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Research article

Preparation and use of complex titanium-containing coagulant from quartz-leucoxene concentrate

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Abstract. The search for the new high-efficiency reagents for wastewater treatment is a challenging and urgent task. Titanium-containing coagulants represent a new trend in water treatment and have a much higher efficiency that the traditional aluminium and iron-containing coagulants. The high cost of reagents significantly hinders their implementation. Complex titanium-containing reagents are coagulants prepared by modifying the traditional coagulants by adding 2.5-10.0 wt.% titanium compounds. In this work, titanium tetrachloride prepared from quartz-leucoxene concentrate was prehydrolyzed with subsequent double decomposition with sulfuric acid. The resulting mixture of hydrochloric and sulfuric acids was neutralized with aluminium hydroxide/oxide to form a self-hardening mixture (chemical dehydration). The sample of a complex sulfate-chloride titanium-containing coagulant was a mixture of $AlCl_3 \cdot 6H_2O$ – 5-20 wt.%, $Al_2(SO_4)_3 \cdot 18H_2O$ – 70-90 wt.% and $TiOSO_4$ – 2.5-10.0 wt.%. It was proved that by changing the ratio of aluminium oxide/hydroxide and titanium tetrachloride at the stage of prehydrolysis and double decomposition, it is possible to obtain samples of a complex coagulant with different contents of the modifying additive of titanium compounds. An assessment of the coagulation properties of the complex reagent demonstrated its higher efficiency in cold water compared to aluminium sulfate. Studies on the use of the complex titanium-containing coagulant in the process of wastewater treatment from phosphate anions and suspended matter demonstrated its higher efficiency as compared to that of traditional reagents. The advantages of the prepared reagent are a reduction in the effective dose of the reagent, minimization of residual concentrations of pollutants in purified water, intensification of the processes of sedimentation and filtration of coagulation sludge. Purified water can be reused in the recycling water supply system. The use of quartz-leucoxene concentrate and titanium tetrachloride obtained from it as the source material would not only minimize the cost of the resulting complex coagulant, but also take a step towards the implementation of the Zero Waste concept.

Keywords: complex titanium-containing coagulant; quartz-leucoxene concentrate; chemical dehydration; water purification; dephosphatization

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Introduction. Considerable attention is paid to the search for the new high-efficiency reagents for water purification and treatment processes. Most common among the water purification processes is the coagulation and its special case – flocculation. Introduction of the salts of aluminium, iron, titanium, silicon, etc., into the dispersed system leads to hydrolysis with formation of hardly soluble hydroxides of the corresponding metals, which are capable of aggregating dispersed particles around due to neutralization of the surface charge. Depending on the type of reagent used, in addition to neutralization processes, adsorption and flocculation can also occur.

In spite of their relatively high efficiency, the traditional coagulants based on aluminium and iron salts are no longer able to optimally perform their assigned tasks, and also have a number of major disadvantages. The strict residual MPC standard, a narrow pH range and a low efficiency in cold water are the main disadvantages of aluminium salts, while iron salts have strong abrasive properties and can form highly soluble compounds with organic ligands [1-3]. Titanium salts, despite an almost complete lack of shortcomings and the highest efficiency, are not commonly used due to their high cost [4-6].

This situation can be overcome by the use of complex titanium-containing reagents - aluminium or iron salts modified by the addition of titanium compounds in the amount of 2.5-10 wt.%. The key advantage of such reagents is a possibility of producing them from various mineral raw materials and technogenic waste, which will significantly reduce the cost [7, 8]. Titanite (sphene), ilmenite and other concentrates can be used as potential raw materials for preparing complex titanium-containing coagulants. These raw materials are used to produce titanium dioxide from ilmenite as well as tanning agents, sorbents and other products from titanite.

Quartz-leucoxene is a large-tonnage by-product of the development of the Yarega oil-titanium field with titanium compounds content of 7-11 wt.%. In the course of flotation enrichment, a quartz-leucoxene concentrate with titanium dioxide content to 45-50 wt.% was obtained. Reserves of quartz-leucoxene, according to various estimates, are approximately 260 million tons [9].

The chemical nature of the mineral (close intergrowth of silicon and titanium dioxides) determines a high chemical resistance and makes it impossible to use the traditional sulfuric acid processing method [10, 11].

Currently, a number of technologies were developed for the enrichment or processing of quartz-leucoxene concentrate to prepare rutile concentrate:

• Autoclave leaching [9].
• Magnetic separation and reduction roasting [9, 12].
• Plasma-thermal reduction [13].
• Hydrofluoride leaching [14, 15].
• Silicon reduction [16].

Undoubtedly, the most successful trend of processing quartz-leucoxene concentrate is a comprehensive solution developed by a team of authors under the leadership of prof. G.B. Sadykhov at the A.A.Baikov Institute of Metallurgy and Materials Science, RAS. The proposed technology comprises the stages of magnetizing enrichment and autoclave leaching of quartz-leucoxene concentrate to produce rutile concentrate and acicular wollastonite [9, 11, 12].

The rutile concentrate is directed to the stage of selective chlorination to produce a mixture of titanium, silicon, aluminium, etc., chlorides. The resulting $TiCl_4$ after rectification purification can be used to produce technical titanium dioxide [17, 18]. Unfortunately, the multi-stage cleaning system considerably increases the cost of the process.

As an alternative processing trend, one should mention the pyrometallurgical processing of quartz-leucoxene concentrate to produce titanates for the needs of various industries [19].

To increase the economic attractiveness of the chlorination/rectification process, it seems most appropriate to develop an alternative technology for producing innovative reagents with increased cost and market demand.

The main goal of this work is to develop a technology for the synthesis of a complex titanium-containing coagulant using titanium tetrachloride obtained by selective chlorination of quartz-leucoxene concentrate as a raw material. An additional goal is to check the efficiency of the complex reagent in wastewater treatment processes and compare its activity with traditional aluminium-containing coagulants.

Methods. As the initial precursor for the synthesis of the complex reagent, a sample of titanium tetrachloride containing an admixture of silicon tetrachloride was used in the amount up to 0.4 wt.% obtained by selective chlorination of quartz-leucoxene concentrate from the Yarega oil-titanium field.

In the experiment, samples of aluminium oxide and hydroxide produced by Sigma-Aldrich (Germany) and sulfuric acid produced by "Component-Reaktiv" (Russia) were used. Aluminium sulfate and aluminium oxychloride produced by Kemira (Finland) were chosen as a sample for comparison of coagulants.

Identification of the phase composition of solid samples was performed using a DRON 3 N X-ray diffractometer manufactured by NPO "Burevestnik" (Russia) (copper anode Kα-1.5418Å).

The determination of metals in acidic solutions and wastewater was accomplished by atomic emission spectroscopy with magnetic plasma on a "Spectrosky" device manufactured by the GK "Skygrad" (Russia) [20].

Test coagulation (Jar tests of at least three repetitions for each water) was performed on a laboratory flocculator JLT4 Velp Scientifica (Italy). Mixing time of coagulant and wastewater was 2 min, flocculation phase (slow coagulation) 8 min, sedimentation 30 min.

Determination of suspended matter concentration was performed by the gravimetric method, and also on a portable turbidimeter-haze meter Hanna HI 98 703 HANNA Instruments (Hungary).

The filtration rate was determined by passing a given volume of wastewater treated with coagulants through the "White Ribbon" filter (15 μm) for 60 s. Sedimentation time of sludge was estimated using a ZOMS-KFK-3-01 photocolorimeter (Russia).

To assess the coagulation efficiency of complex and traditional coagulants, a sample of river water taken in the warm and cold seasons was used, wastewater from the gas purification unit of the moulding and steel melting shop at the machine-building plant as well as household wastewater subjected to deep biological treatment and directed to the stage of reagent dephosphatization.

Discussion of results. To obtain a solid sample of a complex titanium-containing coagulant, an innovative synthesis technology was applied (Fig.1) including prehydrolysis of titanium tetrachloride, double decomposition and hydrolysis of titanium oxysulfate:

$$TiCl_4 + 4H_2O \rightarrow 4HCl + Ti(OH)_4;$$

$$TiCl_4 + 2H_2SO_4 \rightarrow Ti(SO_4)_2 + 4HCl;$$

$$TiOSO_4 + 3H_2O \rightarrow H_2SO_4 + Ti(OH)_4.$$

Titanium tetrachloride, hydrolytic hydrochloric or sulfuric acid will react with aluminium hydroxide/oxide to form aluminium sulfate and chloride:

$$Al_2O_3 + 6HCl \rightarrow 2AlCl_3 + 3H_2O;$$

$$Al(OH)_3 + 3HCl \rightarrow AlCl_3 + 3H_2O;$$

$$2Al(OH)_3 + 3H_2SO_4 \rightarrow Al_2(SO_4)_3 + 6H_2O \text{ (12 } H_2O \text{ moles lacking)};$$

$$Al_2O_3 + 3H_2SO_4 \rightarrow Al_2(SO_4)_3 + 3H_2O \text{ (15 } H_2O \text{ moles lacking)}.$$

Neutralization of sulfuric acid with aluminium hydroxide or oxide leads to formation of aluminium sulfate; however, since only 18-aqueous crystalline hydrate is in a stable form, the deficient hygroscopic moisture will be absorbed from the reaction mixture, and it would be possible to obtain solid products (chemical dehydration).

The key feature of the proposed technology is a possibility of rejecting the drying processes and obtaining a reagent consisting of two water-soluble metal cations with a charge of 3+ and 4+ for aluminium and titanium, respectively.

Phase composition of the resulting sample of complex titanium-containing coagulant is presented in the diffractogram (Fig.2).

Data on the chemical composition of coagulant samples (a set of results of X-ray diffraction and atomic emission analyses) obtained from aluminium oxide and hydroxide at various ratios of aluminium compounds and sulfuric acid are presented in Table 1.

Comparing the data in Table 1 and Fig.2, it can be concluded that, regardless of the type of the compounds used, the main phase in the complex reagent is aluminium sulfate – 70-90 wt.%, while the product contains aluminium chloride – 5-20 wt.% and water-soluble titanium oxysulfate – 2.5-10 wt.%. The presence of aluminium compounds in the form of sulfate and chloride in the reagent will probably increase the efficiency of coagulation due to a synergistic effect.

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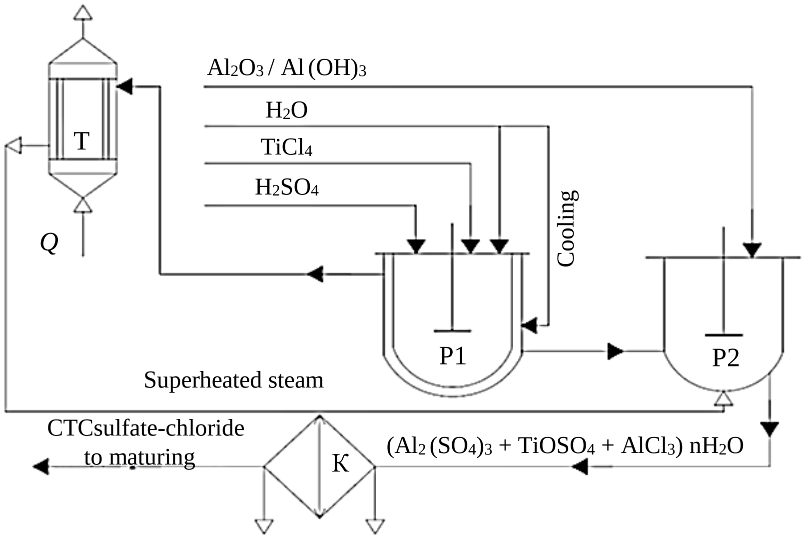

Fig.1. Scheme for the synthesis of a complex titanium-containing coagulant
P1 – double decomposition reactor; P2 – neutralization reactor
(chemical dehydration); T – heat exchanger; K – crystallizer

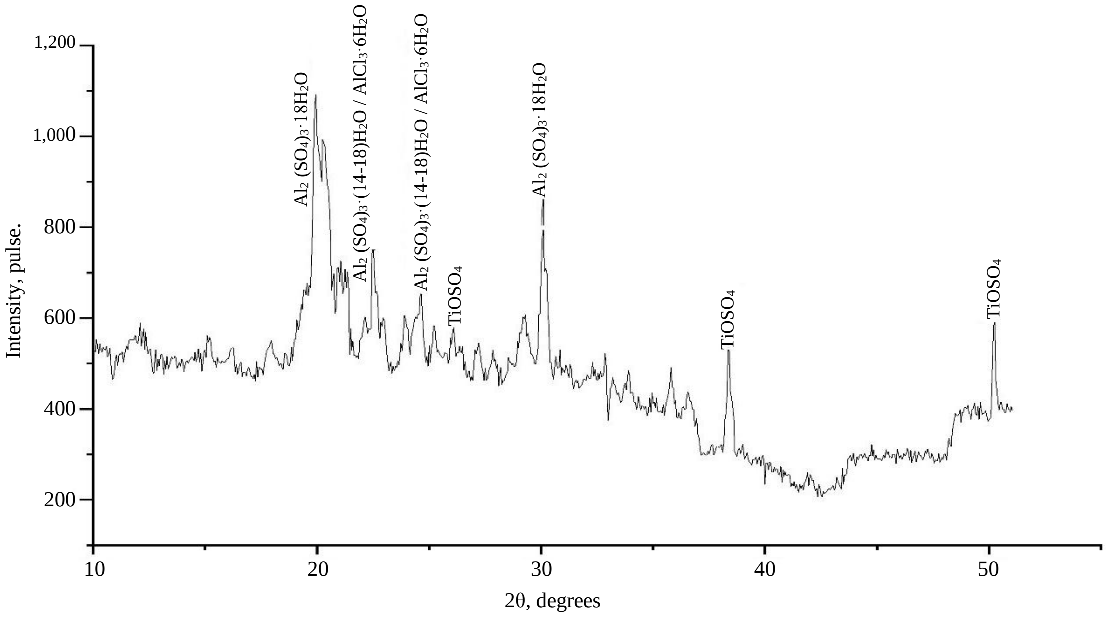

Fig.2. Diffractogram of a coagulant sample

Table 1

Phase composition of samples of complex titanium-containing coagulants

Al-containing component	Mass ratio $(Al_2O_3/Al(OH)_3)/TiCl_4$	Content of main components, wt.%			
		$AlCl_3{\cdot}6H_2O$	$Al_2(SO_4)_3{\cdot}18H_2O$	Ti-components	Insoluble part
Al_2O_3	2.3/1	5.5±0.35	91.7±5.78	2.6±0.22	0.2±0.04
	2/1	10.1±0.64	84.6±5.33	5.0±0.42	0.3±0.06
	1.1/1	15.1±0.95	76.8±4.84	7.6±0.64	0.5±0.10
	0.6/1	19.8±1.25	69.4±4.37	10.1±0.85	0.7±0.14
$Al(OH)_3$	7/1	5.7±0.36	91.7±5.78	2.5±0.21	0.1±0.02
	3/1	10.5±0.66	84.6±5.33	4.8±0.40	0.1±0.02
	1.6/1	16.1±1.01	76.2±4.80	7.5±0.63	0.2±0.04
	1.1	21.7±1.37	67.7±4.27	10.4±0.87	0.2±0.04

The final stage of experiments was the assessment of the efficiency of the prepared samples of complex titanium-containing reagents in water purification processes.

The first object of study was water from the surface water intake collected in the warm and cold seasons. The initial content of suspended matter was 6.9 mg/dm^3 (autumn), pH – 6.54 and 12.3 mg/dm^3, pH – 6.71 (summer).

From the data in Table 2 it is clear that the use of the sample of the complex titanium-containing coagulant makes it possible to neutralize the effect of water temperature and significantly increase the efficiency of the base reagent – aluminium sulfate (70-90 % of the composition of the complex reagent). This phenomenon can be accounted for by the expansion of the range of coagulant hydrolysis products, polycondensation (flocculation) processes as well as neutralization (nucleation) phenomena [21-23].

Table 2

Efficiency of natural water purification from suspended matter, %

Coagulant	Coagulant dose, mg (Me_XO_Y)/dm^3	Purification efficiency, %	Suspended matter concentration, mg/dm^3	Filtration rate, ml/min	Sedimentation time, min
	Cold period (water temperature 8-9 °C)				
Aluminium sulfate	15	65.8	2.36	35	8
Aluminium oxychloride	12	84.3	1.08	46	5
CTCsulfate-chloride	10	93.8	0.43	55	4
	Warm period (water temperature 18 °C)				
Aluminium sulfate	12	94.2	0.71	43	5
Aluminium oxychloride	9.5	98.3	0.21	54	4
CTCsulfate-chloride	8	98.4	0.20	60	3

Data on the efficiency of dephosphatization of household wastewater subjected to deep biological treatment are presented in Fig.3.

Fig.3 shows that the use of the complex titanium-containing coagulant allows minimizing the residual content of phosphate ion, which is primarily due to a low solubility of titanium phosphate compared to aluminium phosphate. In addition to the growing efficiency, the use of the complex reagent also allows reducing by 10-25 % the dose of coagulant required to achieve the $MPC_{fish\ farming}$ standard.

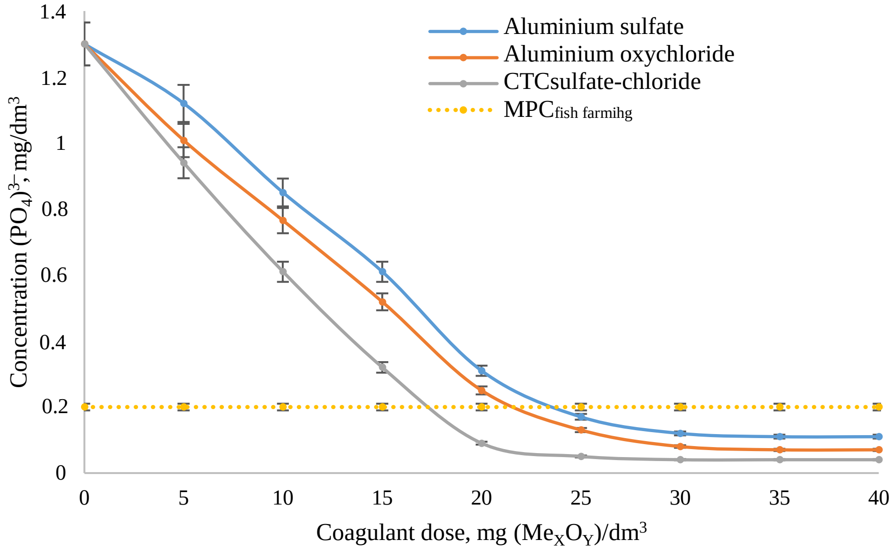

Fig.3. Reagent dephosphatization of wastewater

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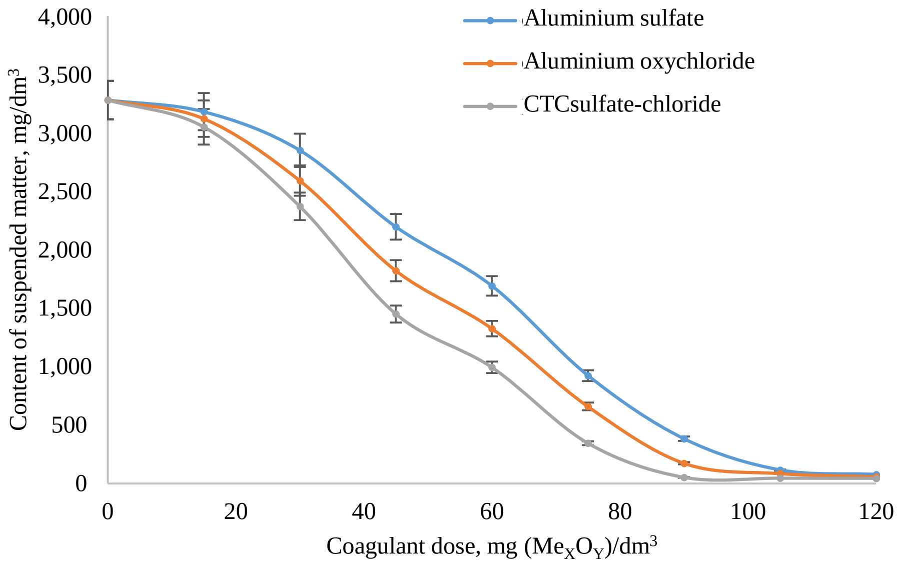

Fig.4. Residual concentrations of suspended matter

The next step was the assessment of the efficiency of the complex coagulant using a sample of wastewater from the area for preparing the core mixture and electro arc steel melting at the machine-building plant in the Moscow Region. The initial content of suspended matter represented by oxides of iron, silicon, aluminium and calcium in water of wet gas purification system was 3,280 mg/dm^3. Data on residual concentrations of suspended matter in wastewater, depending on the type and dose of the coagulant used, are presented in Fig.4.

The graph in Fig.4 clearly shows that the use of the complex coagulant allows not only reducing the consumption of reagents, but also minimizing the residual content of suspended matter in water. Purified water can be sent for reuse in the gas purification system, and, due to the lowest residual concentration of suspended matter in the recirculated water, abrasive wear of the equipment will be much less [24, 25]. The coagulation sludge formed when using the complex reagent settled on average 10-15 % faster, which in the future will significantly increase the productivity of the treatment facilities. Filtration rate of coagulation sludge when using the new reagent was on average 20 % higher than when using traditional reagents.

The composition of coagulation sludge obtained using the complex reagent will be almost identical to coagulation sludge from the use of aluminium sulfate, with the exception of the addition of titanium dioxide in the amount of 5-10 wt.%, while both sludges have the 4th hazard class and can be placed for permanent storage at landfills. In addition, the sludge obtained when using the complex titanium-containing coagulant can be used as a precursor for thermochemical synthesis of titanium aluminates (a component of ceramics, a catalyst) [19].

Conclusion. As part of the work, a technology was proposed for the preparation of innovative complex titanium-containing coagulants using titanium tetrachloride as the raw material obtained from quartz-leucoxene concentrate – large-tonnage mineral waste from the shale oil extraction process at the Yarega oil-titanium field. The use of quartz-leucoxene concentrate will make it possible to take a step towards the implementation of the Zero Waste concept within the framework of circular economy [26, 27].

The possibility of obtaining a solid sample of the complex coagulant including the processes of prehydrolysis of titanium tetrachloride and its double decomposition in combination with chemical dehydration was established. It was proved that the composition of the complex reagent remained virtually unchanged when using both aluminium hydroxide, and oxide. It was proved that the main phase in the complex reagent was aluminium sulfate (70-90 wt.%), while the product contained aluminium chloride and 2.5-10.0 wt.% water-soluble titanium oxysulfate.

Studies on the coagulation ability of the complex titanium-containing coagulant showed that in the efficiency of removing suspended matter, the complex reagent surpassed the traditional coagulants based on aluminium sulfate or oxychloride. It was ascertained that titanium-containing reagents were less sensitive to low water temperatures compared to the base aluminium sulfate.

It was proved that the use of the titanium-containing reagent allowed minimizing the residual concentrations of phosphate anion during after-purification of water that passed the stage of deep biological treatment. The resulting coagulation sludge settled to the bottom faster and had a higher filtration rate, which will significantly increase the efficiency of the treatment facilities.

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