



Establishing Test Procedures for Determining Precipitate Sedimentation Rates at Varying Temperatures

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Abstract

A laboratory setup and test procedure for determining precipitate sedimentation rate at varying temperatures are presented. The objective is to improve on conventional sedimentation study apparatus as well as generate reproducible sedimentation data under controlled thermal conditions. The setup comprises a transparent graduated cylindrical tube for sedimentation, a transparent water bath for heat transmission, a magnetic stirrer for uniform heat distribution, a temperature-controlled heating mantle for heat generation, a thermometer and a digital stop watch for timing. The procedure involves monitoring gravitational sedimentation of precipitate formed in a reaction medium over time. The setup has been applied to study the influence of temperature on the sedimentation profiles of aluminium and iron (II) phosphate precipitate suspensions. The setup is suitable for quantifying sedimentation rates of insoluble inorganic compounds and assessing the physical stability of pharmaceutical suspensions.

Keywords: Precipitate sedimentation, Heating setup, Test procedure, Temperature, Aluminium phosphate, Iron (II) phosphate

Introduction

Precipitation processes-whether from concentrated, saturated or sub-saturated solutions-typically involve intense nucleation of dissolved ions into stable solid-phase (precipitate) particles (Nduna et al., 2013). Sedimentation (or settling) of precipitate particles follows immediately after the precipitation process and continues until the final particles are formed. Precipitate sedimentation may therefore be defined as the gravitational settling of particles formed from solution medium to the bottom of containing vessel. The phenomenon is a crucial physico-chemical process in water purification technology, where it facilitates the removal of impurities rendered settleable by precipitation or flocculation. Sedimentation study provides important information such as particle size, precipitate sedimentation rate, erythrocyte sedimentation rate and other physical instability characteristics such as compaction of drug materials in pharmaceutical suspensions. Previous studies (Jain, 2014; Obunwo et al., 2014; Obunwo & Iboroma, 2015; Konne et al., 2017) have examined the influence of several factors such as variations in reactant, additive or particle concentration, on sedimentation behaviours. However, studies investigating sedimentation under varying temperature conditions remain limited. This scarcity may be attributed to the lack of simple, unautomated, and reproducible methodologies for observing and quantifying sedimentation in basic laboratory settings. In response to this gap and the need to study sedimentation behaviour of some alkaline-earth phosphate precipitate suspensions at different temperatures, an experimental sedimentation setup was improvised using conventional laboratory apparatuses and appliances (Iboroma, 2019). The setup comprises a cylindrical tube, water bath, magnetic stirrer, heating mantle, thermometer and stop watch. It was employed subsequently by Amadi (2022) to determine sedimentation rates of insoluble aluminium and iron (II) phosphates at 30°C, 40°C and 50°C. The present article aims to present a detailed description of the setup and test procedure for determining sedimentation rate profiles of aluminium and iron (II) phosphate precipitate suspensions at different temperatures, thereby making this methodology accessible and reproducible for the broader scientific community.

Precipitate Sedimentation Setup

Plate 1 shows the manual precipitate sedimentation measurement setup. It consists of transparent graduated cylindrical tube (1), transparent water bath (2), magnetic stirrer (3), digital temperature-controlled heating mantle (4), thermometer (5) and digital stopwatch (6). The digital stopwatch indicates time, while the thermometer displays the working temperature. The digital temperature-controlled heating mantle, which is tunable, provides

regulated heat, while the magnetic stirrer distributes this heat evenly through the water bath. The graduated cylindrical tube serves as the container where sedimentation of precipitate occurs. Plate 2 is a simplified schematic representation of the photograph shown in Plate 1, illustrating the heating components and their relationship with the sample (precipitate). At any given temperature setting on the heating mantle, the magnetic stirrer distributes heat through the water bath to the graduated cylindrical tube. The movement made by the precipitate in the cylindrical tube is manually recorded by the experimenter.

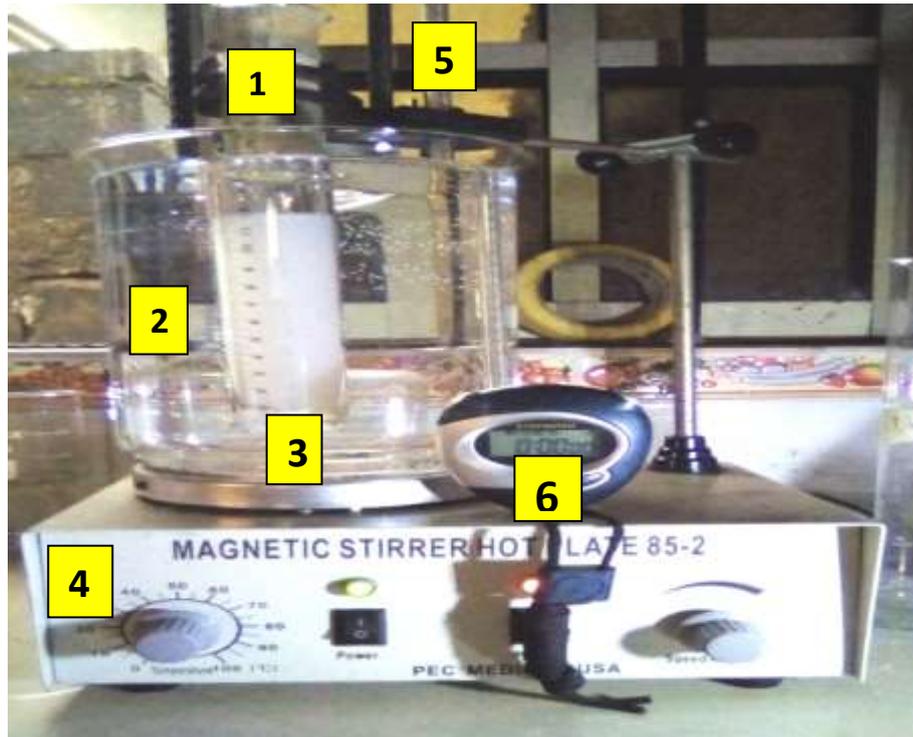


Plate 1: Manual (Heating) Precipitate Sedimentation measurement Setup

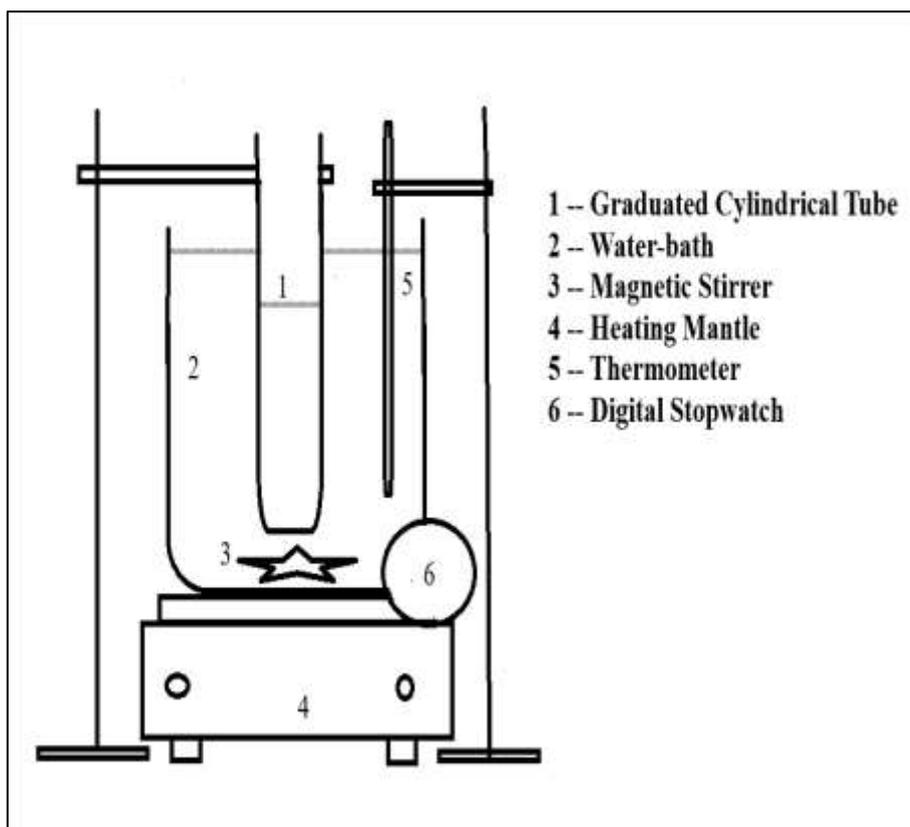
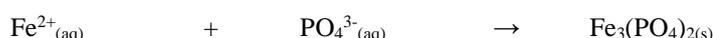
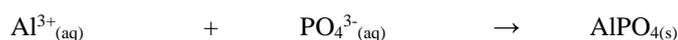


Plate 2: Schematic Representation of Manual (Heating) Precipitate Sedimentation measurement Setup

Precipitate Sedimentation Test Procedure

To describe precipitate sedimentation test procedure, the record of aluminium and iron (II) phosphate precipitates sedimentation test operations performed by Amadi in 2022 was reviewed. Accordingly, the heating components (heating mantle, water bath and magnetic stirrer) were appropriately positioned and connected to an alternating current source. The graduated cylindrical tube and thermometer were clamped in the water-bath as shown in Plate 2. Temperature control knob on the heating mantle was adjusted to 30°C setting, and the actual working temperature was reached when the thermometer stabilized at 30°C. The magnetic stirrer control knob on the heating mantle was also adjusted to achieve uniform heat distribution. The digital stopwatch was placed within view to measure time.

Analytical-grade JHD chemicals-aluminium chloride ($[\text{AlCl}_3 \cdot 6\text{H}_2\text{O}]$, M.W. = 241.43g/mol), iron (II) chloride ($[\text{FeCl}_2 \cdot 4\text{H}_2\text{O}]$, M.W. = 198.81g/mol) and disodium hydrogen phosphate ($[\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}]$, M.W. = 358.14g/mol)-were used to form aluminium phosphate $[\text{AlPO}_4]$ and iron (II) phosphate $[\text{Fe}_3(\text{PO}_4)_2]$ precipitate suspensions. Solutions were prepared as follows: 0.5M solution of aluminium chloride from 120.73g, 0.05M solution of iron (II) chloride from 9.94g, and 0.5M and 0.05M solutions of disodium hydrogen phosphate from 179.07 and 17.91g, respectively, each dissolved in de-ionized water and made up to one litre in a volumetric flask. To form aluminum phosphate, 30ml of 0.5M solution of disodium hydrogen phosphate (as PO_4^{3-} precursor) was mixed with 30ml of 0.5M solution of aluminium chloride (as Al^{3+} precursor). For iron (II) phosphate, 30ml of 0.05M disodium hydrogen phosphate solution was mixed with 30ml of 0.05M iron (II) chloride solution (as Fe^{2+} precursor). The precipitate formation reactions are given below:



Each suspension was stirred for one minute with a glass rod to obtain a homogeneous precipitate mixture, then introduced into the graduated cylindrical tube and allowed to settle. Sedimentation under gravity was monitored manually, recording the height of the precipitate (hp) or interface between precipitate and supernatant every minute-up to 10 minutes for aluminium phosphate suspension and 25 minutes for iron (II) phosphate suspension-until complete settling occurred. Initial height (hp_0) at time zero (t_0) and subsequent heights (hp_i) after each time

interval (t_i) were recorded. The test was repeated at 40°C and 50°C. Plots of sedimentation height (h_p) versus time (t) for 30°C, 40°C and 50°C settings were generated to show the influence of temperature on sedimentation rate.

Results

Figure 1 shows the sedimentation profiles of aluminium phosphate precipitate at 30°C, 40°C and 50°C. Figure 2 shows that of iron (II) phosphate under the same conditions. For both precipitates, sedimentation rate increased with temperature. At 50°C, sedimentation occurred fastest, followed by 40°C and 30°C, which exhibited the slowest rate.

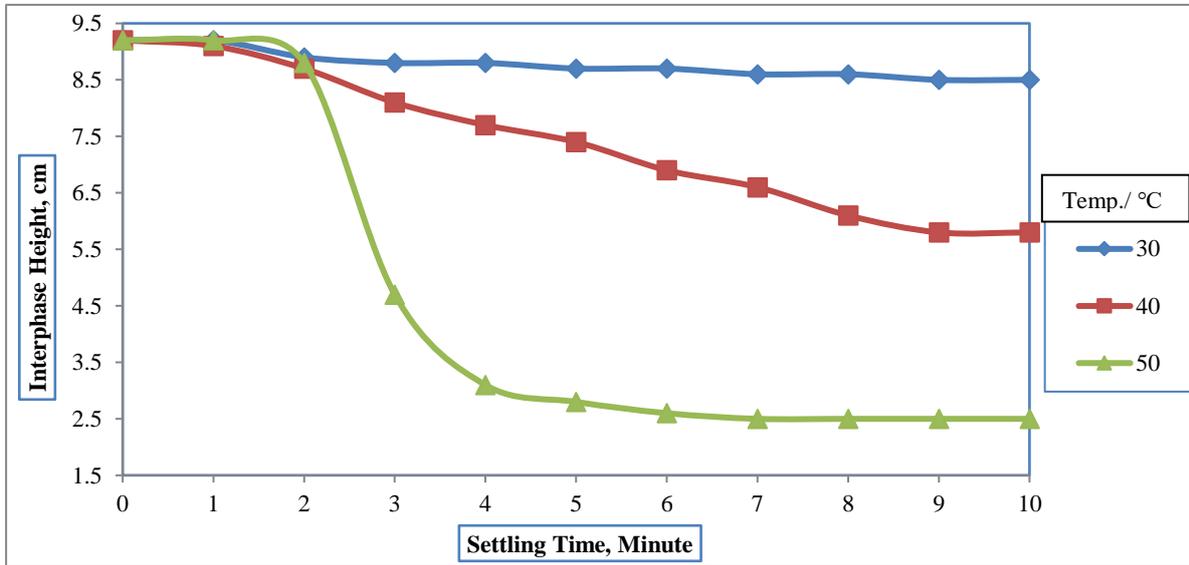


Figure 1: Sedimentation Profile of Aluminium Phosphate as Function of Setup Temperature

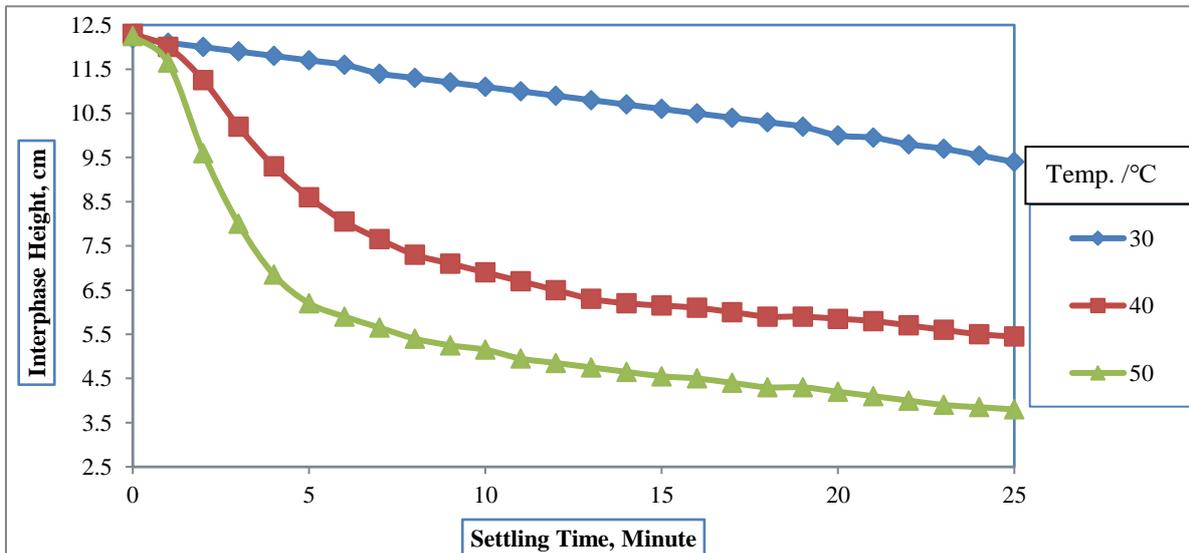


Figure 2: Sedimentation Profile of Iron (II) Phosphate as Function of Setup Temperature.

Discussion

Sedimentation of precipitate is a well known phenomenon. Traditional measurements often use measuring cylinders or jars under ambient temperature (Obunwo et al., 2014; Obunwo & Iboroma, 2015; Konne et al., 2017). However, the present heating setup, assembled from standard laboratory apparatuses and appliances, illustrated in plate 2, is novel. The setup enables stable, controlled-temperature sedimentation measurements. Transparent components also allow visual monitoring of colour, texture, and volume of the precipitate. Developed in May 2018 at the photochemistry laboratory, Rivers State University, Port Harcourt, the setup represents an improvement over conventional ambient devices, allowing sedimentation rate measurements at multiple temperatures (Figures 1 and 2) while minimizing external fluctuations. Comparative tests demonstrated that

results (not given here) obtained with mounted heating components differed markedly from those without. This confirms also that temperature stabilization significantly affects sedimentation behaviour. The setup can thus be applied in soil analysis, inorganic sedimentation rate measurements, and evaluation of pharmaceutical suspension stability. Its major limitation lies in operator dependence due to manual monitoring and data recording. Nonetheless, it provides a foundation for further automation and optimization by the scientific community.

Conclusion

A simple laboratory setup and test procedure for determining sedimentation rates of precipitate particles under controlled temperatures have been developed. The setup employs a tunable digital heating mantle and auxiliary components for stable heat generation and distribution. The procedure involves tracking precipitate sedimentation over time, producing reliable data that illustrate the influence of temperature on sedimentation behaviour. The setup proved efficient for determining precipitate sedimentation rates at controlled temperatures. It is suitable for studying sedimentation of insoluble inorganic compounds and evaluating the physical stability of pharmaceutical suspensions.

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