

Article

A Study of the Kinetics, Structure, and Morphology of the Effect of Organic Additives on Barium Sulfate Precipitation Reactions in Propan-1-ol–Water and Ethanol–Water Mixture Solutions

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Abstract: Barium sulfate precipitation in production industrial facilities and in petroleum reservoir systems is a dangerous operational problem. The solvent acts as a second component in the system during the onset of crystallization from solution, significantly impacting the kinetics. Few studies have investigated the influence of organic solvents on barium sulfate precipitation. In this study, we examine the precipitation reaction of barium sulfate in mixed propan-1-ol–water and ethanol–water solvents at 303.15 K. The temporary conductivity variation is determined to follow the kinetics of the precipitation reaction and the germination time of barium sulfate crystals. A large variation is found in the precipitation reaction rate and induction time due to differences in physical parameters such as polarity and dielectric constants between water and organic solvents. This difference in physical properties leads to a stronger variation in intermolecular interaction between the solute and the solvent, especially in hydrogen bonds. The solid phase obtained at the end of the precipitate reaction is characterized through FTIR, XRD, and SEM techniques, demonstrating an important difference in the structure, morphology, and agglomeration phenomena of the precipitate obtained in water–ethanol and water–propan-1-ol mixtures compared to pure water solutions.

Keywords: barium sulfate; precipitation; organic solvent; kinetic; morphology



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1. Introduction

The phenomenon of precipitation is a fundamental process of chemistry, often encountered in industry, especially when working in aqueous environments where water is one of the main constituents of the system. All thermodynamic and kinetic parameters of the precipitation process are related to the solubility of the solute in the solvent. The properties of the solute and solvent influence the interaction between salts and solvents. Solvents are identified by the Debye–Hückel technique based on their physical characteristics, including density, dielectric constant, viscosity, relative permittivity, and miscibility [1]. Since solvent mixtures are used in many industrial processes and laboratory operations, the solutes' solubility in mixed solvents is crucial from a practical standpoint. In the petroleum industry, the precipitation of barium sulfate in pipelines during the production phase is a

notable example of scale formation in a mixed solvent. Indeed, studies have shown that the aqueous phase in oil slicks is rich in organic material due to the solubility of gases in water; contact between crude oil and formation water occurs [2,3] during water extraction in petroleum production.

Extensive studies have involved barium sulfate precipitation and co-precipitation phenomena in aqueous solutions [4–14]. The study of organic solvent effects on barium sulfate precipitation has received minimal experimental attention. In 2008, Jons et al.'s studies show that in the methanol–water mixture solution, the solubility of barium sulfate decreases, the crystal growth and nucleation times are faster, and a large difference in the morphology occurs when the amount of methanol increases [15]. In 2016, Esam A. G.'s study confirmed that the solubility of barium sulfate decreases in an ethanol–water mixture compared to a pure water solution [16]. Contrary to previous studies, in the case of calcium sulfate, Gomis et al. demonstrated that the solubility of calcium sulfate increases in the presence of ethanol [17].

It is well understood that in barium sulfate, previous studies have confirmed the effect of ethanol and methanol on the barium sulfate precipitation process.

However, studying the effects of adding these organic solvents requires more attention and greater synchronization between the physical properties of the solvents and the precipitation process. To achieve this goal, we propose studying the effect of two solvents with different physical properties: ethanol as a total miscible solvent and propan-1-ol as a partially miscible solvent with water (Table 1). In this paper, we present a kinetic, structural, and morphological study divided into two parts. In the first part—the solution study—we conduct a conductivity analysis to identify the effect of organic solvents on the kinetics of nucleation and crystal germination during the precipitation reaction. In the second part—the solid study—we present the characterization of the solid phase obtained using FTIR, XRD, and SEM techniques in order to determine the effect of the presence of organic solvents on the structure and morphology of the barite crystals.

Table 1. Physical properties of different solvents.

Solvent	Dipole Moment (D)	Dielectric Constant	Miscibility with Water
Water	1.85	78.5	---
Ethanol	1.69	24	Total
Propan-1-ol	1.68	20	Partial

2. Experimental Section

2.1. Experimental Unit

Figure 1: 1: Conductivity cell using JENWAY 4520 (Bibby Scientific Ltd., Stone, Staffs, UK); 2: electrode conductivity; 3: magnetic stirrer; 4: heating head; 5: reactor of 500 mL; and 6: thermostatic bath.

2.2. Chemical Reagents

Analytical-grade BaCl_2 and Na_2SO_4 , ethanol, and propan-1-ol were supplied by SIGMA-ALDRICH (Germany), FLUKA (Honeywell International Inc.), and Lab-Scan (Ireland).

2.3. Experimental Procedure

To monitor the precipitation of barium sulfate, 100 mL of Na_2SO_4 solution and a 100 mL of BaCl_2 with a volume V (10 mL or 50 mL) of the organic solvent were introduced into a cylindrical reactor designed to resemble pipeline geometry under magnetic steering

(500 tr/min). We use a very high stirring speed to quickly obtain a homogeneous mixture between the organic and aqueous phases. Subsequently, the temperature for the reaction is set at the working temperature (at $T = 303.15$ K) using a bath equipped with a thermostat, since the different parameters of the precipitation reaction can change if the temperature is modified. We chose to work at temperatures near 303 K and at atmospheric pressure to ensure the environment closely resembled the precipitation conditions found at the surface of oil wells, in distribution pipes, or within the pipes of desalination units. In addition, we compared our results to the majority of previous work related to the precipitation of barite in the mixture phase [15–17]. At the end of each precipitation reaction, the solution was filtered under a Buchner system to recover the solid phase. The precept obtained was dried in an oven at a temperature equal to 105 degrees for 24 h to eliminate all solvent molecules, until the weight of the powder no longer decreases. This result is supported by the absence of any signal related to the solvent molecule in the infrared spectrum. At the start time, 100 mL of a BaCl_2 solution, prepared at the working temperature, was added to 100 mL of a Na_2SO_4 solution to initiate the precipitation reaction. The conductivity of the solution was measured at different times to monitor the precipitation reaction at $T = 303.15$ K and $\text{pH} = 6.7$ (in the presence of 50 mL ethanol, $\text{pH} = 6.6$, and in the presence of 50 mL propanol, $\text{pH} = 6.4$). SO_4^{2-} is an inactive base; the literature shows that within the range of 3–9, barite nucleation and growth are not affected by pH variation due to the addition of an organic solvent [18]. The pH must not exceed 7 to prevent the precipitation of BaCO_3 or $\text{Ba}(\text{OH})_2$. The composition of all working solution was illustrated in Table 2.

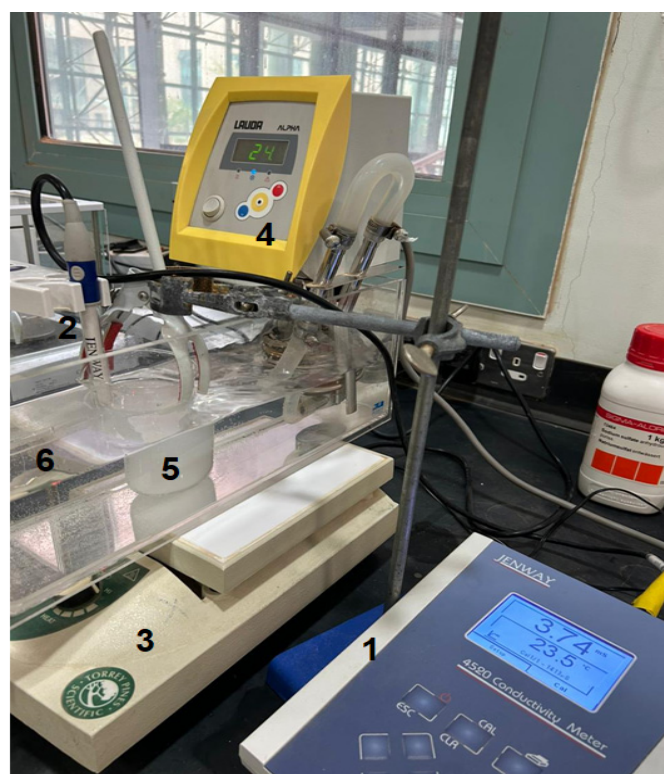
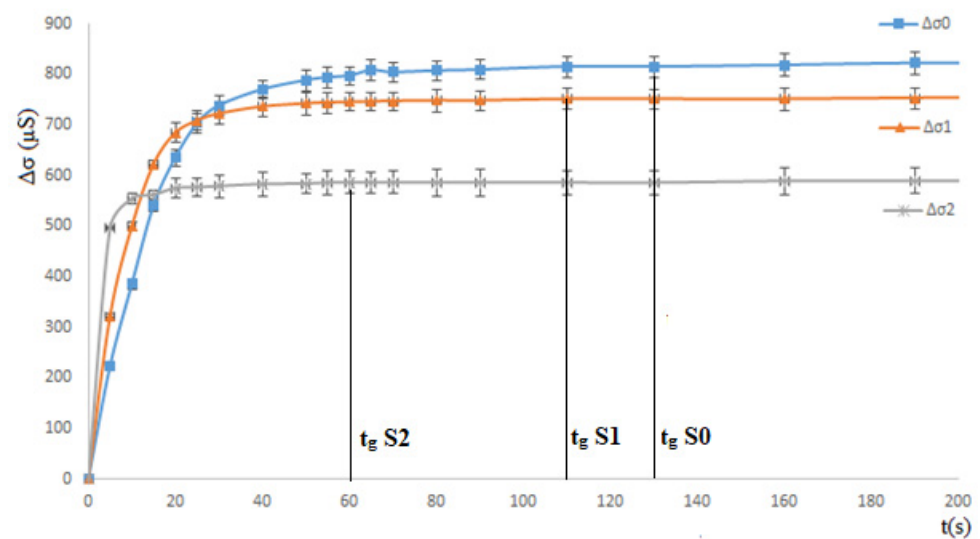
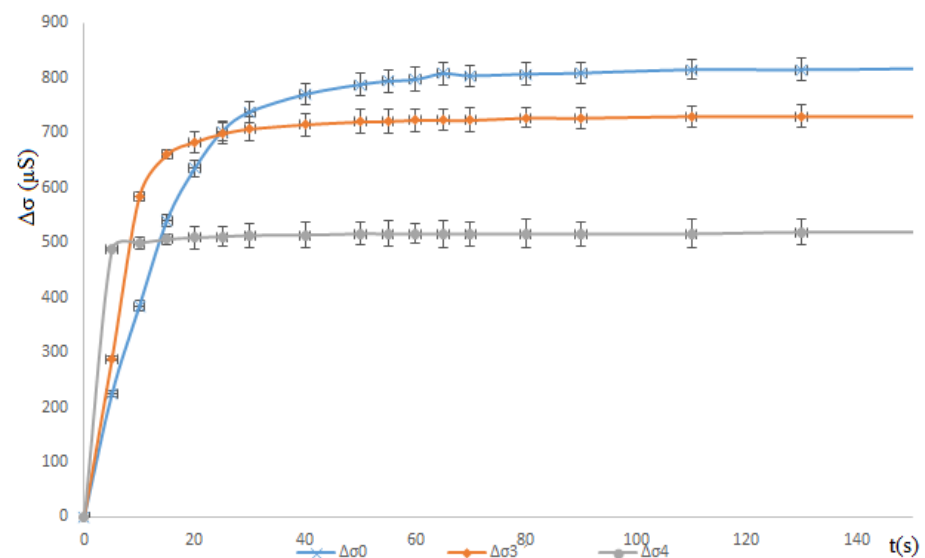


Figure 1. Experimental unit.

Table 2. Composition of working solutions.

Composition Solution	V of BaCl ₂ (mL) (5×10^{-3} M)	V of Na ₂ SO ₄ (mL) (5×10^{-3} M)	V (mL) of Add Organic Solvent	Molarity of Added Organic Solvent (M)	Mass of Precipitate
Solution S0	100	100	0	0	0.2131
Solution S1	100	100	10 mL of Ethanol	0.68	--
Solution S2	100	100	50 mL of Ethanol	3.43	0.1940
Solution S3	100	100	10 mL of Propan-1-ol	0.53	--
Solution S4	100	100	50 mL of Propan-1-ol	2.66	0.1882

All experiments were performed in triplicate; all conductivity data are expressed as the mean $\pm$ standard deviation in Figures 2 and 3.

**Figure 2.** Temporal delta conductivity variation ($\Delta\sigma = \sigma_0 - \sigma_t$) in barium sulfate precipitation at $T = 30^\circ\text{C}$ for S0, S1 (10 mL ethanol), and S2 (50 mL ethanol).**Figure 3.** Temporal delta conductivity change ($\Delta\sigma = \sigma_0 - \sigma_t$) in barium sulfate precipitation at $T = 30^\circ\text{C}$ for S0, S3 (10 mL propan-1-ol), and S4 (50 mL propane).

2.4. Theoretical Background

The value of barium sulfate supersaturation can be expressed by the following equation:

$$\Omega_{BaSO_4} = \frac{\gamma_{Ba^{+2}} [Ba^{+2}] \gamma_{SO_4^{-2}} [SO_4^{-2}]}{k_{sp, BaSO_4}} \quad (1)$$

The $k_{sp, BaSO_4} = 1.14 \times 10^{-10}$ at $T = 303$ K in pure water [19].

The specific activity coefficient can be calculated according to the extended Debye–Hückel equation. The Davies equation is an empirical extension of the Debye–Hückel theory, which can be used to calculate activity coefficients of electrolyte solutions at concentrations higher than 0.001 M [20].

$$\log \gamma_i = AZ_i^2 \left(\frac{\sqrt{I}}{1 + \sqrt{I}} - 0.31 \right) \quad (2)$$

$$I = \frac{1}{2} \sum_{ions} m_i Z_i^2 \quad (3)$$

$A = 1.82 \times 10^6 (\epsilon T)^{-3/2} = 0.4961 \text{ mol}^{-1/2} \text{ kg}^{1/2}$ at $T = 303$ K.

$\gamma = 0.9991$ (In dilute solutions, the activity coefficient is almost equal to 1);

$\Omega_{BaSO_4} = 13,682$ in our synthetic solutions.

2.5. Analysis Techniques

At ambient temperature, the solid phase was analyzed using XRD, FTIR, and SEM techniques. XRD was performed using Cu K α radiation ($\lambda = 0.15418$ nm) and a Philips X'PERTPRO diffractometer in step scanning mode. The scanning range for the XRD patterns was $2\theta = 5\text{--}90^\circ$. A fixed counting period of 4 s and a tiny angular step of $2\theta = 0.017^\circ$ were employed. Software “Fullproof” [21] and HighScore Plus (version 3.0) were used to calculate the XRD reflection positions and for the solid phase matching. Infrared spectra of the samples in KBr pellets were obtained through diffuse reflectance, accumulating 40 scans on an Affinity-1C Shimadzu spectrophotometer, within the range of $400\text{--}4000 \text{ cm}^{-1}$ at a resolution of 4 cm^{-1} . Scanning electron microscopy (SEM) images were obtained using a Philips FEI Quanta 250 model SEM device at an accelerating voltage of 30 KV.

3. Results and Discussions

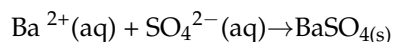
This study is devoted to the analysis of barium sulfate precipitation reactions in the presence of certain organic fluids (ethanol and propan-1-ol) (Table 2). This investigation is based on conductivity measurements in the liquid phase, in addition to a morphological and spectroscopic exploration of the solid phase obtained at the end of these reactions.

3.1. Solution Study

Given that solution composition differs for each experiment, in order to compare results, it is more effective to present the $\Delta\sigma$ conductivity variation instead of the solution conductivity curves. Delta conductivity ($\Delta\sigma$) is the difference between the initial conductivity and the conductivity at time t : $\Delta\sigma = \sigma_0 - \sigma_t$.

Therefore, we investigated the effects of two organic solvents with differing miscibility in aqueous solution (ethanol and propan-1-ol) on the reaction involving barium sulfate precipitation. We showed their effects one by one. Indeed, the precipitation rate of barium sulfate was investigated using the conductimetric technique, where the supersaturation is

larger than 1. The rate of linear growth of barium sulfate crystals is limited by a process occurring at the crystal–solution interface through the following reaction:



3.1.1. Ethanol Effect: Total Miscibility

The effect of ethanol on barium sulfate precipitation is assessed by monitoring the conductivity measurements in synthetic solutions during the precipitation reaction. The temporal variation of $\Delta\sigma$ curves in the various solutions S0, S1, and S2 is shown in Figure 2.

Figure 2 shows that ethanol influences the kinetics and thermodynamic of barite precipitation. All curves are distinguished by the existence of two zones. The first corresponds to a nonlinear variation in the conductivity as a function of time, corresponding to nucleation and to crystalline growth evolution ($t_{\text{ind}} + t_g$). The second step, which relates to the linear part of the curve, corresponds to the equilibrium state (end of the reaction). As the supersaturation coefficient is higher in our case ($\Omega_{\text{BaSO}_4} = 13,682$), the induction time is very small, which is why the induction step is not detectable [22].

On one hand, it is understood that ethanol added in different volumes affects the kinetics and thermodynamic of the barite precipitation reaction. Thus, when we increase the volume of ethanol, the total $\Delta\sigma$ variation decreases ($233 \text{ mS}\cdot\text{cm}^{-1}$ between S0 and S2). Therefore, the presence of ethanol decreases the mass of the precipitate obtained ($m_0 = 0.2131 \text{ g}$, $m_2 = 0.1940 \text{ g}$), which implies an increase in solubility. This result was expected due to the decrease in the dielectric constant and the polarity of the mixture (Table 1), as well as the difference in the value of the free energy of solvation [15–17]. This difference in polarity and dielectric constant leads, at the microscopic scale, to variations in the hydrogen bonds associated with water and ethanol molecules. Consequently, these factors directly affect the precipitation phenomenon. Some of the hydrogen bonds separating the molecules of water are disrupted when an ethanol molecule is added to the water. Although there are new attractions that occur between ethanol and water molecules, the structure of the water is significantly altered due to these new attractions differing from the ones that are broken.

On the other hand, for the same reason, Figure 2 illustrates that the kinetics of barite crystallization in ethanol–water mixtures for the supersaturated solution are affected. In the first step of the precipitation reaction (the crystal growth step), the rate of the precipitation reaction increases as the volume of the added ethanol increases. In addition, the crystal germination time, t_g , in S1 ($t_g = 110 \text{ s}$) and S2 ($t_g = 60 \text{ s}$) is noted to be less than in S0 ($t_g = 130 \text{ s}$). In this respect, the results of our study indicate that the dielectric environment can play a critical role in determining the relative stability of the crystalline phases (the nucleation process). These results imply that both nucleation and growth occur more quickly in ethanol than in water, which can also affect the morphology of barite particles. These results align with the precipitation of barite in the presence of methanol. Jones et al. linked this modification to the variation in the surface tension factor of barium sulfate particles [15]. The conductivity measurements show that the water–ethanol solutions exhibit both faster nucleation and crystal growth. It is anticipated that adding ethanol will alter the surface tension of the barite particles. The Gibbs–Thomson equation, the basis of classical nucleation theory, states that surface tension can influence the nucleation rate J by

$$J = v_D C_N \quad (4)$$

where v_D is the rate of impingement of ions on the nuclei, and C_N is the concentration of critical nuclei.

$$C_N = C e^{\frac{\Delta G}{k_B T}} \quad (5)$$

$$\Delta G^c = \frac{16 \pi \gamma^3 V_0^2}{3 k_B^3 T^3 \ln(S^*)^2} \quad (6)$$

where γ is the surface tension, C is the solution concentration, $S^* = C/C_{eq}$ is the supersaturation, and V_0 is the volume of a unit cell.

Referring to Equations (5) and (6), factors that can significantly affect the nucleation rate in water and water–ethanol mixtures at the same temperature and supersaturation levels are the variations in surface tension γ and solubility [15]. On one hand, as demonstrated in a different study [23], surface tension decreases in the ethanol–water mixture compared to that of pure water. On the other hand, solubility increases [15], directly implying an increase in the nucleation rate, in agreement with our experiment results.

3.1.2. Propan-1-ol Effect: Partial Miscible

In the case of propan-1-ol addition, Figure 3 shows the same phenomenon observed with the addition of ethanol. When the volume of propan-1-ol increases, the total $\Delta\sigma$ variation decreases (307 mS.cm^{−1} between S0 and S4). Therefore, the presence of propan-1-ol decreases the mass of the precipitate ($m_0 = 0.2131$ g, $m_4 = 0.1882$ g). Propan-1-ol demonstrates a greater effect compared to the addition of ethanol due to the significant difference in the dielectric constant and polarity between pure water and propan-1-ol (Table 1). These results imply that both nucleation and growth occur more quickly in the presence of propan-1-ol (the nucleation process) than in pure water, which can also affect the morphology of barite particles obtained at the end of the reaction [15–17]. These variations can be attributed to the difference in solubility and in surface tension γ (Equations (4)–(6)).

3.2. Solid Study

The solid phase obtained was characterized by FTIR, XRD, and SEM techniques. We chose to characterize the solid phase recovered from the solutions S0, S2, and S4.

3.2.1. FTIR

The infrared spectra of barium sulfate precipitate obtained from solutions S0, S2, and S4 (Figures 4–6) show several peaks. Peak assignments were made based on the literature data [15,24].

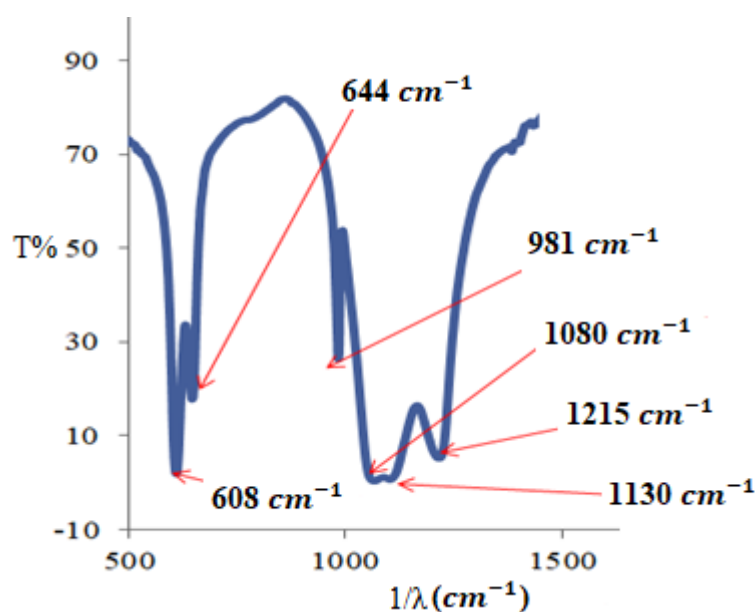


Figure 4. FTIR transmittance spectrum of pure BaSO_{4(s)} obtained in pure water (S0).

Figure 4 shows the FT-IR spectra of the pure BaSO_4 obtained from solution S0. The literature states that the sulfate group has three fundamental vibrational modes (T_d molecular point group). There is one nondegenerate mode (V1) at 981 cm^{-1} assigned to the symmetric stretching vibration of the SO_4^{2-} group (mode (V2) is inactive); one doubly degenerate mode (V4) in the low frequency domain at 608 cm^{-1} and 644 cm^{-1} , corresponding to deformation vibration; and one triply degenerate mode (V3) at 1080 cm^{-1} , 1130 cm^{-1} , and 1215 cm^{-1} assigned to the antisymmetric stretching vibration of the SO_4^{2-} group [24].

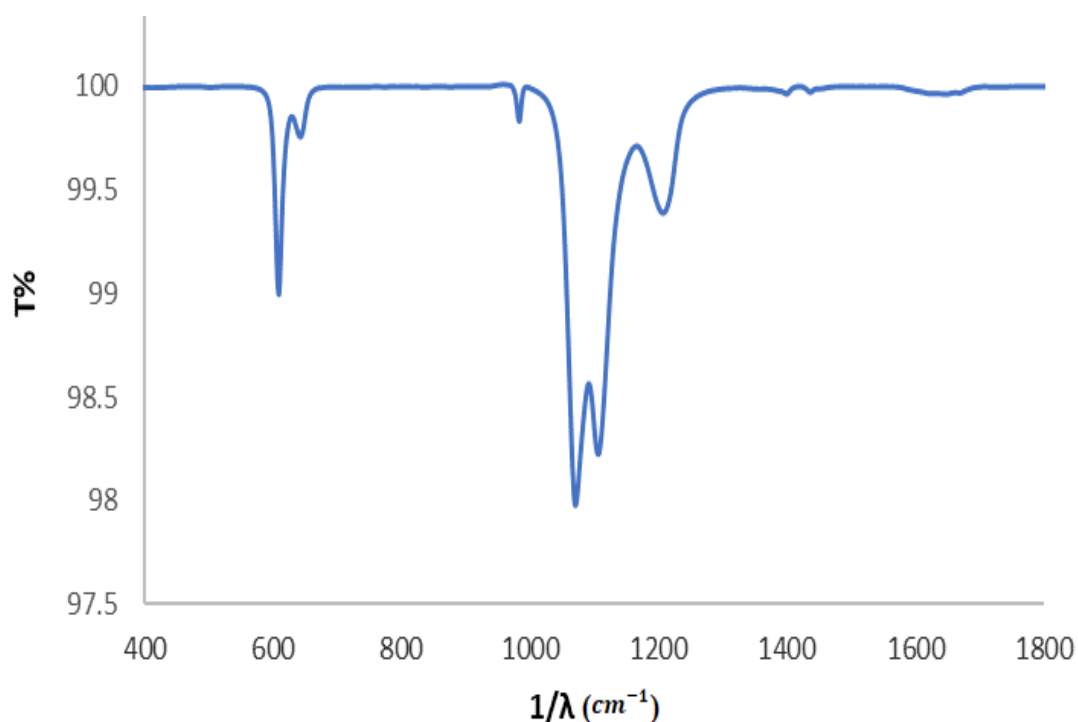


Figure 5. FTIR transmittance spectrum of pure $\text{BaSO}_{4(s)}$ obtained in the presence of ethanol (S2).

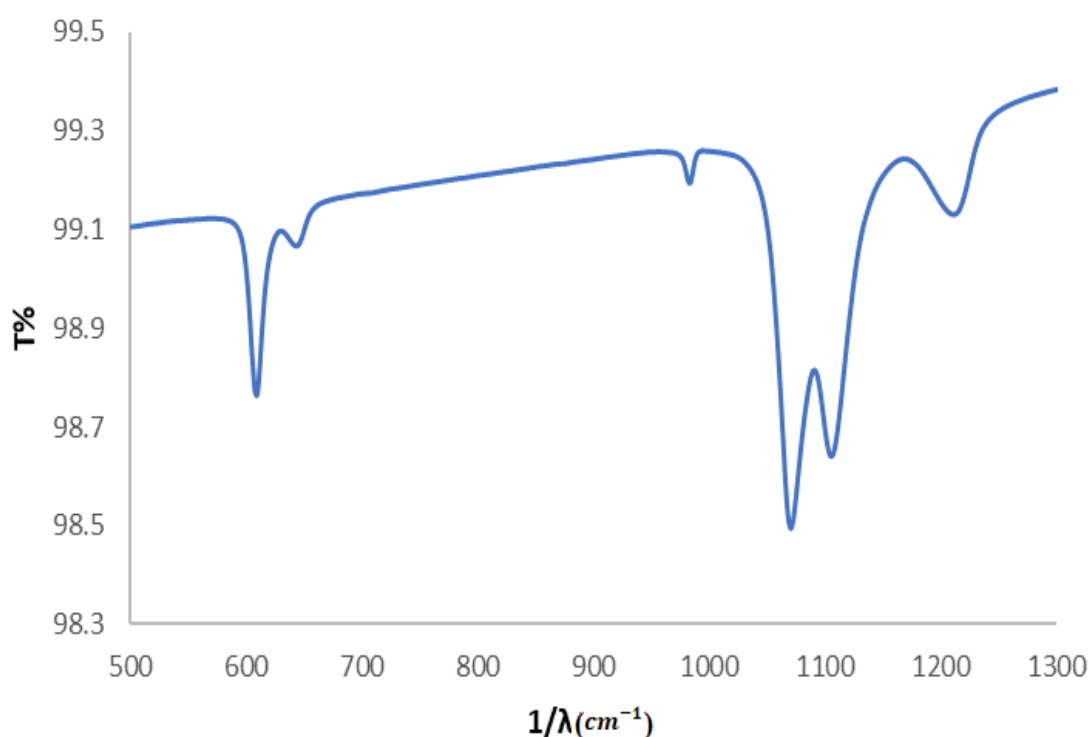


Figure 6. FTIR transmittance spectrum of $\text{BaSO}_{4(s)}$ obtained in the presence of propan-1-ol (S4).

The comparison of the three spectra in Figures 4–6 shows that the presence of ethanol and propan-1-ol leads to a difference in the antisymmetric vibration frequencies (V3). This difference can be attributed to a structural difference in the crystal lattice attributed to the presence of organic solvents [15,24]. That indicates a modification in morphology and in the lattice parameters. Given the presence of all the pics related to the vibrations of the sulfate group in the barite network, we can confirm that the solid phase obtained from the S2 and S4 solutions is barium sulfate. To confirm these results, we examined the solid deposits obtained using the X-ray diffraction technique to characterize the structure and the electron microscopy technique to analyze the morphology.

3.2.2. XRD

The identification of the solid phase obtained from S2 and S4 was carried out by matching it with highScore Plus, identified as the barium sulfate ICSD pattern-code 01-080-0512 [25]. Figures 7 and 8 confirm the BaSO_4 structure for both solid phases obtained from S2 and S4.

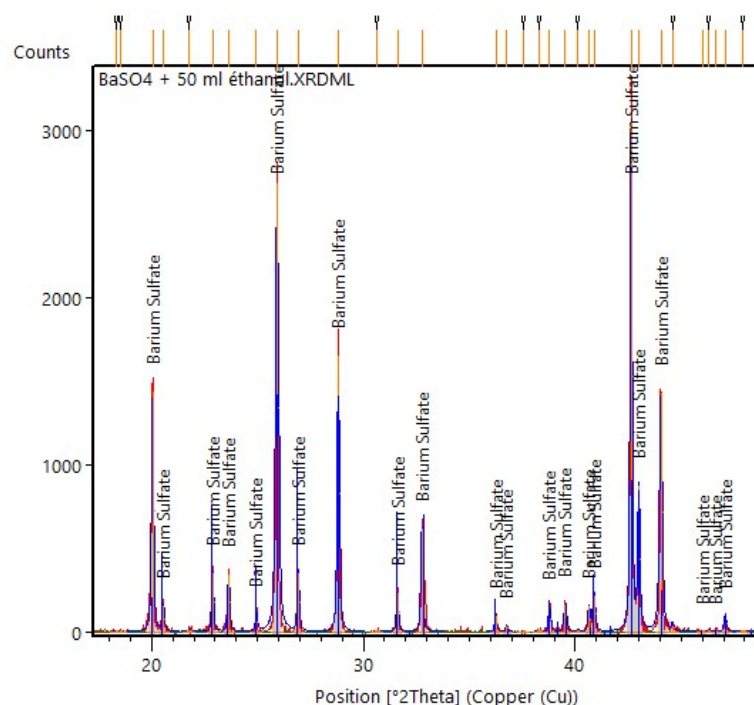


Figure 7. Qualitative characterization of the solid obtained from S2 via HighScore plus software.

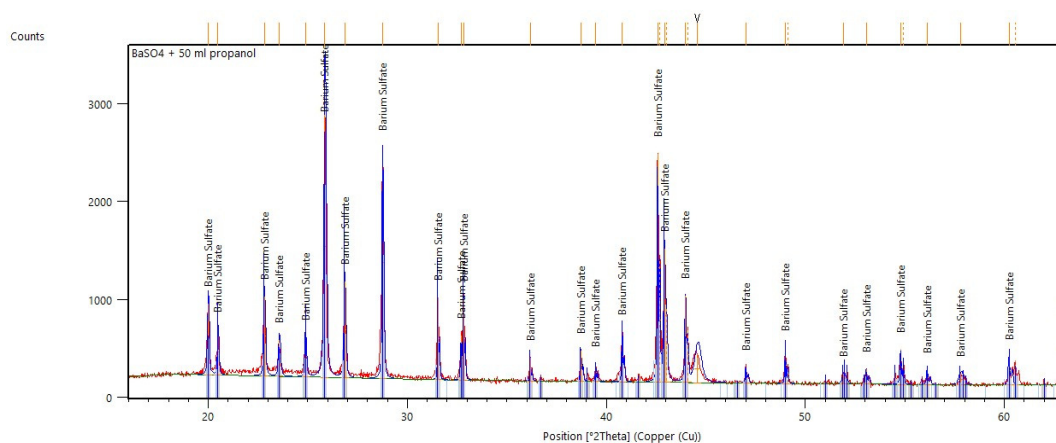


Figure 8. Qualitative characterization of the solid obtained from S4 via HighScore plus software.

Figure 9 shows that the presence of an organic solvent causes a slight shift in the XRD peaks to higher 2θ angles [9]. The peak shift arises from lattice distortions involving a slight compression of the crystal lattice, which results in a decrease in the unit cell volume. In addition, we can observe a modification in the position of the principal peak. These results allow us to predict that the morphology of barite will change in the solutions S2 and S4 because the main orientation of germination will be modified due to a modification in the electrostatic field [15,26]. Indeed, XRD results confirm the FTIR results.

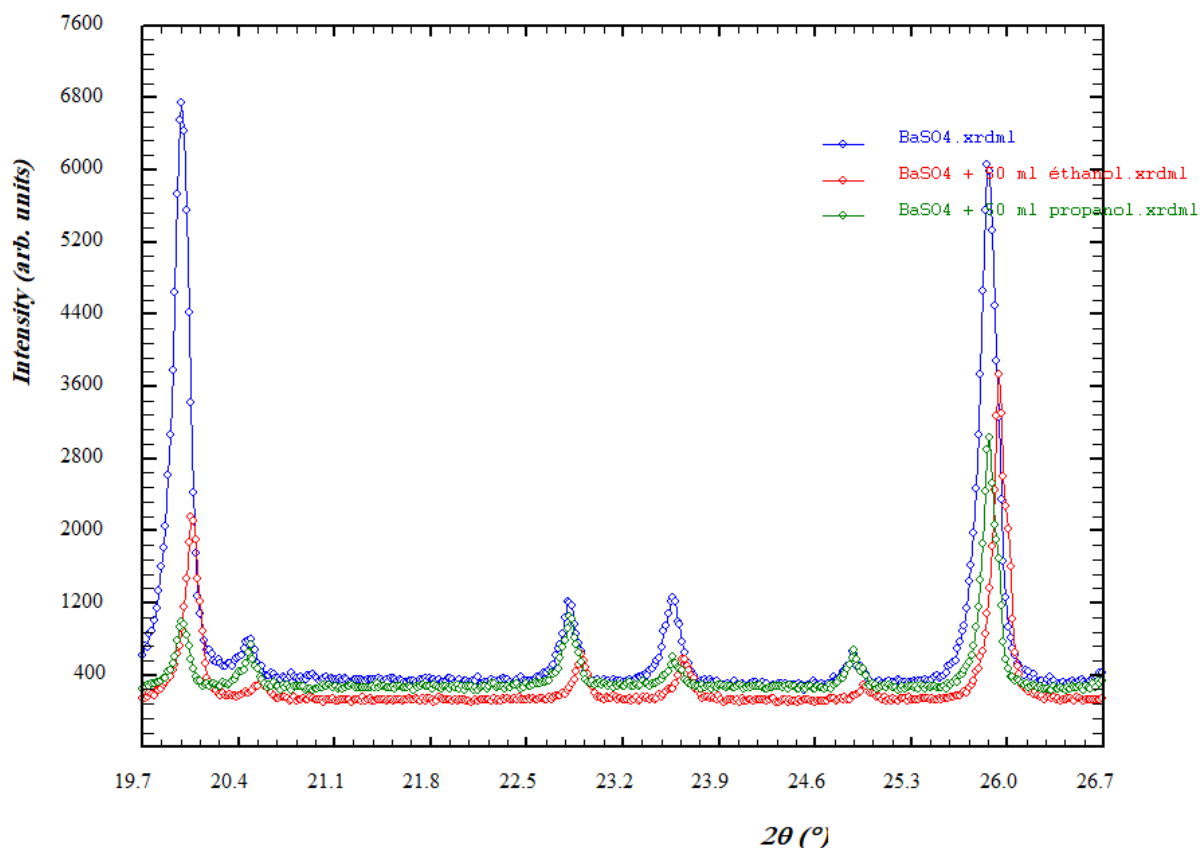


Figure 9. Superposition of XRD patterns of barite obtained from S0, S2, and S4.

3.2.3. SEM

The solid phase obtained from solutions S0, S2, and S4 was also examined using the SEM technique to confirm our FTIR and XRD results related to morphology interpretation.

SEM images depicted in Figures 10–12 indicate that the morphologies of solid barite obtained from S2 and S4 are different compared to S0. We observed a strongly anisotropic growth of two-dimensional nuclei [5]. The variations in the solvent surface contacts of polar crystals must be linked to a noticeable differential in growth rate at their opposing hemihedral faces. According to a specially designed, tailor-made inhibitor theory, a solvent acting as an adsorbed additive would inhibit the regular deposition of incoming molecular layers and hence prevent crystal formation at that face [27]. Thus, the difference in interaction is attributed to the difference in crystal growth direction.

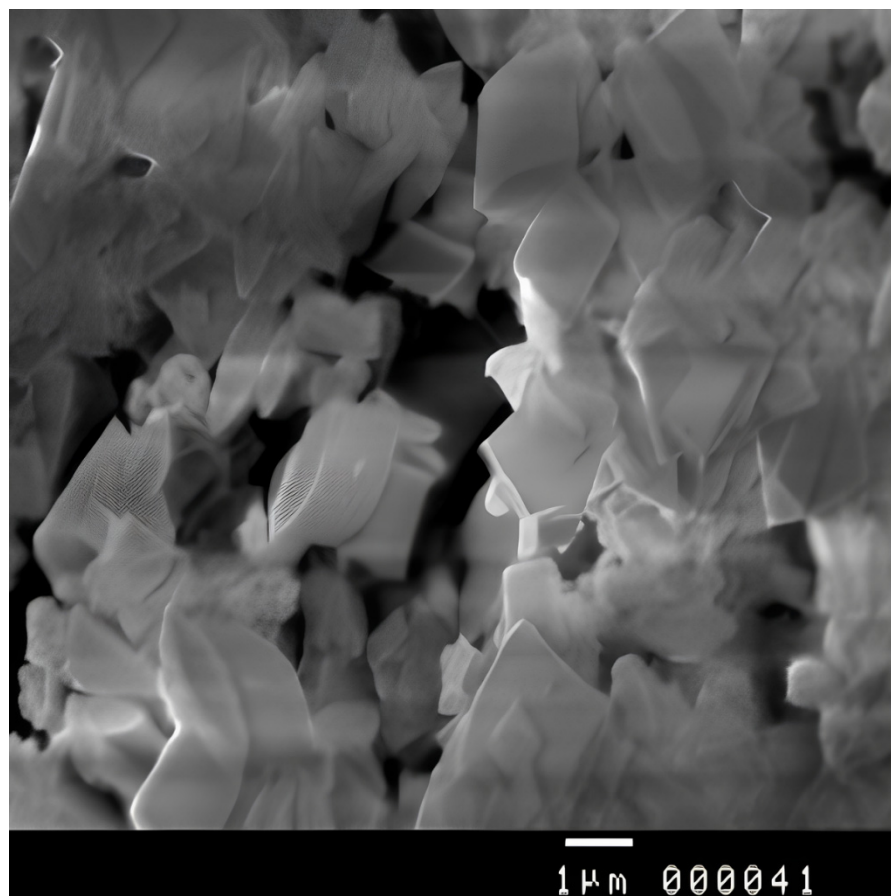


Figure 10. SEM image of a barite solid obtained from S0.

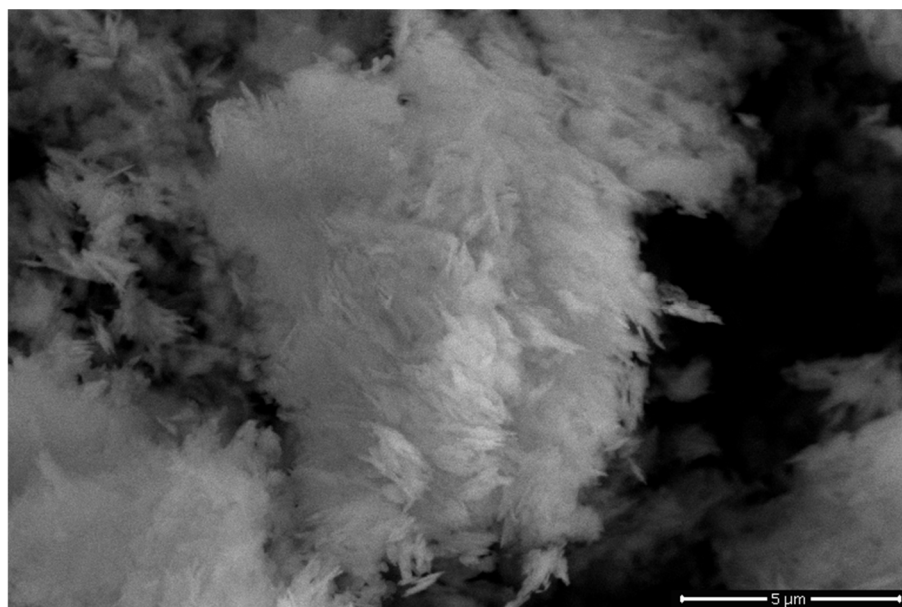


Figure 11. SEM image of a barite solid obtained from S2.

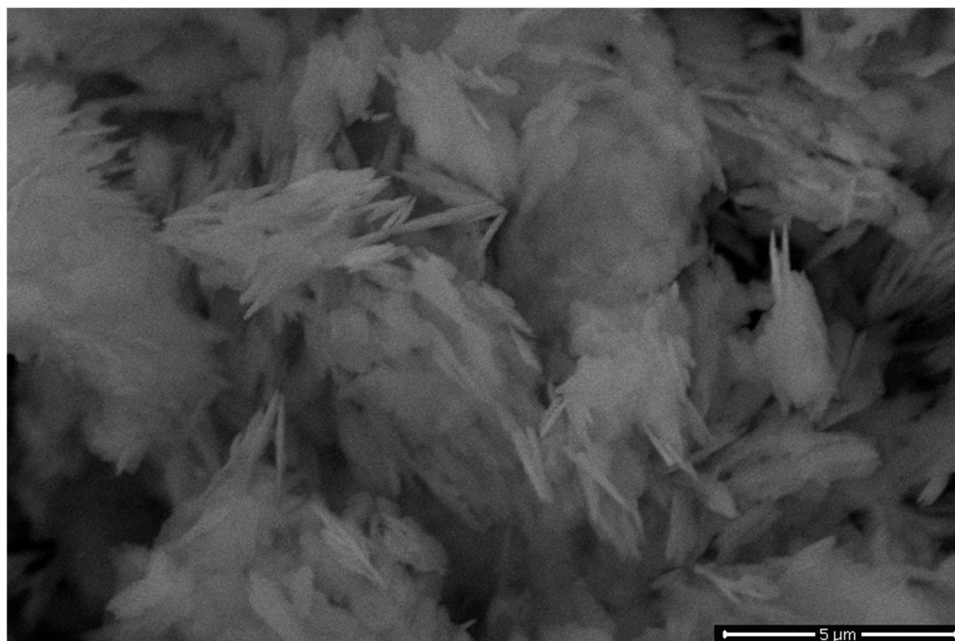


Figure 12. SEM images of a barite solid obtained from S4.

The agglomeration of solid barite is more pronounced in S2 and S4 than in S0. This result can be attributed to differences in crystal–crystal interactions in various solutions (dynamics of collisions), which affect the development of crystalline bridges and inter-particle consolidation during the agglomeration of barite due to differences in crystal growth rates (Figures 2 and 3). Crystal agglomeration and aggregation disturbances depend on crystal growth rates [10,28,29].

SEM images illustrated in Figures 11 and 12 show that in the presence of ethanol, a homogeneous morphology can be achieved from S2 (total miscibility of solvent), while a heterogeneous morphology can be achieved from S4 (partial miscibility of solvent). This result can be attributed to the difference in miscibility between ethanol and propan-1-ol in aqueous solution.

All solid characterization techniques confirm that there is a structural and morphological difference attributed to the presence of organic solvents, according to the conductivity study.

4. Conclusions

This work explores the influence of adding organic solvents (ethanol and propanol-1-ol) into barium sulfate precipitation in an aqueous medium. The difference in physical parameters, such as polarity and dielectric constant, between water and these organic solvents provides a perturbation in the intermolecular interaction between solute and solvent, especially the hydrogen bond. Further analysis of the conductivity variation in the saturated solution of barium sulfate shows that stronger variations in intermolecular interactions between the solute and the solvent affect the kinetics of the nucleation process and the mass of the precipitate obtained. It can be seen that the water–ethanol and water–propanol solutions exhibit faster nucleation and crystal growth. In addition, when the volume of ethanol increases, the total $\Delta\sigma$ variation decreases (indicated by a decrease in the mass of the precipitate obtained), implying an increase in barite solubility. Solid phase analyses using FTIR, XRD, and SEM techniques confirm the conductivity results. For the solid phase obtained, FTIR and XRD spectra illustrate a slight difference in water–ethanol and water–propan-1-ol mixtures compared to pure water. Both techniques confirm the formation of barite in all solvent mixtures. SEM images confirm all previous analyses and

show a significant difference in morphology and agglomeration phenomena due to the presence of organic solvent. The difference in morphology between the solids obtained from S2 and S4 arises from the differing miscibility with water of ethanol and propan-1-ol.

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Data Availability Statement: The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest: The authors declare no conflicts of interest.

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