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TECHNOLOGICAL SCHEME AND MATERIAL BALANCE OF THE PRODUCTION OF NITROGEN-CALCIUM FERTILIZERS FROM SODA PLANT SLUDGE WASTE

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Abstract

Today, the utilization of waste from soda production enterprises and the production of high-quality and profitable products based on them is an urgent problem. Because by utilizing these wastes, a very large environmental problem can be eliminated. In addition, it can be learned from our ongoing research that these wastes are actually raw materials. In particular, this article presents a technological scheme and material flow calculations for obtaining liquid fertilizers containing nutrients such as nitrogen and calcium from sludge waste generated during soda production.

Keywords: *Liquid nitrogen-calcium fertilizers, ammonization, crystallization, filtration, ammonia, nitric acid slurry, calcium nitrate*

Introduction

In recent years, the European Union (EU) and other developed countries have been increasingly interested in renewable energy sources, waste-free technologies, and resource-efficient production methods. In order to ensure economic and environmental efficiency, it is important to optimize the consumption of raw materials, determine the profitability of semi-finished and finished products, analyze them, and manage their material balance of technological processes (European Commission. 2020; ISO 14051:2011).

In the soda ash production industry, large volumes of sludge waste are generated during many technological stages. These wastes mainly contain calcium carbonate ($CaCO_3$), calcium hydroxide ($Ca(OH)_2$), sodium salts, and other useful components, which can be recycled as secondary raw materials (Yunusov A. S., et al., 2019; Yunusov A. S., et al., 2021). However, in practice, these sludges are often dumped into the environment, occupying land resources and increasing environmental hazards (Mamatqulov B., Karimova D., 2020).

At the same time, one of the important factors of sustainable industrial development is the saving of resources through waste recycling and their return to the economy (UNEP. 2019). In this context, the development of a technology for the production of nitrogen-calcium fertilizers from soda ash sludge is scientifically and practically relevant. Such fertilizers contain nitrogen and calcium, which are important in increasing soil fertility, especially in neutralizing acidic soils (Xodjayev B., 2022; Kolesnikov A. A., 2018).

The proposed technological approach allows simultaneously reducing waste, ensuring environmental safety and achieving economic efficiency. At the same time, the material balance compiled for the stages of the process allows for a scientific assessment of production efficiency (Khusanov D. R., 2023). This article reviews the technological scheme for the production of fertilizer from soda ash sludge, its main stages and material balances, and analyzes their efficiency.

The ongoing research work is devoted to the processing of sludge waste generated during the brine washing process at the JV "Kungroat Soda Plant" LLC and the production of liquid nitrogen-calcium fertilizers based on it. In this regard, all technological parameters from raw materials to the process of obtaining finished products have been determined and the results have been presented in the articles (Ergashev M. T., Rajabov Sh.Sh., Mirzakulov X.Ch., 2024; Ergashev M. T., Rajabov Sh.Sh., Mirzakulov X.Ch., 2025).

Research methodology and methods

The main goal of this scientific research work is to develop a technology for producing nitrogen-calcium fertilizer using soda ash sludge waste and determine the material balance of the process. The research was conducted based on the following stages and methods:

1. Experiments with reagents for obtaining nitrogen-calcium fertilizer: The main components of the sludge and their ability to react with other nitrogen compounds were tested. Conditions – factors such as pH level, temperature and mixing speed were determined experimentally.

2. Material balance calculations: The mass of incoming and outgoing substances according to the technological process was calculated. The amount of fertilizer produced was compared with the amount of sludge, ammonium nitrate and other reagents. This created a material balance scheme for production and ensured maximum recycling of waste.

3. Product quality assessment: The physicochemical properties of the obtained nitrogen-calcium fertilizer samples (solubility, crystal size, nitrogen and calcium content) were analyzed in the laboratory in accordance with GOST standards.

Main part: When nitric acid slurry is evaporated to 85% of its initial mass, the content of soluble substances in the composition is as follows: $Mg(NO_3)$-2–9.5%; $Ca(NO_3)2$–56.1%; $Al(NO_3)_3$–0.4%; Fe_2O_3–0.1%; NH_4NO_3–6.1%. The resulting solution can be used as a liquid nitrogen-calcium fertilizer (LNCF-A) containing magnesium.

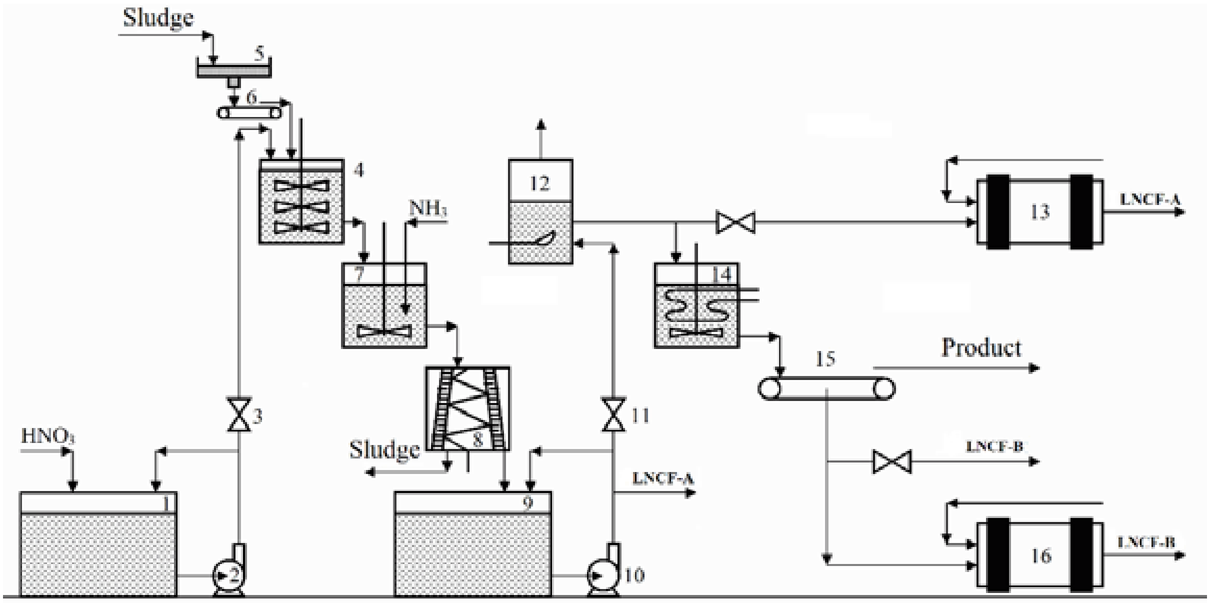
The resulting solution can be crystallized to obtain calcium nitrate, and the resulting crystals can be filtered and used as a liquid nitrogen-calcium fertilizer (LNCF-B) containing magnesium.

The results of the research work provided the basis for developing a technology for producing liquid nitrogen-calcium and nitrogen-calcium-magnesium fertilizers using sludge waste from a soda plant. The figure below shows the basic technological scheme for the production of liquid nitrogen-calcium fertilizers with and without the separation of calcium nitrate.

The description of this technological scheme is as follows: From the nitric acid tank (1), unconcentrated nitric acid is supplied to the reactor (4) through a pump (2) through a flow meter (3). Sludge waste from the soda plant is supplied from special tanks (5) for sludge collection via a weigher (6). The nitric acid standard is supplied at 100–105% to convert all cations in the sludge into nitrates. Filter washing water is also supplied there. Due to the exothermic nature of the process, the temperature in the reactor (4) is maintained at 45–55 °C. The residence time of the reaction mixture in the reactor is 15–30 minutes.

Then the reaction pulp is sent by gravity to the ammonizer (7), where the excess acid is neutralized with gaseous ammonia. Neutralization is carried out to pH = 5.5–7. In the ammonizer, the conversion of iron and aluminum nitrates into hydroxides, i.e. their transition to insoluble compounds, also occurs. Then the neutralized pulp is transferred by gravity to a continuously operating centrifuge (8). In the centrifuge, the pulp is separated into liquid and solid phases. The liquid phase is transferred by gravity to a collection tank (9) for the neutralized extract. The insoluble residue of the slurry is sent to the solid phase waste collection. The neutralized extract collected from the collection tank (9) is sent to the consumer as a liquid nitrogen-calcium fertilizer (LNCF-A) or for further processing.

Figure 1. *Basic technological scheme for processing filter press sludge into nitrogen-calcium fertilizers*



Further processing of LNCF-A involves evaporation to remove part of the calcium nitrate or to obtain a granular product.

Evaporation of LNCF-A occurs in a foam evaporation unit (FEU) (12), where LNCF-A is directed by a pump (10). If it is necessary to reduce the amount of calcium nitrate in the product, LNCF-A, after evaporation in FEU (12), is sent to the crystallizer (14) for cooling. In the crystallizer (14), calcium nitrate crystals are formed, which are separated from the solution on a belt vacuum filter (15). After separating the crystals, the liquid phase is sent to the consumer as liquid nitrogen-calcium fertilizer LNCF-B or sent for further processing.

Thus, the presented technological scheme allows obtaining three types of fertilizers differing in the ratio and composition of nutrients.

In the ongoing research work, a material balance was developed to determine the consumption of raw materials, the profitability of semi-finished products and finished products. Also, as mentioned above, samples of two types of fertilizers (LNCF-A and LNCF-B) were presented in the research work.

In particular, Table 1 shows the material balances for the production of LNCF-A and calcium nitrate (for 1000 kg of finished product).

Table 1. *Material balance of LNCF-A production (per 1000 kg of finished product)*

Sludge	326,19	–	–	–	–	–	–	–
HNO_3	–	478,16	–	57,38	–	–	–	–
H_2O	–	353,43	–	433,14	–	415,75	409,06	6,69

CO_2	–	–	90,16	–	–	–	–	–
E.Q.	–	–	–	49,03	–	49,03	0,00	49,03
$Mg(NO_3)_2$	–	–	–	22,18	–	22,18	21,82	0,36
$NaNO_3$	–	–	–	29,92	–	29,92	29,43	0,49
$Ca(NO_3)_2$	–	–	–	433,06	–	433,06	426,12	6,95
$Fe(NO_3)_3$	–	–	–	16,63	–	0,42	0,42	0,00
$Al(NO_3)_3$	–	–	–	26,28	–	1,31	1,28	0,03
NH_3	–	–	–	–	14,09	–	–	–
NH_4NO_3	–	–	–	–	–	113,71	111,88	1,83
$Fe(OH)_3$	–	–	–	–	–	7,18	–	7,18
$Al(OH)_3$	–	–	–	–	–	9,17	–	9,17
∑	326,19	831,59	90,16	1067,62	14,09	1081,71	1000,00	81,71

As can be seen from the table above, to produce 1000 kg of LNCF-A, we need 326.19 kg of dry sludge waste. To decompose sludge waste at an acceptable acid concentration and rate, 478.16 kg of acid is required. Since the acid concentration is 57.5%, 353.43 kg of water is added to the acid and enters the process. During the decomposition process, 90.16 kg of CO_2 is released in the gaseous state. In the next stage, i.e., the ammonization process, 14.09 kg of ammonia is added and the mixture is sent to the filtration process. During the filtration process, mainly insoluble residue is removed with a certain amount of moisture. In addition, it was found that the insoluble residue contains small amounts of $Ca(NO_3)_2$, $Fe(OH)_3$ and $Al(OH)_3$. The composition of the resulting 1000 kg of LNCF-A fertilizer was as follows. $Mg(NO_3)_2$–21.82 kg; $NaNO_3$–29.43 kg; $Ca(NO_3)_2$–426.12 kg; $Fe(NO_3)_3$–0.42 kg; $Al(NO_3)_3$–1.28 kg; NH4NO3–111.88 kg and 409.06 kg of water.

The material balance of the proposed technology for obtaining LNCF-B fertilizer proceeds in the following stages (Table 2).

As can be seen from Table 2, to produce 1000 kg of LNCF-B, we need 748.22 kg of dry sludge waste. To decompose sludge waste at an acceptable acid level and concentration, 1096.82 kg of nitric acid is required. Since the acid concentration is 57.5%, 810.70 kg of water in it is added to the acid and enters the process. During the decomposition process, 206.81 kg of CO_2 is released in the gaseous state. As a result, 2448.93 kg of suspension is formed. In the next stage, i.e., the ammonization process, 32.32 kg of ammonia is added. As a result of the addition of ammonia, NH_4NO_3 is also formed in the suspension and the total volume of the suspension increases to 2481.26 kg. After that, the mixture is sent to the filtration process. During the filtration process, 112.45 kg of insoluble residue is removed with a certain amount of moisture. This leads to a partial loss of soluble substances present in the solution. As a result, a total mass loss of 187.43 kg is observed at this stage. In the next stage, a crystallization process was carried out to separate $Ca(NO_3)_2 \cdot 4H_2O$ in a crystalline state. As a result, 573.44 kg of water was evaporated and 720.39 kg of $Ca(NO_3)_2 \cdot 4H_2O$ crystals were filtered and separated. The composition of the remaining solution, i.e. 1000 kg of LNCF-B fertilizer, was as follows: $Mg(NO_3)_2$–46.98 kg; $NaNO_3$–63.35 kg; $Ca(NO_3)_2$–490.25 kg; $Fe(NO_3)_3$–0.90 kg; $Al(NO_3)_3$–2.76 kg; NH_4NO_3–240.87 kg and 154.90 kg of water.

In conclusion, in the current research work, a material balance was established to determine the cost of raw materials, the profitability of semi-finished products and finished products. In particular, to obtain 1 ton of LNCF-A fertilizer, a dry sample of 326.19 sludge waste can be obtained by decomposing it with 478.16 kg of 57.5% nitric acid. In addition, it was determined that 654.90 kg of

Table 2. *Material balance of calcium nitrate and LNCF-B production (per 1000 kg of finished product)*

Name	Stream number according to the scheme												
	1	2	3	4	5	6	7	8	9	10	11	12	13
Sludge	748,22	–	–	–	–	–	–	–	–	–	–	–	–
HNO_3	–	1096,82	–	131,61	–	–	–	–	–	–	–	–	–
H_2O	–	810,70	–	993,56	–	953,67	938,32	15,35	364,88	573,44	165,06	154,90	10,16
CO_2	–	–	206,81	–	–	–	–	–	–	–	–	–	–
H.o.	–	–	–	112,45	–	112,45	–	112,45	–	–	–	–	–
$Mg(NO_3)_2$	–	–	–	50,87	–	50,87	50,05	0,82	50,05	–	50,05	46,98	3,08
$NaNO_3$	–	–	–	68,62	–	68,62	67,50	1,12	67,50	–	67,50	63,35	4,15
$Ca(NO_3)_2$	–	–	–	993,37	–	993,37	977,43	15,94	977,43	–	522,35	490,25	32,10
$Fe(NO_3)_3$	–	–	–	38,15	–	0,95	0,95	0,00	0,95	–	0,95	0,90	0,06
$Al(NO_3)_3$	–	–	–	60,29	–	3,01	2,94	0,07	2,94	–	2,94	2,76	0,18
NH_3	–	–	–	–	32,32	–	–	–	–	–	–	–	–
NH_4NO_3	–	–	–	–	–	260,83	256,64	4,19	256,64	–	256,64	240,87	15,77
$Fe(OH)_3$	–	–	–	–	–	16,46	0,00	16,46	–	–	–	–	–
$Al(OH)_3$	–	–	–	–	–	21,03	0,00	21,03	–	–	–	–	–
$Ca(NO_3)_2 \cdot 4H_2O$	–	–	–	–	–	–	–	–	–	–	654,90	–	654,90
Σ	748,22	1907,52	206,81	2448,93	32,32	2481,26	2293,83	187,43	1720,39	573,44	1720,39	1000,00	720,39

$Ca(NO_3)2 \cdot 4H_2O$ can be obtained by obtaining 1 ton of LNCF-B fertilizer.

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