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DRY SINTERING OF NEPHELINE - A NEW MORE ENERGY EFFICIENT TECHNOLOGY

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Abstract

A new more energy efficient technology for dry sintering of nepheline and limestone has been developed in collaboration between Pikalevo Soda, FLSmidth and the Russian National Mineral Resources University. The new technology is a result of laboratory research activities and semi-industrial tests conducted by FLSmidth in Denmark and USA.

The technology tested semi-industrially consists of a 3-stage preheater with calciner and rotary kiln. This type of equipment has already been in operation in the cement industry for more than 40 years.

The tests proved a success due to the ability to control feed chemistry and particle size distribution as well as temperature profile and retention time in kiln system. Control of the above parameters is the basis for achieving high quality sintering and required alumina extraction.

This paper presents the results of the semi-industrial tests and compares it to the existing wet technology applied at the Pikalevo site.

Introduction

Up to today sintering of nepheline and limestone has been done by the means of wet technology at several sites in Russia; Achinsk, Pikalevo and in earlier days also Volkhov. In spite of the fact that nepheline contains substantially less alumina than bauxite, very good utilization of the chemical components is obtained with the complex process applied at both the Achinsk and Pikalevo site. Table I shows the chemical composition of Russian nephelines and average bauxite. Nonetheless with regards to energy efficiency the wet technology is today unable to compete with newer technology.

Table I. Chemical composition of nephelines and bauxite

	Al_2O_3	SiO_2	Fe_2O_3	CaO	Na_2O	K_2O
Nepheline ore (Achinsk)	26.5	40	4.8	7.9	11.8	2.7
Nepheline concentrate (Pikalevo)	28.3	44	3.5	1.5	12.8	7.6
Bauxite, average	55	4	20	2	-	-

The utilization of the components in the complex processing of nepheline is illustrated in the following figure 1. The figure shows the inputs to the process and the approximate distribution of outputs. As alumina is the focus of this paper this is highlighted.

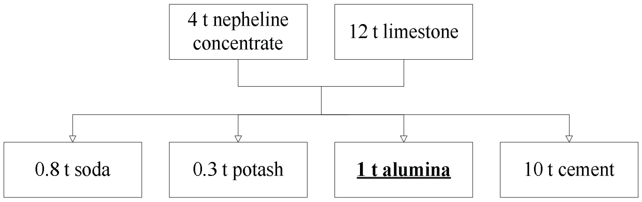

Figure 1. Inputs and outputs of the complex processing of nepheline.

The limestone used for the sintering process is approximately half of the 12 tons while the other half is used in the further processing of belite (dicalcium silicate, Ca_2SiO_4, C_2S) slurry to produce cement.

Laboratory research and semi-industrial testing has been the keystone in the effort to develop a dry technology for nepheline sintering. The result of this test work is that the dry technology is a valid replacement for the wet technology as it was the case in the cement industry many years ago.

In this paper the dry technology solutions as well as process parameters for the semi-industrial testing will be presented and compared to the wet technology applied at the Pikalevo site.

Processing of nepheline to alumina

The processing of nepheline to alumina contains many sub-processes. The sintering of nepheline and limestone is the first part of the production of alumina. In figure 2 the flow sheet for the complex processing of nepheline is shown. The processes that this paper deals with are marked with bold in the figure. In addition to this, the sintering process is also responsible for generating the CO_2, which is needed for carbonisation.

The sintering process itself produces alkali aluminates (sodium aluminate, $NaAlO_2$ and potassium aluminate, $KAlO_2$) and belite. The reaction is:

$$4CaCO_3 + (Na, K)_2O \cdot Al_2O_3 \cdot 2SiO_2 \rightarrow 2(Na, K)AlO_2 + 2Ca_2SiO_4 + 4CO_2 \quad (1)$$

The final sintering takes place in a rotary kiln at a burning zone temperature of 1250-1325°C. There is a very narrow temperature range where the final sintering takes place and attention to the amount of liquid phase is crucial as this is responsible for the final reactions and structure of the sinter.

Preceding the burning zone is a preheating and calcining zone. In the preheating zone the material is heated up and the moisture in the raw mix is removed. In the calcining zone the most important reaction is the decomposition of calcium carbonate.

$$CaCO_3 \rightarrow CaO + CO_2 \qquad (2)$$

This reaction is also central in cement production and is one of the reasons for the good synergies that are present when comparing the two processes. It is to a great extent the same equipment applied and thereby process experiences from cement production can be used in the nepheline sintering process.

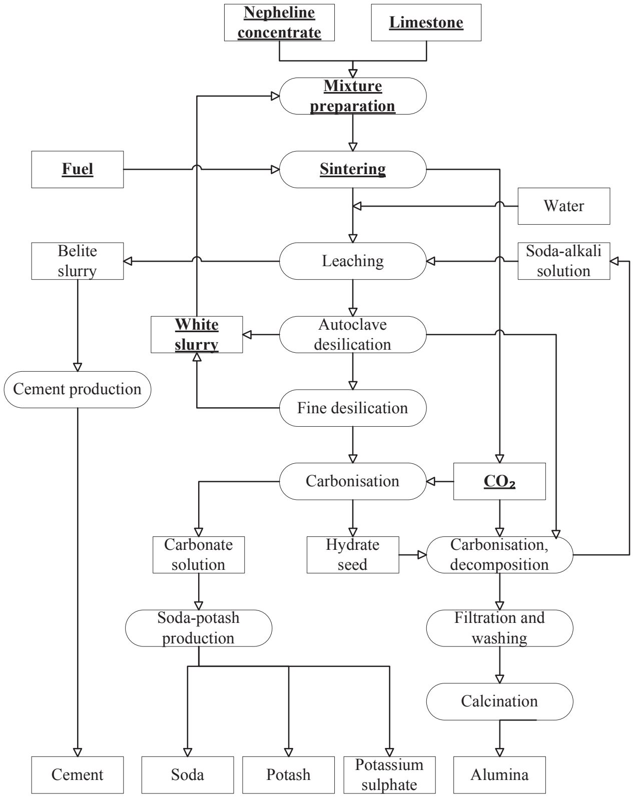

Figure 2. Flow sheet showing the complex processing of nepheline.

Current wet sintering technology at Pikalevo

At the Pikalevo site the raw mix preparations are made up of two steps. First limestone is ground separately with white slurry in an open circuit system followed by co-grinding with nepheline in the last step also in an open circuit system.

White slurry is a waste product from the desilication, but as it contains residual alumina it is re-introduced to the process to minimize waste products and achieve as close to 100% utilization of the chemical components as possible. An example of the chemical composition of white slurry is shown in table II.

Table II. Example of the chemical composition of white slurry

	Al_2O_3	SiO_2	Fe_2O_3	CaO	Na_2O	K_2O
White slurry	21.5	10	0.5	31.5	3	6.5

The particle size distribution of the raw mix feed, determined as the residue on a 90µm Alpine sieve is 15.1%. Alpine sieving has traditionally been used in the cement industry as a process parameter and important when comparing a fineness of a raw mix.

The nepheline and limestone is a homogeneous raw mix, as a consequence of the co-grinding. Once ground, the mix is stored in basins and final adjustments with soda are done in order to obtain the right chemistry. The modules that Pikalevo target in the raw mix feed are:

Alkali module:	1.03 ± 0.02
Lime module:	1.97 ± 0.03

Figure 4 shows a simplified flow sheet of the Pikalevo wet sintering system, where all reactions take place in the rotary kiln, limiting process control.

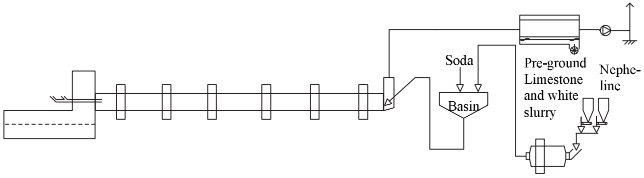

Figure 3: Pikalevo; wet sintering of nepheline.

The temperature profile of the rotary kiln is:

Preheating zone:	220-1000°C gas temperature
Calcining zone:	1000-1200°C gas temperature
Burning zone:	1200-1400°C gas temperature

The solid material retention time in the kiln is 2½-3 hours, resulting in an alumina extraction of 87-89% in the sinter. The heat consumption is approximately 1290 kcal/kg sinter.

As mentioned earlier the process was in earlier days also applied at the Volkhov site, using the same raw materials as at the Pikalevo site. Approximately 40 years ago the dry process technology (long dry kiln) was attempted at the Volkhov site, without success. The assumption is that the challenging part was the raw mix homogenization and chemistry control. As a wet process is not continuous, but rather a bulk type of operation it is simpler to operate and homogenize as well as adjust chemistry in the basins. However in the years that have passed since the attempt the dry process technology with regards to homogenization and chemistry control has improved dramatically.

Dry technology experience from the cement industry

The experiences from cement production that are applicable to the nepheline and limestone sintering cover the whole dry cement technology flow sheet – from raw material crushing to product cooling. An example is illustrated in figure 4 together with a picture of a cement plant in figure 5.

In dry cement production the chemical control of the raw materials is sustained by bulk material analyzers and homogenizing storages. Further into the process after raw mix grinding and drying it can be supported once again by online x-ray analyzers and homogenizing silos. The result is a chemically known and well homogenized raw mix feed ready for preheating.

The preheater set-up with cyclones, calciner and the associated instrumentation together with the control system provides the means for monitoring of every stage and controlling the process with regards to temperature and pressure. As the material in the preheater is suspended in the airflow every particle is preheated and calcined. The degree of calcination is directly related to the calciner temperature and therefore a very important control parameter. The result is a well homogenized and calcined material ready for sintering.

The outcome is an improved process control when comparing to the wet technology.

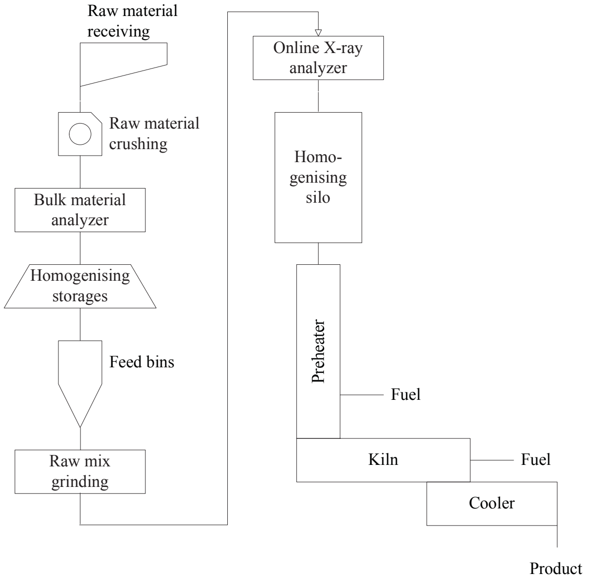

Figure 4. Simplified flow sheet example of dry technology from the cement industry.

Figure 5. Picture of a cement plant with preheater and calciner kiln system.

Additionally the dry technology also allows for larger production units, meaning that the production of one preheater calciner kiln system can produce the same as e.g. three or more wet kilns. Today in the cement industry units designed for 12,000 tpd is a possibility.

The main driver for conversion to dry technology in the cement industry has been the better energy efficiency. The heat consumption is reduced 50% compared to a wet cement production.

Dry technology laboratory research

Laboratory research was conducted as preparation to the semi-industrial test and with the main purpose to provide a basis for raw mix feed design and initial process parameter settings with regards to retention time and burning zone temperature. Additionally the testing will also be the basis for the final industrial system design together with the experiences from the semi-industrial test.

A series of tests were conducted as well as burning tests on a raw mix feed sample from Pikalevo. The results are illustrated in the graphs below. The chemistry of the sample was analyzed to be:

Alkali module: 1.00
Lime module: 1.96

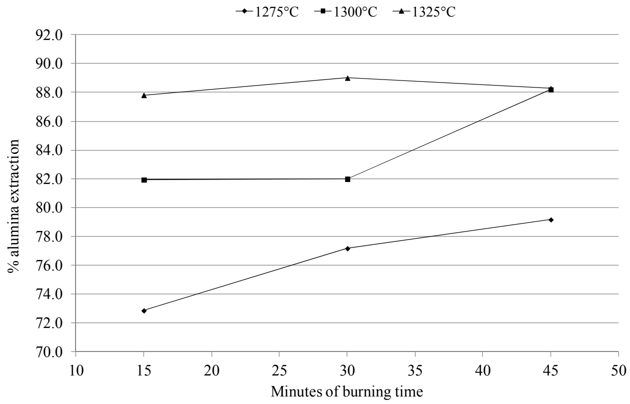

Figure 6. Burning of raw mix sample from Pikalevo at different temperature and time.

The result of the burning tests with the raw mix sample from Pikalevo, illustrated in figure 6, shows that the best conditions were a burning temperature of 1325°C with a duration of 30 minutes. All further laboratory burning tests were carried out under these conditions.

Figure 7 illustrates the results of the burning test where separate dry grinding of nepheline and limestone was applied. Nepheline was ground in a ball mill and limestone in a vertical mill. The chemical modules targeted were the same as for Pikalevo.

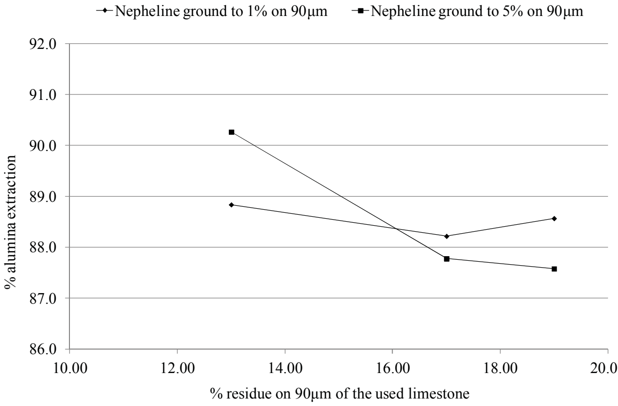

Figure 7. Burning of raw mix samples with different particle size distribution.

The separate grinding gives better control of the particle size distribution as nepheline is twice as hard as limestone. The raw mix feed preparation is essential and two conditions are of major importance:

1. The particle size distribution of ground nepheline and ground limestone is similar so the reactions particle to particle have optimal conditions.
2. The raw mix feed is homogeneous.

The separate grinding is used to ensure similar particle size distribution for the two components. Separate grinding is also applied at Achinsk and was also at Volkhov in earlier days when nepheline sintering was still in operation.

The data illustrated in figure 7 shows no significant increase in alumina extraction when grinding nepheline finer. Nevertheless there is a significant increase when grinding limestone finer. The tendency for product quality to increase with finer grinding of the raw mix is also seen in the cement industry. In the end it becomes a compromise between the electrical consumption versus the increased product quality.

Based on the laboratory research results the decision was to proceed with nepheline ground to 5% on 90μm and limestone ground to 13% on 90μm, measured by Alpine sieving. This meant that this was the particle size distribution to aim for in the semi-industrial testing.

The tests especially showed that an emphasis on the raw mix feed preparation and homogenization is vital to the success of the future work to be conducted.

Semi-industrial test of dry technology

A semi-industrial test was conducted in the beginning of 2013 with the main objective to demonstrate that the sintering of nepheline and limestone can be accomplished in a dry process utilising cyclone preheater, calciner and rotary kiln.

The key success factors were:

1. Demonstrate the continuous operation in the kiln system.
2. Produce a sinter with the required extraction of alumina.
3. Collect data for optimization with regards to the final industrial system design.

Separate grinding test set-up and raw mix preparation

The vertical mill test carried out in the laboratory proved it difficult to grind the rather soft limestone from Pikalevo. Consequently the decision was to grind the limestone in a drier crusher, which is a hammer mill type crusher with the advantage of handling very moist and soft raw materials. This proved to be the perfect match.

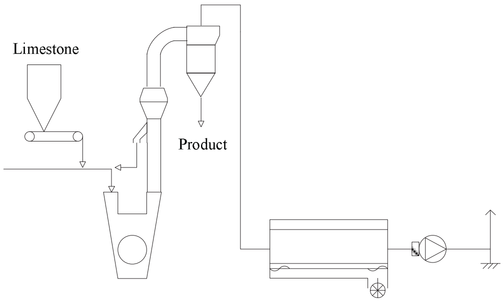

Figure 8. Drier crusher test set-up.

In figure 8 an outline of the closed circuit test set-up can be seen.

The closed circuit ball mill system is excellent for nepheline grinding as the nepheline is dry and fine. The grinding took place in a system as illustrated in figure 9.

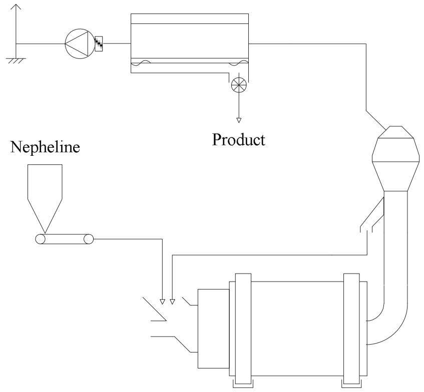

Figure 9. Ball mill test set-up.

Subsequent to the grinding, the chemical analysis of the ground materials was determined and the final raw mix comprising of the four components; nepheline, limestone, dry white slurry and dry soda, was prepared by pneumatic blending. The blending took place in a fluidized bed, where local high velocity zones are induced to activate mixing and create a homogenized mix. The modules targeted for the raw mix were the same as at Pikalevo. This is similar to the process in a homogenization silo for an industrial operated system.

The particle size distribution of the raw mix feed measured as the residue on a 90μm Alpine sieve was 8.7%. The particle size distribution determined by laser diffraction is very similar to the one used at Pikalevo, while the 90μm Alpine residue is somewhat lower. As a result, seen from the laser diffraction, the closed circuit grinding of the raw mix is more uniform ranging from 0.363-209μm compared to the Pikalevo raw mix ranging from 0.316-550 μm.

Pyro system test set-up

The pyro system designed for the semi-industrial test is in principle identical to an industrial operated system. It consists of a 3-stage preheater with calciner and a rotary kiln. The major difference to an industrial operated system is the missing product cooler and tertiary air duct connected to the calciner. These two pieces of equipment are in an industrial operated system part of stabilizing temperature profile and reducing fuel consumption as a major part of the combustion air for both main burner and calciner is preheated by these features.

Figure 10 and 11 shows the pyro system set-up, while figure 12 is a picture taken of the flame and burning zone in the rotary kiln during operation.

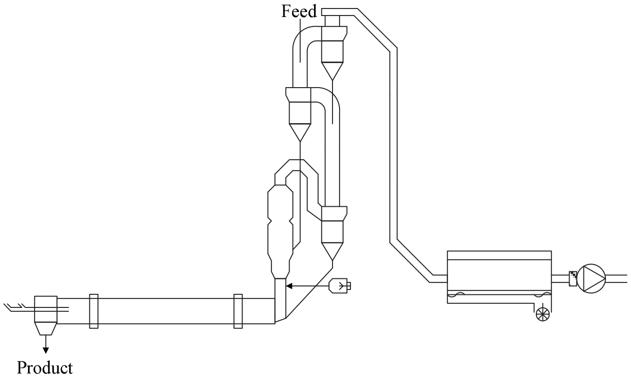

Figure 10. Pyro system test set-up.

Figure 11. Picture of pyro system set-up.

The solid material retention time in a preheater is only seconds while in the rotary kiln it was adjusted to 1½ hours. This is significantly less than the 2½ hours retention time at Pikalevo. It is expected that the retention time in the industrial operated system can be reduced even further as the features as discussed above; product cooler and tertiary air ducts are present, resulting in a more stable operation. For a cement kiln the solid material retention time is approximately 30 minutes in total.

Figure 12. Picture of burning zone.

Results of the semi-industrial testing

During the course of the 10 day test, samples were collected from feed to product including discharge from every cyclone stage and bag house dust. All operational data was logged and changes in the process parameters were documented. This data is the basis for the discussion of the results. The results of the best and most stable period of the semi-industrial test are illustrated in four graphs; figures 13-16.

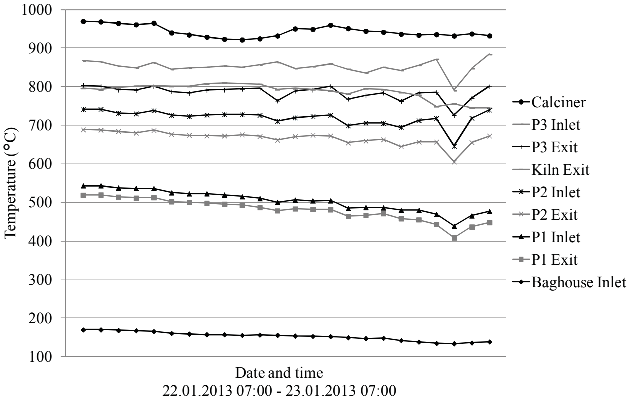

Figure 13. Preheater and calciner temperature profile.

Semi-industrial test systems are more vulnerable to minor fluctuations than industrial operated systems, as dimensions are smaller and heat loss is higher. Also the systems have to be versatile in order to serve a number of different applications. The most common challenge in semi-industrial test systems is blockages and maintaining a stable temperature profile. This was also the case for this system and experience has been gathered from this.

Nevertheless regarding the period documented the data illustrates an ability to maintain a stable temperature profile enabling effective sintering and required product quality. The temperature profile also corresponds to the zones mentioned earlier for the wet rotary kiln at Pikalevo. The kiln exit temperature is lower than expected for an industrial operated system as well as the calciner temperature.

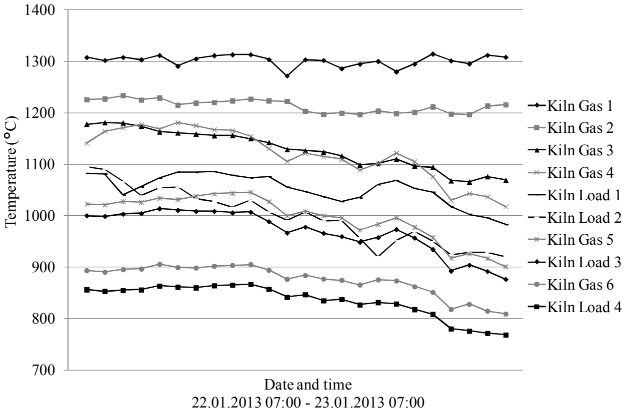

Figure 14. Kiln temperature profile.

The approximate temperatures expected for an industrial operated system are:

Kiln exit: 1100°C
Calciner: 900°C

The lower temperatures during the semi-industrial test can be explained by the increased heat loss of the test system and the differences in the equipment installed contra an industrial operated system as mentioned earlier. In the effort to obtain the above mentioned temperatures the burning zone temperature increased leading to difficulties in maintaining the optimal sintering temperature.

The burning zone temperature window for sintering is narrow and needed special attention and control when trying to optimize process parameters. Ring formations were observed on several occasions due to an increase in liquid phase making it necessary to lower the burning zone temperature.

The lime and alkali modules illustrates that the chemistry in the system is stable and controllable. At Pikalevo the dust return feed point is to the burning zone, however in the semi-industrial test it was to the lowest preheating stage ensuring that the dust would be carried into the kiln. The results indicate that the return feed point is suitable, as the alkali module is stable in the sinter produced and does not vary significantly from the raw mix modules.

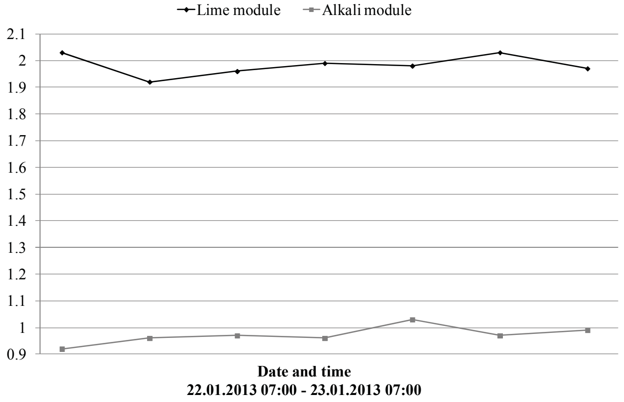

Figure 15. Lime and alkali modules in sinter.

For the given period the product quality results were stable and as required. At Pikalevo the variation in alumina extraction in the sinter is in the range of 87-89% while it for the semi-industrial test was in the range of 86-90%.

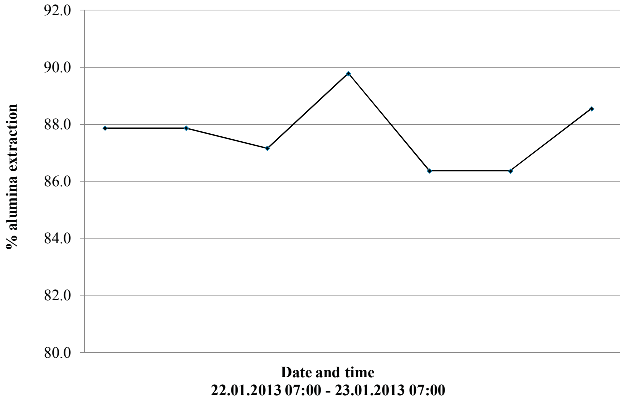

Figure 16. Alumina extraction.

The heat consumption of an industrial operated system has on the basis of the above results been theoretically calculated to be approximately 610 kcal/kg. Compared to Pikalevo with a heat consumption of approximately 1290 kcal/kg this gives a reduction of 680 kcal/kg. That is a significant reduction in heat consumption to lower than half of what it is today.

Conclusion

Dry sintering of nepheline and limestone is a new approach to the sintering process resulting in a much more energy efficient technology. The process has been proven in a semi-industrial test and the alumina extraction results are in line with what is achieved today at the Pikalevo site where a wet technology is applied.

Additionally when comparing the two technologies the following process knowledge and experience has been transferred:

1. Separate grinding resulting in similar particle size distribution of the nepheline and limestone
2. Enhanced focus on raw mix homogenization and chemical control to ensure optimal conditions for sintering
3. Control of calcination degree and temperature profile
4. Special care to the temperature profile in the rotary kiln as the burning zone is narrow
5. Retention time is needed to allow for the sintering to reach completion

Further optimization is possible and industrial operated systems would be custom built which should enable the dry process to achieve excellent performance.

A new more energy efficient technology for sintering of nepheline and limestone has been developed and proven semi-industrially, with a more than 50% reduction in heat consumption. Furthermore the dry technology offers the possibility to have bigger production units.

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