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Waste disposal of chlorine and caustic soda production

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ABSTRACT

A major problem for most petrochemical and chemical companies is the disposal of sludge waste generated during the filtration, precipitation, and neutralization stages that are stored in large quantities in sludge ponds or on industrial sites. In the production of chlorine and caustic soda, solid and liquid sludge wastes are generated along with the target product. The chemical composition of chlorine and caustic soda production sludge was determined, the phase composition of the sludge and its structure were studied. On the basis of on the results of the research, a fundamental technological scheme for sludge processing was developed and "Recommendations for the design of the scheme and basic equipment of the sludge processing unit for soda-caustic treatment of brine from chlorine and caustic soda production by the membrane method" were issued. Taking into account the importance of rational use of natural resources and environmental protection, new methods of utilization and processing of chlorine and caustic soda production sludge were developed, which allows obtaining a target product that can replace scarce and expensive components.

Keywords: sludge waste, sludge utilization, caustic soda production, membrane method.

INTRODUCTION

Solid wastes generated in the production of chlorine and caustic soda by the membrane method include sludge from the stages of brine saturation, brine clarification, sulfate removal from brine, and brine filtration. According to the design data, the yield of solid waste from the production of caustic soda by the membrane method is 138.58 kg per 1 ton of 100% NaOH [Permanent technological regulations, 2013; Thomas et al., 2005].

In terms of particle size, mineral sludge constitutes heterogeneous colloidal dispersed systems in which the solid phase is finely dispersed gypsum, calcium carbonate, soluble and insoluble salts of calcium, sodium, and metal hydroxides. Currently, sludge is removed from the process in special containers and transferred to third-party organizations for disposal [Crook et al., 2016]. The analysis of scientific, technical, and patent information made it possible to determine that chlorine and caustic soda production sludge can be used in various chemical industries, taking into account the relevant physicochemical, physical, mechanical, and toxicological properties of the waste [Thiel et al., 2017, Pathak et al., 2016]. However, none of these products has been widely used on an industrial scale due to the high corrosive activity of chlorides in sludge. The main areas of use of chlorine and caustic soda production sludge include:

- as concrete hardening intensifiers [Malovanyy et al., 2020];
- as concrete hardening intensifiers [Hamidi et al., 2017];

- as additive that allows the use of mortars at low temperatures;
- as a plasticizer of cement mortars;
- as anti-frost agents that prevent freezing during transportation of materials [Balasubrahmanyam et al., 2021];
- as drilling and washing solutions [1Malovanyy et al., 2022];
- as nutrient additives in soils [2Malovanyy et al., 2022];
- as anti-icing mixtures [Chelyadyn et al., 2024].

However, the analysis of the technical documentation requirements for the characteristics of building materials and the results of experimental studies of building materials obtained from soda sludge did not always yield positive results [Wang et al., 2014, Vasiichuk et al., 2022].

The aim of this research is developing the acceptable ways to utilize chlorine and caustic soda sludge, which allows obtaining a target product with minimal investment that can replace scarce and expensive components without compromising the performance and environmental characteristics of the final products.

Research materials

The first stage of the study involved determining the chemical composition of chlorine and caustic soda production sludge selected for laboratory experiments under production conditions. Chemical methods of analysis (weight and volume) were used to determine the physical and chemical properties of the sludge. The content of Ca^{2+} and Mg^{2+} in the sludge was determined by complexometric titration in the presence of a solid indicator of chromogen black, Cl^- – using the mercurometric method in the presence of a mixed indicator of diphenylcarbazone with bromphenyl blue, and insoluble residue and SO_4^{2-} – using the gravimetric method. Quantitative determination of carbonates and bicarbonates was performed by direct acidimetry.

The phase composition of the sludge was studied using an infrared spectrometer in the frequency range of 400–4000 cm^{-1} using a Specord-80IR device. Electron microscopic studies of the structure of the samples (crystal optical analysis) were carried out on a DS-130C scanning electron microscope analyzer.

The study of the sludge utilization process for the production of calcium chloride was carried out at a laboratory installation, the scheme of which is shown in Figure 1.

The glass reactor (3) is equipped with a stirrer (6), which rotates at a constant speed of 300 rpm. To continuously monitor the pH of the solution during the dissolution of the sludge, a glass electrode connected to a pH meter of the pH-150 type fixed in the reactor (5). The temperature of the solution was measured using an electronic thermometer, which was immersed in the solution in the reactor (3). Abgas hydrochloric acid to maintain pH 1–2 was supplied from the burette (2), mounted on the laboratory stand (1).

The procedure of the experiment was as follows. The reactor (3) was filled with abgas hydrochloric acid. The sludge in an amount proportional to the volume of acid (determined stoichiometrically) was poured into the reactor in portions. The dissolution process was considered complete when the pH of the medium was within 4–5. After the dissolution reaction, the suspension was separated by settling, the amount of undissolved sludge and liquid phase was determined, and samples of calcium chloride were taken to determine its chemical composition. A day later, the precipitate was separated from the solution and, its chemical composition was determined.

The dissolution kinetics of the sludge was evaluated by the rate of transition of calcium and magnesium ions into solution.

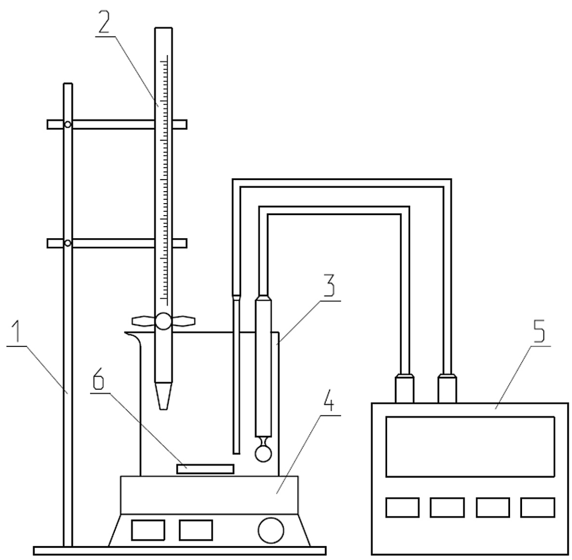

Figure 1. Laboratory setup for determination of sludge dissolution kinetics. 1 – laboratory tripod; 2 – burette with hydrochloric acid; 3 – reactor; 4 – magnetic stirrer; 5 – pH meter; 6 – stirring device

RESEARCH RESULTS AND DISCUSSION

When determining the chemical composition of sludge waste, it was found that the sludge taken from different stages of production have different chemical compositions (Tables 1, 2, 3, 4).

The phase composition of the sludge from the filter press was studied using infrared spectroscopy and crystal optical analysis.

Simple crystals, mostly tablet-shaped (0.03 to 0.2 mm in size), can be observed in the sludge samples. The immersion preparation shows large clusters of tightly adhering grains that form large aggregates (Figure 2).

Table 1. Chemical composition of the sludge from the saturator, % wt

No.	Indicator	Analysis results
1	Appearance	Crumbly mass of dark gray color
2	NaCl	33.95
3	H_2O	4.36
4	$CaSO_4$	7.10
5	Fe_2O_3	0.29
6	Insoluble residue	54.30

Table 2. Chemical composition of filter sludge, % wt

No.	Indicator	Analysis results
1	Appearance	Crumbly mass from white-pink color
2	Sodium carbonate and bicarbonate	0.73
3	H_2O	44.00
4	SO_4^{2-}	0.60
5	Ca^{2+}	1.20
6	Mg^{2+}	0.07
7	Cl^-	14.80
8	α-cellulose	38.6

Table 3. Chemical composition of the sludge from the filter press, % wt

No	Indicator	Analysis results
1	Appearance	Crumbly mass of gray color
2	NaCl	13.50
3	H_2O	22.70
4	Na_2SO_4	0.50
5	$CaCO_3$	62.00
6	$Mg(OH)_2$	1.10
7	Insoluble residue	0.20

The measured crystal-optical constants correspond to gypsum $CaSO_4 \times H_2O$. Refractive indices $N_p = 1.520 \pm 0.002$, $N_g = 1.529 \pm 0.002$, birefringence $\Delta = N_p - N_g = 0.009$.

The infrared absorption spectrum of the sludge contains a number of intense absorption bands: 420, 460, 610, 670, 1130, 1630, 2120, 2240, 3440, and 3590 cm^{-1} (Figure 3). These bands fully correspond to the spectral pattern of $CaSO_4{\cdot}H_2O$ gypsum. The peaks in the region of 610, 670, and 1130 cm^{-1} indicate the presence of SO_4^{2-} ion. The absorption maxima in the region of 3590 and 3440 cm^{-1} correspond to the valence, and the maximum of 1630 cm^{-1} to the strain vibrations of OH^-. A small peak in the region of 1400 cm^{-1} corresponds to the vibrations of the carbonate group of CO_3^{2-}. Absorption bands of other phases are absent.

On the basis of the results of the conducted research, the dissolution curve shown in Figure 4. Figure 4 shows that for the complete dissolution of the sludge, it is necessary to maintain a pH of no higher than 3.5, the required contact time of the reagents is 1–3 hours. On the basis of the results of experimental studies, the material balance of the sludge dissolution process with abgas hydrochloric acid was calculated. As a result of dissolving the sludge from the filter press stage with hydrochloric acid, a solution of calcium chloride is formed, which contains impurities of magnesium chloride and sodium chloride.

The concentration of the calcium chloride solution will depend on the moisture content of the sludge supplied for processing. The sludge moisture content after the filter press is 22.8 ± 5%.

Amount of sludge generated during soda-caustic brine treatment:

- 431.04 t/month = 13.9 t/day = 0.580 t/hour.

1. This sludge contains:

- $CaCO_3$ – 359.6 kg;

Table 4. Chemical composition of the sludge from the filter press (stage 02), % wt

No.	Indicator	Analysis results
1	Appearance	Crumbly mass from white-pink color
2	NaCl	8.70
3	H_2O	33.10
4	$CaSO_4$	58.20

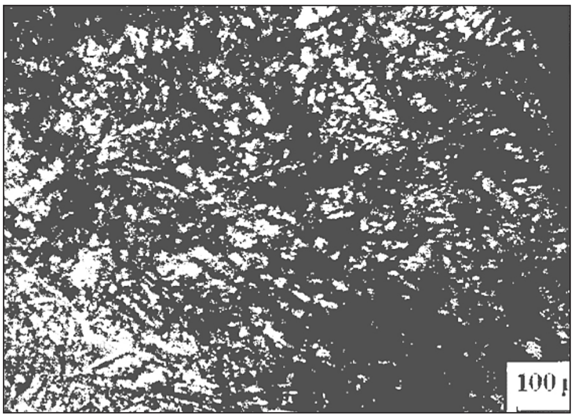

Figure 2. Micrograph of the immersion preparation of the sludge from the filter press

- $Mg(OH)_2$ – 6.38 kg;
- H_2O – 131.7 kg;
- NaCl – 78.3 kg;
- insoluble residue – 1.16 kg;
- Na_2SO_4 – 2.9 kg.

2. The amount of hydrochloric acid required to dissolve the sludge. For sludge dissolution, it is advisable to use 26% abgas hydrochloric acid with a density of 1129 kg/m³ [Vasiichuk et al., 2022, Kurylets et al., 2022]. The hourly consumption of acid is:

- to convert calcium and magnesium compounds into chlorides:

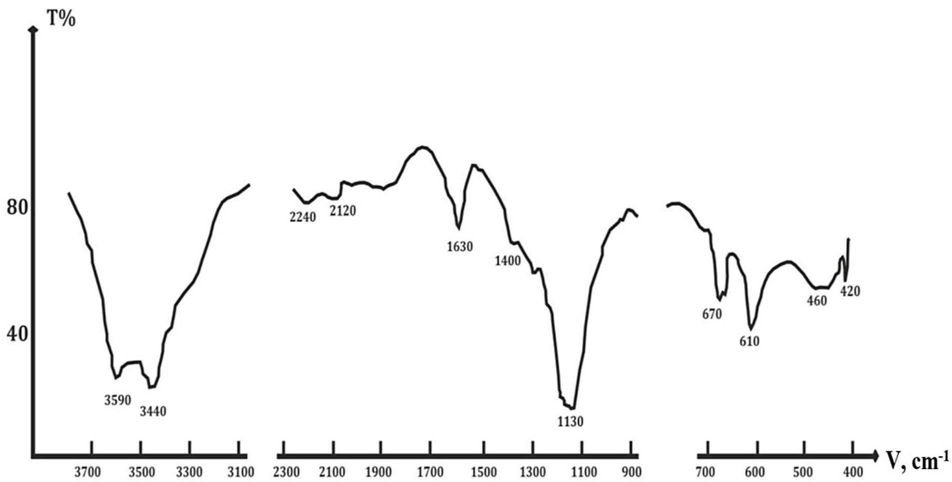

Figure 3. Infrared absorption spectra of the sludge from the filter press

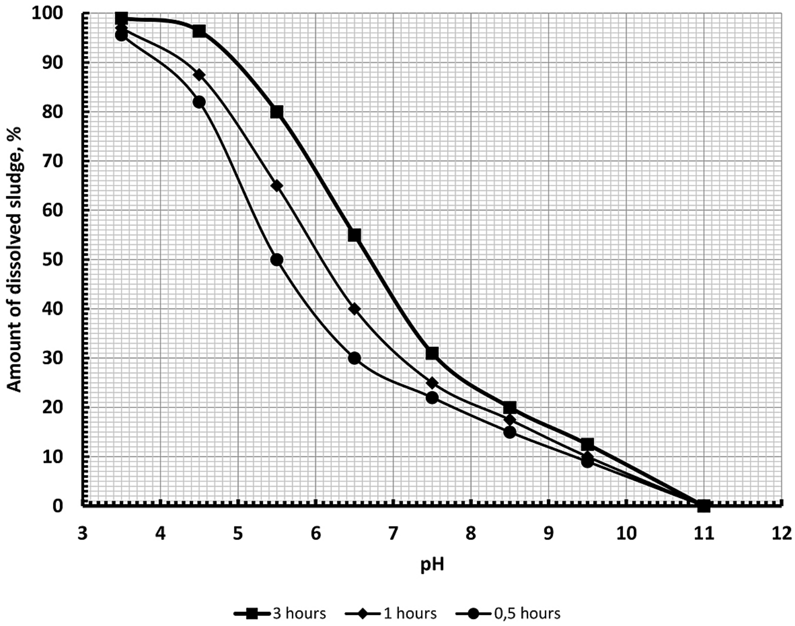

Figure 4. Dependence of the amount of dissolved sludge on pH

$$\frac{359.6 \cdot 73 \cdot 100}{100 \cdot 27 \cdot 1.129} + \frac{6.38 \cdot 73 \cdot 100}{58 \cdot 27 \cdot 1.129} = 887.5 \frac{l}{hour}$$

3. To bring the solution to pH = 1...2:

- to achieve pH = 1, the required H^+ concentration is 10^{-1} mol/l, or 0.1·36.5 = 3.65 g/l = 3.65 kg/m^3. Given that the acid is 26%, we have:

$$\frac{3.65 \cdot 100}{26} = 14.04 \frac{kg}{m^3}$$

or

$$\frac{14.04}{1.129} = 12.41 \frac{l}{m^3}$$

- for pH = 2, the H^+ concentration will be 10^{-2} mol/l, or 0.01·36.5 = 0.365 g/l = 0.365 kg/m^3. Given that the acid is 26%, we have:

$$\frac{0.365 \cdot 100}{26} = 1.404 \frac{kg}{m^3},$$

or

$$\frac{1.404}{1.129} = 1.241 \frac{kg}{m^3}$$

The amount of solution entering the reactor pos. 3 (Figure 1):

887.5 · 1.129 + 580 = 1582 kg/hour
or 15821.3 = 2117 l/hour ≅ 1.2 m^3/hour,

where: 1.3 is the density of the resulting solution (according to laboratory tests), g/cm^3.

The hourly consumption of acid to bring the pH of the solution to 1–2 will be

- at pH=1, 12.4·1.2=15 l/hour,
- at pH=2, 1.24·1.2=1.5 l/hour.

Total acid consumption:

887.5+15 = 902.5 l/h or 0.9 m^3/hour,
887.5+1.5 = 889 l/h or 0.89 m^3/hour.

When the sludge is fed into the reactor(3) (Figure 1), the acid in solution (pH = 1–2) reacts with the sludge according to the reaction equations:

$$CaCO_3 + 2HCl = CaCl_2 + CO_2 + H_2O \quad (1)$$
$$Mg(OH)_2 + 2HCl = MgCl_2 + 2H_2O \quad (2)$$

In the reactor (3) receives 12 m^3/hour of solution with pH = 1–2. Sludge with pH = 5–7 is fed to the same reactor (3) (Figure 1). When the pH is increased to 7, it will react with the sludge:

$$\frac{(3.65 + 0.365) \cdot 1.2}{2} = \frac{2.41 kg}{hour} HCl$$

This acid will dissolve calcium carbonate and magnesium hydroxide.

Only calcium carbonate, which is more abundant in the sludge, was calculated:

$$\frac{2.41 \cdot 100}{2 \cdot 36.5} = 3.3 \frac{kg}{hour}$$

This requires sludge:

$$\frac{3.3 \cdot 580}{359.6} = 5.32 \frac{kg}{hour}$$

4. The amount and composition of the solution formed.

1.2 m^3/hour of solution will be formed, containing:

$$\frac{359.6 \cdot 111}{100} = 399.16 \text{ kg } CaCl_2$$

$$\frac{6.38 \cdot 95}{58} = 10.45 \text{ kg } MgCl_2$$

i.e.

$$\frac{399.16 \cdot 100}{1.2 \cdot 1.3 \cdot 1000} = 25.59\% \ CaCl_2$$

$$\frac{10.45 \cdot 100}{1.2 \cdot 1.3 \cdot 1000} = 0.7\% \ MgCl_2,$$

and the residual amount of sodium chloride and insoluble residue brought with the sludge.

5. Polyacrylamide (PAA) flocculants are widely used in water treatment and waste disposal technologies, as they are highly efficient and inexpensive [Kurylets et al., 2022]. Calculation of PAA consumption. The working solution of hydrolyzed polyacrylamide contains 1–3 kg/m^3 of polyacrylamide. PAA is dosed into the settling tank at the rate of 1–3 g/m^3, i.e. 1 l/m^3, which will be 1.2 l/hour. Consumption of reagents and products in Table 5.

Description of the basic technological scheme of sludge processing

On the basis of on laboratory studies, a continuous technological scheme for dissolving sludge from soda-caustic brine treatment with hydrochloric acid is recommened (Figure 5). The sludge filtered on a filter press, consisting mainly of calcium carbonate and magnesium hydroxide, is continuously fed into a reactor with an intensive stirring device of note 2 (Figure 1). Hydrochloric

Table 5. Summary table of reagent consumption and products formed

No.	Name of reagents	Daily consumption
1	Sludge	13.9 ton
2	Abgaseous hydrochloric acid (26%)	28.8 m^3
3	Polyacrylamide, 8% commercial grade	1.2 kg
4	Calcium chloride solution	28.8 m^3

acid is dosed into the reactor by gravity from the pressure tank of abgas hydrochloric acid (1).

From reactor (2), the solution is fed by gravity to the intensive mixing reactor (3), where this solution is mixed to neutralize the excess acidity with a part of the soda-caustic treatment sludge. The acidity (pH) in reactor (2) is measured and adjusted automatically. The slurry formed in the reactor (3) flows by gravity into the central pipe of the settling tank (4), where it is dosed from the pressure meter (5) through the diaphragm mixer (6) hydrolyzed polyacrylamide (HPAA). The thickened part of the suspension from the sump (4) is discharged into a collector with a stirrer (7), from where it is pumped to the reactor (2).

The clarified part of the slurry from the settling tank note 4 is fed to the finished product collector (8). On the basis of the results of the research, "Recommendations for the design of the scheme and main equipment of the sludge processing unit for soda-caustic brine purification of chlorine and caustic soda production by the membrane method" were issued.

Table 6. Physicochemical properties of calcium chloride obtained as a result of sludge dissolution

No.	Name of the indicator	$CaCl_2$ solution synthesized	$CaCl_2$ solution according to GOST 450-77
1	Appearance	The solution is yellowish-gray in color, slightly cloudy	The solution is yellowish-gray or greenish in color, transparent or with slight turbidity
2	Mass fraction of calcium chloride, %, not less	22.5	35
3	Mass fraction of other chlorides, including $MgCl_2$, in terms of NaCl, %, not more	7.5	3
4	Mass fraction of undissolved residue in water, %, not more	2.5	0.15
5	Density, kg/m^3	1271	not standardized
6	pH	6.5	not standardized

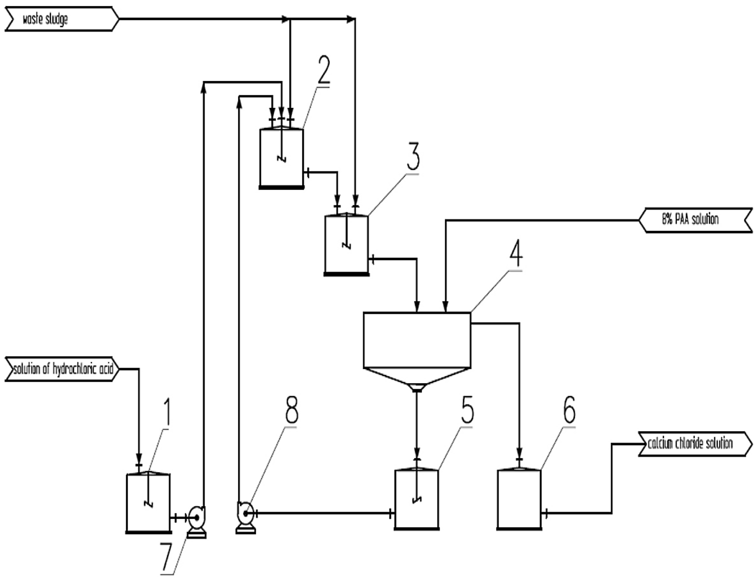

Figure 5. Schematic diagram of sludge dissolution. 1 – pressure tank of abgas hydrochloric acid; 2 – reactor; 3 – reactor; 4 – sump; 5 – pressure tank of HPAA; 6 – diaphragm mixer; 7 – sludge collector; 8 – calcium chloride collector

Calcium chloride obtained as a result of sludge dissolution has the following physical and chemical properties (Table 6):

According to laboratory data, the calcium chloride solution obtained as a result of sludge dissolution does not comply with GOST 450-77 “Calcium chloride technical. Technical conditions”. Therefore, the resulting solution cannot be used as a target product.

Alternatively, the resulting product can be discharged into an acidic sewer. In this way, caustic soda can be saved, which is used to neutralize abgas hydrochloric acid [Kurylets et al., 2022].

The design amount of gaseous HCl formed as a result of desorption in terms of 100% HCl is 1152 kg/hour.

$$HCl + NaOH = NaCl + H_2O \quad (3)$$

The amount of 100% NaOH consumed: mNaOH = (1152 (amount of 100% HCl) · 40 (mol. wt. of NaOH))/36.5 (mol. wt. of HCl) = 1253.5 kg/hour 100% NaOH. The amount of NaCl formed: mNaCl = (1152 (qty. 100% HCl) · 58.5 (mol. wt. NaCl))/36.5 (mol. wt. HCl) = 1846.4 kg/hour 100% NaCl.

The amount of water produced: mH_2O = (1152 (amount of 100% HCl) · 18 (mol. wt. of NaCl)) / 36.5 (mol. wt. of HCl) = 568.1 kg/hour 100 % H_2O. The amount of water required to dilute the chlorides obtained as a result of neutralization: 2000 mg – 1 kg; 1846400000 mg - X kg; mH_2O = ((1846400000 · 1) / 2000) – 568.1 = 922631.9 kg/hour. In the case of neutralization of stage 4 sludge with abgas hydrochloric acid, 1253.5 kg/hour of 100% NaOH and 922631.9 kg/hour of H_2O will be saved.

CONCLUSIONS

The laboratory studies to investigate the possibility of utilizing caustic soda sludge by the membrane method for its use as a building additive or any finished product enabled drawing the following conclusions:

Studies of chemical composition proved that the main components of caustic soda production sludge samples by the membrane method are

- sludge from the saturator: insoluble residue – 54.3% by weight; $CaSO_4$ – 7.1% by weight; NaCl – 4.36% by weight; H_2O – 22.7% by weight;
- - sludge from the filter press (stage 1): $CaCO_3$ – 62.0% wt.; NaCl – 13.5% wt.; Na_2SO_4 – 0.5 wt.; $Mg(OH)_2$ – 1.1 wt.; H_2O – 4.36% wt;
- sludge from the filter press (stage 2): $CaSO_4$ – 58.2% by weight; NaCl – 8.7% by weight; H_2O – 33.1% by weight.

The sludge from the saturator cannot be further processed due to the high content of insoluble residue, represented mainly by clay compounds, sand, and other substances. Alternatively, it can be considered as a material for storage in the unopened part of the quarry or as a material for reclamation work at solid waste disposal sites (solid waste landfills).

In the process of burning the sludge from the filter press (stage 1) at a temperature of 1000 °C, a sintered mass of light gray color was formed. Crushing and subsequent grinding produced a fine powder, which did not produce the expected result as a result of the reaction with water (no lime was formed during the sludge firing process).

As a result of dissolving the filter press sludge with hydrochloric acid (stage 1), a solution of calcium chloride is formed, which contains impurities of magnesium chloride and sodium chloride. The resulting solution of calcium chloride does not comply with GOST 450-77 “Calcium chloride technical. Technical conditions” and cannot be used as a target product. It is proposed to dissolve this sludge with abgas hydrochloric acid (a waste product of vinyl chloride monomer production) and discharge the resulting solution into the acid-base wastewater treatment system. In the process of dissolving the sludge with acid, the company will save 466.4 kg/hour of 100% NaOH and 336 kg/hour of H_2O, which are used to neutralize 1593 kg/hour of acidic wastewater.

The use of filter press sludge (stage 2) as a plasticizer is proposed, which makes it possible to eliminate lime in the preparation of mortars in which cement is the binder. Sodium chloride, which is part of this sludge, acts as a hardening accelerator. Studies of the physical and mechanical properties of the resulting mortar have established that the developed mortar according to DSTU B V.2.7-239: 2010 belongs to grade 25, with a design grade of 100.

The use of filter press sludge (stage 2) as an additive that allows the elimination of filler in the preparation of construction concrete with cement as a binder was proposed.

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