

Reviewer 3

Open Review

(x) I would not like to sign my review report

() I would like to sign my review report

English language and style

() Extensive editing of English language and style required

(x) Moderate English changes required

() English language and style are fine/minor spell check required

() I don't feel qualified to judge about the English language and style

	Yes	Can be improved	Must be improved	Not applicable
Does the introduction provide sufficient background and include all relevant references?	()	(x)	()	()
Is the research design appropriate?	()	()	(x)	()
Are the methods adequately described?	()	()	(x)	()
Are the results clearly presented?	()	(x)	()	()
Are the conclusions supported by the results?	()	()	(x)	()
Comments and Suggestions for Authors				

Despite the authors have corrected a number of comments, there are some answers related to the insufficient interpretation of the X-ray diffractometry data, based on which conclusions of the work are formed.

- For table 1 “Measurement uncertainty – 5 %” is not correct because, for example, pH value has significantly lower measurement error. Moreover, excessive accuracy is written for most data. Replace, for example 255.00 with 255.
- “XRD diffractogram” is not correct. Please, use XRD pattern.
- On the XRD patterns (fig 3 and fig 4) corresponding to the Hazenite phase peaks absent. Fitting of the XRD pattern in the case of Struvite phase is of poor quality. Generally, for quantitatively phase analysis is require to show the Rp and Rwp factors, which are define the fitting quality.
- It is necessary to describe the procedure for calculating the quantitative phase composition. Which RIR or Rietveld method were used?
- For all phase fractions calculated written in the Table 4 values have excessive accuracy.

Submission Date

16 September 2021

Date of this review

07 Nov 2021 00:22:42

Answers:

In **Table 1** Measurement uncertainty – 5 % was meant up to 5 %.

The reviewer is right that for the pH value the measurement uncertainty is lower. In our case, the measurement uncertainty was 2.5 %, i.e. 8.12 ± 0.2 . In the text we corrected the pH value measurement uncertainty as 'up to 3 %'. This measurement uncertainty is always stated outside the tables to make the tables clearer. Based on the reviewer's recommendations, we removed the zeros after decimal point.

Higher quality XRD patterns were added and we renamed the 'diffractograms' to 'XRD patterns'.

XRD comments were added in the paper (Materials and Methods Section):

Powder X-ray diffraction analyses were carried out using a Bruker-AXS D8 Advance instrument with a $2\theta/\theta$ measurement geometry and the positionally sensitive detector LynxEye under the following conditions: radiation $\text{CuK}\alpha/\text{Ni}$ filter, current 40 kV, 40 mA voltage, step mode with a step of $0.014^\circ 2\theta$, and a total time of 15 seconds per step. Analysis of diffraction patterns was performed using the EVA software (Bruker-AXS) and the database PDF-2, release 2011 (International Centre for Diffraction Data). The Rietveld method using the TOPAS software, version 4.2 (Bruker) was applied to verify the qualitative analyses and for its quantification.

We added comments on XRD into the Results and Discussion Section.

For better readability of data in Figures 2 and 3, we exported the data into MS Excel and enlarged the curves to make the peaks more visible.

We added Rietveld reliable factors in the text. They are: R_{wp} 14.89, R_p 11.26 in molar ratio 1:1, and R_{wp} 14.89, R_p 11.26 in molar ratio 1:1.3. We believe that for the purposes of WWTP technologies, the XRD values are workable (we needed to confirm the abundance of struvite) – see studies by Meira et al. 2020, Heraldry et al. 2017) where the peaks are also asymmetrical. The peaks may be asymmetrical due to the occurrence of several struvite modifications, but this is not the subject of this study.

Meira, R.C.S.; Paz, S.A.P.; Corrêa, J.A.M. XRD-Rietveld analysis as a tool for monitoring struvite analog precipitation from wastewater: P, Mg, N and K recovery for fertilizer production. *Journal of Materials Research and Technology*. **2020**, Volume 9. <https://doi.org/10.1016/j.jmrt.2020.10.082>.

Heraldry, E.; Rahmawati, F.; Heryanto ; Putra, D.P. Application of quantitative XRD on the precipitation of struvite from Brine Water. *IOP Conf. Ser.: Mater. Sci. Eng.* **2017**, Volume 172. 012015. <https://doi.org/10.1088/1757-899X/172/1/012015>.