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#### REVIEW

PhD dissertation of M.Sc. Ahmed Subrati, titled:

**„Electrochemically - derived graphite oxide: oxidation, functionalization,  
oxygen clustering, Ni - electrodeposition and characterization”**

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Doctoral dissertation of M.Sc. Ahmed Subrati, performed at the NanoBioMedical Center of the Adam Mickiewicz University, Poznań, is an extensive - over 250-page monograph. The work as a whole was divided into 5 independent chapters, in the first of which the author introduced the reader to the general scope of the work, its goals, as well as presented the reasons and justification for the need to take up the topic, and listed elements of scientific novelty. My first, general impression of the dissertation project, the proportion of the content constituting the literature review to the results of the doctoral student's own work, tables, figures, charts and photos is very positive. Chapters 2-5, concerning the works carried out as part of the doctoral dissertation, have been arranged classically, i.e. for each of them there is a literature part, an experimental part, a separate section with the results along with their discussion, conclusions and literature list. The specific layout of the work makes it easy to obtain complete information about the dissertation - both taken from the available literature and obtained in the course of the author's practical activities aimed at achieving the effects listed in the first chapter of the dissertation and described later, as well as about many other aspects related to the topic of theses, regarding the methodologies used to obtain materials

and methods of assessing their properties. In my opinion, this approach makes it easy for the reader to analyze the content of the dissertation.

In chapters 2-5, the proportion of content relating to literature review to experimental content varies. However, in general the literature part accounts for about 60% of the dissertation and the experimental part is about 40%. The literature review is up-to-date, which is confirmed by numerous items from the years 2021 and 2022, and the thorough one - the author of the dissertation cites nearly 200 source items - mostly scientific publications from journals with international access, mostly from the last two decades, but with many older, including even from the second half of the nineteenth century. The literature review provides key information on the experimental part of the work, both very specific and general, including fundamental and historical. In my opinion, the literature part of each chapter of the dissertation is a well prepared introduction to the topic of the dissertation and to the author's activities.

Both the literature review and other fragments of the dissertation clearly indicate the research aspect of the work, in which particular emphasis was placed on the preparation and modification of graphite oxide in terms of hydrogen storage, which was emphasized in the first parts of the dissertation. I believe that the subject undertaken by the author fits in with the direction of research intensively researched in recent years, which proves the topicality of the topic.

Numerous illustrations, tables, diagrams and other graphics are generally prepared in a thoughtful and careful manner, which facilitates the reception of the content, although I have a few critical remarks in this regard, which I will mention later in the review. As a result, the listed ones constitute a generally reader-friendly form of the dissertation. The objectives and scope of the work, indicated before the chapters containing research parts of the dissertation, are specified in detail and clearly, giving the reader a clear general view of its subject and scope, and, importantly, the elements of scientific novelty.

Analysis of the doctoral dissertation by Mr. Ahmed Subrati confirms its experimental character, as evidenced by a wide range of typically laboratory activities and a very wide spectrum of instrumental methods, including methods such as XPS, NMR, TPD, SEM / EDX, cyclic voltammetry (CV) and many others, used for performing an impressive number of measurements and analyzes of various properties of materials obtained and tested during the work. Both the above-mentioned and other methods used in the work have been selected reasonably, correctly and applied in a manner and to the extent that do not raise any objections.

In my opinion, some elements of the dissertation deserve special attention and high evaluation. I appreciate the author devoting a lot of attention to the process of enriching the reduced material obtained from graphene with nitrogen, and in particular the attempt to explain the mechanism of this process. I highly appreciate the use of a very extensive spectrum of advanced research methods by the doctoral student, which was very beneficial to the convincing interpretation of his observations regarding the conducted work and research on the properties of materials. There are various examples in the dissertation, including the analytical confirmation (by XRD and HR-TEM) of obtaining the material in the form of a hybrid material Ni nanoparticles / graphite oxide (chapter 5) and the experimental part of chapter 4, where the author used several research methods (DSC, SEM, XRD, XPS, Raman spectroscopy) and on basis of obtained results he presented a discussion on the reduction of graphite oxygen and the accompanying phenomena with the participation of oxygen. The discussion of the obtained results contained in the experimental parts of the dissertation is presented in a user-friendly way and with the indication of source literature, the combination of which with the results of Author's own work gave a very good effect in my opinion. I believe that the above-mentioned represent a convincing confirmation of Mr. Ahmed Subrati's scientific maturity.

The research described in the doctoral dissertation is part of the scientific discipline of chemical science, which is confirmed by the scope of research undertaken by the doctoral student, including research on chemical compounds, their transformations, reactions and properties resulting from their atomic structure. Research done by the author of the dissertation, including those concerning the processes of obtaining and testing materials for the purpose of storing energy carriers, have been an intensively recognized and developed research subject in recent years, practiced by numerous teams around the world. After reading the content of the dissertation, including the review of the literature from recent years, I have no doubts that the research on obtaining, characterizing and applying the materials being the subject of the dissertation contains a significant amount of scientific novelty, and their taking up by the PhD student is fully justified. The quantity and quality of the research results presented in the dissertation and their wide scope prove the great commitment of the Author in the implementation of the doctoral dissertation.

Realizing that the preparation of the reviewed doctoral dissertation is a big challenge, it is understandable that the Author did not avoid imperfections in it or doubtful provisions. These already appear in the summary, where the reduced graphite material was compared to

a known object, but doped with nitrogen, which in my opinion is difficult to imagine. Other shortcomings that caught my attention are:

- illegible drawings / charts or their elements, e.g. images in Fig. 2.33 (page 62) difficult to interpret due to their small size, illegible descriptions on the spectra in Fig. 3.14 page 91, some fragments illegible Fig. 5.20 on page 229,

- on page 60 The author has included a table with the results of the XPS spectrum analysis, but he did not include the spectrum itself, which results in a certain deficiency. I believe that presenting the reader with the source material (XPS spectrum) should be an integral part of his analysis,

- the conclusions to chapters 1-3 are, in my opinion, summaries rather than the author's thoughts on the research carried out. I believe that the presentation by the Author of his actual conclusions would certainly make the content of the dissertation more attractive,

- the concluding remarks (pp. 241-243) seem to cover the content that should be included in the chapter conclusions. I believe that this is an unnecessary element of the dissertation, requiring the reader to re-analyze the content presented in the earlier fragments of the dissertation, including the description of its outline (pp. 7-8).

During the study of the dissertation, there were also doubts, which I ask the author to clarify during the defense of the doctoral dissertation:

- what was the purpose of describing in detail (pp. 232, 233) how to use a particular software, since the reader does not have access to it?

- the summary shows that the storage of hydrogen in the obtained and tested materials is an important element of the work. Meanwhile, throughout the following chapters, the Author reports on storing energy in a different form. Why is the abstract of the work directly referring to the use of the tested materials for hydrogen storage, but the dissertation does not include the expected research results?

Despite the above comments, I believe that the doctoral dissertation by Mr. Ahmed Subrati is a work of a well-thought-out structure, with many values, mainly cognitive. After analyzing the dissertation, I can say that the collection of the experimental material required a lot of work and long-term research, mainly in the field of chemistry, but also in materials science. The content of the dissertation shows that the work was carried out consistently and covered several problems, including the preparation of materials, testing their properties and possible use. Based on the research results contained in the dissertation and the current state of knowledge about nanomaterials, in particular those being the subject of the dissertation, I

conclude that the presented material - the doctoral dissertation, broadens the knowledge in this field.

Taking into account the very rich experimental material contained in the work and the results of the research and their interpretation, I believe that the doctoral dissertation broadens the knowledge in the field of exact and natural sciences - in the discipline of chemical sciences. I also believe that the doctoral dissertation of Mr. Ahmed Subrati Fri "Electrochemically - derived graphite oxide: oxidation, functionalization, oxygen clustering, Ni - electrodeposition and characterization", is an original solution to a scientific problem and confirms his general, theoretical and practical knowledge in this scientific discipline, as well as the ability of the doctoral student to independently conduct scientific work. In view of the above, I believe that the doctoral dissertation entrusted to me for review meets all the requirements for doctoral dissertations in accordance with Art. 13 sec. 1 of the Act of March 14, 2003 on academic degrees and academic title as well as degrees and title in arts, as amended. I recommend to the Scientific Council of the Chemical Sciences Discipline AMU the admission of Mr. Ahmed Subrati to the next stages of the doctoral process.

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