

Industrial Trials Results of Scandium Oxide Recovery from Red Mud at UC RUSAL Alumina Refineries

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Abstract

UC RUSAL pioneered the development of technology to extract scandium oxide from red mud with a pilot plant arranged at one of the company alumina refineries. Pilot production comprises of a site of scandium concentrate production and a site of scandium oxide production. In the paper, the results of industrial trial of scandium oxide production process from red mud are given, in particular: processes of scandium concentrate production and its re-cleaning yielding a commercial product – scandium oxide. In the course of industrial trials, pilot batches of scandium oxide with scandium content 99.9 % were obtained. From the pilot batch of scandium oxide aluminium-scandium master alloy was produced corresponding to the requirements of GOST R 53777-2010 for master alloy of AlSc₂ (A). The developed process, which was implemented at pilot scale is competitive and can be easily integrated into the alumina production. This process does not yield acidic or toxic effluents and provides competitive production cost of scandium oxide

Keywords: red mud, scandium oxide production.

1. Introduction

In recent years, there has been a trend of increasing the demand for scandium oxide and scandium-containing materials. According to [1], in 2017 the scandium oxide market was estimated at 16.3 t/year, and in 2018 it was estimated at 18.9 t/year. By 2028, the demand for scandium is forecast to increase up to 300 t/year in terms of scandium oxide due to its wider application in such industries as shipbuilding, aerospace, aircraft engineering, 3D printing and others [1].

Currently, scandium is mostly produced in China from solutions resulted from the processing of ilmenite concentrates at titanium dioxide production sites and from industrial solutions at zirconium production sites. At the same time, it is known that most of the world's scandium volume is contained in bauxite and its process waste, which is red mud (RM) [2]. It is only the RUSAL alumina refineries located in Russia that annually produce over 2 million tonnes of red mud containing about 240 tonnes of Sc. Unlike other ore and man-made sources of scandium, red mud has a number of advantages, namely, it has already prepared for processing and does not require any extraction and grinding costs.

For more than 50 years, researchers from different countries have been working on the problem to recover scandium from red mud. During this time, various technologies have been developed, including those that include both the scandium pre-concentrating stages through the use of enrichment processes or pyrometallurgical processes and technologies involving direct leaching of scandium with acid and salt solutions [3]. Unfortunately, almost all of these technologies have been tested on a laboratory and large laboratory scale where the amount of processed red mud is calculated in grams and kilograms. Such scale allows researchers neither to obtain an objective assessment of the technology being developed both in terms of the specific consumption of agents and energy costs nor to assess the influence of various factors on the quality of the end product (presence of impurities and their effect on each of the stages of the process, loss of agents and commercial product with effluents and waste, accumulation of impurities in solutions during their recycling process and their impact on subsequent process cycles, impact of the key equipment design and its operation modes on the process performance indicators). All these factors can be investigated only when creating a pilot plant to be operated in a continuous mode, which will make it possible to fine-tune and optimise each stage of the process.

UC RUSAL is the first company in the world to have designed and built a pilot plant (PP) to produce scandium oxide from red mud, which is capable of processing several thousand tonnes of red mud per year to produce up to 100 kg/year of scandium oxide. For several years of plant operation, such processes as leaching of scandium from red mud, concentrating of scandium by sorption from liquors and slurries, recovery of scandium concentrate and removing of impurities from it to produce scandium oxide were fine-tuned; pilot lots of scandium oxide with a purity of $\text{Sc}_2\text{O}_3 = 99.0 - 99.9$ wt.% in the calcined product were produced and tested at customers' sites, and fundamentally new equipment designs were created and tested.

This paper presents the results of the pilot field tests of the technology to produce scandium oxide from red mud that were carried out at an own pilot production site located at one of the UC RUSAL alumina refineries.

Unlike competitors' technologies, the RUSAL's technology has a number of advantages, namely:

- own stable source of raw materials (red mud from the process implemented at the alumina refineries and waste red mud stored in red mud disposal areas);
- the technology was developed using own R&D resources of the company;
- this is the world's only technology implemented to produce scandium oxide from the red mud delivered from alumina refineries;
- in contrast to other known technologies for the production of scandium, it does not provide for the use of acids at the stage of scandium recovery and concentrating; therefore, the technology does not have any acidic and salt effluents;
- the red mud, from which scandium was extracted, is prepared for further processing into other commercial products or for storage in an 'ultra-dry' way, which allows reducing the capital expenditure for the construction of red mud disposal areas by approximately 30%;
- exhaust kiln gases can be used as agents, which cuts operating costs and reduces carbon dioxide emissions;
- scandium oxide of a specified quality can be produced for the needs of own production of Al-Sc alloys and master alloys; and
- flexibility to increase production as the market for scandium develops.

2. Experiments

The basic process flow diagram of pilot production of scandium oxide from red mud is shown in Figure 1 and includes the following process areas:

- filtering the source red mud slurry;

- reslurrying the red mud with a spent leach liquor;
- leaching of scandium from the red mud;
- filtering the leached red mud slurry to produce a scandium product solution;
- sorption of scandium from the solution or slurry on an ion exchange resin;
- desorption of scandium from the ion exchange resin to produce a scandium commercial recycled product;
- carbonisation of the spent liquor after the sorption process to produce a spent leach liquor;
- removing of impurities from the scandium commercial recycled product;
- hydrolytic precipitation of the scandium concentrate;
- filtration of the scandium concentrate;
- recycling of the desorbing solution from the spent liquor after the hydrolysis process;
- stepwise removing of impurities from the scandium concentrate to produce a scandium oxalate; and
- thermal decomposition of scandium oxalate to produce a commercial product in the form of scandium oxide.

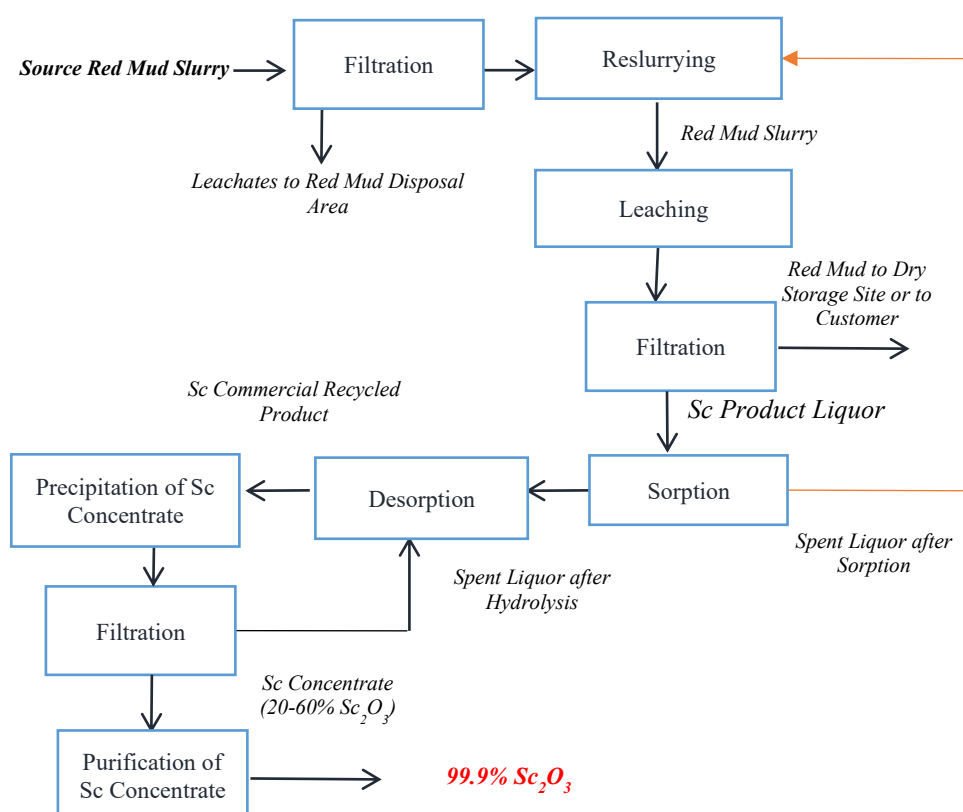


Figure 1. The basic process flow sheet of pilot scandium oxide production from red mud.

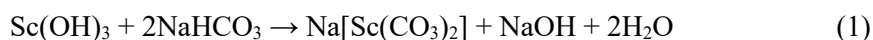
The hardware of the pilot production site includes both standard tanks (reactors, vessels), filter presses and some unique equipment that has been created for this production site and designed for carrying out continuous sorption of scandium from solutions (columns) and slurries (a sorption unit with a suspended layer of sorbent, the so-called Pachuca tank), a carboniser to treat the spent liquor after the sorption process with carbon dioxide with a view of obtaining an optimal ratio of $\text{NaHCO}_3 / \text{Na}_2\text{CO}_3$ in the liquor, which is supplied to the new red mud leaching stage (spent leach liquor).

A detailed description of the technology for producing scandium oxide from red mud on a pilot field scale is given in references [4-7].

2.1. Leaching of Scandium from Red Mud

Leaching of scandium from red mud was carried out by using a solution containing a sodium carbonate and sodium bicarbonate mixture and through a simultaneous aeration of the slurry with carbon dioxide (the so-called carbonisation leaching method).

This method is based on the scandium ability to dissolve in the presence of an excessive amount of carbonates and hydrogen carbonates of alkaline elements to form anionic complexes like $[\text{Sc}(\text{CO}_3)_n]^{(2n-3)-}$ ($n \geq 2$), Equation (1) [7]. The aeration of slurry with carbon dioxide during the leaching process makes it possible to shift the equilibrium of the chemical reactions for dissolving $\text{Sc}(\text{OH})_3$ from the solid phase of red mud due to the formation of an additional amount of sodium bicarbonate according to Equation (2) and contributes to a higher degree of recovery of scandium into the solution:



According to the results of the research conducted by the authors, the carbonisation leaching of scandium from red mud with a solution containing a NaHCO_3 and Na_2CO_3 mixture results mostly in the recovery of scandium which is sorbed on the surface of red mud [8].

The pilot production process starts with preparation of red mud for leaching. For this purpose, the source red mud slurry washed down and prepared for mud disposal at an alumina refinery is fed into the buffer tank and filtered by the filter press (Figure 2, right); then, the red mud is reslurried in a spent leach liquor containing a sodium carbonate and sodium bicarbonate mixture in the reslurry reactor. The spent leach liquor is produced through aeration of the spent liquor after sorption with carbon dioxide in the carboniser reactor (Figure 2, left), which allows achieving an optimal ratio of $\text{NaHCO}_3 / \text{Na}_2\text{CO}_3$ in the liquor.

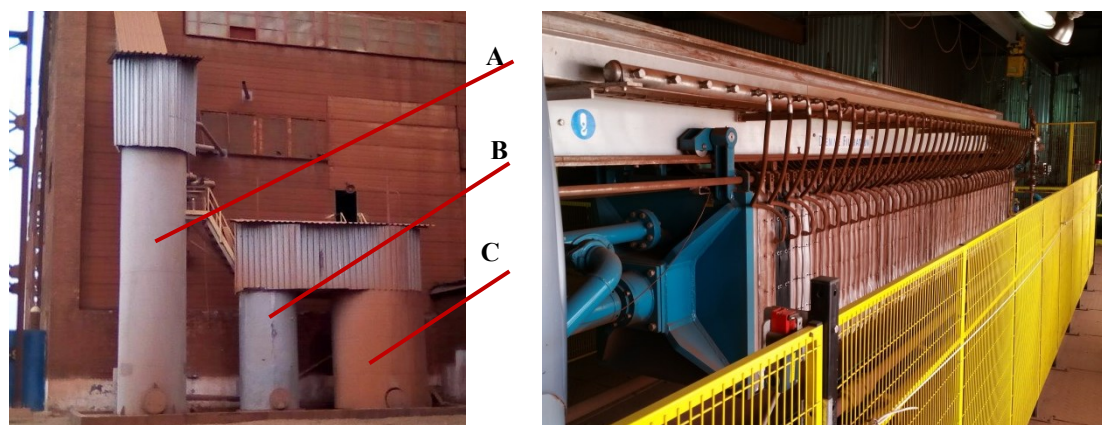


Figure 2. Area to leach scandium from red mud. Left: A - a carboniser, B - a spent liquor agitator, C - red mud slurry agitator; right: filter press to filter red mud slurry.

The red mud slurry produced in the reslurry tank is supplied into the leaching reactor. 2 to 5 tonnes of red mud slurry in terms of dry red mud can be charged at one time.

During the operation of the PP, different modes for leaching of scandium from red mud were fine-tuned, and the effect of the following parameters on the degree of recovery of scandium was investigated:

- the process temperature;
- the liquid/solid ratio;
- the total concentration of the leaching agent ($\text{Na}_2\text{O}_{\text{total}}$ in NaHCO_3 and Na_2CO_3) and $\text{NaHCO}_3 / \text{Na}_2\text{CO}_3$ ratio in the solution and, respectively, the pH of the liquor;
- the duration of the leaching process; and
- the initial concentration of scandium and impurities entering the new leaching cycle.

According to experimental data, the increase in temperature and concentration of the leaching agent increases the degree of recovery of scandium from red mud into the solution. Figure 3 shows the dependence between the degree of recovery of scandium from red mud and the concentration of the leaching agent at different temperatures at the optimal 3-hour long duration of the leaching process. As can be seen, the maximum degree of recovery is achieved by increasing the concentration of $\text{Na}_2\text{O}_{\text{total}}$ over 60 g/l at a temperature above 75 °C.

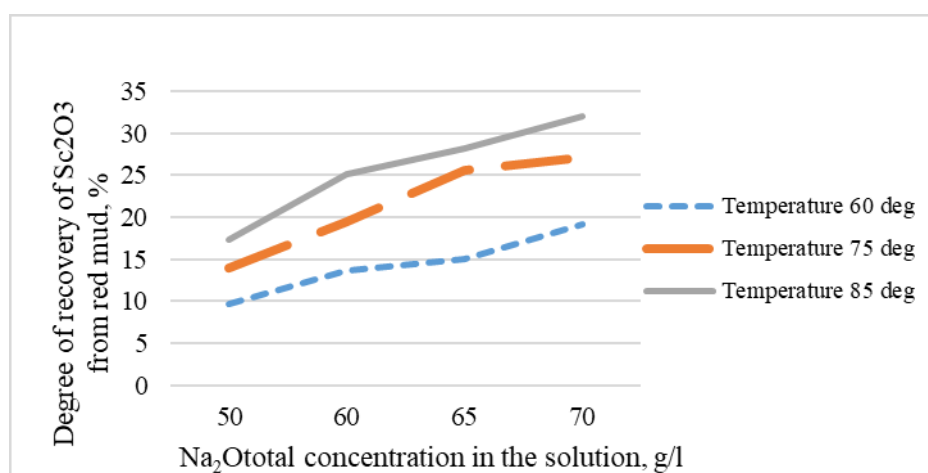


Figure 3. The effect of temperature and $\text{Na}_2\text{O}_{\text{total}}$ concentration on the degree of scandium recovery from red mud.

According to the pilot field test results, the optimal parameters of the leaching process have been determined, namely: the liquid/solid ratio is not less than 3.5, the $\text{Na}_2\text{O}_{\text{total}}$ concentration in the liquid phase of the mud slurry is 60 - 70 g/l with a ratio of $\text{NaHCO}_3 / \text{Na}_2\text{CO}_3$ is about 1.5 - 2, the process duration is 3 hours, and the process temperature is 80 - 85 °C. Carrying out the leaching process with these parameters made it possible to achieve a degree of scandium recovery from red mud into the solution of at least 30 %.

After the leaching process, the red mud slurry is fed to a filter press where it is filtered; the resulting filtrate is a product solution having a Sc_2O_3 content of 8 - 15 mg/l, which is supplied to the stage of scandium concentrating by sorption, and the washed down cake of the leached red mud is disposed of in a red mud disposal area of an alumina refinery (or supplied to customers if there is a demand for it).

The modes of washing down the source and leached red mud using the filter press, which were fine-tuned at the pilot plant, made it possible to reduce the consumption of agents more than twofold, which significantly reduced the production cost of scandium oxide.

2.2. Concentrating of Scandium by Sorption

The process of scandium concentrating by sorption is based on the ability of some ion exchange resins to selectively sorb scandium from solutions on their surface. For scandium, the most selective sorbents are phosphorus-containing ion exchange resins due to the formation of strong scandium complexes with phosphorus-containing functional groups of ion exchangers [9]. Based on the results of our earlier laboratory tests of the scandium sorption and desorption processes in static and dynamic conditions, an ion exchange resin brand with the best performance characteristics, Lewatit TP-260 (produced by Lanxess, Germany), was selected for the pilot field tests.

The initial parameters of the scandium sorption and desorption processes (temperature, solution feed rate, concentration of sodium carbonate in the desorbing solution) were determined according to the results of laboratory studies, the results of which are given in paper [10]. The optimum temperature of the process involving sorption of scandium from solutions containing a sodium carbonate and sodium bicarbonate mixture at a linear throughput rate of the solution of 1 to 2 m/h is 60 °C.

The optimum temperature of the scandium desorption process (40 to 50 °C) is due to the need to use desorbing solutions with a high concentration of sodium carbonate (320 - 350 g/l for Na₂CO₃) in order to achieve the maximum degree of scandium recovery for producing a commercial recycled product.

The concentrating of scandium by sorption was carried out as described below. The Sc-containing product solution obtained after filtration of the leached red mud slurry was supplied to a pressure vessel, from which it was continuously fed at a certain linear speed to the bottom of the sorption column (Figure 4, right) and passed through the ion exchange resin loaded into it, with the concentration of scandium in the solution decreased as it moved upwards through the resin layer, reaching a minimum value at the outlet of the sorption column, while the maximum content of scandium on the resin was reached in the bottom of the column. After the ion exchange resin had been saturated with scandium, part of the resin from the bottom of the sorption column was pumped out by airlift into the top of the desorption column for recycling.



Figure 4. Photo showing a concentrating unit of scandium by sorption. Left: tops of the sorption and desorption columns; right: bottoms of the sorption and desorption columns.

The spent liquor obtained at the outlet of the sorption column was fed into the carboniser, where it was carbonated with carbon dioxide to produce a spent leach liquor with a NaHCO₃ / Na₂CO₃ ratio of about 1.5 - 2.

After the desorption stage, the recycling of the resin was carried out in the desorption column; for this, the desorbing solution, which was a concentrated solution of sodium carbonate, was fed into the bottom of the said column (Figure 4, right). In the desorption and sorption columns, the solution and resin counter-current flow is implemented: the desorbing solution first passes through the lower layer of the ion exchanger containing the minimum amount of scandium and desorbs scandium from the surface of the ion exchanger. At the outlet of the top of the column (Figure 4, left), a commercial recycled product with a 100 to 800 mg/l content of scandium in terms of Sc₂O₃ was obtained. The recycled resin was pumped by airlift from the bottom of the desorption column into the top of the sorption column.

The scandium sorption and desorption were carried out continuously in a spaced-out bed placed in the columns under stabilised conditions, namely:

- the sorption and desorption temperature remained constant due to the thermal insulation of the columns, pressure vessels, solution supply pipelines and due to the fact that the columns were equipped with an automatic temperature control system and the possibility of heating the product and desorbing solutions in the pressure tanks was provided;
- the feed rate of the product and desorbing solutions was easily adjusted and remained constant due to the automatic flow control system according to the readings of the flow meters;
- the volumes of the ion exchange resin pumped between the columns were monitored according to the readings of the level gauges located in the columns; and
- the material flows in the process areas for leaching of scandium from red mud, sorption and desorption, preparing a spent leach liquor, extracting the scandium concentrate were balanced, which made it possible to provide the continuity of the whole process.

The resin and solution counter-current flow implemented at the sorption stage made it possible to achieve the maximum degree of scandium recovery on the resin at its minimum concentration in the spent liquor after sorption and the maximum exchange capacity of the resin for scandium in the lower layers of the ion exchanger; this provides a high concentration of scandium in the commercial recycled product and then, in the scandium concentrate.

Table 1 shows the experimental data on the effect of the feed rate of the product and desorbing solutions on the concentration of scandium in the spent liquor and commercial recycled product, respectively.

Table 1. The effect of the feed rate of product and desorbing solutions on the scandium sorption and desorption processes.

Linear feed rate of the solution to the sorption unit, m/h	Average concentration of Sc ₂ O ₃ in the spent liquor, mg/l	Linear feed rate of the solution to the desorption unit, m/h	Average concentration of Sc ₂ O ₃ in the commercial recycled product, mg/l
1	0.4	0.2	327
1.5	0.8	0.25	215
2	1.9	0.3	156
5	3.5	0.4	83

As can be seen, with an increase in the feed rate of the solutions, the degree of scandium recovery at the sorption and desorption stage decreases. To achieve maximum Sc throughput the authors selected the following optimal linear feed rates: 1.5 m/h for the product solution and 0.25 to 0.3 m/h for the desorbing solution.

The technological characteristics of the sorption/desorption processes that cannot be implemented in a laboratory were fine-tuned during the pilot field tests. For example, the authors obtained data on the effect of impurities contained in the solution on the sorption of scandium and the change in the sorption capacity of the resin as iron and titanium impurities accumulated on it; fine-tuned the practices of recycling the resin for removing impurities from it, pumping the resin between the columns using airlifts of various designs; optimised parameters for washing down the resin and forcing out the pore moisture to prevent dilution of the spent liquor and the commercial recycled product while minimising the consumption of sodium carbonate to adjust the composition of the spent leach liquor and desorbing solution.

The stabilisation of parameters of the process to concentrate scandium by sorption made it possible to obtain reliable data on the degree of scandium recovery at the sorption and desorption stages as well as stabilise the quality of the commercial recycled scandium product according to the content of scandium and impurity components.

2.3. Extracting of Scandium Concentrate

The extracting of scandium concentrate from the commercial recycled product obtained at the stage of scandium concentrating by sorption was carried out in two stages by hydrolysis which included the addition of a sodium hydroxide solution and the maintenance of the solutions at an elevated temperature. The first stage included the precipitation of impurity components, the main of which were titanium and iron.

The commercial recycled scandium product containing at least 100 mg/l of scandium oxide was fed to a hydrolysis reactor, where the solution was treated with sodium hydroxide until the pH reached about 10 - 10.5; as a result, the impurities of iron, titanium and other elements precipitated in the form of insoluble hydroxides, whereas scandium remained in the solution [4]. The slurry produced in the hydrolysis was filtered using a filter press, while the precipitate was supplied to the red mud leaching stage to prevent scandium losses, and the filtrate, which was an impurity-free commercial recycled product, was fed to the second hydrolysis stage.

While fine-tuning the process of removing impurities from the commercial recycled product, the process parameters (temperature, soak time, amount of sodium hydroxide, etc.) which allowed precipitating the impurities of titanium and other components to the fullest extent possible, while leaving scandium in the solution, were being selected. As can be seen from Table 2, at the first stage of hydrolysis, the titanium content in the solution was reduced by 5 - 9 times, while the zirconium concentration decreased slightly.

Table 2. The experimental data on the process of removing impurities from the commercial recycled product at the 1st hydrolysis stage.

Experiment No.	Before/after hydrolysis	Concentration of components in the commercial recycled product, mg/l		
		Sc ₂ O ₃	ZrO ₂	TiO ₂
1	Before	188	54	41
1	After	165	10	10
2	Before	113	246	195
2	After	105	230	34
3	Before	156	211	247
3	After	149	190	38
4	Before	263	239	248
4	After	261	233	44
5	Before	177	185	197
5	After	166	165	21

Following the removing of impurities, the commercial recycled scandium product was fed into the hydrolysis reactor to implement the second hydrolysis stage and extract scandium concentrate. To achieve this, sodium hydroxide was added to the solution until the pH in the solution reached about 12 - 12.5, and the solution was kept at a temperature of 70 - 80 °C [4]. The scandium concentrate slurry was fed to precipitators (Figure 5, left), and then the transparent part of the liquid phase was poured into the reactor to prepare a desorbing solution, and the condensed scandium concentrate slurry was filtered using a filter press (Figure 5, right).



Figure 5. The photo showing the scandium concentrate extraction area. Left: precipitators for the preparation of scandium concentrate slurry before its filtration; right: filtration of scandium concentrate.

The chemical composition of some lots of scandium concentrate produced during the pilot field tests is shown in Table 3. The scandium concentrate quality varied depending on the quality of the commercial recycled product and the conditions of the first and second hydrolysis stages.

Table 3. The chemical composition of the pilot lots of scandium concentrate, wt.%.

Lot No.	Sc ₂ O ₃	ZrO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	Na ₂ O	SiO ₂	H ₂ O
1	27.9	11.1	1.02	0.14	1.5	2.3	21.1	1.3	48.0
2	24.3	11.4	0.78	0.21	2.2	3.0	20.8		55.3
3	22.5	11.5	2.1	0.1	1.4	2.3	25.7	1.0	56.9
4	23.1	12.4	1.8	0.079	1.1	3.9	20.5	1.1	52.8
5	32.4	10.2	1.3	0.13	3.2	4.4	10.8	2.4	46.1
6	25.9	4.6	0.32	0.065	2.6	4.4	28.8	0.7	55.5

During the pilot field tests, the optimal parameters of the scandium concentrate extraction process were selected, namely:

- sodium hydroxide consumption at the first and second hydrolysis stages depending on the commercial recycled product quality;
- soak time;
- scandium concentrate precipitation time;
- parameters of washing down the scandium concentrate; and
- modes of aeration of the spent liquor with carbon dioxide after hydrolysis to obtain a desorbing solution therefrom.

2.4. Obtaining Scandium Oxide from Scandium Concentrate

A pilot plant (Figure 6) was created to carry out the pilot field tests of the scandium concentrate purification process and produce pilot lots of scandium oxide. The plant is capable of producing at least 100 kg/y of scandium oxide from scandium concentrate obtained from red mud.

The technology for removing impurities from scandium concentrate is described in paper [11] and patents [4-5]. It includes the following process stages:

- washing down the scandium concentrate;
- selective leaching of scandium out of the scandium concentrate with a sulphuric acid solution;
- separating the scandium-containing solution from the acid-insoluble residue;
- precipitating the $\text{Na}_3\text{Sc}(\text{SO}_4)_3 \times 5\text{H}_2\text{O}$ double scandium sulphate by adding sodium sulphate;
- dissolving the double scandium sulphate in water and precipitating iron and thorium impurities from the resulting solution by adding barium compounds;
- precipitating scandium hydroxide from an impurity-free Sc-containing solution by treatment with sodium hydroxide solution;
- obtaining a scandium oxalate; and
- thermal decomposition of scandium oxalate to produce a scandium oxide product.



Figure 6. The pilot plant for the production of scandium oxide. Left: a scandium concentrate re-cleaning unit; right: a scandium oxide batch in the furnace.

During the pilot field tests conducted at the pilot plant for the production of scandium oxide, the scandium concentrate obtained at the pilot plant was processed, with various parameters of each process stage being fine-tuned. This made it possible to achieve the maximum commercial yield of scandium oxide from the concentrate to the oxide and minimise the consumption of agents (sulphuric acid, sodium hydroxide) for the purification of scandium concentrate due to the selective recovery of scandium from scandium concentrate and stepwise removing of impurities. To avoid scandium loss, all spent liquors after the precipitation of their compounds were fed to the stage of scandium concentrating by sorption.

During the operation of the pilot plant, some pilot lots of scandium oxide were produced (Figure 6, right); the content of scandium oxide in the lots was 99.0 - 99.9 wt.%. The chemical composition of one of the lots of scandium oxide is shown in Table 4.

According to the pilot field test results, the costs for removing impurities from scandium concentrate depending on the chemical composition of the scandium concentrate and the requirements for the quality of scandium oxide were determined.

**Table 4. The chemical composition of a pilot lot of scandium oxide,
produced by UC RUSAL, wt.%.**

Sc ₂ O ₃	Zr	Si	Al	Fe	Ti	Ca	Mg	K	Na	Th	∑Ln
99.9	0.038	0.001	0.0024	0.0075	0.0018	0.0016	0.001	0.001	0.022	0.015	0.002

Pilot lots of scandium oxide produced by the pilot plant were successfully tested at potential customers' sites. At the aluminium master alloy production area of RUSAL ETC in Krasnoyarsk, an aluminium scandium master alloy the quality of which meets the requirements of GOST R 53777-2010 for AlSc2 (A) master alloys was produced (Figure 7).



**Figure 7. The photo of an Al-Sc master alloy produced
by RUSAL as per GOST R 53777-2010.**

3. Conclusion

The operation of the pilot plant for the production of scandium oxide from red mud made it possible to fully fine-tune the technology which was developed by the UC RUSAL own engineering centre. Pilot plant testing allow optimisation: reduce operating costs for the production of scandium oxide by several times by achieving the maximum degree of scandium recovery in each of the process areas, optimising the process parameters and also test the new equipment.

The pilot field tests were continuously carried out for 4 years. This made it possible to obtain reliable data on the consumption of agents and energy resources, commercial product yield, identify the features of individual process stages, which arise in a closed process cycle, develop pilot lots of scandium oxide and test them at potential customers' sites.

According to the results of the pilot field tests, the authors obtained data that formed the basis of a feasibility study for the construction of an industrial scale production facility to produce scandium oxide from red mud.

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