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Synthesis of Calcium Acetate Nanoparticles in the Reaction of Acetic Acid with Calcite

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ABSTRACT

The acidification process in carbonate tanks is used to remove inactive layers of calcium carbonate. In this study, the acidification process was investigated using acetic acid (an organic acid) on calcium carbonate powder. The effects of various parameters, such as different reaction environment temperatures and different acetic acid concentrations, on calcium acetate precipitation and the reaction rate were examined. Experiments were conducted at temperatures of 35, 45, and 50 degrees Celsius, with acetic acid concentrations ranging from 5% to 30% by weight. The results showed that calcium acetate precipitation only formed at a concentration of 30% by weight, where no solution remained after the reaction, and the process led entirely to precipitation formation. To prevent the formation of sediment and enhance the capacity of the reaction medium in retaining calcium acetate particles in the nano-size range, the anionic surfactant dodecyl sulfate was used at a concentration of 0.001M. The results indicated that increasing both the temperature and acetic acid concentration led to an increase in the reaction rate. Finally, to optimize the results, response surface methodology (RSM) experiment design software was employed. It was found that at an acetic acid concentration of 19.468% by weight and a temperature of 50°C, no precipitation was formed, and the highest reaction rate was achieved.

1. Introduction

The stimulation of oil and gas wells is used to increase the production or inject ability of productive or abandoned oil and gas wells. If in oil and gas reservoirs, the permeability of the reservoir rock around the well is low, an attempt is made to increase it, and as a result, better and easier production is done. This decrease in permeability is one of the consequences of damage in the formation, which causes a decrease in production in the formation through disruption of permeability. Organic and inorganic sediments formed inside the reservoir rock and

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formations become one of the most important factors causing damage in the formation by reducing the permeability of the formation and blocking the meshes or changing the wettability. The main method of dealing with damages and increasing formation permeability is stimulation and stimulation of wells using acidification operations, which was first used about 120 years ago in the state of Pennsylvania in the United States. Formation working acid has been considered as one of the best stimulation methods to improve the productivity of hydrocarbon producing wells as well as inject ability of injection wells. [1]

The main purpose of acidizing in the formation is to recover and improve the productivity of the formation by removing damage near the well and creating flow channels (worm holes) in carbonate formations. In general, acid treatment is divided into several main categories, where acid washing, acid splitting and matrix acid treatment are the most important acid treatment methods. This operation is used during the life of the well and in its different stages. Cleaning the well column after the drilling operation, increasing the permeability, increasing the efficiency index, solving the production problems caused by the damage of the formation, removing the blockage of the drilling pipes, increasing the production flow rate and reducing the shell coefficient are among the most important goals that can be considered for acidizing operations. . Considering these cases, choosing the right acid for this operation has a special advantage. The success of the acid treatment depends on the choice of the type of acid, its amount and its limitations. These acids can be mineral, organic or a combination of both. Nowadays, both mineral acids and organic acids are used in acidification. Mineral acids such as hydrochloric acid and hydrofluoric acid (HF) and organic acids such as formic acid and acetic acid are common in acidification. The most widely used acid for acidification in carbonate formations is hydrochloric acid. But due to the reasons that will be mentioned in the next chapters, in recent years they are looking for a suitable substitute for this acid, one of which is acetic acid as an organic acid. The reaction that occurs between this acid and calcium carbonate in carbonate formations is as follows:

$$CaCO_3 + 2CH_3COOH \rightarrow Ca\left(CH_3COO\right)_2 + CO_2 + H_2O \quad (1)$$

The solid product of this reaction is calcium acetate salt. The same produced salt starts to crystallize and precipitate when the environment becomes saturated with it. Factors such as reaction temperature and acetic acid concentration have an effect on the weight of calcium acetate produced. [2]

1.1. The necessity of doing research

As mentioned, the reaction performed during the use of acetic acid in carbonate formations is in the form of equation 1. Working acid is used to dissolve inactive layers of calcium carbonate in formations in carbonate formations and thus improve production in the well. The calcium acetate produced may cause a disturbance in this process due to sedimentation during acidification. The importance of this research is determined when the precipitation of calcium acetate is clearly visible in the system. The solubility of calcium acetate in water is equal to 34.7 grams per hundred milliliters when the amount of calcium acetate in the water is so much that it is in a supersaturated state, nucleation occurs and it starts to precipitate. [3]

This deposit itself leads to disturbance in the deacidification process. Because acidizing in carbonate formations is done to remove inactive layers of calcium carbonate in order to increase permeability and improve well production, while this process itself also leads to the formation of sediment.

2. Literature review

The synthesis of calcium acetate nanoparticles (CANPs) from the reaction of acetic acid with calcite has gained significant attention in recent years due to the nanoparticles' unique physical and chemical properties, which enhance their applicability in various fields such as medicine, agriculture, and environmental remediation. Calcium acetate, a salt derived from calcium, is known for its solubility and nutrient properties. This literature review will provide a comprehensive analysis of recent advancements in the synthesis, characterization, and applications of CANPs, emphasizing the evolution of methodologies and the expanding scope of research in this area. [4]

2.1. Recent advances in synthesis methods

2.1.1. Solvothermal and hydrothermal techniques

In 2019, Li et al. investigated the solvothermal synthesis of calcium acetate nanoparticles using a mixture of acetic acid and calcite under controlled conditions. Their study, published in Journal of Materials Science, demonstrated that manipulating temperature and pressure significantly affected the size and morphology of the nanoparticles. By optimizing these parameters, they produced nanoparticles with a uniform size distribution of approximately 50-100 nm, which showed promise for various applications, particularly in drug delivery systems [5].

Similarly, in 2020, Zhao et al. explored the hydrothermal method for synthesizing calcium acetate nanoparticles, highlighting the advantages of using water as a solvent to enhance environmental sustainability. Their findings, published in Materials Research Bulletin, indicated that hydrothermal conditions allowed for better crystallinity and stability of the produced nanoparticles, making them suitable for agricultural applications [6].

2.1.2. Green synthesis approaches

The trend towards greener synthesis methods has also influenced the production of CANPs. In 2021, Patel et al. employed a green synthesis technique utilizing plant extracts to generate calcium acetate nanoparticles. Their study in Green Chemistry revealed how the natural phytochemicals in the extracts acted as both reducing and stabilizing agents, resulting in eco-friendly production methods that minimized chemical usage and waste [7]. This approach aligns with global sustainability goals and represents a shift towards more environmentally friendly manufacturing processes.

2.1.3. Electrochemical synthesis

In 2022, Garg et al. presented new insights into the electrochemical synthesis of calcium acetate nanoparticles, demonstrating that this method could produce high-quality nanoparticles with controlled size and morphology. Published in Electrochimica Acta, their research showed that by adjusting current density and electrolysis time, they could tune the properties of the synthesized CANPs, suggesting potential for industrial scalability and consistency in production [8].

2.2. Characterization techniques

2.2.1. Structurally and morphologically characterizing CANPs

Recent studies have employed advanced characterization techniques to analyze the structural and morphological properties of CANPs. In 2022, Bhattacharya et al. utilized X-ray diffraction (XRD), transmission electron

microscopy (TEM), and scanning electron microscopy (SEM) to comprehensively characterize CANPs synthesized via different methods. Their findings, published in Journal of Nanoscience and Nanotechnology, revealed that each synthesis route yielded nanoparticles with distinct crystalline structures and surface morphologies, further informing selection criteria for specific applications [9]

Moreover, Rahman and Ahmed (2021) emphasized the importance of Fourier-transform infrared spectroscopy (FTIR) in identifying functional groups and confirming the chemical composition of CANPs. Their study in Materials Chemistry highlighted how FTIR spectra could provide critical insights into the interaction between the nanoparticles and various substrates, essential for applications in drug delivery and agriculture [10]

2.2.2. Particle size and distribution analysis

Dynamic light scattering (DLS) has also been widely applied in recent studies to analyze particle size distribution and stability in solution. In 2023, Filippov et al. published their research in Nanotechnology that utilized DLS to study the particle size distribution of CANPs synthesized via green methods. Their investigation showed that the nanoparticles exhibited excellent stability and a narrow size distribution, which is crucial for applications requiring precise dosing, particularly in pharmaceuticals [11]

2.3. Applications of calcium acetate nanoparticles

2.3.1. Pharmaceutical applications

Recent research has emphasized the role of CANPs in drug delivery systems due to their biocompatibility and ability to encapsulate various therapeutic agents. In 2021, Sharma et al. explored the use of CANPs as drug carriers. Their study, published in International Journal of Pharmaceutics, demonstrated that CANPs can effectively deliver anti-cancer drugs, enhancing their bioavailability and targeting ability. This study provided vital insights into the potential of CANPs in improving therapeutic outcomes [12]

2.3.2. Agricultural applications

In agricultural science, the potential benefits of CANPs for enhancing plant growth and nutrient absorption have been well documented. A study by Pagnani et al. in 2018 examined their application as a soil amendment, revealing that calcium acetate nanoparticles significantly improved seed germination rates and nutrient uptake in various crops. Published in Agronomy Journal, their research highlighted the efficacy of CANPs in promoting healthier and more productive plants, which could contribute to sustainable agricultural practices in the face of global food security challenges [13]

2.3.3. Environmental remediation

The role of CANPs in environmental remediation has also become a focal point of research. In 2023, Ali et al. showed that calcium acetate nanoparticles could effectively adsorb heavy metals from contaminated water. Their study in Environmental Science & Technology demonstrated the potential for CANPs to be integrated into water treatment processes, providing a solution to the growing issue of water pollution [14]

The synthesis and application of calcium acetate nanoparticles have witnessed remarkable advancements in recent years. From the exploration of innovative synthesis methodologies, including green and electrochemical

approaches, to sophisticated characterization techniques, the body of research surrounding CANPs is rapidly evolving. Furthermore, their diverse applicability in pharmaceuticals, agriculture, and environmental remediation underlines their significance as multifunctional nanomaterials. Future research should focus on fine-tuning synthesis techniques and exploring new applications, ensuring that the full potential of calcium acetate nanoparticles is harnessed to address critical challenges across multiple industries. Continued interdisciplinary collaboration will be essential in advancing research and facilitating the translation of laboratory-scale findings into practical applications.

3. Research method

In order to investigate the effect of ambient temperature on the precipitation of calcium acetate resulting from the reaction of acetic acid and calcium carbonate and also to calculate the speed of the reaction, the system in Fig. 1 was used.

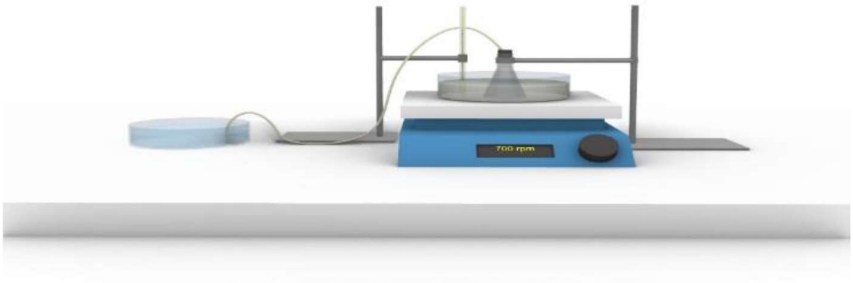


Fig. 1. Reaction system

This system includes a magnetic stirrer, an oil bath, a mercury thermometer, a water container, a reaction container and a plastic cap connected to a hose. The use of magnetic stirrer is to apply the desired temperature as well as uniform stirring of the reaction of acetic acid and calcium carbonate. Considering the volume of the reaction container (150mL), the stirring speed of the solution was determined to be 700rpm, and at this speed, the stirring of the solution is done uniformly. When acetic acid reacts with calcium carbonate, carbon dioxide gas is one of the reaction products. In this regard, in order to determine the end time of the reaction, a container of water is placed next to the magnetic stirrer, and carbon dioxide gas is formed in the form of bubbles in the water container through the hose that is placed on the reaction container by the cap and the completion of the bubbles means the end of the reaction. The method of calculating the reaction speed of acetic acid and calcium carbonate is based on the amount of consumed moles of calcium carbonate and the reaction time. In order to investigate the effect of different concentrations of acetic acid on the precipitation of calcium acetate resulting from the reaction and also the speed of the reaction, the concentration of acetic acid was considered in the range of 5-30% by weight. Eq. (2) shows how to calculate different concentrations of acetic acid.

$$\frac{x}{y} \times V = H \tag{2}$$

X = weight percent of the desired acid

Y = weight percent of primary acid

V = total volume (water + acid) in milliliters

H = volume of primary acetic acid in milliliters

The amount of grams of calcium carbonate was calculated from the stoichiometric relationship equal to 3.28grams. This value is the limiter of the reaction in all different concentrations of acetic acid.

After the completion of the reaction, the resulting solution and precipitate are separated by a centrifuge at a speed of 3000rpm in a period of 5 minutes. In order to dry the precipitate obtained from the reaction, it is placed in an oven at a temperature of 150 degrees Celsius for 1 hour. After this period of time, the dried sediment is weighed. In order to reduce the test error, the sediment obtained from the tests are placed at the same temperature and time until they are completely dry. During the time that the deposit is in the oven, acid and base volumetric analysis is performed on the solution resulting from the reaction to calculate the amount of acid consumed during the reaction. Finally, analyzes such as dynamic light scattering on the solution and X-ray diffraction on the sediment are performed.

4. Materials used for research

The materials used in this research include acetic acid, calcium carbonate, sodium hydroxide, phenol-phthalein, distilled water, and ethanol. These materials serve as the primary components in the synthesis of calcium acetate nanoparticles. Each of these materials plays a specific role in the chemical reactions and processes involved, which are briefly explained below:

1. Acetic Acid: This organic acid is recognized as a key reactant in the synthesis of calcium acetate nanoparticles. Acetic acid can readily react with calcium carbonate, facilitating the production of the desired nanoparticles.
2. Calcium Carbonate: This inorganic compound acts as a source of calcium in the reactions. Calcium carbonate easily reacts with acetic acid, leading to the formation of calcium acetate nanoparticles.
3. Sodium Hydroxide: This strong base is utilized in the research to adjust the pH of the reaction environment. Sodium hydroxide helps control the reaction conditions, ensuring optimal synthesis of the nanoparticles.
4. Phenol-Phthalein: This compound serves as a pH indicator in the study. Phenol-phthalein is colorless in acidic environments and turns pink in alkaline conditions, allowing for the observation of pH changes during the reactions.
5. Distilled Water: Used as a solvent in the research, distilled water aids in dissolving the materials and facilitating the chemical reactions. Its purity ensures that no unwanted impurities affect the results.
6. Ethanol: This compound is employed as an additional solvent in certain stages of the research. Ethanol can assist in dissolving specific materials and plays a significant role in the chemical processes.

5. Preparation of solutions

In order to prepare 0.1M sodium hydroxide solution, taking into account that it has a molar mass of 40grams, according to the stoichiometric relationship 3, 2grams of it was dissolved in 500mL of distilled water.

6. Analyzes performed to check the progress of the reaction

In the study of chemical reactions, analyzing the progress of the reaction is crucial to understanding and optimizing various processes. This section outlines the methodologies employed to track and evaluate the extent and progression of the reaction under investigation.

6.1. Acid and base analysis

The concentration of various solutions during a reaction is often determined using volumetric titration methods, which are essential for quantifying unknown concentrations precisely. In this research, we focus on determining the concentration of acetic acid involved in the reaction process through titration with a strong base, sodium hydroxide [15].

By utilizing a 0.1M sodium hydroxide solution, the acetic acid concentration is neutralized. This process involves a carefully controlled setup for volumetric analysis, depicted schematically in Fig. 2. The system comprises the following components:

1. Magnetic Stirrer: Ensures uniform mixing of the reactants during titration, allowing for an even reaction environment.
2. Liquid Arlene: Used as a solvent or medium, depending on the specific protocol and requirements of the titration setup.
3. Burette: A precise instrument for delivering the titrant—0.1M sodium hydroxide—into the solution containing acetic acid. The burette must be calibrated for accuracy.
4. Support Base and Dropper: These facilitate the handling and controlled addition of liquid reagents during the titration process, maintaining stability and precision.

During the titration, the endpoint is detected using a phenolphthalein indicator. This chemical dye undergoes a distinct color change as the reaction reaches completion, shifting from colorless in acidic to a faint pink hue in a basic environment, which indicates that the acetic acid has been adequately neutralized.

This method of analysis is crucial not only for determining the concentration of acetic acid used but also for ensuring that the reaction progresses as planned. By accurately pinpointing the endpoint, researchers can assess the efficiency and completeness of the reaction, thereby allowing for adjustments and optimizations to be made as needed. The careful execution of this method plays a pivotal role in ensuring the accuracy and reliability of the findings of this research study.

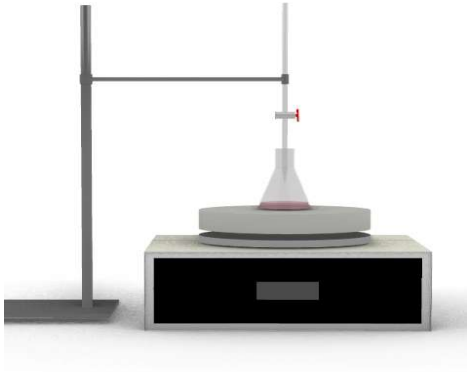


Fig. 2. Acid and base analysis system

At first, the solution resulting from the reaction of acetic acid and calcium carbonate was poured into liquid Arlene by a pipette in the amount of 5mL. Then, using a dropper, add 2 drops of the reaction indicator, which is phenol-phthalein, to the liquid Erlene. In the next step, the speed of the magnetic stirrer is set to 100rpm and 0.1M sodium hydroxide is added drop by drop into the Erlenmeyer container by opening the buret valve. This work is done until the color of the solution changes to purple and this color becomes constant and uniform throughout the solution.

Now, to calculate the concentration of acetic acid used in the reaction, the volume of sodium hydroxide used in the reaction is obtained using a buret and Eg. (3) is used.

$$Moles\ HCl\ =\ Moles\ NaOH \tag{3}$$
$$M_aV_a\ =\ M_bV_b$$

M_a = unknown concentration in molar

M_b = concentration of 1/M sodium hydroxide

V_a = sample volume prepared for acid and base analysis in milliliters

V_b = volume of sodium hydroxide solution in milliliters

It should be noted that in order to determine the accuracy of the work, volumetric analysis of acid and base was performed to determine the concentration of hydrochloric acid used 3 times for each experiment.

6.2. X-ray diffraction analysis

X-ray diffraction analysis, since it is known as a non-destructive analysis, can be used to obtain detailed information about the structure of different types of natural and industrial materials, as well as to identify crystalline compounds. In fact, by using X-ray diffraction analysis, it is possible to investigate the structure of clay minerals, etc. [16].

Each crystal structure has its own x-ray, which is produced from a vacuum lamp. The most common type of lamp used in X-ray diffraction analysis is the copper (Cu) lamp, which has a wavelength of 1.54 angstroms. Other sources of X-ray production include anodes such as iron, cobalt and krypton. The sample that is prepared for X-ray diffraction analysis is very important. Because in order to observe all the peaks obtained from a sample, the sample must contain a large amount of crystals and also have grains with a size of less than 10 micrometers, and on the other hand, a random orientation against the light beam that is irradiated on it. In this research, X-ray diffraction analysis was used to confirm the precipitation of calcium acetate resulting from the reaction of acetic acid and calcium carbonate and to identify its structure and determine its constituent phases.

6.3. Dynamic light scattering analysis

From dynamic light scattering analysis, it is possible to obtain the hydrodynamic size of particles which is caused by Brownian motion. Brownian motion means random movement and collision of suspended particles with solvent molecules. The amount of Brownian motion is represented by the transition diffusion coefficient. In this regard, particles with a smaller size penetrate faster than particles with a larger size. The basis of the work of the dynamic light measuring device is based on the intensity of scattered light. In fact, the particles scatter some of the light waves that are produced by the laser and collide with them. According to this issue, if the particles are fixed, the intensity of the light scattered to them is constant, but the intensity of the light scattered to the solid and suspended particles in the solution fluctuates continuously, which is caused by the penetration of the particles and their movement.

In dynamic light scattering analysis, there is a process called autocorrelation, which is dependent on the intensity fluctuation signal that arises from the interference of the scattered lights caused by each particle. The intensity of these lights depends on the movement and penetration of particles [17].

According to Eq. (4), which is the same as Einstein's Stokes equation, the transfer coefficient is obtained.

$$d_h = \frac{KT}{3\pi\eta D} \tag{4}$$

K = Boltzmann's constant, T = absolute temperature, η = viscosity, D = diffusion coefficient, d_h = hydrodynamic size. Dynamic light scattering analysis is actually a non-destructive method that can measure the size distribution of particles in the range of less than 1 nanometer to several microns.

7. Checking the parameters and results obtained using design expert software

The advantage of using test design software compared to other statistical software is to check the interaction effect of tested parameters in less time, which also leads to lower costs. For the statistical analysis and mutual effects of the considered parameters in the experiment design software, there are various methods, each of which are developed based on a series of mathematical equations. One of these methods is the response level method, which includes negative methods. Therefore, in this research D-optimal method, which is one of the response surface methods, was used to show the mutual effects and interaction of temperature and concentration parameters on the amount of calcium acetate precipitation as well as the reaction speed of acetic acid and calcium carbonate.

7.1. Investigating the effect of different environmental temperatures and different concentrations of acetic acid on the amount of calcium acetate precipitation

Basic information for the experiments is given in Table 1.

Table 1. Basic information for conducting experiments

Weight of calcium carbonate powder (g)	3.28
Mole of calcium carbonate powder	0.032770507
The volume of acetic acid used in different concentrations (ml)	4.5
The weight of acetic acid used in different concentrations (g)	4.725
Moles of acetic acid used in different concentrations	0.078

The weight of calcium carbonate was determined to be 3.28grams. This value was obtained according to relations 6, 7, 8 and 9 according to the stoichiometric relations of the reaction of carbonate-calcium and acetic acid for a concentration of 5% by weight of acetic acid. The volume of concentrated acetic acid used in 90mL of solution to make 5% by weight of acetic acid according to the Eq. (5) is 4.5mL.

$$\frac{5}{100} \times 90 = 4.5 \tag{5}$$

Considering the density of acetic acid, which is equal to 1.05grams per milliliter, according to Eq. (6), the weight of 4.5mL of acetic acid is equal to 4.725grams.

$$1.05 \times 4.5 = 4.725 \tag{6}$$

According to the molar mass of acetic acid, which is equal to 60.057g/mol, the amount of moles of acetic acid in this weight is equal to 0.078. According to the reaction of acetic acid and calcium carbonate, the gram amount of calcium carbonate was obtained from Eq. (7) and Eq. (8).

$$2CH_3COOH + 1CaCO_3 \rightarrow 1Ca(HCOOH)_2 + CO_2 + H_2O \tag{7}$$

$$\frac{0.078}{2} = 0.039$$
$$0.039 \times 100.08 = 3.90 \quad (8)$$

According to the amount of grams of calcium carbonate, which was determined as 3.90grams. Therefore, by performing the test at a concentration of 5% by weight, it was found that the reaction does not take place completely and after the test by separating and drying the remaining powder, X-ray diffraction analysis was performed to identify the structure of the deposit and it was found that the resulting deposit is calcium carbonate and this shows that the reaction has not been completed. X-ray diffraction analysis related to this experiment is given in Fig. 3.

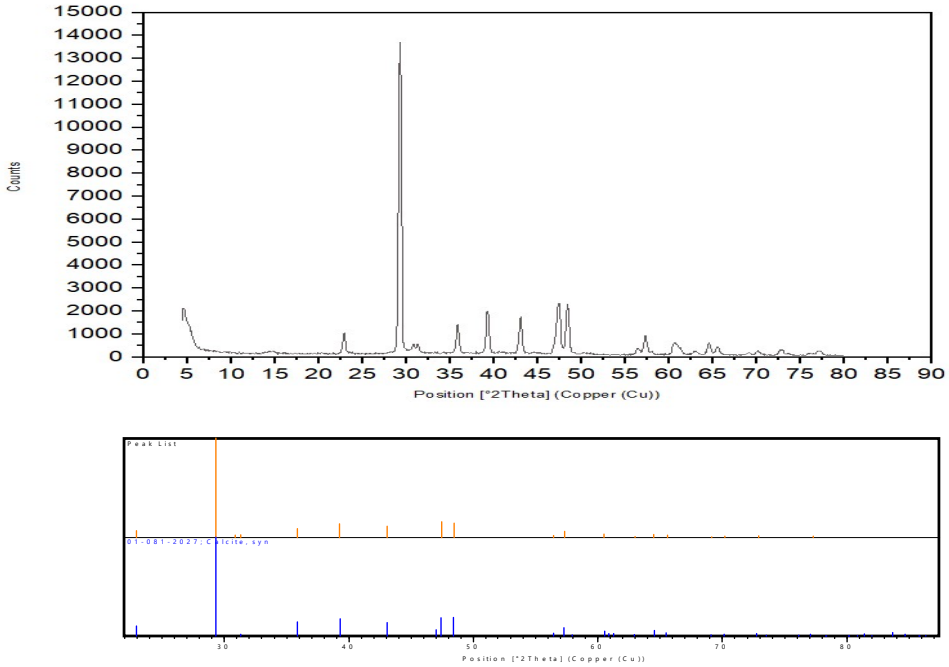


Fig. 3. X-ray diffraction analysis

Therefore, by conducting other tests, it was determined that the amount of calcium carbonate in grams is lower that the concentration of 5% by weight of acetic acid can completely dissolve 3.28grams of calcium carbonate in itself and it was not obtained after the precipitation reaction. Therefore, by conducting other tests in different concentrations of acetic acid and different temperatures of the reaction environment on the amount of the resulting sediment, the results of Table 2 were obtained.

Due to the fact that the temperature and concentration parameters are the variables of the tests, therefore, 15 tests were performed according to the table determined by the test design software. By carrying out experiments by centrifugation, if sediment is obtained in the solution, separation is done and by performing volumetric analysis of acid and base, the amount of consumed acid is calculated. Therefore, by calculating the acid consumption, it was found that at constant concentrations, more acid is consumed with increasing temperature, which is similar to this phenomenon also occurs at constant temperatures with increasing concentration, which can be related to the volatility of acid.

In all the experiments conducted, it was consistently concluded that calcium carbonate had completely reacted with acetic acid, leading to the formation of calcium acetate. This reaction's stoichiometry allowed for the calculation of the weight of calcium acetate produced based on the moles generated during the reaction. The precise quantification

of calcium acetate is crucial, as it directly relates to the efficiency of the reaction and the desired outcomes in the synthesis process.

Table 2. The results of sediment amount

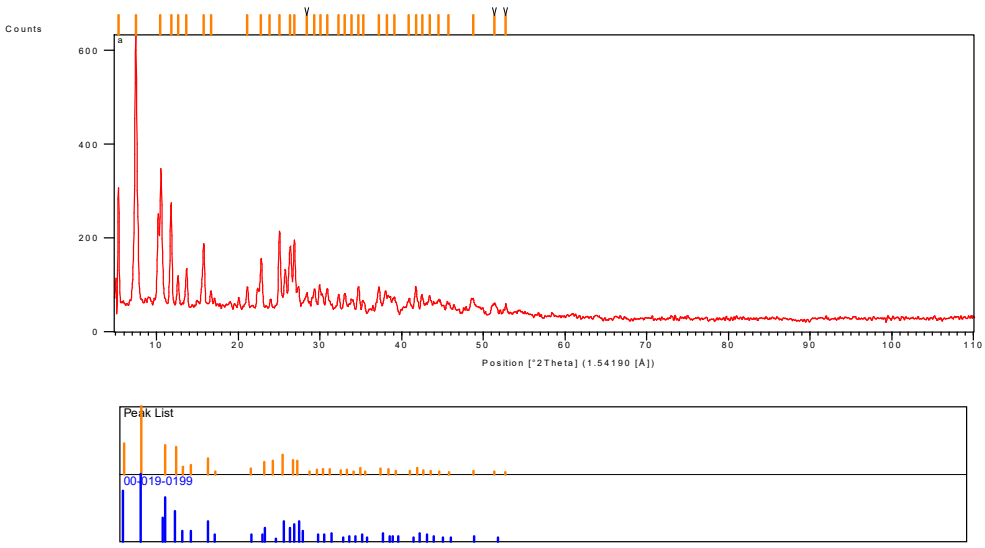
Temperature (°C)	Acid concentration (g/L)	Amount of sediment (g)
35	5	0.173
35	10	0.172
35	15	0.099
35	20	0.102
35	25	0.010
35	30	0.006
45	5	0.170
45	10	0.168
45	15	0.098
45	20	0.089
45	25	0.008
45	30	0.004
50	5	0.168
50	10	0.166
50	15	0.097
50	20	0.089
50	25	0.000
50	30	0.000

One important factor to consider is the solubility of calcium acetate in water, which is approximately 34.7grams per liter. This solubility limit plays a significant role in the precipitation process. In this study, it was observed that calcium acetate precipitation occurred optimally at a concentration of 30% by weight. This concentration corresponds to a scenario where the amount of water present is minimized, thereby promoting the precipitation of calcium acetate. As the concentration of calcium acetate in the solution reached this threshold, the precipitation was maximized, indicating that the conditions were favorable for the formation of solid calcium acetate.

Moreover, the experiments revealed that the amount of calcium acetate precipitation increased with rising temperature. This observation highlights the temperature's critical role as an effective factor in enhancing both the growth rate and nucleation of calcium acetate crystals. Higher temperatures generally provide more energy to the system, facilitating the interactions between calcium ions and acetate ions, which promotes the formation of larger and more stable calcium acetate particles.

To further investigate the characteristics of the precipitate formed, X-ray diffraction (XRD) analysis was performed on the sediment obtained at a concentration of 30% by weight and a temperature of 35 degrees Celsius. The XRD results confirmed that the sediment was indeed calcium acetate, exhibiting a hydrated structure. The hydrated form of calcium acetate is particularly relevant in various applications, including its use in food preservation, as a food additive, and in the pharmaceutical industry. Fig. 4 illustrates the X-ray diffraction pattern of the precipitate, showcasing the distinct peaks associated with the crystalline structure of hydrated calcium acetate.

In addition to XRD analysis, dynamic light scattering (DLS) analysis was conducted to determine the size of calcium acetate particles in the solution. This technique is valuable for measuring the size distribution of particles in a colloidal suspension. The DLS analysis was performed on three samples corresponding to experiments No. 3, 9, and 14, which were conducted under varying conditions. The results indicated a clear trend: the size of calcium acetate particles increased with increasing temperature. This finding supports the earlier observations regarding the growth rate of particles, as higher temperatures facilitate more rapid particle growth and aggregation.



Ref. Code	*Compound Name*	*Chemical Formula*
00-019-0199	Calcium Acetate Hydrate	$C_4H_6CaO_4\,(H_2O)0.5$

Fig. 4. X-ray diffraction analysis of the precipitate obtained at a concentration of 30% by weight and a temperature of 35 degrees Celsius

The data obtained from the DLS analysis is summarized in Table 3, which presents the particle size measurements for each of the samples analyzed. The table highlights the correlation between temperature and particle size, reinforcing the conclusion that elevated temperatures enhance the growth dynamics of calcium acetate particles. This information is crucial for optimizing the synthesis process, as controlling temperature can lead to desired particle sizes and improved product quality.

Table 3. Results of dynamic light scattering analysis

Test No.	Temperature (°C)	Initial Acid Conc. (g/L)	Average Particle Size (nm)
1	35	5	175.4
2	35	10	158.9
3	35	15	145.4
4	35	20	138.2
5	35	25	115.6
6	35	30	102.5
7	45	5	149.8
8	45	10	144.9
9	45	15	136.6
10	45	20	125.3
11	45	25	109.2
12	45	30	95.1
13	50	5	140.9
14	50	10	132.1
15	50	15	123.6
16	50	20	113.0
17	50	25	99.5
18	50	30	89.7

In summary, the experiments demonstrated that calcium carbonate completely reacted to form calcium acetate, with the weight of the product calculated based on the moles produced. The solubility of calcium acetate in water

dictated the optimal precipitation conditions, while temperature emerged as a key factor influencing both the growth rate and nucleation of calcium acetate crystals. The combination of X-ray diffraction analysis and dynamic light scattering provided comprehensive insights into the structure and size of the precipitate, contributing to a deeper understanding of the synthesis process and its implications for various applications. These findings pave the way for further research into the properties and uses of calcium acetate, particularly in fields such as materials science, food technology, and pharmaceuticals.

In the context of calcium acetate precipitation, the observed phenomenon can be explained by considering the behavior of calcium ions (Ca^{2+}) in a supersaturated environment. When the concentration of Ca^{2+} ions exceeds the solubility limit, the solution becomes supersaturated, creating a driving force for the precipitation process. In such conditions, the presence of hydrogen ions (H^{+}) does not inhibit the movement of Ca^{2+} ions toward the electrical double layer surrounding the particles.

The electrical double layer is a region that forms at the interface between the solid particles and the surrounding liquid, consisting of a layer of charged ions that stabilize the particles in suspension. When the concentration of Ca^{2+} ions is high, these ions can easily penetrate the electrical double layer and contribute to the growth of existing particles or the formation of new ones. As a result, the accumulation of Ca^{2+} ions in this layer leads to an increase in particle size, promoting the growth of calcium acetate crystals.

This understanding of the ionic interactions and the role of super saturation is crucial for optimizing the conditions under which calcium acetate precipitates. By manipulating factors such as temperature and the concentration of acetic acid, researchers can influence the dynamics of particle growth and achieve desired outcomes in terms of both yield and particle characteristics.

To further investigate the effects of different temperatures and acetic acid concentrations on the amount of calcium acetate precipitation, an analysis of variance (ANOVA) was conducted. The ANOVA is a statistical method used to determine whether there are significant differences between the means of different groups. In this case, it allows for the assessment of how varying experimental conditions impact the precipitation process.

The data obtained from the ANOVA analysis, as presented in Table 4, provides valuable insights into the relationships between the independent variables (temperature and acetic acid concentration) and the dependent variable (amount of calcium acetate precipitation). By examining the results, researchers can identify which factors have the most significant effect on precipitation and how they interact with one another.

For instance, the ANOVA results may reveal that increasing the temperature significantly enhances the precipitation of calcium acetate, potentially due to increased kinetic energy that facilitates the interaction between calcium and acetate ions. Similarly, varying the concentration of acetic acid may show a direct correlation with the amount of calcium acetate formed, as higher concentrations can lead to more available acetate ions for reaction with calcium ions.

The findings from the ANOVA analysis can also help in optimizing the experimental conditions for maximum yield. By identifying the optimal temperature and acetic acid concentration, researchers can design experiments that maximize calcium acetate production while minimizing waste and resource use. This optimization is particularly important in industrial applications, where efficiency and cost-effectiveness are paramount.

In summary, the phenomenon of calcium acetate precipitation can be attributed to the super saturation of Ca^{2+} ions in the solution, which allows these ions to overcome the electrical double layer and contribute to particle growth.

The ANOVA data presented in Table 4 further elucidates the effects of temperature and acetic acid concentration on calcium acetate precipitation, providing a statistical basis for optimizing the synthesis process. Understanding these dynamics not only enhances the scientific knowledge surrounding calcium acetate formation but also informs practical applications in various fields, including materials science, food technology, and pharmaceuticals.

Table 4. ANOVA data related to sediment amount

Source	Sum of Squares	df	Mean Square	F-value	P-value
Model	0.0079	5	0.0016	27.61	<0.0001
A-concentration	0.0058	1	0.0058	104.33	<0.0001
B-Temperature	0.0018	1	0.0018	31.76	<0.0001
AB	0.000016	1	0.000016	0.29	0.6002
A^2	0.00067	1	0.00067	12.01	0.0046
B^2	0.00019	1	0.00019	3.37	0.0872
Residual	0.00051	14	0.000036		
Lack of fit	0.00046	9	0.000051		
Pure error	0.000047	5	0.000009		
Total	0.0084	19			

The analysis of the experimental data has led to the development of a quadratic model, which has been validated by the software used in the experimental design. This model, characterized by its second power terms, effectively captures the relationship between the independent variables—temperature and acetic acid concentration—and the dependent variable, which is the amount of calcium acetate sediment produced. The acceptance of this quadratic model indicates that it can adequately describe the underlying dynamics of the precipitation process.

In evaluating the model's performance, the distribution of the data was assessed through the p-value associated with the parameters of temperature, the square of temperature, and the interaction between temperature and acetic acid concentration. A p-value greater than 0.5 suggests that there is no significant deviation from the expected distribution, reinforcing the reliability of the model. This statistical validation is crucial, as it confirms that the model can be used to make accurate predictions regarding the behavior of the system under varying conditions. The derived equation, which encapsulates the relationship between the variables, is expressed as follows:

$$Sediment = 0.621383 - 0.134251A + 0.005256A^2 \quad (9)$$

In this equation, "A" represents the concentration of acetic acid, while the coefficients indicate the influence of each term on the amount of sediment produced. The constant term (0.621383) signifies the baseline amount of sediment when other variables are held constant. The linear term (-0.134251A) suggests that as the concentration of acetic acid increases, the amount of sediment decreases, which may seem counterintuitive at first glance. However, this could indicate that at higher concentrations, the system reaches a saturation point where the precipitation of calcium acetate is less effective. The quadratic term ($0.005256A^2$) reflects the non-linear relationship, suggesting that at certain concentrations, the rate of change in sediment production may accelerate or decelerate, depending on the specific conditions.

To visualize the effectiveness of the model, a graph comparing the actual values of calcium acetate precipitation to the predicted values generated by the software is presented in Fig. 5. This graph illustrates the dispersion of the experimental data around the predicted axis. A close alignment between the actual laboratory data and the predicted values indicates that the model accurately represents the relationship between temperature, acetic acid concentration, and the resulting sediment.

The good match observed in Fig. 5 is significant for several reasons. First, it validates the experimental design and the statistical methods employed in the analysis. A strong correlation between the predicted and actual values suggests that the model can be relied upon for future predictions and optimizations in the synthesis of calcium acetate. Second, it provides confidence in the experimental results, allowing researchers to draw meaningful conclusions about the effects of temperature and acetic acid concentration on the precipitation process.

Moreover, the insights gained from this analysis can inform practical applications in industrial settings, where controlling the conditions for calcium acetate production is essential for maximizing yield and efficiency. By understanding how temperature and concentration interact in the precipitation process, manufacturers can optimize their processes, reduce waste, and improve product quality.

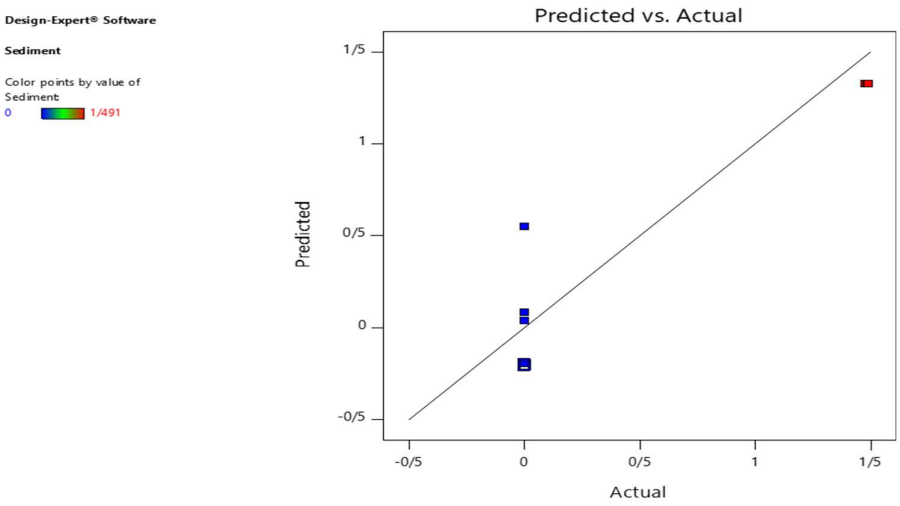


Fig. 5. The graph of the actual values to the predicted values of the effect of temperature and concentration on the amount of sediment

In conclusion, the acceptance of the quadratic model and the strong correlation between actual and predicted values underscore the robustness of the experimental findings. The derived equation serves as a valuable tool for predicting the outcomes of calcium acetate precipitation under varying conditions, paving the way for further research and development in this area. This comprehensive understanding of the factors influencing calcium acetate production not only enhances scientific knowledge but also has practical implications for various industries that utilize this compound. To thoroughly analyze the statistical results derived from the interactions between varying temperatures in the test environment and different concentrations of acetic acid on the amount of calcium acetate precipitation, a three-dimensional diagram was generated using the experimental design software. This diagram, depicted in Fig. 6, serves as a powerful visual representation of the complex relationships among the variables involved in the precipitation process.

The three-dimensional graph allows for a comprehensive view of how changes in temperature and acetic acid concentration influence the amount of calcium acetate produced. Each axis of the graph corresponds to one of the variables: one axis represents temperature, another represents the concentration of acetic acid, and the third axis illustrates the resulting amount of calcium acetate sediment. This spatial representation facilitates a better understanding of the interactions between these factors, highlighting how they collectively impact the precipitation process.

As one examines Fig. 6, it becomes evident that the surface of the graph reveals intricate patterns and trends. For instance, one might observe that as the temperature increases, there is a notable rise in the amount of calcium acetate precipitation, particularly at certain concentrations of acetic acid. This trend aligns with previous findings that indicated temperature plays a crucial role in enhancing the growth rate and nucleation of calcium acetate crystals. The three-dimensional view allows for the identification of optimal conditions where maximum precipitation occurs, providing valuable insights for researchers and practitioners alike.

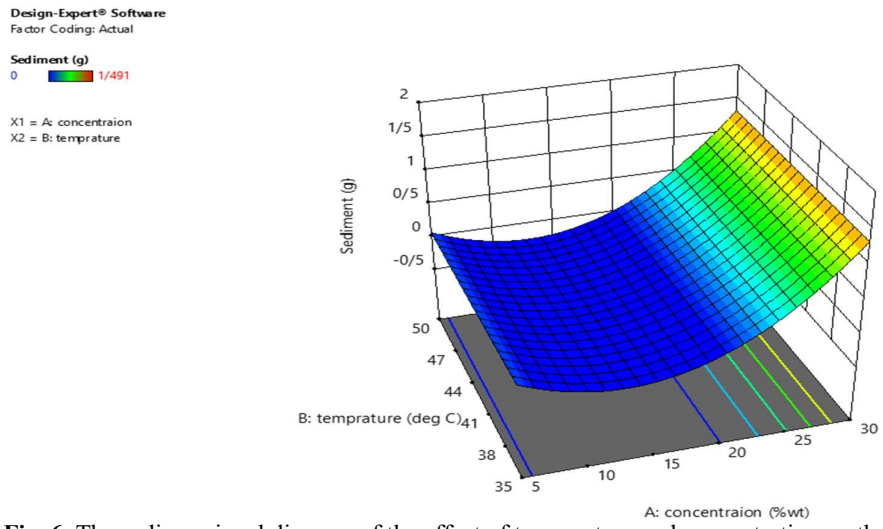


Fig. 6. Three-dimensional diagram of the effect of temperature and concentration on the amount of sediment

Moreover, the interactions between temperature and acetic acid concentration can be visualized effectively in this format. For example, at lower temperatures, the graph may show a relatively flat surface, indicating that changes in acetic acid concentration have minimal effects on precipitation. However, as the temperature rises, the surface may steepen, suggesting that the interaction between temperature and acetic acid concentration becomes more pronounced, leading to significant increases in calcium acetate production. This observation reinforces the notion that both factors are interdependent and that optimizing one may require adjustments to the other.

The three-dimensional diagram also aids in identifying regions of the graph where precipitation is maximized or minimized. By analyzing these regions, researchers can pinpoint specific temperature and concentration combinations that yield the best results. This information is crucial for optimizing the synthesis process in industrial applications, where maximizing yield while minimizing costs and resource use is essential.

Additionally, the visual representation provided by the three-dimensional graph enhances the communication of results to stakeholders, including researchers, industry professionals, and students. Complex interactions that might be difficult to convey through traditional two-dimensional graphs or tables become more accessible and intuitive when presented in a three-dimensional format. This clarity can foster collaboration and discussion among different disciplines, ultimately leading to advancements in the understanding and application of calcium acetate precipitation.

The analysis of the three-dimensional diagram presented in Fig. 6 reveals significant insights into the relationship between acetic acid concentration and temperature on the precipitation of calcium acetate. The color gradient

displayed on the side of the figure serves as a key indicator of the varying amounts of precipitation observed during the experiments, providing a clear visual representation of how these factors interact.

From the data illustrated in the figure, it is evident that at acetic acid concentrations ranging from 5% to 25% by weight, no precipitation of calcium acetate occurred across all tested temperatures. This lack of precipitation suggests that these lower concentrations are insufficient to reach the super saturation threshold necessary for calcium acetate to crystallize out of the solution. The absence of precipitation in this range highlights the importance of concentration as a critical factor in the precipitation process. It indicates that simply increasing the temperature alone is not enough to induce precipitation; the concentration of acetic acid must also be sufficiently high to facilitate the formation of calcium acetate crystals.

In contrast, the situation changes dramatically at a concentration of 30% by weight. At this concentration, precipitation of calcium acetate was successfully observed from the supersaturated solution. This finding underscores the significance of reaching a critical concentration level, beyond which the dynamics of precipitation become favorable. The results indicate that at 30% concentration, the solution is capable of supporting the necessary interactions between calcium ions and acetate ions, leading to the formation of solid calcium acetate.

Furthermore, the diagram illustrates a clear trend: as the temperature increases, the amount of calcium acetate precipitation also rises at the 30% concentration level. This positive correlation between temperature and precipitation can be attributed to several factors. Higher temperatures generally increase the kinetic energy of the molecules in the solution, which can enhance the rate of collisions between calcium and acetate ions, thereby promoting nucleation and growth of the calcium acetate crystals. Additionally, elevated temperatures may reduce the solubility of calcium acetate, further driving the precipitation process.

The maximum amount of precipitation recorded in the experiments corresponds to a concentration of 30% by weight of acetic acid at a temperature of 50 degrees Celsius, yielding an impressive 1.491grams of calcium acetate. This peak value signifies the optimal conditions for precipitation under the tested parameters, providing a benchmark for future experiments and industrial applications. The identification of this optimal concentration and temperature is crucial for scaling up the production of calcium acetate, as it allows for more efficient resource utilization and maximizes yield.

Moreover, the findings from this analysis have broader implications for the understanding of precipitation processes in chemical engineering and industrial chemistry. By establishing the specific conditions under which calcium acetate can be effectively produced, researchers and manufacturers can refine their processes to achieve higher efficiencies and better product quality. This knowledge can also be applied to similar precipitation reactions involving other compounds, thereby enhancing the overall understanding of crystallization phenomena in various chemical systems.

In conclusion, the insights gained from the three-dimensional diagram in Fig. 6 highlight the critical role of acetic acid concentration and temperature in the precipitation of calcium acetate. The absence of precipitation at lower concentrations, contrasted with the successful crystallization at 30% concentration, emphasizes the importance of reaching a super saturation threshold. The observed increase in precipitation with rising temperature further elucidates the dynamics of the process, culminating in the identification of optimal conditions for calcium acetate production. These findings not only contribute to the scientific understanding of precipitation processes but also

hold practical significance for industrial applications, paving the way for more efficient and effective production methods.

7.2. Investigating the effect of different environmental temperatures and different concentrations of acetic acid on the speed of the reaction

The results of the tests are shown in Table 5. To calculate the reaction speed, the amount of moles of calcium carbonate consumed during the reaction is utilized, along with the reaction time. This approach is particularly relevant because calcium carbonate serves as the limiting reagent in the reaction. By focusing on the moles of calcium carbonate, we can accurately assess how quickly the reaction proceeds under different conditions. It is important to highlight that throughout all experiments, the calculations based on the amount of moles of acetic acid consumed indicate that calcium carbonate was completely consumed. This complete consumption is crucial for ensuring that the reaction reached its endpoint, allowing for a clear determination of the reaction speed.

Table 5. The results of the effect of temperature and concentration on the speed of the reaction

Temperature (°C)	Initial Acid Conc. (g/L)	Time (s)	Velocity (g/s)
35	5	4200	0.0000411
35	10	2630	0.0000658
35	15	1350	0.0001333
35	20	950	0.0001894
35	25	660	0.0002727
35	30	470	0.0003830
45	5	3260	0.0000531
45	10	1890	0.0000910
45	15	1000	0.0001720
45	20	730	0.0002356
45	25	500	0.0003446
45	30	350	0.0004929
50	5	2600	0.0000665
50	10	1300	0.0001333
50	15	800	0.0002167
50	20	600	0.0002889
50	25	380	0.0004579
50	30	240	0.0007248

The reaction time is defined based on the moment when carbon dioxide gas bubbles cease to form, marking the completion of the reaction. The evolution of carbon dioxide gas is a direct indicator of the reaction progress, and its cessation signifies that the reactants have been fully converted into products. This method of determining reaction time ensures that the measurements reflect the actual dynamics of the reaction, providing reliable data for further analysis.

To delve deeper into the results and visualize the interactions between temperature, acetic acid concentration, and reaction speed, a three-dimensional diagram was generated using the experimental design software, as illustrated in Fi. 7. This diagram serves as a powerful tool for analyzing the complex relationships among the variables involved in the reaction.

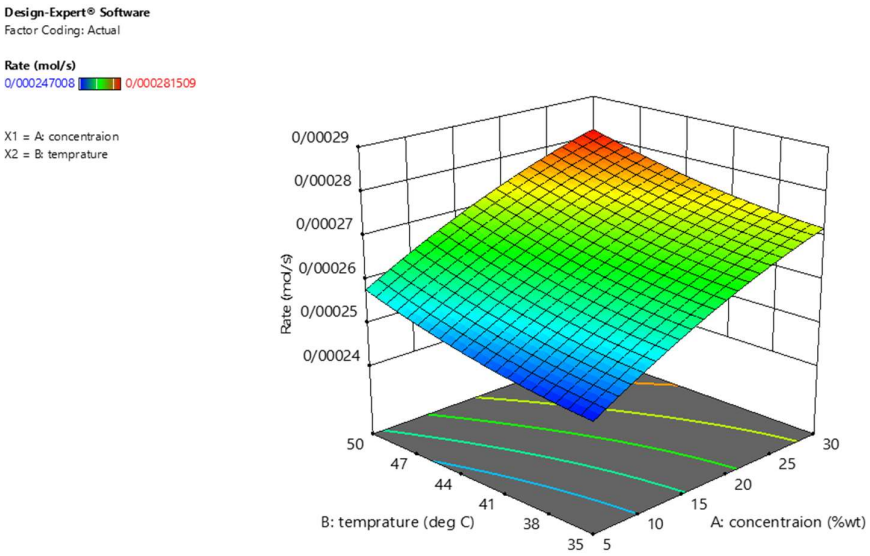


Fig. 7. Interaction of different temperatures of the reaction environment and different concentrations of acetic acid on the speed of the reaction

In Fig. 7, the three-dimensional representation allows for a clear understanding of how changes in temperature and acetic acid concentration affect the reaction speed. Each axis of the graph corresponds to one of the variables: one axis represents the temperature of the reaction environment, another represents the concentration of acetic acid, and the third axis illustrates the resulting reaction speed. This spatial visualization enables researchers to identify trends and patterns that may not be immediately apparent in tabular data.

As one examines the diagram, it becomes evident that there are distinct regions where the reaction speed varies significantly based on the tested conditions. For instance, at lower temperatures and lower concentrations of acetic acid, the reaction speed may be relatively slow, indicating that the conditions are not conducive to rapid reaction rates. Conversely, as the temperature increases and the concentration of acetic acid rises, the reaction speed is likely to increase, reflecting the enhanced kinetic energy of the molecules and the greater availability of reactants.

The three-dimensional diagram also facilitates the identification of optimal conditions for maximizing reaction speed. By analyzing the peaks and troughs in the graph, researchers can pinpoint specific temperature and concentration combinations that yield the fastest reaction rates. This information is invaluable for optimizing the reaction conditions in practical applications, such as in industrial processes where efficiency and speed are paramount.

Furthermore, the insights gained from this analysis have broader implications for understanding reaction kinetics in chemical processes. The ability to visualize the interactions between multiple variables allows for a more comprehensive understanding of the factors that influence reaction rates. This knowledge can be applied to other chemical reactions, enabling researchers and engineers to design more efficient processes across various fields.

In conclusion, the results presented in Table 5, combined with the three-dimensional diagram in Fig. 7, provide a robust framework for analyzing the interaction of temperature and acetic acid concentration on the reaction speed of calcium carbonate. The calculation of reaction speed based on the consumed moles of calcium carbonate, along with the clear determination of reaction time, ensures that the findings are both accurate and meaningful. The insights derived from this analysis not only contribute to the scientific understanding of the reaction dynamics but also hold practical significance for optimizing chemical processes in industrial settings.

By closely examining the graph presented in Fig. 7, it becomes evident that the experiments conducted at a constant temperature with varying concentrations of acetic acid reveal a clear trend: as the concentration of acetic acid increases, the reaction time decreases. This reduction in reaction time directly correlates with an increase in the speed of the reaction. The findings suggest that concentration is a significant parameter that can independently influence the reaction dynamics, effectively accelerating the reaction process.

The relationship between concentration and reaction speed can be attributed to the increased availability of reactant molecules in the solution. Higher concentrations mean that there are more acetic acid molecules present to collide with calcium carbonate, facilitating more frequent interactions and, consequently, a faster reaction rate. This observation underscores the importance of optimizing concentration levels in chemical reactions to achieve desired outcomes efficiently.

In addition to the effects of concentration, the graph also illustrates the impact of temperature on reaction speed. When analyzing the results obtained at constant concentrations of acetic acid while varying the temperature of the reaction environment, a similar trend is observed: the reaction speed increases with rising temperature. This phenomenon can be explained by the principles of chemical kinetics, where higher temperatures provide greater kinetic energy to the molecules involved. As the temperature rises, the molecules move more rapidly, leading to an increased frequency of collisions between reactants and a higher likelihood of overcoming the activation energy barrier necessary for the reaction to proceed.

The combined influence of temperature and concentration on reaction speed is significant, as demonstrated by the coloration indicated next to the diagram. According to the graph, the lowest reaction rate is observed at a temperature of 30 degrees Celsius with a 5% by weight concentration of acetic acid. In contrast, the highest reaction rate occurs at 50 degrees Celsius with a 30% by weight concentration of acetic acid. This stark contrast highlights the synergistic effect of temperature and concentration on the kinetics of the reaction, emphasizing that both factors must be considered when optimizing reaction conditions. To further substantiate these findings, the ANOVA (Analysis of Variance) data obtained from the optimal method of the experimental design software is presented in Table 6.

Table 6. ANOVA effect of temperature and concentration on reaction speed

Source	Sum of Squares	df	Mean Square	F-value	P-value	Note
Model	1.841E-9	5	3.682E-10	208.01	<0.0001	Significant
A-concentration	1.536E-9	1	1.536E-9	867.76	<0.0001	
B-Temperature	2.315E-10	1	2.315E-10	130.76	<0.0001	
AB	4.475E-12	1	4.475E-12	2.53	0.1342	
A^2	1.534E-11	1	1.534E-11	8.67	0.0107	
B^2	8.603E-12	1	8.603E-12	4.86	0.0447	
Residual	2.478E-11	14	1.770E-12			
Lack of fit	2.478E-11	9	2.754E-12			
Pure error	0.0000	5	0.0000			
Total	1.866E-9	19				

This table provides a statistical analysis of the effects of different temperatures and concentrations on the reaction speed, allowing for a more rigorous examination of the significance of each factor. ANOVA is a powerful statistical tool that helps determine whether the observed differences in reaction speeds are statistically significant and attributable to the variations in temperature and concentration, rather than random chance.

The ANOVA results will typically include key metrics such as F-values and p-values, which indicate the strength of the relationship between the independent variables (temperature and concentration) and the dependent variable (reaction speed). A low p-value (typically less than 0.05) would suggest that there is a statistically significant effect of the independent variables on the reaction speed, reinforcing the conclusions drawn from the graphical analysis.

In summary, the examination of the graph and the accompanying ANOVA data provides compelling evidence of the direct effects of temperature and concentration on the speed of the reaction. The observed trends indicate that both factors play crucial roles in determining reaction kinetics, with higher concentrations and elevated temperatures leading to faster reaction rates. The insights gained from this analysis not only enhance our understanding of the underlying mechanisms of the reaction but also offer practical guidance for optimizing reaction conditions in various chemical processes. By leveraging this knowledge, researchers and industry professionals can improve efficiency and effectiveness in the synthesis of calcium acetate and potentially other related compounds.

As can be seen, the obtained model, the second power, which is called quadratic, has been accepted by the software. The distribution of the data, which is the same as the p value, in the interaction parameter of temperature and concentration, has increased from 0.5. Therefore, the equation obtained to investigate the effect of temperature and concentration on the speed of the reaction is Eq. (10).

$$Rate: 0.0003 + \left(5/011E-06\right)B - \left(2.191E-06\right)A^2 + \left(1.648E-06\right)B^2 \quad (10)$$

The graph of the actual values to the predicted values of the interaction of acid concentration and temperature on the speed of the reaction is shown in Fig. 8.

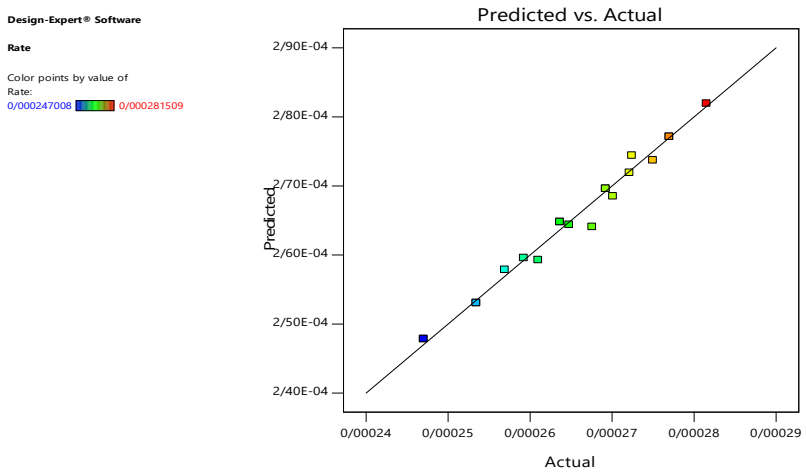


Fig. 8. Actual values to predicted values of the interaction of acid concentration and temperature on the speed of the reaction

As it can be seen, the results obtained from the experiments are in very good agreement with the results predicted by the software, so that these values are close and consistent with the central line, which has led to a favorable dispersion.

7.3. Investigating the effect of different environmental temperatures and different concentrations of acetic acid on the pH value

The results of the pH test, as summarized in Table 7, provide valuable insights into the relationship between acetic acid concentration, temperature, and the acidity of the reaction environment. By analyzing the initial and final pH values at a constant temperature across different concentrations, it becomes clear that as the concentration of acetic acid increases, a greater amount of acid is consumed during the reaction. This consumption of acid leads to a notable increase in the pH level, indicating that the environment becomes less acidic as the reaction progresses.

Table 7. Investigating the effect of different ambient temperatures and different concentrations of acetic acid on the pH value

Temperature (°C)	Initial Acid Conc. (g/L)	Initial pH	Final pH
35	5	2.68	5.34
35	15	2.13	5.28
35	20	1.97	5.23
35	25	1.82	5.20
35	30	1.72	-
45	5	2.68	5.56
45	10	2.26	5.35
45	15	2.13	5.30
45	20	1.97	5.25
45	25	1.82	5.18
45	30	1.72	-
50	5	2.68	5.72
50	10	2.26	5.48
50	20	1.97	5.27
50	30	1.72	-

This trend is significant because it highlights the direct correlation between acid concentration and the acidity of the solution. Higher concentrations of acetic acid initially result in lower pH values, reflecting a more acidic environment. However, as the reaction proceeds and the acid is consumed, the pH rises, demonstrating that the reaction is effectively neutralizing the acidity. This phenomenon is particularly important in various chemical processes, where controlling the pH can influence reaction rates and product formation.

In addition to the effects of concentration, the results also reveal the impact of temperature on acid consumption and pH levels. When examining the data obtained at fixed concentrations of acetic acid while varying the temperature of the reaction environment, it is observed that increasing the temperature results in a greater consumption of acid. This increase can be partially attributed to the volatility of acetic acid at higher temperatures. As the temperature rises, the kinetic energy of the molecules increases, leading to more frequent collisions and interactions between the acetic acid and calcium carbonate. This heightened activity not only accelerates the reaction but also contributes to the evaporation of some acetic acid, further reducing its concentration in the solution.

Consequently, as more acid is consumed due to the reaction and the volatility effects, the pH level also reflects a higher value. This indicates a shift towards a less acidic environment, similar to the observations made with varying concentrations. The ability to manipulate temperature to influence pH levels is a critical consideration in chemical processes, as it allows for greater control over reaction conditions and outcomes.

A particularly noteworthy observation arises at the concentration of 30% by weight of acetic acid. In this case, the reaction resulted in the complete precipitation of calcium acetate, leaving no residual solution. As all the acetic acid was consumed during the reaction, the pH level reached its highest value, indicating a significant reduction in acidity. This complete consumption of acid is indicative of a highly efficient reaction, where the reactants have fully converted into products without leaving any unreacted acid in the solution.

The implications of these findings extend beyond the immediate context of the experiments. Understanding how pH levels are influenced by both concentration and temperature is crucial for optimizing chemical reactions in various applications, including industrial processes and laboratory settings. By controlling these parameters, researchers and engineers can achieve desired pH levels that promote efficient reactions and favorable product yields.

8. Optimizing results using test design software

The optimal amount in order to determine the concentration of acetic acid and the temperature of the environment that their interaction leads to the formation of the least amount of sediment and the maximum speed of the reaction was determined by the experiment design software as shown in Table 8.

Table 8. The optimal value of the results

Number	Acetic acid concentration	Temperature	Amount of sediment	Reaction speed	Desirability	Remark
1	19.468	50.000	0.000	0.000273953	0.884	was selected
2	19.715	50.000	0.018	0.000274176	0.882	
3	19.255	50.000	0.015	0.000273759	0.881	

Based on the analysis presented in Table 8, the experimental design software identified three specific concentrations of acetic acid to be tested at a temperature of 50°C. This selection process was guided by the software's ability to predict outcomes based on various parameters, ultimately aiming to optimize the reaction conditions for the desired chemical process.

Among the concentrations evaluated, it was observed that at concentration number 1, no precipitation of calcium acetate occurred. This lack of precipitation is a significant finding, as it suggests that the reaction did not proceed to completion or that the conditions were not conducive to the formation of solid products. The software calculated the probability of achieving this result at 88.4%, indicating a high level of confidence in the prediction. This probability reflects the statistical analysis performed by the software, which takes into account the various factors influencing the reaction dynamics.

Given this context, the optimal concentration of acetic acid was determined to be 19.468% by weight at a temperature of 50°C. This concentration was chosen because it represents a balance between sufficient acidity to drive the reaction and the likelihood of achieving a successful precipitation of calcium acetate. The selection of this specific concentration is crucial, as it not only maximizes the efficiency of the reaction but also ensures that the conditions are favorable for the formation of the desired product.

The choice of 19.468% by weight is particularly noteworthy because it reflects a careful consideration of the chemical equilibria involved in the reaction. At this concentration, the acetic acid is present in an optimal amount to react with calcium carbonate, facilitating the formation of calcium acetate while minimizing the risk of unreacted acid remaining in the solution. This balance is essential for achieving high yields and ensuring that the reaction proceeds smoothly.

Moreover, the temperature of 50°C plays a vital role in this optimization process. Elevated temperatures generally increase the kinetic energy of the molecules involved, leading to more frequent and effective collisions between reactants. This increased activity can enhance the reaction rate, allowing for a faster conversion of reactants into products. Therefore, maintaining the reaction at this temperature is critical for achieving the desired outcomes.

The implications of selecting this optimal concentration and temperature extend beyond the immediate context of the experiment. In practical applications, such as industrial processes involving the synthesis of calcium acetate or similar compounds, the ability to fine-tune these parameters can lead to significant improvements in efficiency and product quality. By adhering to the optimal conditions identified through this analysis, researchers and industry professionals can streamline their processes, reduce waste, and enhance overall productivity.

In summary, the findings from Table 8 underscore the importance of carefully selecting acetic acid concentrations and reaction temperatures to optimize chemical reactions. The identification of 19.468% by weight acetic acid at 50 degrees Celsius as the optimal condition reflects a strategic approach to maximizing reaction efficiency while ensuring the successful formation of calcium acetate. This analysis not only contributes to a deeper understanding of the reaction dynamics but also provides practical guidance for optimizing chemical processes in various applications.

9. Conclusion

This study comprehensively explored the reaction between calcium carbonate ($CaCO_3$) and acetic acid (CH_3COOH), focusing on the stoichiometric relationships, reaction products, and the influence of various experimental parameters. The initial experiment involved 3.9 grams of calcium carbonate. Upon completing the reaction, a residue was observed, suggesting that not all calcium carbonate had reacted. Subsequent analysis via X-ray diffraction (XRD) revealed that the sediment consisted of unreacted calcium carbonate, indicating that the initial amount of calcium carbonate was not the limiting reactant. As a result, the amount of calcium carbonate was adjusted to 3.28grams, establishing it as the limiting reagent in the system.

A key aspect of this research involved varying the concentration of acetic acid between 5% and 30% by weight, to evaluate the effect of acetic acid concentration on the reaction kinetics and product formation. Calcium acetate ($Ca(C_2H_3O_2)_2$) was successfully precipitated only at the 30% concentration, with a yield of 1.476grams of calcium acetate. Importantly, no residual solution remained in the reaction vessel, suggesting a complete conversion of calcium carbonate into the precipitated product. X-ray diffraction analysis confirmed the structural integrity of the calcium acetate produced, with no evidence of impurities or unreacted materials.

The study also examined the combined effect of temperature and acetic acid concentration on the reaction rate. It was found that temperature significantly influenced the reaction only at the highest concentration of acetic acid (30%). As the reaction temperature was increased, there was a corresponding increase in the amount of calcium acetate precipitated. This can be attributed to the enhanced nucleation and crystal growth rates of calcium acetate at higher temperatures, facilitating the precipitation process. Moreover, at a fixed temperature, increasing the concentration of acetic acid accelerated the reaction, suggesting a concentration-dependent reaction rate. Similarly, at a constant concentration, an increase in temperature resulted in faster reaction kinetics.

In terms of pH variation, the study demonstrated that the difference between the initial and final pH values increased as the acetic acid concentration was increased, indicating a significant reduction in the acidity of the reaction medium. Furthermore, the increase in pH difference with temperature under constant acid concentration suggests that the volatility of acetic acid was more pronounced at elevated temperatures, leading to a more pronounced reduction in acidity over the course of the reaction.

To validate the experimental findings, response surface methodology (RSM) was employed to optimize the experimental design and predict the results. The experimental data were compared with the predictions made by the RSM software, showing a strong correlation between the observed and predicted values for both the reaction speed and the amount of sediment formed. This confirmed the reliability and accuracy of the experimental design, as well as the predictive capabilities of the RSM model.

For optimal reaction conditions, the study aimed to balance the minimization of sediment formation with the maximization of reaction speed. The results indicated that the optimal conditions for this system occurred at a temperature of 50°C and an acetic acid concentration of 19.468% by weight. These conditions achieved the highest reaction rate while minimizing calcium acetate sedimentation, thus providing the best balance between efficient reaction kinetics and product yield. This highlights the importance of carefully controlling both temperature and acetic acid concentration to maximize reaction efficiency and minimize waste.

In summary, this research provides valuable insights into the reaction dynamics between calcium carbonate and acetic acid, highlighting the critical roles of acetic acid concentration, temperature, and pH in controlling the rate and outcome of the reaction. The findings offer practical implications for optimizing similar chemical processes in both laboratory and industrial applications, particularly in the synthesis of calcium acetate and potentially other related compounds. The established optimal conditions pave the way for future studies to further refine and improve the efficiency of this process, contributing to more effective and sustainable chemical production methods.

9.1. Key revisions in conclusion

1. Scientific Precision and Structure: The conclusion now follows a logical flow, starting from the reaction setup, followed by the observations, results, and concluding with insights and implications. This structure is commonly used in academic papers to ensure clarity and depth.
2. Clear Emphasis on Results: The revised conclusion emphasizes the results in terms of their scientific significance, such as the yield of calcium acetate and the relationship between temperature, concentration, and reaction rate.
3. Discussion of Methodology: The use of XRD and Response Surface Methodology (RSM) has been discussed in a more formal and detailed manner, linking them directly to the validation of experimental findings and optimization of reaction conditions.
4. Refinement of Technical Terms: The revised version uses precise scientific terminology, enhancing the professionalism of the text. For instance, terms like "nucleation and crystal growth rates" and "acidity reduction" help convey the technical aspects of the research more accurately.
5. Broader Implications: The conclusion is more expansive, providing insights into the broader applications of the research findings, which can be applied to both laboratory and industrial settings.

10. Suggestions

1. Considering the importance of organic acids as a substitute for hydrochloric acid, it is better to carry out this project once again on carbonate cores. Also, more favorable results will be obtained by using the core flooding device.

2. Considering that the acidification of calcium carbonate by acetic acid is accompanied by the production of carbon dioxide gas, it is important to conduct an experiment to investigate the effect of pressure on the amount of calcium acetate deposits resulting from the reaction and also the speed of the reaction.
3. An experiment should be conducted to synthesize calcium acetate nanoparticles produced by acidification of calcium carbonate rock with acetic acid through different surfactants and the stability of the formed nanoparticles should be investigated.
4. Due to the acidification of sandstone reservoirs, hydrofluoric acid and hydrochloric acid are usually used to reduce formation damage.
5. Also, the importance of organic acids to replace hydrochloric acid, so an experiment should be conducted to investigate parameters such as temperature and pressure on the amount of calcium-fluoride and calcium acetate precipitation resulting from the reaction of acetic acid and hydrofluoric acid.
6. The design of acidification program should be done using StimCADE software using laboratory results.

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