



# Adsorption of Zn(II) ions on Calcium Silicate Precipitation Tube (CaSPT) produced through 'silica garden' route



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## ABSTRACT

Zn(II) adsorption in aqueous medium has been reported on Calcium Silicate Precipitation Tube (CaSPT), grown by 'silica garden route'. Adsorption varies nonlinearly with adsorbent dose and initial adsorbate concentration. Adsorption data fit better with Langmuir isotherm model which returns Zn(II) loading capacity of 229.6 mg g<sup>-1</sup> at 0.01 g CaSPT. Adsorption kinetics follows pseudo-second order rate equation with activation energy of 8.56 kJ mol<sup>-1</sup>, typical for a physisorption process. The adsorption process is endothermic and spontaneous. CaSPT was successfully tested with regard to Zn(II) removal on a real life effluent sample collected from an electroplating industry.

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## 1. Introduction

Growth of self organized, apparently gravity defying, tree like tubular structures from the reaction of some metal salt crystals with aqueous sodium silicate is popularly known as 'silica garden' phenomenon. This phenomenon, essentially a precipitation reaction, is known for more than at least three centuries and a number of metal ions have been reported to produce silicate precipitation tubes (Coatman et al., 1980; Balkose et al., 2002). However, the applicability of this interesting class of compounds has remained less explored and only a few have been sporadically used as catalyst (Collins et al., 1999), heavy metal ion adsorbent (Parmar et al., 2011; Parmar et al., 2012), etc.

Zn(II) is a heavy metal ion that occurs naturally in air, water and soil. It is well known as a micronutrient for living organisms. However, the presence of Zn(II) in the biosphere beyond acceptable limits, presumably due to anthropogenic activities, may cause serious health problems (Nriagu, 2007).

Adsorption is a widely accepted and practiced technique for heavy metal ion removal in aqueous medium. With reference to Zn(II) adsorption, a wide range of adsorbents has been used that includes red mud (Gupta and Sharma, 2002), bauxite ore (Rout et al., 2009a), aluminum plant sand (Mohapatra et al., 2009), water washed and chemically treated clay (Samir, 2008) and low grade manganese ore (Rout et al., 2009b), to name a few. Calcium Silicate Precipitation Tube (CaSPT), prepared

through 'silica garden' route has recently been found to be a potential adsorbent for a number of heavy metal ions (Parmar and Bhattacharjee, 2014), Zn(II) being one of them. This communication reports detailed investigation on Zn(II) adsorption on CaSPT in aqueous medium, including adsorption kinetics and thermodynamics. The objective of this study was to explore in detail the potential of CaSPT as an adsorbent for Zn(II).

## 2. Materials and methods

### 2.1. Adsorbent [CaSPT]

Calcium Silicate Precipitation Tube (CaSPT), synthesized through 'silica garden' route was used as adsorbent. The details of its synthesis and characterization may be found elsewhere (Parmar et al., 2011).

### 2.2. Adsorbate [Zn(II)]

AR grade ZnSO<sub>4</sub>·7H<sub>2</sub>O, was used for making 1000 mg l<sup>-1</sup> stock Zn(II) solution from which other solutions were prepared through serial dilution as appropriate. 18 MΩ ASTM Grade 1 water was used for making the solutions.

### 2.3. Instrumentation

SEM (Hitachi 3400 N) and TEM (Philips C M 200) were respectively used for imaging the CaSPT microtubes and crushed particles. Flame

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atomic absorption spectrometer (GBC AVANTA), was used for Zn(II) estimation.

#### 2.4. Adsorption experiments

All adsorption experiments were carried out in 50 ml batches. Zn(II) solution of desired strength (initial concentration,  $C_0$ ), pH and a known weight ( $m$ ) of the powdered CaSPT were taken in a stoppered conical flask and shaken in a horizontal shaker over a period of pre-decided time for adsorbate–adsorbent contact.

For kinetic study, contact time was varied from 5 to 180 min at 25, 30, 40 and 50 °C, while for other experiments it was 60 min. After shaking, contents of the flask were allowed to settle for 30 min., filtered and the filtrate was analyzed for Zn(II) concentration (final concentration,  $C_e$ ). Zn(II) captured by CaSPT was calculated by subtraction ( $C_0 - C_e$ ). All experiments, unless specified, were carried out at room temperature (27 °C).

Adsorption–desorption hysteresis of Zn(II) on CaSPT surface was developed in the pH range of 2–9. Dilute NaOH and HCl were used for making pH adjustments. For adsorption, process described above was followed in the pH range of 2–9. For desorption, Zn(II) was first adsorbed over CaSPT without any pH adjustment for 60 min. The pH of the Zn(II) solution during this was 5.6. This was followed by pH adjustment of the mixture in the range 2–9, another contact session of 60 min in the horizontal shaker, settling for 30 min, filtration and analysis of the filtrate for Zn(II) concentration.

If  $C_0$ ,  $C_e$  and  $C_e'$  are the initial, post adsorption and post desorption Zn(II) concentrations respectively, then

Amount of Zn(II) adsorbed/trapped over CaSPT =  $(C_0 - C_e) / 20$  mg,

Amount of Zn(II) desorbed from CaSPT =  $(C_e' - C_e) / 20$  mg,

Amount of Zn(II) still trapped over CaSPT after desorption =  $(C_0 - C_e') / 20$  mg.

#### 2.5. Plot of adsorption–desorption hysteresis

Adsorption–desorption hysteresis was obtained by plotting the amount of Zn(II) retained by CaSPT surface after carrying out adsorption and desorption experiments in the pH range of 2–9, as per the procedure outlined in Section 2.4. If the amount of Zn(II) retained by CaSPT surface at a particular pH is not different from what observed after desorption in the same pH, one may assume that there is no desorption and Zn(II)–CaSPT sludge is stable at that pH. However, if Zn(II) retention by CaSPT after desorption at a particular pH is less than the corresponding value post adsorption at the same pH, it reflects Zn(II) leaching from the CaSPT surface and destabilization of the Zn(II)–CaSPT sludge at that pH. On the contrary if the Zn(II) retention by CaSPT after desorption at a particular pH is more than what observed post adsorption in the same

pH, it signals further adsorption and stabilization of Zn(II)–CaSPT sludge. This analysis comes handy while dealing with disposal of sludge.

#### 2.6. Optimization program SOLVER

SOLVER optimization program built in MS-Excel was used for the purpose of solving various equations to obtain the associated parameters. Sum of the square of errors between the experimental and calculated data was set as error function which was minimized by successive change of equation parameters. The set of parameters returning minimized error function was reported as the optimized set of parameters.

### 3. Results and discussion

#### 3.1. Characterization of CaSPT

Details on synthesis, characterization and surface property of CaSPT may be found elsewhere (Parmar et al., 2012). Figs. 1a, b and c show the synthesized CaSPT, scanning and transmission electron micrographs of 'as grown' CaSPT. From Fig. 1b it is apparent that the tube is brittle in nature with different exterior and interior morphology. The porous nature of CaSPT is also evident in Fig. 1b and c. These tubes were finely crushed before using as adsorbent.

#### 3.2. Effect of adsorbent dose

Effect of adsorbent dose (0.01–0.08 g) on Zn(II) uptake by CaSPT over a Zn(II) concentration range of 50.6–327 mg l<sup>-1</sup>, has been shown in Fig. 2. Metal uptake increases nonlinearly with increase in adsorbent weight. Complete Zn(II) removal could be effected with 0.07 g CaSPT from 50 ml of 100 mg l<sup>-1</sup> Zn(II) solution in 60 min.

#### 3.3. Effect of initial Zn(II) concentration

Zn(II) uptake by varying amounts of CaSPT over a concentration range of 50.6–327 mg l<sup>-1</sup> has been shown in Fig. 3. Zn(II) uptake is non linear with respect to initial Zn(II) concentration. This non linearity increases with increase in adsorbent weight. Figs. 2 and 3 together clearly indicate that Zn(II) adsorption on CaSPT is a non linear function of adsorbent weight and initial Zn(II) concentration. This observation will be discussed in greater detail in later part of this communication.

#### 3.4. Effect of pH on adsorption–desorption

Though desorption is the reverse process of adsorption it may or may not follow the same path as adsorption on changing pH. It should also be understood that while adsorption at a particular pH may be directly calculated from the difference between initial ( $C_0$ ) and post

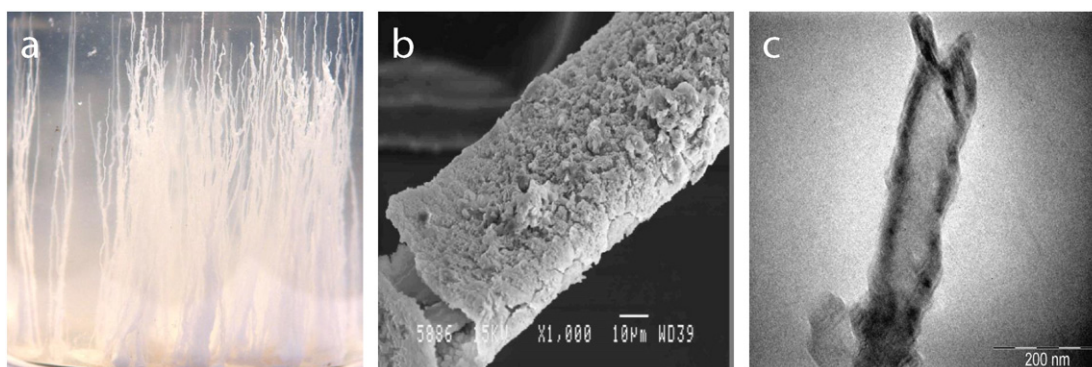


Fig. 1. CaSPT grown in the laboratory, (b) SEM image of 'as grown' CaSPT, (c) TEM image of crushed particle of CaSPT.

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