

External examiner's review of a Master's thesis

Student's name and surname: Taha Jumaah
Degree programme: Environmental Engineering
Degree course:
Specialization
(if the degree course is divided into specializations):
Department: Department of Environmental Protection Engineering
Supervisor of the Master's thesis: doc. Ing. Jiří Pecha, Ph.D.
External examiner of the Master's thesis: prof. Ing. Petr Zámotný, Ph.D.
Academic year: 2022/23

Title of the Master's thesis:
Analytical monitoring of triglyceride ethanolysis reaction

Assessment of the Master's thesis using the ECTS grading scale:

Assessment criteria	Assessment according to the ECTS
1. Fulfilment of the assignment criteria	B – Very good
2. Level of quality of the formal aspects of the thesis, including the level of linguistic quality	C – Good
3. Amount, topicality and relevance of the literature sources consulted	B – Very good
4. Description of experiments and implementation methods	C – Good
5. Level of quality of processing of the results	A – Excellent
6. Interpretation of the results achieved and discussion thereof	B – Very good
7. Formulation of the conclusion of the thesis	B – Very good

Select the option the submitted thesis for defence and propose the following assessment:

B – Very good

Comments on the Master's thesis:

The theoretical assignment of the thesis was aimed at the critical review of the analytical techniques suitable for online monitoring of a transesterification process. The thesis approaches this task in a broad manner, describing the background of biofuels production, classification and listing the possible analytical methods with special focus on FT-IR and 2D COS. The critical aspect of this review is mostly limited on providing examples of successful FT-IR usage for analysis of methanolysis transesterification, discussing the suitability of the method for an online measurement. Also the potential benefit of 2D COS is discussed on the example of alternative processes. The detailed description of the 2D correlation maps interpretation is provided, but it refers on definitions of terms and symbols which are not explained or defined and thus this part is not self-explanatory.

The experimental part is rather brief. While the essential overview of the experimental procedures is clear, some experimental and instrumental details are not provided. Namely the scheme would be welcome in the description of the analytical method. From the description, it seems the analysis was not done fully online during the timeframe of the reaction but rather online during the timeframe of sample withdrawal.

The essential part of the results and discussion section comprise the obtained spectra at different reaction conditions, their description with pointing out the regions of interest and their 2D COS maps, identifying regions suitable for quantitative analysis. There is generally missing an information to which experimental conditions the particular map is assigned, but the general nature of spectra changes is clear and it is properly discussed and attributed to the reaction chemistry. Further the regression data between various composition characteristics and absorbances in specific bands are reported and evaluated. It is not clear, which method was used to obtain the composition data (there are hints it could be GC, but no details are provided). Further a calibration model is developed using 2D peak and validated on unseen data.

Unfortunately, no details about the nature of such data is provided.

The language quality of the thesis is mostly good with some interpunction and formatting imperfections. The quality of the artwork is mixed. While the 2D maps follow the general rules expected of an scientific publication, other figures are of poor technical quality and they are not mutually standardized.

In conclusion, the thesis achieved assigned objectives and it was successful in developing possible method of online monitoring of the transesterification process. On the bad side, the results are poorly documented and the results would benefit from more thorough description and discussion related to reaction-related effects of the developed method performance.

Questions to be asked by the external examiner of the Master's thesis:

1. The model reaction system was based on the rapeseed oil only. How much applicable the obtained results are for other fat systems?
2. The waste fats may contain different impurities. The spectral methods may be more sensitive to those impurities compared to chromatography. Could you comment on sensitivity of developed approach to impurities.
3. The random validation data are not specified. Were they selected as the random portion of the obtained bulk data or were they obtained of specific experiment (same or different conditions)?
4. The discussion around Fig. 27 compare the performance of raw and corrected data vs refractive index method. Would the conclusion change, if the comparison was made vs the GC data?

In Prague on 26. 05. 2023

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prof. Ing. Petr Zámotný, Ph.D.
Zámotný, Ph.D.
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Page 2/3

Signature of the external examiner of the Master's thesis