OPTIMIZATION OF CHEMICAL PROCESS PARAMETERS FOR DEFOLIANT PRODUCTION USING SODA INDUSTRY BY-PRODUCTS.

Majidov Hayotjon Bakhtiyor ugli

E-mail: majidovhayot532@gmail.com

Doctoral student of the Department of Chemical Technologies at Navoi State University of Mining and Technology

Pardayev Ulugbek Khairullo ugli

E-mail: pardayevulugbek125@gmail.com

A student of the Chemistry program at the Faculty of Natural Sciences, Uzbekistan-Finland Pedagogical Institute.

Toshmurotova Pokiza Farhod kizi

E-mail: farxodkarabekov967@gmail.com

A student of the Chemistry program at the Faculty of Natural Sciences, Uzbekistan-Finland Pedagogical Institute.

Khusanov Eldor Safariddinovich

Doctor of Philosophy (PhD) in Technical Sciences, Senior Lecturer at the Department of Chemistry, Faculty of Natural Sciences, Uzbekistan-Finland Pedagogical Institute.

Annotation: This study presents the optimization of chemical process parameters for the synthesis of defoliants utilizing secondary by-products from the soda industry. Industrial residues such as calcium chloride, sodium carbonate, and sulfur-containing filtrates were selected as primary reactants. The experimental work focused on identifying optimal reaction temperature, pH, molar ratios, and reaction time to maximize defoliation efficiency and minimize environmental impact. A response surface methodology (RSM) was applied to evaluate the effect and interaction of multiple process variables on the yield and biological performance of the synthesized defoliants. The results demonstrated that a reaction temperature of 75–80°C, pH 6.5–7.5, and a molar ratio of 1:2 ($CaCl_2$ to sulfur source) provided the highest defoliation rates with minimal by-product formation.

Key words: Defoliants; Process optimization; Soda industry by-products; Chemical synthesis; Green chemistry; Calcium chloride; Sulfur compounds; Response surface methodology (RSM); Sustainable agrochemicals; Circular economy.

Introduction: The global agricultural sector increasingly demands efficient and environmentally sustainable agrochemicals, including defoliants that enhance harvesting efficiency and crop quality. Conventional defoliants, though effective, often rely on expensive synthetic routes and petrochemical feedstocks, raising both economic and ecological concerns. As a result, research into alternative

synthesis strategies using industrial by-products has gained momentum within the framework of green chemistry and the circular economy.

The soda industry, particularly through processes such as the Solvay method, produces substantial volumes of secondary waste materials—including calcium chloride ($CaCl_2$), sodium carbonate (Na_2CO_3), and sulfur-rich filtrates—that are often underutilized or discarded. These inorganic residues possess promising chemical reactivity and structural potential for use as precursors in agrochemical synthesis. However, the practical conversion of such by-products into high-performance defoliants requires careful process design and optimization to ensure both efficacy and safety.

This study aims to optimize the chemical synthesis parameters for producing effective defoliants using soda industry waste streams. By systematically varying reaction conditions—such as temperature, pH, molar ratio, and reaction time—the study seeks to identify optimal settings that maximize defoliant yield and biological activity. Response Surface Methodology (RSM) is employed as a robust statistical tool to model the interactions between process variables and assess their impact on product performance.

The novelty of this research lies in its integration of waste valorization with process optimization to develop cost-effective, scalable, and eco-friendly defoliant production technologies. The study contributes to the growing body of literature on sustainable agrochemical innovation and industrial ecology by offering a viable route for converting waste into high-value agricultural inputs.

Literature review: Defoliants have played a pivotal role in mechanized agriculture by facilitating the uniform shedding of leaves before harvest, particularly in cotton and other fiber crops. Conventional defoliants such as thidiazuron, ethephon, and tribufos have been widely used, but their chemical synthesis often involves non-renewable feedstocks, multistep reactions, and hazardous intermediates, making their production costly and environmentally burdensome.

To address these limitations, recent research has focused on the application of green chemistry principles to agrochemical synthesis. One promising approach involves the utilization of industrial by-products, which not only reduces production costs but also aligns with sustainability goals (Tiwari & Singh, 2018). Among various industrial waste sources, the soda industry generates substantial quantities of chemically active by-products such as calcium chloride, sodium bicarbonate residues, and sulfur-rich filtrates. These substances are often regarded as waste, but several studies have shown their potential for reuse in construction materials, water treatment, and chemical manufacturing.

Despite growing interest in waste-based synthesis, the specific application of soda industry residues in defoliant production remains underexplored. Limited studies suggest that calcium and sulfur-based compounds can exhibit bioactive properties relevant to plant physiology, potentially disrupting leaf retention mechanisms. However, these studies have rarely included a systematic process optimization framework, such as Response Surface Methodology (RSM), to fine-tune reaction parameters and enhance product performance.

Optimization techniques such as RSM and factorial design have proven valuable in chemical engineering applications, allowing researchers to assess the influence of multiple variables and their interactions with minimal experimental trials. When applied to agrochemical development, these tools enable data-driven decisions to improve product yield, stability, and effectiveness while reducing environmental and economic costs.

This review highlights the gap in current literature regarding the integration of soda industry by-products into optimized defoliant synthesis protocols. The present study aims to address this gap by combining waste valorization with systematic parameter optimization, offering a novel and sustainable pathway for agrochemical innovation.

Methodology: Secondary by-products from soda ash production—primarily calcium chloride ($CaCl_2$), sodium carbonate (Na_2CO_3), and sulfur-containing filtrates—were obtained from a local soda industry plant. These materials were preliminarily purified through sedimentation, filtration, and drying processes to remove suspended solids and impurities. The chemical composition of each by-product was confirmed using X-ray fluorescence (XRF) analysis.

To identify the most favorable reaction conditions for defoliant synthesis, a central composite design (CCD) within the framework of Response Surface Methodology (RSM) was employed. The four main process variables selected were:

- Reaction temperature (60–90°C)
- Reaction time (2–6 hours)
- Molar ratio of $CaCl_2$ to sulfur source (1:1 to 1:3)
- pH of the reaction medium (adjusted from 5.5 to 8.5 using dilute HCl or NaOH)

Statistical software (Design-Expert® 13) was used to generate the experimental matrix and analyze interactions between variables. A total of 30 experimental runs were conducted.

Batch reactions were performed in 500 mL glass reactors equipped with magnetic stirrers and temperature control. Pre-weighed amounts of $CaCl_2$ and sulfur filtrate were mixed under continuous stirring. The pH was adjusted prior to heating, and the reaction was maintained under constant conditions based on the experimental design. The resulting product was cooled, filtered, washed with distilled water, and dried at 60°C for 12 hours.

Greenhouse experiments were conducted using cotton plants (*Gossypium hirsutum* L.) to assess the biological activity of each synthesized defoliant. Plants were sprayed with aqueous formulations of the compounds, and defoliation percentage was recorded after 72 hours. Control groups (untreated and commercial defoliant-treated) were included.

Results: The optimization of defoliant synthesis using soda industry by-products yielded significant insights into the influence of chemical process parameters on product performance and efficiency. Based on the central composite design, the experimental data revealed that both the molar ratio of calcium chloride to sulfur source and the reaction temperature had the most significant effects on the defoliation rate ($p < 0.01$). The optimal formulation was achieved at a molar ratio of 1:2, reaction temperature of 78°C, reaction time of 4.5 hours, and pH 6.8, which resulted in a defoliation rate of 86.4% within 72 hours of application. At these conditions, the synthesized compound exhibited minimal phytotoxicity and favorable application properties. In the bioassay trials, the synthesized defoliant performed comparably with commercial standards. Plants treated with the optimized formulation showed over 85% leaf abscission within 72 hours, while exhibiting no boll damage or visual phytotoxicity. In contrast, the commercial defoliant showed 78% defoliation with moderate leaf tip necrosis in 25% of treated samples. The environmental safety assessment revealed that the synthesized product left negligible residues in soil (0.6 mg/kg) and showed low aquatic toxicity, with Daphnia magna LC_{50} values exceeding 100 mg/L, suggesting minimal ecological risk.

Table 1. Optimal Process Parameters.

№	Parameter	Optimal Value
1	Molar Ratio ($CaCl_2$: Sulfur Source)	1:2

2	Reaction Temperature (°C)	78
3	Reaction Time (hours)	4.5
4	pH	6.8

Table 2. Defoliation Efficiency Comparison.

№	Treatment	Defoliation Rate (%)	Leaf Tip Necrosis Observed	Boll Damage
1	Synthesized Defoliant	86.4	No	None
2	Commercial Defoliant	78.0	Yes (25%)	Moderate

Table 3. Environmental Safety Indicators.

№	Parameter	Synthesized Defoliant	Commercial Defoliant
1	Soil Residual Toxicity (mg/kg)	0.6	2.4
2	*Daphnia magna* LC_{50} (mg/L)	102.5	48.7

Table 4. Statistical Model Summary.

№	Model Metric	Result
1	Significant Parameters	Molar Ratio, Temperature
2	R^2 Value	0.964
3	*p*-value (Molar Ratio)	< 0.01
4	*p*-value (Temperature)	< 0.01

Discussion: The results of this study highlight the effectiveness of using soda industry by-products as a sustainable and economically viable resource for defoliant synthesis. The application of Response Surface Methodology (RSM) provided a robust statistical framework for understanding the influence of key process variables on defoliant performance. Among the parameters investigated, the molar ratio of calcium chloride to sulfur-containing filtrate and the reaction temperature emerged as the most influential factors affecting defoliation efficiency, as confirmed by ANOVA analysis ($p < 0.01$).

The optimal synthesis conditions—molar ratio of 1:2, temperature of 78°C, reaction time of 4.5 hours, and pH 6.8—resulted in a high defoliation rate of 86.4% within 72 hours of application. These findings suggest that controlling stoichiometric balance and thermal energy input is critical for promoting the formation of bioactive compounds capable of inducing leaf abscission. The absence of leaf tip necrosis and boll damage further indicates that the synthesized defoliant is both effective and crop-safe under optimal conditions.

Importantly, the synthesized defoliant demonstrated superior environmental performance compared to commercial formulations. Soil residue levels were significantly lower (0.6 mg/kg), and aquatic toxicity testing showed high LC_{50} values (>100 mg/L for *Daphnia magna*), indicating minimal ecological hazard. These advantages are attributed to the inorganic origin and low persistence of the active components, as well as the controlled synthesis pathway employed.

From an industrial perspective, the process aligns well with circular economy principles by valorizing waste materials and reducing dependency on synthetic raw materials. The high R^2 value (0.964) of the

predictive model confirms that the optimization process was statistically sound and can be reliably applied to scale-up studies. Moreover, the simplified and energy-moderate reaction conditions make this approach attractive for commercial adoption in sustainable agrochemical production.

Conclusion: This study successfully demonstrated the feasibility of synthesizing effective defoliants from soda industry by-products through a statistically optimized chemical process. By applying Response Surface Methodology (RSM), key parameters such as molar ratio, reaction temperature, time, and pH were optimized to maximize defoliation efficiency while ensuring environmental safety and product quality. The optimal formulation—1:2 molar ratio, 78°C, 4.5 hours, and pH 6.8—resulted in a defoliation rate of 86.4% with no adverse phytotoxic effects on cotton crops.

Economically and ecologically, this approach supports sustainable agriculture and circular economy goals by transforming industrial waste into valuable agrochemical inputs. The strong predictive power of the statistical model ($R^2 = 0.964$) further confirms the reliability of the optimized process for potential industrial scale-up. Future research should explore field-scale applications and long-term environmental interactions to validate commercial implementation.

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