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## Synthesis of Hydroxyapatite Using Recycled Wash Water

*Abstract:* Hydroxyapatite is a biocompatible calcium-phosphate material widely used in medicine, pharmacy, and advanced biomaterials engineering. At the same time, its synthesis by homogeneous precipitation is associated with the generation of significant amounts of wash water containing residual nitrogen- and phosphorus-containing compounds. In the context of circular economy and low-waste chemical production, the valorization and recycling of such water streams represent an important technological challenge. This work investigates the synthesis kinetics of Hydroxyapatite using calcium saccharate as a calcium precursor and urea as an *in situ* source of ammonium hydroxide under conditions approximating a Continuous Stirred-Tank Reactor (CSTR). The formation of Hydroxyapatite was confirmed by infrared spectroscopy and X-ray phase analysis. Kinetic studies based on turbidity measurements established that the rational precipitation time is 50–60 min, which ensures the completion of nucleation and crystal growth processes. The purification of the resulting Hydroxyapatite precipitate requires no fewer than seven consecutive centrifugal washing cycles, leading to the generation of substantial volumes of wash water. It was shown that these water fractions contain residual ammonium phosphates, urea, and organic saccharate compounds, making them suitable for secondary valorization. Two utilization pathways are proposed: conversion into a suspended mineral-organic NP fertilizer and concentration by membrane technologies, particularly reverse osmosis, followed by permeate recycling into the synthesis process. The proposed approach may enable the integration of Hydroxyapatite synthesis with nutrient recovery and wash water recycling, significantly improving process sustainability and supporting the principles of zero-waste chemical technology.

*Keywords:* hydroxyapatite, calcium saccharate, homogeneous precipitation, recycled wash water, NP-fertilizer, reverse osmosis, circular economy.

### **Abbreviations:**

**CSTR** is Continuous Stirred-Tank Reactor.

## Introduction

Under modern conditions of the development of advanced low-tonnage industrial synthesis methods for chemical materials, the implementation of resource-saving and low-waste approaches is becoming increasingly relevant. One of the key directions in the realization of sustainable development principles is the reuse of process water within industrial cycles. In certain cases, the source of such wastewater is the technological washing of target products after filtration or centrifugation.

A similar situation is observed in the synthesis of hydroxyapatite by the homogeneous precipitation method (*Serbiienko et al., 2021*).

Hydroxyapatite ( $Ca_5(PO_4)_3OH$  або  $Ca_{10}(PO_4)_6(OH)_2$ ) belongs to the calcium phosphate mineral family forming the hard tissues of vertebrate organisms (*Kumar et al. 2022; Precious et al., 2024*). Synthetic hydroxyapatite is a biocompatible material with promising potential in 3D-printing technologies (*Serbiienko et al., 2022*).

The development of industrial low-tonnage hydroxyapatite synthesis technologies using recycled wash water corresponds to modern trends in the progressive greening of chemical production, as well as to the principles of the circular economy and the concept of zero-waste production.

Of particular importance is the establishment of the regularities governing the influence of the composition of recycled wash water on the physicochemical properties of the resulting product. Equally significant is the possibility of valorizing wash or wastewater into secondary-value products, such as complex fertilizers.

The study aims to investigate the possibility of Hydroxyapatite synthesis using recycled wash water and to develop proposals for water reuse with the simultaneous production of a liquid NP fertilizer formulation, as well as to substantiate the prospects for implementing such an approach in low-waste technologies for the production of calcium phosphate materials.

To achieve this purpose, the study focuses on several interrelated research tasks:

- to obtain hydroxyapatite by homogeneous precipitation using calcium saccharate, diammonium phosphate and urea as initial reagents;
- to determine the kinetics of hydroxyapatite formation by monitoring changes in the light transmittance of the reaction medium;
- to identify the rational duration of precipitation required for the completion of nucleation and crystal growth;
- to confirm the phase composition of the obtained precipitate by infrared spectroscopy and X-ray phase analysis;
- to evaluate the technological necessity of repeated centrifugal washing for removing residual impurities;
- to analyse the composition and potential value of the resulting wash water and primary centrifugate;
- to propose possible schemes for integrating wash water recycling, membrane concentration and nutrient recovery into the hydroxyapatite production process.

The theoretical significance of the study lies in the clarification of the relationship between homogeneous precipitation conditions, urea hydrolysis, pH generation, precipitation kinetics and

the formation of hydroxyapatite as the dominant calcium-phosphate phase. The work contributes to the scientific understanding of hydroxyapatite synthesis under conditions close to ideal mixing, where urea acts as an internal precursor of ammonium hydroxide and provides a more uniform increase in alkalinity throughout the reaction volume. In addition, the study develops the theoretical basis for considering wash water not merely as a technological waste stream, but as a secondary resource containing residual nitrogen- and phosphorus-bearing compounds that may be involved in further material or nutrient recovery cycles.

The practical significance of the results is associated with the possibility of improving the environmental and technological efficiency of low-tonnage hydroxyapatite production. The determination of a rational precipitation time of 50–60 minutes can be used for process optimisation and future scale-up. The confirmation that no fewer than seven centrifugal washing cycles are required for effective purification provides a practical basis for designing the washing stage and estimating water consumption. At the same time, the proposed utilisation pathways for wash water—conversion into a suspended mineral-organic NP fertiliser or concentration by membrane technologies with subsequent permeate recycling—may reduce wastewater discharge, increase material efficiency and support the transition towards low-waste and circular chemical production.

*Thus*, the study is aimed not only at obtaining hydroxyapatite as a valuable biocompatible calcium-phosphate material, but also at developing a more sustainable technological concept in which the synthesis process is integrated with wash water recycling, nutrient recovery and secondary product formation. Such an approach corresponds to the principles of circular economy, resource conservation and zero-waste chemical technology.

### Methods

The study was based on the method of homogeneous precipitation of hydroxyapatite from a mixture of aqueous solutions of calcium saccharate, diammonium phosphate, and urea, followed by heating to a temperature of 60°C and above without mandatory reactor thermostating (*Serhiienko et al., 2021*).

At the preparation stage, an aqueous solution of calcium saccharate was separately obtained. The calcium saccharate solution was prepared by the interaction of calcium oxide (0.53 g) with an aqueous sucrose solution (3 g of sucrose, 97 g of distilled water).

Subsequently, aqueous solutions of calcium saccharate, ammonium hydrogen phosphate, and urea were mixed in distilled water within a laboratory-scale CSTR, followed by heating of the resulting homogeneous reaction medium to a temperature not lower than 80°C or at the boiling point (in our case 103°C). Hydroxyapatite synthesis was carried out under constant stirring for 1.5 hours.

As a result, a white precipitate was formed, while the reaction medium became viscous. After completion of the synthesis, the reaction mixture was cooled, the Hydroxyapatite precipitate was washed with distilled water and separated from the liquid phase using a laboratory centrifuge. The washed Hydroxyapatite precipitate was dried in a forced-convection drying oven at 60°C.

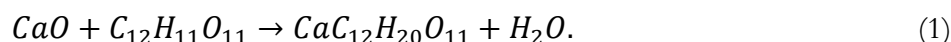
During the precipitation process, turbidity measurements were carried out to determine the completion time of Hydroxyapatite precipitation kinetics.

### Literature Review

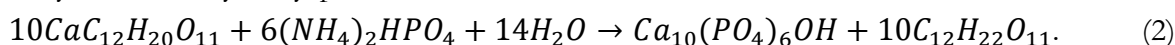
The homogeneous precipitation method makes it possible to control the particle size of precipitates, ranging from highly dispersed to conventional dimensions. Under such conditions, a large number of crystallization centers are formed, leading to the generation of highly dispersed precipitate particles.

To a certain extent, this model is close to the CSTR concept (*Fedenko et al., 2026*). In this model, instantaneous and complete mixing of the entire reaction volume is assumed, resulting in uniform concentrations of reactants, products, and temperature throughout the reactor.

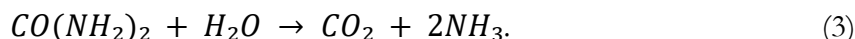
In the case of hydroxyapatite precipitation, the following chemical interactions occur: obtaining calcium saccharate for reaction



synthesis of Hydroxyapatite



To create an alkaline medium ( $pH \geq 8$ ) for the implementation of reaction (2), any alkali, even a weak one, is suitable. However, the use of ammonium hydroxide is expedient, since it is volatile and can be easily removed from the hydroxyapatite precipitate during drying. The sources of ammonium hydroxide are gaseous ammonia introduced into the reaction medium during stirring or aqueous ammonia solution. If ammonium hydroxide is introduced in the form of gaseous ammonia or aqueous ammonia, inevitable local supersaturation with alkali occurs at the point of reagent introduction. Instantaneous and uniform distribution of alkali throughout the entire reactor volume requires forced and rather complex programmed hydrodynamic conditions (*Rashid et al., 2020*). Such a regime does not guarantee uniform alkali distribution in the reactor within an almost zero-time interval. The introduction of gaseous ammonia or aqueous ammonia therefore does not correspond to conditions close to those of an ideal mixing reactor. The problem lies in the fact that aqueous solutions of calcium saccharate and diammonium phosphate are first mixed to form a homogeneous mixture, and only then the alkaline reagent is added. It is precisely at this stage that concentration heterogeneity arises and the correspondence to the ideal mixing reactor conditions is violated. An alternative source of ammonium hydroxide is the well-known hydrolysis reaction of urea upon heating (*Sigurdarson et al., 2018; Mazzei et al., 2020*).



If an aqueous urea solution is introduced at room temperature into the reaction mixture consisting of aqueous calcium saccharate and diammonium phosphate solutions and then mixed, the reaction mixture becomes homogeneous. Under such conditions, reaction (2) does not proceed immediately. Upon subsequent heating and stirring of the homogeneous mixture of aqueous calcium saccharate, diammonium phosphate, and urea solutions at  $60^\circ C$ , urea hydrolysis according to reaction (3) begins, resulting in ammonia formation and the release of hydroxide groups. Even under moderate stirring, uniform temperature throughout the reactor volume is established very rapidly. Likewise, the concentration of hydroxide groups in the reactor is established almost instantaneously. Thus, the use of urea in hydroxyapatite synthesis by the homogeneous precipitation method largely corresponds to the conditions of an ideal mixing reactor. It is specifically urea that enables the formation of hydroxyapatite in nanosized form.

A drawback of using urea is that even under boiling conditions of the reaction mixture it does not decompose completely; therefore, it must be used in a certain excess. In addition, in the case

of nanosized hydroxyapatite formation, additional volumes of wash water are required to obtain hydroxyapatite free from residual impurities of the precursor substances. This raises the issue of wash water utilization. In (*Serhiienko et al., 2021*), neither the multiplicity of washing cycles nor the utilization of wash water was considered. Likewise, the kinetics of hydroxyapatite formation by the homogeneous precipitation method were not addressed. The present work examines the precipitation rate of hydroxyapatite, the rational number of washing cycles, and proposals for wash water recycling.

The issue of wash water recycling is directly related to the fact that these waters contain dissolved residual diammonium phosphate and urea, both of which are fertilizers. Considering that saccharates also remain in solution, such a composition can be classified as mineral-organic fertilizer. This classification makes it possible to consider the utilization of wash water as a fertilizer product.

Wash waters generated during Hydroxyapatite synthesis by precipitation and containing residual concentrations of Ammonium phosphate and Urea may therefore be regarded as a by-product liquid fertilizer, namely a complex binary nitrogen-phosphorus fertilizer.

Residual ammonium phosphates represent a biologically available form of phosphorus readily assimilated by plants, as well as a source of ammonium nitrogen. Residual urea represents a biologically available and prolonged amide form of nitrogen for root uptake. The combination of ammonium phosphates and urea forms a balanced nutrient system, namely:

- readily available phosphorus for plants;
- immediate ammonium nitrogen;
- prolonged nitrogen release from urea;
- possible enhancement of the utilization coefficient of N and P due to synergistic effects.

Such a composition is functionally close to liquid NP-fertilizers.

From a technological perspective, the use of urea as an internal source of ammonium hydroxide ensures a more uniform increase of local pH throughout the entire reaction volume, which is fundamentally important for achieving conditions close to those of a Continuous Stirred-Tank Reactor (CSTR). This minimizes local supersaturation zones and promotes the formation of a large number of homogeneous nucleation centers. Such a mechanism is particularly important for the formation of finely dispersed Hydroxyapatite, since the uniform generation of  $\text{OH}^-$  directly affects the morphology, particle size distribution, and crystallinity of the precipitate.

## Results

The results of measuring by spectrophotometer Unico101 light transmittance (%) in the reaction medium during Hydroxyapatite precipitation are presented in Table 1.

The kinetic studies make it possible to recommend 50–60 minutes as the rational time required for complete Hydroxyapatite precipitation according to reactions 1–3. During this period, crystallization centers are formed and Hydroxyapatite crystals grow.

The unwashed Hydroxyapatite precipitate obtained in the experiments was studied by infrared spectroscopy using a Shimadzu IRAffinity-15 spectrometer (*Figure 1*).

The unwashed Hydroxyapatite precipitate contains impurities of organic calcium complexes, ammonium phosphates, and urea (*Linstrom & Mallard, 2025; John Wiley & Sons, 2025*).

Separately, the composition of the Hydroxyapatite precipitate subjected to seven consecutive centrifugal washing cycles was studied by infrared spectroscopy (*Figure 2*).

The spectral profiles are consistent, and the dominant crystalline phase is Hydroxyapatite.

The washed Hydroxyapatite precipitate was further investigated by X-ray phase analysis (*Figure 3*).

The interpretation of the data was carried out based on (*ICDD Powder Diffraction File (PDF-4+ / PDF-2), Crystallography Open Database (COD)*).

The obtained infrared spectroscopy and X-ray phase analysis data are consistent with the results previously reported in (*Serhiienko et al., 2021*). The precipitate obtained and washed at least seven times is Hydroxyapatite.

### Discussion

The results of the kinetic studies make it possible to recommend 50–60 minutes as the rational time required for the complete precipitation of hydroxyapatite from homogeneous solutions according to reactions 1–3.

During this period, the formation of crystallization centers and the growth of hydroxyapatite crystals take place.

The investigation of the crystal-chemical composition was not the objective of this study. The examination of the chemical composition by means of X-ray phase analysis and infrared spectroscopy demonstrates that the dominant solid phase is hydroxyapatite. A challenging stage of the synthesis is the purification of this precipitate from all residual reagent impurities present in the reaction solution. For effective precipitate separation, the use of a centrifuge and no fewer than seven consecutive washing cycles is required.

The washing process is inevitably accompanied by the formation of a significant amount of wastewater. These waters should also include the first fugate, which represents the reaction solution remaining after the separation of the hydroxyapatite precipitate. The problem of generating substantial volumes of wastewater and wash water requires the development of appropriate approaches for its resolution.

### Conclusions

The wastewater generated during the synthesis and washing stages of Hydroxyapatite production may be effectively utilized by several technological approaches.

A. One possible route is the conversion of the obtained Hydroxyapatite into a suspended mineral-organic NP fertilizer. Such a fertilizer is characterized by the prolonged release of its phosphate component and may be applied in closed-soil systems for precision plant nutrition. This approach is functionally attractive due to the combined fertilizing effect of Hydroxyapatite, residual ammonium phosphates, and urea. Hydroxyapatite itself may serve as a slow-release phosphorus source, while the dissolved ammonium phosphate and urea components provide immediately available and prolonged nitrogen nutrition. Thus, the resulting system may function as a binary NP fertilizer with combined short-term and long-term nutrient release effects. However, this approach should be considered rather palliative, since it does not fully solve the problem of obtaining chemically pure Hydroxyapatite as an independent product. In addition, maintaining the long-term colloidal stability of the Hydroxyapatite suspension requires additional investigation and

optimization of stabilizing systems. Among the promising stabilizing agents are polyhexamethylene guanidine succinate compounds, which may improve suspension stability while preserving the fertilizing functionality of the product (Natiashnyi *et al.*, 2023).

B. A more technologically attractive and industrially scalable route is the integration of all generated water fractions, including wash water and the primary centrifugate, followed by their homogenization and membrane treatment, particularly by reverse osmosis. Such an approach can potentially provide concentration of dissolved residual compounds by at least 5–7 times (Tyvonenko, A. *et al.*, 2023). In the performed experiments, depending on reactor loading, 1 to 3 g of Hydroxyapatite were obtained, while the total collected wastewater volume reached no less than 700 ml. Under such conditions, membrane concentration may yield approximately 100–200 ml of concentrate and 500–600 ml of purified permeate. Based on mass-balance estimation, the concentrate may theoretically be regarded as a non-standardized liquid NP fertilizer containing 8-12% N and up to 5% P<sub>2</sub>O<sub>5</sub>. This route not only minimizes wastewater discharge but also increases the overall material efficiency of the process through nutrient recovery.

The kinetic investigations performed in this work make it possible to recommend 50–60 minutes as the rational duration of homogeneous precipitation for complete Hydroxyapatite formation under the studied conditions. Within this period, the formation of crystallization centers and subsequent crystal growth reach practical completion. The obtained kinetic parameters may be considered as a basis for scaling up the process and for developing automated process control systems based on turbidity measurements.

The performed infrared spectroscopy and X-ray phase analysis confirmed that the dominant solid phase after synthesis and repeated washing is Hydroxyapatite. The obtained analytical data demonstrate the feasibility of Hydroxyapatite synthesis under homogeneous precipitation conditions and confirm the applicability of the CSTR model as an adequate approximation of the hydrodynamic conditions in the reactor. The use of urea as an internal precursor of ammonium hydroxide ensures uniform pH generation throughout the reaction medium, minimizes local supersaturation zones, and promotes the formation of highly dispersed Hydroxyapatite particles.

A critical technological stage of the process is the purification of Hydroxyapatite from residual reaction components. Experimental results show that no fewer than seven consecutive centrifugal washing cycles are required to achieve sufficient purity of the target product. However, this stage is directly associated with the generation of considerable amounts of wash water, which creates an additional environmental and technological challenge.

In general, industrial Hydroxyapatite production by the homogeneous precipitation method from calcium saccharate under conditions approximating a CSTR may consist of the following technological stages:

- synthesis of calcium saccharate;
- preparation of diammonium phosphate solution;
- preparation of urea solution;
- mixing of calcium saccharate, diammonium phosphate, and urea solutions in a stirred and heated reactor;
- heating of the homogeneous reaction medium to a temperature not lower than 60°C, preferably 80–100°C;

- maintaining the reaction medium at the selected temperature for 50–60 minutes until completion of Hydroxyapatite precipitation;
- cooling of the reaction medium;
- centrifugation and separation of the Hydroxyapatite precipitate;
- addition of purified water followed by repeated centrifugal washing of the precipitate for 6–7 cycles, with separate collection of wash water after each cycle;
- drying of the completely washed Hydroxyapatite precipitate;
- homogenization and purification of the accumulated wash water by membrane separation methods;
- concentration of nutrient-containing wastewater fractions;
- conditioning and packaging of the concentrate as a liquid NP fertilizer product;
- return of the permeate to the stages of calcium saccharate, diammonium phosphate, and urea solution preparation.

*Thus*, the proposed technological concept combines Hydroxyapatite synthesis with an integrated wash water recycling strategy and simultaneous recovery of a secondary-value fertilizer product. Such an approach corresponds to the principles of circular economy, resource conservation, nutrient recovery, and zero-waste chemical synthesis. The obtained experimental data confirm the technological feasibility of integrating Hydroxyapatite synthesis with internal water recycling without compromising the formation of the target calcium-phosphate phase.

Further studies should be focused on the detailed investigation of long-term wash water recycling effects, impurity accumulation in the closed-loop system, membrane concentration efficiency, and the agronomic properties of the resulting NP fertilizer concentrate. Particular attention should also be paid to the influence of recycled wash water composition on the physicochemical characteristics, phase purity, and morphology of Hydroxyapatite obtained in subsequent synthesis cycles.

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### **Conflict of Interest**

The author declares that there is no conflict of interest.

### **Acknowledgements:**

Not applicable.

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### Appendix

Table 1. – Kinetics of changes in the percentage of light transmittance during hydroxyapatite formation.

Time, min	0,5	3	5	10	20	30	45	60	75	90
Percentage of transmittance	92	91	49	42	40	36	35	34	34	34

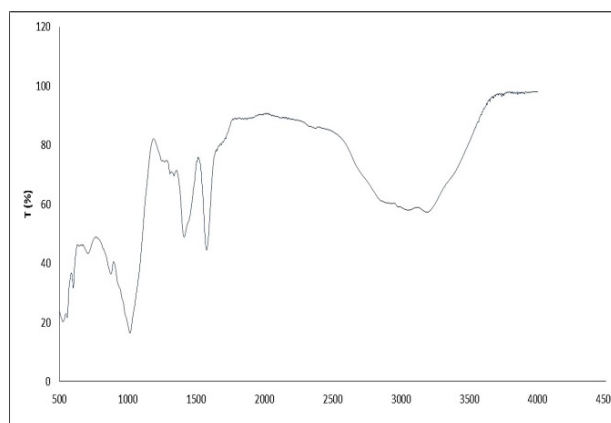


Figure 1. Infrared spectrum of unwashed hydroxyapatite

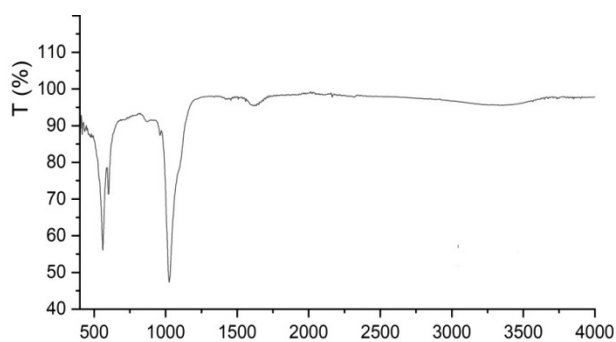


Figure 2. Infrared spectrum of washed hydroxyapatite

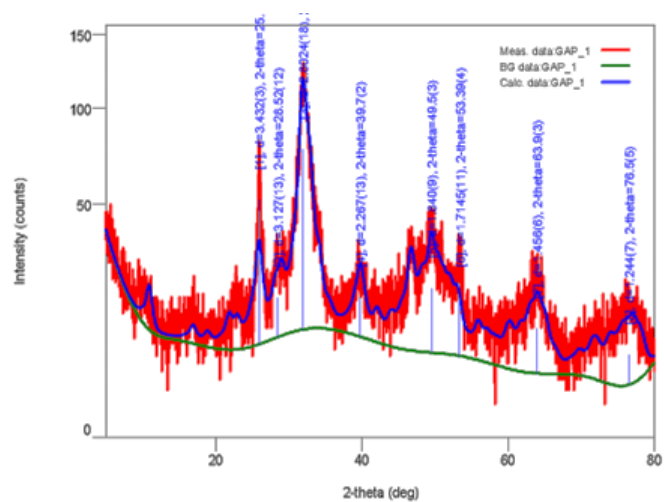


Figure 3. Results of X-ray phase analysis of the hydroxyapatite precipitate