BENEFICIATION AND PROCESSING OF NATURAL AND TECHNOGENIC RAW MATERIALS

Research paper

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Enrichment of Algerian kaolin using froth flotation method

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Abstract
The objective of this research is to study the removal of coloring impurities from Algerian kaolin ore (Tamazert kaolin, TK) located in the eastern region of Algeria, utilizing froth flotation process. The results obtained from XRF, SEM, and XRD characterization demonstrate that this local material is an alumino-silicate containing kaolinite, along with impurities such as Fe_2O_3 (> 2.7 % by weight) and TiO_2 (0.28 % by weight) which contribute to its coloring. After homogenization, crushing and milling, several froth flotation tests were conducted on TK. The results revealed that Tamazert kaolin exhibits favorable performance with froth flotation, in order to improve its properties. Based on the results obtained, it can be concluded that all fractions can be treated effectively using froth flotation with an optimal mass yield (weight recovery) of 79.84 % in concentrate for the fraction of 20–40 μm. Iron and titanium, the main coloring impurities in Tamazert kaolin, were reduced from 2.7 % to 0.08 % by weight for Fe_2O_3 in the fraction 20-40 μm, and from 0.28 % to 0.04 % by weight for TiO_2 in the same fraction, as determined by the optimum test. The significant reduction in coloring impurities (Fe_2O_3 and TiO_2) achieved through the flotation process confirms that iron is present in a free state in Tamazert kaolin. It can be ultimately confirmed that the froth flotation process can be a potentially effective process to improve the quality of Tamazert kaolin ore by removing Fe_2O_3 and TiO_2 with satisfactory results which meet the requirements of local companies.

Keywords
Tamazert kaolin, froth flotation, coloring impurities, Fe_2O_3, TiO_2

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ОБОГАЩЕНИЕ, ПЕРЕРАБОТКА МИНЕРАЛЬНОГО И ТЕХНОГЕННОГО СЫРЬЯ

Научная статья

Обогащение каолиновой руды месторождения Тамазерт (Алжир) методом пенной флотации

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Аннотация
Целью данного исследования является изучение процесса удаления окрашивающих примесей из каолиновой руды месторождения Тамазерт (тамазертский каолин, ТК), расположенного в восточном регионе Алжира, с использованием процесса пенной флотации. Исследования методами рентгенофлуоресцентной спектрометрии, сканирующей растровой электронной микроскопии и рентгенофазового анализа показали, что данный местный материал представляет собой алюмосиликат, содержащий каолинит, а также примеси Fe_2O_3 (> 2,7 % по весу) и TiO_2 (0,28 % по весу), которые способствуют его окрашиванию. После гомогенизации, дробления и измельчения ТК был подвергнут серии испытаний по пенной флотации. Результаты показали, что тамазертский каолин приобретает благоприятные характеристики после применения пенной флотации для улучшения его свойств. На основании полученных результатов можно сделать вывод, что все фракции могут быть эффективно обогащены методом пенной флотации с оптимальным массовым выходом по весу (извлечением по весу) 79,84 % в концентрат для фракции 20–40 мкм. Содержания железа и титана, являющихся основными окрашивающими примесями в тама-

зертском каолине, были снижены посредством флотационной обработки с 2,7 до 0,08 % по весу для Fe_2O_3 во фракции 20–40 мкм и с 0,28 до 0,04 % по весу для TiO_2 в той же фракции, что было показано в оптимальном испытании. Значительное снижение содержаний окрашивающих примесей (Fe_2O_3 и TiO_2), достигнутое в процессе флотации, подтверждает, что железо присутствует в тамазертском каолине в свободном состоянии. В итоге можно утверждать, что процесс пенной флотации может быть потенциально эффективным способом улучшения качества каолиновой руды Тамазерта путем удаления Fe_2O_3 и TiO_2 с удовлетворительными результатами, отвечающими требованиям местных компаний.

Ключевые слова
тамазертский каолин, пенная флотация, окрашивающие примеси, Fe_2O_3, TiO_2

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Introduction

Kaolin is a finely granulated rock, usually white and chemically inert. It is widely used in many industrial fields [1, 2] due to its favorable properties such as natural whiteness, fine particle size, plasticity, non-abrasiveness and chemical stability. The main impurities associated with commercial kaolin are quartz, feldspar, muscovite, biotite, titanium oxides and iron oxides or hydroxides, such as goethite, hematite and magnetite [3, 4].

There are many techniques for the beneficiation of kaolin ore. These include selective flocculation, magnetic separation, bleaching and flotation to remove colored impurities. The presence of iron oxides in kaolin has a deleterious effect on the color of the clay which declines in brightness with increasing iron content [2, 5, 6].

Flotation is undoubtedly the most important and versatile mineral separation technique, and both its use and application are continually being expanded to treat greater tonnages and to cover new areas [7].

Flotation is a separation process which exploits natural and induced differences in surface properties of the minerals, whether the surface is easily wetted by water, i.e. hydrophilic, or repels water, i.e. hydrophobic. If hydrophobic, the mineral particle can attach to air bubbles and be floated. The system is complex, involving three phases (solids, water, and air) and the interaction of chemical and physical variables [7].

The success of flotation processes is dependent primarily on the tendency of surface-active species to concentrate at the water-fluid interface, and on their capability to make selected non-surface-active materials hydrophobic by means of adsorption or association [8]

Froth flotation is the dominating mineral beneficiation technique and has achieved great commercial success [9]. In froth flotation, first a pulp of crushed and ground particles in water is conditioned with the desired flotation reagents including pH modifiers and surfactants. Then it is agitated in a cell in the presence of air sucked or fed into the impeller zone where the air is well dispersed due to intense agitation in that zone. The air bubbles collide with particles and are attached to those that are hydrophobic or have acquired hydrophobicity. The bubble-particle aggregates rise to the top of the cell and are removed by skimming [8].

In froth flotation, the parameters that can affect the efficiency of the separation are as follows [10]: the nature of the ore, dose of reagents, particle size, pulp density, conditioning time, pH…etc.). The optimum size range of froth flotation varies with the flotation process parameters and flotation machine types. Too coarse or fine sizes are not suitable for flotation, thus, the suitable flotation size is subject to upper and lower limits [11].

Flotation is a process used for the removal of TiO_2, especially when this mineral is colored by iron [12].

Kaolinite is hydrophilic. Thus with the addition of a small amount of chemical dispersant to negate the edge charges due to broken bonds, it will disperse readily in water. Ionic and/or polar non-ionic surfactants can be applied to the surfaces of the kaolinite to modify them to produce particles which have hydrophobic ororganophilic characteristics [13].

Flotation [13–15] is also used to improve the whiteness of kaolin intended for the paper industry.

The purpose of the present study is to improve (if possible) the quality of kaolin ore from the Tamazert deposit (Fig. 1) using the froth flotation technique based on the recovery of iron. A consequent objective is to improve the whiteness and the brightness level wished by the most industrial applications.

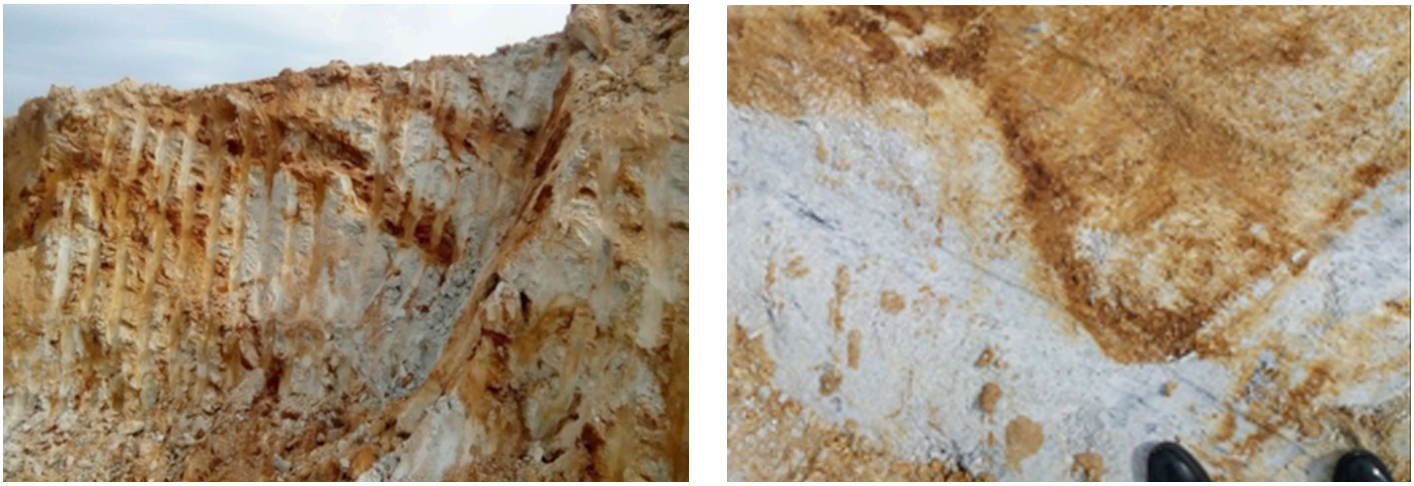

Fig. 1. Kaolin deposit of Tamazert

Materials and experimental methods

Characterization of Tamazert kaolin

In this study, the representative sample of kaolin ore was collected from Tamazert mine situated in Jijel city, East of Algeria and studied using X-Ray Fluorescence (XRF), X Ray Diffraction (XRD) and Scanning Electron Microscope (SEM) coupled with EDX.

The X-Ray Fluorescence (XRF) was realized with a Thermo Niton XL3t XRF Analyzer. The Scanning Electron Microscopy (SEM-EDX) was performed using JEOL JSM-5600. The surface of the thin section of sample was metallized (Au). The X-ray diffraction (XRD) was performed using PHILIPS-X'Pert MPD System diffractometer equipped with Cu-Kα (λ = 1.54 Å) radiation which was generated at 40mA. 45kV generator settings were performed to characterize the kaolin sample.

Scanning electron microscopy (SEM) was used to observe the morphology of Tamazert kaolin. The XRD was used to determine the mineralogical composition, while the XRF was used to determine the chemical composition.

Used Materials

The samples were milled using Retsch RS 100 laboratory miller, and then sieved to obtain five fractions: (0–20, 20–40, 40–80, 80–100 and 100–120 μm). For the test, the most commonly used materials are:

Flotation cell (KHD HUMBOLDT WEDAG AG) with a volume of the tank is 1.5 liters), pH-meter, Electronic balance, Magnetic agitator and Oven.

Used reagents

Most minerals are not water-repellent in their natural state and flotation reagents must be added to the pulp. The most important reagents are collectors which adsorb on mineral surfaces, rendering them hydrophobic and facilitating bubble attachment. Regulators are used to control the flotation process. These either activate or depress mineral attachment to air bubbles and are also used to control particle dispersion and the pH of the system. Frothers enable producing fine bubbles required to increase collision rates and allow maintaining a reasonably stable froth [7]. The chemical reagents used to carry out the flotation tests are presented in Table 1. All reagents were supplied by the laboratory of mineral processing, polytechnic school on Mieres, university of Oviedo, Spain.

Table 1

Used reagents in froth flotation of TK

Reagent	Formula	Role
Oleic acid	$C_{18}H_{34}O_2$	Collector
Methyl Isobutyl Carbinol (MIBC)	$C_6H_{14}O$	Frother
Sodium Metasilicate (pentahydrate)	$Na_2SiO_3 \cdot 5H_2O$ $Na_2SiO \cdot 5H_2O$	Depressor
Kerosene	$C_{10}H_{22}$	Activator
Sodium Hydroxide	NaOH	pH regulator

Experimental procedure of flotation

Each kaolin sample powder (**250 g**) was introduced in the flotation cell with 1000 g (1L) of water to form a pulp (slurry) which contains 25% of solid by weight. The diluted Sodium Hydroxide was added prudently to adjust the flotation pulp at 9.5. The machine is left running and 9 ml of Sodium Meta-Silicate added for about 5 minutes, then 4 droplets (≈0.84 g) of Kerosene to render our useful mineral (kaolin) hydrophobic for duration of 2 minutes. 13 droplets of Oleic

Acid (≈0.338 g) are carefully added to the cell and conditioned for 5 minutes; Oleic Acid plays the role of a collector. Ultimately, 3 droplets of Methyl Isobutyl Carbinol (MIBC) are added and the suspension was conditioned for 2 minutes. Then the air tap is opened to create the foam (air bubbles) required for flotation (Fernando Pita, 2017). This operation is known as bubbling. All tests were performed at room temperature and the flotation machine was adjusted to a rotational speed of 450 rpm for the all tests. Flotation tests were carried out in a KHD HUMBOLDT WEDAG AG flotation machine.

The zeta-potential of kaolinite is negative in a wide range of pH values and kaolin can be floated by amine collectors in acidic and alkaline medium [16–18]. The dried concentrates and tailings were analyzed. The results obtained are used to calculate grade, yields and recovery rates.

The concentrates and tailing obtained after the flotation operations were dried in an oven at 105°C during 24 hours. All these steps are summarized in Fig. 2.

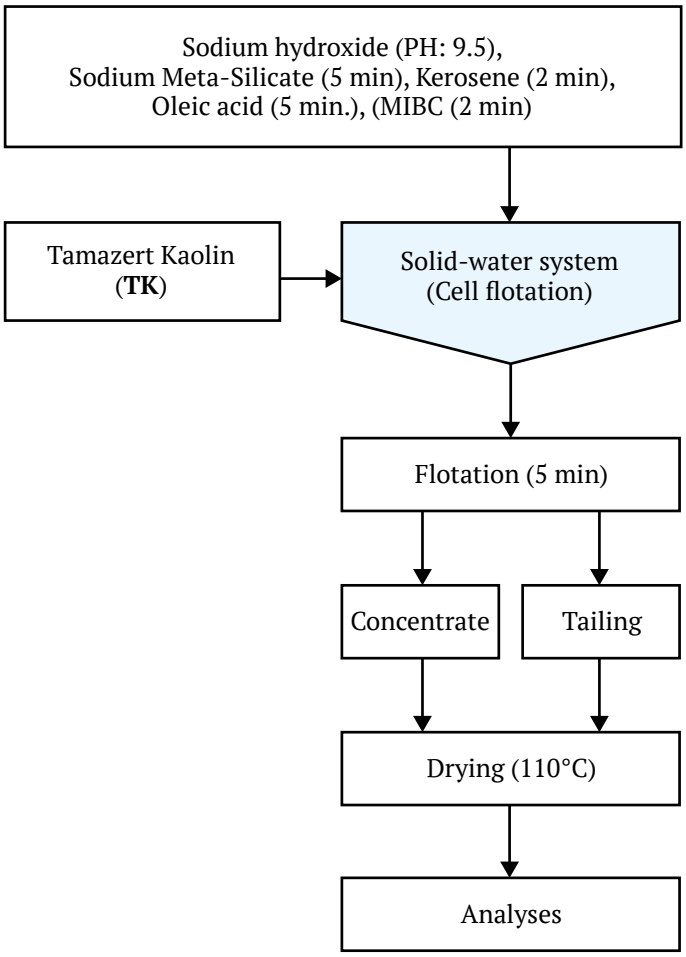

Fig. 2. A flowchart summarizing the steps of the kaolin flotation (TK)

Results and discussions

Characterization results

X-Ray fluorescence (XRF)

The chemical analyses of the kaolin sample are listed in Table 2. The kaolin sample was rich in SiO_2 at 46.70% and Al_2O_3 at 32.67%. The SiO_2/Al_2O_3 ratio of this sample was equal to 1.43. SiO_2/Al_2O_3 ratio in kaolinite mineral is usually 1.18[1] (Kaolinite Mineral Data). X-ray fluorescence measurements to determine the oxide composition of kaolin were carried out using Thermo Niton XL3t XRF Analyzer.

Chemical analyzes reveal that Tamazert kaolin has a high content of Fe_2O_3 (2.70 %), SiO_2 (46.70 %) and K_2O (2.58 %). TiO_2 content is low, 0.28 %.

The loss on ignition of TK is relatively low (9.36 %). It is less than the value of the loss on ignition of pure kaolinite [19], indicating the low presence of organic matter. This may be due to the presence of micas.

The value of the SiO_2/Al_2O_3 ratio (1.27) of TK is differs from the corresponding value of ideal kaolinite (1.18). This may be connected with the presence of alumina in this kaolin.

The presence of Fe_2O_3 produces the brownish color and decreases brightness and whiteness. The low content of TiO_2 makes the TK kaolin a very attractive raw material for a number of industries.

Scanning Electron Microscopy (SEM-EDX)

Figure 3 presents the SEM micrographs of the Tamazert kaolin (TK). The chemical elements proportion of kaolin was established by the Energy Dispersive X-ray (EDX) method. The Scanning Electron Microscopy (SEM-EDX) analysis was used to determine the morphology and composition of kaolin using JEOL-JSM 6610-LV coupled with an OXFORD INCA-ENERGY microanalyser.

In general terms, the morphology of kaolin presents hexagonal shaped kaolinite plates. According to the results of scanning electron microscopy (SEM-EDS), Fig. 3, A, the micrograph presented an imperfect structure of kaolinite. It thus shows that the particles have irregular and flat platelet shapes. There are also quartz particles surrounding the kaolinite particles. According to Amigo et al., 1994 [20], Tamazert kaolin has more or less low crystallinity. Fig. 3, B shows the presence of Hematite as impurity (Fe_2O_3) in this kaolin.

X-ray diffraction (XRD)

X-ray diffraction (XRD) analysis was performed using PHILIPS-X'Pert MPD System diffractometer with CuKα radiation adjusted at 40 mA and 45 kV generators. Fig. 4 shows that the mineral components of Tamazert kaolin were mainly kaolinite, quartz, muscovite and illite.

[1] Kaolinite Mineral Data. Available online: http://www.webmineral.com/data//Kaolinite.shtml#.W1CcIfZuLIU

Flotation test results

The products obtained from flotation tests (froth and tailing) were dried in an oven at 105 °C for 24 hours. The foam composed of kaolin particles (hydrophobic) is recovered by overflow of the scum on the surface of the flotation cell, while the tailing (hydrophilic) remains in the bottom of the cell and consists of mainly iron and titanium. Controlling the airflow to the flotation cell during the initial flotation period was found to be very crucial both for loading air bubbles with minerals, as well as for detachment. In addition, a foaming time of 5 minutes or more is necessary for the satisfactory formation of the foamy layer.

The results of the material balance and yields of flotation for each test are reported in Table 3. The chemical analysis of the concentrates products of flotation process are reported in Table 4.

Table 2

Chemical composition (wt %) of kaolin ore (TK)

Sample	SiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	LOI	SiO_2/Al_2O_3
TK	46.70	36.67	2.70	0.02	0.21	0.12	0.19	2.58	0.28	0	9.36	1.27
Kaolin: KGa-1b, (low-defect)	44.20	39.70	0.21	0	0.03	0	0.013	0.05	1.39	0	13.49	1.11
Ideal kaolinite*	46.55	39.50	–	–	–	–	–	–	–	–	–	1.18

LOI: measured at 1000 °C.
ORIGIN: Tuscaloosa formation , County of Washington, State of Georgia, USA
Ideal kaolin: Refers to a high-purity, fine-grained, absence of impurities, high whiteness and brightness, with excellent plasticity, thermal stability, and other specific properties suited to its intended application, such as ceramics, paper, paint, or pharmaceuticals productions.

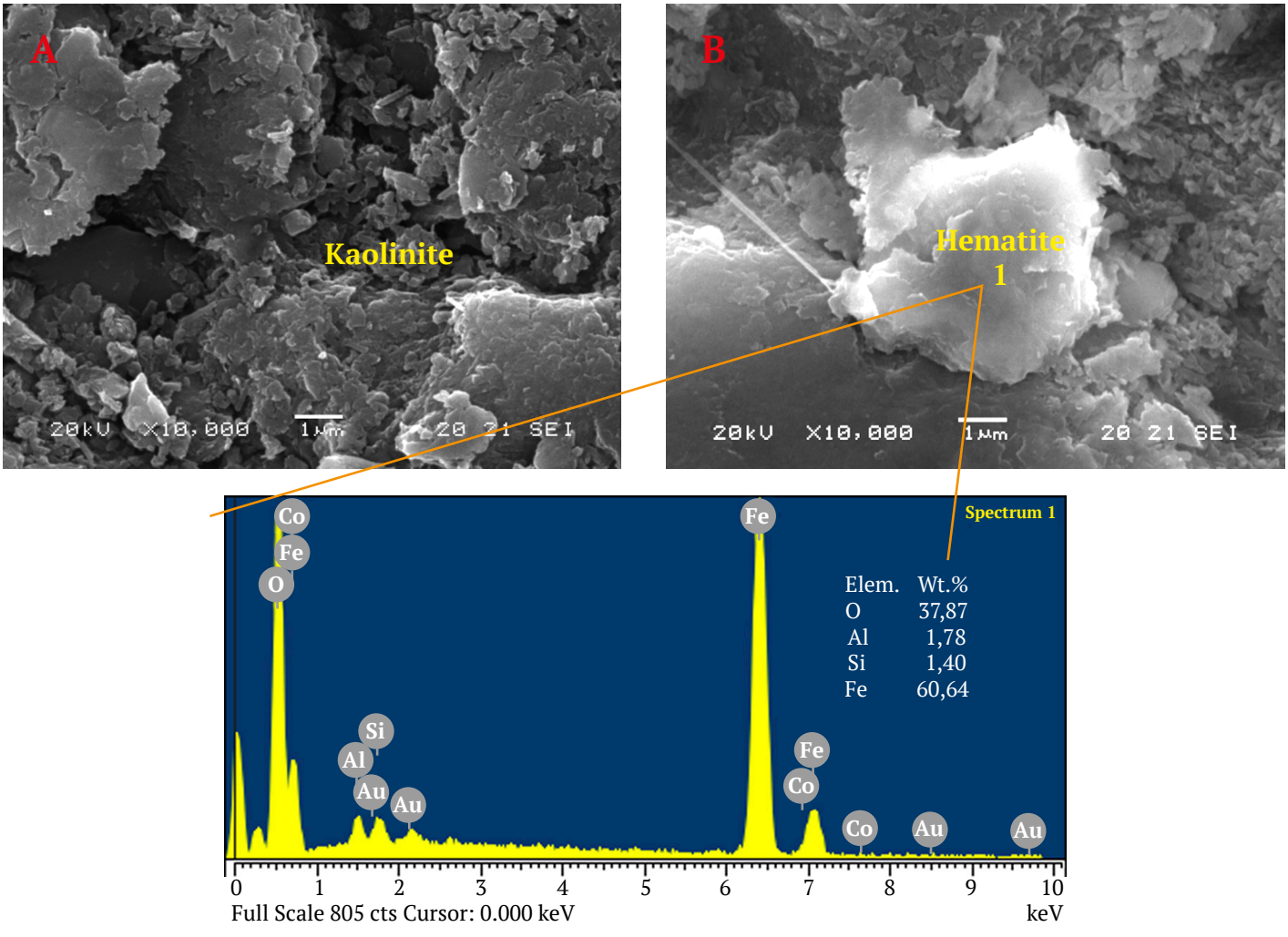

Fig. 3. SEM-EDX of Tamazert kaolin (TK)

Calculation of technological indicators

Each separation product can be characterized by qualitative and quantitative indices. For this reason, several equations were used [21]:

– material balance equation for separation products:

$$Q_f = Q_c + Q_t; \quad (1)$$

– balance equation for yields, %:

$$\gamma_f = \gamma_c + \gamma_t; \quad (2)$$

– yield of concentrate, %

$$\gamma_c = \frac{Q_c}{Q_f} \cdot 100; \quad (3)$$

– yield of tailings, %:

$$\gamma_t = \frac{Q_t}{Q_f} \cdot 100, \quad (4)$$

Q_f – masse of feed, t; Q_c – masse of concentrate, t; Q_t – masse of tails, t ; γ_f – yield of feed, %; γ_c – yield of concentrate, %; γ_t – yield of tailings, %.

Tamazert kaolin shows a good flotation performance. The yields and the contents results of the concentrates and tails of Tamazert kaolin (TK) after flotation process are illustrated in Figs. 5 and 6. The optimum yield value is in the fraction between 20 and 40 μm with 79.84 wt. % and 19.92 % by wt.% for the concentrate and the tailing respectively.

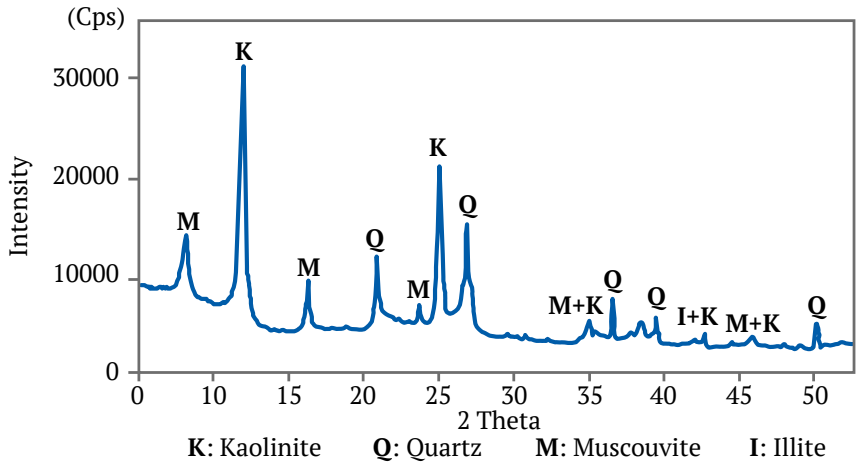

Fig. 4. XRD pattern of Tamazert kaolin (TK)

Table 3

Material balance and yields of the flotation tests

Test N°	1		2		3		4		5	
Fraction, μm	0–20		20–40		40–80		80–100		100–120	
Material balance, g	Q_{c1}	Q_{t1}	Q_{c2}	Q_{t2}	Q_{c3}	Q_{t3}	Q_{c4}	Q_{t4}	Q_{c5}	Q_{t5}
	190.5	58.9	199.6	49.8	185.6	63.7	162.5	85.7	148.6	100.5
Q_F, g	249.4		249.6		249.5		249.2		249.5	
Yields, %	γ_{c1}	γ_{t1}	γ_{c2}	γ_{t2}	γ_{c3}	γ_{t3}	γ_{c4}	γ_{t4}	γ_{c5}	γ_{t5}
	76.25	23.30	79.84	19.92	74.24	25.26	65	34.28	59.44	40.20

Table 4

Material balance and yields of the flotation tests

Fraction, μm	Oxide, wt.%											
	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	MgO	CaO	Na_2O	K_2O	MnO	P_2O_5	LOI	SiO_2/Al_2O_3
0–20	49.13	38.81	0.15	0.05	0.10	0.06	0.07	1.05	00	00	10.58	1.26
20–40	49.38	38.97	0.08	0.04	0.11	0.06	0.09	1.11	00	00	10.19	1.27
40–80	48.95	38.79	0.13	0.09	0.12	0.08	0.11	1.23	00	00	10.42	1.26
80–100	48.14	36.85	0.19	0.12	0.15	0.10	0.12	1.28	00	00	12.98	1.30
100–120	47.31	36.72	0.28	0.20	0.19	0.10	0.16	1.41	00	00	13.59	1.29

It can be seen from Table 3, Figs. 5 and 6 that, on the whole, the yield of concentrate decreases with increasing kaolin particle sizes. There is general agreement: flotation efficiency decreases with increasing particle size [22].

The content of K_2O decreased from 2.58 to 1.05 wt. % in the fraction < 20 μm; this indicate a significant removal of illite and/or muscovite.

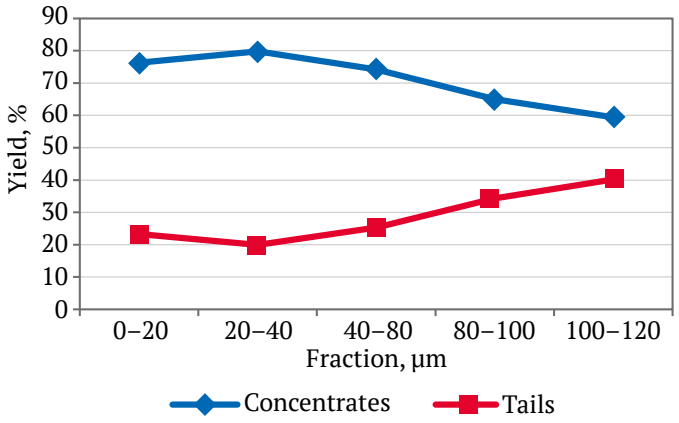

Fig. 5. Yields of the concentrates and tailings of Tamazert kaolin (TK)

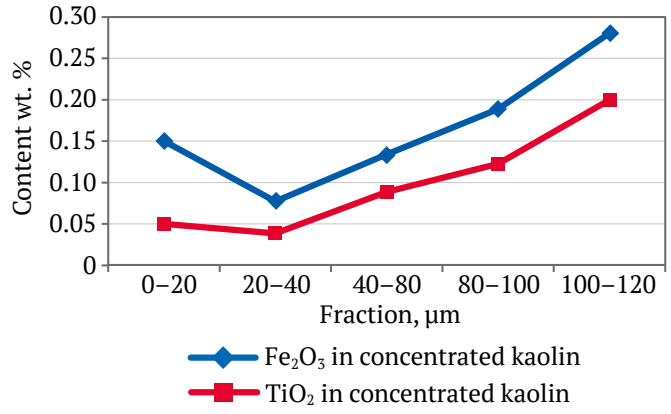

Fig. 6. Fe_2O_3 and TiO_2 contents in concentrated Tamazert kaolin after flotation process

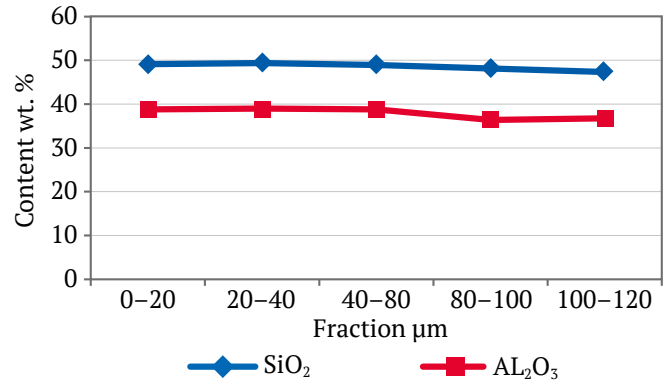

Fig. 7. Al_2O_3 and SiO_2 contents in concentrated of Tamazert kaolin after flotation process

The chemical results (Table 1 and Table 4) and Figure 7 show that the SiO_2 and Al_2O_3 contents of the analyzed sample increased from 46.70 to 49.38 wt.% and 36.67 to 38.97 wt.%, respectively, in the fraction of 20–40 μm. This means logically that the kaolinite content will increase and the whiteness also will be increased.

All these analyses show that the dimensions of particles have a significant influence on the enrichment of Tamazert kaolin (TK) situated between 0 to 40μm.

Comparing the chemical analysis of ideal and Georgian kaolin (KGa-1b) and chemical analysis of processed Tamazert kaolin (TK) can confirm that the flotation beneficiation gives satisfactory results.

Conclusion

Most commonly, the presence of Fe_2O_3 and TiO_2 are the first impurities that cause the coloring of kaolin and reduce its commercial value and industrial utilization. Several tests were performed on the Tamazert kaolin sample using froth flotation process, in order to meet commercial specifications.

The chemical analysis showed that Tamazert kaolin (TK) is an alumino-silicate with contents of 46.70 % SiO_2 and 36.67% Al_2O_3. The impurities as Fe_2O_3 and TiO_2 with 2.7% and 0.28%, respectively, give the brownish color to Tamazert kaolin (low quality) which is not suitable for many industries.

The results obtained using froth flotation are very encouraging. The test results showed that after the enrichment of samples, iron and titanium (the main coloring impurities in Tamazert kaolin) were removed from 2.7 to 0.08 wt.% for Fe_2O_3 in the fraction of 20–40 μm and from 0.28 to 0.04 wt.% for TiO_2 in the same fraction as the optimum test. These results confirm that iron is not substituted in the structure of kaolin and that it is in the free-state.

The contents of SiO_2 and Al_2O_3 after froth flotation of the samples analyzed were increased from 46.70 to 49.38 wt.% and 36.67 to 38.97 wt.%, respectively, in the fraction of 20–40 μm. This means logically that the kaolinite content will increase and the whiteness and brightness will also increase.

Moreover, this study highlighted the importance of dimensional distribution, and has a significant influence on beneficiation of Tamazert kaolin (TK) using froth flotation process. The best results were obtained in the fraction from 0 to 40μm.

Comparing the chemical analysis of Georgian kaolin (KGa-1b) and ideal and treated Tamazert kaolin (TK) confirmed that the froth flotation process can be a potentially effective process to remove Fe_2O_3 and TiO_2 with satisfactory results.

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