SEDIMENTATION STUDY OF TRIBASIC MAGNESIUM PHOSPHATE PRECIPITATE IN AQUEOUS MEDIUM

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IJASR 2019
VOLUME 2
ISSUE 2 MARCH – APRIL **ISSN: 2581-7876**

Abstract – Sedimentation data of bioactive materials are essential in the preparation of their suspension drugs. In the present work, sedimentation rate of tribasic magnesium phosphate (TMP) precipitate was determined with respect to magnesium-to-phosphate (Mg:P) volume ratio, stirring and temperature, using the simultaneous ion variation method. It was found that TMP precipitate sedimentation is unaffected by stirring but influenced by high temperature conditions. It is also influenced by Mg:P volume ratio if high volume of magnesium ions is mixed with low volume of phosphate ions. In three consecutive stirrings, TMP precipitate showed 0% increase in sedimentation rate but exhibited 100% decrease between 30oC and 50oC rise in temperature. Evaluated sedimentation rate of TMP precipitate remained relatively low (0.029cmminute-1 - 0.089cmminute-1) when Mg:P volume fraction was varied from 0.1 to 0.8 except the highest volume fraction (0.9) which gave high value (0.650cmminute-1). Change in viscosity resulting from particle-medium and particle-particle interactions is the main factor identified in this study. The results may be employed as physical stability information in formulation of magnesium phosphate suspensions.

Keywords: sedimentation, magnesium-to-phosphate, stirring, temperature, tribasic magnesium phosphate, precipitate

Introduction

Sedimentation data of bioactive materials provide useful information vital for describing their physical stability characteristics [1]. They are also employed in the determination of molecular weight of proteins and other macromolecules of biological interests [2].

Magnesium phosphates are gaining attention as bone resuscitation materials [3] with comparable or in some cases better properties than calcium phosphates [4]. As such they are also being developed as suspension drugs, for the same purpose. Suspension drugs show sedimentation phenomenon before and during storage. A number of factors including size, density and concentration of particles, viscosity and density of medium as well as precipitation conditions (temperature, stirring, mixing ratio, reactant concentration and pH) affect sedimentation [5 - 7]. Interactions among particles and with the aqueous environment also influence sedimentation behaviour of particles in suspension. Previous study dedicated to exploring sedimentation behaviour of magnesium phosphate with respect to magnesium-to-phosphate volume ratio, temperature and stirring is unavailable. Obunwo et al. [6] had presented the settling velocities of tribasic calcium and strontium phosphates precipitates. In the present work, sedimentation rate of tribasic magnesium phosphate (TMP) precipitate was determined by sedimenting TMP precipitate in its mother liquor, using the Simultaneous Ion Variation Method. The aim was to provide sedimentation data of TMP precipitate, to add to existing data on sedimentation.

Materials and methods

Analar grade magnesium nitrate hexahydrate (Mg(NO3)2.6H2O) and tribasic sodium phosphate (Na3PO4) from BDH Chemicals Limited, Poole, England were procured. Thermostated precipitate sedimentation measurement apparatus set up for this purpose [1] was used for the study. Precipitate sedimentation measurement procedure describing the Simultaneous Ion Variation Method [6] was employed.

Magnesium-to-Phosphate (Mg:P) Volume Ratio Effect: Into the sedimentation apparatus, 10ml of 0.6M Mg(NO3)2 solution was mixed with 90ml of 0.6M Na3PO4 solution to form tribasic magnesium phosphate(TMP), [Mg3(PO4)2], precipitate. The precipitate was stirred for 120 seconds and monitoring of the sedimentation process commenced immediately after mixing. The precipitate displacement was recorded every

minute, for 18 minutes. The procedure was repeated at 20:80; 30:70; 40:60; 50:50; 60:40; 70:30; 80:20 and 90:10 magnesium ions to phosphate ions volume ratios. The volume combinations were coded 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, and 0.9 in mole fraction terms, to represent the reagents mixing or volume ratios in the sedimentation apparatus set at 30oC.

Stirring Effect: In the determination of the effect of stirring on the residence time, the 30:70 volume ratio was selected for the tests. The selection was based on interface clarity, indicating that it gave relatively high particle size distribution. As it was earlier, the precipitate formed was stirred for 120 seconds and the displacement of the suspension was monitored and recorded every minute, for 18 minutes. The precipitate was re-suspended by stirring and the procedure repeated two more times. The experiments were carried out in the sedimentation apparatus set at 30oC.

Temperature Effect: In the determination of the effect of temperature, the 30:70 metal-to-phosphate volume ratio was also selected. Tests were performed in the sedimentation apparatus set at 5oC intervals between 30oC and 50oC. Monitoring and recording processes were followed.

Plot of change in precipitate height, [-(〖hp〗_i-〖hp〗_0)] versus time, (t_i-t_0), gave sedimentation profile. Where hpi is the height of the precipitate suspension at a given time, ti, and hp0 is the initial height of the suspension at time zero, t0. Sedimentation rate was determined from the slope of the plot of change in precipitate height against time (profile).

Results and discussion

Magnesium-to-phosphate volume ratio: The tribasic magnesium phosphate (TMP) sedimentation monitored at different magnesium-to-phosphate (Mg:P) volume ratios and time are presented in Figure 1. Figure 2 shows the evaluated sedimentation rate of TMP precipitate plotted against Mg:P volume fraction. The results showed that sedimentation of TMP precipitate was influenced by Mg:P volume ratio when high volume of magnesium ions was mixed with low volume of phosphate ions. The profile corresponding to 90:10 Mg:P volume ratio produced the fastest sedimentation. This was distantly followed by the 10:90 Mg:P volume ratio. The 50:50 Mg:P volume ratio produced the slowest sedimentation, in agreement with the evaluated TMP precipitate sedimentation rate (Figure 2). It is also in agreement with the visual observations that highest particle saturation was observed when equal volumes of magnesium and phosphate ions were mixed. It was generally observed that precipitate content varied in the reaction mixture as Mg:P volume ratio was varied, with the 50:50 and 90:10 volume ratios respectively producing the highest and lowest particle saturation in all the tests. Precipitate concentration (saturation) increased from 0.1 to 0.5 and decreased thereafter from 0.6 to 0.9. Thus, a perfect curvi-linear relationship between TMP precipitate sedimentation rate and magnesium-to-phosphate volume ratio, with the 10:90 and 90:10 volume ratios producing the highest sedimentation rate was expected. However, this was not completely achieved possibly due to the interaction of TMP precipitate with the aqueous medium.

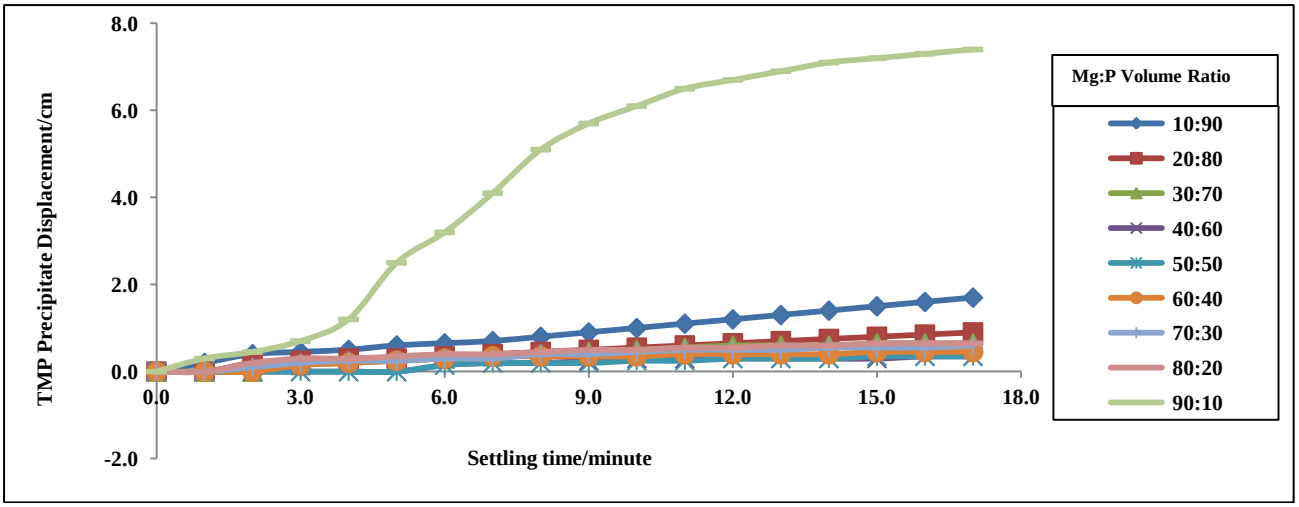

Figure 1: Sedimentation Profiles of TMP Precipitate for different Mg:P Volume Ratios.

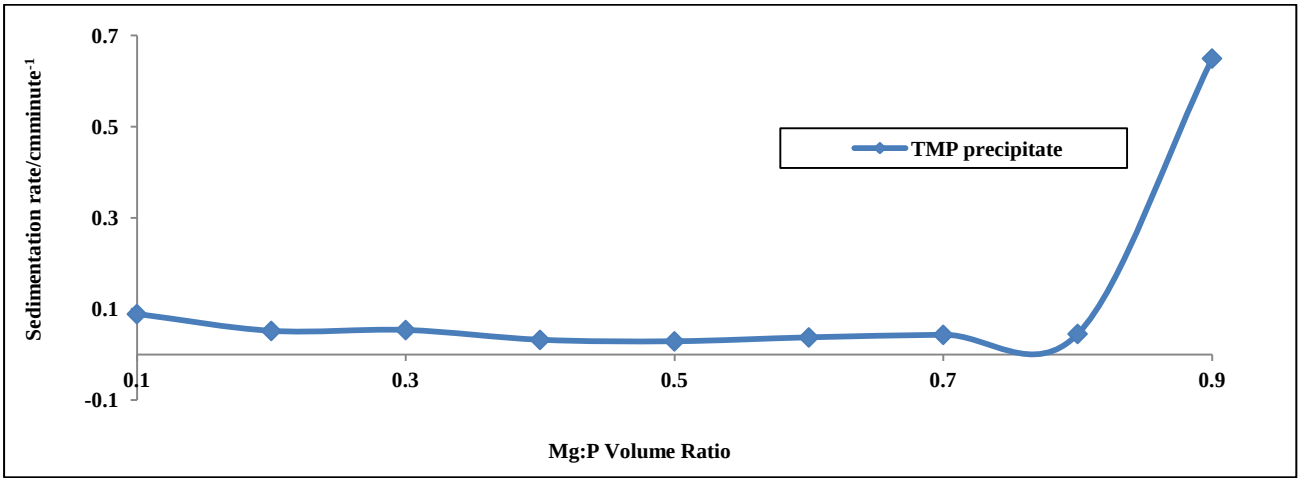

Figure 2: Sedimentation Rate of TMP Precipitate as a function of Mg:P Volume Fraction.

Stirring: Results of TMP precipitate sedimentation profile in three consecutive stirring conditions are presented in Figure 3. The results showed that sedimentation of TMP precipitate was unaffected by stirring conditions. Figure 3 indicates that sedimentation profiles for first, second and third stirrings were similar (that is 0% increase). This suggests that TMP precipitate was strong and stable at that composition and temperature. Ordinarily, if the particles were weak, breakage will occur and slower sedimentation would have resulted in the process of controlled stirrings. On the other hand, if the particles were not stable, crystal growth, and thus faster sedimentation would have occurred during the long residence time. Similar observation and inference were drawn by Obunwo et al. [6] when they determined the sedimentation rate of tribasic calcium phosphate precipitate in three consecutive stirring conditions.

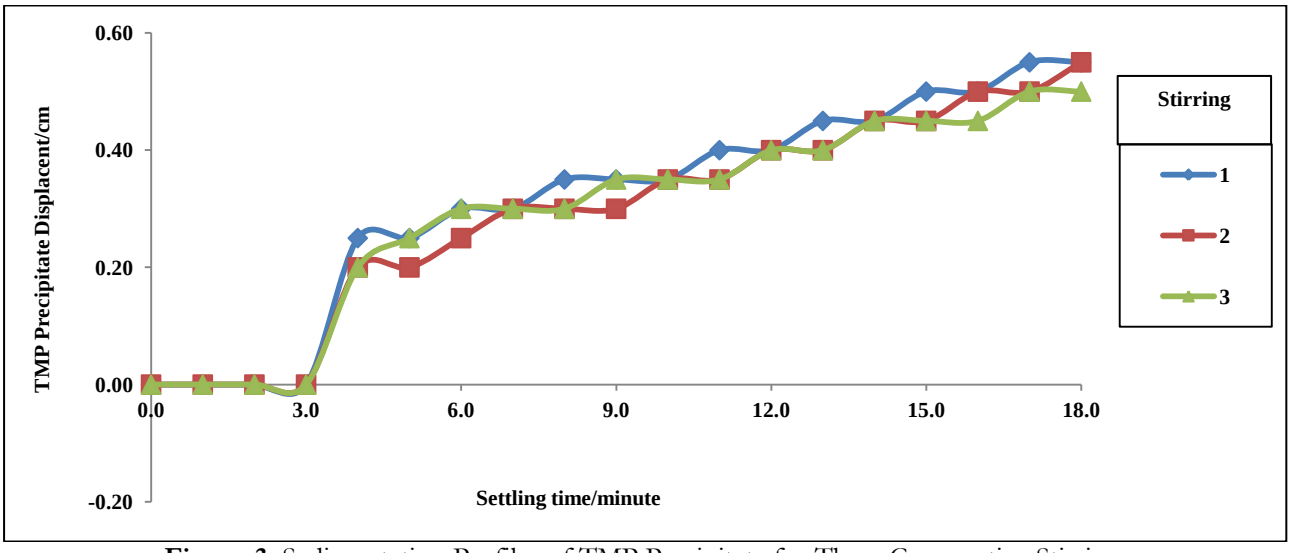

Figure 3: Sedimentation Profiles of TMP Precipitate for Three Consecutive Stirrings.

Temperature: The TMP precipitate displacements with time at various temperatures are presented in Figure 4. Sedimentation rate profile of TMP precipitate plotted against temperature is also presented in Figure 5. The results (Figure 4 and 5) indicate that sedimentation of TMP precipitate was low and poorly influenced by temperature variations. Profiles of 30 and 40oC (Figure 4) show longer sedimentation times while those for 35 and 45oC stopped shortly after commencement. The profile of 50oC shows almost no sedimentation. It was observed that the TMP precipitate formed upon mixing was gelatinous. The mixture also exhibited various degrees of viscosity which was observed to be highest at 50oC. The irregular and low sedimentation observed as the temperature was raised may be due to the interaction of the gelatinous TMP precipitate with the aqueous medium. The TMP precipitate suspension which was initially less viscous at 30oC became highly viscous above

50oC with no observable sedimentation. It is assumed that heat caused TMP precipitate to interact with the aqueous solution, made it become more diffuse and allowed water to penetrate the surface thereby converting it into a jelly-like suspension which could not settle. The 100% drop in sedimentation rate (Figure 5) observed when medium temperature was varied between 30 and 50oC also means that an increase in viscosity might have occurred and was responsible for the observed decrease. Influence of temperature on sedimentation is based upon the fact that viscosity of fluids decreases with rising temperature and thus faster sedimentation [8,9]. However, this generalization did not hold for TMP precipitate as temperature-induced particle-medium and particle-particle interactions affected the process. In an aqueous environment of this nature solution-driven interactions of precipitate with H+ and OH- affect sedimentation behaviours [10,11]. At higher temperatures solubility of particles in suspension medium may increase and the possibility of transformation into new products with entirely different properties may also result. Besides, additional viscosity is imparted to a suspension system if solid concentration is relatively high and may out-weigh the influence of temperature on viscosity.

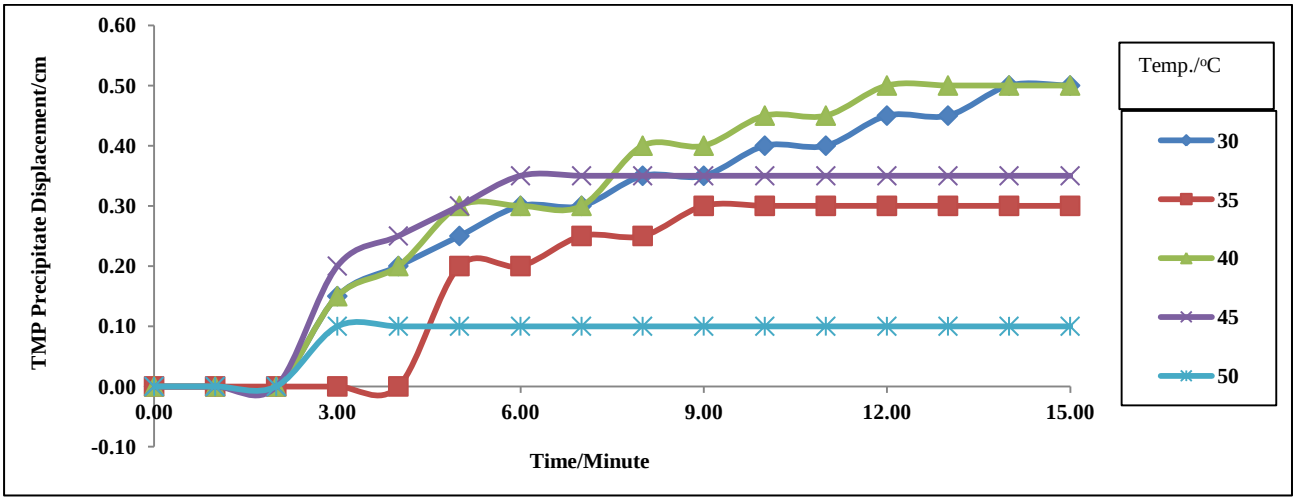

Figure 4: Sedimentation Profile of TMP precipitate at various Temperatures.

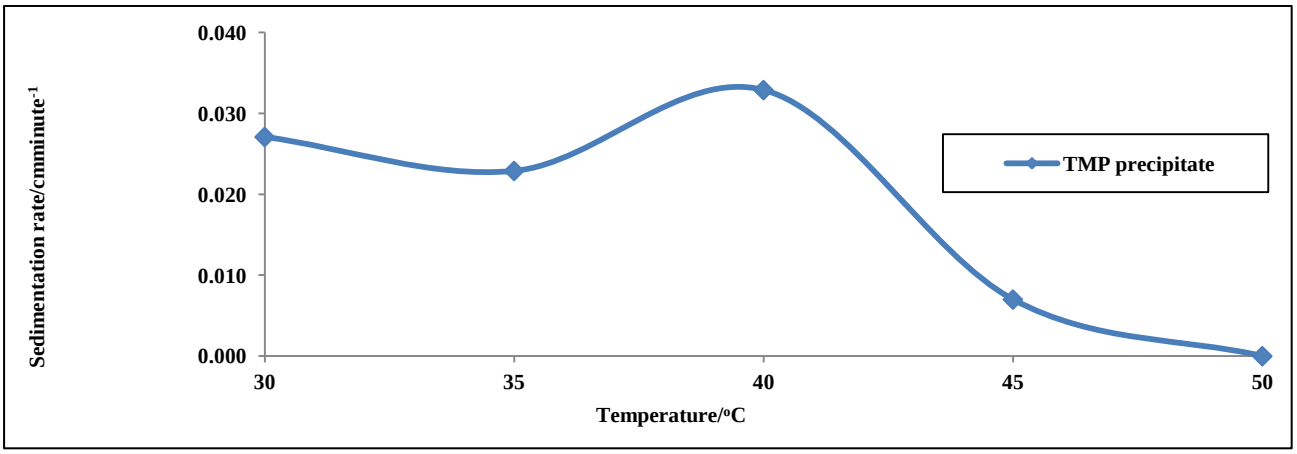

Figure 5: Sedimentation rate of TMP precipitate as a function of Temperature.

Conclusion

The sedimentation rate of tribasic magnesium phosphate (TMP) precipitate has been determined with respect to magnesium-to-phosphate (Mg:P) volume ratio, stirring and temperature using simultaneous ion variation method. The results show that TMP precipitate exhibits no sedimentation above 50°C and is unaffected by stirring. The results also suggest that TMP precipitate exhibits high sedimentation rate when magnesium-phosphate composition of mixture is ninety percent in excess of magnesium. Particle-medium and particle-particle interactions contributed immensely to the viscosity of the medium. The information obtained in this work can be exploited in the formulation of magnesium phosphate suspensions in pharmaceutical industries.

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