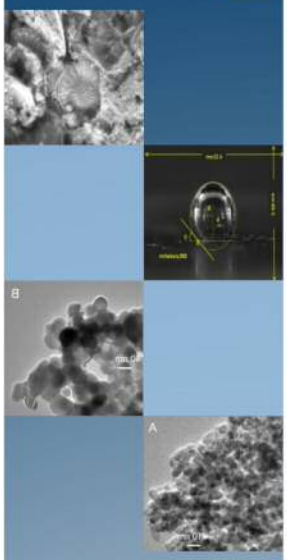

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RECENT DEVELOPMENTS IN METALLURGY, MATERIALS AND ENVIRONMENT

CHAPTER 14

Purification of Kaolin Clays by Means of Electrochemical Techniques

Flores Segura J. C., Reyes Cruz V. E., Legorreta García F., Hernández Cruz L.E. and Veloz Rodríguez M. A.

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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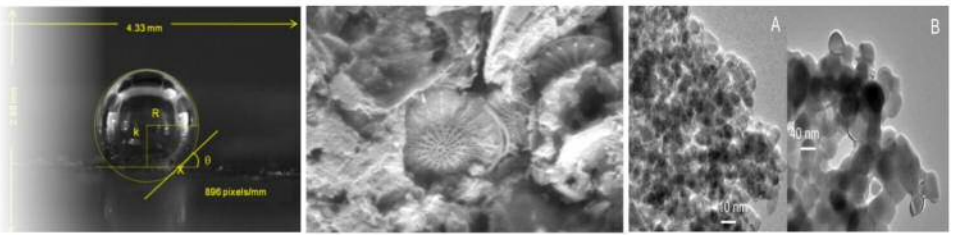

Materials

CHAPTER 14

Purification of Kaolin Clays by Means of Electrochemical Techniques

Flores Segura J. C., Reyes Cruz V. E., Legorreta García F., Hernández Cruz L.E., and Veloz Rodríguez M. A.

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Abstract

In the present work a kaolin clay sample was studied and its treatment was carried out with the purpose of separating iron oxides contained in it. This kaolin clay comes from Agua Blanca de Iturbide in Hidalgo State (Mexico). The clay was classified and treated to obtain micrometrical particles. This material in solution was studied by voltammetry, this solution showed a cathodic process attributed to reduction of iron in kaolin. Likewise, a subsequent process of macroelectrolysis at a controlled cell potential, showed that, when process is finished, iron is solved; helping to increase the purity of kaolin. The mineral original was characterized by X-ray diffraction (XRD), particle size analysis (PSA), scanning electron microscopy (SEM) and chemical analysis by high dispersion spectroscopy of inductively coupled plasma (ICP). The obtained product after treatment was characterized by ICP.

Keywords: Clays; Kaolin; Purification; Electrochemistry

1. Introduction

The kaolin is a clay product from decomposition of feldespar rocks, it is a silicate of hydrate of aluminum whose main component is the kaolynite, its formula is $2H_2O{\bullet}Al_2O_3{\bullet}2SiO_2$, it has a wide application in several industries; its consumption at world level was estimated for 2013 by Freedonia

Group on 24.8 million metric tons. Big locations exist in countries such as: United States, England, Brazil, Indonesia, China, etc., where kaolin is extracted and treated to purify it, and then to take maximum advantage of its properties [1].

In Mexico there are also important mineral veins, mainly in states of Veracruz, Guanajuato, and Hidalgo [2]. In Hidalgo there is a vein in the municipality of Agua Blanca de Iturbide [3], where kaolin presents an important quantity of impurity, which degrades its value and its properties (mainly its whiteness due to iron oxides) [4].

Wet purification process, done in most of plants where kaolin clays are treated around the world, includes mechanical methods as extraction, degritting, mixed, classified, decanted and dryed [1], some or the combination of the following ones: high intensity magnetic separation, flotation, lixiviation and flocculation [4-9].

Within the purification processes those that have acquired greater importance are: lixiviation process which consists on treating kaolin with acids in order to reduce iron oxides content and this way to whiten the polluted clays [7, 9], and that of flotation which consists on adding a hydrophobic surfactant to kaolin solution and to make pass air bubbles that catch the impurities [6, 8]. The application of several purification techniques causes the increase of the costs for its treatment [2].

Claudio Cameselle and collaborators in 2007 [10] found that application of electric potential in combination with use of oxalic acid in an electroremediation cell, helps to whiten kaolin increasing its whiteness to 80%, besides minimizing the quantity of reagents used in common treatment of purification for kaolin clays. The electroremediation process consists on placing a moistened soil between two holes with an electrolyte, so the transport mechanisms caused by application of an electric field concentrate pollutants on some of electrodes, these mechanisms are: electroosmosis, electromigration, electrophoresis, and diffusion.

The results obtained by Camesselle reveal that the electrochemical processes can be an alternative to confront the problem of purification of polluted kaolin. However, these studies also indicate that high energy consumption is required in the process when creating fronts with low pH.

For this reason, in the present investigation the problem is approached carrying out characterization and electrochemical studies of a solution of kaolin clay coming from the municipality of Agua Blanca, Hidalgo. It is necessary to mention that the application of these techniques in an electrolytic cell represents a decrease in the quantity of used energy, in comparison with the electroremediation treatment.

The kaolin clay was first mechanically processed until obtaining micrometrical particles. These particles were carried into solution. The corresponding voltammetric study was performed and also a chronopotentiometric study with the solution of kaolin clay in an electrolytic cell, with the purpose of knowing energy conditions on which iron oxides decrease. With the information obtained from these studies, it was carried out a macroelectrolysis study with the kaolin clays solution in order to carry out the removal of iron oxides.

2. Experimental procedure

Mechanical preparation consisted on wet sieve of the kaolin clay (from Agua Blanca, Hidalgo and marketed by the company "Molinos y Moliendas de Pachuca") with sieve -400 mesh to obtain particles with a smaller size of 37 μm. X-Ray diffraction was carried out with Inel X-ray diffractometer, model Equinox 2000, with radiation source Cu-Kα. Sample was dried at 120 °C. It was pulverized in an agate mortar and prepared in the equipment sampler. Clay morphology was observed by means of a scanning electron microscope, brand Jeol, model JSM 6300. The sample dried off at 120 °C was previously covered with gold. For the analysis of particle size, a small quantity of sample was placed in deionised water and it was exposed to ultrasonic bath during 3 min, then it was processed by means of analyzer of particle size by laser ray diffraction brand Beckman Coulter, model LS13320.
For chemical analysis, samples were digested with HF and H_3BO_3. Then, they were analyzed with an inductively coupled plasma spectrometer, brand Perkin Elmer, model Optima 3000XL.
For electrochemical study the techniques of cyclic voltammetry and chronopotentiometry were used and were done in an electrochemical cell of 3 electrodes. The electrodes used are: as working electrode a silver badge, as counterelectrode a DSA and as reference electrode SCE. The solution used for the voltammetric study was kaolin clays at 25% of solids in quantities of 300 mL. In the performing of electrochemical studies a potentiostat-galvanostat PAR263A was used. The techniques were applied through equipment software.
For macroelectrolysis study a power source brand GW Instek and 2 electrodes were used with a cell potential obtained from chronopotentiometric study.

3. Results and discussions

3.1 Characterization of the kaolin clay

Diffractogram of analyzed kaolin clay is described in Figure 1. The sample presents a high kaolynite content (JCPDS 01-078-1996) and, quartz (JCPDS 03-065-0466) in its main phases.

On the other hand, it is corroborated that this clay possesses an important quantity of impurities among those highlight the maghemite and iron or titanium oxides, being iron oxides those that contribute to the beige-reddish coloration to clays, when they are in significant quantities.

In Figure 2 the results obtained from analysis of particle size by laser ray diffraction are shown.

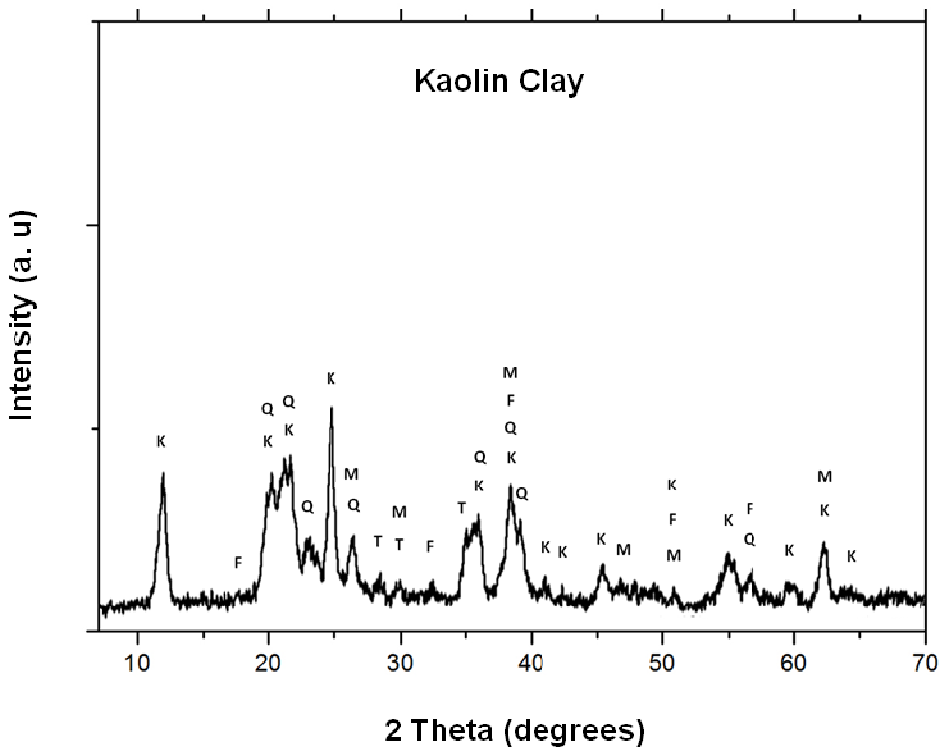

Figure 1. Diffractogram of X-ray for kaolin clay from the municipality of Agua Blanca, Hidalgo. (K=kaolynite; Q=quarz; M=Maghemite; T=titanium oxides; and F= iron oxides).

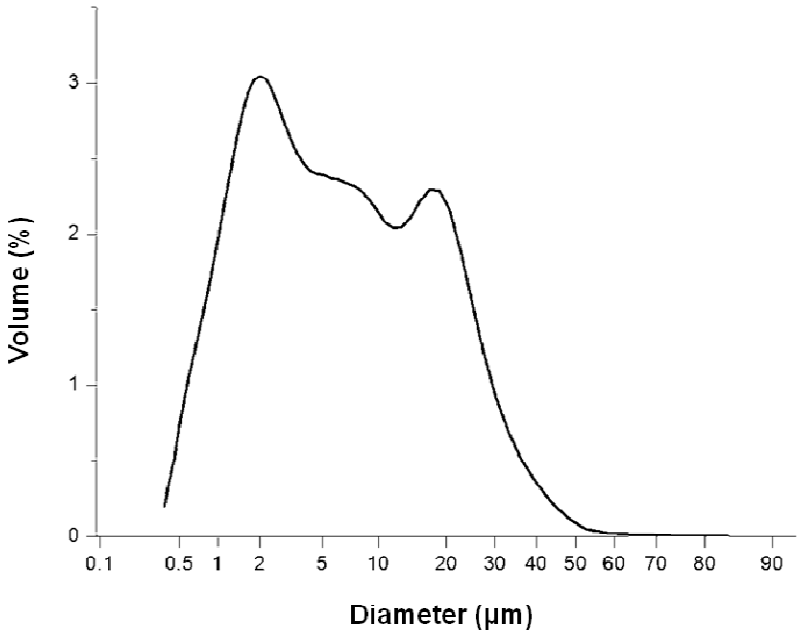

Figure 2. Distribution of particle size for the kaolin clay.

In Figure 2 two populations of main particles are observed, one with size of approximately 2µm that occupies the biggest percentage in the volume of the sample. The second majority population corresponds to particles with size of approximately 20µm, although the presence of particles can also be appreciated with sizes up to 60 µm and more. These sizes can be presented due to the loads of the fine particles causing interactions between them and forming accumulations of major size. This corresponds with that observed in the microscopy images obtained by scanning electron microscopy (SEM).

In Figure 3 microscopy of kaolin sample is presented. It was obtained with the SEM looking for accumulations of kaolin of major sizes, between 30 and 60 µm.

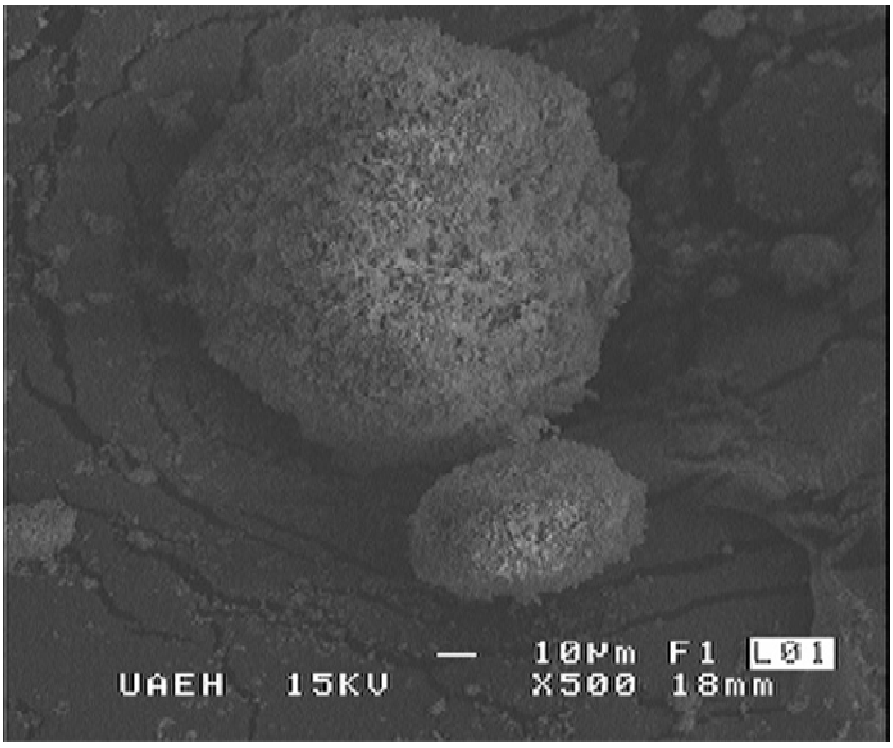

Figure 3. Microscopy of kaolin clay obtained with SEM.

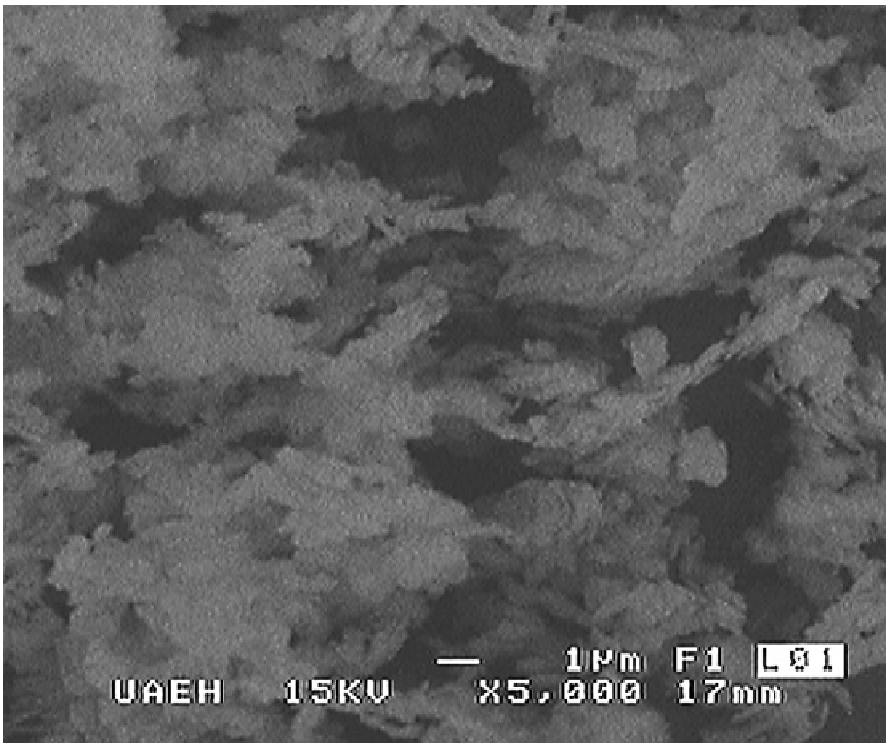

Figure 4. Microscopy of the kaolin clay obtained with the (SEM) at 5000 increases.

In Figure 4, the same sample of that in Figure 3 is observed, with more magnification although in low resolution. In the figure it is possible to observe stacking of plates which is characteristic of kaolynite.

The obtained results of chemical analysis, Table 1, show high contents of iron and titanium. Also, the proportion in weight observed between alumina (Al_2O_3) and silica (SiO_2) does not correspond with the formula of kaolynite, which is 1:2, this is due to the quartz presence in excess. It is assumed that all alumina present is forming part of the kaolynite.

% Wt			
Fe_2O_3	**TiO_2**	**Al_2O_3**	**SiO_2**
0.6834	0.6934	23.3334	18.8347

Table 1. Chemical composition of the sample of kaolin clay from Agua Blanca de Iturbide, Hidalgo, Mexico, obtained after acid digestion.

3.2 Electrochemical studies

In Figure 5 voltammetric response of kaolin solution is shown. It was performed at a scanning speed of 20 mV s^{-1} when the scanning begins in cathodic direction.

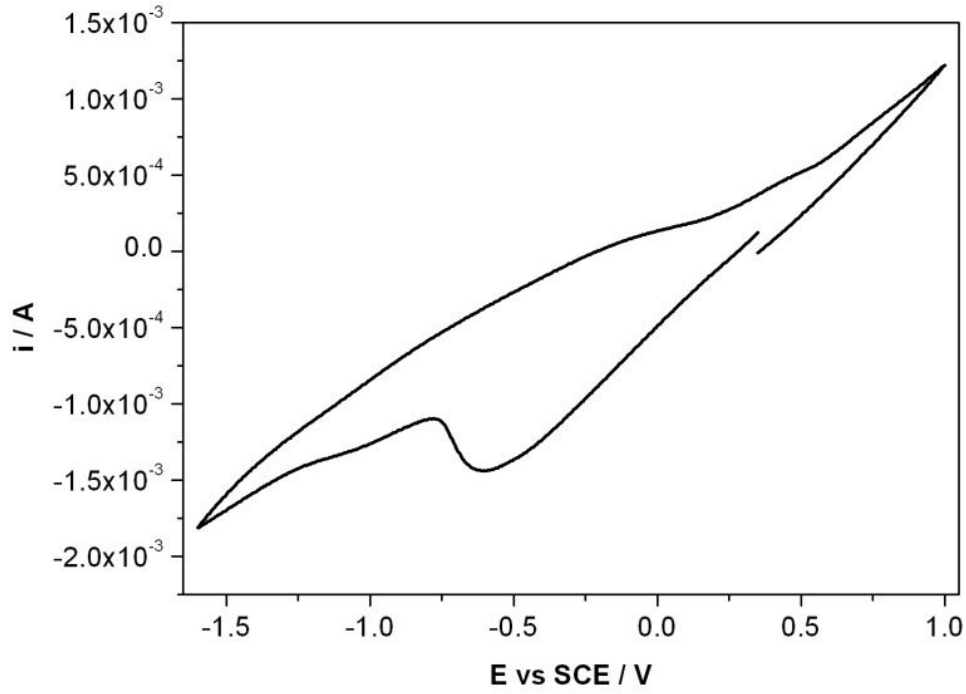

Figure 5. Voltammogram obtained on a surface of Ag in a kaolin (from Agua Blanca) solution to a scanning speed of 20 mVs^{-1}.

In Figure 5 two reduction processes are observed, one in the potential range of 0.280 to -0.780 V vs SCE and the other in the range of -0.780 to -1.6 V vs SCE. The first process is attributed to the reduction of iron species present in kaolin and the second to reduction of the medium. When the potential scanning is inverted, an oxidation process is observed, which begins at -0.167 V. This is attributed to the oxidation of the reduced products of the kaolin in the direct scanning.

These results indicate that reduction of iron present in kaolin could be carried out in the potential range between 0.28 and -0.780 V and at a current range from 0 to $-1.46x10^{-3}$ A.

In order to knowing the cell potential that should be used in an electrochemical arrangement of two electrodes, a chronopotentiometric study was carried out. The figure 6 shows the chronopotentiometric transient of kaolin reduction when a current of -1.46x10-3 A is imposed to the Ag electrode.

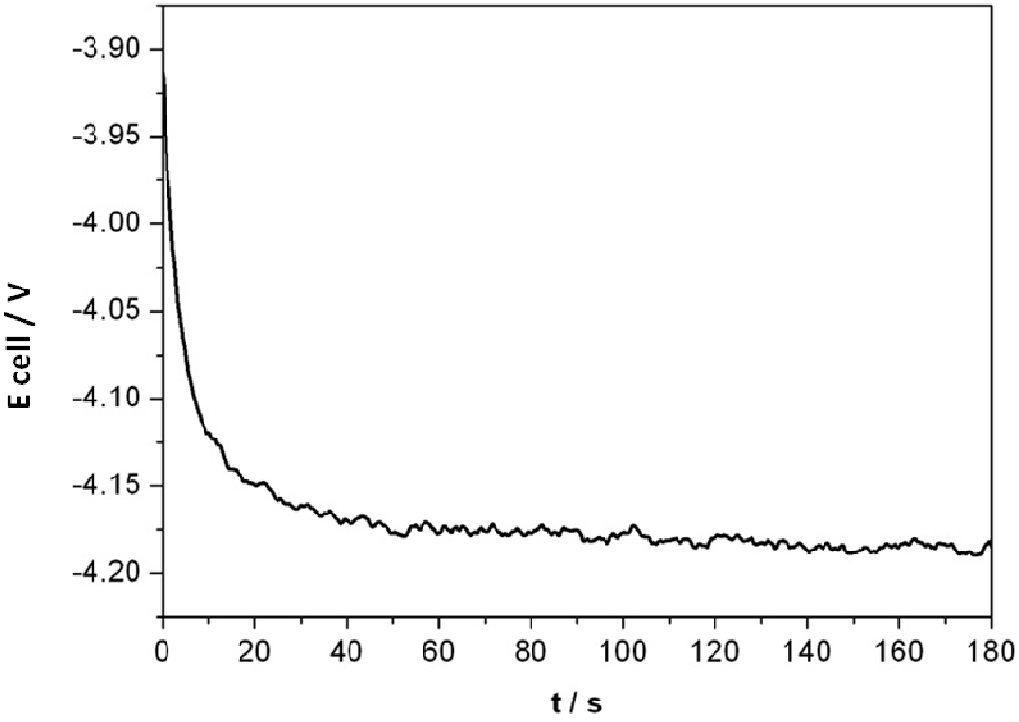

Figure 6. Chronopotentiometric transient obtained on a Ag surface in a kaolin (from Agua Blanca) solution when a current of -1.46x10-3 A was imposed.

In the figure 6, a considerable decrease of the cell potential is observed up to -4.16 V vs SCE in the interval of time from 0 to 30 s. At times greater than 30 s a constant potential is presented for the rest the electrolysis time. This behaviour indicates that only one species is being reduced and that it is not being finished in the solution; in this case the species is attributed to the iron oxide.

With the information of these results the process of macroelectrolysis of kaolin was carried out to obtain a higher transformation of iron reduced species present in the mineral.

In Table 2 are shown the results from chemical analysis with inductively coupled plasma (ICP) carried out to kaolin samples subjected to macroelectrolysis when a cell potential is imposed in the interval from 3.5 to 4.3 V.

APPLIED POTENCIAL	IRON OXIDE CONTENT ANALYZED	PERCENTAGE OF IRON OXIDE REMOVED
0V	0.6834	0
3.5V	0.6156	9.91850
3.7V	0.5859	14.2717
3.9V	0.5760	15.7075
4.1V	0.5656	17.2363
4.3V	0.5797	15.1689

Table 2. Results of the analyses carried out to kaolin samples subjected to macroelectrolysis when a cell potential is imposed in the interval from 3.5 to 4.3 V.

In Table 2 it is observed that as the cell potential increases the quantity of iron oxides present in the sample diminishes, until certain point. It is observed clearly that when increasing the cell potential from 3.5 V there is a tendency to decrease of content of iron oxides, until a maximum on 4.1 V. This is the cell potential given in the chronopotentiogram of the solution. This corroborates that iron oxides are the species decreasing in the kaolin. Once this point is overpassed, at 4.3 V, it is assumed that the reduction of the medium is also beginning, causing decrease in the reduction of the iron oxides. It is important because controlling the cell potential the quantity of iron oxides removed could be greater giving place to the obtaining of kaolin with more purity degree.

4. Conclusions

The results obtained by chemical analysis and XRD of kaolin clay indicated that a high kaolynite content is present, besides the quartz and impurities, these mainly from iron and titanium oxides. The microscopies obtained by SEM revealed the presence of particles with sizes bigger than 30μm, even though in their majority they correspond to fine particles, this was corroborated with the analysis of particle size that showed great extent of micrometrical particles with size of 2μm approximately.

The electrochemical studies (voltammetry and chronopotentiometry) showed that solution of micrometrical kaolin particles has a good response to reduction processes of the species present in this mineral. The potential interval where the species of iron in the kaolin decrease was located from 3.5 to 4.3 V.

The chemical analyses with coupled plasma after macroelectrolysis at controlled cell potential indicated that the electrochemical techniques are a viable alternative to purify the kaolin because as the potential increases the quantity of removed iron increased, achieving in this first studies a 17% of iron oxides removing.

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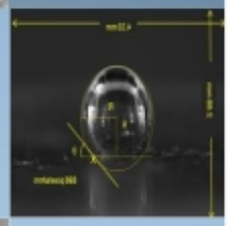

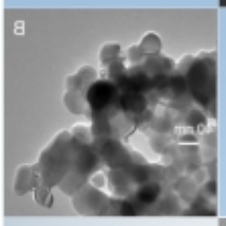

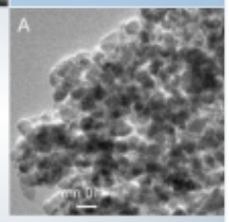

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Jorge López-Cuevas
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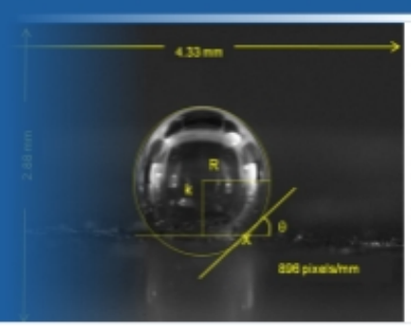

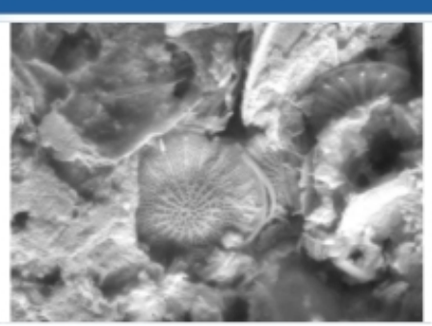

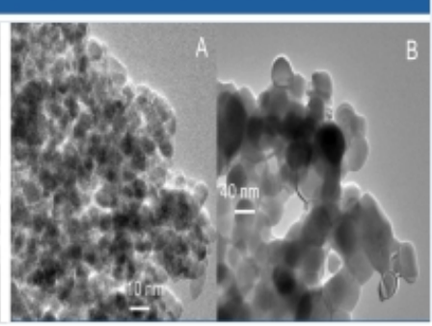

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