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REVIEW OF DISSERTATION THESIS

Dissertation Thesis: Carbon - modified plastic materials for food packaging

Study Programme: P2301 Mechanical Engineering

Study Branch: Materials Engineering **Author: Ing.** Michal Marek Szczypinski

University: Technical University of Liberec, CZ

Faculty: Faculty of Mechanical Engineering

Supervisor: prof. RNDr. Stanislaw Mitura, DrSc.

Department: Department of Material Science **Reviewer:** Prof. Ing. Otakar Bokůvka, PhD.

Reviewers Workplace: University of Žilina, FME, DME, Žilina, Slovakia

Topicality of the theme: The theme of dissertation thesis is very topical. The process from the manufacturer through the retail chains to the customer often leads to a deterioration of the useful properties of food products. The reason is often the use of unsuitable packaging materials especially when storing products. This fact is undesirable from an economic and ecological point of view. There is a waste of food vs. a growing human population and a gradual loss of natural resources. One of the possibilities to increase the shelf life of food products is development of nanodiamond / polymer coatings on plastic packaging films with an antioxidant effect that inhibits the rancidity of fats. Targeted experimental research in this area is desirable from the point of view of materials engineering.

Choice of methods and equipment: The Ph.D. student, Ing. M. M. Szczypinski used suitable and appropriate experimental devices, equipment and methods (e. g. Differential scanning calorimetry, viscosity measurement, Raman spectroscopy, plasma treatment, surface tension spectral analysis, sessile drop technique, scratch – test, evaluation of antioxidant properties and so on.). The amount of the experimental work was very extensive. The student Ing. M. M. Szczypinski, showed strong experimental skills, deep critical analysis, ability to evaluate the experimental results and to draw correct and innovative conclusions.

Results, new technical knowledge, contribution in the field of science: The present thesis clearly contributes to add scientific knowledge. A method was developed to obtaining polymer coatings with the addition of nanodiamonds on the packaging film with excellent anti – rancid properties. The coatings are characterized by satisfactory adhesion on the polyethylene packaging film and due to the addition of an appropriate amount of nanodiamond, the correct coefficient of friction thanks to which they can be mechanically processed on the packaging lines and welded. These coatings are safe for health and environment.

PhD. thesis aim fulfilment: The aims of the dissertation thesis (Chapter 2, p. 9) have been completed successfully. Original results have been obtained (Chapter 4, p. 28 - 75)), final summary and conclusions are quoted in Chapter 5, p. 76 - 79.

Remarks and questions: The present doctoral thesis, submitted for final defense, has a very good level. The terminology is conformable with the field of study "Materials Engineering". The dissertation thesis is divided in a balanced and suitable way (introduction - one page, objectives of dissertation - one page, theoretical part – 17 pages, experimental part – 47 pages, summary and conclusion - 4 pages). The results were published in 12 scientific journals (7 times in the WoS). In view of this facts, the assumption that these scientific papers have undergone a demanding review process. The graphical level including the figures is very good. The references (in Attachment) quote 78 papers (71 after 2000 year), this fact is positive. There are not the serious objections to this dissertation thesis; only tiny "beauty" mistakes occurred, e.g., p. 1, stores [1]. vs. p. 12, species. [22 - 24] and further at work; p. 15, Fig. 3.5, is not quoted in the text; p. 17, (3.2), γ_{ls} vs. γ_{sl} is solid...[41]; p. 22, Fig. 3.10. Figure 3.10 shows; p. 25, Fig. 3.13 and List of Figures, p. 26; p. 36, shown Table 4.5...in Table 4.5; p. 55, Fig. 14.5?

The questions which can help in the next scientific work:

- you can specify, Ing. Michal Marek Szczypinski (cover of Thesis), mgr inž Michal Szczypinski ("page" 3, 4) and "List of author publications", Szczypinski, M. [2, 5, 7, 9, 10] vs. Szczypinski, M.M.?
- when using these coatings, you can determine the expected shelf life of the products?
- what the costs are in comparison with the packaging technology used so far?
- your opinion on the recyclability?
- the dissertation was part of a specific scientific project?

Conclusion: The Dissertation Thesis entitled "Carbon – modified plastic materials for food packaging" submitted for defense by Ing. Michal Marek Szczypinski, meets all requirements. I recommend this thesis for defence and after successful defence I recommend graduation to Ph.D. (Philosophiae Doctor) degree to Ing. Michal Marek Szczypinski, in the Study Branch "Materials Engineering".

Žilina, March 26, 2021

Prof. Ing. Otakar Bokůvka, PhD.

reviewer

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REVIEW OF DISSERTATION THESIS

Carbon- modified plastic materials for food packaging

mgr inż. Michal Marek Szczypinski

Thesis Supervisor: prof. RNDr. Stanislaw Mitura, DrSc

The theme of dissertation thesis focuses on the development of nanodiamond/polymer coatings on plastic packaging films with an antioxidant effect that inhibits the rancidity of fats. Bearing in mind problem related to wasting of food because a shelf life was passed, the presented work should have big impact on improved property of commercial packaging materials, especially for storing food products, and finally on economic and ecological aspect of our daily life.

In my opinion the original contribution of thesis in the science is showing of possibility of formation of polymer coatings with the addition of nanodiamonds on the packaging film with excellent anti-rancidity properties.

While reading the work, some shortcomings can be noticed. On page 7, M.Sc. Michal Marek Szczypinski has listed abbreviations, however, the abbreviations should be also defined at it's the first appearance in the text of work, which he does not always do. The second example, while reading the work, I did not find an explanation of what it is DLVO theory. Also, in the list of quoted literatures appears two times position: MITURA, Katarzyna, et al. Interactions between carbon coatings and tissue. Surface and Coatings Technology, 2006, 201.6: 2117-2123.

After reading the work, I have also a question:

- Coatings include nanodiamonds was deposited on commercial foil with imprint. Did the presence of this imprint on foil affect the results of differential scanning calorimeter (DSC) measurements?
- In thesis author present the results concerning measurements of friction coefficient.
 What was counter body during the tests?
- Does the author have a graph illustrating how the transmittance of the samples (foil, foil with coatings) changed at each stage of the experiment?

Concluding, the presented dissertation thesis entitled "Carbon -modified plastic materials for food packaging" fulfills the requirements for the Ph.D. degree, in the Study Branch "Materials Engineering".

Lodz, April 8, 2021

Posudek disertační práce

Title of Dissertation: Carbon-modified plastic materials for food packaging

Author: mgr inž Michal Szczypinski

Supervisor: prof. RNDr. Stanislaw Mitura, DrSc.

Opponent: Doc. Ing. Ludmila Kučerová, Ph.D.

The Thesis aims to develop nanodiamond-polymer coatings on plastic packaging films with an antioxidant effect that inhibits the rancidity of fats. This topic can be considered original, very interesting, and up-to-date, as an advancement in packaging materials would play a very important role for the whole food industry enhancing sustainability and having also a positive effect on human health and the environment.

The Thesis has a conventional layout, with an Introduction, Objectives, Theoretical part, and Experimental part ending with the Summary and conclusions. Considering the formal side of the work, the Thesis is very carefully written, without typos, and with minimal language problems. Graphs and images are of good quality, they explain the main ideas well and effectively visualize the experimental results.

The theoretical part deals with the overview of colloid and surface chemistry, properties of functionalized nanodiamonds, and tribology of packaging films, and contains a very good explanation of the basic principles necessary for the successful development of antioxidant coatings containing nanodiamond particles. Slightly missing is a review chapter describing coatings currently commercially used or developed by other researchers.

The main part of the Thesis is chapter 4 – The Experimental part. The experimental work is clearly and thoroughly described and it is easy to follow the progress of the work. Generally, suitable methods were used in the experimental work and they were applied properly. The experimental program is rather large and the application of various testing and analytical methods is well explained and justified. I appreciate the detailed Infracted spectral analysis in Chapter 4.4.1., where not only individual spectra were given, but also the comparative images (such as Fig. 4.12.) were provided. This really improved the possibility of comparison and evaluation of the results and the accompanying data description was very well wrapped up. On the other hand, I was missing representative images and row/measured data for several other experimental methods used in the work. Instead, in several cases, only the textual description of the method was provided, followed by the tables with already evaluated results. It would be more credible and support the repeatability of the experiments to offer also the actual documentation of how the measured data looked like and how exactly was their evaluation carried out. For example, the images of the drop for the Pedant drop method, or image of the drop with evaluated angles for Free surface energy and wettability test or graphs from tribological tests with marked Lc1, Lc3 could have been included and print-outs from evaluation software might have been used when necessary.

The objectives of the Thesis were stated in the second chapter and were fulfilled during the experimental work.

However, the objectives are rather general, without any threshold values which should be necessarily obtained for the practical application of the developed polymer coatings. This is one of the main throwbacks of the thesis, that except for a friction coefficient, obtained values are only compared

relatively within the experimental coatings, but they are not assessed against any reference values or discussed with relation with the coatings already commercially used.

The Dissertation thesis is also missing any discussion of the archived results and conclusions. The last chapter only sums up the experiments that were carried out and obtained results without any discussion.

In connection with the above-given comments, I have the following questions to the submitted Dissertation thesis:

- 1. Which of your coatings had the most promising set of properties for a practical application?
- 2. When doing this evaluation, to which of analysed property/properties would you give more weight and why?
- 3. Could you relate this best candidate to the properties of coatings currently used/required in the food packaging industry?

The publication activities of the postgraduate student could not be evaluated as the list of publications was not delivered with the thesis.

The postgraduate student proved to master appropriate skills and knowledge and proved his ability of independent research work in his field of expertise. The work **fulfils** the requirements of a dissertation thesis and therefore **I recommend** submitted a Dissertation thesis for the defence.

In Pilsen,

18.4.2021

Ludmila Kučerová