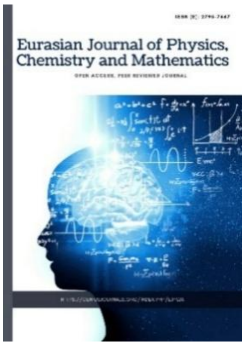

Preparation of calcium peroxide from liquid waste of the Kungrad soda plant and use in the pulp industry

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ABSTRACT

Soda ash is the main product of the chemical industry, which is used in glass production, the metallurgical, the pulp and paper industry and other. Currently,industry uses the Solvay method to produce soda ash. But the main disadvantage of the Solvay method is the formation of a large amount of liquid waste,the so-called distillation liquid. The problem is solved by the fact that with continuous stirring of the distiller liquid by a circulation pump of a thermostabilized reactor volume, a 55% solution of hydrogen peroxide is introduced into the distiller liquid at a temperature of 4 °C, bringing the pH to 5.3, then ammonia gas is bubbled into the distiller liquid to achieve the precipitation of calcium peroxide hydrate from the solution at pH up to 9.15, followed by separation of calcium peroxide hydrate on the filter surface, washing of the precipitate and dehydration of the precipitate at 120-130 °C . This method can be used at soda ash production plants to recycle soda ash production waste (distiller liquid) to produce calcium peroxide CaO_2 . The proposed method allows, by using gaseous ammonia and combining the functions of mixing and transporting distillation liquid using a circulation pump, to reduce the precipitation time of calcium peroxide hydrate to 1 minute and thereby increase the amount of commercial calcium peroxide by reducing its dissolution in the resulting ammonium chloride solution in a heat-stabilized volume reactor.

Keywords: Solvay method, distiller liquid, sentrifugat, calcium peroxide, liquid waste

1. Introduction

Soda ash is the common name for the technical grade anhydrous sodium carbonate (Na_2CO_3). In the 18th century soda ash was produced by LeBlanc process based on roasting salt cake with carbon and limestone.[1]. The largest soda ash production companies in the world were in China [2], the USA and Turkey. Belgian company Solvay holds a good lead with a capacity of 7.50 Mt/year and further announced an expansion of 600,000 tonnes/y to the Green River region of Wyoming, USA and 200,000 tonnes/y at Devnya, Bulgaria in 2019. Among these, nearly 73% of soda ash productions adopted the Solvay process [3]. Ammoniasoda process uses ubiquitous limestone ($CaCO_3$), industrial salt (NaCl), and common ammonia gas (NH_3) as raw materials [4], [5]. In Uzbekistan, the main producer of soda ash is the Kungrad soda plant, located in the Republic of Karakalpakstan in the village of Eleabad.

Figure 1. Above view of liquid waste from soda production (according to Google Earth)

Currently, Uzbekistan's needs for soda ash are estimated at approximately 60-70 thousand tons per year. Currently, industry uses the Solvay method to produce soda ash.The Solvay Process, named after its inventor Ernest Solvay (1838–1922), aims at the production of soda ash (sodium carbonate, Na_2CO_3), which is a major commodity and an essential raw product for many industrial applications (above all: the production of glass) and even used in household applications (e.g. detergents). Thus, the Solvay Process is one of the most important inorganic chemical processes. Where no natural sodium carbonaceous minerals (e.g. trona, $Na_2CO_3{\cdot}NaHCO_3{\cdot}2H_2O$; or nahcolite, $NaHCO_3$) or natural occurring sodium carbonate-bearing brines [6] are available, there is no real alternative to the production of soda ash in the Solvay Process. In 2000, 59 % of the worldwide soda ash production was synthesized in the Solvay Process, 30 % were produced by processing natural sodium carbonate minerals, and 11 % were produced using other methods [7]. An important aspect of the Solvay process (the most popular process of synthetic soda ash production) is the recovery of ammonia from the filtration liquid (with the aim of calcium hydroxide): $2NH_4Cl+Ca(OH)_2 \rightarrow 2NH_3\uparrow+2H_2O+CaCl_2$ [8]. Soda sludge (SS), which is composed of $CaCO_3$, $Ca(OH)_2$, $CaSO_4{\cdot}0.5H_2O$, and NaCl, etc., is generated from the production of sodium carbonate in the industry [9]. Its yield is reported to be 50–80 million tons annually all over the world [10], [11].The ammonia soda process waste stream is the distillation waste stream produced by the ammonia soda process [12], [13]. The Solvay process is a well-known process used for soda ash synthesis, using sodium chloride, and limestone as raw materials in the presence of a basic medium (ammonia). Recovery of ammonia is considered the most significant step. So in order to achieve this step, the filtrate has to be treated with calcium hydroxide suspension, and the recovered ammonia is recycled back into the process. The resulting suspension (Solvay Wastewater) is considered the main waste stream of the Solvay process [14]. This waste stream, which contains $CaCl_2$ (main constituent, concentration reaches 120 g/l [15], unprocessed NaCl, $CaCO_3$, $Ca(OH)_2$ and other substances), creates a huge problem for the ammonia-soda plants since waste liquids in most cases are directly discharged as sewage to the nearest river, lake, or sea. Therefore, it leads to environmental pollution causing serious health hazards and waste of resources[16]. Ammonia-soda process uses ubiquitous limestone ($CaCO_3$), industrial salt (NaCl), and common ammonia gas (NH_3) as raw materials [17]. The schematic diagram of the entire ammonia-soda process. Firstly, solution mining of salt deposits is prepared as original brine, thereafter sent to absorption tower with ammonia gas. Meanwhile, limestone is calcined in lime kiln, providing CO_2 for the carbonation of ammonia-saturated brine in carbonating tower. Herein, solutions of sodium bicarbonate ($NaHCO_3$) and ammonia chloride (NH_4Cl) are formed, whose crystals are separated from filtered fluid by centrifuges or vacuum filters. NaCl + NH_3 + H_2O + CO_2 → $NaHCO_3$ ↓ + NH_4Cl

Afterwards, the crude bicarbonate decomposes to sodium carbonate and carbon dioxide is sent to a calcinator. $2NaHCO_3 \rightarrow Na_2CO_3 + H_2O + CO_2\uparrow$

The special part of the ammoniasoda process lies in the recovery of ammonia gas from the filtered fluid. To obtain ammonia gas, lime milk was prepared in advance of mixing the liquid in a blender, which could be produced by treating the calcined lime from the burning of limestone in a hydrator. $CaO + H_2O \rightarrow Ca(OH)_2$. The filtered fluid contains NH_4Cl, which is sent to a distillation tower together with $Ca(OH)_2$ solution. The evolved ammonia gas is vented back into the absorption tower in the first step, achieving the simple recycle of ammonia. $Ca(OH)_2 + 2NH_4Cl \rightarrow CaCl_2 + 2H_2O + 2NH_3\uparrow$ However, distiller waste is produced at the same time, whose effluent is commonly pumped to setting basins and solid wastes are deposited accumulatively, causing severe environmental issues [18]. The Solvay method is inexpensive, widespread and easy to obtain.[19], [20]. The reactions are carried out at low temperatures and close to atmospheric pressure.The method is well studied, the technological processes are debugged and stable [21]. The resulting soda ash is of high quality at a relatively low cost. For 1 ton of soda ash produced, there is approximately 9–10 m^3 of distiller liquid. Soda production waste, sludge and distiller liquid, are stored in special storage tanks called “white seas”[22]. This affects the environment.[23] sewage sludge is rich in pathogenic microorganisms and toxic pollutants, with the potential to cause serious risks to health. In order to acquire class A solids, and fulfill the demands of the Environmental Protection Agency, sludge must be stabilized and detoxified prior to its final disposal, or use for land application [24]. As a solution for the production of calcium peroxide, we used a model liquid corresponding in composition to the main waste from the production of soda ash from the Kungrad soda plant (distiller liquid), having the following composition per 1 liter - 115.4 g $CaCl_2$, 67.6 g NaCl, 1.11 g $Ca(OH)_2$, 0.596 g $CaCO_3$, 0.993 g Na_2SO_4 the rest H_2O. In the proposed utility model, in contrast to known methods, a smaller amount of initial reagents is used, since the molar ratio of $CaCl_2$ ⍰ H_2O_2 is used equal to 1:1, and from experiments using the proposed method it follows that an increase in the molar ratio does not lead to a significant increase in the yield of calcium peroxide CaO_2. Calcium peroxide CaO_2 : Used in food, beverage, agriculture and cosmetics. Calcium peroxide (CaO_2) is a widely used agent toward contaminants removal in soil and groundwater due to its strong oxidizing properties and basicity [25]. The high oxidative potential of calcium peroxide allows it to be used for water purification and disinfection. Calcium peroxide is an oxidizing agent in the rigid form of hydrogen peroxide [26]. Calcium peroxide which is a powerful chemical agent known for its thermal stability, versatility, ecofriendly nature. It is recognized as an efficient solid source of hydrogen peroxide [27]. Calcium peroxide (CaO_2) has attracted increasing interest recently due to its excellent ability of oxygen supplement and outstanding biocompatibility [28], [29]. The additive is excluded from food production, but is widely used in poultry farming, pharmaceuticals, and agriculture.

2. Materials and Methods

The instruments used for the study were IR-Fourier spectrometer Japan, Shimadzu “IRAffinity-1”, high-performance, energy-dispersive, X-ray, fluorescence spectrometer - Japan, Rigaku NEX CG EDXRF and distiller liquid of the Kungrad soda plant,H_2O_2(60%). Measurements of the phase characteristics of the sample under study were carried out using a Panalytical Empyrean powder X-ray diffractometer. All control over the operation of the equipment is carried out via a computer using the Data Collector program, and the analysis of radiographs was carried out using the High Score program with a PDF 2013 database. X-ray phase analysis of the studied samples was performed on a Panalytical Empyrean X-ray diffractometer equipped with a Cu tube (K□1= 1.5406 Å). The measurements were carried out at room temperature in the range of angles 2□, in the range from 5° to 90° in step-by-step scanning mode with a step of 0.013 degrees and a signal accumulation time at a point of 5 s. The study of reflection and light transmission used the Eye one pro X.Rite device.

2.1. Paper Title

Preparation of calcium peroxide from liquid waste of the Kungrad soda plant and use in the pulp industry

2.2. Authorship

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2.3. Abstract & Keywords

Abstract. Soda ash is the main product of the chemical industry, which is used in glass production, the metallurgical, the pulp and paper industry and other. Currently,industry uses the Solvay method to produce soda ash. But the main disadvantage of the Solvay method is the formation of a large amount of liquid waste,the so-called distillation liquid. The problem is solved by the fact that with continuous stirring of the distiller liquid by a circulation pump of a thermostabilized reactor volume, a 55% solution of hydrogen peroxide is introduced into the distiller liquid at a temperature of 4 °C, bringing the pH to 5.3, then ammonia gas is bubbled into the distiller liquid to achieve the precipitation of calcium peroxide hydrate from the solution at pH up to 9.15, followed by separation of calcium peroxide hydrate on the filter surface, washing of the precipitate and dehydration of the precipitate at 120-130 °C . This method can be used at soda ash production plants to recycle soda ash production waste (distiller liquid) to produce calcium peroxide CaO_2 . The proposed method allows, by using gaseous ammonia and combining the functions of mixing and transporting distillation liquid using a circulation pump, to reduce the precipitation time of calcium peroxide hydrate to 1 minute and thereby increase the amount of commercial calcium peroxide by reducing its dissolution in the resulting ammonium chloride solution in a heat-stabilized volume reactor.

Keywords: Solvay method, distiller liquid, sentrifugat, calcium peroxide, liquid waste

2.4.1. Figures

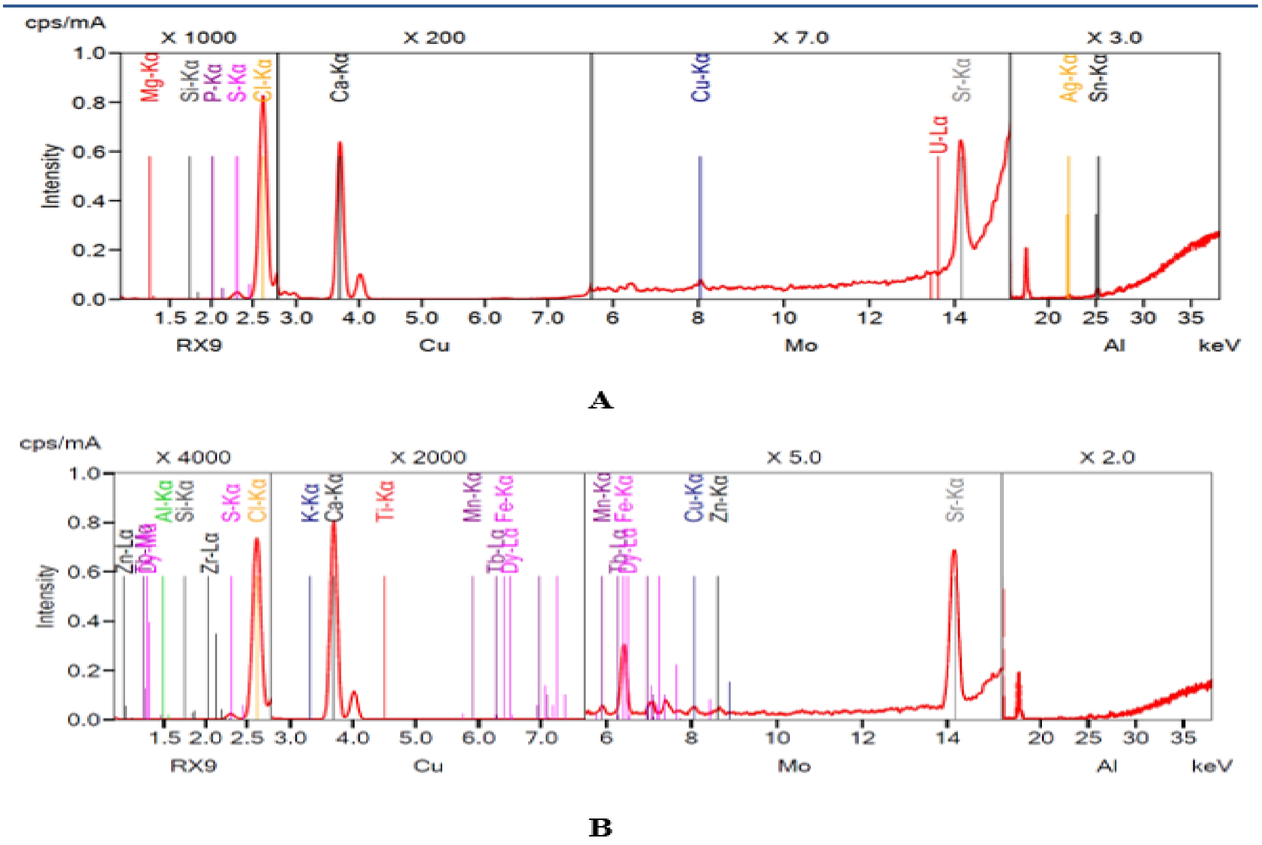

Figure 1. *:* A-Energy-dispersive X-ray fluorescence spectrum centrifugate of distiller liquid;
B- Energy-dispersive X-ray fluorescence spectrum of calcium peroxide obtained from liquid waste

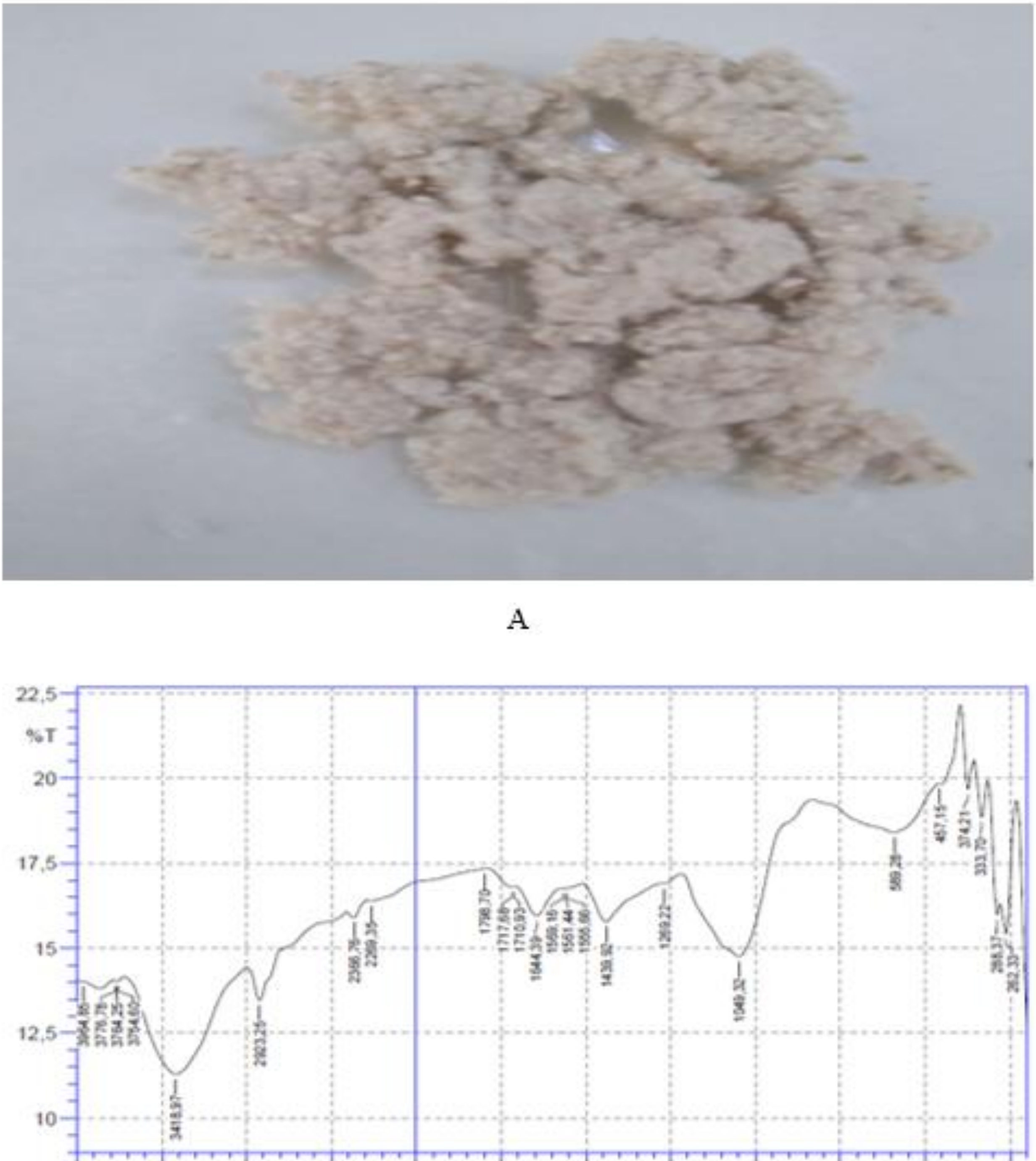

Figure 2: *A*-Source cellulose; *B*-Infrared spectrum of the source cellulose

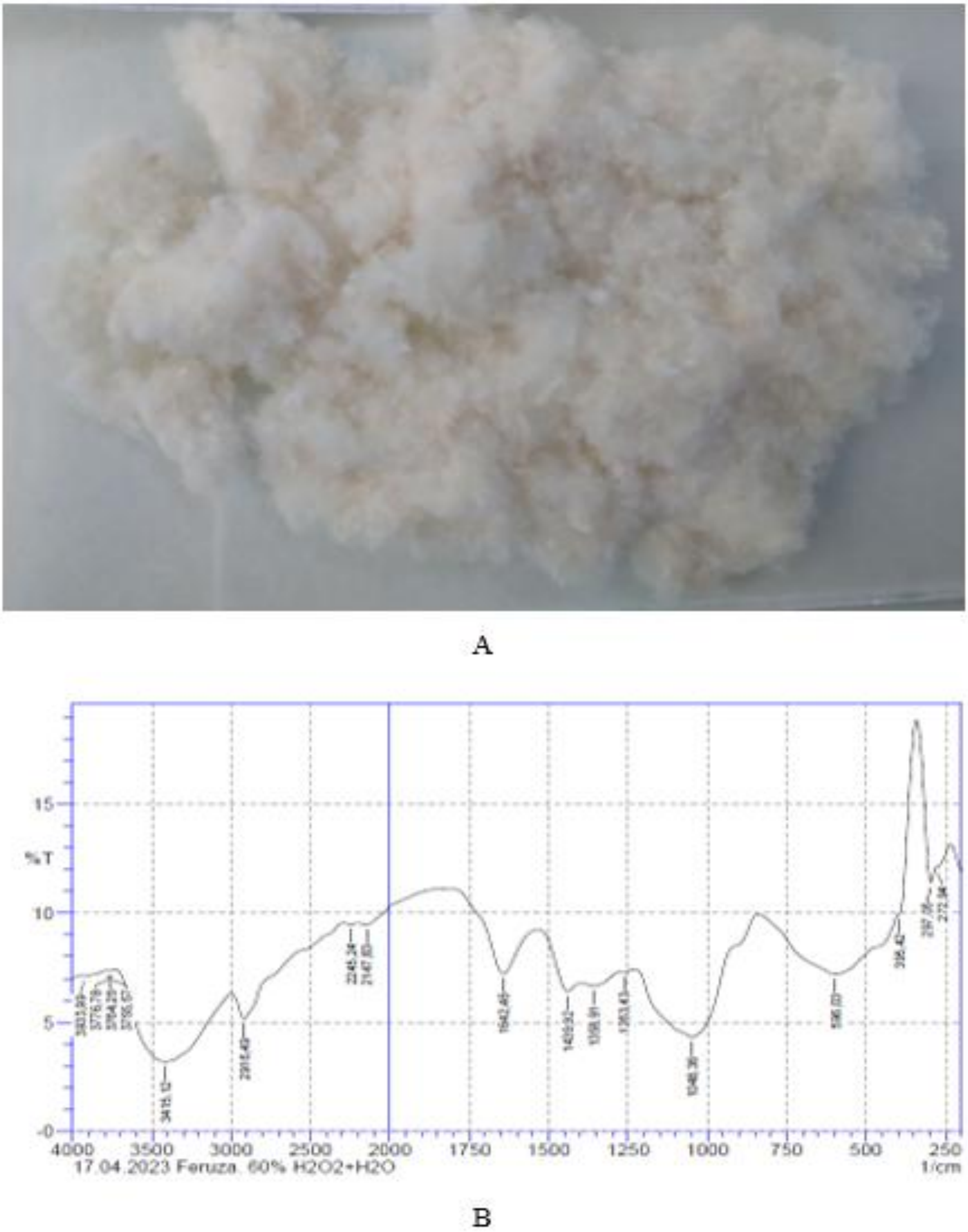

Figure 3: A-Bleached cellulose with calcium peroxide (60%H_2O_2+waste);
B- Infrared spectrum of the bleached cellulose with calcium peroxide (60%H_2O_2+waste)

Table 1. Elemental composition of calcium peroxide obtained from liquid waste from the Kungrad soda plant

№	Component	S%	Cl%	Ca%	Cu%	Zn%	Sr%	H_2O%
1	Centrifugate Dis.liquid	0.17	9.01	3.96	0.000517	0.000482	0.00163	86.8
2	calcium peroxide	0.359	6.03	42.4	0.0023	0.0015	0.0044	51.2028

Table 2. Mineral composition of liquid waste from the Kungrad soda plant

№	Mineral composition	quantity
1.	NaCl	0.155
2.	$CaCl_2$	0.028
3.	SrO	0.357
4.	ZnO_2	0.051
5.	$CaCO_3$	0.155
6.	$CaCl_2$	0.057
7.	Fe_2S_3	0.019
8.	FeS	0.010

3. Result

This problem is solved by the fact that with continuous stirring of the distiller liquid by a circulation pump of a thermostabilized reactor volume, a 55% solution of hydrogen peroxide is introduced into the distiller liquid at a temperature of 4°C, bringing the pH to 5.3, then ammonia gas is bubbled into the distiller liquid to achieve the precipitation of calcium peroxide hydrate from the solution at pH to 9.15, followed by separation of calcium peroxide hydrate on the filter surface, washing of the precipitate and dehydration of the precipitate at 120-130 °C. Pour 16 ml of liquid waste + NH_3 16;24;32;40;48 into a 250 ml cylindrical glass. H_2O_2 (60%) 10 ml and stir in a magnetic stirrer for 10 minutes at a speed of 1100. A white suspension is formed and elemental analysis was studied.It should be noted that the reaction time greatly affects the yield of the product. In the reaction time of up to 1.5 minutes, the highest content of the resulting calcium peroxide hydrate CaO_2 in the sediment is observed. Increasing the reaction time leads to a decrease in the yield of calcium peroxide CaO_2. This is caused by the fact that calcium peroxide CaO_2 dissolves in the resulting ammonium chloride solution NH_4Cl. Thermal stabilization of the reactor volume around 4 °C also leads to an increase in the yield of calcium peroxide CaO_2 in the sediment.

4. Conclusion

The proposed method allows, by using gaseous ammonia and combining the functions of mixing and transporting distillation liquid using a circulation pump, to reduce the precipitation time of calcium peroxide hydrate to 1 minute and thereby increase the amount of commercial calcium peroxide by reducing its dissolution in the resulting ammonium chloride solution.The choice of the sequence of use of ingredients and their quantitative characteristics are based on data obtained from control (laboratory) tests. The use of a temperature of 4 °C is optimal from a technological point of view due to the ease of control of thermal stabilization, while the use of lower temperatures leads to an increase in the cost of the technology.The use of ammonia gas and a reaction time of up to 1 minute is optimal, since it is with this ratio that the maximum positive result is achieved - maximizing the yield of calcium peroxide hydrate, which, in turn, allows solving the underlying problem. proposed technology– saving the consumption of ammonia and hydrogen peroxide for the production of calcium peroxide.

5. Acknowledgements

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