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Synthesis and Purification of High-Purity Sodium Citrate: Optimization of Laboratory Process Parameters

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ABSTRACT

A laboratory-scale procedure for the synthesis of high-purity sodium citrate from chemically pure sodium carbonate and analytical-grade citric acid was developed and evaluated. The process is based on controlled neutralization followed by solution concentration, crystallization, solid–liquid separation, and drying, enabling the production of sodium citrate in dihydrate or anhydrous form. The influence of the reactant molar ratio, solution pH prior to crystallization, and neutralization temperature on process performance was systematically studied. Optimal conditions were identified as a Na_2CO_3:$C_6H_8O_7$ molar ratio of 3.06:2.00, a pH range of 7.8–8.2 at 25 °C, and a neutralization temperature of 35–45 °C. Under these conditions, uniform crystal growth and favorable filtration properties were achieved, resulting in a yield of 82–84% of the theoretical value and a purity of 99.5–99.6%. Deviations from the optimal parameters led to the formation of acidic citrate species or impaired crystallization and filtration, causing a decrease in yield and purity. The proposed method provides a reproducible approach for obtaining high-purity sodium citrate suitable for analytical, pharmaceutical, and technological applications.

1. Introduction

Sodium citrate ($Na_3C_6H_5O_7$), the trisodium salt of citric acid, is widely used in medicine, the food industry, analytical chemistry, and pharmaceutical formulations [1]. Owing to its buffering capacity and metal-complexing properties, sodium citrate is employed as a pH stabilizer, acidity regulator, and preservative (food additive E331), as well as a reagent in titrimetric and photometric analytical methods. In medical practice, it is commonly applied as an anticoagulant for blood preservation, while in pharmaceutical formulations it serves as an auxiliary component for regulating acid–base balance [2,3].

The practical applicability of sodium citrate in analytical and pharmaceutical fields is critically dependent on its purity. Products intended for such uses are required to meet the specifications of chemically pure or analytical grade quality, which implies strict limitations on the presence of heavy metals, insoluble matter, and organic impurities. At present, a significant proportion of high-purity sodium citrate is produced industrially outside many regions, resulting in increased reliance on imported materials and reduced flexibility in laboratory-scale supply [4,5].

Sodium carbonate (Na_2CO_3, chemically pure) represents an accessible and economically favorable precursor for sodium citrate synthesis. It readily reacts with citric acid ($C_6H_8O_7$, analytical grade) to form trisodium citrate according to the reaction:

$$3Na_2CO_3 + 2C_6H_8O_7 \rightarrow 2Na_3C_6H_5O_7 + 3H_2O + 3CO_2\uparrow$$

This neutralization process is accompanied by the evolution of carbon dioxide and requires careful control of pH, temperature, and reagent dosing in order to obtain crystalline sodium citrate with high purity and reproducible physicochemical properties [6,7].

Despite the apparent simplicity of the reaction, deviations from optimal process parameters may lead to incomplete neutralization, formation of acidic citrate species, or deterioration of crystallization and filtration behavior. Therefore, the development of a reliable laboratory-scale procedure that ensures high product purity while maintaining satisfactory yield remains an important practical task.

The aim of the present study is to develop a laboratory process for the synthesis of high-purity sodium citrate based on sodium carbonate and to evaluate the influence of key technological parameters—reactant ratio, pH, neutralization temperature, and crystallization conditions—on the yield and quality of the final product.

2. Materials and Methods

2.1 Materials

Chemically pure sodium carbonate (Na_2CO_3) and analytical-grade citric acid ($C_6H_8O_7$), used either in anhydrous form or as the monohydrate ($C_6H_8O_7 \cdot H_2O$), served as the starting reagents. Demineralized water with a total dissolved solids content below 0.1 mg L^{-1} was used as the solvent throughout the experiments. The synthesis was based on the neutralization of citric acid with sodium carbonate, resulting in the formation of trisodium citrate, water, and carbon dioxide.

Stoichiometric calculations were performed assuming a Na_2CO_3:$C_6H_8O_7$ molar ratio of 3:2. When citric acid monohydrate was employed, the reagent mass was recalculated on an equivalent basis. A slight excess of sodium carbonate (not exceeding 2–3%) was allowed to ensure complete neutralization and stabilization of the solution pH. A typical batch consisted of 2.0 L of water, 384.2 g of anhydrous citric acid (2.00 mol) or 420.3 g of citric acid monohydrate in equivalent amount, and 318.0 g of sodium carbonate (3.00 mol).

2.2 Synthesis Procedure

The synthesis was carried out in a glass or enamel-lined reactor equipped with a heating–cooling jacket, an anchor-type stirrer operating at 60–120 rpm, and a gas outlet. Citric acid was initially dissolved at 20–25 °C, after which the temperature was raised to 30–35 °C. Sodium carbonate was added either portionwise as a solid or as a 20–25 wt.% aqueous solution at 35–45 °C under continuous pH monitoring.

The target pH value prior to crystallization was maintained in the range of 7.8–8.2 at 25 °C. After the cessation of carbon dioxide evolution, the reaction mixture was held at 40 °C for an additional 15–20 min to ensure completion of neutralization. When discoloration was observed, 1–2 g of chemically pure powdered activated carbon was added, and the suspension was stirred for approximately 10 min.

The hot solution was filtered at 40–45 °C using a vacuum Nutsche filter and subsequently concentrated under reduced pressure at 45–55 °C to a total solids content of 40–55%, as determined by solution density. Crystallization was initiated by controlled cooling at a rate of 0.2–0.5 °C min^{-1} to 10–15 °C, with the addition of seed crystals in an amount of 0.2–0.5 wt.% relative to the dissolved solids. The suspension was aged for 60–90 min to complete crystal growth.

The resulting crystals were separated by vacuum filtration and washed with cold demineralized water in an amount corresponding to 5–10% of the wet cake mass. To facilitate dehydration, a brief wash with cold anhydrous ethanol (5–10% in minimal volume) was applied when required.

2.3 Drying and Product Handling

Drying was performed in a laboratory oven at temperatures ranging from 40 to 150 °C, depending on the desired product form. Sodium citrate dihydrate was obtained by drying at 60–80 °C to constant mass. The anhydrous form was prepared by drying at 120–130 °C under reduced pressure (10–30 kPa) for 2–4 h, with the temperature not exceeding 150 °C. The dried product was sieved through a 0.5–1.0 mm mesh and stored in airtight containers.

2.4 Quality Control and Yield Determination

Quality control included measurement of the pH of a 5 wt.% aqueous solution, which was required to fall within the range of 7.5–9.0 at 25 °C. The mass fraction of the main component was determined by titration with 0.1 mol L^{-1} hydrochloric acid using methyl orange as the indicator. Insoluble impurities were quantified by filtration followed by drying of the residue at 105 °C to constant mass. Chloride and sulfate contents were assessed by phototurbidimetric or nephelometric methods, while heavy metals were determined photometrically using dithizone.

Moisture content was controlled according to the product form: for the dihydrate, based on the stoichiometric crystallization water content; for the anhydrous salt, by Karl Fischer titration or drying to constant mass. The product yield was calculated based on citric acid consumption, using conversion factors of $n(C_6H_8O_7) \times 258.06$ for anhydrous sodium citrate and $n(C_6H_8O_7) \times 294.10$ for the dihydrate, with explicit indication of the product form and drying conditions.

3. Results and Discussion

The influence of key technological parameters, including the reactant molar ratio, the pH of the solution prior to crystallization, and the neutralization temperature, on the yield and quality of sodium citrate was systematically investigated. The obtained results made it possible to identify optimal operating conditions that ensure maximum product yield while maintaining a high degree of purity and to confirm the reproducibility of the process under laboratory conditions.

3.1 Effect of Reactant Molar Ratio

Table 1 summarizes the effect of the Na2CO3:C6H8O7 molar ratio on the final pH, product yield, and purity.

Table 1: Effect of reactant molar ratio (Na_2CO_3:$C_6H_8O_7$) on sodium citrate yield and purity

Molar ratio	Final pH (25 °C)	Yield, % of theoretical	Purity, %
2.8:2.0 (–6.7% Na_2CO_3)	7.2–7.4	76–78	99.1–99.3
2.9:2.0 (–3.3%)	7.4–7.6	79–81	99.3–99.4
3.00:2.00	7.7–7.9	82–83	99.5–99.6
3.06:2.00 (+2%)	7.8–8.1	83–84	99.5–99.6
3.12:2.00 (+4%)	8.0–8.3	82–83	99.4–99.5

3.24:2.00 (+8%)	8.3–8.6	80–81	99.2–99.4
2.8:2.0 (–6.7% Na2CO3)	7.2–7.4	76–78	99.1–99.3
2.9:2.0 (–3.3%)	7.4–7.6	79–81	99.3–99.4
3.00:2.00	7.7–7.9	82–83	99.5–99.6
3.06:2.00 (+2%)	7.8–8.1	83–84	99.5–99.6

Analysis of the data presented in Table 1 indicates that the reactant molar ratio is a determining factor governing both yield and purity of sodium citrate. When sodium carbonate was deficient (molar ratios of 2.8–2.9:2.0), incomplete neutralization of citric acid occurred, leading to the formation of acidic citrate species (NaH_2Cit and Na_2HCit). Under these conditions, the final pH remained within 7.2–7.6, the yield did not exceed 79–81%, and the product exhibited increased titratable acidity accompanied by additional losses during crystal washing.

At the stoichiometric ratio of 3.0:2.0, complete neutralization of citric acid was achieved, the solution prior to crystallization was clear, and the yield increased to 82–83% while maintaining high purity (99.5–99.6%). The most favorable performance was observed with a slight excess of sodium carbonate (+2%), where the yield reached 83–84% without any loss in purity. Under these conditions, crystallization proceeded uniformly, resulting in well-formed crystals with good filtration properties.

Further increases in alkali excess (+4–8%) led to a rise in the final pH to 8.0–8.6, which was associated with residual alkalinity, the need for more intensive washing, and a gradual decrease in yield to 80–82%. In addition, signs of co-precipitation of impurities and increased conductivity of the wash waters were observed, indicating a reduction in the effective purity of the final product.

3.2 Effect of pH Prior to Crystallization

The influence of the pH of the solution before crystallization on yield and purity is presented in Table 2.

Table 2: Effect of solution pH prior to crystallization on sodium citrate yield and purity

pH (25 °C)	Yield, % of theoretical	Purity, %
7,0–7,3	77–79	99,1–99,3
7,4–7,7	80–82	99,3–99,5
7,8–8,0	82–84	99,5–99,6
8,1–8,2	82–83	99,5–99,6
8,3–8,5	80–82	99,3–99,5
> 8,5	78–80	99,2–99,4

The results demonstrate that the pH of the solution prior to crystallization is a critical parameter affecting both yield and product quality. At lower pH values (7.0–7.3), an excess of citric acid remained in solution, favoring the formation of acidic citrate salts. This resulted in a reduced yield of 77–79% and a decrease in purity to 99.1–99.3%, along with increased titratable acidity of the filtrate and the need for additional washing steps.

In the pH range of 7.4–7.7, the process approached stoichiometric conditions, the solution remained clear, and crystallization was stable; however, the yield was still below the optimal level (80–82%). Maximum yield and purity were achieved in the pH interval of 7.8–8.0, where complete neutralization was ensured, the content of foreign salts was minimized, and relatively large, well-filterable crystals were formed. Under these conditions, the yield reached 82–84% with a purity of 99.5–99.6%.

At pH values of 8.1–8.2, high yield and purity were preserved, although careful control of wash water conductivity was required to exclude residual alkalinity. Further increases in pH (8.3–8.5 and above 8.5) led to deterioration of process characteristics: crystals became finer, slurry viscosity increased, filtration slowed, and mechanical losses intensified, causing a decrease in yield to 78–82%.

3.3 Effect of Neutralization Temperature

The effect of neutralization temperature on sodium citrate yield and purity is summarized in Table 3.

Table 3. Effect of neutralization temperature on sodium citrate yield and purity

Temperature, °C	Yield, % of theoretical	Purity, %
20–25	77–79	99,2–99,3
30–34	80–81	99,3–99,4
35–40	82–84	99,5–99,6
41–45	82–83	99,5
50–55	80–81	99,3–99,4
> 55	77–79	99,1–99,2

The data indicate that neutralization temperature has a pronounced effect on reaction kinetics, crystallization behavior, and overall process performance. At low temperatures (20–25 °C), dissolution of citric acid and neutralization proceeded slowly and were accompanied by intensive evolution of finely dispersed carbon dioxide, which caused solution turbidity, complicated filtration, and reduced yield to 77–79% with purity not exceeding 99.2–99.3%.

Moderate temperature increase to 30–34 °C accelerated the reaction and improved solution clarity; however, the yield remained suboptimal at 80–81%. The most favorable results were obtained in the temperature range of 35–40 °C, where neutralization proceeded smoothly, carbon dioxide evolution was uniform, and crystallization was stable. Under these conditions, the yield reached 82–84% with a purity of 99.5–99.6%.

At temperatures of 41–45 °C, high yields (82–83%) were maintained, although careful control of gas evolution was required to avoid foaming. Further temperature increase to 50–55 °C significantly intensified reaction rates, increasing the risk of local overheating, formation of fine crystals and inclusions, and a reduction in yield to 80–81% with purity of 99.3–99.4%. The least favorable performance was observed above 55 °C, where partial degradation of citric acid occurred, the solution darkened, and both yield (77–79%) and purity (99.1–99.2%) decreased.

Overall, a neutralization temperature range of 35–45 °C was identified as optimal, providing a favorable balance between reaction kinetics, crystallization behavior, and product quality, and ensuring stable and reproducible process performance.

4. Conclusion

A laboratory-scale process for the preparation of high-purity sodium citrate from sodium carbonate and citric acid was developed and experimentally validated. The study demonstrates that the Na_2CO_3:$C_6H_8O_7$ molar ratio, the pH of the solution prior to crystallization, and the neutralization temperature are the key parameters governing both product yield and purity. Insufficient amounts of sodium carbonate result in acidic conditions and the formation of acidic citrate species, which is accompanied by increased titratable acidity of the filtrates and a reduction in yield. Conversely, excessive alkalinity increases the ionic background of the system and complicates crystal washing, leading to losses in both yield and purity.

The optimal operating conditions were identified as a Na_2CO_3:$C_6H_8O_7$ molar ratio of 3.06:2.00, a pH range of 7.8–8.2 at 25 °C, and a neutralization temperature of 35–45 °C. Under these conditions, the maximum yield of 82–84% relative to the theoretical value was achieved while maintaining a high product purity of 99.5–99.6%.

It was also shown that lowering the pH below 7.3 reduces the yield to 77–79% and decreases purity to 99.1–99.3%, whereas increasing the pH above 8.5 promotes the formation of fine crystals, increases slurry viscosity, and slows filtration, resulting in mechanical losses and reduced yield. Overall, the selected optimal parameters ensure reproducible production of sodium citrate with high purity and maximum yield, making the proposed laboratory procedure suitable for analytical and technological applications.

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Declaration of authors’ conflict of interest:

The authors declared no conflict of interest.

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