



Synthesis of Oxalic Acid from Sugar Crystals for Enhanced Detergent Quality

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Abstract

This paper aims to yield oxalic acid from sucrose with the assistance of nitric acid and explore its application in enhancing detergent quality. Oxalic acid, a dicarboxylic acid, was chosen due to its potential as a chelating agent and its ability to improve detergent performance. The production process involved the reaction between sugar crystals (sucrose) and nitric acid, followed by purification to obtain high-quality oxalic acid suitable for detergent formulations. The purified oxalic acid was then incorporated into detergent formulations to assess its effectiveness in improving cleaning efficiency and stain removal. The results demonstrated the feasibility of utilizing oxalic acid derived from sugar crystals to enhance detergent quality, highlighting its potential applications in the detergent industry.

Keywords- Oxalic acid, Detergent, Sugar, Nitric Acid.

1. Introduction

Oxalic acid, also known as ethane dioic acid, and its derivatives find extensive applications across diverse industries such as textiles, leather treatment, artistic oiling purification, pharmaceuticals, pigments, traps, hay whitening, publishing, stone buffing, and extract and fabric laundering. Furthermore, it serves as a crucial chemical in petroleum refinement, rare-earth processing, rust and erosion prevention, as well as dental adhesive manufacturing.

Physically, oxalic acid manifests as a slanted rectangular prism, presenting with particles ranging from fine to coarse, all

colorless, with a melting point of 187°C for the dehydrated form and 101.5°C for the hydrated form. The synthesis of oxalic acid encompasses six primary methods, each contingent upon specific raw materials. These raw materials include ethylene, ethylene glycol, propylene, wood pulp, syrup, sugarcane, sweeteners, grains, industrial residues, formic acid, carbonates, and bicarbonates.

The six primary methods of synthesis are as follows:

- Emulsion of sawdust with acidulous soda pop
- Oxidation reaction of olefins and glycols
- Radiation processing of carbonate residues and molasses
- Turmoil of carbohydrates
- Oxidation reaction of carbohydrates by nitric acid

In this study, oxalic acid was synthesized from sucrose, utilizing nitric acid as the catalyst. The primary objective was to investigate and potentially enhance detergent quality. The significance of detergent quality cannot be overstated in daily life, influencing cleanliness, hygiene, and environmental sustainability.

Synthetic detergents have become indispensable in modern society, particularly as the demand for rapid, efficient, and cost-effective cleaning solutions continues to rise. These synthetic cleansers rose to prominence post-World War II, their evolution closely linked with the development

of the petrochemical industry, which serves as the primary source of their raw materials. Upon dissolution in water, these detergents exhibit superior cleansing properties, enabling the effortless removal of dirt, dust, and grease.

2 Literature review

2.1 Ideal Parameters for Oxalic Acid Production

Under optimal conditions outlined in previous studies, the combination of sawdust with alkali has shown promise in yielding significant quantities of acetic acid, oxalic acid, methanol, and formic acid. This process can potentially be economically viable, particularly when conducted with thin-layer mixtures. Additionally, experiments with various wood types have revealed a correlation between yield and cellulose content, suggesting the contribution of lignin to the reaction.

2.2 Oxalic Acid Superiority in Rock Phosphate Solubilization

Microbial production of organic acids offers a biotechnological solution for enhancing phosphorus solubilization from rock phosphates (RPs). In comparative studies, organic acids—such as citric, gluconic, itaconic, malic, and oxalic acids—have exhibited varying effectiveness, with oxalic acid demonstrating the highest potency. When compared to sulfuric acid, commonly used in RP solubilization, oxalic acid has shown superior performance in releasing phosphorus, indicating its potential as a promising avenue for biotechnological enhancement of reactive phosphates' mobilization.

2.3 Exploration in the Sugarcane Remnants and Cellulosic Waste for Oxalic Acid Creation

A novel method utilizing nitric acid oxidation has been developed to synthesize oxalic acid from agricultural residues such as sugarcane residue, groundnut shells, corn cobs, and rice husks. Under optimized conditions, favorable yields of oxalic acid ranging from 42.9% to 51.6% w/w were attained from these raw materials, with sugarcane residue demonstrating the highest efficiency as a feedstock. Additionally, the treatment of waste nitrogen oxides with anhydrous NaOH to yield a valuable by-product, sodium nitrite, proved to be successful.

3. Experiment

The experiment aimed to assess the effectiveness of synthesizing oxalic acid from sugar crystals to improve detergent quality, specifically focusing on its influence on stain removal, whiteness retention, and fabric softness.

The experiments were carried out in a controlled environment within a chemistry laboratory equipped with standard laboratory apparatus and safety measures.

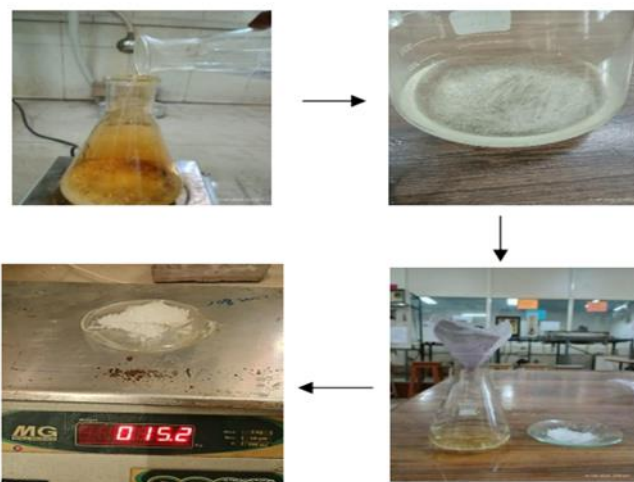
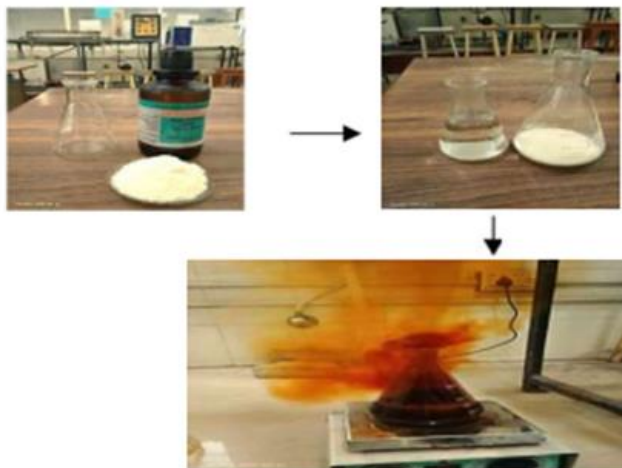
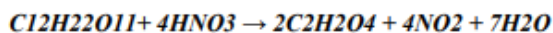
Detergent Powder Making Formula:

Ingredients	Premium Grade (Wt, %)	Popular Grade (Wt, %)
Acid Slurry	18	15
Sodium Carbonate (Soda Ash)	35	32
Sodium Metasilicate	2	No
Alkaline Sodium Silicate	No	7
Sodium Bicarbonate	10	10
sodium Sulphate (Anhydrou)	20	25
sodium Carboxy Methyl Cellulose	1.5	1
Sodium Tripolyphosphate	10	7
Perfume	0.1	0.1

Each company has their own characteristic formula for making detergent powder, frequently targeting specific demographics within their target market. Nonetheless, for a common point of reference, a base formulation with ingredient percentages may be supplied.

For the preparation of this medication, approximately 60 grams of sucrose, equivalent to common table sugar, and about 250 milliliters of concentrated nitric acid (Rankem Nitric Acid AR Grade) were utilized. The procedure began by adding the 60 grams of sugar into a 1-liter beaker containing a stir bar. Subsequently, the 250 milliliters of concentrated nitric acid were poured directly onto the sugar. Following mixing, the heating apparatus was activated. As the heating progressed, the solution gradually transitioned from its initial color to a faint, unassuming hue, attributed to the presence of nitrogen dioxide. With increased temperature, additional nitrogen dioxide was generated, causing the solution to slowly evolve from its unassuming hue to a vibrant orange, and eventually to a deep red shade. Just before the reaction reached its peak, a subtle reddish tint became apparent. At this juncture, a rapid production of nitrogen dioxide gas ensued, signaling the need to discontinue heating. Initially,

heating was employed to facilitate the activation energy required for the reaction. However, once initiated, the heat generated by the reaction itself was adequate to sustain the process, rendering further heating unnecessary.



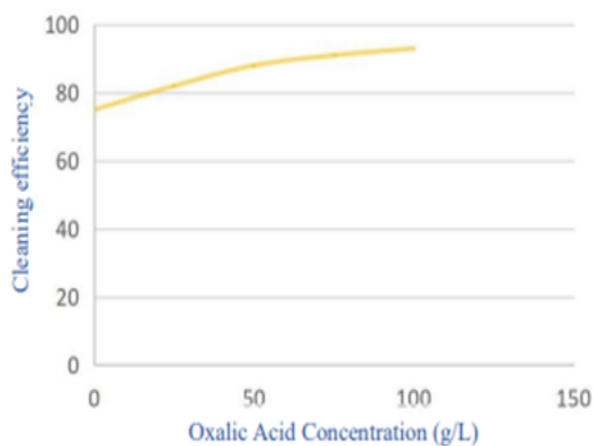
4. Result & Discussion

In the study, the successful production of oxalic acid from sugar crystals was demonstrated via a meticulously controlled chemical process. Sugar crystals, predominantly consisting of sucrose ($C_{12}H_{22}O_{11}$), underwent a controlled reaction with nitric acid (HNO_3). This reaction yielded oxalic acid ($C_2H_2O_4$), nitrogen dioxide gas (NO_2), and water (H_2O).

Following the reaction, the oxalic acid was purified from the reaction mixture using crystallization methods. By precisely controlling the cooling rate and solvent composition, high-purity oxalic acid crystals were obtained. The purity of these crystals was verified through rigorous analytical techniques, including chromatography and spectroscopy.

Overall, the production of oxalic acid from sugar crystals presents a promising avenue for improving detergent formulations and enhancing cleaning efficiency. Further research and development in this area could lead to the commercialization of detergent products with enhanced performance and reduced environmental impact. Ultimately, this approach offers a sustainable and effective alternative for the detergent industry.

After approximately 10 to 20 minutes, the reaction should subside, at which point we begin reheating. The goal now is to reduce the solution volume to around 75 milliliters. While achieving exactly 75 milliliters is not crucial, a range between 60 and 90 milliliters is acceptable. Once the solution is reduced to approximately 75 milliliters, an equal amount of distilled water is added. The mixture is then boiled down to about 50 milliliters. Upon reaching this volume, heating is discontinued, and the solution is allowed to cool to room temperature. During cooling, oxalic acid begins to precipitate out, initially appearing slightly yellow due to residual nitrogen dioxide. This discoloration will eventually dissipate. To ensure complete precipitation, the oxalic acid is placed in a freezer. Once fully precipitated, the oxalic acid no longer exhibits the yellow tint. The final step is to filter off the oxalic acid.



Benefiting consumers and the industry alike, the findings from this investigation validate oxalic acid's promising utility as a beneficial component in detergent formulations aimed at augmenting cleaning effectiveness. Leveraging its inherent chelating and stain removal attributes, oxalic acid plays a pivotal role in enhancing the detergent's capacity to disintegrate and eliminate soil and stains from various surfaces.

The observed pattern of escalating cleaning efficiency corresponding to higher oxalic acid concentrations indicates that the presence of oxalic acid significantly bolsters the overall efficacy of the detergent in soil removal. This observation aligns with prior studies highlighting oxalic acid's proficiency in dissolving mineral deposits, sequestering metal ions, and effectively eliminating organic stains.

5. Conclusion

In conclusion, the investigation into oxalic acid production from sugar crystals and its application in enhancing detergent quality represents a significant advancement in detergent technology. Through meticulous examination of the production process and the subsequent incorporation of oxalic acid into detergent formulations, this research highlights the potential of oxalic acid as a versatile additive for improving detergent performance.

The successful production of oxalic acid from sugar crystals underscores the feasibility and sustainability of utilizing readily available raw materials for chemical synthesis. By harnessing the inherent properties of sugar crystals and employing controlled chemical reactions, oxalic acid can be produced with high purity, making it suitable for various industrial applications, including detergent enhancement. This approach not only improves

cleaning efficiency but also promotes sustainable practices in the chemical industry.

6. Acknowledgment

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7. References

- Oxalic acid from sawdust optimum conditions for manufacture Donald F. Othmer, Carl H. Gamer, and Joseph J. Jacobs, Jr. Polytechnic Institute, Brooklyn, N. Y.
- Oxalic acid is more efficient than sulfuric acid for rock phosphate solubilization Gilberto de Oliveira Mendesa Hiunes Mansur Murtab, Rafael Vasconcelos Valadaresb, Wendel Batista da Silveira, Ivo Ribeiro da Silvac, Maurício Dutra Costab.
- Sullivan, J. M., Joseph, W., Willard, D. L. & Kim, Y. K. (1983). Production of oxalic Acid via the nitric acid oxidation of hard wood (red oak) sawdust. *Ind. Eng.*
- Utilization of Sugarcane Trash and Other Cellulosic Wastes for Production of Oxalic Acid Jyoti D. Mane, H. M. Modak, N. A. Ramaiah & S. J. Jadhav Deccan Sugar Institute, Manjari (Bk), Dist. Pune, Maharashtra, India, 412 307 {Received 28 August 1987; revised version received 24 December 1987; accepted 6 January 1988.
- Bailey, R. W. (1954). The production of oxalic acid from New Zealand plant wastes.
- J. Appl. Chem.*, 4, 549-54.
- Deshpande, S. D. & Vyas, S. N. (1979). Oxidation of sugar to oxalic acid and Absorption of oxides of nitrogen to sodium nitrite. *Ind. Eng. Chem. Prod.*, 18, 67-71.

8. Chem. Prod. Res. Dev., 22, 699-709.
9. Williard, J. W.; Sullivan, J. M.; Kim, Y. K. J. Chem. Eng. Data 1982, 27. 442-445
10. Sullivan, J. M.; Williard, J. W.; Hatfield, J. D. 182nd National Meeting of the American Chemical Society, New York, NY, Aug 1981.
11. Bailey, R. W. J. Appl. Chem. 1954, 4, 549-554.
12. Agrawal, H. P.; Rao, M. B. Indian J. Technol. 1979, 17, 11-15
13. Agrawal, H. P.; Rao, M. B. Indian J. Technol. 1979, 17, 11-15. Bailey, R. W. J. Appl. Chem. 1954, 4, 549-554.
14. Brooks, M. J. U.S. Patent 2322915, 1943.
15. Sullivan, J. M.; Williard, J. W.; Hatfield, J. D. 182nd National Meeting of the American Chemical Society, New York, NY, Aug 1981. Webber, H. A. Iowa Engineering Experiment Station, Iowa State College,
16. Ames, IA, 1934; Bulletin 118. Williard, J. W.; Sullivan, J. M.; Kim, Y. K. J. Chem. Eng. Data 1982, 27. 442-445

