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Article title: Kinetics and morphology analysis of struvite precipitated from aqueous solution under the influence of heavy metals: Cu<sup>2+</sup>, Pb<sup>2+</sup>, Zn<sup>2+</sup>

Article reference: JECE2019

Journal title: Journal of Environmental Chemical Engineering

Corresponding author: Professor Athanasius Priharyoto Bayuseno

First author: Dr. Dyah S Perwitasari

Accepted manuscript available online: 22-NOV-2017

DOI information: 10.1016/j.jece.2017.11.052

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Dear Professor Bayuseno,

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**Reviewer #1:**

**Comment #1:**

This article addresses the use of struvite precipitation to remove metals from waste water, it is not clear to me what this article will add to our current knowledge and what kinds of wastewater has the potential to use this technique, please see my below comments;

**Response:**

We agree with Reviewer #1, and hence to eliminate ambiguity we changed the title.

**Original version**

Kinetics and morphology analysis of struvite precipitated from wastewater containing heavy metals.

**Revised version**

Kinetics and morphology analysis of struvite precipitated from aqueous solution under the influence of heavy metals:  $\text{Cu}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Zn}^{2+}$

**Comment #2:**

General: language needs to be improved.

**Response:**

We agree with Reviewer #1 that the language needs improvement. We have thoroughly checked the language and have consulted the following sources as references

1. Silyn-Roberts, H. 2000. Writing for Science and Engineering: Papers, Presentations and Reports, Butterworth-Heinemann, Oxford etc.
2. Weissberg, R. And Buker, S.1990. Writing Up Research: Experimental research report writing for students of English, Prentice Hall Regents, Englewood Cliffs, NJ 07632.

### Comment #3:

Abstract: please address the purpose and the key results of the study, revise the language.

### Response to Comment #3:

We agree to revise the abstract as well as the language.

#### Original version

The present study examined the influence of metal ions ( $\text{Cu}^{2+}$ ,  $\text{Pb}^{2+}$ , and  $\text{Zn}^{2+}$ ) on kinetics and morphology of struvite precipitation from the synthetic wastewater. Kinetics of struvite precipitation in the presence of metal ions [0, 1, 10, 50 and 100 ppm] was evaluated through the change of pH in the respective solution. In the experiment, the precipitating solid was then characterized by the XRPD Rietveld method. The morphology of crystals was studied by SEM equipped by EDX analysis. Results showed that the high proportion of struvite and minor impurity of sylvite was produced in the precipitate. The typical prismatic morphology of struvite was shown with sizes in the range of 10 and 60  $\mu\text{m}$ . The increase of ion concentrations in the supersaturated solutions, corresponding to the reduction of Mg content, made slower metal removal rates and reduced mass scale. The adsorption of the suspended metals to the surface of struvite crystals was apparently retarded and followed by distortions in the morphological characteristics. Here, recovery of phosphorus and ion metals from the wastewater through struvite precipitation potentially reduces heavy metal pollution in the environment and at the same time produce the valuable resources.

#### Revised version

The present study examined the influence of metal ions ( $\text{Cu}^{2+}$ ,  $\text{Pb}^{2+}$ , and  $\text{Zn}^{2+}$ ) on kinetics and morphology of struvite precipitated from aqueous solutions containing equimolar ratios of struvite components:  $\text{Mg}^{2+}$ ,  $\text{NH}_4^+$ ,  $\text{PO}_4^{2-}$ . Kinetics of the struvite precipitation in the presence of metal ions [0, 1, 10, 50 and 100 ppm] was evaluated through the change of pH of the precipitating solution. The kinetic evaluation demonstrated that the precipitation satisfactorily followed the first-order kinetic with respect to  $\text{Mg}^{2+}$ . It was found that for the three metal ions tested, the higher the concentrations of the metal ions: 0, 1, 10, 50, 100 ppm, the less the crystals obtained and the lower the rate constants. Depending on the concentrations of the metal ions added into the solution, the rate constants varied from 4.344 to 1.056  $\text{h}^{-1}$  which agree with most published values. It was postulated that the metal ions were adsorbed onto the surface of the crystals and hence retarded the growth. The crystals obtained were characterized using SEM-EDX and XRD. The characterization revealed that the precipitates were mainly struvite of various sizes (between 10 and 60  $\mu\text{m}$ ) with sylvite as impurities. It is envisaged that the present study would add to the understanding of the removal of metal ions from industrial wastewater through struvite precipitation.

**Comment #4:**

The introduction does not explain and present the effect of the three metals on struvite precipitation, please add some of the key results from literature.

**Response:**

We do not agree with Reviewer #1. The effect of  $\text{Cu}^{2+}$ ,  $\text{Pb}^{2+}$ , and  $\text{Zn}^{2+}$  on struvite precipitation was the objective of our study, and therefore should be placed on the discussion section. However, we have added in the Introduction the occurrence of the three metals in wastewater, especially the industrial effluents.

**Comment #5:**

what kinds of wastewater have the combination of these metals and the contaminants.

**Response:**

It is known that metal ions are generally present in industrial wastewater. The Introduction has been revised to include this information.

**Original version**

**1. Introduction**

Wastewater contains a particularly high concentration of phosphate, potassium, and ammonia which may cause eutrophication of surface waters [1]. The phosphorus concentrations in the wastewater can be reduced through mass crystallization of sparing struvite  $[\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}]$  on a mechanical mixing equipment [2, 3]. In addition, the wastewater containing heavy metals [namely, copper (Cu), lead (Pb) and zinc (Zn)], could have a negative effect on health, environment, equipment, and the aesthetic quality of the water bodies. Correspondingly, traces of heavy metals contaminated wastewater must be treated prior to its release to the environment [4, 5].

Toxic heavy metals from inorganic effluent can be stabilized through conventional methods such as precipitation, cementation, sedimentation, filtration, coagulation, flotation, ion exchange and adsorption [6, 7, 8]. However, these methods often lead to incomplete treatment, high-energy consumptions, and still produce an output of toxic sludge or waste products [9]. In this way, the conventional precipitation of heavy metals to yield hydroxides have shown the ineffectiveness in metal immobilization, especially when their concentrations are low in the solution. Alternatively, heavy metals in the wastewater can be removed simultaneously from the wastewater with a recovery of potassium, phosphate and ammonia using the precipitation agents [e.g., KOH and Mg (OH)<sub>2</sub>], to produce struvite and/or struvite-K [KMgPO<sub>4</sub>·6H<sub>2</sub>O-(KMP)] [10]. Currently, the recovery of six heavy metals [Cu, Ni, Pb, Zn, Mn, Cr (III)] from solutions can be enhanced by utilizing the self-synthesized struvite under pH variations of 6.0 to 10.0. Results indicated that more than 95 % of those six heavy metals could be taken out from the solution by struvite precipitation [11], and the precipitating solid contains the highest quality of struvite (97 %) which could be potentially utilized as fertilizer [12].

Correspondingly, several kinds of reactor systems have been developed to facilitate the wastewater treatment for not only sustainable recovery of phosphorus but also the absorption of metals and organics into the surface of crystal struvite [5]. Consequently, the heterogeneous nucleation mechanism and crystal growth of struvite may be influenced by the presence of heavy metals. The exchange between ionized metals such as cadmium, zinc, and nickel with magnesium can occur in the crystal lattice of struvite [3]. Therefore, the interaction between metals and struvite during co-precipitation and adsorption have become a great concern. Knowledge of the heterogeneous nucleation in terms of the speed of nucleation and the crystal growth has received much attention for reducing the activation energy due to the catalytic effect of metal ion particles

[2]. The presence of heavy metals may deteriorate the morphology of the precipitates and, in certain cases, cause the precipitation of a less stable phase instead of struvite [13]. Microstructure and property evaluation results of selected heavy metals incorporated into the crystal lattice of struvite are required to control the quality of recovered products. The characterization methods are needed here to examine the struvite quality flushing out of the reactors. Mineralogy, morphology and final particle size are the key factors in assuring for phosphorus recovery efficiency [2].

Analysis of kinetics, phase compositions and morphology of precipitates from the synthetic wastewater in the presence of three metal ions (Cu, Pb, and Zn) was presented in this paper. Kinetics analysis of struvite precipitation was estimated in the presence of three metal ions, through the measurement of pH over times, hence providing information on the retardant effect of the tested metals. In particular, the influence of three metal ions on the mineralogy and morphology of the precipitating solid was examined. The precipitating solid was then subjected to mineralogical phase evaluation using XRPD Rietveld method, and morphology and elemental chemical analysis by the SEM/EDX method.

#### Revised version

Industrialization and urbanization cause a serious problem for the environment and especially the life forms due to excessive release of heavy metals into water sources. Heavy metal ions are known to be non biodegradable and may accumulate in living tissues. Among these eco-toxic metals are  $Pb^{2+}$ ,  $Cu^{2+}$ , and  $Zn^{2+}$ . Many industrial activities for the manufacture of batteries, ammunition, various metal products and ceramic discharge  $Pb^{2+}$  into the water bodies [Pehlivan et al, 2009]. Copper is used extensively in various industries including refineries, paper and pulp, fertilizer, mining, and electroplating [Zehra et al, 2015]. Meanwhile, industrial effluents from

pulp and paper mills, organic and inorganic chemical plants, petroleum and petrochemicals, steel work foundries, and steam generation power plants may contain  $Zn^{2+}$ [Ajmal et al, 2011].

**Comment #6:**

State at the end of the introduction the novelty of this study, what this article is adding to our current knowledge in this field.

**Response:**

At the end of the introduction we have added the following statement to show the novelty of the study.

“So far there is no report on the influence of three metal ions,  $Pb^{2+}$ ,  $Cu^{2+}$ , and  $Zn^{2+}$ . on struvite precipitation. Therefore, the objectives of this study were to examine the struvite precipitation under the individual influence of these metals from a kinetic and morphological point of view. This study could be useful for determining the struvite precipitation as a means of fouling prevention and mitigation in which the effluent contains heavy metal ions.”

**Comment #7:**

page 6 line 5: correct "30 OC"

**Response:**

Correction has been made as requested.

**Revised version:** 30°C.

**Comment #8:**

page 6 line 23: please explain "The powder samples were initially deposited by carbon"

**Response:**

Correction has been made as requested.

**Original version**

The powder samples were initially deposited by carbon

**Revised version**

Samples with different particle sizes were embedded in epoxy on a glass slide and then sputtered with gold for SEM analysis.



**Comment #9:**

page 7 line 19: What do you mean by "Subsequently, the pH change is insignificant at the conclusion of the precipitation"

**Response:**

The statement is to explain that toward the end of the precipitation experiment, the pH of the solution remains almost constant.

**Original version**

Subsequently, the pH change is insignificant at the conclusion of the precipitation"

**Revised version**

From about 60 to 90 min toward the end of the experiment, the pH level for the three metal ions tested was almost constant, indicating the completion of the precipitation.

**Comment #10:**

Section 3.1 is confusing.

In the results, the author did not explain how these metals and pH impact (mechanism) on struvite formation.

It seems to me that only section 3.1 addresses the results of this study (experimental work),

**Response**

we agree with Reviewer #1. Section 3.1 together with section 3.2 has been re-written.

**Original version**

**3.1 Effect of pH on metal absorption in struvite crystal**

The time profiles of pH in the solution in the presence of different concentrations of Cu are shown in Fig. 1a. In the absence of Cu, a rapid reduction of the pH was observed during the first 10 minutes corresponding to the removal of magnesium from solution. Accordingly, the pH reduction was caused by struvite precipitation. Subsequently, the pH change is insignificant at the

conclusion of the precipitation. With increasing concentration of Cu (1-100 ppm), pH changes towards constant values as a consequence of the magnesium removal, of which was apparently at a lower rate than that in the absence of Cu. Similar trends were also observed in the time pH profile in the absence and presence of Pb and Zn (1-100 ppm) in the aqueous solution (Figures 1b and c). Apparently, Cu, Pb, and Zn were absorbed on the struvite surface leading to pH reduction. However, the insignificant pH change was observed and may be assigned to the lower metal uptake on the surface of struvite. Accordingly, the removal efficiency of the metal ions could be lower. Here initial pH plays an important role for adsorption of the metal ions. The significant removal efficiency of heavy metals was reported at pH 1.0-4.0 but the efficiency is improved slightly with pH > 4.0 [7]. In this way, ion exchange between Mg and the metal may occur in struvite surface.

### 3.2. Adsorption kinetics of metals

Struvite crystallization can be well represented in Eq.(1) [20]. Hence, the crystallization kinetics of struvite can be expressed as either the rate of reduction of  $Mg^{2+}$  or the increment rate of  $H^+$ . Correspondingly, the magnesium concentration  $[Mg^{2+}]$  left in the solution can be linked up to the observed pH values as reported in Eq. (2). Instead, the rate constant could have a substantial negative correlation between the quantities of metal ions dissolved in the solution [13]. Therefore, the adsorption kinetics of metal ions onto struvite surface were examined by the kinetic model of the first-order of Eq. (2).

Figure 2a shows the line of best fit for the typical experimental run of initial 10 minutes in the absence of metal ions. The struvite crystallization is well exemplified by the first-order kinetics model with a correlation coefficient ( $R^2$ ) of 0.9775. During 10 to 70 minutes, the reaction followed the first order rate kinetic model with  $R^2$  of 0.9549. Consequently, two forms of the kinetic may

follow the crystallization of struvite. The only single stage of the kinetic adsorption data in the presence of 1 ppm metal ions could be fitted to Eq. (2) with the best correlation coefficients (Figure 2b). The adsorption rate constant of Cu was calculated from the slopes of the straight lines, and the values were presented in Table 3. The experimental runs at 30 °C and in the absence of metals yields the lines corresponding to the first order rate equation, of which the calculated rate value of 4.334 h<sup>-1</sup> is in close agreement with results presented in the literature [21, 22, 23]. Moreover, the rate constant of Cu (1-100 ppm) present in the solution was all lower than that in the absence of Cu.

Further, the adsorption rate constants of Pb and Zn (1-100 ppm) were similarly calculated from the slopes of the straight lines, and the values were listed in Tables 4-5. Apparently, the rates of three metal ions decreased with increasing concentrations from 1 to 100 ppm. Moreover, the percentage of inhibition for struvite growth could be estimated from the calculation between the highest rates (0 ppm) and the lowest rates (100 ppm) of Cu, Pb and Zn as follows:  $[(4.344-1.512)/4.344] \times 100\% = 65.19\%$ ;  $[(4.344-1.338)/4.344]= 69.42\%$ ;  $[(4.344-1.056)/4.344=75.6\%]$ , respectively. The highest inhibition of growth rate was found for Zn. This rate-limiting step of the adsorption may be controlled by a physical adsorption process [8]. Correspondingly, the ions could be adsorbed onto the surface during developing crystal nuclei and thus prohibiting their outgrowth beyond the critical size for further growth [24]. This effect could be seen in the result of mass scale precipitated in the solution due to the presence of Zn (Table 5). However, the adsorption processes could be much slower than the dispersion processes, of which temperature is the primary factor to control the exchange of metal ions onto struvite [8]. Accordingly, the time-dependent adsorption kinetics study of metal ions onto struvite at different temperatures should be taken.

## Revised version

### 3.1 Effect of pH on metal adsorption in struvite crystal

The time profiles of pH in the solution in the presence of different concentrations of  $\text{Cu}^{2+}$  are shown in Fig. 1a. In the absence of  $\text{Cu}^{2+}$ , a rapid reduction of the pH was observed during the first 10 min corresponding to the removal of magnesium from solution. The pH reduction was caused by struvite precipitation. From about 60 to 90 min toward the end of the experiment, the pH level for the three metal ions tested was almost constant, indicating the completion of the precipitation. With increasing concentration of  $\text{Cu}^{2+}$  (1-100 ppm), the pH decreases gradually until stabilized because the crystallization process of struvite is inhibited by the addition of metal ions concentration. Similar trends were also observed in the time pH profile in the absence and presence of  $\text{Pb}^{2+}$  and  $\text{Zn}^{2+}$  (1-100 ppm) in the aqueous solution (Figures 1b and c). It was postulated that the metal ions were adsorbed onto the surface of the crystals and hence retarded the growth. Accordingly, the removal efficiency of the metal ions could be lower. Here initial pH plays an important role for adsorption of the metal ions. The significant removal efficiency of heavy metals was reported at pH 1.0-4.0 but the efficiency is improved slightly with pH > 4.0 [7]. In this way, ion exchange between  $\text{Mg}^{2+}$  and the heavy metal ions may occur in struvite surface.

### 3.2. Adsorption kinetics of metals

Struvite crystallization can be well represented by Eq.(1) [24]. Hence, kinetics of the struvite precipitation was evaluated through the change of pH of the precipitating solution. The kinetic evaluation demonstrated that the precipitation satisfactorily followed the first-order kinetic with respect to  $\text{Mg}^{2+}$  as reported in Eq. (2). It is known, the rate constant, could have a substantial

negative correlation between the quantities of metal ions dissolved in the solution [13]. Therefore, the adsorption kinetics of metal ions onto struvite surface were examined by the kinetic model of the first-order of Eq. (2). Figure 2a shows the line of best fit for the typical experimental run of initial 10 min in the absence of metal ions. The struvite crystallization is well exemplified by the first-order kinetics model with a high correlation coefficient ( $R^2$ ) of 0.9775 (Fig.2a-left). During 10 to 70 min, the reaction followed the first order rate kinetic model with  $R^2$  of 0.9549 (Fig.2a-right). Hence, two forms of the kinetic may follow the crystallization of struvite. Kinetic adsorption data (0-60 min) in the presence of 1 ppm metal ions could be fitted to Eq. (2) with the best correlation coefficients (Figure 2b). The adsorption rate constant of  $\text{Cu}^{2+}$  was calculated from the slopes of the straight lines, and the values were presented in Table 3. The experimental runs at 30 °C and in the absence of metals yields the lines corresponding to the first order rate equation, of which the calculated rate value of  $4.334 \text{ h}^{-1}$  is in close agreement with results presented in the literature [25, 26, 27]. Moreover, the rate constant of  $\text{Cu}^{2+}$  (1-100 ppm) present in the solution was all lower than that in the absence of  $\text{Cu}^{2+}$ .

Further, the adsorption rate constants of  $\text{Pb}^{2+}$  and  $\text{Zn}^{2+}$  (1-100 ppm) were similarly calculated from the slopes of the straight lines, and the values were listed in Tables 4-5. Obviously, the rates of the three metal ions tested decreased with increasing concentrations from 1 to 100 ppm. Moreover, the percentage of inhibition for struvite precipitation could be estimated from the calculation between the highest rates (0 ppm) and the lowest rates (100 ppm) of  $\text{Cu}^{2+}$ ,  $\text{Pb}^{2+}$  and  $\text{Zn}^{2+}$  as follows:  $[(4.344-1.512) / 4.344] \times 100\% = 65.19\%$ ;  $[(4.344-1.338) / 4.344] = 69.42\%$ ;  $[(4.344-1.056) / 4.344 = 75.6\%]$ , respectively. The maximum inhibition of growth rate was found for  $\text{Zn}^{2+}$ . This adsorption may be controlled by a physical adsorption process [8]. It is assumed, the ions could be adsorbed onto the surface during developing crystal nuclei and thus prohibiting their

outgrowth beyond the critical size for further growth [28]. This effect could be seen in the result of mass scale precipitated in the solution due to the presence of  $Zn^{2+}$  (Table 5).

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**Reviewer #2:**

**Comment #1:**

In this manuscript, the authors tested the effects of three kinds of metal ions ( $Cu^{2+}$ ,  $Pb^{2+}$ , and  $Zn^{2+}$ ) on crystal growth of struvite. They found that all the tested metal ions can inhibit struvite crystallization. However, I noticed that Muryanto and Bayuseno (2014) have investigated the influence of  $Cu^{2+}$  and  $Zn^{2+}$  on crystallization kinetics and morphology of struvite, and this manuscript did not make a noticeable progress. Moreover, there are several key questions as listed below that the authors should pay attention to and make them clear in the manuscript.

**Response**

The advancement from the previous study (Muryanto and Bayuseno (2014)) is shown by the findings on rate constants for the three metal ions investigated. In addition, the present study compared characterization of the precipitates with predicted precipitation using Visual Minteq.

**Comment #2:**

1. Page10 Line46-49: The mineral sylvite is unidentified by Visual Minteq, but appears in the products. I believe this results from the incorrect washing method. As the mass of the products is used to evaluate the inhibitory capacity of heavy metal ions, the authors should try to remove this impurity. Centrifugation is recommended.

**Response:**

The recovered precipitates were filtered and then washed lightly with distilled water, followed by air drying. It was assumed that only chlorides and alkali ions were removed from the surface of the precipitates during washing, leaving the struvite crystals mass unaffected. The mass of

sylvite in the recovered products was assumed to be negligible. This washing procedure was the same as reported in our previous studies [1, 2].

Refs:

1. D.S.Perwitasari, L.Edahwati, S.Sutiyono, S.Muryanto, J.Jamari and A.P.Bayuseno, Phosphate recovery through struvite-family crystals precipitated in the presence of citric acid: mineralogical phase and morphology evaluation, *Environ.Technol.* 38, 22 (2017) 2844-2855. <http://dx.doi.org/10.1080/09593330.2017.1278795>
2. A.P. Bayuseno and W.W.Schmahl, Improved understanding of the pozzolanic behaviour of MSWI fly ash with Ca(OH)<sub>2</sub> solution, *Int.J.Environ.Waste Manage.* 15 (2015) 39-66.

Comment #3:

2. Page10 Line49-56: The heavy metal-bearing minerals did not be detected by XRPD. This could explain that the metal content is under X-ray detection limit. However, XRD cannot detect the amorphous materials. Hence, FT-IR should also be conducted to examine the phase composition of the products.

Response:

We do not agree with Reviewer #2 on this statement. Although the metal content is not evident through XRPD (see Fig 3a), it is shown in EDX analysis (see Fig 5b). Hence an FT-IR analysis is of necessity not required.

Comment#4:

3. Page11 Line12-15: SEM/EDX analysis confirmed the existence of Cu<sup>2+</sup>, but cannot provide information about the surface composition of struvite. This is because the analysis depth of EDX can be 20 <math>\mu\text{m}</math>. To determine the element composition on struvite surface, XPS should be conducted.

Response:

We agree with Reviewer #2, that SEM/EDX cannot provide information about the surface composition of struvite. The statement: “Additionally, the resulting struvite structure may accomodate cation of Cu <sup>2+</sup> as is shown by the EDX spectrum (Fig.5b).” was changed.

Original version

“Additionally, the resulting struvite structure may accommodate cation of  $\text{Cu}^{2+}$  as is shown by the EDX spectrum (Fig.5b).”

**Revised version**

The EDX spectrum for struvite precipitated with the addition of one of the metal ions ( $\text{Cu}^{2+}$ ). (Fig.5b) shows traces of  $\text{Cu}^{2+}$ . This demonstrates that  $\text{Cu}^{2+}$  could either have been adsorbed onto the surface of the crystals or embedded into the crystal lattice.

**Comment #5:**

4. Page 13 Line 7-10: The authors reported that "All the particles observed in the solids have a prismatic crystal in nature". The morphology of sylvite is not identified in the manuscript.

**Response:**

We agree with Reviewer #2. The statement has been changed.

**Original version** Page 13 lines 7 – 15.

"All the particles observed in the solids have a prismatic crystal in nature. These kind of crystals could be influenced by the pH solution and the concentration of magnesium, ammonium, phosphate and potassium.”

**Revised version**

Through SEM, all the particles precipitated show morphological characteristics of struvite [Tao et al, 2016]. Other precipitates, e.g. sylvite is not visually detected due to their presence being in trace amounts only.

Ref.:

W.Tao, K.P.Fattah and M.P.Huchsermeier, Struvite recovery from anaerobically digested dairy manure: A review of application potential and hindrances. *J. Environ. Manage.* 169 (2016) 46-57.

**Comment #6:**

5. There are also some imprecise statements and grammatical errors in this manuscript. For example, "ion metals" should be "metal ions" (Page 1, Line 47). " $\text{KMgPO}_4 \cdot 6\text{H}_2\text{O}$ " should be " $\text{KMgPO}_4 \cdot 6\text{H}_2\text{O}$ " (Page 2, Line 44). "Mg and the metal" should be " $\text{Mg}^{2+}$  and the heavy metal ions" (Page 7, Line 46). So the manuscript should be carefully checked.



In short, there are some key points that need to be addressed by a major revision before acceptance for publication in Journal of Environmental Chemical Engineering.

**Response:**

We agree with Reviewer #2. All the incorrect statements and grammatical mistakes have been corrected.

**Original version** Page 1 lines 47

“ion metals”

**Revised version**

metal ions

**Original version** Page 2 lines 44

"KMgPO<sub>4</sub>\*6H<sub>2</sub>O"

**Revised version**

KMgPO<sub>4</sub>\*6H<sub>2</sub>O

**Original version** Page 7 lines 46

"Mg and the metal"

**Revised version**

Mg<sup>2+</sup> and the heavy metal ions

\*\*\*\*\*

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