International Journal of Materials and Chemistry 2024, 14(2): 24-29
DOI: 10.5923/j.ijmc.20241402.03

Development of Technology for Obtaining Sodium Peroxide Sulfate

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Abstract The aim of the work is to study the theoretical foundations for obtaining $Na_2SO_4 \cdot 0.5H_2O_2$ by liquid-phase and solid-phase methods and to study its bleaching properties. In order to obtain bleaches, a theoretical analysis of the systems: sodium sulfate - hydrogen peroxide - water and potassium carbonate - hydrogen peroxide - water was carried out. Based on the chemical analysis of the compositions of liquid and solid phases, isothermal diagrams of the solubility of these systems at 10 and 20℃ were drawn. In order to obtain synthetic detergents and theoretical analyzes of the literature data, the mutual solubility of salts in the ternary system sodium sulfate - hydrogen peroxide - water was studied by the isothermal method. The obtained data of chemical analysis were used to determine the compositions of solid phases according to Schreinemakers method and drawing diagrams of solubility at 25℃. The solid-phase process of treating sodium sulfate (thenardite) powder with hydrogen peroxide at various rates was also studied. The liquidus curve of the solubility diagram for the sodium sulfate-peroxide-hydrogen-water system is characterized. The mineralogical composition of the products obtained by the liquid-phase and solid-phase methods was determined by X-ray phase, thermal catalytic and electron microscopic methods of analysis. X-ray phase analysis was used to determine the mineralogical composition of the products, thermal analysis was used to determine the thermal stability of the obtained samples, and electron microscopic analysis was used to determine the particle sizes and their compositions. The physics - mechanical properties of the obtained products are determined, which are the initial moisture content, apparent density, density with compaction, angle of inclination and hygroscopic point.

Keywords Sodium sulfate, Hydrogen peroxide, Sodium peroxysulfate, System, Synthetic detergent, Surface active agent, Ratio and crystal

1. Introduction

Currently, wide ranges of synthetic detergents are produced in the world, but they consist mainly of tripolyphosphate - a substance that does not wash itself, but only prepares water for washing. Washes the rest - surfactants, bleaches, enzymes and other ingredients. Tripolyphosphate is the cheapest, but also the most dangerous of them for human health and the environment. In many countries, the use of tripolyphosphate in synthetic detergents is prohibited. In this regard, the creation of cheap and environmentally friendly synthetic detergents from local materials is an urgent problem.

In recent years, among household chemical goods, one of the dynamically developing, constantly increasing amount of production is a group of bleaching agents. Modern bleaching agents should be considered as independent agents for bleaching linen, and as auxiliary agents for processing linen during the washing process. These goals are achieved with optical and chemical bleaches.

The bleaching effect is also achieved chemically. Oxidative bleaching has gained practical importance. Currently, it is carried out using peroxides and hypochlorites. The essence of bleaching lies in the fact that active oxygen radicals interact with the chromophore parts of pollutant molecules, turning them into uncolored compounds or white compounds.

The source of hydrogen peroxide is its aqueous 30% solution. Hydrogen peroxide is currently one of the most important oxidizing agents widely used in chemistry and chemical technology. Its main advantage is ecological compatibility; the main disadvantage is the inconvenience during transportation due to the high (60-70%) water content. In this regard, along with a solution of hydrogen peroxide, solid peroxides are also used - sodium perborate, sodium percarbonate, urea peroxide and others [1-5].

2. Materials and Methods

Based on this, in order to obtain synthetic detergent and theoretical analyzes of the literature data [6-9], we studied the mutual solubility of salts in the ternary system sodium

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Received: Apr. 9, 2024; Accepted: May 5, 2024; Published: May 7, 2024
Published online at http://journal.sapub.org/ijmc

sulfate - hydrogen peroxide - water was studied by the isothermal method [10-12] at 20℃ (table and figure). Chemical analysis of liquid and solid phases was carried out by known methods of analytical chemistry. The data obtained were used to determine the compositions of the solid phases according to Schreinemakers and drawing diagrams of solubility at 25℃.

The phase equilibrium in the system was established with continuous mixing and thermostated after 0.5 and 1 days, respectively. Based on the chemical analysis of the compositions of the liquid and solid phases, an isothermal diagram of solubility of this system at 25℃ is formed. In the sodium sulfate system - hydrogen peroxide - water at 20℃ is the formation of a new compound of the composition $Na_2SO_4 \cdot 0.5H_2O_2$. The liquids curve of the solubility diagram of the sodium sulfate system - hydrogen peroxide - water at the studied temperature is characterized by the presence of three branches of crystallization of the original components and the new compound of the composition - $Na_2SO_4 \cdot 0.5H_2O_2$. The connection formed in the studied system is identified by the chemical method. To obtain the compound, a solution of the composition figurative point N was prepared: Na_2SO_4-40.0%, H_2O_2-30.0% and H_2O-30% which was stirred at 35℃ for 10 minutes and cooled to 25℃. The formed solid phase from the suspension was separated by filtering, washed, and dried at 60℃.

3. Results and Discussion

Chemical analysis of the solid phase, selected from the alleged crystallization of the $NA_2SO_4 \cdot 0.5H_2O_2$ compound, gave the following results: defined, %: Na – 30,42; O_2 –5,90. For $Na_2SO_4 \cdot 0.5H_2O_2$, it is calculated, %: Na –30,46; O_2 – 5,96.

Table 1. Nodular points of the isotherm of the system $Na_2SO_4 \cdot$ - H_2O_2 - H_2O at a temperature of 25℃

№	The composition of the liquid phase, weight %			Solid phase
	Na_2SO_4	H_2O_2	H_2O	
1	16,20	-	83,80	$Na_2SO_4 \cdot 10H_2O$
2	27,0	56,22	16,78	$Na_2SO_4 \cdot 10H_2O$+ $a_2SO_4 \cdot 0{,}5H_2O_2$
3	30,0	12,50	57,5	$Na_2SO_4 \cdot 0{,}5H_2O_2 + H_2O_2$

It was also studied the process of a solid-phase method for obtaining sodium sulfate peroxide by processing sodium sulfate (thenardite) powder with hydrogen peroxide at a rate of 80, 100 and 110%. The data obtained are shown in Table 2.

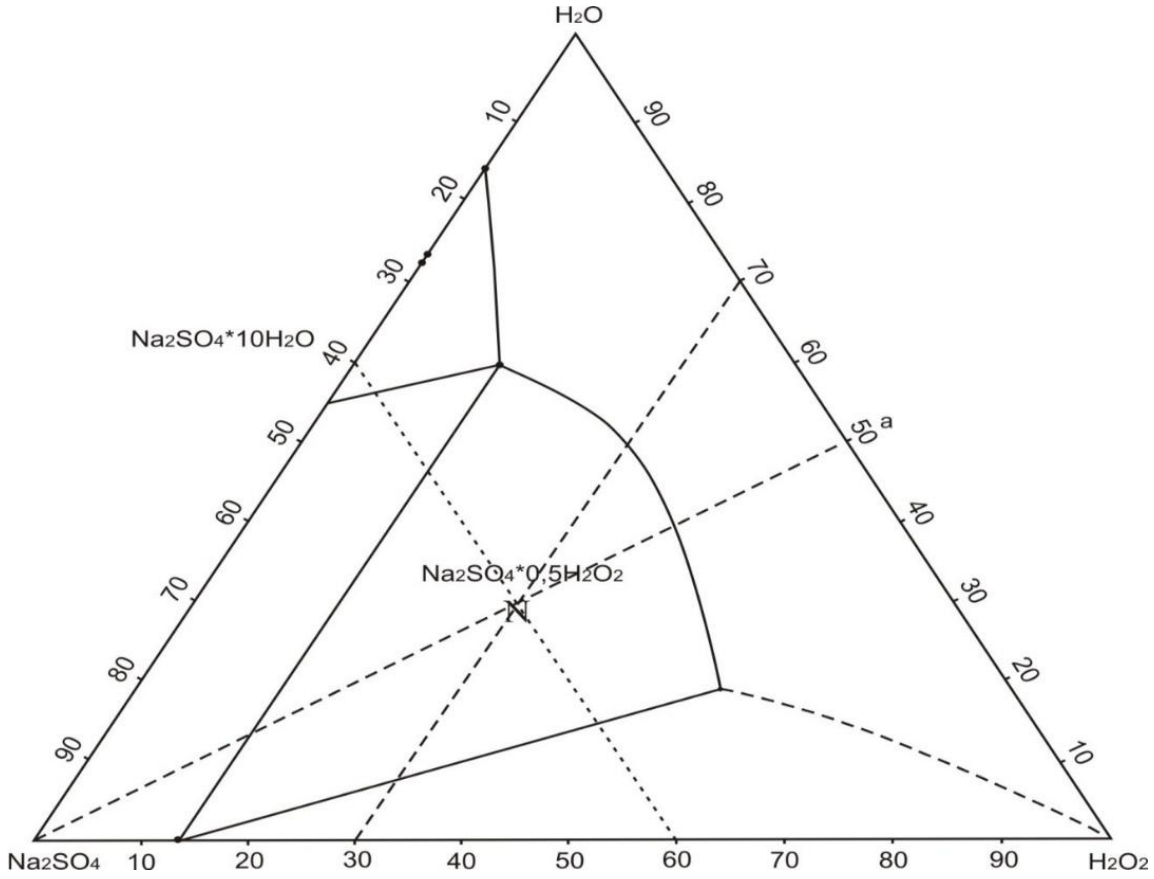

Figure 1. Isothermal diagram of system solubility Na_2SO_4-H_2O_2-H_2O at 25℃

Table 2. Influence of technological processes on the quality of peroxysulfate

№	Hydrogen peroxide concentration, weight %	Rate of hydrogen peroxide relative to $Na_2SO_4 \cdot H_2O_2$	Loss of hydrogen peroxide, weight %	Content of active oxygen,%		Humidity of the product after the process, %
				wet product	dry product	
1	40	80	31,5	3,27	4,10	6,62
2	50		20,8	3,72	4,91	4,21
3	60		19,6	5,71	5,01	2,51
4	40	100	50,6	5,36	3,91	8,12
5	50		43,4	5,52	4,12	6,51
6	60		30,5	5,73	4,83	5,04
7	40	110	51,6	5,41	3,14	11,01
8	50		45,6	5,60	3,25	8,11
9	60		36,5	5,80	4,81	6,01

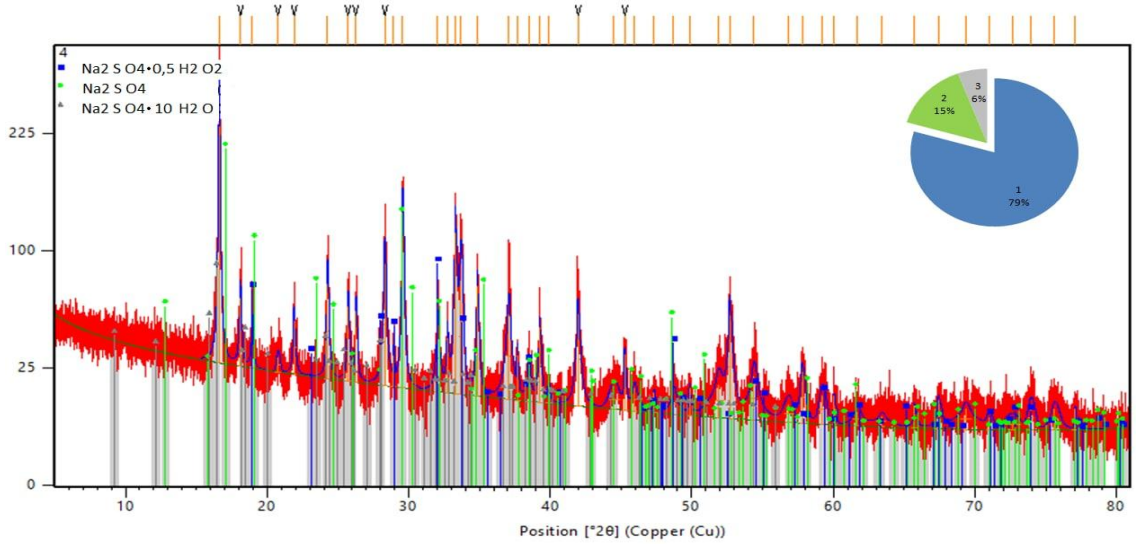

Figure 2. X-ray pattern of samples

Table 2 shows that the content of active oxygen in wet and dry products ranges from 3.27-5.73 and 3.12-5.01%, respectively. The moisture content of the product does not exceed 2.51-6.62% at a hydrogen peroxide rate of 80% in the range of the latter's concentration of 40-60%. With an increase in the rate of hydrogen peroxide from 100% to 110%, the ranges of humidity fluctuations increase and amount to 5.04-8.12% and 6.011-11.01%, respectively. In a dry product, with an increase in the rate of hydrogen peroxide, the content of active oxygen in products decreases.

This is explained by the fact that with an increase in the rate of hydrogen peroxide, the moisture content of the products increases and the content of hydrogen peroxide in the liquid phase increases proportionally, which is easily released from the product during drying. Accordingly, with an increase in the norm from 80.90 to 110%, the loss of hydrogen peroxide increases from 14.6, 20.8 and 31.5 to 35.5, 78.6 and 51.6%, respectively, at a hydrogen peroxide concentration of 40% and 60%.

Based on the foregoing, it can be concluded that with a decrease in the concentration and an increase in the rate of hydrogen peroxide, peroxide losses increase and the product yield relative to hydrogen peroxide is reached more than 80% at a rate of 80% use of hydrogen peroxide with a concentration of at least 50%.

Table 3. Mineralogical composition of the reaction products according to X-ray phase analysis

№ Sample corresponds to the table numbers	Ref.Code	Names of minerals	Content, weight %
3	01-085-1732	$Na_2SO_4 \cdot 0{,}5H_2O_2$	81,01
	01-086-0803	Na_2SO_4	13,10
	01-074-0937	$Na_2SO_4 \cdot 10H_2O$	7,10
4	01-085-1732	$Na_2SO_4 \cdot 0{,}5H_2O_2$	79,02
	01-086-0803	Na_2SO_4	13,10
	01-074-0937	$Na_2SO_4 \cdot 10H_2O$	5,10
9	01-085-1732	$Na_2SO_4 \cdot 0{,}5H_2O_2$	68,03
	01-086-0803	Na_2SO_4	18,11
	01-074-0937	$Na_2SO_4 \cdot 10H_2O$	13,90

To determine the mineralogical composition of the samples, X-ray phase analysis was used [13-15] (Fig. 2 and Table 3),

which showed that the mineralogical composition of the obtained product consists of $Na_2SO_4 \cdot 0.5H_2O_2$, $Na_2SO_4 \cdot 10H_2O$ and Na_2SO_4. At temperatures above 35℃ mirabilite completely transforms into thenardite after a certain period of time. The main constituent mineral of the samples is $Na_2SO_4 \cdot 0.5H_2O_2$, the content of which is more than 80%.

Morphological studies of the surface of the samples were carried out using a scanning electron microscope SEM - EVO MA 10 (Zeiss, Germany). For the experiment, on a round holder made of a metal alloy, on top of which an aluminum foil with a double-sided adhesive surface was glued, which sample powders were applied. This experiment is to study the microstructure of the surface, as well as to determine the size of microparticles. During the measurement, an accelerating voltage (EHT - Extra High Tension) of 20.00 kV was applied; the working distance (WD-working distance) was 8.5 mm. The measurement was carried out in the mode of detecting secondary electrons (SE1-secondary electrons detector). The images were acquired at various scales using the Smart SEM software.

The results of the study are illustrated in fig.3.

Figure 3 clearly shows the main particles with an average size of 572 and 495.5 mm, covered with minerals of sodium peroxide sulfate $Na_2SO_4 \cdot 0.5H_2O_2$, and inside the particles there is thenardite. When using 60% hydrogen peroxide, plate-shaped $Na_2SO_4 \cdot 0.5H_2O_2$ particles with dimensions of 5-15 mm are formed (Fig.3. sample.3 b), as well as mirabilite particles with dimensions of 100x80x300 mm., (Fig.3. sample. 9 d) [16].

To determine the thermal stability of the obtained samples, thermal analytical studies were carried out on a Netzsch Simultaneous Analyzer STA 409 PG instrument. Sample holders TG, TG-DTA are equipped with a thermocouple for direct temperature measurement in the sample/reference crucible. Temperature range: 25-3700C. Thermoanalytical studies of samples in the amount of 5-10 mg were carried out in an inert nitrogen atmosphere with a nitrogen flow rate of 50 ml/min [17-20].

Figure 4 shows that the decomposition of the product starts at a temperature of 60.1℃ and continues to a temperature of 133.1℃, and the total mass loss is 6.0-7.0%.

Figure 4 shows that the decomposition of the product begins at a temperature of 60.1℃ and continues to a temperature of 133.1℃, and the total weight loss is 6.0-7.0%.

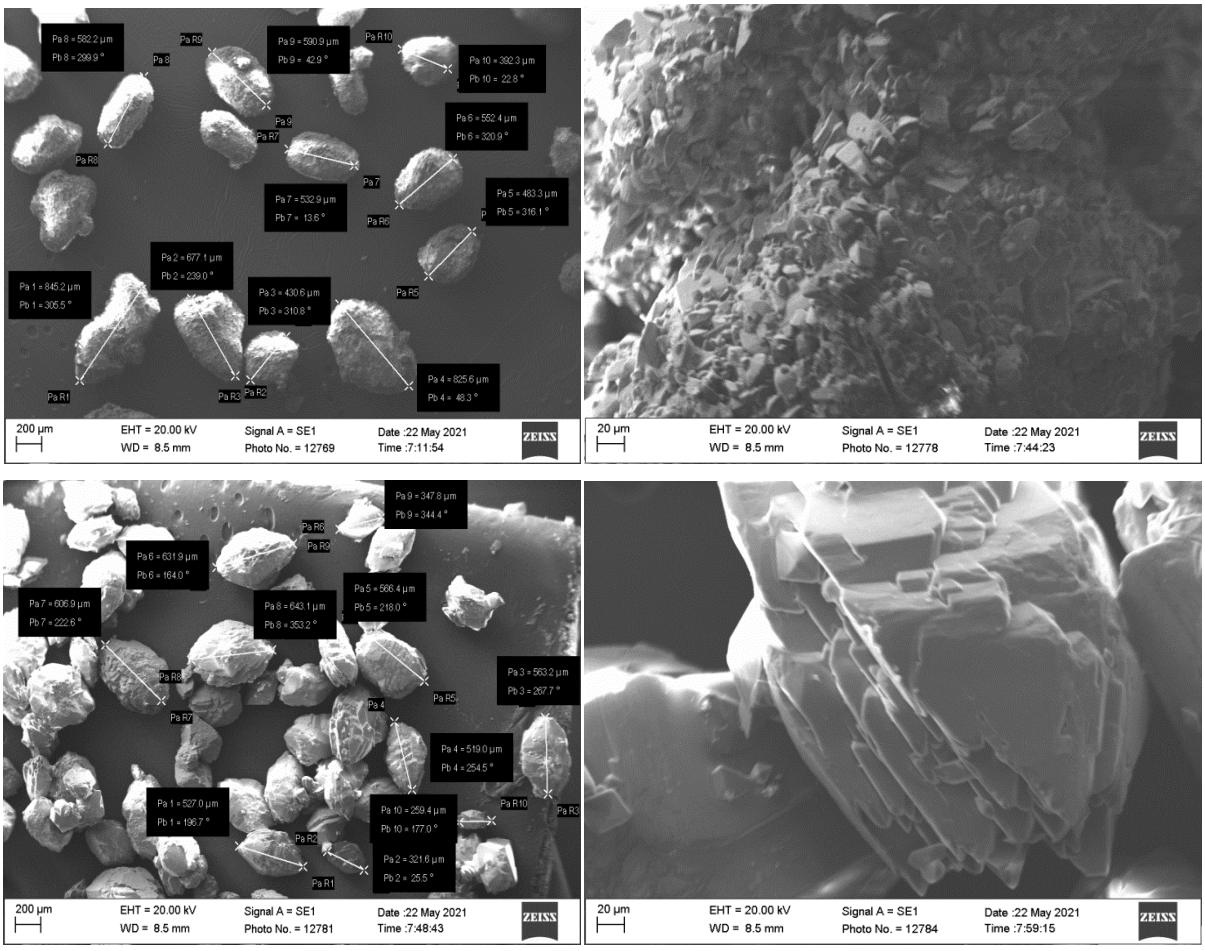

Figure 3. Microscopic images of samples 3.9. Sample numbers correspond to those in table 2

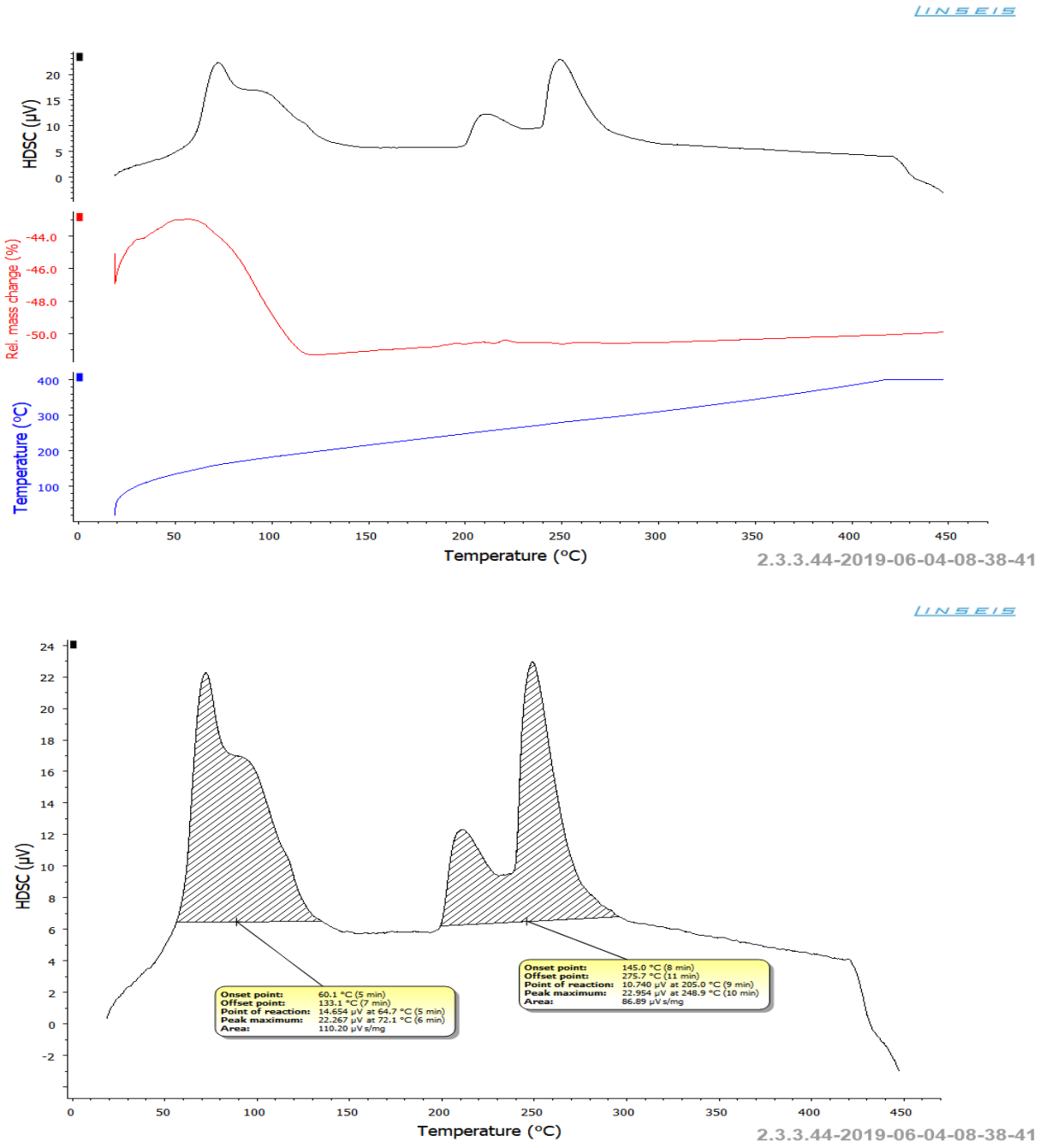

Figure 4. Derivatogram of products (numbers of samples correspond to numbers in tab. 2, sample 9)

Table 4. Physical and chemical properties of $Na_2SO_4 \cdot 0.5H_2O_2$

Technical indicators	Technical specifications $Na_2SO_4 \cdot 0{,}5H_2O_2$
Initial humidity, %	0,13
Apparent density, g/cm3	0,8557
Density of compaction, g/cm3	1,1000
Angle of inclination, hail.	35
Hygroscopic point, %	65

This process on the HDSC curve looks like a doublet maximum characterizing the decomposition of mirabilite and sodium sulfate peroxide with the release of water and elemental oxygen. Also on the HDSC curve, a second doublet maximum is observed in the temperature range 145-275.7°C, which is characteristic of the phase transformation of sodium sulfate without weight loss.

4. Conclusions

The physical and mechanical properties of the obtained product are also determined (Table 4). The data obtained show that the hygroscopic point of the product is 65%, i.e. it corresponds to low-hygroscopic substances.

Thus, the conducted studies show that $Na_2SO_4 \cdot 0.5H_2O_2$

develops good bleaching properties and can be obtained in two ways: liquid-phase and solid-phase.

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