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Nitric Acid Decomposition Process of Sludge Waste of the Soda Plant

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ABSTRACT: Today, air and environmental pollution is an urgent problem in the world. One of the main reasons for this is various types of waste from industrial enterprises. Various waste-free technologies are being created to overcome these. However, the goal cannot be achieved only by creating zero-waste technologies. Because there is a lot of industrial waste collected until now, and many types of waste are released from industrial enterprises that work in the old way. Processing these wastes and obtaining quality products based on them remains a challenge. In particular, 15 different types of solid, liquid and gaseous wastes are released from the soda production enterprise using the "Solve" method. This article is devoted to the study of the process of decomposition of sludge waste with nitric acid in enterprises producing caustic soda using the ammonia method. Various technological factors affecting the decomposition process are considered. Using modern chemical analysis methods, he determined the composition of the initial and resulting products. Optimum technological parameters for the decomposition of these wastes with nitric acid have been determined by studying the acid norm, temperature, time and solid-liquid phase ratio. Based on the obtained results, preliminary information was obtained for processing these wastes with acid and obtaining liquid nitrogen fertilizers.

I. INTRODUCTION

The "Solve" method of producing calcined soda was created in 1840. In 1861, Belgian chemist Ernest Solve received a patent for the invention of soda production technology, and based on his invention, the first soda factory was built in Belgium in 1865. Until now, that is, since 159 years, the majority of soda enterprises produce soda in this way [1, 2]. The main advantage of this method is the ubiquitous availability of raw materials, sodium chloride and limestone. Due to the simplicity of the technological process and the possibility of obtaining clean and high-quality soda, this method cannot be avoided. At the same time, the main drawback of the method is the large amount of generated waste [3]. About 15 types of wastes come out of this type of enterprises, and among them, the most are sludge wastes from the filter-press device during the purification of distillation liquid [4] and sodium chlorine solution [5]. The processing of industrial waste, especially the waste of soda production enterprises, is a priority. Because the waste piles around the soda production enterprises seriously harm not only people, but also the flora and fauna there. Processing waste as secondary raw materials and obtaining various products based on them is one of the important tasks facing mankind.

Today, many scientists around the world are conducting research on the processing of soda ash production waste and obtaining products based on them [2, 3, 6, 7]. However, most of the research works are focused on the production of silicate and construction materials [8, 9, 10]. In particular, the inclusion of Cl^- and various compounds (NaCl, $CaCl_2$) in the amount of 2% in the initial state of these wastes into cement and concrete accelerates the hardening period of cement [11, 12]. For this reason, studies were conducted on the use of solid carbonate slurry containing chlorine anion as a cement strength enhancer in soda production [13]. In addition to calcium and sodium chloride, this waste contains a large amount of secondary calcium carbonate, which has been proven in the scientific works of a number of scientists [14]. In addition, waste sludge and limestone sand together have been found to accelerate the hardening of portland cement and slag portland cement. Addition of waste up to 3% to the composition of slag portland cement leads to an increase in the strength of the cement stone both in the initial and later periods of solidification [15, 16]. The optimal amount of the additive (3%) has been found to increase initial strength by 73% and brand strength by 19% [17].

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Taking into account the above, the production of liquid nitrogen fertilizers based on the processing of sludge waste of soda production enterprises with nitric acid was determined as the goal and tasks of the research. In this regard, the waste of JSC "Qo'ng'irot soda zavodi" located in the Konghirot district of the Republic of Karakalpakstan was determined as a research object. Because this enterprise also produces soda using the "Solve" method. In the first part of our scientific research work, we carried out experiments on returning the NaCl salt contained in the original composition of this type of waste to the soda enterprise by washing it with water and a circulating solution and we mentioned the results in our previous articles [18].

II. RESEARCH METHODS

The raw materials used in this research work were soda ash waste and nitric acid. Chemical analysis processes were carried out in laboratory conditions based on the requirements of the relevant state standard in studying the process of nitric acid decomposition of the sludge waste of the soda enterprise.

In particular, the determination of the amount of CaO and MgO was carried out by the volumetric titrometric method: the indicator was determined by titration with a 0.05 N solution of Trilon B in the presence of dark blue chromium [19, 20].

The amount of iron and aluminum oxides was determined by complexometric analysis method [21, 22]. To determine the amount of iron, by titration with Trilon-B in the presence of sulfosalicylic acid, and to determine the indicators of aluminum, it is based on re-titration of the excess amount of Trilon-B with zinc sulfate solution and titration in the presence of a flame-colored indicator during shaking.

The amount of gold-sulfur in the samples was determined by the gravimetric method with the participation of barium chloride [23, 24].

The amount of water in the samples was determined by drying in a drying cabinet at a temperature of 100-105°C to a constant weight [25].

The insoluble residue in the samples was determined by the gravimetric method. This method is based on filtering the samples mixed in hydrochloric acid for 30 minutes, separating the residue on the filter paper and determining its weight [26].

III. EXPERIMENTAL RESULTS

The second stage of the research consists in the decomposition of sludge waste with nitric acid and the production of liquid micro and complex fertilizers based on them. To do this, the chemical composition of these waste was studied first. The results of the chemical composition were table 1:

Table 1. Chemical composition of sludge

Sample	Chemical composition in terms of dry matter content, wt.%							Water, % wt.
	Mg^{2+}	Ca^{2+}	Al^{3+}	Fe^{3+}	SO_4^{2-}	CO_3^{2-}	i.r.	
Sludge	3,94	32,62	0,05	0,15	0,72	53,08	3,17	25,06

For the production of liquid micro and complex fertilizers from sludge waste, which are defined as the goals and objectives of the research, it is important to study the various technological factors (temperature, acid norm and solid-liquid ratio) that affect the decomposition process.

Experiments on the decomposition of sludge waste were carried out with an acid norm in the range of 75-110% of the stoichiometric norm for the main oxides CaO + MgO present in its composition. Separate experiments are carried out on mixing time, temperature, thermodynamic processes in the decomposition process and other technological processes important for production.

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In the process of decomposition, the degree of transition of components into solution (γ) is determined by the following formula:

$$\gamma_X = \frac{m_1 \cdot C_{X_1} - m_2 \cdot C_{X_2}}{m_1 \cdot C_{X_1}}$$

Here C_{X_1} – the % amount of components in the initial raw materials, C_{X_2} – % amount of components in the insoluble residue, m_1 and m_2 are the weight of the initial dry slurry and the weight of the insoluble residue.

In the first table, the effect of nitric acid rate on the chemical composition of solid and liquid phases is studied. In this experiment, the temperature was set at a constant 40 °C, and the mixing time was set at a constant 30 minutes. Nitric acid with a concentration of 57.5% was used in the experiment.

Table 2. Effect of nitric acid norm on the chemical composition of the phases formed during the decomposition of sludge waste

HNO_3 norm, %	Chemical composition. %											S:L ratio
	Liquid phase					An insoluble residue						
	Mg^{2+}	Ca^{2+}	Al^{3+}	Fe^{3+}	SO_4^{2-}	Mg^{2+}	Ca^{2+}	Al^{3+}	Fe^{3+}	CO_3^{2-}	SO_4^{2-}	4,47
75	1,26	8,76	0,00	0,03	0,05	3,74	38,27	0,12	0,28	49,70	1,28	8,45
80	1,37	9,90	0,01	0,02	0,04	4,85	51,96	0,19	0,48	28,74	2,18	14,67
85	1,43	10,73	0,01	0,02	0,04	5,64	62,56	0,29	0,76	8,48	3,48	21,45
90	1,45	11,23	0,01	0,02	0,03	5,46	61,65	0,37	1,01	0,97	4,68	31,36
95	1,46	11,67	0,01	0,01	0,03	4,80	51,44	0,49	1,37	–	6,24	44,17
100	1,44	11,79	0,01	0,01	0,02	3,86	37,95	0,64	1,77	–	8,03	55,34
105	1,41	11,60	0,01	0,01	0,02	2,93	27,93	0,74	2,05	–	9,13	64,59
110	1,40	11,56	0,01	0,01	0,01	2,90	27,92	0,76	2,09	–	9,15	4,47

As can be seen from this table, as the acid norm increases, Ca^{2+} and Mg^{2+} ions in the insoluble residue decrease. In particular, Ca^{2+} and Mg^{2+} ions in the insoluble residue at 75% of the acid norm were 38,27% and 3,74%, while at 105% of the acid norm, the amount of these substances was 27,93% and 2,93%, respectively. We can see that it has decreased to 93% and passed into the liquid phase. In addition, the amount of Al^{3+}, Fe^{3+} and SO_4^{2-} contained in the insoluble residue increases due to the decrease of Ca^{2+} and Mg^{2+}. That is, Al^{3+} increases from 0,12% to 0.74%, Fe^{3+} increases from 0,28% to 2,05%, and SO_4^{2-} increases from 1,28% to 9,13%. Also, the content of CO_3^{2-} in the insoluble residue sharply decreases with the increase of the acid norm, and it was found that at 95% acid norm, the complete gas phase has passed.

The content of the main substances Ca^{2+} and Mg^{2+} ions in the composition of the liquid phase increases as the acid norm increases. In particular, the Ca^{2+} ion in the composition of the liquid phase was 8,76% at 75% of the acid norm and increased to 11,60% at 105% of the acid norm. In the results of the experiment presented in the table, the reason for the low change of the substances in the liquid phase depends on the increase in volume. That is, with an increase in the acid norm, the total volume of the mixture increases, and sharp changes in the composition of the insoluble residue have little effect on the composition of the liquid phase. From the table above, it was determined that 105% of the acid level is acceptable. At values higher than that, the transition of the components to the liquid phase was less.

That is, the maximum transition rate of Mg^{2+}, Ca^{2+}, Al^{3+}, Fe^{3+}, SO_4^{2-} at the rate of 105% nitric acid is 99,46; 99,37; 45,30; 68,30; 61,14%, respectively.

One of the technological factors effecting the acid digestion process is the temperature, which allows to create an optimal technological regime norm by ensuring an optimal temperature level in the process of sludge waste decomposition and reducing heat consumption by setting a minimum temperature regime. This experimental work was carried out at the rate of 105% of nitric acid, which was acceptable in the results of the research conducted above. The mixing time was fixed at 30 minutes. Experiments were carried out between temperatures of 20°C-60°C. As a result of the experiments, it was

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found that the temperature has a significant effect on the degree of transition of Mg^{2+}, Ca^{2+}, Al^{3+}, Fe^{3+} and SO_4^{2-} to the liquid phase (Table 3).

Table 3. Effect of temperature on the composition of formed phases during the interaction of nitric acid and sludge waste

Temperature °C	Chemical composition. %.											Solid:Liquid ratio
	Liquid phase					An insoluble residue						
	Mg^{2+}	Ca^{2+}	Al^{3+}	Fe^{3+}	SO_4^{2-}	Mg^{2+}	Ca^{2+}	Al^{3+}	Fe^{3+}	CO_3^{2-}	SO_4^{2-}	
20	1,37	11,25	0,006	0,016	0,039	4,44	39,68	0,69	2,23	–	7,69	43,58
25	1,38	11,34	0,007	0,022	0,040	4,09	36,97	0,71	2,07	–	8,09	46,34
30	1,39	11,43	0,008	0,026	0,042	3,76	34,06	0,72	2,02	–	8,48	49,18
35	1,40	11,52	0,008	0,028	0,045	3,34	31,05	0,74	2,04	–	8,82	52,20
40	1,41	11,60	0,009	0,029	0,049	2,93	27,93	0,74	2,05	–	9,12	55,35
45	1,42	11,67	0,009	0,031	0,055	2,52	24,59	0,76	2,07	–	9,33	58,79
50	1,43	11,73	0,010	0,033	0,065	2,13	21,79	0,76	2,08	–	9,25	62,01
55	1,43	11,77	0,010	0,034	0,077	1,86	19,31	0,76	2,11	–	8,89	65,04
60	1,43	11,81	0,011	0,034	0,089	1,67	17,23	0,76	2,15	–	8,43	67,69

As can be seen from Table 2, the rate of transition of metal cations and sulfate ions to the liquid phase increases with the increase in the temperature of sludge waste decomposition. This is explained by the increase in the rate of interaction of the initial reagents with an increase in temperature. In particular, when the temperature decomposition process was carried out at 20°C, the composition of the liquid phase was Mg^{2+}-1,37%, Ca^{2+}-11,25%, and the composition of the insoluble residue was Mg^{2+}-4,44%, Ca^{2+}-39,68%. In the experiments conducted at a temperature of 60°C, the composition of the liquid phase changed to Mg^{2+}-1,43%, Ca^{2+}-11,81%, and the composition of the insoluble residue changed to Mg^{2+}-1,67%, Ca^{2+}-17,23%. Here, the rate of transition of the components into the liquid phase increases as the temperature increases during the decomposition process, it can be explained as follows. That is, as the temperature increases, the solid-liquid ratio also increases. From this we can understand that due to the increase in volume, the amount of components in the liquid phase is partially decreasing at high temperature. Thus, with an increase in temperature, there is almost the same increase in the coefficient of transition to solution of all components of sludge waste.

However, as the temperature increases, the loss of nitric acid increases due to its decomposition and transition to the gas phase, which can negatively affect the sanitary and hygienic conditions in the enterprise. Taking into account the above, the optimum temperature was determined to be 35-45°C, both from the point of view of the decomposition level of sludge waste and from the point of view of reducing the loss of nitric acid, and important data for further experimental processes were obtained.

Time is another factor affecting the process of decomposition of sludge waste with nitric acid. Because by determining how much time is spent in the decomposition process, it serves to optimize the subsequent technological parameters and the production process.

In this way, the effect of time on the decomposition process was studied using the acceptable parameters of the above experiments. That is, the standard of nitric acid was 105%, and the temperature was 40°C.

Table-4. The effect of time on the composition of solid-liquid phases in the process of decomposition with nitric acid

Time, min	Chemical composition.%									
	Liquid phase					Insoluble residue				
	Mg^{2+}	Ca^{2+}	Al^{3+}	Fe^{3+}	SO_4^{2-}	Mg^{2+}	Ca^{2+}	Al^{3+}	Fe^{3+}	SO_4^{2-}
Decomposition temperature – 40°C										
5	0,64	7,32	0,002	0,007	0,011	5,03	30,39	0,12	0,35	1,19
10	1,10	10,06	0,004	0,013	0,021	9,69	57,34	0,46	1,36	4,91
15	1,29	10,87	0,006	0,019	0,029	6,63	47,74	0,59	1,70	6,59
20	1,35	11,19	0,007	0,024	0,036	5,01	41,08	0,64	1,83	7,57
30	1,41	11,60	0,009	0,029	0,049	2,93	27,93	0,74	2,05	9,12
60	1,42	11,61	0,011	0,036	0,073	2,92	27,92	0,75	1,99	9,24

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From the table above, it can be seen that at all temperatures, the transition of the components to the liquid phase was rapid at the beginning of the decomposition process. Little changes are observed in 30 minutes and above. The duration of the process optimall is 30 minutes. The reason for this is that there is little difference between the results of the 30-minute decomposition experiment and the 60-minute experiment. Moreover, the duration of the process exceeding 30 minutes does not increase the transition of Mg^{2+}, Ca^{2+} into the solution, while Al^{3+}, Fe^{3+} and SO_4^{2-} continue to move into the solution in small amounts.

A number of scientists have conducted studies on the processing of waste containing calcium and magnesium ions with nitric acid. But there are aspects of our work that are different from their research work. Among them is the diversity of waste composition that we use.

For example, Chinese professor Hong Yang with his research team [27] and other scientists [28, 29] proposed decomposition with nitric acid to process waste containing $CaCO_3$. However, various parameters such as nitric acid concentration, reaction time and temperature in the decomposition process have not been paid much attention. The main results are focused on the synthesis of obtained calcium nitrate using liquid waste. In addition, as a source of unreacted nano-calcium carbonate and silicon in waste, Na_2SiO_3 is recommended as an improver of various physical and mechanical properties of concrete.

In addition, studies were conducted on the carbonization of waste containing mainly calcium and magnesium [30]. From the experiments, it can be seen that the thermodynamic properties of the formed dicarbonate minerals are improved. As a result, it is possible to obtain products that are resistant to different environments (acidic, alkaline) [31].

In his research, Mark Z. Jacobson [32] decomposed sea salt with HNO_3 acid using equilibrium modeling method. He estimated the composition of solid liquid and gas phases that can be formed. He studied the effect of time on this process and obtained the desired results. To test these hypotheses, published nitric acid consumption model coefficients for local inorganic aerosol concentration and size distribution measurements were compared with the above data by other scientists [33]. According to the results of the comparison, $NaNO_3$ and $Ca(NO_3)_2$ formation increases with time.

If we compare the research results of the above scientists in the process with nitric acid decomposition with the results of our research, we can list the following main differences.

Few scientists have conducted research on the decomposition of sludge waste from soda plants with nitric acid. Factors such as basic technological parameters (time, temperature, and acid rate, etc.) for the decomposition process were not taken into account in the conducted research. This means that the research work is not studied in depth.

The optimal time, acid rate, temperature and the optimal mode of action of solid-liquid phases for the process of decomposition of sludge waste with nitric acid were not established. The main drawback of this study was the dates. We have eliminated this shortcoming and provided optimal modes.

The main product $Ca(NO_3)_2$ is formed as a result of decomposition, but no scientist has proposed the production of mineral fertilizer based on this product.

IV. CONCLUSION

In conclusion, the process of decomposition of sludge treated with sodium chloride of soda ash enterprises with nitric acid and the production of liquid micro and complex fertilizers based on them, which is considered the first technological stage, has been fully studied. It was determined that the optimal time for the decomposition process is 30 minutes, the norm of nitric acid is 105%, the temperature is 40° C, and the ratio of solid-liquid phases is 1:55.35. Important information was obtained for the planned further experimental procedures. This is a proposed solution to partially eliminate the environmental problem of the enterprise. When research and development is fully completed, large amounts of waste can be eliminated and economic benefits can be obtained.

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