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SEDIMENTATION BEHAVIOUR OF BARIUM PHOSPHATE PRECIPITATE IN AQUEOUS SOLUTION

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Abstract – The sedimentation rate of barium phosphate (BP) precipitate in aqueous medium has been determined with respect to barium-to-phosphate (Ba:P) volume ratio, concentration of sodium carboxymethylcellulose (Na-CMC), temperature and stirring, using the batch-wise 'Simultaneous Ion Variation Method'. The results show that BP precipitate sedimentation is strongly influenced by stirring and high temperature conditions but weakly affected by Na-CMC biopolymer. It is also influenced by Ba:P volume ratio if low volume of barium or phosphate ions is mixed with high volume of phosphate or barium ions. Sedimentation rate of BP precipitate increased by 79% between first and second stirrings, 179% between 30 and 50oC rise in temperature and 42% between 200 and 2000ppm Na-CMC addition. BP precipitate formed at 0.1, 0.7 and 0.8 Ba:P volume fractions produced high sedimentation rate values (4.46cmminute-1, 4.49 cmminute-1 and 4.96 cmminute-1) while other volume fractions (0.2, 0.3, 0.4, 0.5 and 0.6) gave lower values (2.46 cmminute-1, 3.74 cmminute-1, 1.96 cmminute-1, 1.90 cmminute-1 and 2.42 cmminute-1). These results have been discussed based on change in amount of particles formed, interactions between particles in suspension and with aqueous medium.

Keywords: barium phosphate, stirring, temperature, Ba:P volume ratio, sodium carboxymethylcellulose, sedimentation rate

Introduction

Sedimentation rate is an important parameter useful for describing physical stability behaviours of suspensions [1-3] and in modeling fluidization and solid/liquid flow operations in environmental engineering [4].

Sedimentation behaviours of precipitates of several inorganic compounds have been studied with many interpretations. In most studies [4 - 7] the rate has been found dependent on concentration of solutions before mixing and degree of particle saturation in mixture after mixing. In continuation of our work on sedimentation of alkaline-earth metal phosphates [3, 8], we have determined the sedimentation rate of barium phosphate (BP) precipitate with respect to barium-to-phosphate (Ba:P) volume (mixing) ratio, concentration of sodium carboxymethylcellulose (Na-CMC) biopolymer additive, temperature and stirring, using the batch-wise 'Simultaneous Ion Variation Method'. The objective of this paper was to report the sedimentation rate data of BP precipitate which hitherto have not been presented in the literature.

Materials and Methods

Analytical grade barium nitrate [Ba(NO3)2] and tribasic sodium phosphate [Na3PO4] were procured and used without further purification. Sedimentation test procedure describing the batch-wise Simultaneous Ion Variation Method [9] was adopted and the apparatus for measurement of sedimentation of precipitate at different temperatures [8] was also used.

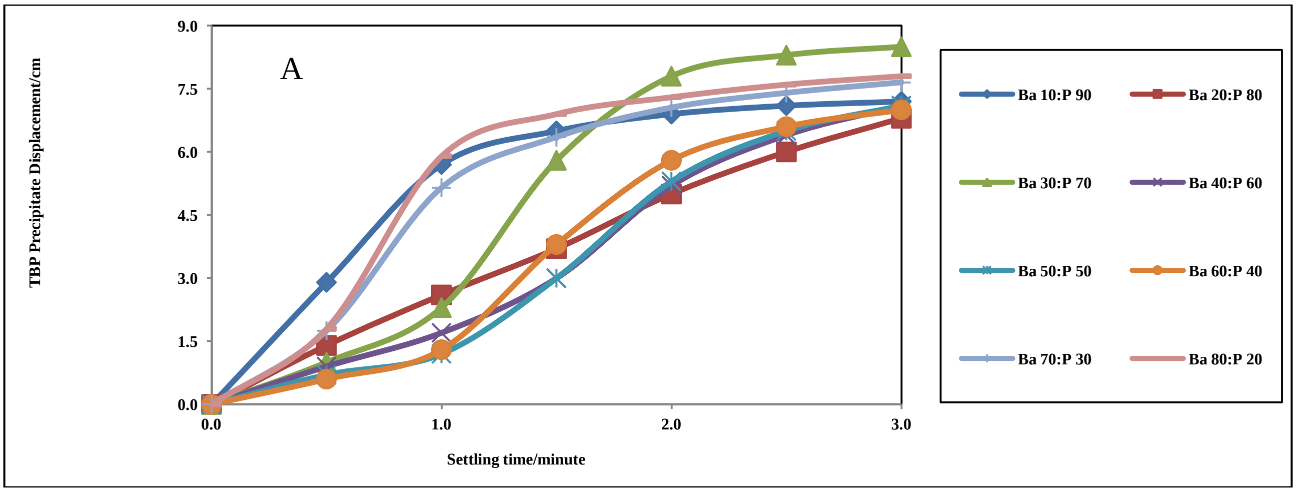
Treatments: Effect of barium-to-phosphate (Ba:P) volume ratio was investigated by mixing sub-saturated (0.1M) solutions of barium nitrate with tribasic sodium phosphate in various (10:90, 20:80; 30:70; 40:60; 50:50; 60:40; 70:30; 80:20 and 90:10) volumes ratios. This gave barium phosphate (BP) precipitate suspension of various Ba:P compositions (0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, and 0.9), in mole fraction terms. The precipitate was stirred and monitoring of sedimentation process commenced immediately. In the evaluation of the effect of sodium carboxymethylcellulose (Na-CMC) biopolymer, the 50:50 Ba:P volume ratio was selected after several trial runs. The selection was based on suspension interface clarity in the presence of Na-CMC biopolymer. Fixed volume (1ml) of different concentrations (200ppm - 2000ppm) of Na-CMC solution was added first to the phosphate ion solution. Resulting solution was then mixed with the barium ion solution. Monitoring and recording processes were followed. For temperature, 30:70 Ba:P volume ratio was selected. Selection was based on interface clarity in the absence of Na-CMC biopolymer. Tests were performed in the precipitate sedimentation measurement apparatus set initially at 30oC and were repeated at 35, 40, 45 and 50oC. For stirring, the 30:70 volume ratio was again selected for the test. Upon termination of the first test, the precipitate was re-suspended by stirring the second time and the procedure repeated two more times. The experiments were carried out in the precipitate sedimentation measurement apparatus set at 30oC.

In each experiment, the precipitate formed was stirred for 30 seconds and the displacement of the suspension was monitored and recorded every minute, and terminated when compression settling was minimal. Plots of change in precipitate height against time gave sedimentation profile. Sedimentation rate was then determined from the slope of the plot of change in height of precipitate against time.

Results and Discussion

Barium-to-phosphate: The BP precipitate sedimentation monitored at different barium-to-phosphate volume ratios are presented in Figure 1A. The evaluated sedimentation rate results are presented in Figure 1B. The results (Figure 1A) showed that sedimentation of BP precipitate was influenced by Ba:P volume ratio. Also, Figure 1B shows that BP precipitate sedimentation exhibited a curvi-linear relationship with Ba:P volume fraction with the 0.8 volume fraction producing the highest sedimentation rate (4.96cmminute-1) and was closely followed by 0.7 and 0.1 volume fractions with 4.49 cmminute-1 and 4.46 cmminute-1 respectively. Rate data for BP precipitate at 0.9 volume fraction was not recorded due to the absence of a clear suspension zone but by observation adjudged to have settled faster than other volume ratios. Also, the precipitate exhibited various degrees of particle saturation which were visually observed to be highest at 0.4 and 0.5 volume fractions.
The following order in sedimentation was obtained for BP precipitate:
Ba:P: 80:20 > 70:30 ≈ 10:90 > 30:70 > 20:80 ≈ 60:40 > 40:60 ≈ 50:50.

High particle saturation slows sedimentation down due to increase in viscosity resulting from particle-particle and particle-medium interactions that occur in suspension media [3]. Determination of precipitate content was not performed. However, by intuition particle saturation observed in each reaction mixture matched measured sedimentation data. Increase in precipitate concentration as Ba:P volume ratio was varied apparently imparted additional viscosity to the mixtures and could possibly the reason slow sedimentation occurred in Ba:P volume ratios having high particle saturation. The curvi-linear BP precipitate sedimentation rate profile (Figure 1B) also suggests that, though interaction of the particles with the aqueous environment [6] may have considerable effect, disperse phase content [10] may have been the most important physical parameter that affected the viscosity and thus sedimentation. Similar inferences have also been made on calcium and strontium phosphate precipitates [9] for which sedimentation rate profiles were curvi-linear. Sedimentation rate is a unique function of concentration-the greater the concentration of particles in suspension, the greater the inter-particle interactions and the slower the sedimentation.



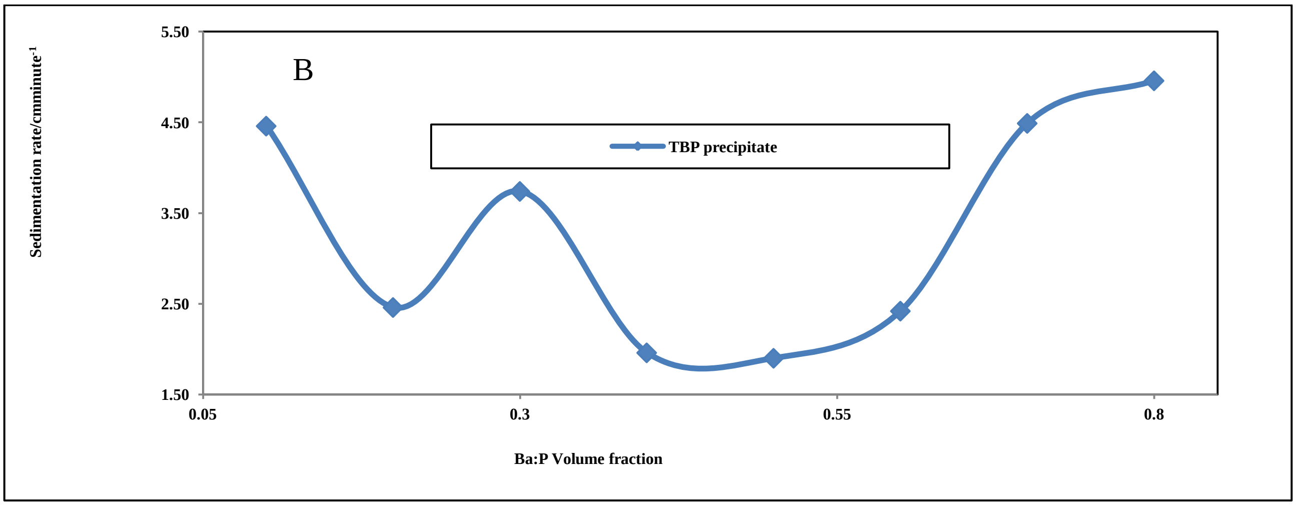
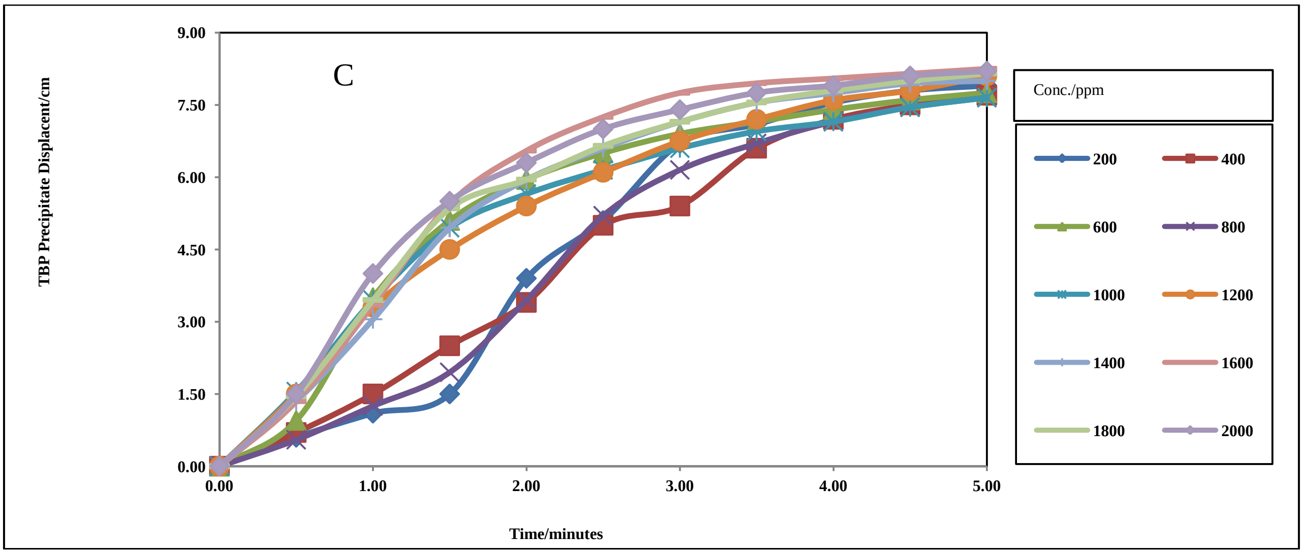


Fig. 1: Sedimentation (A) and Sedimentation Rate (B) profiles of BP Precipitate at Various Ba:P Volume Fractions

Concentration of sodium carboxymethylcellulose: The results of BP precipitate sedimentation in solutions containing various amounts of sodium carboxymethylcellulose (Na-CMC) are displayed in Figures 2C and D. The results show that BP precipitate sedimentation was weakly influenced by Na-CMC. Figure 2C indicates that BP precipitate containing low concentration of Na-CMC (that is profiles for 200ppm, 400ppm and 800ppm) had longer sedimentation times while those containing higher (that is 1000ppm, 1200ppm, 1400ppm, 1600ppm, 1800ppm, 2000ppm) had relatively shorter sedimentation times. Figure 2D also showed that between 200 and 2000ppm of Na-CMC addition, sedimentation rate of BP precipitate increased from 2.05cmminute^{-1} to 2.91cmminute^{-1} (42% increase). This describes a weak destabilization effect on BP precipitate suspension in presence of Na-CMC, possibly due to mild interaction of BP precipitate with the Na-CMC molecules. In an earlier study, Iboroma *et al.* [8] reported a strong destabilization effect on tricalcium phosphate precipitate in the presence of Na-CMC biopolymer. Affinity between calcium ion and carboxylate molecule of Na-CMC was adduced as responsible for the destabilization. The results of this study suggest that Na-CMC molecule has mild affinity for barium ion considering the weak destabilization inferred. They also mean that Na-CMC will not be a good stabilizing agent for suspensions containing barium considering the mild interaction observed.



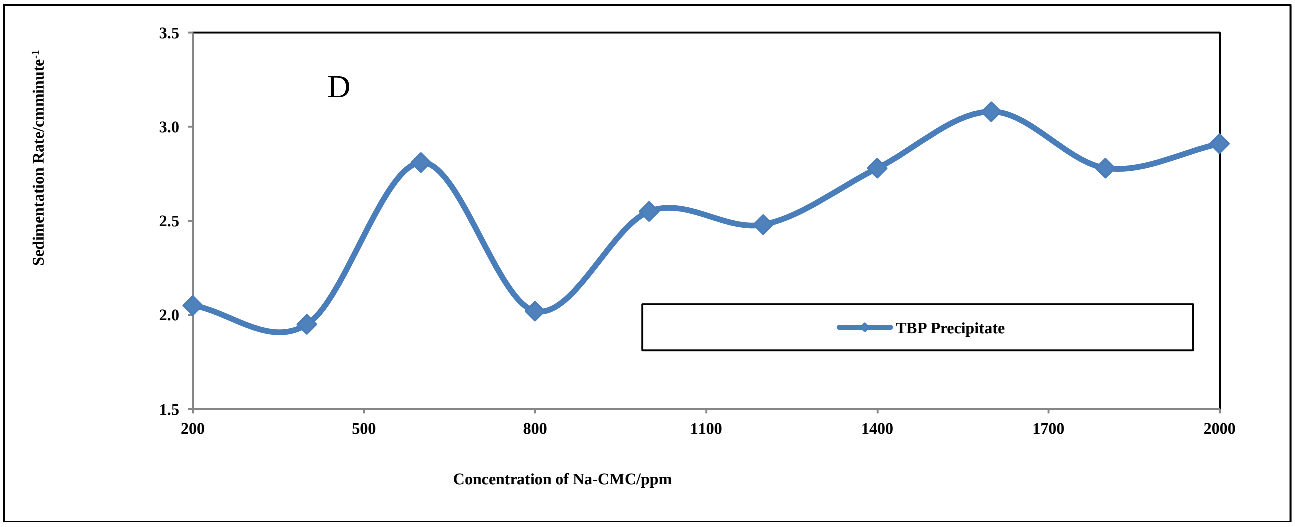
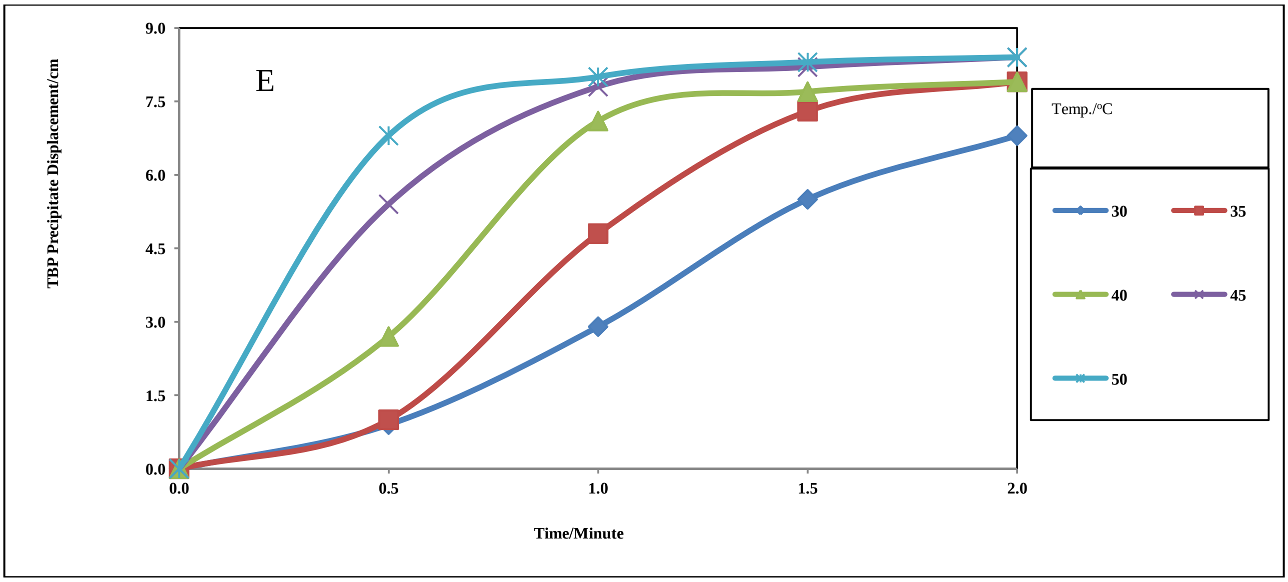


Fig. 2: Sedimentation (C) and Rate (D) Profiles of BP Precipitate as Function of Added Na-CMC Biopolymer

Effect of Temperature: Figures 3E and F display the sedimentation results of BP precipitate monitored at different temperatures ranging from 30 to 50°C. The results (Figures 3E and F) show that sedimentation of BP precipitate was influenced by temperature variations. In Figure 3E, profile for 50°C shows the fastest sedimentation followed by 45, 40 and 35, while that for 30°C is the slowest. As may be seen, BP precipitate reached equilibrium sedimentation in less than 1 minute at 50°C. Figure 3F shows the evaluated sedimentation rate results of BP precipitate at different temperatures. When the temperature of the precipitation medium was raised from 30°C to 50°C the sedimentation rate of BP precipitate formed increased from 2.90cm/minute to 8.00cm/minute (i.e. 176% increase in sedimentation rate). Decrease in viscosity of the medium, as temperature was raised, may be responsible for the faster sedimentation. Viscosity of fluids decreases with rising temperature and may lead to faster sedimentation of particles in suspension [10, 11]. In an earlier study, Iboroma *et al.* [3] reported slower sedimentation of trimagnesium phosphate precipitate with rise in temperature. According to the researchers, at high temperature, trimagnesium phosphate precipitate interacted with the aqueous disperse-medium and the suspension became viscous with no sedimentation at 50°C. The high sedimentation rate observed in this study is therefore an indication of low interaction of BP precipitate with the medium (water).



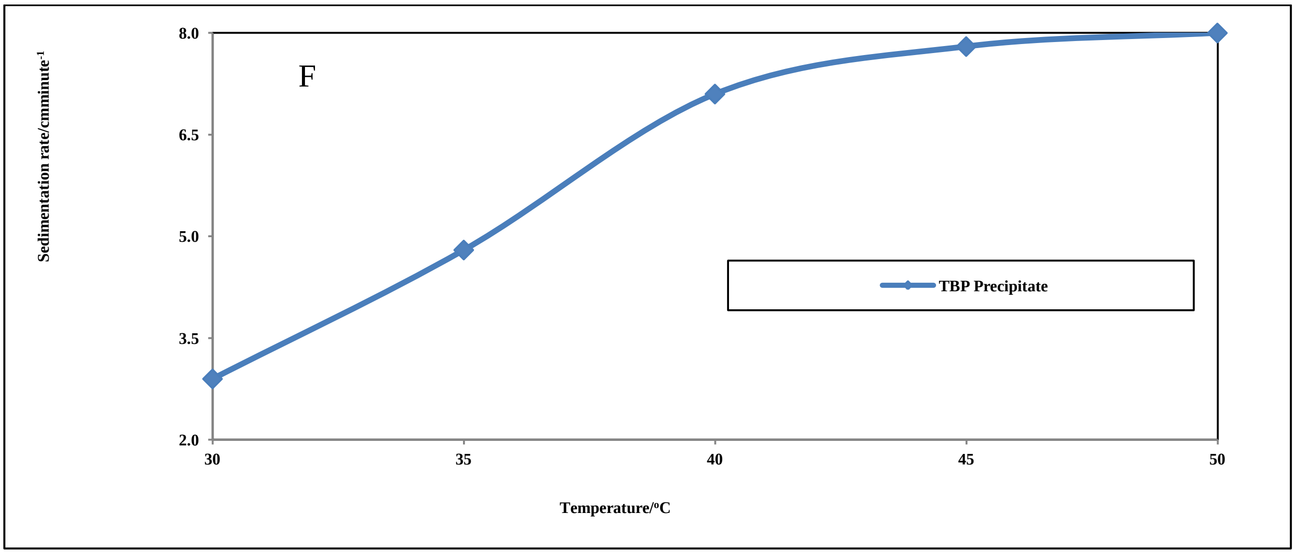


Fig. 3: Sedimentation (E) and Rate (F) Profiles of BP Precipitate at various Temperatures

Effect of Stirring: The results of BP sedimentation profile in two consecutive stirring conditions are presented in Figure 4. These results indicate that sedimentation of BP precipitate was influenced by stirring conditions. As may be seen in Figure 4 that profile for stirring 2 is higher than that of stirring 1. Data for the third stirring condition was not realized due to rapid dispersion of the precipitate particles. The suspension interface disappeared soon after the third stirring was undertaken. However, it was observed that the precipitate settled fast and the supernatant became clear in less than three minutes after the third stirring. Between the first and second stirrings, sedimentation of BP precipitate increased by 79%. These observations suggest that BP precipitate is unstable to stirring. However, the instability inferred is not to mean that BP precipitate is weak but the particles might have changed or grown from a more colloidal state to crystalline form in the process of controlled stirring and longer residence time in the mother solution. Precipitate particles grow in size by processes of dissolution and deposition. Smaller particles dissolve and re-deposit on surfaces of larger ones, resulting in particle growth, which over time leads to faster sedimentation [6].

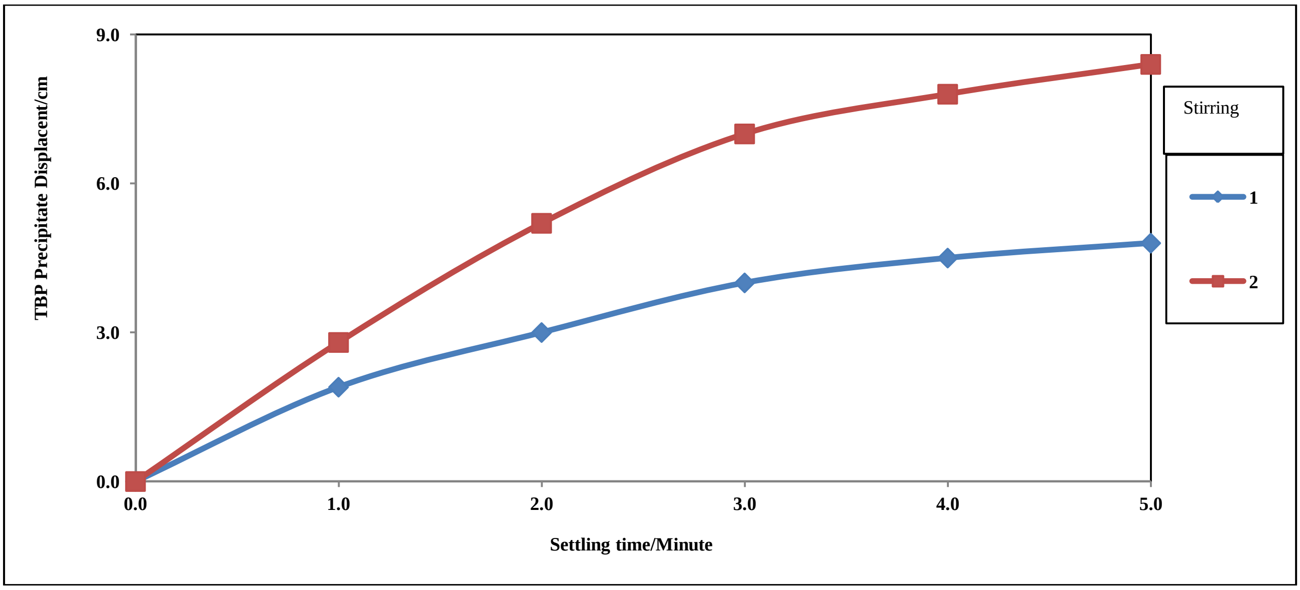


Fig. 4: Residence Time of BP Precipitate.

Conclusion

Sedimentation rate of BP precipitate has been evaluated with respect to Ba:P volume ratio, concentration of Na-CMC biopolymer, temperature and stirring using the batch-wise Simultaneous Ion Variation Method. The results show that BP precipitate is physically unstable to stirring and high temperature conditions; exhibits high sedimentation rate if barium-phosphate composition of mixture is low (0.1) and high (0.7 and 0.8). The results also suggest that BP precipitate weakly destabilizes in the presence of Na-CMC biopolymer. Change in medium viscosity resulting from changing particle concentration, interactions between particles in suspension and with the medium affect sedimentation of precipitate. This information may be employed in formulation of barium phosphate suspension fluids.

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