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29.6.2022

Statement on the dissertation entitled "STRUVITE PRECIPITATION FROM PROCESSED DAIRY WASTES" by Claver Numviyimana

The dissertation was prepared by Claver Numviyimana, who realized the research in WUST in a frame of the Maria Skłodowska-Curie Action H2020-ITN, project REFLOW "Phosphorus recovery for Fertilizers from Dairy processing wastes", No 814258, October 2019 - September 2022

The topic of the dissertation: STRUVITE PRECIPITATION FROM PROCESSED DAIRY WASTES, is highly topical due to the challenges related to increasing price of fertilizers caused by supply shortages. Therefore, more and more efforts are directed to alternative sources of fertilizers. Especially, nutrients are found in side streams such ash municipal and industrial sludge, agricultural waste and various ashes. I this thesis phosphorous was recovered from the dairy wastes as struvite

Dissertation consists of 124 pages, 7 chapters, 21 figures, and 16 tables. On the whole, the thesis is clear and logically written and contains all the necessary parts from the theory through methods and results to final conclusions and future research.

Chapter I Theoretical part, presents the literature review about the state-of-the-art methods of P recovery and struvite precipitation. All the important P recovery methods are explained and summarized and limitations related sludge utilization in agriculture discussed. Major drawbacks of commonly used chemical precipitation of P using iron salts is highlighted. Technologies used for P recovery as struvite are described in detailed and their drawbacks and benefits compared. In addition, review contains excellent analysis of the effect of conditions, such as presence of interfering ions and addition of Mg and P salts, on the struvite precipitation efficiency and quality of the final product. Removal methods of interfering ions are presented as well as effects of the treatment of wastes, with focus on thermochemical treatment, before struvite precipitation. Finally, existing full-scale processes for struvite production from aqueous phase, ash, and sludge are briefly summarized.

The summary of the literature review highlights the gaps of knowledge related to utilization of alum and iron loaded sludge along with the phosphorous recovery, which is also one of the main research questions of this study.

Chapter II Methodology, defines the experiments, instrumental, and modeling methods used in the study. Especially, existing data and literature was effectively used in the selection of the

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methods used. Experiments conducted are comprehensive. Studied dairy waste samples as well products (both liquid and solids) from thermochemical treatment and final struvite compositions are characterized with proper methods (ICP, SEM, XRD…) and all the important information presented. Treatment and synthesis methods are clear and well justified. Furthermore, final struvite products are tested as fertilizers which complements the research excellently.

In addition, several theoretical models were used in this thesis in order to verify the experimental data and optimize and design the experiments.

Chapter 4. Results and discussion, contains detailed description of the results obtained with illustrative figures and tables and comparison with the literature.

Chapter 4.1. Phosphorus recovery from cheese production wastewater presents the results from characterization of cheese production wastewater, the pH and foreign ions effects on struvite precipitation, and struvite precipitation with ammonium sorption onto zeolites. Presented results are comprehensive showing optimum pH and level of Ca:P ratio to maximize the struvite precipitation as well as minimize the effect of foreign ions. Removal of ammonium by zeolite sorbent helped to reach lower levels of P and ammonium in the precipitation effluent and inhibited release of Ca, Na and K into the reaction solution. Importantly, this precipitation process with sorption produced a fertilizer containing both struvite and ammonium loaded zeolite.

Chapter 4.2. Phosphorus recovery from thermochemically processed sludge presents results from the characterization of sludge and their thermo-chemical processing products, phosphorus species in the liquor, phosphorus extraction and iron removal efficiencies, precipitation and related DOE results, effect of dilution and residence time, characterization of recovered products from the liquor, and struvite precipitation from incinerated sludge ash.

Characterization of sludge, hydrochar, HTC liquor and ash revealed their contents of nutrients and other elements. Sludge contains high amount of P due to its precipitation with iron salt, but the most interesting residue was the HTC liquor that can be used for struvite precipitation. Its high iron content and complexity was emphasized and taken into account in further studies where iron was precipitated with oxalic acid. Utilization of oxalic acid decreased the amount of iron and calcium in liquor allowing production of better-quality fertilizer. In addition, the amount of oxalic acid needed was optimized.

Chapter 4.2 contains results of optimizing the struvite production from the HTC-liquor and comprehensive characterization i.e. elemental composition and structures of the product. Additionally, studies were made to dissolve incinerated sludge ash and precipitate struvite from

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the ash leachate. Combination of dairy sludge ash and magnesite for struvite precipitation yielded better nutrient profile in the final product. As summary, the main result obtained in this chapter is that after optimizing conditions fertilizers accepted on the markets could be produced.

Chapter 4.3. Qualification of phosphorus fertilizers presents the results from the In-vitro nutrient release assay and in-vivo nutrients use efficiencies. The best nutrients availability was obtained for the struvite precipitated after oxalic acid extraction. Comprehensive analysis was made using required efficiency parameters and results were compared to control sample. Especially, it was important to reduce the amount of iron that made efficiency parameters on plant worse.

Chapter 4.4. Business case and cost effectiveness presents the model of coupled dairy sludge hydrothermal processing and struvite precipitation and illustration of industrial plant set-up for iron recovery as oxalates and phosphorus recovery as struvite. Costs for the struvite production and P recovery are calculates and compared to previously reported results and products market prices. Recovering of iron and calcium oxalates makes the process even more advantageous and appealing.

On the whole results and discussion part in this thesis is excellently written with illustrative, high-quality figures and tables and all the results interpreted carefully and in justified manner.

Chapter 5. Conclusions, clearly presents the main conclusions obtained based on the conducted research. The main achievement was the optimized production of fertilizer from the dairy waste verified with complete characterization, qualification as fertilizer, and cost analysis.

Chapter 6. Recommendations for further research, indicates that the future research could be conducted for struvite precipitation from the EBPR sludge since it does not contain metals that should be removed thus reducing the amount of treatment steps. There is clearly possibility for scaling-up of the process since the costs estimated are not too high. In addition, the prices of the fertilizers are very high at the moment, and it is highly important to find alternative ways to produce them from the secondary resources, which in addition reduces the amount of nutrients released into the aqueous environment.

The author, Claver Numviyimana, has high four quality papers related to his thesis published in scientific refereed journals. In addition, he has participated in several conferences and awarded with three different scholarships. Therefore, he has already excellent merits to be graduated as PhD.

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Based on the notification presented in this statement, I state that the reviewed doctoral thesis of Claver Numviyiman, M.Sc., entitled 'STRUVITE PRECIPITATION FROM PROCESSED DAIRY WASTES' meets the requirements for doctoral dissertations specified in Art. 187 of the Act of July 20, 2018 Law on Higher Education and Science (Journal of Laws of 2018, item 1668, as amended). Therefore, I am applying to the Chemical Engineering Discipline Council at the Wroclaw University of Science and Technology for admission to the next stages of the procedure for the award of a doctoral degree in the field of engineering and technical sciences in the scientific discipline of chemical engineering."

Questions related:

1. Why raw incinerated sludge ash was not tested as fertilizer?
2. Could other adsorbents than zeolite be used in the ammonium removal?
3. Why column (dynamic) tests were not conducted in struvite precipitation

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