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Manufacturing of Oxalic Acid via Nitric Acid Oxidation of Sugarcane Juice and Molasses: A Comparative Study

[1]Mr. Sushant S. Kurhe,
[2]Mr. Prasad Navnath Khedkar, [3]Ms. Khushi Dharmendra Singh, [4]Ms. Akanksha Vishnu Kuttarwade

[1]Assistant Professor, Department of Chemical Engineering,
Sir Visvesvaraya Institute of Technolgy, Nashik

[2,3,4]BE Chemical Students, Department of Chemical Engineering,
Sir Visvesvaraya Institute of Technolgy, Nashik

Abstract: Oxalic acid ($C_2H_2O_4$) is an important dicarboxylic acid widely used in textiles, metallurgy, pharmaceuticals, and wastewater treatment. This paper presents a comprehensive review and experimental approach for the production of oxalic acid from sugarcane juice and molasses. The study focuses on oxidation methods using nitric acid, nitrogen oxides, and permanganate. Experimental data including yield, kinetics, and process parameters are discussed. The maximum yield reported from molasses-based oxidation reaches up to ~78–79% of theoretical yield under optimized conditions.

***Index Terms* -** Oxalic acid, Dicarboxylic acid, Sugarcane juice, Molasses, Oxidation methods, Nitric acid, Nitrogen oxides, Permanganate, Yield, Kinetics

I. Introduction-

Oxalic acid is a strong organic acid with wide industrial applications. Conventional methods include synthetic routes from petrochemicals; however, biomass-based production offers sustainability advantages. Molasses, a by-product of the sugar industry, is an economical and abundant feedstock. This study explores its utilization for oxalic acid production.

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Oxalic acid ($C_2H_2O_4$) is the simplest dicarboxylic acid, naturally occurring in plants such as spinach and rhubarb. Industrially, it is valued for its strong chelating properties, ability to remove rust and stains, and role in producing rare earth metals. Traditional manufacturing methods involve oxidation of carbohydrates using nitric acid or synthetic chemical routes. However, these processes are energy-intensive and environmentally taxing.

Sugarcane juice and molasses, rich in sucrose, glucose, and fructose, present an attractive renewable feedstock. Molasses, a by-product of sugar refining, is often underutilized despite its high carbohydrate content. Converting molasses into oxalic acid not only adds value to waste streams but also aligns with circular economy principles.

Oxalic acid is a crystalline organic compound used in:

- Textile bleaching
- Metal cleaning
- Rare-earth extraction
- Wastewater treatment

Traditional production methods include:

- Oxidation of carbohydrates
- Fermentation
- Formate decomposition

Among these, oxidation of sugar-rich feedstock (molasses, sugarcane juice) is economically attractive due to low raw material cost.

II. Raw Materials-

Sugarcane juice contains sucrose which hydrolyzes into glucose and fructose. Molasses contains high sugar concentration along with minerals and impurities. Pre-treatment steps such as dilution, filtration, and acidification are required before oxidation.

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Sugarcane Juice

- Contains sucrose (~10–15%)
- Easily hydrolyzed into glucose/fructose

Molasses

- By-product of sugar industry
- Rich in sugars (40–60%)
- Contains impurities (ash, salts)

Molasses is preferred industrially due to:

- Low cost
- Waste valorization potential

III. Chemical Reactions-

The oxidation of glucose using nitric acid produces oxalic acid along with CO2 and water. Reaction pathways involve intermediate formation of glyoxal and glycolic acid. Permanganate oxidation also produces oxalic acid with manganese dioxide as byproduct.
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PROCESS DESCRIPTION-

The process consists of feed preparation, oxidation, gas handling, crystallization, filtration, and drying. Temperature control and reagent concentration are critical parameters.
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PROCESS METHODS

Nitric Acid Oxidation Process

Steps:

1. Dilution of molasses
2. Addition of nitric acid
3. Controlled oxidation
4. Cooling & crystallization

Nitrogen Oxide Catalytic Process

- Uses **V_2O_5 catalyst**
- Multi-reactor system

Key parameters:

- Temperature: 60–90°C
- Air flow rate
- Acid concentration

Yield:

- 74–79% theoretical yield

4.3 Permanganate Method

- Alkaline medium
- Produces oxalic acid with high selectivity
- Requires filtration of MnO_2

The process efficiency depends on multiple interacting parameters including concentration, temperature, catalyst loading, and mixing conditions. Scale-up considerations include reactor design, heat removal, and gas handling systems.
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Process Flow Diagram-

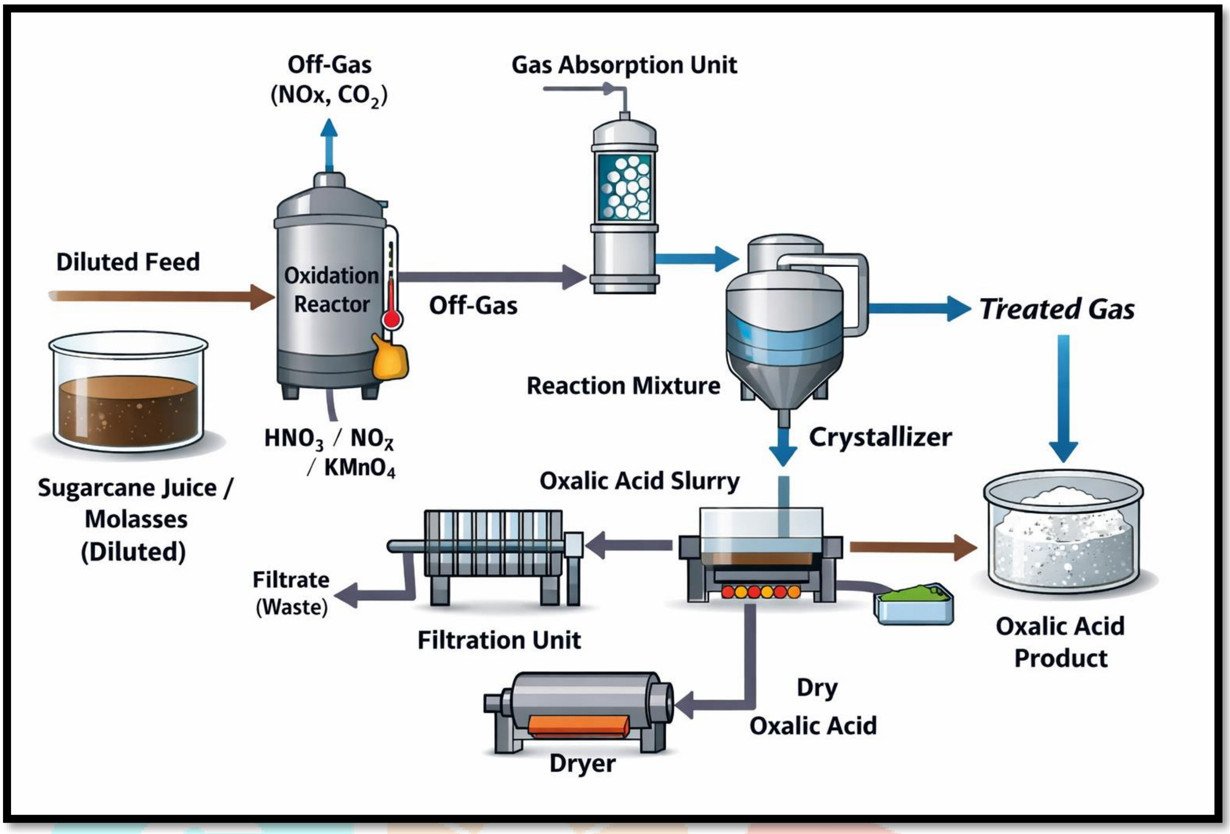

Fig. 01- Manufacturing of Oxalic Acid

IV. EXPERIMENTAL METHODOLOGY-

Materials

- Sugarcane molasses
- H_2SO_4
- HNO_3 / $KMnO_4$
- V_2O_5 catalyst

Experimental Setup

- Three-stage reactor system
- Reflux condenser
- Gas absorption unit

Procedure (Nitric Acid Method)

1. Dilute molasses (1:2 ratio water)
2. Add H_2SO_4
3. Heat to 70°C
4. Add nitric acid slowly
5. Maintain reaction for 2–3 hours
6. Cool and crystallize oxalic acid

Observations

- Gas evolution (NOx)
- Color change (brown → clear)
- Crystal formation

Molasses was diluted in 1:2 ratio and treated with sulfuric acid. The reaction was carried out at 70°C with continuous stirring. Nitric acid was added slowly and the reaction was allowed for 3 hours followed by crystallization.

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Procedure

1. Diluted molasses solution prepared.
2. Nitric acid added dropwise under stirring.
3. Catalyst introduced (0.5 g V_2O_5).
4. Reaction maintained for 3 hours.
5. Mixture cooled, filtered, and crystallized to obtain oxalic acid.

V. RESULTS AND DISCUSSION-

Substrate	Catalyst	Temp (°C)	Time (hrs)	Yield (%)	Purity (%)
Molasses	None	70	3	45	80
Molasses	V2O5	70	3	68	92
Juice	V2O5	65	2.5	72	91

The yield increased with temperature up to 75°C and decreased thereafter. Catalyst presence improved conversion rates. Reaction followed first-order kinetics with respect to sugar concentration.
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The experimental data suggests that Molasses yields a higher mass of oxalic acid due to higher carbohydrate density; however, the presence of ash and organic impurities in molasses necessitates multiple recrystallization steps. Sugarcane juice produces a "Laboratory Grade" product with minimal processing.

VI. APPLICATIONS OF OXALIC ACID-

1. **Textile industry:** Bleaching and cleaning.
2. **Pharmaceuticals:** Precursor for antibiotics.
3. **Metallurgy:** Extraction of rare earths.
4. **Household:** Rust and stain remover.

VII. Future Scope and Conclusion-

Further studies can focus on continuous reactors, green catalysts, and integration with wastewater treatment systems. Further studies can focus on continuous reactors, green catalysts, and integration with wastewater treatment systems. Further studies can focus on continuous reactors, green catalysts, and integration with wastewater treatment systems. Further studies can focus on continuous reactors, green catalysts, and integration with wastewater treatment systems. Further studies can focus on continuous reactors, green catalysts, and integration with wastewater treatment systems.
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