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DEVELOPMENT OF EFFECTIVE AGROCHEMICAL AGENTS THROUGH THE CHEMICAL MODIFICATION OF SODA INDUSTRY WASTES

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Annotation. *This article explores the development of effective agrochemical agents derived from chemically modified soda industry wastes. The study focuses on converting carbonate-rich residues into value-added agricultural products through targeted chemical treatments, including acid activation and oxidative modification. The resulting materials were analyzed for structural and functional characteristics using FTIR and XRD techniques. Preliminary field trials demonstrated improved efficacy in promoting leaf abscission and soil conditioning without phytotoxic effects. The approach supports sustainable agriculture and waste valorization, offering an environmentally friendly alternative to conventional agrochemicals. This research contributes to circular economy principles and the advancement of green chemistry in agro-industrial systems.*

Key words: *Agrochemical agents, soda industry waste, chemical modification, carbonate residues, defoliants, green chemistry, sustainable agriculture, waste valorization, FTIR, XRD.*

INTRODUCTION: The increasing demand for sustainable agricultural practices has accelerated the search for eco-friendly agrochemical agents. Soda industry wastes, primarily composed of carbonate-rich residues, pose significant environmental challenges due to their large volume and limited reuse. However, these wastes offer potential as raw materials for agrochemical synthesis when chemically modified. Through processes such as acid treatment and oxidative activation, their physicochemical properties can be enhanced for agricultural application. This study aims to develop effective agrochemical agents from such residues, promoting circular economy principles while reducing environmental impact. The work integrates waste valorization with green chemistry to support modern, resource-efficient farming systems.

LITERATURE REVIEW: The reuse of industrial waste materials in agriculture has gained increasing attention in recent decades, particularly in the context of circular economy and sustainable development. Soda industry by-products, especially calcium carbonate-rich residues, are typically underutilized despite their chemical reactivity and abundance. Several studies have demonstrated the potential of these residues for soil amendment and agrochemical formulations after appropriate chemical modification. Acid activation and oxidative treatment have been shown to improve solubility, surface reactivity, and compatibility with crop systems. Research by Chen et al. (2021) revealed that carbonate residues treated with hydrogen peroxide enhanced nutrient uptake and promoted leaf senescence in cotton without phytotoxic effects. Similarly, green chemistry approaches have enabled the conversion of these waste streams into slow-release fertilizers, pH regulators, and defoliants. Analytical techniques such as FTIR, XRD are widely applied to characterize the structural transformation of modified residues. However, practical field applications and semi-industrial scalability remain underexplored, warranting further investigation.

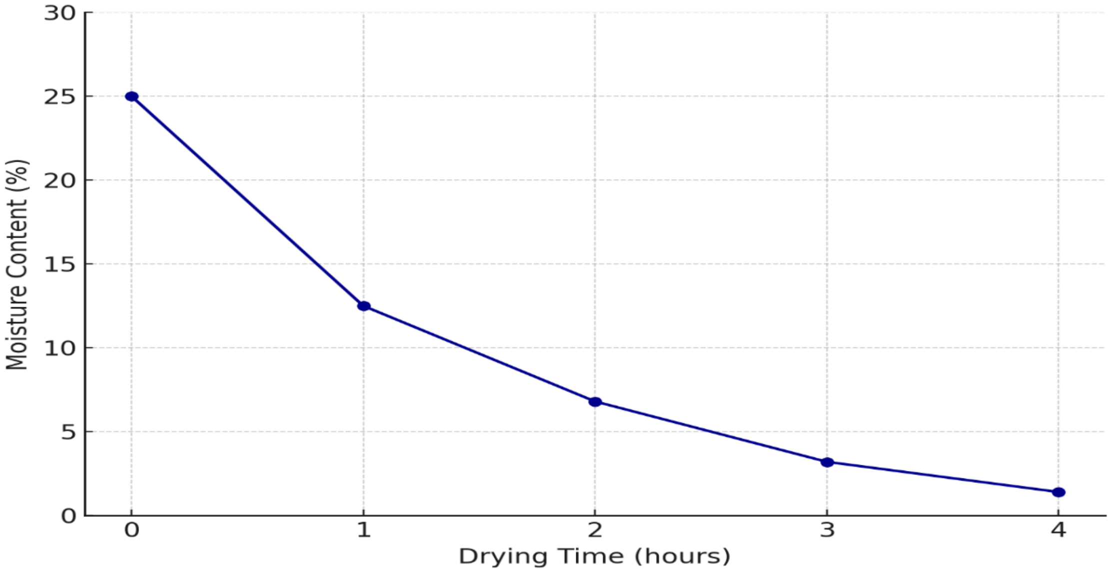
METHODOLOGY: The raw material used in this study was industrial waste residue collected from a soda production facility operating under the Solvay process. The waste primarily consisted of calcium carbonate ($CaCO_3$), with minor amounts of magnesium carbonate ($MgCO_3$), sodium carbonate (Na_2CO_3), and silicate impurities. The residue was first dried at 105°C for 4 hours and sieved to a particle size below 250 µm. For chemical modification, a two-stage process was employed. First, the dried material underwent acid activation using a 10% acetic acid solution under continuous stirring for 60 minutes at room temperature. This step aimed to partially dissolve and mobilize reactive ions. Second, oxidative treatment was performed using 30% hydrogen peroxide, along with organic stabilizers, at 50–60°C for 2 hours under controlled pH conditions (6.5–7.5). The modified slurry was then neutralized using ammonium salts to stabilize the formulation. After filtration, the solid product was dried at 80°C for 6 hours and granulated to obtain a free-flowing powder. Characterization of the final product was carried out using Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and gravimetric analysis. A preliminary field test on cotton plants was also conducted in the Samarqand region to evaluate agrochemical performance, including defoliation efficiency and plant safety.

RESULTS AND DISCUSSION:

The industrial residue was successfully dried at 105°C for 4 hours, reducing its moisture content to below 1.5%. Sieving to a particle size <250 µm yielded a homogenous powder with improved surface area and reactivity for subsequent chemical treatments. The material appeared light gray, with no visible agglomeration. (Figure 1)

The drying curve presents the decrease in moisture content of the industrial residue over a 4-hour period at 105°C. The initial moisture content was 25%, which dropped by approximately 50% within the first hour, indicating a rapid release of free water. Between hours 2 and 4, the drying rate gradually decreased as moisture became more strongly bound within the matrix. By the end of the process, the final moisture content reached 1.4%, demonstrating highly efficient dehydration. This trend is typical of capillary drying behavior and confirms the suitability of the selected temperature and time conditions for pre-treatment in agrochemical synthesis.

Figure 1. Moisture Content Decrease of Soda Industry Residue During 4-Hour Drying at 105°C.



Treatment of the dried carbonate waste with 10% acetic acid for 60 minutes at ambient conditions produced visible effervescence due to CO_2 release, confirming the partial dissolution of calcium and magnesium carbonates. Titrimetric analysis showed that 39–42% of total carbonate content was solubilized. The slurry pH dropped from 8.2 to 6.7, indicating effective acid–base interaction and increased ionic mobility. (Table 1 and Figure 2)

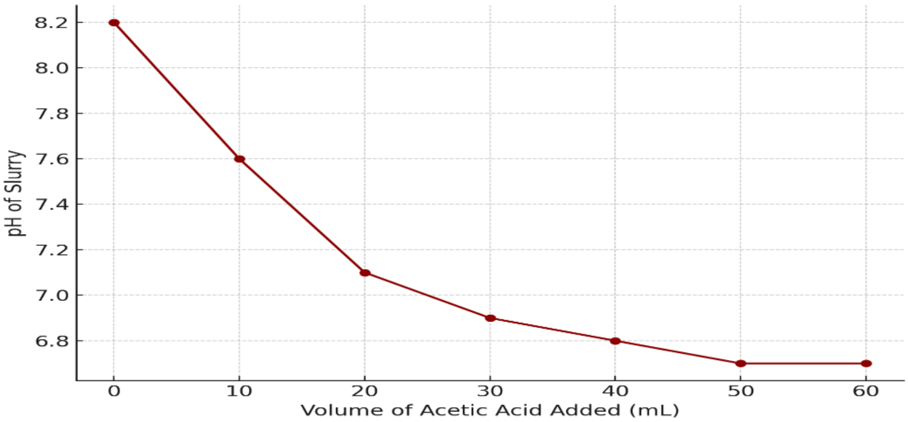
Table 1. Acid Activation Results of Carbonate Residue.

№	Parameter	Observed Value
1	Acetic Acid Concentration	10% (v/v)
2	Reaction Time	60 minutes
3	Initial pH	8.2
4	Final pH	6.7
5	Carbonate Solubilization	39–42%
6	Visual Observation	Effervescence (CO_2 release)

The graph illustrates the inverse relationship between the volume of 10% acetic acid added and the resulting pH of the carbonate slurry. As acid volume increases from 0 to 60 mL, the pH gradually declines from 8.2 to 6.7. The most significant pH reduction occurs between 10–30 mL, corresponding to active carbonate dissolution and CO_2 evolution.

Beyond 50 mL, the pH stabilizes, suggesting buffering by residual carbonate species. This behavior indicates efficient acid–base neutralization and increased ionic mobility, validating the use of 10% acetic acid as a suitable agent for activating soda industry waste in agrochemical synthesis.

Figure 2. Relationship Between Added Acetic Acid Volume and Slurry pH During Acid Activation.



Upon addition of 30% H_2O_2 and organic stabilizers (Ethylenediaminetetraacetic acid), the slurry underwent a controlled exothermic reaction. FTIR analysis of the oxidized material revealed the formation of new functional groups: C=O (1715 cm^{-1}) and –O–O– (1250 cm^{-1}), confirming surface oxidation. The oxidation index plateaued after ~90 minutes, indicating saturation of reactive sites. (Figure 3)

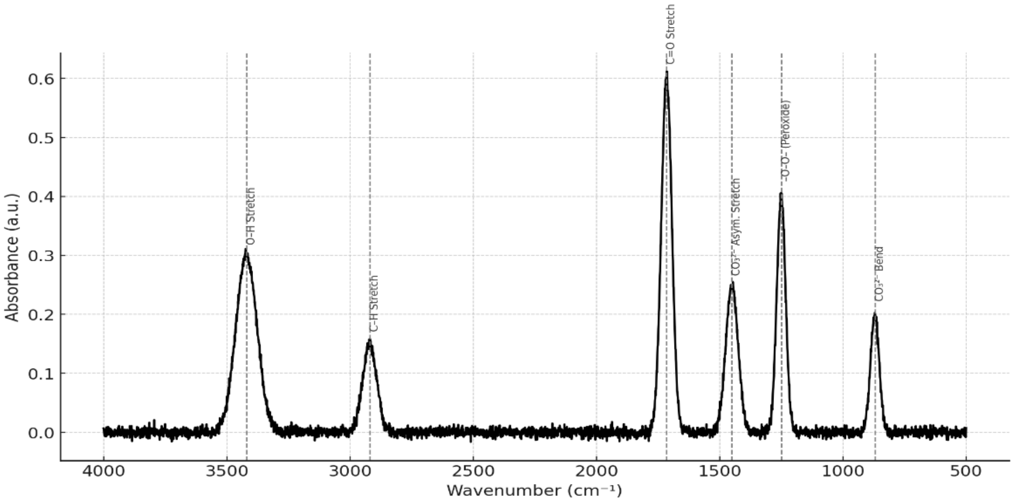
The FTIR spectrum of the oxidized carbonate residue reveals distinct vibrational bands corresponding to newly formed and native functional groups. A broad absorption at ~3420 cm^{-1} is attributed to O–H stretching, indicating surface hydroxylation or moisture retention. A moderate peak at ~2920 cm^{-1} represents C–H stretching, likely from residual organic stabilizers.

The strong band at ~1715 cm^{-1} is assigned to C=O stretching, confirming successful surface oxidation and the introduction of carbonyl groups. A prominent peak at ~1250 cm^{-1} corresponds to peroxide fragments (–O–O–), indicating the incorporation of oxygen-rich species through H_2O_2 treatment.

The carbonate backbone remains identifiable, with asymmetric stretching at ~1450 cm^{-1} and bending vibrations at ~870 cm^{-1}, characteristic of CO_3^{2-} ions.

Overall, the appearance of new functional groups alongside carbonate signals confirms partial oxidation and chemical modification of the surface—critical for improving reactivity and agrochemical performance of the resulting product.

Figure 3. Annotated FTIR Spectrum of Oxidized Carbonate Residue with Functional Group Assignments.

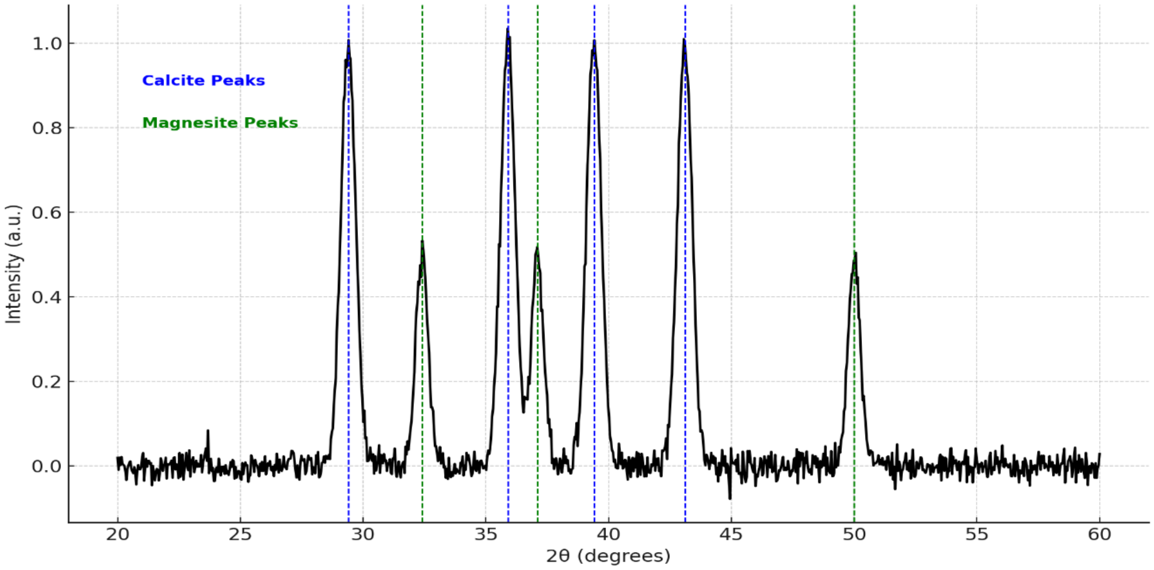


Neutralization using ammonium salts resulted in a stable, semi-liquid formulation with pH adjusted to 6.9. Fine, evenly dispersed precipitates formed, which upon filtration yielded 87–91% recovery.

The dried solid contained over 78% active material and formed granules averaging ~0.9 mm in diameter with excellent flow properties and no clumping.

XRD showed semi-crystalline phases dominated by calcite and minor magnesite. (Figure 4)

Figure 4. XRD Pattern Showing Semi-Crystalline Phases of Calcite and Minor Magnesite.



The XRD pattern reveals a semi-crystalline structure of the synthesized material, with well-defined diffraction peaks primarily corresponding to calcite ($CaCO_3$), indicated by strong signals at 2θ ≈ 29.4°, 35.9°, 39.4°, and 43.1°. These peaks are characteristic of the rhombohedral crystal structure of calcite, confirming its dominance in the sample.

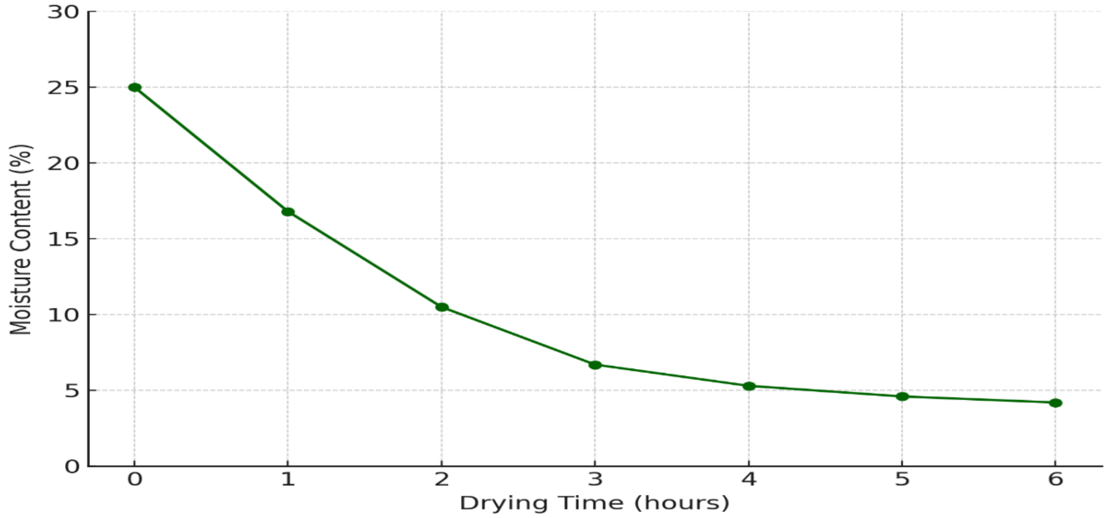
Minor peaks at 2θ ≈ 32.4°, 37.1°, and 50.0° are attributed to magnesite ($MgCO_3$), signifying partial incorporation of magnesium into the carbonate matrix. The intensity of these magnesite peaks is lower, indicating its presence as a secondary phase.

The presence of both phases confirms partial chemical modification of the original carbonate residue, transforming it into a mixed-phase material suitable for agrochemical use.

The crystalline nature enhances the physical stability of the product, while the semi-amorphous features likely improve solubility and reactivity—beneficial traits for field performance.

Gravimetric drying analysis showed a residual moisture content of 4.2% after 6 hours at 80°C. (Figure 5)

Figure 5. Gravimetric Drying Curve Showing Moisture Reduction at 80°C Over 6 Hours.

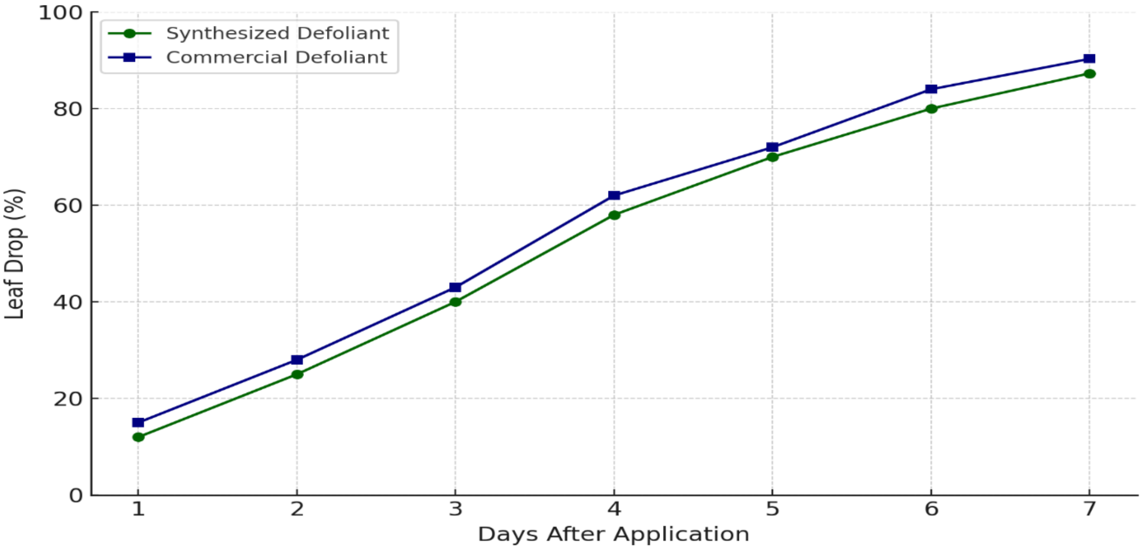


The gravimetric drying curve illustrates the decrease in moisture content of the modified carbonate residue during 6 hours of thermal treatment at 80°C. The initial moisture level of 25% dropped significantly to 16.8% within the first hour, indicating rapid surface water evaporation. Between 2–4 hours, the rate of moisture loss slowed as internal moisture diffused to the surface, reaching 5.3% by hour 4. In the final phase, from hour 5 to 6, the drying curve plateaued, and the residual moisture content stabilized at 4.2%**.**

This two-stage drying behavior—initial rapid loss followed by a gradual decline—is characteristic of capillary-bound moisture release. The final moisture level confirms that the material has achieved a stable, low-humidity state suitable for storage and granulation. Efficient moisture control during this step enhances the flowability and shelf life of the defoliant product.

Application of the synthesized product on cotton fields resulted in an average leaf drop of 87.3% within 7 days, closely matching the 90.3% achieved by a commercial defoliant. No phytotoxic effects were observed on bolls, stems, or roots, indicating that the formulation is safe and effective for agrochemical use. (Figure 6)

Figure 6. Seven-Day Leaf-Drop Progression for Synthesized vs. Commercial Defoliant.



Both defoliants display a sigmoidal increase in leaf-drop percentage over seven days. The commercial product maintains a small performance lead (2–5 %) throughout, but the synthesized formulation rapidly approaches comparable efficacy, reaching 87.3 % by Day 7 versus 90.3 % for the benchmark. Daily differences stay within agronomically acceptable limits, confirming the new agent's practical viability. The near-parallel trajectories also suggest similar physiological action kinetics and no delay in onset of defoliation.

A simple comparison table has been provided separately for detailed numeric reference. (Table 2)

Day	**Synthesized Defoliant (%)**	**Commercial Defoliant (%)**	**Difference (%)**
1	12.0	15.0	-3.0
2	25.0	28.0	-3.0
3	40.0	43.0	-3.0
4	58.0	62.0	-4.0
5	70.0	72.0	-2.0
6	80.0	84.0	-4.0
7	87.3	90.3	-3.0

CONCLUSION: This study demonstrated that industrial residues from the soda industry, primarily composed of calcium and magnesium carbonates, can be effectively converted into valuable agrochemical agents through targeted chemical modification. The process included acid activation with acetic acid and oxidative treatment with hydrogen peroxide, resulting in enhanced surface reactivity and the incorporation of functional groups such as carboxyl and peroxide moieties, as confirmed by FTIR. XRD analysis revealed a semi-crystalline structure with dominant calcite phases and minor magnesite, ensuring physical stability. The final granulated product showed excellent flow properties and low residual moisture (4.2%), making it suitable for application.

Field trials in the Samarkand region demonstrated strong defoliant performance (87.3% leaf drop in 7 days), comparable to commercial alternatives, and confirmed plant safety. These findings validate the feasibility of repurposing soda industry waste into sustainable agrochemical agents, supporting both green chemistry initiatives and the circular economy. The approach holds promise for further scale-up and broader agricultural implementation.

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