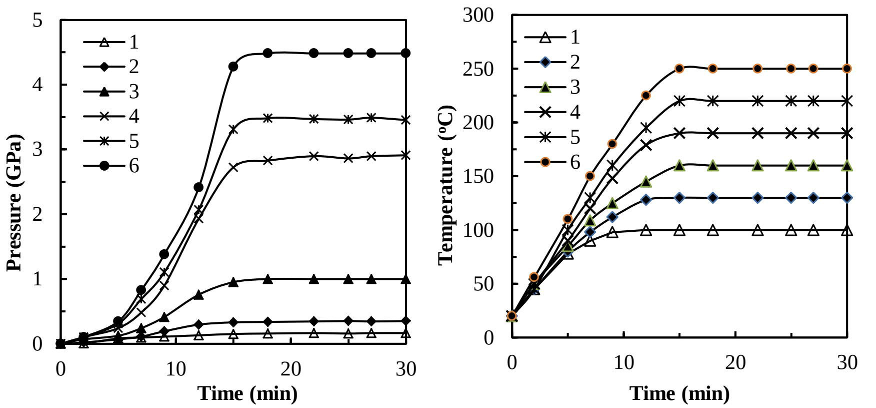

Fig. 4. MAPL tests for time versus pressure and temperature

Table 7. Results of MAPL tests for exctration of Fe and Ti characterizez by the removal ratios

No	Fe (%)	Ti (%)
1	70.64	57.65
2	79.57	70.59
3	94.89	70.00
4	95.11	70.88
5	95.74	70.59
6	95.53	70.88

The color measurements of the concentrates were performed after calibration using Konica Minolta CM-5. The whiteness L and chromaticity indices a and b represent red, green, blue, and yellow colors. The values were determined based on the color vision of the human eye. Some colors are evaluated differently ($\Delta E^{*}ab$ and the human eye). "E" values reveal this difference. Also, Chroma and Hue values are shown in Table 8 by the following equations: Chroma= $\sqrt{a^2+b^2}$, Hue $= b/a$

Whiteness index in ceramic industries should be over 90% with low iron contents (under 5%) (Philips, 1989; Bundy and Ishley, 1991). Whiteness (L), Chroma and Hue values were nearly stable, especially after test 3 in Table 8. The average values of whiteness, Chroma and Hue (90.75±0.09, 0.524±0.003, and 0.195±0.001) were determined, which are quite suitable for an industrial usage. This situation confirmed that the increasing whiteness (> 90%) was the result of the iron and titanium removal ratios from the feldspar. It was shown that feldspar concentrates of MPAL tests was rightfully suitable for ceramic and glass industry. The leaching experiments showed that the pressure at the high temperatures had a strong effect on the MAPL tests compared with AL tests. Similar studies were performed by several researchers using conventional or agitational leaching (Bonney (1994), Calderon et al. (2005), Lee et al. (2006), and Tuncuk et al. (2013)) on purification of kaolin, especially. Veglio et al. (1998) and Aslan and Bayat (2009) investigated the removal of iron from quartz sand. On the other hand, the iron removal ratios of similarly conducted studies were lower than the MAPL test results from this study.

Table 8. Average results of color measurements of MPAL tests

No	*L*	*a*	*B*	*E*	Chroma	Hue
1	90.51	-0.50	-0.04	-0.48	0.480	-0.042
2	90.61	-0.51	-0.10	-0.55	0.502	0.080
3	90.66	-0.51	-0.13	-0.67	0.520	0.194
4	90.71	-0.51	-0.12	-0.71	0.526	0.250
5	90.76	-0.51	-0.13	-0.71	0.524	0.231
6	90.87	-0.51	-0.14	-0.68	0.526	0.250

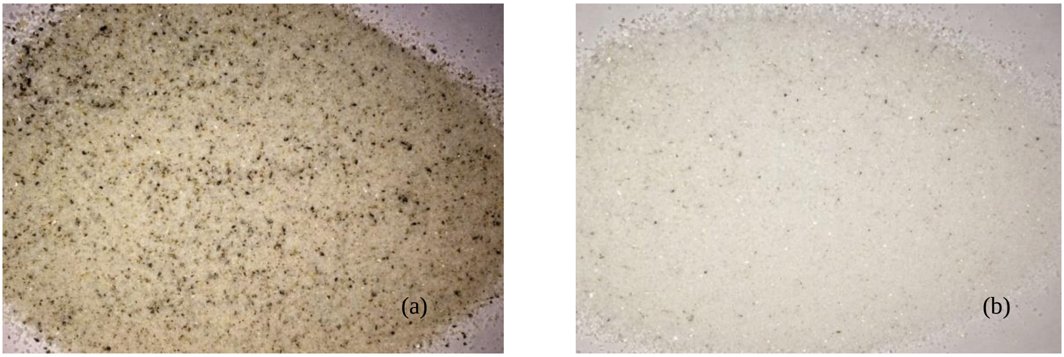

Fig. 5. Color comparison of the feed (a) and concentrate (b) of the MAPL test (No. 5)

Therefore, it should be accepted that the organic acid leaching using a microwave can be a good choice to reduce the Fe and Ti contents of feldspar ores for glass and ceramic making processes. Additionally, the operation times of MAPL tests were very short. However, energy requirements of MAPL compared with AL may be optimized for industrial applications.

Conclusions

The results obtained from this study show that the iron and titanium impurities in feldspar ores can be efficiently removed by the AL or MAPL techniques using organic acids. The removal ratios of Fe and Ti by AL were 67.9% and 43.75%, respectively. The optimum result was obtained using the MAPL tests with oxalic acid leaching providing over 95% Fe and 70% Ti removal ratios. The proposed leaching conditions of this method were 4% solid/liquid ratio, 1.1 mol/dm^3 oxalic acid concentration, 45 min leaching time, and 160 ^oC temperature. Although this study was focused on the beneficiation of the -500+75 μm feldspars size fraction. The -75 μm feldspar fraction, which is discharged into the tailings in the glass making and ceramic industries, could also be considered after overcoming furnace problems in these industries.

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