

## Treatment of Apatite Nepheline Ore Wasteenrichment Waste

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### Abstract

The study demonstrated the efficiency of using the cascade reaction method applied to polymineral systems treatment, including technogenic waste of apatite nepheline ore. The conditions for minerals decomposition by sulphuric acid, content, and properties of forming solid phases have been researched. The sequence for carrying out production operations has been defined; the parameters and their operation modes were optimized. It is shown that by gradually increasing the sulphuric acid concentration and temperature the process can be carried out stepwise so that the products of one reaction participate in another reaction. This method provides complete use of initial mineral wastes and conversion of components in the form of wide range of useful products.

**Keywords:** mineral waste, nepheline, apatite, titanite, cascade reaction method

### 1. Introduction

The necessity for the rational use of mineral resources is caused by their scarcity and non-reproducibility. Under the conditions of highly intensive field exploitation, minor part of minable raw material is used. In recent years Mining, Minerals and Sustainable Development project has gained world support (MMSD Project, 2002). In respect to the use of mineral resources, this conception provides disposal of mining waste since under exploitation of natural resources is source of a considerable waste amount having adversely effect on the environment (Tirutu-Barna et al., 2007; McLellan et al., 2009). Waste deposits not only deprive useful area from economic circulation, but also represent the pollution sources of air, soil, underground, and surface water (Branwart, and Malmstrom, 2001; Stewart et al., 2003; Broadhurst & Petrie, 2010; Young et al., 2000; Norgate et al., 2007). In this regard, the researches on waste disposal followed by obtaining the new types of inorganic materials are of current interest. Generally, mineral wastes are processed to recover the most valuable component thus not contributing to the complete waste disposal and slightly decreasing the environmental load.

The offered waste treatment processing method is based on the cascade reactions principle, consisting in the fact that a share of products resulting from serial-parallel reactions is a raw material for the subsequent technologic stages and resource for getting new products. It is significant that as far as all or the majority of mineral components being the result of chemical transformations form final products, non-waste and low-waste technologies can be considered. The new method can be applied for mining waste utilization. In order to justify it, it is necessary to conduct a particular complex of physical and chemical researches that includes the following stages:

- Determination of amount for each mineral component composing the test material;
- Definition of function for each mineral component;
- Selection of chemical reactions leading to final product formation;
- Study of kinetics and mechanism of final products formation;
- Selection of optimum components ratio for targeted synthesis of products with the tailored properties.

The possibility of cascade method application is shown in this work by the example of apatite nepheline ore wastes treatment (Kola peninsula, Russia). This waste is formed during ore flotation concentration with apatite and nepheline concentrate production. They are scarcely treated and, in the form of aqueous suspension, are sent to waste facilities. The new flow scheme of wastes disposal will allow producing a whole series of cheap products used in paint and construction materials industry, and sewage and processing waters purification of

suspensions and pernicious contraries.

## 2. Material and Method

Nepheline wastes (fraction up to 100  $\mu\text{m}$ ) with composition (weight %): nepheline –30-35, aegirine –30-35, titanite –20-25, apatite –5-7 were used in this study. Apatite and nepheline are concentrated in the fine fraction (up to 30  $\mu\text{m}$ ). The coarser fraction consists of titanite and aegirine. 10-40%  $\text{H}_2\text{SO}_4$  was used in decomposition processes.

The results of preliminary studies of individual concentrates were used to prepare a synthetic concentrate mixture similar to industrial wastes. The experiment was carried out in a glass reactor connected with a stirrer and a thermometer, under reflux. In the reactor, a calculated amount of sulphuric acid was initially placed and after that, the corresponding amount of concentrate was added. The obtained suspension was heated (if necessary) under stirring during 2-8h. The solid product was separated and both solid and liquid phases were analysed. The pH of solutions was measured with Orion pH meter 611. The chemical composition of mineral waste was determined using AAS-300 atomic absorption spectrometer. Both the Initial and final materials were identified with Siemens D 5000 Powder X-ray diffractometer, with Cu  $K\alpha$  radiation. The final material was examined by SEM SEM-EDS a JEOL 3400.

## 3. Results and Discussion

The level of mineral waste decomposition was determined from different solubility of the minerals in sulphuric acid. Thereby, gradual leaching of components from minerals into liquid phase is taking place with their subsequent conversion into compounds, in which they individually or together with other compounds form final products. The alternation of chemical (solid phase interreacting with sulphuric acid) and physical (resulting suspension separation) reactions allows effecting process with preparation of intermediate and final product according to low waste technology.

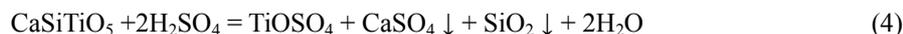
Principal chemical reactions involved in the method are as follows:



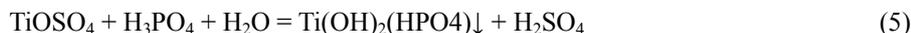
nepheline



apatite



titanite



Complexity and instability of system composition under study are the reason for particular challenges when developing technology. That is why an amount of each mineral component that composes waste under study was initially determined. Further interreacting kinetics of each mineral with selected sulphuric acid solution as well as mechanism of residues formation was studied and their optimum ratio for streamlined synthesis to produce tailored final products was defined.

### 3.1 Stage I - Nepheline Dissolution

First, the solubility studies involved nepheline as the most reactive component of the waste (Taylor and Eggleton, 2001; Tole et al., 1986; Jena et al., 2014; Hamilton et al., 2001). Nepheline was added to a sulphuric acid solution with a concentration of 50-150 g/l and consumption of 40 to 100% of the theoretically justified quantity in order to fix the acid-soluble components in sulphates.

Nepheline adding in solution of sulphuric acid was carried out for 15-20 minutes with the following stirring of reaction suspension for 1 hour without heating. When using sulphuric acid with a concentration of less than 100 g/l  $\text{H}_2\text{SO}_4$  leaching of main components proves to be incomplete and their content in liquid phase does not exceed 20 g/l in aluminium and silicium when converted into oxides(in terms of oxides). Acid strengthening to 150 g/l is accompanied by nearly complete leaching of mineral components. The crystallization process of aluminium sulphates and potassium alum takes place simultaneously. Under these conditions, silicium is prone to hard filterable colloid systems formation. At the consumption of sulphuric acid less than 85% the degree of

nepheline decomposition dips down and suspension tendency to polymerization with producing of colloids increases. Increase in acid consumption more than 85% of stoichiometry contributes to increase in transition degree of main components into solution to 90%, but resultant acid solutions are unusable (Figure 1), for example, as coagulum flocculants, since low pH of such solutions may lead to acidification of purifying water. According to findings, optimum conditions for nepheline decomposition are as follows:  $H_2SO_4$  concentration - 100 g/l at the rate of 85% of stoichiometric rate (Table 1).

Table 1. Nepheline dissolution by sulphuric acid

$H_2SO_4$ , initial concentration, $g \cdot l^{-1}$	$H_2SO_4$ , % of stoichiometric value	components in liquid phase, $g \cdot l^{-1}$		release to solution, %	
		$Al_2O_3$	$SiO_2$	$Al_2O_3$	$SiO_2$
50	50	11.83	21.25	50.24	51.13
	85	10.92	18.82	79.17	76.85
	100	10.12	17.24	84.41	82.56
100	50	29.54	50.35	62.32	60.52
	85	24.96	44.27	89.71	90.43
	100	21.85	39.45	92.24	94.75
150	50	52.16	88.26	73.43	70.77
	85	33.41	56.34	79.82	76.62
	100			gelatinization	

As the nepheline components pass to solution, the suspension pH increases to 1.8-2.1. After stirring for 30-60 min, the suspension pH grows to 2.2-2.4. The undecomposed nepheline particles are separated by filtration. The solution represents a stable colloid system containing, g/l:  $Al_2O_3$  -20-30,  $SiO_2$  -40-50,  $Na_2O$  -10-15,  $K_2O$  -5-10 (Berkowitz et al., 2005; Otterstedt et al., 1987; Matijevic et al., 1971). The level of nepheline decomposition was determined from the leaching level of the most acid-resistant aluminium and silica oxides. Under optimal conditions, the decomposition achieves 90-95%. The obtained colloid solutions can be used for thickening of fine suspensions and for treatment of industrial wastewater (Gao et al., 2002; Velyaev et al., 2011).

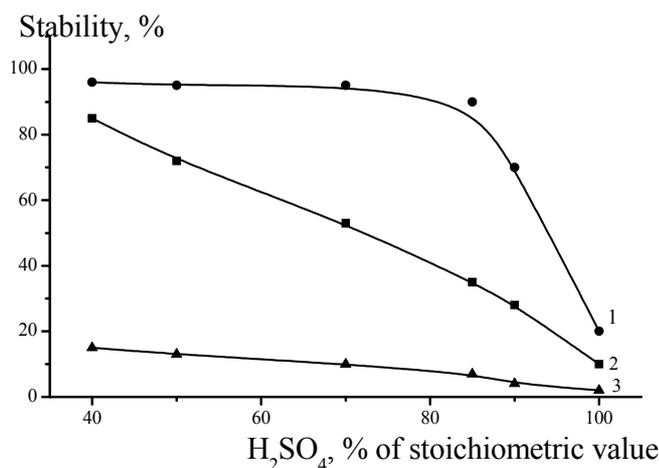


Figure 1. Stability to dilution of solutions obtained through nepheline dissolution in sulphuric acid with a concentration of (g/l) 1-50, 2-100, 3-150

### 3.2 Stage II - Apatite Dissolution

The second stage is interaction of apatite with sulphuric acid solution. The process of sulphuric acid decomposition of apatite results in phosphoric acid with a concentration of 28-50% in  $P_2O_5$  and in calcium sulphate in the form of gypsum (Evenchik & Brodskii, 1987; Pozin, 1974).

Sulphuric acid concentration impact and rate have been studied, as well as process time impact on apatite decomposition degree and structural features of calcium phase. For this purpose when stirring concentrated

sulphuric acid in amount of 110% of its stoichiometric amount necessary for calcium banding in the form of gypsum was slowly added (~30 min) into aqueous suspension of apatite. Resulting concentration of  $H_2SO_4$  in liquid phase is 10-40%. Due to heat release, the suspension temperature increases to 75-80 °C. The process lasts 2-6h. The process duration and temperature affect the recovery of fluorine, evolving in the form of hydrofluosilicic acid with gas-vapour phase ( $SiO_2+6HF \rightarrow H_2SiF_6+2H_2O$ ) and decreasing medium aggressivity, depends on hold-up time and temperature. Resulting gypsum is separated by filtrating and washed with water at S:L=1:3. Apatite decomposition data is shown in Table 2.

Table 2. Regimes of apatite dissolution affecting the composition of liquid and solid phases

Dissolving conditions		Chemical composition of liquid phase, $g \cdot l^{-1}$				Chemical composition of solid phase, %			
$H_2SO_4$ , %	$\tau$ , h	$P_2O_5$	$SO_3$	$F^-$	$SO_2$	$P_2O_5$	$SO_3$	$SiO_2$	CaO
10	2	The filtrates are unstable				-	-	-	-
40	2					1.92	40.82	1.59	35.1
40	4	203.43	3.16	0.96	0.53	1.86	41.57	0.63	36.5
10	4	162.91	3.97	0.82	0.48	1.94	42.73	0.45	39.5
10	6	227.44	2.48	0.41	0.32	1.55	35.24	0.27	36.6

Sulphuric suspension holding for 2 hours at a temperature of 80°C regardless of  $H_2SO_4$  concentration is insufficient for complete solid phase precipitation. With increase of reacting mass holding time to 4 h the system is close to equilibrium and calcium precipitates almost completely. The higher sulphuric acid concentration, the more phosphorus content is in the liquid phase. In the case of process time of 6 h, ultimate recovery of phosphorus from apatite is reached (Figure 2).

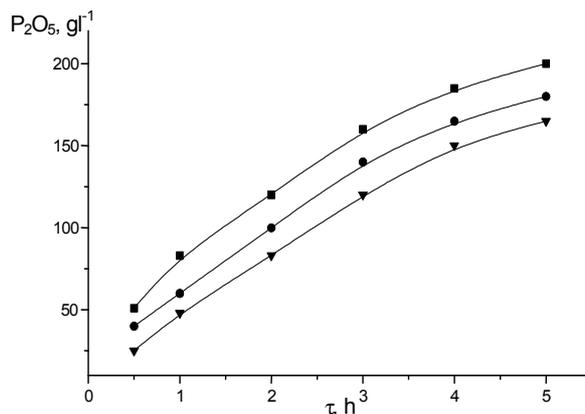


Figure 2. The effect of sulphuric acid concentration on  $P_2O_5$  extraction from apatite.

$H_2SO_4$  % (▲-10, ●-20, ■-40).

XRD analysis shows that main phase in all samples is gypsum -  $CaSO_4 \cdot 2H_2O$ , according to crystal optical analysis its particles is of needle prismatic form from 4-28  $\mu m$  to 5-64  $\mu m$ , are prone to aggregation (Figure 6c). Impurity phase is anhydrite, the amount of which increases with increase in sulphuric acid concentration.

The studies has proved that sulphuric acid concentration increase for more than 20% in  $H_2SO_4$  is unfeasible since during real wastes processing titanite will be composed together with apatite (Figure 3). For that reason the optimum conditions for apatite dissolution are as follows: 15-20%  $H_2SO_4$ , temperature 80o C; reaction time 6 h. Gypsum precipitation is well aggregated, low-level in relation to the mother solution.

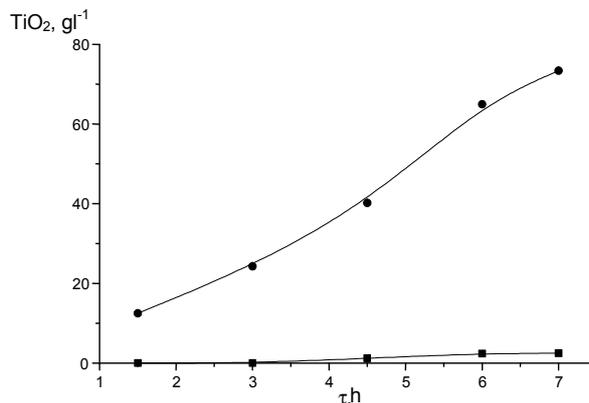


Figure 3. Titanium (IV) leaching from titanite during its dissolution ( $T_{\text{boil}}$ ) by  $\text{H}_2\text{SO}_4$ , % (■-15, ●-40)

### 3.3 Stage III - Titanite Dissolution

Choosing conditions for titanite dissolution is based on the necessity of titanium (IV) transition into liquid phase. Previously it had been established that this process occurs under rigid conditions: sulphuric acid high concentration – 1000-1700 g/l  $\text{H}_2\text{SO}_4$ , elevated temperature – 140-200°C (Motov and Maximova, 1983). Then the concentration of sulphuric acid is about 350 g/l,  $\text{H}_2\text{SO}_4$  titanium (IV) in liquid is unstable and precipitates in the form of titanium hydroxide. Herewith, mineral decomposition degree does not exceed 50% (Motov & Maksimova, 1983).

For the research, 400-600 g/l  $\text{H}_2\text{SO}_4$  was chosen. This concentration provides high stability of titanium (IV) in liquid phase and leads to preparation of all-purpose titanium-containing precursor (as solution), which acts as a basis for subsequent syntheses of various titanium-containing products (Lazareva et al., 2006).

The test object was fine-grained titanite ( $\text{TiO}_2$  – 35-37%). The dissolution process was carried out at 105-115°C during 7-8 h, at a ratio of titanite and acid of  $T:V_L = 1:3$ . At that point leaching of titanite components takes place into liquid sulphuric acid phase with the later formation of reaction solid products – calcium sulphate and amorphous silica. Owing to high solubility, titanium (IV) is concentrated in liquid phase.

Figure 4 shows the relationship between titanium extraction from titanite and the sulphuric acid concentration. Initially, the process occurs at the surface of fine-grained mineral particles with components leaching in liquid phase gradually forming solid products.

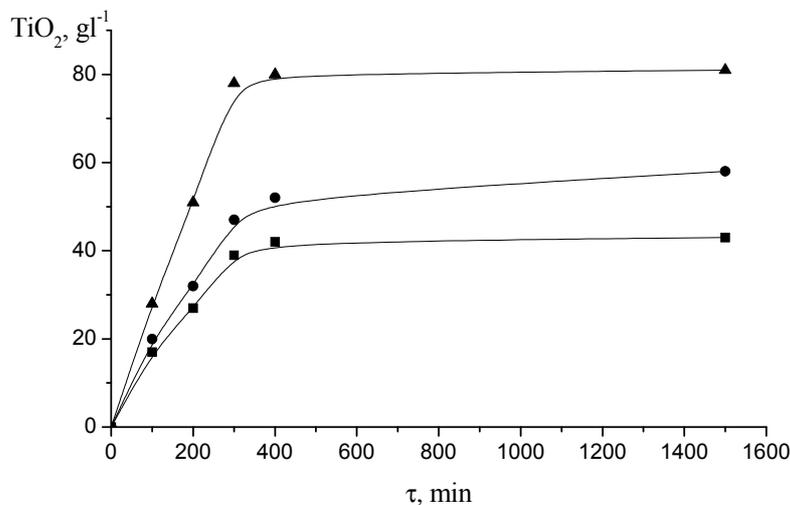


Figure 4. The effect of sulphuric acid concentration on the rate of titanium leaching to the liquid phase.  $\text{H}_2\text{SO}_4$ , g/l (▲-400, ●-500, ■-600)

Temperature rise and concentration of  $H_2SO_4$  have a positive effect on reaction speed. This stage time is about 60 minutes. According to XRD analysis, there is mainly titanite in solid phase and anhydrite in fewer amounts. With increase in reaction products in reaction mass, the process slows down. According to SEM data, products are formed in the form of individual phases, and forms porous shell at the surface of undecomposed sphene particles as well (Figure 5).

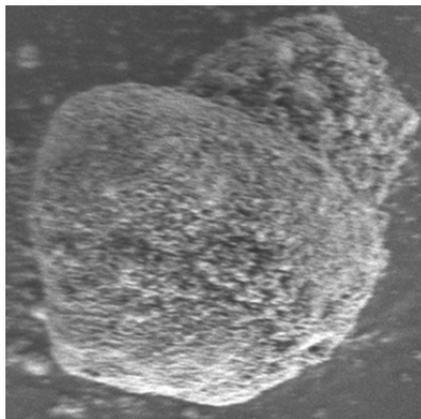


Figure 5. SEM image of titanite particle after 60 min of mineral dissolution

The reaction rate becomes slower with increasing amount of the reaction products due to diffusion of the acid through the layer of newly-formed phases.

Approximately, in 5 h decomposition rate is so low that one can consider pseudoequilibrium state of the system that can be tilted by external influence only. The optimal concentration of sulphuric acid, permitting to leach the maximum of titanium, should be considered to be 600 g/l  $H_2SO_4$ .

#### 4. The Flow Sheet and Target Products

Flow chart of mineral wastes treatment with final products obtaining is shown in Figure 6.

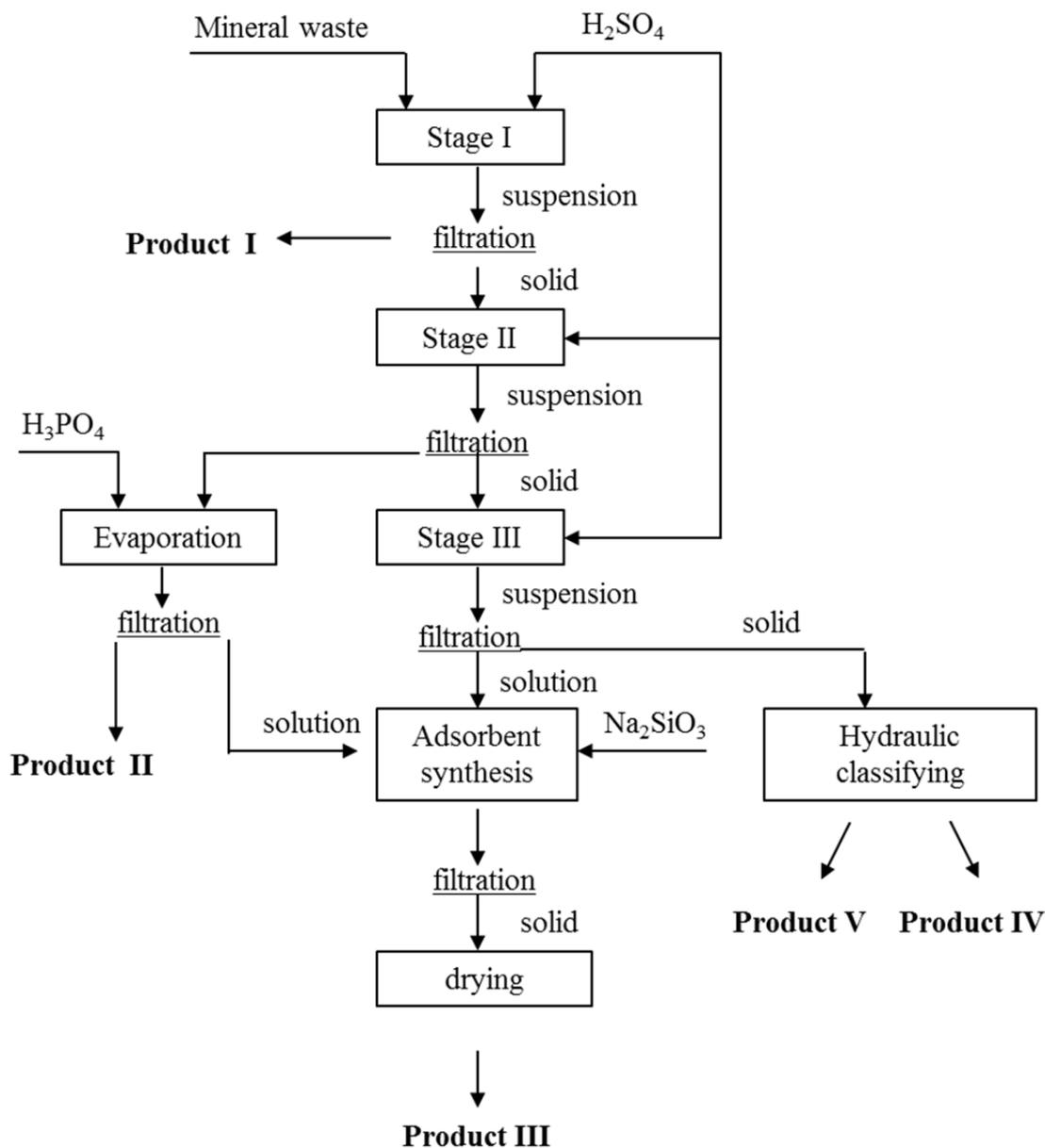
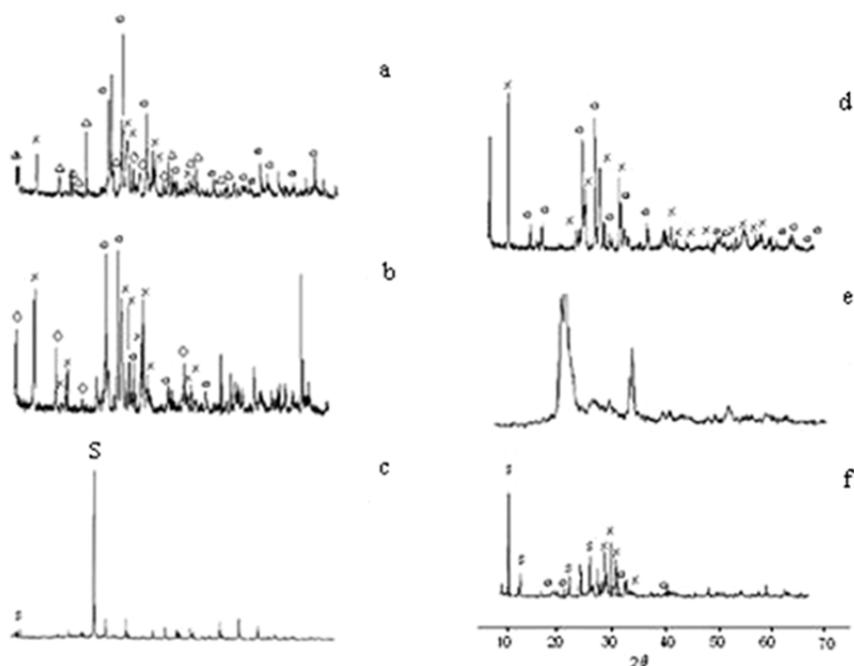


Figure 6. Schematic flow chart of apatite nepheline mineral waste utilization

The process proceeds in a periodic mode. At the first stage of decomposition wastes composed of minerals mixture (Figure 7a) are treated with 100 g/l H<sub>2</sub>SO<sub>4</sub> solution at the solid-liquid stage ratio of S:L=1:6.



o – titanite; Δ – nepheline; ◇ – apatite; x – aegirine; s – CaSO<sub>4</sub>

Figure 7. XRD powder patterns for (a)-apatite nepheline mineral waste, (b)-residue after nepheline dissolving, (c)- gypsum, (d)- residue after apatite dissolving, (e)-titanium phosphate, (f)- the residue after third stage

Under the chosen conditions the nepheline decomposition degree is about 90%. When pH reaches 2.2-2.4, the solid phase is separated by filtrating. The liquid phase is an aluminium and silicium colloid solution, which is watered to 10 g·l<sup>-1</sup> of SiO<sub>2</sub> in order to increase the aggregative stability. The efficiency of using this colloid solution as a coagulum flocculants (Product I) is shown in Table 3.

Table 3. Purification properties of aluminosilicium coagulant

Impurities	Before purification, mg·l <sup>-1</sup>	After purification, mg·l <sup>-1</sup>	Purification efficiency, %
Oil	0.53	0.27	49.0
Suspensions	87.60	14.20	83.8
Nitrate	1.41	1.00	29.1
Sulphate	69.81	63.62	8.9
Chloride	17.20	14.61	15.1
Phosphate	2.93	0.89	69.6
Ferrum	3.50	0.99	71.7

Represented data allows recommending aluminosilicic coagulum flocculants for cleaning industrial waste of suspensions and oil (Zouboulis and Tzoupanos, 2009; Zakharov, et al., 1996; Morgunov, et al., 2006) After the nepheline dissolution (Figure 7b), the solid phase is loaded into a 15% H<sub>2</sub>SO<sub>4</sub> solution. at S:L=1:1. The suspension is heated to 80°C and stirred for 6 h. In the process of heating decomposition of the residual quantity of nepheline and apatite with the formation of phosphoric solution containing aluminium and silicium takes place. Calcium sulphate is crystallized in the form of gypsum and settles out (Figure 7c), which is separated from the solution by filtrating. Orthophosphoric acid is added into the solution to precipitate aluminium and silica gel phosphates. Resultant residues wash off mother liquor and heat-treated (Product II). By varying the treatment regimes it is possible to obtain products different functional properties. (Table 4) (Kalendova, 2003; Yeh & Chang, 2008; Tolmachev, 1998).

Table 4. The effect of treatment regime on the functional properties of Product II

Heat up °C	Chemical composition	Areas of application
40-50	Acidicaluminiumhydrophosphate, amorphous silica	Anticorrosion paint primers, underpaint putty
90- 120	Basic aluminiumhydrophosphate, amorphous silica	Water-based paints (dull), coupling agents
400-550	aluminium phosphate	Water- and organic-based paints (gloss)

The solid residue after apatite separation, (Figure 7d) is treated with sulphuric acid with a concentration of 600g/l  $H_2SO_4$  at a solid-liquid ratio of S:L=1:3. The reaction mass is heated to boiling temperature, held for 7 h and the suspension is filtered. Sulphuric filtrate with a titanium (IV) concentration, when converted into  $TiO_2$  –80-100 g/l and 420-470  $g \cdot l^{-1}$   $H_2SO_4$ , is used for synthesis of titanium-containing ion-exchanger material (Product III) (Bortun et al., 1997; Clearfield, 1995; Nilchi et al., 2004).

For this purpose the solution was heated up to 65-75 °C and the corresponding amount of silica agent (molar ratio of  $TiO_2: SiO_2 = 1:0.15-0.2$ ) was added to it. After stirring of solution for 30 min, phosphoric acid ( $TiO_2:P_2O_5 = 1:0.5-2$ ) is added. The low solubility of titanium and silicium phosphates favours the emergence of seeds promoting the formation of composite solid (Reaction 5). Evaporated filtrate after the stage of titanite dissolution is used as a phosphoric reagent. The suspension was agitated for 6 h, the solid then filtered and washed with water (till pH 3.0-3.2). The final solid was dried initially on air and then at 70°C. Precipitation degree of titanium (IV) when producing titanium phosphate is 95-99%. The final silica titanium phosphate composite with formula  $Ti(OH)_{2(1-x)}(HPO_4)_x \cdot ySiO_2 \cdot nH_2O$  can be used as an ion-exchanger (Figure 6e) ((Maslova et al., 2008). The silicium component facilitates pelletizing of the material. The synthesized material has good sorption affinity towards mono- and polyvalent metal cations (Figure 8).

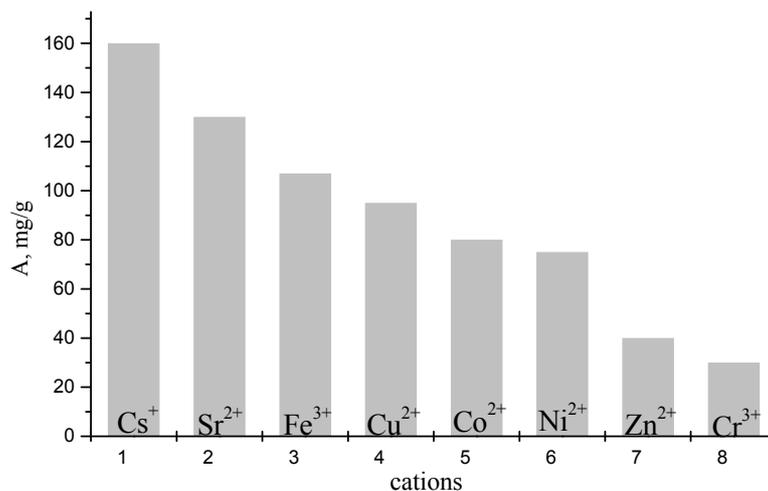


Figure 8. Sorption affinity (A) of some cations by titanium phosphate composition

Residue from the titanite decomposition (Figure 7f) is divided into synthetic and mineral fractions by means of hydraulic classification. The synthetic fraction, collected in catchers, includes calcium sulphate and amorphous silica. The residue is heat treated at 300°C. The resulting Product IV is a white or ecru powder, which can be used as component of dry construction mixtures (Olmez & Erden, 1989; Singh & Garg, 2000; Singh, 1988; Singh & Garg, 1992). The mineral part, mainly consisting of aegerite (Product V), is used in the production of weatherproof fillers of paint and construction materials (Table 5).

Table 5. Characteristics of the products obtained from mineral waste

Product	Components	Field of use
Product I	Colloidal solution of aluminium and silicium—a coagulating-flocculating agent	Thickening of suspensions with a finely divided solid phase. Water purification from oil, suspensions, iron, etc.
Product II	Acid aluminium phosphate, silica gel	Anticorrosion compounds on the organic and water bases
Product III	Titanium hydrophosphate or its composite with silica gel	Sorbent purification of toxic liquid effluents
Product IV	Calcium sulphate, amorphous silica, titanium dioxide	Dry building composites for whitewashing
Product V	Sphene and aegirite concentrates	In the production of pigments, weather-resistant paints and paint primers

## 5. Conclusions

By using the data of mineral composition of polymineral apatite-nepheline ore dressing wastes, there has been developed a method of their recovery, yielding synthetic and mineral products. This involved researching of the kinetics of mineral dissolution in sulphuric acid and mechanism of phase formation in the resulting system. With regard for different solubility of nepheline, apatite, titanite and egrine in acid media, the sequence of mineral separation was determined. There have been determined the parameters ensuring the process completeness. The composition and characteristics of solid and liquid phases obtained during the mineral dissolution have been studied, which permitted to use them directly as precursors in synthesis of such useful products as anticorrosion pigments and binding materials, water and organic paint fillers, and sorbents of the ion-exchange type. Processing of polymineral (not individual) wastes in sulphuric acid has shown that the main process parameters are easily adjustable. On the basis of a physical-chemical study, there has been developed a technology for polymineral wastes processing which meets the environmental requirements.

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