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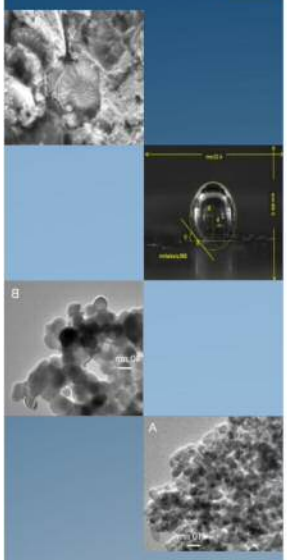

RECENT DEVELOPMENTS IN METALLURGY, MATERIALS AND ENVIRONMENT

CHAPTER 15

Characterization and Study of Gravimetric Separation of Kaolinitic Sand coming from Agua Blanca de Iturbide, Hidalgo (Mexico)

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ÁREA ACADÉMICA DE CIENCIAS DE LA TIERRA Y MATERIALES, INSTITUTO DE CIENCIAS BÁSICAS E INGENIERÍA DE LA UNIVERSIDAD AUTÓNOMA DEL ESTADO DE HIDALGO

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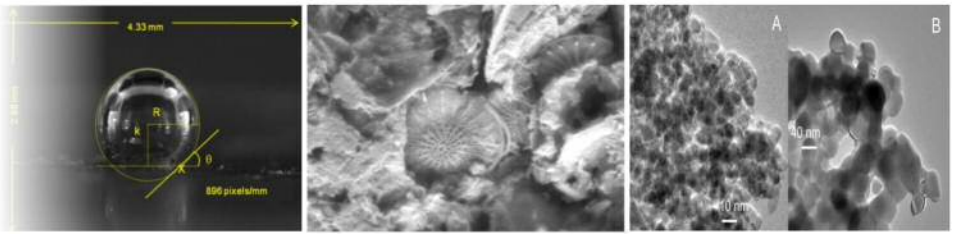

Materials

CHAPTER 15

Characterization and Study of Gravimetric Separation of Kaolinitic Sand coming from Agua Blanca de Iturbide, Hidalgo (Mexico)

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Abstract

In the present research characterization of kaolin waste coming from municipality of Agua Blanca de Iturbide in State of Hidalgo (Mexico) was carried out, the powders analyzed included particles that oscillate between 37 and 500 micrometers. To make their characterization X-Ray Diffraction, Scanning Electron Microscopy, Chemical Analysis with Inductively Coupled Plasma (ICP) and distribution of analysis of sizes per gradation test were used. Gravimetric separation was also carried out in Wilfley concentrating table studying three parameters: flow for dilution water, table inclination and blows per minute (RPM). Obtained products were chemically analyzed, obtaining Fe and Ti oxides, and aluminum silicates.

Keywords: Kaolin; Si Oxide; Gravimetric Separation

1. Introduction

Mexico is one of the countries with great quantity of mineral resources, and not only in case of metallic minerals, but also in the non-metallic ones. Twelve states of Mexican Republic have important kaolin

($Al_2O_3 \cdot SiO_2 \cdot 2H_2O$) deposits, being Hidalgo, Zacatecas, Veracruz, and Guanajuato, states that have figured between the first places in production for this mineral in last years [1]. Within geographic zones with more importance in kaolin deposits in State of Hidalgo is Agua Blanca de Iturbide whose geologic potential extends up to state of Veracruz, for the most part in Huayacocotla, however, the economic value of their minerals is very low due to the great quantity of impurities that contains [1, 2].

In general, kaolin deposits in Mexico are very irregular, they exist in diverse qualities, very heterogeneous particle sizes and with diverse impurities such as, iron, titanium oxides, quartz and silica [3]. One of their main impurities are SiO_2 sands which is a relatively stable compound; it is a by-product that is created during geologic formation of kaolin (clay made up of very fine particles) which means that to be able to obtain it is absolutely necessary to carry out sizes separation whose thick particles correspond for the most part to silica and for this reason it is considered important to process it and to give it an use because these sands generate to the companies an important monetary loss in long term [4, 5].

Basically, their processing is divided in two different paths, according to its final quality: in dry and wet. The first one is mainly used in kaolins with greater silica content and the second one in kaolins with high alumina contents [6-8]. In dry processing only sand is removed, kaolin is classified by particle sizes and it is dried off. In the last one, there are also carried out other steps in which water is used for cleaning to obtain finer product and of greater quality; this last one is used mainly for paper industry [8].

In some kaolin purification plants, after mechanical preparation there is a stage that is called degritting; these sands contain high silica, alumina and other impurities percentage, which represent a problem to environment [9-12]. By this reason in this work silica sands obtained from processing of kaolin mineral coming from Agua Blanca de Iturbide, Hidalgo (Mexico), are physical and chemically characterized, determining their species and separating them by means of Wilfley concentrating table with the purpose of finding them alternative use and the viability of giving them value added instead of discard them.

2. Experimental procedures

As raw material it was used samples of kaolin mineral obtained directly from mines of Agua Blanca de Iturbide, Hidalgo. Material coming from mine reduced its size by means of an Allis Mineral Systems' jaw crusher, model N184T17FB12C, until obtaining 100% of material pieces at - ¼ inches. Then, broken mineral fed to a Denver flotation cell with double-blade propeller, to which was added water until obtaining a homogeneous and thick pulp, without determining the percent of solids in weight. Pulp went by a series of Tyler sieves, with help of water, with the purpose of carrying out sieve analysis.

Head ore constituted by particles of sand, whose sizes ranged between 500 and 37 μm, was sampled and pulverized in agate mortar to then analyze it chemically with a Perkin Elmer brand spectrometer of high dispersion of plasma coupled inductively (ICP), model Optima 3000 XL. o this mineral it was made a crystallographic analysis in an Inel brand X-ray diffractometer, model Equinox 2000, with a step of 0.02º in the range of 10-110º (2θ) using radiation of Cu-Kα (λ = 1.5418 Å) and to determine its morphology, was used a Jeol brand scanning electron microscope, model JSM-6300, also it was carried out semiquantitative chemical analysis by energy dispersion spectroscopy (EDS) to determine chemical composition of powders to corroborate the approximate percentage concentration of iron oxides, (Fe_2O_3), titanium oxides (TiO_2), silica (SiO_2), and alumina (Al_2O_3) in sands sample.

Such sands were separated by means of laboratory Wilfley concentrating table standard type 13 A, and during all tests pulp feeding was constant with a flow rate of sands of 15 kilograms per hour at 18% of solids in weight, studying three parameters: flow for dilution water, table inclination and blows per minute (rpm) provided by tapping mechanism and that it can be voluntarily adjusted by means of a crank (rpm). In all tests two products were obtained: heavy and light, which were filtered in dry and weighed to carry out corresponding characterization and to obtain the recovery percentages in weight.

3. Results and discussion

In this section, results of the tests carried out with the samples in the above mentioned conditions are shown.

3.1 Characterization

The material was processed in dry, coming from the mine, reducing it in size, quartering it and attritioning its pulp, then to make an analysis of particle sizes distribution , as well as chemical analysis for each obtained product of each mesh. The obtained results are shown in Table 1.

Sieves analysis or gradation test that appears in retained percentages in each mesh, indicates that 29.6% of material are sands, material subject of this study. More than 70% in weight of sample show sizes lower than 37 μm, due to origin of clayish material. Please note also that percentages in weight of ranges between 297 and 210 μm, and from 149 a 125 μm show the most important values (4.5%). The average calculated of particle size of sands, corresponds to 68.6 μm.

Size (μm)	wt %	Fe_2O_3	TiO_2	SiO_2	Al_2O_3
Heat mineral analized		0.802	0.512	31.36	58.39
+595	0.00	-	-	-	-
-595 +500	1.70	0.869	0.43	27.00	58.32
-500 +297	3.90	0.875	0.457	24.48	58.71
-297 +210	4.50	0.786	0.421	27.97	55.67
-210 +149	2.80	0.873	0.446	28.00	57.51
-149 +125	4.50	0.926	0.555	29.86	53.86
-125 +105	1.70	0.943	0.519	24.25	58.16
-105 +63	2.80	1.061	0.528	26.31	61.29
-63 +53	3.90	0.895	0.406	28.77	60.43
-53 +44	2.80	0.864	0.428	36.19	53.54
-44 +37	1.10	0.866	0.472	30.26	57.33
-37	70.40	0.788	0.557	35.33	57.21
	100				
Standard deviation		0.075	0.051	2.203	1.432

Table 1 Sieves analysis of mineral mine.

With the aim of working with a narrow range of particle sizes and whose retained percentage was important, there were worked with kaolin sands with sizes that range between 500 and 37 micrometers. Then from now on results will only be referring to work carried out with sands inside this range.

Crystallography study of kaolin sands is shown in Figure 1, analysis shows mainly two mineral species, kaolinite and silica; this last one is detected as the main phase, in quartz, tridymite and cristobalite form.

This fact shows that there is kaolin adhered to sands. Within other impurities detected there can be signs of iron and titanium oxides (Fe_2TiO_4) and greigite (Fe_3S_4). These results suggest proposal of carrying out some processing to sands, with the purpose of increasing the recovery in weight of kaolin clays.

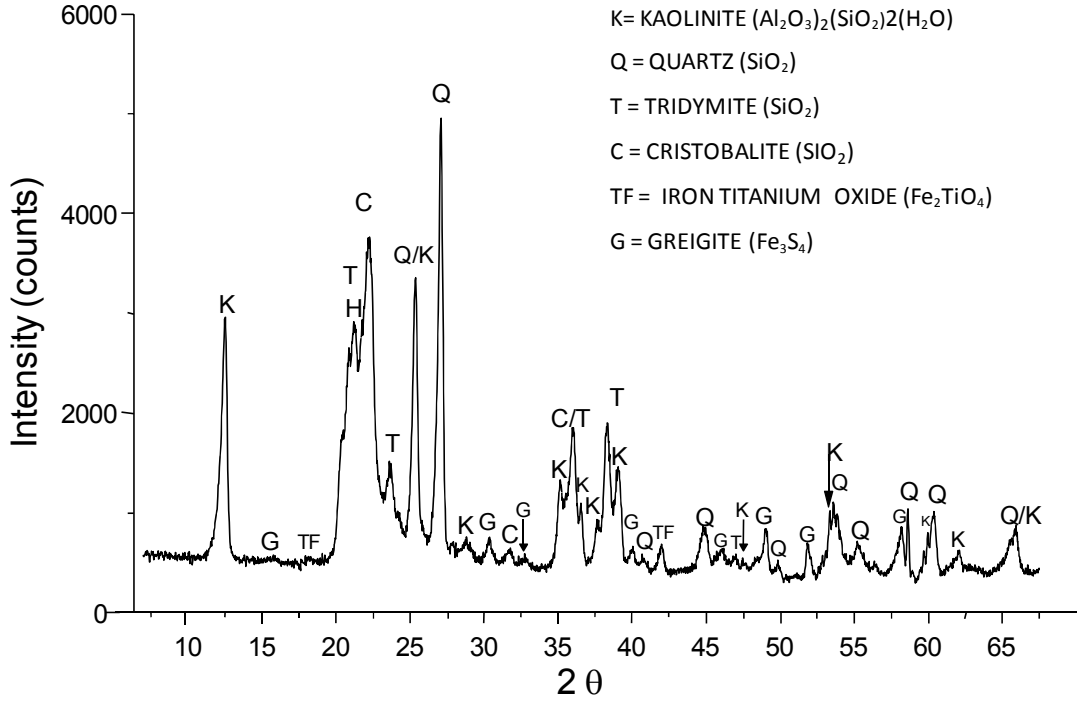

Figure 1. Diffractogram of mineral species of kaolin sands whose particle sizes range between 500 and 37μm.

Also to these sands there were done chemical semiquantitative analysis by energy dispersion spectroscopy (EDS) to determine their chemical composition and to corroborate the approximate percentage concentration of silica and alumina in the sample, these results are shown in Table 2 where there can be appreciated that has 30.140 % de SiO_2 and 55.39 % de Al_2O_3.

In Figure 2 an image is presented at low zooms of morphology of kaolinite sands, which show an irregular shape, a smaller proportion has lengthened form, some are surfaces with a compact consistency and other surfaces are porous. The compact surfaces could correspond to silica particles

and the porous particles to mixture of mineralogical species of silica and kaolin clay. The particle sizes distribution is concordant with that obtained and shown in granulometric analysis.

Sample	Chemical analysis (%)			
	Fe_2O_3	TiO_2	SiO_2	Al_2O_3
Heat mineral analized	0.80	0.302	29.902	57.500
Sands analized by SDE (+/- 0.2%)	0.99	0.240	30.140	55.390

Table 2. Chemical semiquantitative analysis by energy dispersion spectroscopy (EDS), compared with the calculated head.

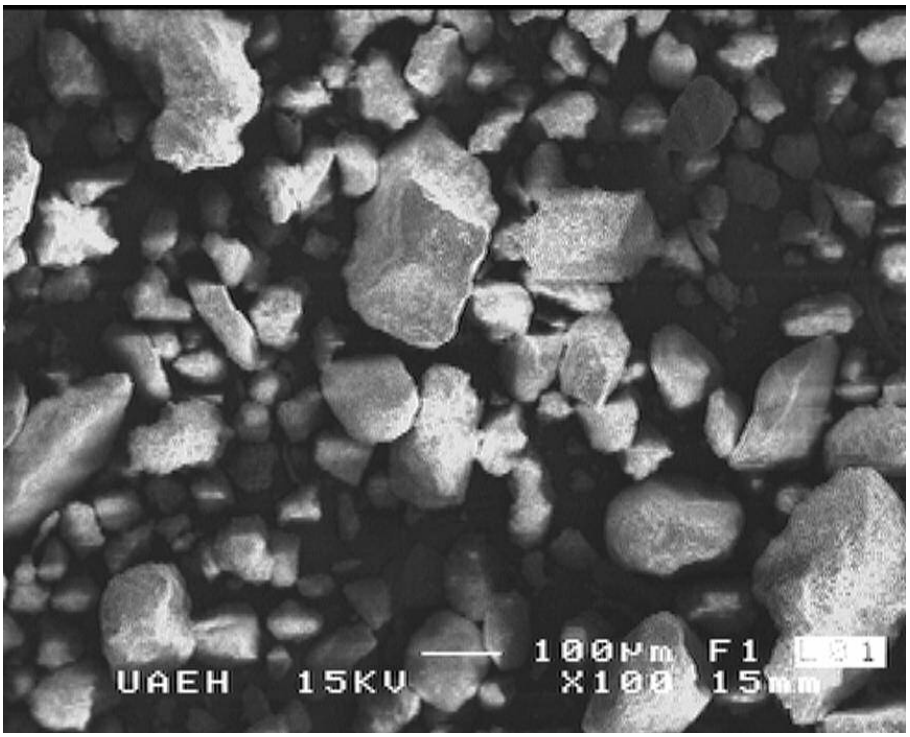

Figure 2. Image of Scanning Electron Microscopy of kaolin sands.

Once characterized the sample of sands, gravimetric separation was done based on formula of concentration approach that for this case was 4, and therefore it was feasible to separate, by means of concentrating table, part of impurities (Fe and Ti oxides) contained in kaolin clays. The analysis of results obtained in each one of the tests, was mostly focused in the weight recoveries obtained and in chemical analyses of light products, due to the decrease of impurities in these products, is a good indication of separation efficiency.

3.2 Study of effect in quantity of dilution water

With the purpose of studying flow rate of diltuion water for pulp in concentrating table, three different flows at 3, 6 and 9 $Lmin^{-1}$ of water were added. During whole test, there were kept constant feeding of 15 kg h^{-1} at 18% of solids in weight, an inclination angle of 8° and the blows per minute in 320 rpm. The chemical analyses of obtained products, that is to say of heavy and light ones, are shown in Table 3.

Dilution water ($L\ min^{-1}$)	Product	wt %	Chemical analysis (%)			
			Fe_2O_3	TiO_2	SiO_2	Al_2O_3
3	Heavy	24.6	0.853	0.412	23.90	62.20
	Light	75.4	0.666	0.260	31.97	57.19
6	Heavy	18.8	1.019	0.449	21.19	60.05
	Light	81.2	0.605	0.268	31.92	55.58
9	Heavy	9.6	1.131	0.549	18.45	67.31
	Light	90.4	0.639	0.271	31.08	49.31
Head mineral calculated		100	0.8	0.302	29.902	57.50

Table 3. Chemical Analysis of the products of concentrating table, when studying dilution water effect.

The results show a low percentage in weight of heavy products; which is due to low concentrations of Fe and Ti. Note that for light products, there is a decrease of contents of Fe_2O_3 and TiO_2 for the three tests carried out at different water flows, however, an increment is shown for silica SiO_2, but the alumina contents Al_2O_3 remain practically stable. In left side bar of Figure 3 percentages of analyses of sample of sands from head mineral are shown, that is to say, before being separated and it is compared with results obtained in light materials.

It shows a maximum reduction of oxides of Fe and Ti to the test on a flow of 6 $Lmin^{-1}$, with recovery of 81.2% for the light materials. This water flow allows an optimal force to sweep the light material, without drag the heavy particles. Therefore, it was decided to fix a flow rate of 6 $Lmin^{-1}$ for the consequential experiments with adequate separation of minerals containing Fe_2O_3 and TiO_2, obtaining an acceptable recovery.

3.3 Study of inclination angle effect

During this study, there was kept constant feeding of 15 kg h^{-1} at 18% of solids in weight, a flow rate of dilution water of 6 $Lmin^{-1}$ and blows per minute in 320 rpm. The angles of 4.5°, 8°, and 11.5° of inclination were calculated. The preliminary tests carried out with angles lower than 8°, demonstrated that there is not flow of pulp, stagnating the mineral in the rifles of table, then there were only worked with 8° and 11.5°. The results of the chemical analyses are shown in Table 4.

The results obtained in this study show a percentage in weight of recovery of lower mineral at an angle of 11.5° because an important part of the mineral is dragged to area of light materials, taking these an important proportion of the undesirable oxides (refer to Figure 4).

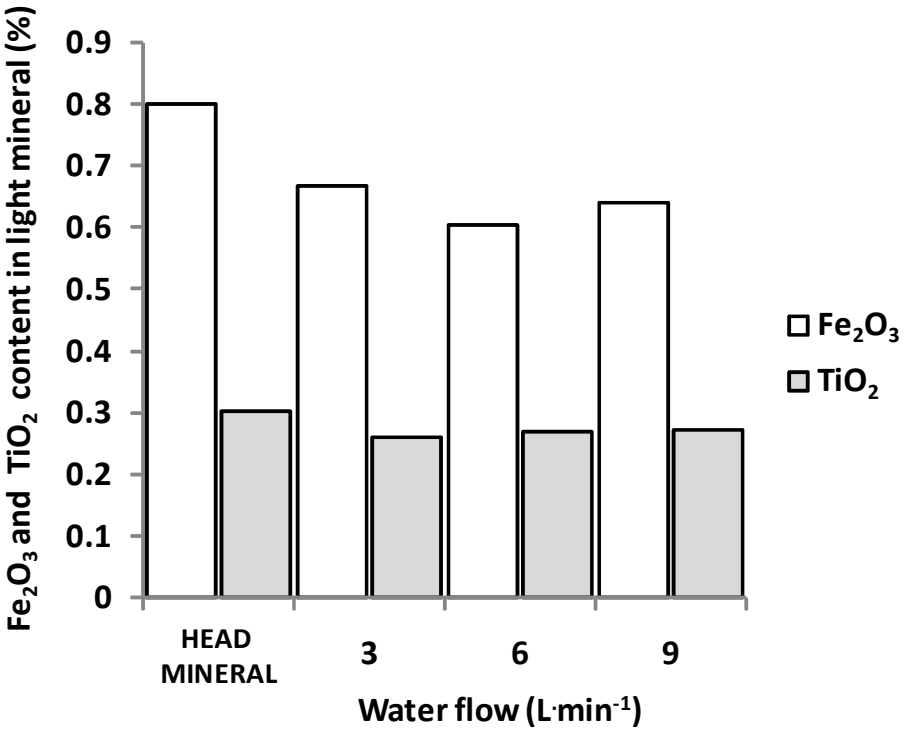

Figure 3. Comparison of the contents of Fe_2O_3 and TiO_2 of light materials, with respect to tests carried out in Wilfley table, varying water flow.

Tilt angle	Product	wt %	Chemical analysis (%)			
			Fe_2O_3	TiO_2	SiO_2	Al_2O_3
11.5 °	Heavy	15.2	1.3	0.8	11.1	64.3
	Light	84.8	0.7	0.4	21.7	57.5
8 °	Heavy	18.8	1.019	0.449	21.19	60.05
	Light	81.2	0.605	0.268	31.92	55.58
Head mineral calculated		100	0.8	0.302	29.902	57.50

Table 4. Chemical analysis of products of Wilfley table, when was studied the inclination angle effect.

While chemical analyses of light products demonstrate decrease in concentrations of Fe_2O_3 and TiO_2, for both inclinations, results with more decrease of such concentrations are presented when using an inclination angle of 8°; the percentage difference in weight between one test and the another is only of 3.6 percentage points for which is decided to use in tests 8° of inclination for the table.

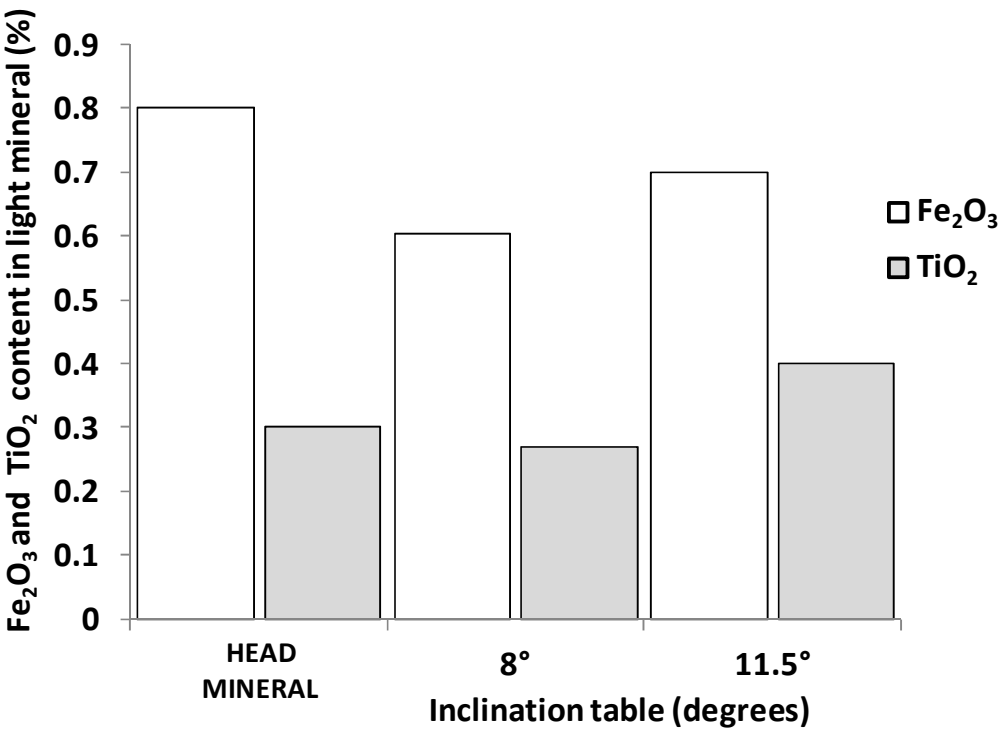

Figure 4. Comparison of contents of Fe_2O_3 and TiO_2 for light materials, with respect tests carried out in Wilfley table, varying inclination angle of the table.

3.4 Study of revolutions per minute effect

During the study of this parameter, there was kept constant feeding of 15 kg h^{-1} at 18% of solids in weight, a flow rate of dilution water 6 $Lmin^{-1}$ and an inclination angle of 8°. The results of chemical analysis of each species are shown in Table 5.

rpm	Product	wt %	Chemical analysis (%)			
			Fe_2O_3	TiO_2	SiO_2	Al_2O_3
220	Heavy	21.2	0.906	0.649	14.802	59.96
	Light	73.9	0.781	0.475	24.174	49.59
270	Heavy	16.8	0.979	0.834	11.677	57.35
	Light	83.2	0.778	0.441	26.485	50.73
320	Heavy	18.8	1.019	0.449	21.19	60.05
	Light	81.2	0.605	0.268	31.92	55.58
Head mineral calculated		100	0.8	0.302	29.902	57.50

Table 5. Chemical analysis of products of concentrating table, when studying revolutions per minute effect.

When comparing the three tests it can be observed in this table that use of 270 rpm shows, in chemical analyses, highest recovery in mineral weight. However the contents of TiO_2 in light products increases. When using 320 rpm, concentrations of evaluated impurities decrease and the recovery in weight is acceptable. The silica contents and alumina don't differ from those of the head. Consequently it has been considered that a good separation is obtained using 320 rpm (see Figure 5)

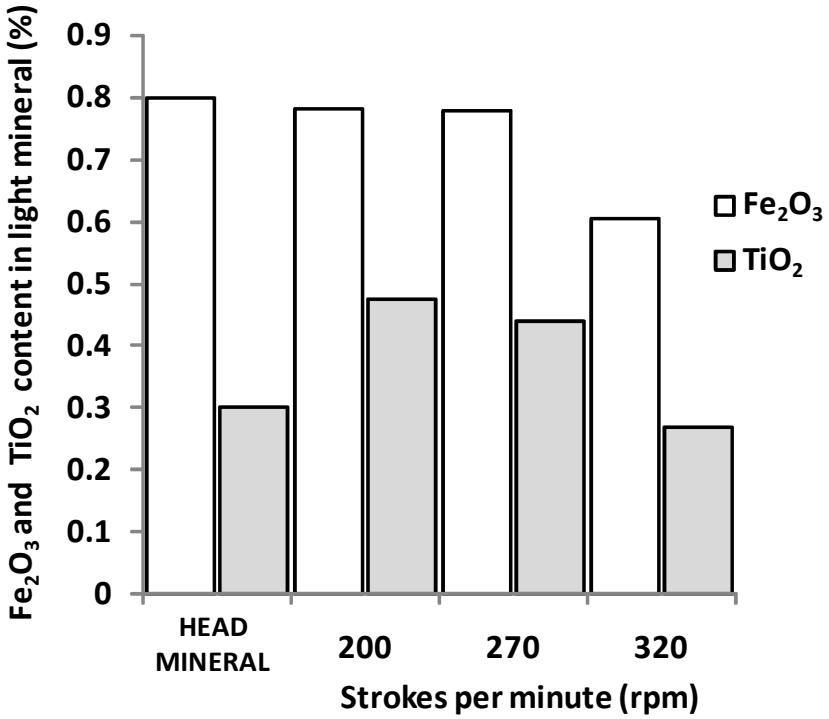

Figure 5. Comparison of contents of Fe_2O_3 and TiO_2 of light materials, with respect to tests carried out in Wilfley table, varying rpm.

4. Conclusions

In this work it was characterized and carried out study of gravimetric separation of kaolin sands coming from Agua Blanca de Iturbide, of Hidalgo State (Mexico). A physical and chemical characterization of origin samples showed mainly two mineral species, kaolinite and silica; this last one was detected as main phase, in quartz form, tridymite and cristobalite. Inside the other detected impurities signs of iron and titanium oxides (Fe_2TiO_4) could be observed and of griegite (Fe_3S_4). The study suggested that it is important to carry out a decrease in size of particles in order to release silica from aluminum silicates. The chemical analyses in obtained products put in evidence the separation of silica particles. The study of gravimetric separation in Wilfley table carried out in this work showed that operation parameters used from the beginning were the most appropriate, under conditions of feeding pulp, 15 kg h^{-1} and 18% of solids in weight. In all cases the use of table was appropriate for separation of Ti and Fe compounds that were found when characterizing by X-ray diffraction and chemical analysis. It is important to highlight that it should be used enough water to drag the material and to make the separation function, however the use of high flows of water impedes the work of rifles of table dragging practically whole material to storage of light products, finding that optimum flow of dilution water was of 6 $Lmin^{-1}$. As for the inclination of concentrating table it was evident that it is not appropriate to work with inclinations from 0 to 7° because this generates that solid material is stagnated and working from that on with inclinations of 9° causes that most of the mineral moves to

storage of light products, finding that optimum angle of inclination is that of 8°. Finally the most appropriate rpm was when working at 320 since better recoveries were obtained.

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