Synthesis of TiO$_2$ nano-powders prepared from purified sulphate leach liquor of red mud

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ABSTRACT

The research work presented in this paper is focused on the development of a purification process of red mud sulphate leach liquor for the recovery of titanium oxide (TiO$_2$) nano-powders in the form of anatase. Initially, titanium was extracted over iron and aluminium from the leach liquor by solvent extraction using Cyanex 272 in toluene, at pH: 0.3 and T: 25 °C, with 40% extractant concentration. Stripping of the loaded, with titanium, organic phase was carried out by diluted HCl (3 mol/L) at ambient temperature. Finally, the recovery of titanium nano-powder, in the form of anatase, was performed by chemical precipitation at pH: 6 and T: 95 °C, using 10 wt% MgO pulp as neutralizing agent. The produced precipitates were characterized by X-ray diffraction analysis (XRD), Fourier transform infrared spectroscopy (FT-IR) and thermogravimetric/differential thermal analysis (TGA/DTA). Their morphological characteristics and microstructure were studied by scanning electron microscopy (SEM). High grade titanium white precipitate, in the form of anatase, was obtained. Iron concentration in the precipitate did not exceed 0.3%, whereas no aluminium was detected.

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1. Introduction

At all the world's 85 alumina plants, 1.0–1.6 tons of red mud is generated per ton of alumina and it is estimated that over 66 million tons of this waste are produced annually in the world. The disposal of such a large quantity of this alkaline waste sludge is expensive (up to 1–2% of the alumina price), as it requires a large disposal area (approximately 1 km^2 per five years for a 1 Mtpy alumina plant) and causes a number of environmental problems [1]. In Greece, the annual production of bauxitic red mud, from the alumina processing plant, is approximately 500,000 tons/year. The red mud slurry has a solids concentration of 500 g/L and bulk density of 1.3 g/L [2,3].

The Laboratory of Metallurgy of the National Technical University of Athens, Greece, has performed a laboratory-scale research on the recovery of titanium from red mud by a leaching process with diluted sulphuric acid, under atmospheric conditions and without using any preliminary treatment. The titanium recovery efficiency, on the basis of red mud weight, reached 64.5% at the following optimum conditions: acid concentration = 3 mol/L, temperature = 60 °C and solid to liquid ratio = 5%. At these conditions, iron leaching

reached 46%, whereas that of aluminium did not exceed 37% [4]. In the present work, the development of a process for the titanium recovery from red mud sulphate leach liquor and the synthesis of titanium oxide TiO$_2$ nano-powder are described.

Titanium dioxide is a widely known material that is mostly used as pigment in paints, plastics, rubber, and paper industries. Besides, titania has been studied extensively because of its wide applications mainly in catalyst supports, fillers, coatings, photoconductors and dielectric materials. In recent years, nanocrystalline TiO$_2$ is well known as a semiconductor with photocatalytic activities and has a great potential for applications such as environmental purification, decomposition of carbonic acid gas and generation of hydrogen gas [5,6]. The properties that make this product so important for the industry are its high refractive index, high reflectivity-brightness and brilliance, chemical inertness, thermal stability and non toxicity.

Two types of manufactured TiO$_2$ are available in the market: anatase and rutile grade. The industrial applications vary, depending on the end product required. The rutile grade is used for products as paints, printing inks, plastics, cosmetics, etc. Anatase grade finds application in paper industry, latex, rubber, pharmaceutical products and soap industry. Anatase-type fine powders have received considerable attention for their high photocatalytic activity and are applied for environmental purification and antibacterial purposes. About 62% of the total TiO$_2$ produced goes to the paint industry. Titanium dioxide pigments, with typical particle size in the order of 200–400 nm, represent over 80% of the world pigment

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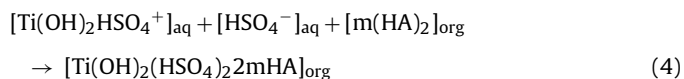*URL:* http://www.hydrometallurgy.metal.ntua.gr (P.E. Tsakiridis).

production and are commercially produced by either the sulphate or the chloride route [7–9].

1.1. Iron and titanium separation from sulphate solution by organophosphorus extractants

The dominant species in sulphuric acid is $TiOSO_4 2H_2O$, which is represented more accurately as $Ti(OH)_3HSO_4$. Titanium species are polymerized according to the reactions [10]:

$$TiOSO_4 \rightarrow (TiOSO_4)_2 \tag{1}$$

$$2TiO(SO_4)_2 \rightarrow [TiO(SO_4)_2]_2 \tag{2}$$

The solvent extraction behaviour of tetravalent titanium from sulphate solutions with organophosphorus acid derivatives such as di-2-ethylhexyl phosphoric acid (D2EHPA) and 2-ethylhexyl phosphonic acid mono-2-ethylhexyl ester (EHEHPA) has been studied by several investigators [11]. The distributions of titanium and aluminium between sulphuric acid solutions and solutions of di-(2-ethylhexyl)-phosphoric acid (D2EHPA) in organic solvents have been investigated under different conditions [12]. Although aluminium was extracted by a cation-exchange reaction, titanium, at low acidity, was extracted by a cation-exchange process and at high acidity by a solvating reaction:

$$[Ti(OH)_2^{2+}]_{aq} + 2[(HA)_2]_{org} \rightarrow [TiOA_4H_2]_{org} + 2H^+_{aq} + H_2O \tag{3}$$

$$[Ti(OH)_2HSO_4^+]_{aq} + [HSO_4^-]_{aq} + [m(HA)_2]_{org}$$
$$\rightarrow [Ti(OH)_2(HSO_4)_2 2mHA]_{org} \tag{4}$$

Islam and Biswas reported equilibrium data for the extraction of Ti(IV) from sulphate solution by D2EHPA and examined the effects of the solution pH and the concentration of the extractant on the distribution ratio [13,14]. Furthermore, they discussed the equilibrium constant in terms of the extraction kinetics. Titanium was found to be extracted as an oligomeric polynuclear complex, with both the HA_2^- and A_2^{2-} ions acting as ligands. Islam et al. also demonstrated that complete extraction of titanium occurs only after 80–100 min of agitated contact, with the rate of extraction being slow at first, then increasing after about 60 min [15]. The extraction of titanium, along with other associated metal ions from leach liquors of titaniferous magnetites, has been studied using D2EHPA as an extractant [16]. Approximately 90% of the titanium in the feed solution was extracted by D2EHPA in a single contact at pH values above 1.5. Selectivity of this extractant for titanium over competing elements was poor. Iron was strongly extracted at pH = 1.5, whereas aluminium extraction was relatively lower.

Jayachandran and Dhadke investigated the liquid–liquid extraction of Ti from sulphate solution using 2-ethylhexyl phosphonic acid mono-2-ethylhexyl ester in toluene (commercially known as PC88A or Ionquest 801 or P507) [17]. The extraction of titanium was found to be quantitative with 0.03 M of PC-88A in toluene in the acidic range of 0.01–0.3 mol/L. Furthermore, the extraction of titanium(IV) from acidic media by 2-ethylhexyl phosphonic acid mono-2-ethylhexyl ester has been also studied [18]. The kinetics of extraction was slow, requiring 60 min for the complete extraction of titanium and was improved with the increase of the extractant concentration in the organic phase. The distribution ratios of titanium presented minimal extraction values at 1–2 mol/L.

However, the so far published literature has given little attention to the titanium extraction from sulphate solutions by dialkylphosphinic acids. The starting point for the "Cyanex" range of extractants is phosphine (PH_3). Monothiophosphinic (Cyanex 302) and the disulphur (Cyanex 301) analogues of bis-(2,4,4 trimethylpentyl) phosphinic acid (Cyanex 272) were introduced in the market in the 1980s. They constitute another important class of extractants,

which have been proved to be better for the separation of Co and Ni than the corresponding phosphonic or phosphoric acid derivatives.

1.2. Synthesis of TiO$_2$ powders

The method most frequently employed for the production of titanium oxide is the precipitation from chloride or sulphate solutions of titanium, by the addition of aqueous solution of ammonia or sodium hydroxide. TiO_2 can be also obtained by other methods such as hydrothermal processing [19] or sol–gel techniques based on the hydrolysis and polycondensation reactions of Ti(IV)-alkoxide [20]. The synthetic routes of TiO_2 production usually result in amorphous solid TiO_2 or anatase or rutile, depending on the preparation route and the experimental conditions. The transformation behaviour from amorphous to anatase or rutile phase is influenced by the synthesis conditions. Phase transformation to the rutile structure takes place when the temperature is raised at least above 450 °C. Baura-Pena et al. studied the synthesis and characterization of hydrated TiO_2, by adding an aqueous solution of sodium hydroxide to a titanium(IV) sulphate solution [21]. The precipitation was accompanied by an exothermic reaction which indicated that the precipitate always formed at temperatures of 90 °C. The X-ray diffraction pattern of this compound showed a poorly crystallized anatase.

Li and Demopoulos examined the precipitation of Ti by aqueous titanium(IV) chloride solutions at different terminal pH (2.5–6.0) with magnesium oxide as base at 95 °C [7]. It was found that forced hydrolysis at 95 °C favours the formation of rutile TiO_2. Neutralization by MgO at 95 °C, on the other hand, favoured the formation of mixed anatase and rutile materials. The anatase fraction (ranging from 0 to 70%) increased with pH elevation (from 2.5 to 6.0). Zhang et al. prepared and characterized nanosized TiO_2 powders from aqueous $TiCl_4$ solutions with 2.5 M dilute NH_4OH at pH = 7 [6]. It was reported that in the presence of a small amount of sulphate ions, the powder was pure anatase and its primary particle size was finer than that of alkoxide-derived powder, moreover the transformation of anatase to rutile was retarded. Both hydrolysis temperature and amount of sulphate ions affected the morphology and crystallization of the powder.

The present research work proposes a process for the titanium recovery from red mud sulphate leach liquor and the preparation of titanium oxide TiO_2 nano-powder. More specifically, titanium can be selectively separated from the leach liquor by solvent extraction using the organophosphinic extractant Cyanex 272. Titanium is recovered from the organic phase by stripping with dilute HCl. It is then precipitated at pH = 6.0 with MgO pulp (10 wt%) at 95 °C, which favours the formation of anatase.

2. Experimental

The leach liquor, which was used during experimentation, had been produced by red mud agitation leaching with dilute sulphuric acid (3 mol/L) at T = 60 °C, with solid to liquid ratio = 5%. It was first pre-neutralised by 10 mol/L NaOH to pH = 0.1 at ambient temperature. After neutralization, the average composition of the leach liquor is given in Table 1. Elements concentration was determined by atomic absorption spectrophotometer (Perkin Elmer 4100). The analysis of Fe (Fe^{2+} and Fe^{3+}) was performed with back-titration using potassium dichromate ($K_2Cr_2O_7$).

2.1. Titanium solvent extraction from red mud sulphate leach liquor

The extractant Cyanex 272, registered by Cyanamid Canada, was kindly supplied by the Holland branch of the company, Cytec

Table 1
Red mud sulphate leach liquor composition after neutralization.

Element	Concentration (g/L)
Ti^{4+}	1.75
Fe^{3+}	10.85
Fe^{2+}	0.45
Al^{3+}	2.17
Cr^{3+}	0.12
Ca^{2+}	0.17
Mg^{2+}	0.05
Mn^{2+}	0.04

Holland. The reactive component $C_{16}H_{34}PO_2H$ is the bis(2,4,4-trimethylpentyl) phosphinic acid. The molecular weight is 290 and the density at $24\,°C$ is $0.95\,g/cm^3$. The extractant was used without further purification. In all cases, toluene was used as organic diluent.

All batch experiments were conducted in a 500-ml five-necked, round bottomed split reactor, which was fitted with a glass stirrer, a vapour condenser, a thermometer and a pH electrode. The two phases (organic and aqueous) were contacted for 60 min by rapid stirring and the pH was controlled by small additions of 5 mol/L NaOH. Heating was provided by an electrical mantle and the temperature of the liquid was controlled by a Pt-100 digital controller.

2.2. Titanium stripping from the loaded organic phase

Titanium recovery from a loaded organic phase was investigated using different strength of stripping agents, such as solutions of sulphuric acid (with or without hydrogen peroxide) and hydrochloric acid. Furthermore, the alternative of titanium recovery by precipitation of TiO_2 from the loaded organic phase, using Na_2CO_3, was also examined. The procedure was the same as that in the extraction runs. The stripping was conducted for 30 min. All experiments were duplicated and the determination of the metal concentration in the loaded phase was achieved by mass balance and checked by complete stripping of the loaded organic phase followed by analysis of the stripped solution.

2.3. Synthesis and characterization of TiO_2 powders

The precipitation tests were performed in a 500 ml, five-necked, round bottomed split reactor, that was fitted with a glass stirrer, vapour condenser, thermometer and a pH electrode. The pH of the solution was slowly raised to a pre-determined equilibrium value (pH = 6), by adding MgO pulp (10 wt%). Hydrolysis was conducted by heating the solution to $95 ± 0.5\,°C$ for 3 h. The temperature of the liquid was controlled by a Pt-100 digital controller and the pH was measured using a Hamilton Chemotrode Bridge 200 combined pH electrode, specially designed for semi-solid and boiling water solutions. The precipitate, after separating from the solution by filtration under vacuum, was washed with water, dried overnight at $110\,°C$ and weighed.

The produced TiO_2 nano-powders were mineralogically analyzed by X-ray diffraction (XRD), using a Bruker D8-Focus diffractometer with nickel-filtered Cu Kα1 radiation (=1.5405 Å), 40 kV and 40 mA. Their morphology was also examined by scanning electron microscopy (SEM) using a Jeol 6380LV Scanning Electron Microscope. Experimental conditions involved 15 kV and 20 kV accelerating voltage. Chemical composition of the samples was carried out by an Oxford INCA Energy Dispersive Spectrometer (EDS) connected to the SEM.

TGA/DTA analysis was conducted at hydrous TiO_2 with a Setaram-Labsys thermal analyzer. Type S-thermocouple is used for temperature measurements in this instrument. The sample was placed in a ceramic crucible and heated from room temperature
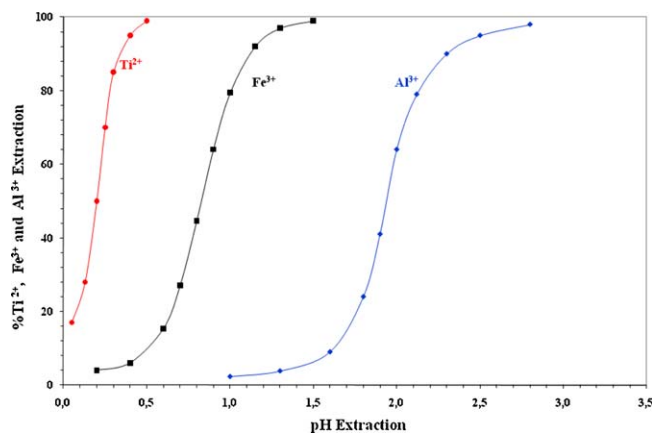

Fig. 1. Ti selectivity over Fe and Al in sulphate leach liquor of red mud with 40% Cyanex 272 at $25\,°C$ (contact time: 60 min, A/O ratio: 1/1).

to $1200\,°C$ at a heating rate of $5\,°C/min$ using air as a medium under static condition. Finally, TiO_2 powders were characterized by a Perkin Elmer Spectrum GX Fourier Transform IR spectrophotometer, in the range of $4000–400\,cm^{-1}$ with 200 successive scans. The spectrometer was equipped with a deuterated triglycine sulphate (DTGS) detector and with an attenuated total reflectance (ATR) unit. The spectra rationed against a potassium bromide (KBr) background. The ATR sampling compartment is a ZnSe crystal (refractive index 2.4) with an angle of incidence of $45°$ oriented horizontally. The size of the rectangular surface area of the ATR crystal is $60\,mm × 10\,mm$.

3. Results and discussions

3.1. Titanium solvent extraction over iron by Cyanex 272

In order to determine the ability of Cyanex 272 to selectively extract titanium from the red mud sulphate leach liquor, extraction tests (based on preliminary experiments) were carried out at various equilibrium pH values, at specific conditions (contact time: 60 min, A/O ratio: 1/1, T: $25\,°C$). The results, presented in Fig. 1, show that Cyanex 272 can efficiently extract titanium in the pH range of 0.3–0.5. In this pH range iron extraction reached 3%, whereas that of aluminium did not exceed 0.2%.

As Cyanex 272 proved to be an efficient extractant for the titanium extraction from sulphate leach liquor, a Mc-Cabe Thiele diagram was constructed and the number of counter-current stages required was predicted. On the basis of conclusions drawn from preliminary experiments, the titanium extraction isotherm was determined at the following conditions: Cyanex 272 concentration = 40% (v/v), diluent = toluene, extraction pH = 0.30 and T = $25\,°C$. Mc-Cabe Thiele diagrams indicated three stages for titanium extraction at a phase ratio A/O = 1.5 (Fig. 2).

3.2. Titanium stripping from the loaded organic phase

Preliminary stripping tests of titanium from the loaded organic phases was conducted using various stripping agents, as it is shown in Table 2, at specific conditions (contact time: 30 min, O/A ratio: 1/1, T: $25\,°C$). Data indicated that, in one stage, 3% H_2O_2 in 1 mol/L H_2SO_4 and 3 mol/L HCl can be used for the recovery of Ti(IV). However, a 3 mol/L HCl solution was preferred, because in the case of hydrogen peroxide/sulphate solution, Ti formed a very stable orange-red titanium hydrogen peroxide complex, which was more difficult to be hydrolyzed and precipitated in the next stage. This coloured complex $[Ti(OH)_4(H_2O_2)SO_4]$ has been used for analysis of titanium and hydrogen peroxide [17].
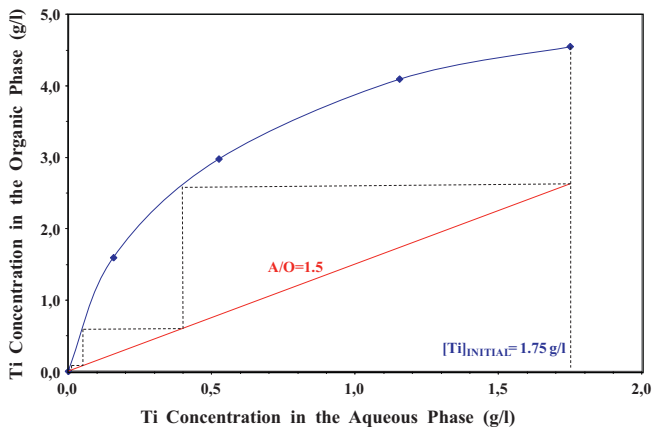
Fig. 2. Mc-Thiele diagrams for titanium extraction by Cyanex 272 at 25 °C.

Table 2
%Titanium recovery from the loaded organic phase using various stripping agents.

Stripping reagent	Titanium (%)	Iron (%)
2 M H_2SO_4	35.34 ± 0.35	3.52 ± 0.22
4 M H_2SO_4	71.52 ± 0.37	12.35 ± 0.25
3% H_2O_2 in 0.5 M H_2SO_4	90.57 ± 0.26	1.46 ± 0.03
3% H_2O_2 in 1 M H_2SO_4	94.28 ± 0.28	3.58 ± 0.05
1.5 M HCl	48.35 ± 0.28	1.85 ± 0.11
3 M HCl	93.76 ± 0.35	3.72 ± 0.18
0.5 M Na_2CO_3	64.25 ± 0.45	2.57 ± 0.02
1 M Na_2CO_3	75.70 ± 0.42	1.52 ± 0.01

Besides stripping with HCl, the stripping with Na_2CO_3 was also studied. Titanium was precipitated as pure metatitanic acid in the aqueous phase during stripping with 0.5 mol/L and 1 mol/L of Na_2CO_3 solution. However, the overall recovery yield was low and reached 76%.

On the basis of conclusions drawn from preliminary experiments, the conditions for the stripping of titanium from the loaded Cyanex 272 were fixed at:

Aqueous phase: 3 mol/L HCl

- Temperature: 25 °C;
- Stirring speed: 800 rpm;
- Cyanex 272 organic phase: $[Ti^{4+}]_{org}$ = 2.58 g/L, $[Fe^{3+}]_{org}$ = 0.65 g/L;
- Reaction time: 30 min.

The Mc-Cabe Thiele interpretation for titanium stripping from the loaded organic phase indicated two stages requirement (Fig. 3), at phase ratio O/A = 1.5.
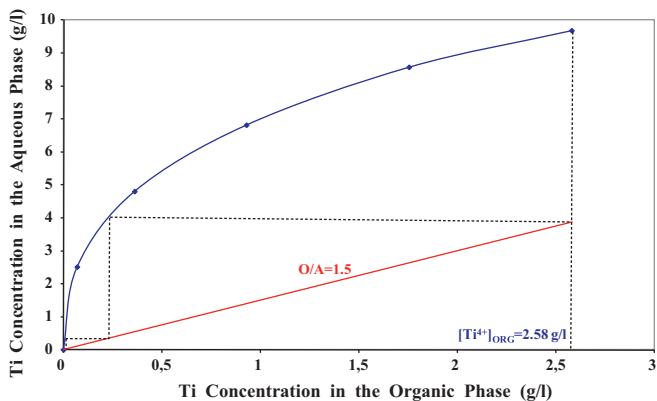

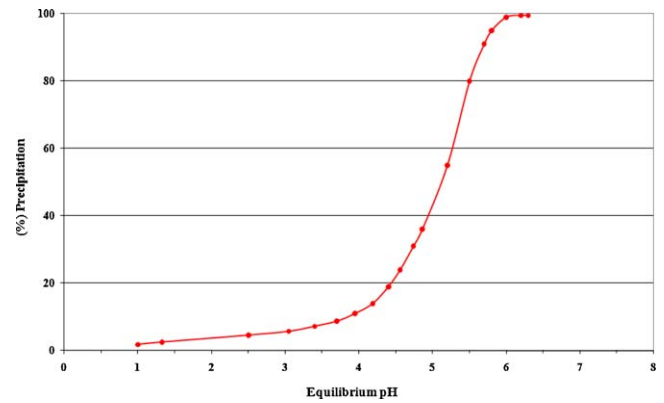**Fig. 3.** Mc-Cabe Thiele diagram for Ti stripping from Cyanex 272 by 3 mol/L HCl at 25 °C.

Fig. 4. Ti precipitation curve from sulphate leach liquor of red mud using 10 wt% MgO pulp at 95 °C.

3.3. Synthesis and characterization of TiO_2 powders

Runs were conducted to draw the precipitation curve of titanium as a function of pH, by the addition of MgO pulp (10 wt%) as neutralizing agent, at 95 °C. Samples that were taken at specific pH values were filtered and the solutions were analyzed. The results, presented in Fig. 4, show that in the pH range of 6.0, titanium is precipitated completely.

The hydrolytic precipitation reactions of metallic ions attain equilibrium very slowly. This is also valid for titanium(IV) and at least 2 h was necessary to obtain reliable equilibrium data. The hydrolysis and condensation reactions started immediately upon mixing, as indicated by the rapid increase in turbidity and the formation of visible flocs, which precipitated at the bottom of the reaction vessel. The addition of MgO pulp produced a gradual increase in pH up to pH = 4.0, whereas in the range of pH = 4.0–6.0 a sudden rise was observed. At pH = 6.0 the precipitation was complete and a white gelatinous precipitate was obtained. Iron concentration in the produced white precipitate did not exceed 0.3%, whereas no aluminium was detected.

The X-ray diffraction data of the precipitates obtained under the experimental conditions mentioned above, after drying at 110 °C is shown in Fig. 5. The characteristic diffraction peaks at 25.3°, 37.8°, 48.0°, 53.9°, 55.1°, 62.7° and 67.0° indicated that the diffraction pattern corresponds to poorly crystallized anatase. It has been pointed out that higher pH favours the anatase phase because of the lower stability of metastable anatase in strong acidic solution [7]. Lower pH favours the formation of rutile phase. Furthermore, the presence of anatase was also attributed to the fact that the precipitate was obtained at 95 °C [21].
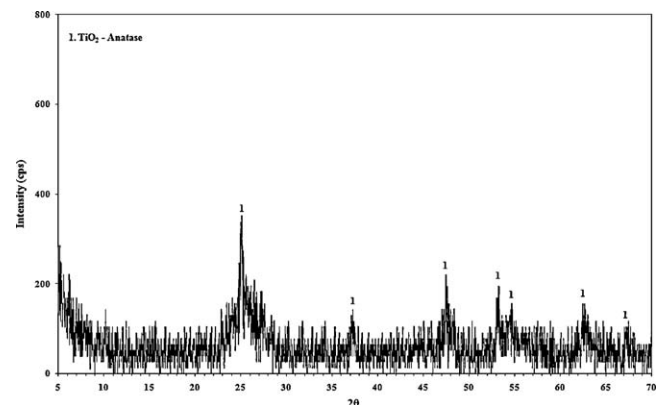

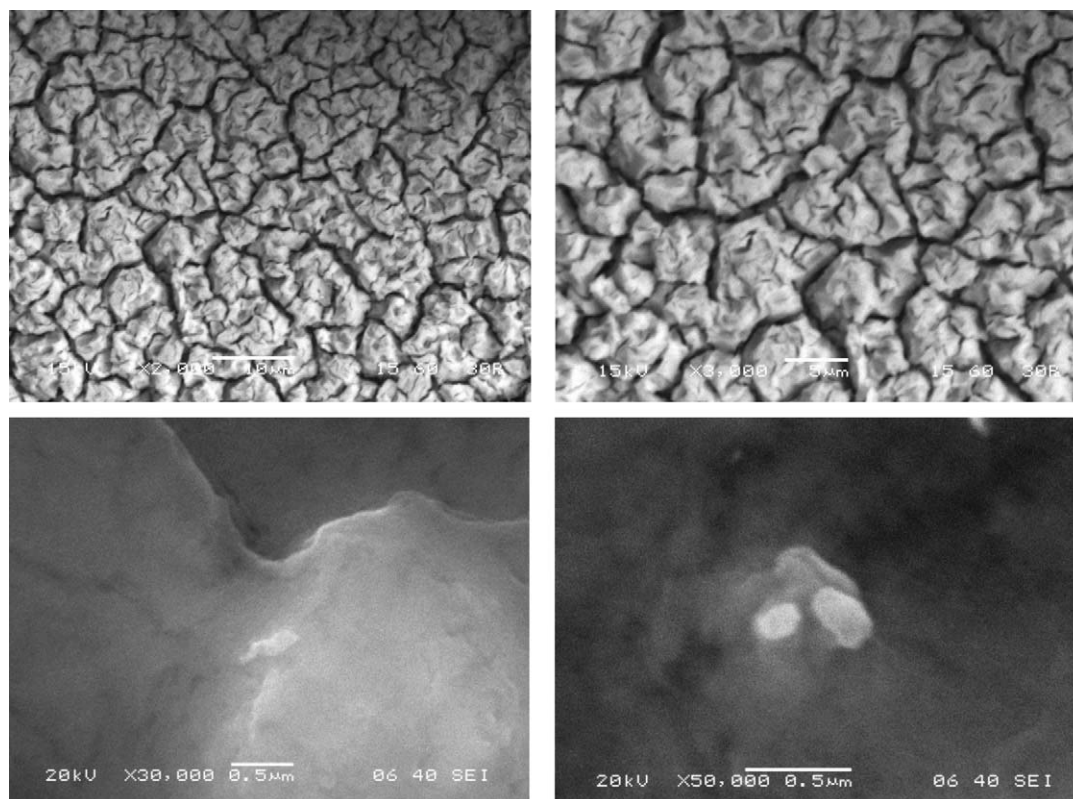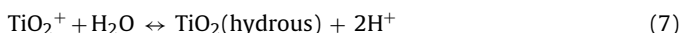**Fig. 5.** X-ray diffraction analysis of the produced titanium precipitate at pH = 6 and T = 95 °C.

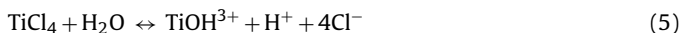**Fig. 6.** SEM micrographs of the produced titanium precipitate at pH = 6 and T = 95 °C.

Scanning electron microscopy with X-ray energy dispersive analysis (EDS) was performed to gain further knowledge of the mineralogical species contained in the mixed precipitate and their morphology. The anatase appeared with the form of aggregates of spherical nano-particles (Fig. 6). It is known, that by hydrolysis of titanium in chloride solution, stable aqueous colloid solutions of 4–6 nm TiO_2 particles can be formed. The hydrolysis may include three steps as shown below [6]:

$$TiCl_4 + H_2O \leftrightarrow TiOH^{3+} + H^+ + 4Cl^- \tag{5}$$

$$TiOH^{3+} \leftrightarrow TiO_2^+ + H^+ \tag{6}$$

$$TiO_2^+ + H_2O \leftrightarrow TiO_2(hydrous) + 2H^+ \tag{7}$$

In acidic medium, the chloride solutions are stable because of the negative surface charge, which stabilizes particles towards agglomeration. By increasing the pH, this surface charge gradually disappears and the particles can form aggregates [22]. The produced aggregates presented an irregular shape and size of about 10–20 μm. Higher magnifications revealed that the primary aggregates were formed by the agglomeration of smaller aggregate subunits with irregular shape and size of about 200 nm.

The FT-IR spectra of the produced TiO_2-anatase precipitate obtained by neutralization with 10% MgO pulp is given in Fig. 7. The spectra exhibited three main peaks. The wide asymmetric band appearing at about 3400 cm^{-1}, was mainly attributed to the presence of –OH groups of water and secondarily to the potential residual hydroxyl group –OH, in the crystal structure of titanium dioxide. The above observation was also confirmed by the presence of the strong band around 1630 cm^{-1}, which is due to hydroxyl bending mode in the adsorbed water, whereas the presence of the minor peak at 3230 cm^{-1}, is related with the small presence of –OH stretching vibrations. The signals in the range of 450–600 cm^{-1} correspond to the bending vibrations of titanium-oxygen (Ti–O–Ti) bonds in titanium dioxide lattice [23,24].

The results of the differential thermal analysis (DTA) and thermal gravimetry analysis (TGA) of the powder precipitated by neutralization at final pH = 6 are given in Fig. 8. The TG diagram shows three main steps for the weight loss. The first one occurred in the range of 30–310 °C (weight loss was about 39% of the total weight), corresponding to the evaporation of physically adsorbed water (30–130 °C) and to the loss of chemically adsorbed water (140–310 °C). The second one, in the range of 310–500 °C (weight loss: 6%), was attributed mainly to the oxy-hydroxides decompositions. Finally, in the range of 500–710 °C (weight loss: 6%) the precipitate product transformation to anatase and rutile takes place. Above 710 °C, the stabilization of sample mass indicates completion of the formation of TiO_2. The endothermic effect in the DTA curve at 135 °C is associated with the loss of the loosely bonded adsorbed water on the particle surface. The endothermic peak at 205 °C should be attributed to the
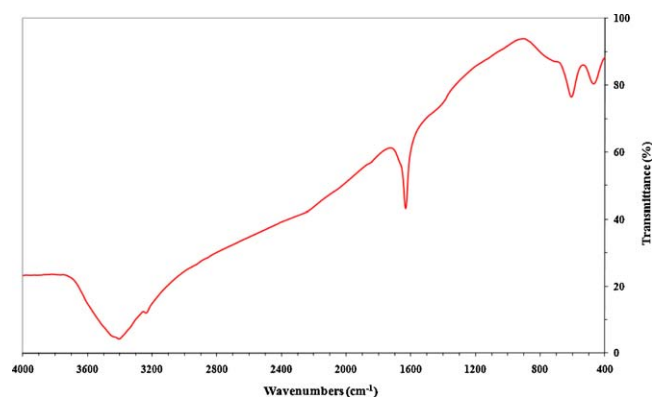

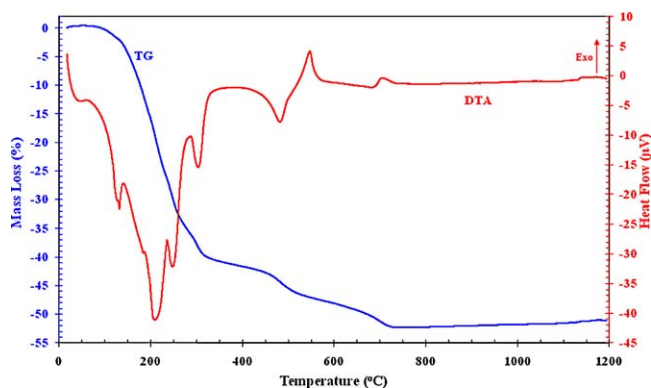**Fig. 7.** FT-IR spectra of titanium precipitate at pH = 6 and T = 95 °C.

Fig. 8. TGA/DTA analysis of titanium precipitate at pH = 6 and T = 95 °C.

transformation of the poorly crystalline phases and the loss of structural water occluded in interplanar regions and lattice interstices. The endothermic peak at 310 °C is associated with the dehydration and the loss of chemically adsorbed water. The exothermic shoulder around 400 °C corresponds to the transformation initiation of amorphous/nano-structured precipitate to crystalline TiO_2, whereas the endothermic peak at 470 °C should be attributed to the oxy-hydroxides dehydroxilation. At 540 °C the transformation of the produced precipitate into anatase phase is completed and proceeds contemporaneously with an increment in the particle size and the subsequent heat release. At around 700 °C the rutile phase start to appear [7,25,26]. Thereafter no significant thermal effects can be detected even up to 1200 °C.

4. Conclusions

Based on the data presented in this paper, a purification process of red mud sulphate leach liquor for the recovery of TiO_2 nano-powders in the form of anatase is suggested. The conclusions from the testwork of the proposed stage are the following:

(1) Titanium can be extracted over iron and aluminium by bis(2,4,4-trimethylpentyl) phosphinic acid. Cyanex 272, diluted in toluene, could extract titanium Ti at pH = 0.3, T = 25 °C and extractant concentration = 40%. According to the Mc-Cabe Thiele diagrams three stages for Ti extraction are required at a phase ratio A/O = 1.5. Iron co-extraction reached 3%, whereas that of aluminium did not exceed 0.2%.
(2) The loaded with titanium Cyanex 272can be stripped with diluted hydrochloric acid (3 mol/L), at ambient temperature. The Mc-Cabe Thiele interpretation for Ti stripping from the loaded organic phase indicated two stages requirement, at phase ratio O/A = 1.5.
(3) High grade titanium oxide white precipitate, was recovered from the strip liquor by hydrolytic precipitation at pH = 6 and T = 95 °C, using MgO pulp as neutralizing agent. Iron concentration in the precipitate did not exceed 0.3%, whereas no aluminium was detected. The nature of the poorly crystallized anatase, precipitated in the form of aggregates of spherical nano-particles, was confirmed by X-ray diffraction analysis.

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