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Comparison between Alumina Production Process from Nepheline Ore in Razgah (Iran) and Khibin & Kiya-Shaltyre Deposits (Russia)

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ABSTRACT

Razgah nepheline-bearing ore deposit is a promising source of alumina in Iran. The VAMI (Russian Aluminium-Magnesium Institute) studies on Razgah representative samples have revealed four types of ore (A, B, C and D) in the deposit. In this paper a technical comparison is made between alumina production process from nepheline ore in Razgah deposit in Iran and Khibin & Kiya-Shaltyre deposits in Russia (as the most important alumina producer from nepheline). Chemical composition, size reduction, sintering, leaching and desilication processes, carbonization, crystallization of aluminium hydroxide, and cement and carbonate production are the main processes to be discussed based on VAMI work. The results of chemical analysis revealed that Razgah samples consist of lower percentage of alumina (about 20 % versus 26.7 to 28.6 %) and more silica (52 to 56 versus 40 to 44 %) compared to Khibin and Kiya Shaltyr deposits in Russia. This difference can cause negative effects on the performance of Razgah nepheline ore processing. Due to a higher clay components content in Razgah ore types (B, C & D), a better grindability was achieved compared to the feed of Achinsk plant in Russia. The sintering temperature for Razgah ore types is 110 to 150 °C more than the temperature for Kiya Shaltyr ore at the Achinsk plant. In leaching process for the nepheline ore of type A, the recovery of values from the sinters were 6 to 8 % higher than B,C and D types. The results confirm a possibility of producing alumina, cement from obtained belite slurry, sodium carbonate, potash and potassium sulfate in the course of Razgah ore processing.

Key words: Nepheline syenite, Alumina, Razgah, Sintering, Leaching, Desilication, Carbonization, Aluminium hydroxide, Cement.

INTRODUCTION

Nepheline syenite is a light-colored feldspathic rock that is used primarily as an addition to the glass batch. It is also used in ceramics and as industrial filler. Composed essentially of the soda and potash feldspars and nepheline, nepheline syenite contributes needed alkalis and alumina to the glass composition. Because it contains a higher proportion of these oxides per unit includes presentation of the position and condition

needed alkalis and alumina to the glass composition. Because it contains a higher proportion of these oxides per unit weight, it is a formidable competitor for feldspar in North America and parts of Western Europe. Nepheline syenite is produced for glass, ceramics, and filler markets by Unimin Canada Limited at Blue Mountain, in south-eastern Ontario [1]. Nepheline has also been a by-product of apatite mining on the Kola Peninsula for many years. In 1951 the production of alumina from nepheline was

commenced in a plant near St. Petersburg (Leningrad). There are now four plants in the Russian Republic, two smaller ones based on nepheline tailings from apatite mining and two larger and more recent plants in central Siberia using other nepheline resources. All four of these plants are integrated complexes that produce alumina for the subsequent production of aluminium metal, sodium and potassium carbonates, and Portland cement. Plants at Volkhov, Pikalevo, and Achinsk are together believed to consume more than 3 Mt of nepheline rock annually [1].

The Razgah nepheline-bearing ore deposit is a different degree pseudoleucitic syenite with a ocellar structure, is mainly formed by ovoidal pieces of fine grains of leucocratic minerals. The deposit is located about 25 km away from Sarab city, near of Razgah village, in Azerbaijan province of Iran. The first exploration studies on ore deposit were carried out from 1977 to 1981 by mines and metals ministry. In 1988, Iranian alumina production and UNIDO Russian experts were performed exploration studies in Razgah deposit. On duration of these studies, a technological sample was prepared from trenches and sent to VAMI Institute (in Russia) for technological studies. Four main types of ore (A, B, C and D) were identified on the basis of the analysis of these samples. The least degree of alteration was observed on the type A that is the first in occurrence and commercial value. Types B&C were distinguished by a higher content of K_2O and lower content of Na_2O respectively as compared with the type A. In type D, the CaO content was increased more than 3.5 times of the average content in the ores A and B types; The Fe_2O_3 increased and the A1203 dropped sharply.

The Shiramin limestone deposit is located in the 60 km south-west of Tabriz and about 2.5 km north of the village of Shiramin. On duration of VAMI studies in Razgah Nepheline deposit, the Shiramin limestone was used as a carbonate raw material in production of alumina and cement. About 400 samples have been collected from the trenches and were used in technological studies.

In this paper according to the VAMI studies in bench scale, a comparison was made between alumina production process from Razgah nepheline ore of Iran and Russian nepheline deposits (as the biggest producer of alumina from nepheline). Size reduction, sintering, leaching and desilication processes, crystallization of aluminium hydroxide, cement and carbonate production are the main processes that are discussed.

Finally the recent activities that are currently in progress on Razgah nepheline deposit by alumina production project and Kanikavan Company as consultant are presented.

EXPERIMENTAL

Sampling Procedure

A sampling procedure was developed by a team of Soviet and Iranian experts based on a study of the

results of the geological exploration. The characteristics samples (R-A, R-B, R-C and R-D) were collected from the main trenches II, III and IV through the most promising section of Razgah deposit. Two series of samples first -3 mm and second -0.1 mm were prepared as characteristics and analysis samples respectively. The final blending of samples was performed by VAMI experts. These representative samples were used as a basic material for technological studies.

The limestone samples were prepared from lengths of intervals formed mainly by pure limestone corresponding by their average quality to requirements for carbonate raw material for alumina production ($SiO_2 < 2\%$, $MgO < 1\%$, $Fe_2O_3 < 0.6\%$, $CaO > 53\%$).

Pre Concentration Studies

Preliminary studies for increase quality of the ore was used to determine possibility of extraction greater amount of reserve, reduce the expenses of selective mining, increase content of useful materials, decrease of harmful content and stabilizing the quality of raw material and subsequently optimize of mining and processing performances. For achieving these aims, wet magnetic separation was used with different strength of magnetic fields.

Size Reduction Studies

The aim of size reduction studies was determining the grindability of the characterize samples of Razgah nepheline ore and Shiramin limestone and selection the capacity of industrial mills. To achieving this aim the grinding kinetics of Razgah samples was studied in bench scale and in relation to the Achinsk alumina plant grinding system (Russia) as reference. The Kiya Shaltyr nepheline ore and Mazulsky limestone are feed components for Achinsk alumina production plant.

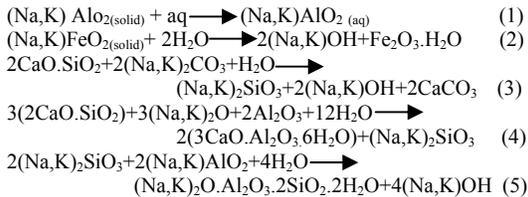
Sintering

For production of alumina, sodium and potassium carbonates and cement a nepheline-limestone feed thermally treated at a temperature close to a melting point. The product is a porous aggregate that ground and leached with alkaline liquor for extraction of valuable materials. The temperature of sintering is an important factor and determines the sinter porosity. Increasing of sinter temperature is accompanied by a considerable partial melting and reduction porosity. Subsequently the recovery of values through leaching is reduced. When the sinter temperature decreases (porosity increase), it causes lower recovery of the values as a degree decreases of reaction rates in interaction of the feed components. Determination of the main sintering process parameters (feed component content, feed size distribution, sintering temperature) and evaluation the quality of sinter produced, were one of the aims of bench scale technological tests in Razgah samples. Three different

sizes (a) normal size: content 5% of +0.08 mm fraction and 85 % of -0.05 mm (b) coarse size: content 10 % of +0.08 mm and 70 % of -0.05 mm (c) fine size: content 1.5 % of +0.08 mm and 90 % of -0.05 mm were used in sintering studies. Normal, coarse and fine sizes were mixed with limestone containing 8 % of +0.08 mm, 15% of +0.08 mm and 3% of +0.08 mm respectively. For selecting an optimum proportion of components the feeds with molar ratios of CaO/SiO₂ equal to 2, 1.97, 1.94 and the molar ration of R₂O/Al₂O₃ equal to 1.03, 1.08 and 1.13 were used on sintering experiments. Sintering the feed in laboratory scale was carried out in a temperature range ensuring the production of sinters corresponding by their physical characteristics to the industrial material.

Leaching Process

The main task of sinter leaching is separation of soluble components from sinter to leaching liquor for production of alumina, carbonates and belite slurry (for cement production) as follows:



Leaching studies included the following sections:

- Determination of optimum leaching parameters (temperature, solid percentage, optimum recovery of aluminium oxide and alkalis)
- Study the effect of raw material mineralogical composition in recovery efficiency of values
- Determination of size distribution of the belite slurry and its settling rate

For studying the above parameters sinters were prepared on basis of four types of ore (types A, B, C&D) and were used in leaching studies.

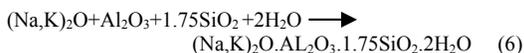
Desilication of Aluminate Liquor

In desilication stage, the silica removes from the aluminate liquor and subsequently amount of silica decrease in final product that is strictly specified by the alumina smelters. The high quality grades of alumina containing not more than 0.02% silica.

The scheme includes two operations:

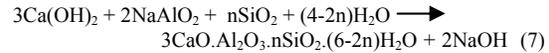
- Autoclave desilication
- Deep desilication

The first operation is based on the removing of the main quantity of silica into sodium (potassium) hydroalumosilicates that is poorly soluble in aluminate liquors according to the following reaction:



The operation is normally run at high temperature (145 to 155°C). After the first desilication, the slurry is

thickened and the alumina liquor feed to the second stage (deep desilication). In the second stage the remain silica removes in the form of hydroalumosilicates according to the following reaction:



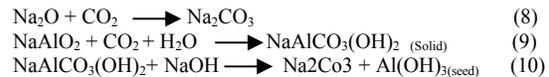
The hydroalumosilicates produced in the first and second stages of desilication is repulped by a sodium carbonate liquor and send to the first of process for recovery of alumina. The liquor is feed to carbonization and precipitation.

Carbonization, Crystallization & Calcination of Aluminium Hydroxide

After the first desilication stage, the liquor divides in two portions. One portion feed to the precipitation of aluminium hydroxide and the other to the second (deep) desilication. After deep desilication, the liquor goes to the tow stage carbonization. The main task of carbonization is:

- Precipitation of aluminium hydroxide seed (at the first stage of carbonization)
- Production of sodium carbonate liquor for separation of sodium carbonate, potash, potassium sulphate and chloride by the poly thermal decomposition method

The carbonization is based on a set of chemical interaction that takes place when passing the CO₂ through the liquor. The sintering kilns off-gases (after dedusting) use in carbonization operation. In the first stage carbonization:



In the second stage:



From the first stage, the seed of Al(OH)₃ feed to the precipitation stage. In the precipitation line, the liquor cool in a vacuum cooler down to the 60-70°C and mix with the aluminium hydroxide seed and held in a precipitator with air agitation. The seed grow and the final aluminium hydroxide precipitates. The liquor is return to the sinter leaching operation and the aluminium hydroxide to the calcination to production alumina.

RESULTS AND DISCUSSION

Chemical Characteristics

The chemical analysis of characteristics samples of Razgah deposit and the nepheline ore are processing currently in Russia are presented in Table 1.

From the Table 1, it can be seen that Razgah samples are consists of a lower percentage of alumina (about

20 % against 26.7 - 28.6 %) in comparison of Khibin and Kiya Shaltyr deposits. Also the amount of silica is more (52-56 % against 40 - 44 %) and relatively small amount of calcium oxide against of Kiya Shaltyr deposit. The said differences can affect the performances of the processing. Alkalis and alumina (with positive effect) and silica (with negative effect) are the most important parameters effect the process. The harmful iron and magnesium oxides percent are meeting the requirements of the VAMI sintering technology to nepheline raw material processing without previous beneficiation. The Razgah R-D sample is marked with increase of iron and magnesium oxides and decrease of alumina percent. The content of sulfur and chlorine that negatively affecting the quality of soda products is lower than Kiya Shaltyr ore.

The nepheline quality depends not only to the content of useful (Al_2O_3 , Na_2O , K_2O and CaO) and harmful (Fe_2O_3 , MgO , TiO_2 , SiO_2 , S , and Cl) components it is also in related to the following molecular relations:

$$\begin{aligned} \text{Alkaline Module } (M_R) &= (\text{Na}_2\text{O} + 0.66 \text{K}_2\text{O}) / \text{Al}_2\text{O}_3 * 1.645 & (1) \\ \text{Calcium Module } (M_{Ca}) &= (\text{CaO} / \text{SiO}_2) * 1.07 & (2) \\ \text{Silica Module } (M_{Si}) &= (\text{SiO}_2 / \text{Al}_2\text{O}_3) * 1.7 & (3) \end{aligned}$$

The above parameters for the referred samples are shown in Table 2. For the best quality ore, the alkaline module has to be near to 1. It causes low alkaline liquors are used as recycled returned into the beginning of the process for feed charge preparation. In Razgah samples, type A has the best alkaline ratio and is better than Kiya Shaltyr ores but inferior the kola nepheline concentrate. The Types B, C and D have a lower alkaline ratio and subsequently lower quality in alumina production. In studied samples, the calcium modules (determines the amount of limestone uses with nepheline in the process) are close to the Kola concentrate and inferior to the Kiya Shaltyr sample. This parameter is high for Type D sample, its only positive character in production of alumina. As shown in Table 2, the silica module which is in inverse relation to the ore quality in alumina production and also determines the amount of belite mud, is about 1.8 times than the Kola and Kiya-Shaltyr samples.

Beneficiation Results

The study of main types of Razgah ores shows that they have hard dressing properties as low leach ability due to close intergrowth of minerals, alteration of coloured minerals that decrease the possibility of their separation from nepheline and presence of montmorillonite in types B, C and D.

Magnetic separation, Flotation, Combined magnetic-flotation methods can be used for beneficiation of nepheline ores.

The results of high magnetic separation are shown in Table3. It can be seen that the amount of Al_2O_3 in concentrate (electromagnetic + non magnetic fraction) produced with high magnetic separation not changed

significantly in comparison to initial ore (from 20 to 20.4 %). The SiO_2 increased (54.3 to 56%) and Fe_2O_3 decreased (3.7 to 1.6 %) as a result of high magnetic separation. The results are shown that this preconcentration not only have additional expenses but also reduction of concentrate yield. So it is necessity to have sintering at higher temperature and higher consumption fuel (due to presence of iron) instead of preconcentration.

By the amount of harmful iron and magnesium oxide the investigated types of rocks meet the requirements of the VAMI sintering technology to the nepheline raw material processed without previous beneficiation as Russian nepheline ores.

Crushing and Grinding Results

The material was crushed first to -20 mm by jaw crusher and then to -3 mm by roll crusher. Wet ball mill was used for more grinding and size reduction. In wet grinding of raw materials, in related to the solids content in the slurry, the slurry can be in three state: (a) no plastic state (the material is not movable and adhere to the mill walls), (b) yielding one (the slurry is moveable but dose not flow) (c) liquid-flowable one (the slurry can flow spontaneously). The yielding state produces the effective grinding. For Achinsk plant, in solid content of 76 to 77 % the slurry is in yielding state. For Iranian samples, the reological properties of R-A characteristic sample and Shiramin limestone is the same to the properties of the raw materials processed in the Achinsk plant. Then solid percentages of 75% were used for grindability tests of type A sample. For samples R-B, R-C and R-D, the presence of clay minerals results in reduction of solid content in slurry (to 64 %) to achieving a yielding state. The results are shown that type A is by 24 % higher grindability than Kiya Shaltyr nepheline ore and the Shiramin limestone features the same grindability as the Mazulski limestone used in Achinsk.

Sintering

On the basis of optimum alumina recovery in leaching process, the optimum feed size distribution for sintering was 5%: +0.08 mm for nepheline and 8% +0.08 mm for limestone. The molar ratios were optimized in $(R_2O / Al_2O_3) = 1.08$ and $(CaO / SiO_2) = 2$. The sintering temperature of the Razgah samples (R-A, R-B, R-C and R-D) was 110 to 150°C more than the Kiya Shaltyr nepheline were used in Achinsk plant and Subsequently for Razgah samples the amount of fuel consumption increased, the capacity of kilns and the porosity of the sinter decreased in contrast to Kiya Shaltyr nepheline sample. The recovery of Alumina (Al_2O_3) from the sinter prepared from type A is by 6 to 8 % higher than the sinters prepared from the B, C and D types.

Leaching

The investigation results of the leaching in a hydro chemical treatment of the sinters (grinded for 5 minutes) are shown in Table 4. The sieve analysis of muds (sinters residue after leaching) for R-A, R-B, R-C, R-D & Achinsk plant sinters are shown that the percent of minus 80 micron for Iranian muds is higher than the Achinsk plant mud (54 to 65 for Iranian mud versus 34 for Achinsk).

The recovery of Al_2O_3 from sinter to leaching solution for Iranian R-A sample(82.8%) is higher than Achinsk plant(78.3 %) and for the other samples (R-B, R-C & R-D) is lower. For all of Iranian samples the recovery of Na_2O and K_2O is lower and higher (respectively) than Russian Achinsk plant sample.

Between different types of Razgah samples, the highest quality was achieved for the type A nepheline ore which gives recovery of the Al_2O_3 from the sinter by 6 to 8 % higher than B, C & D types.

In processing of average composition ore, characterized by content of type A, B& C as 90.3, 7.9& 1.8 % respectively, the recovery of values (Al_2O_3 , Na_2O , K_2O) from the sinter was 81.1,70 & 81.85 % respectively.

Desilication of Aluminate Liquor

The first desilication was carried out in 75 ml laboratory autoclave. The study includes optimization of process temperature and time. The optimized temperature was achieved 180 °C for 3 hours in the presence of a recycle seed of a autoclave mud with content of 16 gr/lit. In this case the ratio of (Al_2O_3/SiO_2) in the liquor reached to 380 units. In the second stage of desilication, the temperature of 90°C for 4 hour in the presence of lime milk (8gr/lit CaO) was determined as the optimized condition. In this stage the (Al_2O_3/SiO_2) ratio reach to 2400 units which is enough for producing a high quality alumina (low silica content). In Russian plants after second stage of desilication the (Al_2O_3/SiO_2) ratio reaches to 1600 to 5000.

Carbonization, Crystallization & Calcination

In carbonization, the process parameters for the Razgah alumina solution are:

- CO_2 concentration in gas fed to the carbonization is 14% by volume
- Carbonization time in the first stage is 6 hours and in second stage 2 hours
- Gas utilization factor is 65 %

In the precipitation stage, the temperature of liquor down to the 60-70°C and then mix with the aluminium hydroxide seed and held in a precipitator with air agitation for 14 hours. The precipitate thickens and filters and is washed to produce aluminium hydroxide. The product calcines in a fluid bed kiln and the Al_2O_3 produce.

Production of Byproducts

The results of laboratory studies shown that there is the possibility of producing of cement & carbonates from Razgah ore that can increase the economical conditions of the project.

CURRENT ACTIVITIES

At present, Kanikavan Shargh Company as consultant contributes in the following projects:

- New technological(bench) tests with the using of bore hole samples of Razgah deposits and Arashtenab limestone (new limestone deposit)
- Material Energy Balance
- Industrial equipment updating
- Detail exploration studies of Razgah and B,C& D blocks of Arashtenab deposits
- Infrastructure studies of alumina, cement and carbonates complex plant
- Plant Environmental impacts assessment
- Feasibility study updating of project

CONCLUSIONS

- 1- The chemical analysis are shown that Razgah samples are consists of lower amount of alumina (about 20 % against 26.7 - 28.6 %) in comparison to Khibin and Kiya Shaltyr deposits. The amount of silica is more than Kiya Shaltyr deposit (52-56 % against 40 - 44 %). The said differences can affect the performances of the processing.
- 2- High magnetic preconcentration for removal of iron and increasing of alumina content in run of mine Razgah nepheline ore, shown that this preconcentration cusses not only additional expenses but also reduction of concentrate yield. So it is necessity to have sintering at higher temperature and higher consumption fuel (due to presence of iron) instead of preconcentration.
- 3- For Iranian samples, the reological properties of R-A characteristic sample and Shiramin limestone is the same to the properties of the raw materials processed in the Achinsk plant in Russia. The results are shown that type A is by 24 % higher grindability than Kiya Shaltyr nepheline ore and the Shiramin limestone features the same grindability as the Mazulski limestone used in Achinsk.
- 4- The sintering temperature of the Razgah samples (R-A,R-B,R-C and R-D) was 110 to 150°C more than the Kiya Shaltyr nepheline were used in Achinsk plant. Subsequently for Razgah samples the amount of fuel consumption increased, the capacity of kilns and the porosity of the sinter decreased in contrast to Kiya Shaltyr nepheline sample. The recovery of Alumina (Al_2O_3) from the sinter prepared from type A is by 6 to 8 % higher than the sinters prepared from the B, C and D types.
- 5- The recovery of Al_2O_3 from sinter to leaching solution for Iranian R-A sample(82.8%) is higher

than Achinsk plant(78.3 %) and for the other samples (R-B, R-C & R-D) is lower. For all of Iranian samples the recovery of Na₂O and K₂O is lower and higher (respectively) than Russian Achinsk plant sample.

- 6- For Razgah sample after two stage desilication the (Al₂O₃/SiO₂) ratio reaches to 2400 unit which is enough for producing a high quality alumina(low silica content). In Russian plants after second stage of desilication the (Al₂O₃/SiO₂) ratio reaches to 1600 to 5000.
- 7- In optimized conditions of precipitation of alumina are: The temperature of liquor 60-70°C, residence time in a precipitator with air agitation 14 hours.
- 8- The bench scale studies shown that there is the possibility of producing of cement & carbonates from Razgah ore that can increase the economical conditions of the project.

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Table 1. Chemical composition of Razgah characteristics samples and Russian nepheline ores

Situation	Deposit	Sample	Chemical Composition (% wt)									
			Al ₂ O ₃	K ₂ O	Na ₂ O	R ₂ O	CaO	MgO	Fe ₂ O ₃	SiO ₂	S	Cl
Iran	Razgah	R-A	20.2	10.3	3.6	10.4	2.4	0.75	3.6	54.7	0.02	0.0075
		R-B	19.9	10.7	1.1	8.2	1.4	0.42	3.9	56.2	0.009	0.006
		R-C	20.2	10.2	1.7	8.4	2.4	0.6	3.8	54.8	0.009	0.004
		R-D	16.6	7.4	1.3	6.2	7.2	0.88	4.4	51.7	0.02	0.002
Russia	Khibin (Kola)	<i>concentrate</i>	28.6	7.6	12.8	17.8	1.3	0.4	3.5	44.0	0.024	0.03
	Kiya-Shaltyr (Achinsk plant)	ore	26.7	2.8	11	12.9	7.9	1.4	4.5	40.2	0.14	0.1

Table 2. Quality indices of Razgah characteristics samples and Russian nepheline ores

Deposit	Samples	Modules		
		Msi	MR2O	Mca
Razgah	R-A	4.6	0.85	0.05
	R-B	4.8	0.68	0.03
	R-C	4.6	0.69	0.05
	R-D	5.3	0.61	0.15
Khibin (Kola)	Conc.	2.61	1.02	0.03
Kiya-Shaltyr	Ore	2.56	0.79	0.21

Table 3. The results of high magnetic separation for Type A sample of Razgah ore

Products	Yield (%)	Chemical Composition (%)								Yield (% wt. of initial)					
		Al ₂ O ₃	K ₂ O	Na ₂ O	CaO	MgO	SiO ₂	TiO ₂	Fe ₂ O ₃	Al ₂ O ₃	K ₂ O	Na ₂ O	CaO	Fe ₂ O ₃	SiO ₂
Initial Ore	100	20	10	3.6	2.2	0.9	54.3	0.63	3.7	100	100	100	100	100	100
High Magnetic	4	8.2	3.1	1.5	1.4	2.2	21.9	7.2	55.5	1.6	1.2	1.7	2.6	60	1.6
Electromagnetic	17.5	18.7	9.3	3.6	3.7	2	54	1	4.9	16.4	16.3	17.6	29.5	23.1	17.4
Non magnetic	78.5	20.9	10.5	3.7	1.9	0.6	56	0.18	0.8	82.0	82.5	80.7	67.9	16.9	81
Nepheline concentrate*	95	20.4	10.4	3.7	2.2	0.6	56	0.27	1.6	96.9	98.8	97.6	95	41.1	98

* Nepheline concentrate = Electromagnetic + Nepheline concentrate

Table 4. Investigation results of the sinters leaching

Sinter	Sieve Analysis of mud (micron)			Recovery from sinter (%)		
	+1000	-1000+80	-80	Al ₂ O ₃	Na ₂ O	K ₂ O
R-A	15.36	24.08	60.56	82.8	74.1	91.2
R-B	16.81	25.11	58.08	77.8	72.6	87.3
R-C	13.06	21.12	65.82	77.5	73.9	90.1
R-D	18.14	27.61	54.25	75	77.1	87.7
Achinsk Plant Sinter obtained under laboratory condition	32	33.6	34.4	78.3	85	82.9