STUDY OF THE PROCESSES OF SEPARATION OF SOLID AND LIQUID PHASES IN THE PROCESS OF CONVERSION

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Abstract. This research investigates the critical processes involved in solid-liquid phase separation during potassium sulfate production through chemical conversion methods. The study examines the settling and filtration characteristics of suspensions formed during two-stage conversion processes, focusing on the clarification rates and filtration efficiency under various operational conditions. Experimental investigations were conducted using graduated cylinders and Buchner funnel systems to determine optimal parameters for phase separation. The results demonstrate that suspension clarification degrees reach 65.8% to 67.3% within 95 seconds for the first stage, while the second stage achieved maximum clarification of 66.25%. Filtration rates varied significantly between stages, with values of 1000-1200 kg/m²·h for the first stage and 2100-2300 kg/m²·h for the second stage. The influence of molar ratios (KCl:$(NH_4)_2SO_4$), temperature (20-60°C), and sediment height on filtration performance was systematically analyzed. The study reveals that filtration rates improve with increasing molar ratios and temperature, though the effect diminishes at higher sediment heights. These findings provide valuable insights for industrial scale-up and process optimization in potassium sulfate manufacturing.

Keywords. *Potassium sulfate, phase separation, solid-liquid separation, filtration kinetics, clarification, suspension rheology, conversion process, double salt formation, ammonium sulfate, potassium chloride, glaserite, industrial crystallization*

Introduction. The production of potassium sulfate (K_2SO_4) through chemical conversion represents a cornerstone process in the global fertilizer industry, with significant implications for agricultural sustainability and food security [1]. The conversion method, particularly involving the interaction between potassium chloride (KCl) and ammonium sulfate ($(NH_4)_2SO_4$), has gained considerable attention due to its economic viability and environmental benefits compared to alternative production routes [2]. However, one of the most challenging aspects of this production method involves the efficient separation of solid and liquid phases, which frequently becomes the rate-limiting step in the overall conversion process [3].

The complexity of solid-liquid separation in potassium sulfate production stems from the formation of various intermediate compounds, including double salts and mixed crystals, which exhibit distinct physical and chemical properties [4]. These intermediate phases significantly influence the separation efficiency, as they affect particle size distribution, suspension stability, and filtration characteristics [5]. Understanding these fundamental mechanisms is crucial for optimizing industrial processes and achieving desired product specifications while maintaining economic competitiveness.

The conversion process typically involves multiple stages, each presenting unique challenges in terms of phase separation due to varying particle morphologies, densities, and chemical compositions of the formed suspensions [6]. The first stage generally involves the formation of intermediate compounds such as glaserite ($K_3Na(SO_4)_2$) or potassium ammonium sulfate, while the second stage focuses on the conversion of these intermediates to the final potassium sulfate product [7]. Each stage requires tailored separation strategies to achieve optimal performance.

Industrial experience has demonstrated that inadequate phase separation can lead to significant economic losses through reduced product yield, increased processing time, and elevated operational costs [8]. Furthermore, poor separation efficiency often results in product contamination and reduced purity levels, which can negatively impact the commercial value of the final product [9]. Therefore, comprehensive understanding and optimization of solid-liquid separation processes are essential for successful industrial implementation.

Literature Review

Previous investigations in the field of potassium sulfate production have primarily focused on the thermodynamic and kinetic aspects of conversion reactions, with relatively limited attention given to the physical separation processes [10]. Research conducted by Zhang et al. [11] demonstrated that the efficiency of solid-liquid separation significantly influences the overall process economics and product quality in sulfate-based conversion systems. Their findings indicated that separation efficiency improvements of even 5-10% could result in substantial economic benefits at industrial scale.

Historical approaches to phase separation in potassium sulfate production have relied heavily on conventional settling and filtration techniques [12]. However, the unique characteristics of the suspensions formed during conversion processes require specialized understanding of their rheological properties and settling behaviors [13]. The formation of double salts during intermediate stages introduces additional complexity to the separation process, as these compounds exhibit markedly different physical properties compared to the final products [14].

Recent developments in separation technology have emphasized the importance of understanding particle size distribution, suspension stability, and the effects of temperature and concentration on separation efficiency [15]. Kumar and Patel [16] investigated the influence of ionic strength on particle aggregation behavior, demonstrating that the interaction between different ionic species in the solution phase can significantly affect the settling characteristics of solid particles. Their work revealed that optimal ionic conditions could enhance settling rates by up to 40%.

The rheological behavior of conversion suspensions has been studied by Rodriguez et al. [17], who found that the apparent viscosity of these systems depends strongly on solid concentration, particle size distribution, and solution chemistry. Their research indicated that proper control of these parameters could lead to significant improvements in both settling and filtration performance.

Furthermore, the economic considerations associated with filtration equipment selection and operation have become increasingly important in industrial applications [18]. The balance between capital investment in separation equipment and operational costs must be carefully evaluated to achieve optimal process performance while maintaining economic viability [19].

Research Methodology

The experimental investigation was designed to systematically evaluate the solid-liquid separation characteristics under controlled laboratory conditions. The study employed a multi-stage approach to examine different aspects of the separation process, including both settling and filtration behaviors.

Clarification experiments were conducted using 100 ml graduated cylinders with precise height markings throughout their entire length. The experimental setup allowed for continuous monitoring of the suspension-clear liquid interface over time. All experiments were performed at ambient temperature (25°C) to ensure reproducible conditions and minimize temperature-related variations in suspension behavior [20].

The degree of clarification was calculated using the following equation:

Clarification Degree (%) = (Height of clear liquid/Total height)×100

Time-dependent measurements were recorded at regular intervals to establish clarification kinetics for different suspension types. The experiments were conducted for both Stage I (conversion of potassium chloride with ammonium sulfate) and Stage II (potassium sulfate extraction) suspensions.

Filtration experiments were performed using Buchner funnels with a filtering surface area of 0.002 m^2 for initial studies and 0.005 m^2 for detailed parametric investigations. The vacuum pressure was maintained at 400 mmHg for comparative studies and 300 mmHg for parametric analysis. Filtration rates were calculated using the following formula:

Results and Discussion

One of the limiting stages of the conversion process in obtaining potassium sulfate is phase separation. In this regard, studies were conducted to determine the rate of their settling and filtration.

Studies on the degree of clarification of suspensions at stages I and II were carried out in a 100 ml graduated cylinder with divisions along the entire height in cm at an ambient temperature of 25°C.

Figures 1 and 2 show the obtained data on the degree of clarification depending on time. The obtained data indicate good separation of the suspensions formed during the conversion of potassium chloride. Thus, the degree of clarification of the suspension based on potassium chloride and sodium sulfate after 95 seconds reaches 65.8%, with a maximum degree of clarification of 67.3%, while the degree of clarification in the second stage for the same period of time reached the maximum value - 66.25%.

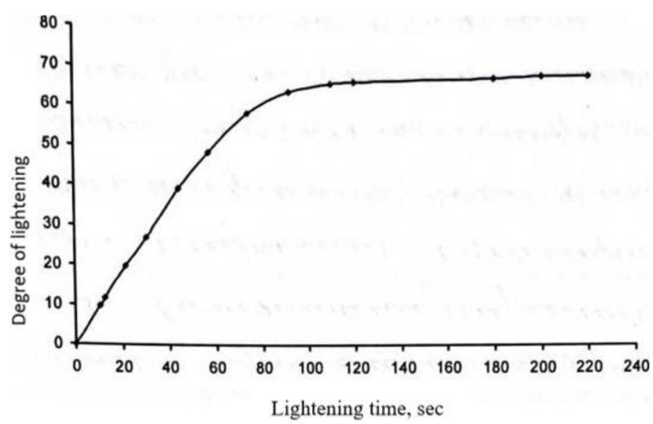

Fig. 1. Change in the degree of suspension clarification depending on time during the production of glaserite for a conversion time of 60 min.

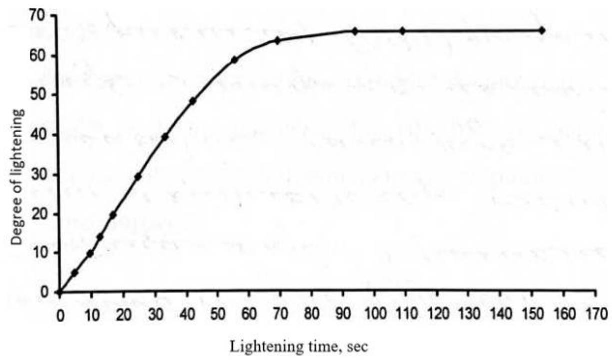

Fig. 2. Change in the degree of suspension clarification depending on the duration of the process

The rate of suspension clarification at the stage of potassium sulfate extraction is twice as fast. This indicates that the obtained products potassium ammonium sulfate and potassium sulfate are homogeneous, do not contain foreign impurities and are very well thickened.

I and II was studied on a Buchner funnel at a vacuum 400 mm of mercury, recording the filtration time. The area of the filtering surface is 0,002 m^2.

The obtained results on filtration were identical to the degree of clarification of suspensions. The filtration rate at the second stage is approximately twice as high as at the first stage and averages 1000-1200 $kg/m^2.h$ and 2100-2300 kg/m2.h, respectively, for the stage of conversion of potassium chloride with sulfate ammonium and the stage of potassium sulfate release.

Table 1 shows the filtration rates of the double salt suspension depending on the initial molar ratio of KCl $(NH_4)_2SO_4$ and the temperature of the filtration process. With an increase in the molar ratio from 1:1 and the temperature from 20 to 60 °C, the filtration rate for both the pulp and the filtrate and the solid phase increases slightly. Thus, if at a molar ratio of KCl $(NH_4)_2 SO_4$ = l:l the filtration rate for pulp increases from 7571.95 kg/m^{2} - h to 7839.52 kg/m^{2} - h, respectively, with an increase in temperature from 20 to 60 °C, then with an initial component ratio of 1:1.5, these indicators change from 8169.74 to 8458.45 kg/m^{2} - h. With the height of the sediment, the filtration rate 6 ммof the suspension decreases significantly and is 3815.89-3882.68 kg/m^2 - h at a molar ratio of KCl:Na_2SO_4 = l :1 in the temperature range from 20 to 60°C, and at the initial at a component ratio of 1:2 is 4418.41-4495.74 $kg/m^2.h$, i.e. no noticeable improvement in filtration rate is observed with an increase in the molar ratio and temperature.

Table 1. The influence of the ratio KCl $(NH_4)_2SO_4$ and temperature on the filtration rate of a double salt suspension at S:L = 1:1

№	Ratio KCI:Na_2SO_4	Temperature, °C	Filtration rate, kg/m^2 h		
			by pulp	by solid phase	by filtrate
			h = 0 mm		
1	1: 1,0	20	7571.95	3849.66	3722.29
		40	7707.04	3953.71	3753.33
		60	7839.52	4021.67	3817.85
2	1: 1,5	20	8169.74	4191.07	3978.67
		40	8315.49	4265.84	4049.65
		60	8458.45	4339.18	4119.27
3	1: 1,7	20	8435.42	4327.37	4108.05
		40	8585.91	4404.57	4181.34
		60	8733.52	4480.29	4253.23
4	1: 2,0	20	8767.52	4497.73	4269.79
		40	8923.94	4577.98	4345.96
		60	9077.36	4656.68	4420.68
h = 6 mm					
5	1: 1,0	20	3815.89	1957.55	1858.34
		40	3849.90	1974.99	1874.91
		60	3882.68	1991.81	1890.87
6	1: 1,5	20	4117.15	2112.09	2005.06
		40	4153.84	2130.91	2022.93
		60	4189.21	2149.06	204015
7	1:1,7	20	4251.04	2180.78	2070.26
		40	4288.93	2200.22	2088.71
		60	4325.44	2218.95	2106.49
8	1:2,0	20	4418.41	2266.64	2151.77
		40	4457.78	2286.84	2170.94
		60	4495.74	2306.31	2189.43

Conclusion

This comprehensive investigation of solid-liquid phase separation in potassium sulfate conversion processes has provided valuable insights into the fundamental mechanisms governing separation efficiency. The study demonstrated that Stage II suspensions exhibit superior separation characteristics compared to Stage I, with clarification degrees reaching 66.25% and filtration rates of 2100-2300 kg/m²·h. The parametric analysis revealed that increasing molar ratios and temperature positively impact filtration performance, with optimal conditions achieved at KCl:$(NH_4)_2SO_4$ ratios of 1:2.0 and temperatures of 60°C. However, sediment accumulation significantly impairs filtration efficiency, reducing rates by approximately 50%. These findings provide crucial information for industrial process optimization and equipment design in potassium sulfate manufacturing facilities.

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