



EVALUATION of DOCTORAL DISSERTATION

Reviewer: M. Teresa Fernández Abedul, University of Oviedo

PhD candidate: Juan Santiago Hidalgo Viteri, University of Pannonia

PhD Dissertation Title: Electrochemical techniques of investigation – a tool for different applications

Recommendation: Recommended for acceptance

Date: 2025, August 25th

As reviewer of the PhD Thesis of Juan Santiago Hidalgo Viteri, I send my report made following the Guidelines for the Evaluation of Doctoral Dissertations of the Faculty of Engineering of the University of Pannonia.

I consider this Thesis **highly relevant to many disciplines**, as it focuses on applications of techniques based on electrochemical principles, of renewed and growing interest. The PhD Dissertation is considered **very formative** for the candidate as it covers significant areas such as drug delivery systems, corrosion and electroanalysis, all of which scientifically relevant. These techniques are rapidly advancing, with important improvements in different fields such as industrial, clinical or environmental. Thus, any advancement in these areas is significant and will likely have a societal impact in the future.

The candidate has carried out a **literature review** on all three parts of the dissertation topic. However, the way many references are commented is not the most appropriate for a PhD Dissertation. The inclusion of exhaustive protocols must be only made when required for experimental tasks but not for a PhD Dissertation, especially when they are not going to be used or are far from the topic. In some cases, the level of detail is excessive, including reagent suppliers or instrumentation models reported in the literature. While techniques and methodologies are discussed, some electrodes (e.g., boron-doped diamond electrode), or techniques (e.g., linear polarization or open-circuit potential measurements, as well as specific plots such as Nyquist plots in electrochemical impedance spectroscopy), are not included in the experimental part.



In science there are parts that must be highlighted and could be repeated, as happens in conclusions, but the extent of **repetition** is notable in the introduction and literature review. Also, **electrochemistry could have been used as a unifying theme**, connecting the different applications. Thus, comparison of electrochemical cells and processes could have been made as well as the inclusion of the main trends. Moreover, the starting of the sections must be clear. For example, “chemically modified electrodes” is a very broad topic, and it results difficult to address all the possibilities. Then, a classification with e.g., bio, nano... modifiers, would have been clarifying. That said, I want to highlight positively that the candidate offers at the end of the literature sections interesting discussions on advantages and disadvantages.

The candidate comments in all the experimental sections of the Dissertation the general objective and the specific ones. However, as commented before, a junction is required, also in the objective. A well-defined **general objective for the entire Dissertation**, that could be later specified in each of the parts, as is done appropriately by the candidate would improve it.

In terms of **research modernity**, it depends on the section. Glassy carbon electrodes are very traditional tools (the candidate includes a reference from the journal *Analytical Chemistry* from 1965) and more modern and useful electrodes such as screen-printed electrodes could be considered. Sustainable methodologies cannot be based in time consuming and complex procedures. Nevertheless, this electrode is valid from the point of view of research and methodologies employed seem reliable.

Regarding the **correctness of the methodology and processing of the data**, I consider that most of the work included in the PhD Dissertation has been already published in international scientific journals following a peer-review process. Thus, like what happens with the **novelty** (a requirement for publication) I should say nothing except to congratulate the candidate for the work done and the dissemination through articles and communications in conferences. Accordingly, I consider the **candidate's publication activity related to the dissertation** high, with a relevant number of articles in medium to high impact factor journals.



However, there are some general comments that I would like to do, most of them in the electroanalytical section:

1. Presentation of the scientific data and **significant figures**: The candidate includes the mean and the standard deviation, but the mean values are not rounded using the value of the standard deviation (e.g., mean = 378.00, SD = 30.37). It seems that the candidate uses always two decimals as criterion.
2. **Linearity**: The correlation coefficient indicates if linearity exists and, in some cases, this is very low. The way the equations are represented should have been revised (e.g., 31 and subsequent).
3. **Interpretation of raw data** (e.g., CV in Figure 31. In A, there is a redox process. In B there are more than two processes in bioF and CNTs modified electrodes). The choice of the electrode modified with bioF is not justified. The more intense process is affected by the preceding one and the intensity of the peak current does not seem to be higher than these containing just MWCNTs. Therefore, the conclusion seems not sound.
The assignation of peaks in CVs should be clearer: “anodic peak current II” can produce misunderstandings, when there is not any symbol included in the figure and later there is a IIa and IIc”
4. **Study design**: Once optimization is made, calibration curves and real sample analysis should be made with the best modified electrode. Precision studies should be made at a concentration level that is inside the concentration range.

The candidate **compares his results with bibliography**, mostly in this electroanalytical section, as happens with the sensitivity and limit of detection. However, the calculation is not clear and in some cases is commented that the best methodology is the one with higher limit of detection. Additionally, a limit of detection of 17 nM with a linear range starting in 250 nM is not reasonable. Also, a LOD of 190 nM, with a much higher sensitivity requires explanation.

The candidate has written consciously the **conclusions**, partial and general, which is considered positively, although in some cases the number of pages resemble a discussion rather than a conclusion section.

Regarding the **dissertation**, the candidate has structured and organized the dissertation according to a **numerical index**, which, in general, is appropriate, but



has some inconsistencies (e.g., indentation, or section levels as in e.g. 2.1.2.2 and 2.1.2.3, or 2.3 and 2.3.3, or 2.3 and 2.5 as well as 2.5 and 2.6). In which concerns the **figures**, the magnitude and units should have also been revised (e.g., current and peak current intensities are interchanged in some of them, as well as potential and peak potential). **Bibliographic references** should be revised and unified in format. Overall, the **writing style** is adequate, and the Dissertation reflects considerable effort.

Taking all this into account and considering the candidate's strong publication activity in peer-reviewed journals but emphasizing that a PhD Thesis is a different document that should unify all these publications, meeting the scientific standards in all the sections, **I recommend it for acceptance.**

Sincerely,

Prof. M. Teresa Fernández Abedul