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#### REVIEW

Doctoral thesis by M. Sc. Eng. Iwona Lupul,

"Removal of selected toxic compounds from water  
by adsorption on activated carbon from agricultural by-products"

"Removal of selected toxic compounds from water by adsorption on activated carbon  
from by-products of agricultural activity"

(Promoters: prof. Grażyna Gryglewicz, Wrocław University of Technology)  
prof. Dr. Jan Yperman, University of Hasselt)

The Ph.D. thesis submitted by Iwona Lupul, made at the Chemistry  
Department of the Wrocław University of Technology and Faculty of Science Hasselt  
Univeristy includes 150 pages. In addition to the text, the thesis includes a total of 71  
drawings, graphs, photographs and diagrams, and 38 tables. 235 papers were  
pointed in the dissertation as used in the doctoral studies.

The reviewed thesis can be categorized as classical . It is divided into a literature section, representing about 30% of the content, and dominating in the work experimental part, including a description of the methodology, a summary and discussion of the results obtained. Author prepared a brief summary, as well as factual introduction to the subject matter of the dissertation. The experimental part opens with a description of the main objectives and scope of the undertaken research. The final pages of the dissertation include conclusions, a list of literature, a list of scientific achievements of the candidate, and a summary written in both English and Polish.

The thesis is characterized by typical experimental character. In addition to widely described technological aspects of multivariate preparation and modifications of materials used in the work, much attention has been paid to the possibilities of their use for the adsorption of several selected organic compounds and hexavalent chromium ions, Cr (VI).

The author paid much attention to finding the relationship between the properties of the precursor and the porous structure of the product, pH and the surface chemistry of the sorbent and the adsorption of organic compounds and Cr (VI). In addition, the author has attempted to explain the mechanisms of adsorption of the above adsorbates.

The fact is that generally, adsorption using carbon adsorbents is not a new topic and is currently not a special novelty. However, it should be pointed that the topic is still current and research on new possibilities for the use of new precursors for this purpose, and the study of their properties, is often carried out by various research teams. For that reason, it can be said without hesitation that the research undertaken by Mrs Iwona Lupul fits into the constantly current field. I believe that attempting to obtain and study porous carbonaceous materials from plant-derived materials along with the potential for their use, although not a remarkable novelty, can certainly be valuable material for the author himself and also for others. Confirmation of the subject matter are the author's works in the form of scientific publications, first of all these published in highly quoted journals.



During the doctoral course, a wide range of modern instrumental techniques such as N<sub>2</sub> adsorption at 77K, SEM, FTIR, XPS, and TPD were used. Both these and other, not mentioned by me, and used in the work, were selected in a thoughtful manner and used for research optimally.

Of the 235 citations listed, the majority are scientific publications from journals of international circulation, mainly from 1990 to 2015. Other, including older and book titles, are definitely a minority. A careful study of the work allowed me to conclude that the literary part contained a number of material relevant to the subject matter of the thesis, directly and indirectly related to the experimental part of the work, and that the review of knowledge constitutes a well prepared introduction to further parts of the dissertation. I believe that the information obtained from the literature was aptly used in the experimental part and constituted an important material helpful in interpreting the obtained results.

The scope of the presented research results proves the great commitment of the student to work. Particular attention should be paid to taking some time consuming methods of material activation.

I appreciate the PhD student's attempt to use renewable raw materials to obtain activated carbons with extensive studies of adsorbent properties, including use for removal of hazardous substances from water. I highly appreciate the ability of a doctoral student in the selection of research techniques and interpretation of the results obtained. I also find it important to propose mechanisms of adsorption of selected adsorbates on porous materials obtained and studies on the effect of properties of raw materials on product properties. The graphic design of the dissertation is impeccable in my opinion, although in the content of the dissertation you can find elements that raise some doubts or insufficient information, but they do not disqualify the work as a whole.

My doubts are raised by the selection of adsorbates used in the studies. The author already in the introduction indicates that the work deals with removal of contaminants, including toxic ones. I have no doubt about selection of the hexavalent chromium for sorption studies. However, other species, i.e. Congo Red dye and the atrazine that is a known herbicide, are synthetic compounds that have been

discontinued or forbidden years ago for different reasons (sustainability, environmental impact). The question here is what led the doctoral student to choose these organic adsorbates, since they are practically absent in natural waters and sewages. Please students to explain and clarify this issue while defending the work and to indicate the significant benefits of the research conducted using the aforementioned compounds.

I also believe that some of the results obtained by the Ph.D. student are already known, as exemplified by the pH effect on the sorption of Cr (VI) ions. My doubts are also related to the mechanism of adsorption of cyanocobalamin indicated by the author, in my opinion insufficiently supported by research. I would like to hear about this issue during defense of the thesis, with an indication of what may be confirm the development of hydrogen bonds between the amine groups of the dye and the surface groups of the activated carbon.

In my opinion, in spite of some shortcomings, Dr. Iwona Lupul's dissertation is a work of considerable cognitive and application value. After reviewing the content of the work I can say that the accumulation of rich experimental material required much of work and long-term research. The material contained in the dissertation shows that the work was carried out consistently and covered a number of stages, including material preparation, properties testing and the possibility of using as a material for the purification of water streams from organic impurities and heavy metal ions. Given the results of the study included in the work, and the current state of knowledge related to research that included the work, I can say that the research presented is broadening the knowledge in this field.

In my opinion, Dr. Iwona Lupul's dissertation to sufficient extent is the original solution to the scientific problem and confirms general theoretical and practical knowledge of chemical technology and the ability to conduct independent research by the candidate. This is the basis for concluding that the thesis entrusted to me for review satisfies the requirements for doctoral dissertations in accordance with Art. 12 of the Law of 14 March 2003 on academic degrees and academic title, and grades and titles in the field of arts, with later changes. Therefore, I recommend to the Faculty of

Chemistry Faculty of Wrocław University of Technology, to admit MSc Eng. Iwona  
Lupul to further phases of the doctoral dissertation.

Szczecin, August 31, 2017

A handwritten signature in blue ink, appearing to be 'Jacek Przepiórski', written in a cursive style.

Jacek Przepiórski