



DOI: 10.31643/2021/6445.35

## Study of influence of the charge granulometric composition on the quality of burned anodes

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### ABSTRACT

Many factors affect the quality of produced anodes: composition and grade of raw materials, recipe, technology, operating parameters, the granulometric composition of charge materials etc. The most significant influence is from the last factor. The article provides research results on the effect of fine fractions on the quality of anodes. The research object is charge of different granulometric composition used to produce anodes at the Kazakhstan Electrolysis plant in Pavlodar. Six types of anode mass with different granulometric composition were prepared for the experiment. The anode mass was made of petroleum coke, pitch, recycled anodes, and dust from filters and grinder produced during coke treatment. Several properties of the specimens were studied. In total, six specimens were made and, specimen № 4 drew particular attention. Its granulometric composition is as following: coarse fractions – 25 %, medium fractions – 20%, fine fractions (grinder dust) – 50 %, and filter dust – 5 %. The results showed that some specifications of anodes, like gas permeability and apparent density that affect anode consumption, are improved as the fineness of dust grinding. For instance, the addition of dust fraction in the charge increases anode density from 1.542 to 1.639 kilogram per cubic decimeters and decreases gas permeability from 1.01 to 0.78 nPm. In addition, the presence of dust fraction minimizes the porosity of the anode block. The results of the investigation help adjust the optimal recipe of anode mass to obtain high-quality anodes.

**Keywords:** anode mass, production of baked anodes, gas permeability, density, granulometric composition, fine fraction.

Received: 04 May 2021

Peer reviewed: 23 July 2021

Accepted: 04 October 2021

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### Introduction

Burnt anodes in an aluminium electrolyzer supply current and participate in the electrochemical process by being burned in formed oxygen. Therefore, one of the main specifications of anodes used as reducing agents during electrolysis of cryolite-alumina melts is the consumption rate.

The relevant literature review showed that increase in anodes density causes a decrease in gas permeability. Consequently, the consumption rate of anodes is reduced during electrolysis of cryolite-alumina melts [1-4].

Anode mass and burnt anodes are made of coal pitches, petroleum coke, and stubs (remnants of anodes) mixed with a recipe. “Green” anode blocks are then formed out of the mixture and are calcinated to produce burnt anode blocks.

There are special requirements for an aluminium electrolyzer:

- as a physical object that must withstand mechanical stress at high temperatures and supply high electrical conductivity;
- as a chemically active electrode that undergoes chemical and electrochemical oxidation during electrolysis, it must resist advanced chemical influences.

These requirements could be satisfied if the electrodes’ structure is built with principles of composite materials, i.e. framework made of coke grain (filler) and binding component (matrix) from coal pitch. According to the theory of composite materials, end-product – composite exceeds by properties both its constituents [4].

Nevertheless, compositional properties of the structure determine some peculiarities of anode’s work under conditions of electrolysis of cryolite-

alumina melt. The most significant peculiarities are as following:

1. Due to the different chemical properties of a coke made of fillers and a coke made of binding material, their oxidation occurs at different rates. This leads to uneven (selective) anode oxidation and shedding off some parts of the filler.

2. Uncompensated shrinkage of matrix's materials during burning causes the formation of many defects (microcracks, pores, bundles) in the structure of coke made of a binding component. This, in turn, triggers mechanical shedding of coke particles.

During the development of an anode mass recipe, one should consider including coke as a filler with a strictly regulated value of reactivity. Appropriate calculation of granulometric composition requires steps like taking into consideration dense packing of grains to prevent high porosity of anode and infiltration of oxidation products into intraporous space.

One of the main tasks of technology is to regulate purposefully the structure and properties of raw materials to reduce uncompensated shrinkage. It is possible to achieve desirable results by varying granulometric composition, filling the matrix with a fine dusty fraction, using high temperature pitch and coke with low thermal expansion coefficient values and changing technological parameters of burnt anode production.

Studying the packing of aggregate grains on geometrical models that consist of homogeneous spheres makes it possible to understand that a minimum quantity of spheres in a unit of volume is achieved when centres match cubic lattice nodes. Meanwhile, a maximum amount of spheres happens by rhombohedral packing. If empty spaces are filled with spheres with corresponding diameters, theoretically, it is feasible to obtain a filling factor infinitely close to one. Using materials of several fractions such as coarse, medium, and dust helps achieve maximum packing.

Literature review showed that granulometric composition significantly affects the quality of a produced anode. An increase of coarse fraction content leads to a decrease in electrical resistance and anode shrinkage during burning. On the other hand, an increase in dust fraction quantity improves anode blocks' mechanical strength and density. Intermediate fractions of coke charge mainly crumble during electrochemical anode oxidation and their amount is regulated [1-6].

It is possible, to sum up, a relation between different components of an anode mass recipe:

– Maximum packing (minimum porosity) is achieved in a system developed according to a relatively balanced straightforward principle, when material from the granular composition has a small size enough to ensure intergranular space. At the same time, the porosity of coarse grains has its influence [6-9].

– Gas permeability of anode material is significantly reduced as fineness and dust content are increased [10].

– Resistance to thermal shock grows if the granular composition has a high percentage of coarse fractions.

A fine fraction in an anode structure affects the "elasticity" of the charge. That property improves compression resistance [11]. For all mentioned reasons, adjustment of optimal granulometric composition significant.

### Experimental part

An object of research is a charge with different granular composition used for anode production at the Kazakhstan electrolysis plant. Table 1 represents a granulometric composition of the charge.

**Table 1** – Granulometric composition of the charge

Sample number	Granulometric composition, %			
	Coarse fraction	Medium fraction	Fine fraction	
			Grinder dust	Filter dust
1	37	40	15	8
2	25	40	25	10
3	30	30	30	10
4	25	20	50	5
5	40	20	22	18
6	40	23	22	15

Equipment used for the research: RDC-161 bench-scale anode production unit; BF12S anode baking furnace; equipment for determination of anode blocks gas permeability; pycnometer.

Six batches of anode mass with different granulometric compositions were prepared for the experiment (Table 1). The fraction classes that were selected are coarse (-14 + 6.3 mm), medium (-6.3 + 0.25 mm), fine fractions (-0.25+0.08 mm), and coke filter dust (-0.074 mm). The coarse fraction consisted of snuff; other fractions were made of coke.

*Determination of reactivity to carbon dioxide.*

Two test samples with a diameter of (50±1) mm and a length of (60±1) mm were prepared. The samples were dried at (120±5) °C within 12 hours and cooled to room temperature. The initial mass of each test samples was measured by weighing each core (m<sub>0</sub>) to an accuracy of ±0.1 g. Then, the muffle furnace was heated to (960±2) °C. The temperature was stabilized by keeping it in the furnace at least for 60 minutes; after, that, the test samples were placed inside. The gaseous flow of carbon dioxide was supplied, and the rate was set at 200 litres per hour through an alumina tube. Mentioned parameters were kept within 7 hours, and the samples were cooled by switching off the gaseous flow. As soon as the furnace temperature cooled to lower than 550 °C, remnants of the test samples (core) and produced dust were taken out, cooled to room temperature, and weighed to an accuracy of ±0.1 g. Each remaining test sample was placed in a separate camera of tripping apparatus with 50 steel balls and was left to turn over for 20 minutes to remove any loose particles. As soon as turning over finished, the camera's content was emptied into a 4 mm sieve, balls were taken out, and remnants of the samples (core) were weighed to an accuracy of ±0.1 g.

*Determination of anode density in xylene by pycnometric method.* Cylindrical specimens of anode mass 50 mm high and 30 mm in diameter were ground up to a particle size of 63 µm, dried in the furnace at 120°C ± 2°C for 8 hours, and cooled with silica gel in a drying cabinet. A dry clean pycnometer without a stopper with 5 g ± 0.1 g of that specimen was placed in a degassing container. Xylene was added drop by drop inside the pycnometer until the content was covered by it to a maximum of 20 mm. After that, the pycnometer was put in a thermostat and heated to a temperature of 25 ± 0.05 °C. As soon as all water was removed, the pycnometer reached test temperature; it was taken out, thoroughly dried and weighed. The experiment was repeated three times with every recipe.

The equation determines density ρ of burned anode expressed in grams per millilitre:

$$\rho = \frac{m_3}{V - \frac{m_4 - (m_0 + m_3)}{\rho_x}} \quad (1)$$

where V – pycnometer volume, ml;

m<sub>0</sub> – a mass of clean, dry, empty pycnometer, g;

ρ<sub>x</sub> – xylene density, g/ml;

m<sub>3</sub> – sample mass, g;

m<sub>4</sub> – a mass of pycnometer with a sample and xylene, g.

Density calculation of sample № 1:

$$\rho = \frac{24.3}{25 - \frac{58.815 - (25 + 24.3)}{1.21}} = 1.418,$$

$$\rho = \frac{23.8}{25 - \frac{58.315 - (25 + 23.8)}{1.21}} = 1.388,$$

$$\rho = \frac{25}{25 - \frac{59.515 - (25 + 25)}{1.21}} = 1.458.$$

Similarly, densities of other samples were determined.

*Determination of gas permeability.* Calculation of gas permeability of the anodes is done using data from measurements:

$$W_{RCR} = \frac{m_0 - m_1}{m_0} \times 100 \quad (2)$$

where m<sub>0</sub> – initial mass the test sample (core), g;

m<sub>1</sub> – a mass of the remaining test sample (core) after tripping, g.

Calculation of gas permeability of burnt anodes prepared with different compositions:

$$W_{RCR (P1)} = \frac{25 - 24.525}{25} \times 100 = 1.9$$

$$W_{RCR (P2)} = \frac{23 - 22.6}{23} \times 100 = 1.73$$

$$W_{RCR (P3)} = \frac{27.3 - 26.8}{27.3} \times 100 = 1.83,$$

$$W_{RCR (P4)} = \frac{21.3 - 21.133}{21.3} \times 100 = 0.78$$

$$W_{RCR (P5)} = \frac{23.23 - 22.951}{23.23} \times 100 = 1.2$$

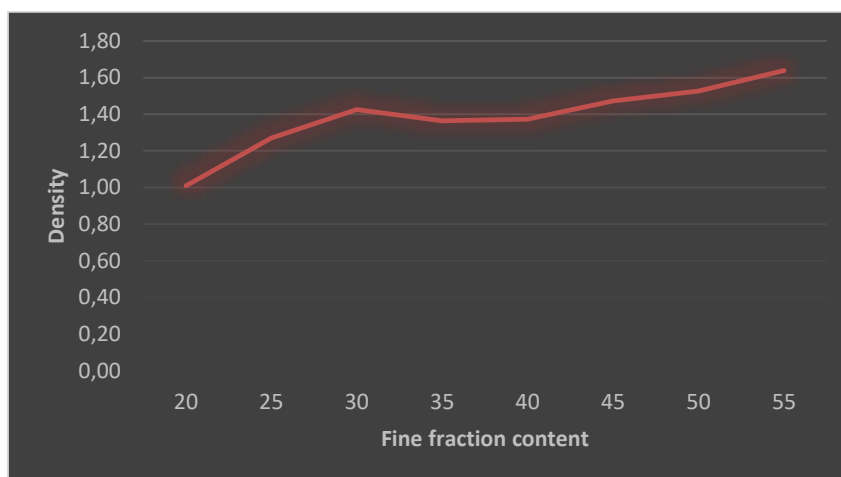
$$W_{RCR (P6)} = \frac{26.33 - 25.915}{26.33} \times 100 = 1.58.$$

## Results and discussion

The results of the experiment showed that granulometric composition affects the density of anodes significantly. It seems that an increase of

**Table 2** – Density of anodes with different granulometric compositions

Sample number	Density 1, kg/dm <sup>3</sup>	Density 2, kg/dm <sup>3</sup>	Density 3, kg/dm <sup>3</sup>	Mean value, kg/dm <sup>3</sup>
1	1.418	1.388	1.458	1.421
2	1.307	1.388	1.400	1.365
3	1.458	1.488	1.517	1.487
4	1.633	1.587	6.88	1.639
5	1.517	1.587	1.476	1.526
6	1.418	1.388	1.310	1.373

**Figure 1** – Plot relationship between anode density and fine fraction content

fine fractions contents increase density of the anode.

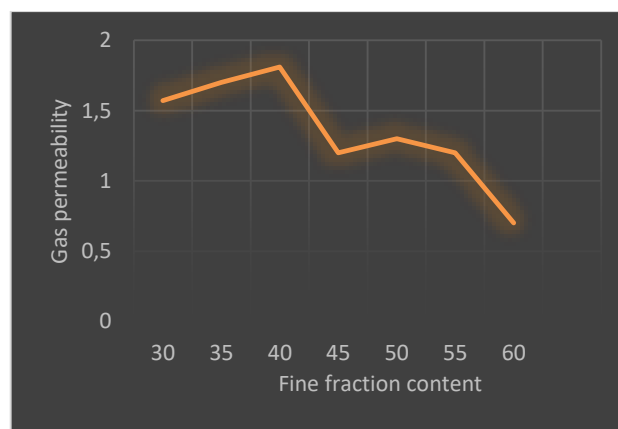
Table 2 shows the result of the experiments related to determination of anode density in xylene by pycnometric method.

Table 3 represents the obtained data from the experiment related to gas permeability of the anodes.

**Table 3** – Gas permeability of burnt anodes with different coke granular compositions

Samples	Sample mass, g	Gas permeability, nPm
Sample № 1	25	1.9
Sample № 2	23	1.73
Sample № 3	27.3	1.83
Sample № 4	21.3	0.78
Sample № 5	23.23	1.2
Sample № 6	26.33	1.58

Based on the calculated values, the plot that represents the dependence of gas permeability on the content of a fine fraction was drawn. The experiment results revealed that an increase of fine fraction content leads to a significant decrease in anode gas permeability.

**Figure 2** – Plot relationship between anode gas permeability and fine fraction content

The results showed that the highest density value has a test sample № 4 (with the highest content of fine fractions). Figures 1 and 2 represent the tendency that an increase in fine fraction content reduces anodes' gas permeability. Further increase in fine fraction content is unacceptable. According to production experience, there is an upper limit of fine fraction content above which deterioration of mechanical properties is observed. The mentioned limit is determined by fineness of applied dust as well [4-5]. High content of fine fractions and dust reduces coarse fraction

**Table 4** – Specification of burned anodes

<i>Present granulometric composition of charge</i>	<i>Density, kg/dm<sup>3</sup></i>	<i>Gas permeability, nPm</i>	<i>Suggested granulometric composition of charge</i>	<i>Density, kg/dm<sup>3</sup></i>	<i>Gas permeability, nPm</i>
Coarse fraction (-14 + 3.5 mm) - 26 %	1.542	1.01	Coarse fraction (-14 + 3.5 mm) – 25 %	1.639	0.78
Medium fraction (-3.5 + 0.25 mm) – 35 %			Medium fraction (-3.5 + 0.25 mm) – 20 %		
Fine fraction (-0.25+0.0 mm) – 39%			Fine fraction (-0.25 +0.0 mm) – 50 %		
			Filter dust (-0.074 mm <) – 5%		

content in anode frame that in turn causes weakening of cohesion between particles. As a result, anodes may undergo deformation after molding or during burning [3, 5].

Granulometric composition of charge for production of higher quality anode masses was chosen based on the research results (Table 4).

Thus, according to the proposed method for producing anode mass, adding an extra fraction of coke – filter dust caught from filters to existing fractions.

Developed recipe will increase anode density from 1,542 kg/dm<sup>3</sup> to 1,639 kg/dm<sup>3</sup> and reduce gas permeability from 1,01 to 0,78 nPm.

## Conclusions

The influence of fine fraction content on the quality of produced anodes was studied experimentally. An increase of anode density was observed as more fine fractions were added to the charge recipe. Moreover, gas permeability was reduced with the addition of fine particles. Gas permeability affects the consumption of anodes during electrolysis of cryolite-alumina melt.

A better option of granulometric composition of coke for production of higher quality anodes at JSC Kazakhstan electrolysis plant was suggested.

**Conflict of interests.** On behalf of all authors, the correspondent author declares that there is no conflict of interests.

*Cite this article as:* Akizhayeva A.S. (2021), Study of influence of the charge granulometric composition on the quality of burned anodes. *Kompleksnoe Ispol'zovanie Mineral'nogo Syr'a = Complex Use of Mineral Resources*. 2021. Volume 4, Issue 319, pp. 13-18. <https://doi.org/10.31643/2021/6445.35>

## Шихтаның гранулометриялық құрамының күйдірілген анод сапасына әсерін зерттеу

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### ТҮЙІНДЕМЕ

Алынатын анодтың сапасына көптеген факторлар әсер етеді, олар: шикізаттың сапасы, рецептура, технология, режимдік параметрлер, шихтаның гранулометриялық құрамы және т.б. Олардың ішінде шихтаның гранулометриялық құрамы ең негізгі орын алады. Бұл мақалада ұсақ фракциялардың анод сапасына әсерін зерттеу нәтижелері келтірілген. Зерттеу объектісі Қазақстан электролиз зауытындағы (Павлодар қ.) әртүрлі гранулометриялық құрамдағы шихта болды. Зерттеу жұмыстары үшін әртүрлі гранулометриялық құрамды анод массасының 6 партиясы дайындалды. Анодты массаны дайындау үшін мұнай коксі, пектер, істен шыққан анод қалдықтары және коксты ұсату кезінде пайда болған фильтрдегі кокс шаңдары мен диірмендегі ұсақ кокстер қолданылды. Зерттеулер барысында осы үлгілердің кейбір қасиеттері зерттелді. Дайындалған үлгілердің ішінен гранулометриялық құрамы келесілерден тұратын: ірі фракциясы – 25 %, орташа фракциясы – 20 %, кішігірім фракциясы (диірмендегі ұсақ кокстер) -50 % және фильтрлердегі ұсақ бөлшектер – 5 % болатын № 4 үлгіге ерекше мән берілді. Зерттеулер нәтижесінде, анод өндірісінің шикізатына кокстың кішкентай (ұсақ) бөлшектерін қосқанда, оның шығынына әсер ететін кейбір қасиеттерінің тығыздығы мен газөткізгіштігінің өзгергені байқалды. Шаң-тозаңдық фракцияны қосқан кезде,

Мақала келді: 04 мамыр 2021  
Сараптамадан өтті: 23 шілде 2021  
Қабылданды: 04 қазан 2021

анодтың тығыздығы 1,542 кг/дм<sup>3</sup> –тен 1,639 кг/дм<sup>3</sup> дейін жоғарылағаны, ал газеткізгіштігінің 1,01-ден 0,78 нРт дейін төмендегені байқалды. Сонымен қатар шикізатқа шаң-тозаңдық фракцияны қосқан кезде анод блоктарының кеуектіліктері төмендейді. Зерттеулер нәтижесі сапалы анод өндіру үшін, анод массасының оңтайлы рецептурасын таңдауға мүмкіндік береді.

**Түйін сөздер:** анодты масса, күйдірілген анод өндірісі, газеткізгіштік, тығыздық, гранулометриялық құрам, ұсақ фракция.

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## Исследование влияния гранулометрического состава шихты на качество обожженных анодов

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**АННОТАЦИЯ**

На качество получаемых анодов влияют многие факторы: состав и качество сырьевых материалов, рецептура, технология, режимные параметры, гранулометрический состав шихтовых материалов и т.д. Особое место среди них занимает гранулометрический состав шихты. В данной статье представлены результаты исследования влияния тонкой фракции на качество анодов. Объектом исследования являлась шихта различного гранулометрического состава для получения анодов на базе КЭЗ (Казахстанский электролизный завод, г.Павлодар). Для эксперимента были приготовлены 6 партий анодной массы с разным гранулометрическим составом. Для приготовления анодной массы использовались нефтяной кокс, пек, оборотные аноды и пыль с фильтров и мельницы, которые образуются при измельчении кокса. Было определено и исследовано некоторые свойства этих образцов. Из приготовленных шести проб особое внимание привлекло проба № 4, где содержание крупной фракций – 25 %, средней – 20 %, мелкой (мельничная пыль) -50 % и фильтровая пыль -5 %. Результаты показывают, что с увеличением тонины помола пыли улучшаются некоторые характеристики анода, в частности газопроницаемость и кажущаяся плотность, которые влияют на расход анодов. Так, при добавлении пылевой фракций в шихту, плотность анодов увеличивается от 1,542 до 1,639 кг/дм<sup>3</sup>, а газопроницаемость снижается от 1,01 до 0,78 нРт. Так же добавление пылевой фракций минимизирует пористость анодного блока. Результаты исследования позволяют подобрать более оптимальную рецептуру анодной массы, для получения более качественного анода.

**Ключевые слова:** анодная масса, производство обожженных анодов, газопроницаемость, плотность, гранулометрический состав, тонкая фракция.

Поступила: 04 мая 2021

Рецензирование: 23 июля 2021

Принята в печать: 04 октября 2021

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