UNIVERSITY OF CHEMISTRY AND TECHNOLOGY PRAGUE
Faculty of Chemical Engineering
Department of Physical Chemistry

To:
Prof. Ing. Jaromíra Chýlková, CSc.
Head of the defence committee
University Pardubice
Faculty of Chemical Technology
Studenstká 573
532 10 Pardubice

Prague, November 9th, 2020

Dear Prof. Chýlková,

I am sending You my assessment of PhD thesis with a title *The use of nanofiltration for separation of heavy metals from wastewater* written by the doctoral candidate Edwin Wallace, MSc.

The topic of the dissertation is beneficial because the effective removal of toxic heavy metals from water using membrane technology has a significant impact on the environment. And, it is of great scientific interest and, potentially, of economic interest as well. In the attachment of this letter, there is a detailed assessment (Annex 1) and a list of comments and remarks and points for further discussion during the defence (Annex 2).

Sincerely

[redacted]

Karel Friess

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Annex No.1

Assessment of the PhD thesis

The use of nanofiltration for separation of heavy metals from wastewater

***Author**: Edwin Wallace, MSc.*
***Supervisor**: prof. Ing. Petr Mikulášek, CSc.*
***Institution**: The University of Pardubice, Faculty of Chemical Technology*

The work deals with the possibility of using nanofiltration (NF) membranes for the separation of toxic heavy metals from wastewater, where zinc sulfate ($ZnSO_4$), zinc nitrate ($Zn(NO_3)_2$), cobalt nitrate ($Co(NO_3)_2$) and nickel nitrate were used as model substances. ($Ni(NO_3)_2$). The separation performances of two commercially available thin-film polyamide NF membranes (AFC 30 and AFC 80) were studied and the structural parameters and charge of the membranes were determined. Two models describing the porous structure of the membrane under different operating conditions were. Furthermore, the influence of different transmembrane pressures, metal concentration in the feed, feed flow rate and pH of the used metal solutions were investigated.

The thesis in the presented form consists of a brief introduction (Chapter 1) and theoretical parts focused on studied topics, i.e. heavy metals (Chapter 2), pressure-driven membrane processes (Chapter 3), phenomena connected with the nanofiltration (NF) processes (Chapter 4) and methods used for characterization and modelling of NF membrane performance (Chapter 5). Chapter 6 summarizing the problems and objectives of the presented work. The experimental part comprises a detailed overview of used materials and methods (Chapter 7) and the summary of obtained results and their discussion (Chapter 8). The final chapter (Chapter 9) concludes the dissertation and highlighted the key findings. In total, the presented thesis comprises of 138 pages, including all text and graphical attachments.

The chosen topic of work of the candidate is attractive in its focus on the global issue associated with water contamination by heavy metal ions. Simultaneously, their effective removal from the wastewater via nanofiltration represents the innovative approach to current techniques. The work emphasizes the finding of relationships and connections between the structure and properties of NF membrane materials and the process parameters (feed concentration, transmembrane pressure, pH, cross-flow velocity). The combination of various experimental techniques and the theoretical models used made it possible to obtain microstructural parameters with the theoretical prediction of properties and direct experimental verification.

Despite the above mentioned, the work gives me an overall unbalanced impression and its scientific contribution can therefore be described as average only. This fact is signed to the chosen structure, from the total number of pages of the thesis (138) only 29 of them are dedicated to own new results and findings (Chapter 8). In total, the author cited 250 references of which only two are his own as the main author and one as the co-author. However, I consider the absence of clear statistical processing of results and determination of experimental errors (as well as their analysis) for the presented results to be more serious. From the texts, tables or figures, it is not fully clear how many times the experiments were performed, whether only once or more times. In the text, the author presents the vague statements only, e.g. page 82 - Pure water flux - "*Differences (experimental error) were below 5%.*" or page 83 Rejection of neutral solutes - "*The obtained results represented an average of two identical experiments and the relative standard deviation was up to 5%*". And, for experiments with metal ions, the text lacks any mention of possible determination errors. Last but not least, I feel negative that the text does not indicate in any way whether the experiments with metal solutions were performed separately for each relevant salt (which I assume) or a mixture thereof. In view of obtained findings on the mono- and bivalent anions rejection, the mixed metal salts experiments could demonstrate the real separation ability of NF membranes for wastewater treatment. The composition of wastewater can rarely be considered a one-salt-solution only.

Regardless of the above mentioned critical points, it is clear that the author has fulfilled the potential of the topic and the results of the work are interesting and beneficial and can contribute to the further progress in the field of membrane wastewater treatment.

Comments on the formal arrangement and language level of the dissertation

Formally and linguistically, the presented work is processed at a high level. It is written clearly and intelligibly, the graphing form of the used figures (adopted and own) is acceptable. However, it contains several unnecessary mistakes. The text of the thesis very often lacks the indefinite articles in given sentences, for instance in page 21 and 22: *In* (a) *similar manner* or *deals with* (a) *phenomenon* or *with* (a) *summary of key findings* or *with* (an) *atomic density* or *in* (a) *living organism*, where the missing articles are indicated in parentheses. Or, conversely, the definite articles reside when presenting all tables and figures (e.g. page 31 The Fig. 2 or The Table 2 on page 34). I would also point out several examples of the wrong use of the third-person singular forms (e.g. page 26 *as it improve the separation*, page 27 *This is due to it high abundance*, page 31 *Pressure driven membrane process are*, page 45 *research have been*, page 49 *The rejection of charged species have been explained*, page 55 *a rough surface are more prone*, page 89 *The application of these equations assume that*, page 90 *the model fit very well*, page 108 *the solution composition play a significant role*, page 111 *the principal factor that influence*, page 112 *The model of rejection of heavy metals agree*, page 113 *it increase the reflection*) and typos or

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misspelled words (e.g. page 29 *descibes*, page 33 *enhnaced*, page 33 *agregate*, page 43 *together with evident on the effects*, page 47 *in the marke*, page 54 *Peinermann* instead of Peinemann, page 55 *Van der Buggen* instead of Van der Bruggen, page 58 *neccessay*, page 58 *neutral and charge species*, page 58 *It should be emphasize that uptil now*, page 59 *model–based process stimulation tools*, page 69 *in terms image forces*, page 73 *COMSOL* vs *COSMOL*, page 81 *recoded*, page 81 *permeate is return to the feed tank*, page 94 *membrane is negatively charge*, page 99 *for both membrane*, page 101 *steric hindered*, page 106 *highly feed concentration*, page 113 *transport is mainly due the diffusion*, page 117 *thickess* and page 119 *polazied layer*. Some sentences, for example, page 74 *Conversely, many applications of NF such as desalination which has a high salt concentration and neglecting the effect of osmotic pressure cannot be possible*, are rather confusing.

However, the above-mentioned shortcomings do not reduce the overall quality of the submitted work.

Conclusion:

In conclusion, despite all the reservations I mentioned above, the dissertation of Edwin Wallace has the required standard. And it can be recommended for defence.

Prague, November 9th 2020

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Annex No. 2

The list of questions and points for discussion

1. In the text of Chapter 2, page 22, the author introduced various heavy metal ions those presences in wastewater is of critical concern (Cd, Cr, Ni, Cu, Pb, As, Co, Hg and Zn). Unfortunately, there is no evidence about the appropriate valence of metal ions because those could appear in water or environment in various valence forms, e.g Cr(II), Cr (III) and Cr (VI). Especially in the case of Cr, its effect is strongly dependent on the valence in which it enters the biologic systems. The Cr (III) form is considered in small amounts to be an essential part of everyday food. And, contrary vice, the Cr (VI) is known as a human carcinogen. Therefore, I would like to know according to which criteria the studied salts of heavy metals were selected and what types of wastewater the simulated conditions correspond to for the possible usage of NF membranes for their purification.

2. Different sets of neutral solutions were used for each membrane to determine structural parameters (Page 88). Why? By measuring both sets for both NF membranes, it would be possible to verify the evaluated parameters.

3. The efficiency of the membrane processes, or the filtration in general, is usually linked to the operating time. In the presented work I lack information about time-intensity or duration of individual experiments, respectively. Also, any example of experimental runs, e.g. a permeate flow over time for one salt at different membrane pressures etc., should be useful.

4. In Table 7, page 80, what is a difference between row 6 and row 9 with values 80 (for AFC 30) and 75 (AFC 80)?

5. In the List of symbols, there are different units for the permeability of pure water (L_p, with units m s^{-1} Pa^{-1}) and solute (P, m s^{-1}). Is it correct?

Prague, November 9th, 2020

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