Referee’s report on PhD. thesis of

Hafiz Shahzad Maqsood

„Cellulose Micro Particles From Jute“

Professor Miroslav Černík

The presented thesis consists of 86 pages divided into 6 major chapters plus conclusions and 2 appendixes (research articles published). The thesis deals with jute fibres and their treatment for production of cellulose fibres and micro crystals. Many various techniques were investigated for jute fibres characterization after oxidation with ozone. Their properties are significantly changed after oxidation.

Chapter 1 (Introduction) is about morphology of lignocellulose fibres, techniques for separation of individual cellulose crystals and fibrils, including oxidation by ozone.

Chapter 2 (Aim and objectives) deals with extraction and characterization of jute micro/nano particles and reinforcement of biopolymers by cellulose particles.

Chapter 3 (Overview of the current state of the problem) shows different techniques for extraction of cellulose micro and nano structures.

Chapter 4 (Methods used and studied materials) deals with ozone treatment of jute fibres, and different views to changes the fibres after the oxidation process.

Chapter 5 (Summary of results achieved) is one of two main chapters of the thesis, where result are present. The jute fibres after oxidation change their colour (lightness value), FTIR spectrum and tenacity. The fibres were additionally treated by enzymatic hydrolysis to produce jute micro crystals. Three types of crystals were obtained (untreated jute micro crystals, chemical treated jute micro crystals and ozone treated jute micro crystals).

Chapter 6 (Evaluation of results and new findings) summarizes the determined results of the thesis. Untreated and ozone treated fibres were compared by SEM pictures, FTIR spectroscopy, mechanical properties, moisture absorption and whiteness index. Similarly jute micro crystals were compared. Their particle size distribution is significantly different, where after treatment significantly smaller particles (submicrone) were produced. The last part evaluates PLA composite films produced by addition of jute micro crystals.

Conclusions highlight results described in the previous chapter. Ozone treatment of jute fibres has significant influence to fibre properties. Tenacity drops significantly and dropping with increasing of oxidation time. Removal of non-cellulosic content (by oxidation) has influence to fibre diameter, fibres after treatment have smaller size. This treatment also leads to easier separation of crystals.

At the end, the author presents the paper on Reinforcement of Enzyme Hydrolyzed Longer Jute Micro Crystals in Polylactic Acid and Reinforcement of ozone pre-treated and enzyme hydrolyzed longer jute crystals in poly lactic acid composite films.
Referee remarks, question and conclusions
The thesis is logically divided into chapter, the content is explained illustratively, and all determined results are simply described. The author shows a story consists of three steps – material oxidation for production of fibres, enzymatic crystal separation and their incorporation into PLA matrix. In all steps the produced material is characterized by all available techniques and different types of treatment is compared.

Questions
1. Why ozone was selected for fibre oxidation? Have you information on various oxidation techniques for non-cellulose material removal from the fibres?
2. The three dimensional surface plots (p.36-39) are not illustrative enough for me. Could you summarize the results of these plots in more clear way?

Imperfections and recommendations
Language of the thesis is very good. I did not find many errors and mistypes. Only sometimes capital letters are used within the sentence without need.

Referee’s conclusion
The presented thesis is logic, has all necessary parts and show the author understand his work and he is able to put results logically into appropriate parts. The thesis shows one topic with different steps (oxidation enzymatic reaction, PLA matrix, characterization). The work significantly contributes to knowledge in the subject. There are no significant recommendations for next author’s work. The language is good and fully understandable.

The thesis is good and meets all criteria to be taken to the defence.

In Liberec (Czech R.) on February 6, 2017

Professor Miroslav Černík
Opponent's review

This opponent's review was elaborated based on Ing. Jana Drašarová, PhD. (dean of Faculty of Textile, Technical University in Liberec) assignment for review Ph.D. dissertation thesis (ref. no. TUL-16/4814/02019, dated 10.11.2016) of Hafiz Shahzad Maqsood, MSc. entitled "Cellulose Micro/Nano Particles from Jute". Tutor of the PhD. student was Ing. Jana Salačová, Ph.D. Above mentioned Ph.D. dissertation thesis deals with the topic of jute fibers application as a filler material in polymer composites. Several pretreatment techniques were used for fiber surface treatment, such as ozone and enzymatic. As a comparison an original jute fibers were used as well. There was found and described mechanism of the disintegration of the natural composite structure of the jute, its chemical modification and surface structure and topology changes. Obtained results were confirming earlier knowledge of the matter on similar products e.g. of the wood fibers bases etc. Author used many characterization techniques as well as the theoretical modeling. Majority of the results was already published in the highly respected scientific journals. The aims of the thesis were fulfilled and completed. I have found some formal errors in the text of the thesis, e.g. in quality of the figures (too small digits at the axes (e.g. Fig. 20, etc.)), text structuring etc. as well as have some questions on the applicant related to the topic studied.

1. It should be beneficial to characterize the fibers color change e.g. by Datacolor instrument and represent the results e.g. in CIE L*a*b scale or other method (page 42, Fig. 18). Can you explain why you have not done so?

2. There is missing formal kinetic modelling of the obtained dependences e.g. Fig. 19. Can you provide these data at the thesis defense and describe their statistical parameters?

3. Similar as in the point 2), there is missing a numerical as well as a statistical evaluation of data dependences in Figs. 22 and 23. Please deliver at the defense results of the ANOVA analysis.

4. With respect to all experiments performed, can you characterize the reproducibility of your measurements?

5. How do you explain negative effect of the jute filler on loading bearing capacity as shown in page 64? Please explain.

6. Explain differences obtained between applied theoretical models and experimental data of modulus vs concentration in Fig. 39?

Based on the thesis, I would like to hear answers from the applicant on the above questions.

Author has published majority of the data obtained during the PhD. studies in relatively high number of scientific papers in well-established journals (4 items published, 2 accepted, and 3 under review). Mr. Hafiz Shahzad Maqsood is the author of 7 conference proceedings.
Based on the latter mentioned facts and by the course of law (Higher Education Law No. 111/1998. Sb.) §47 I recommend to accept the PhD. dissertation thesis of Hafiz Shahzad Maqsood, M.Sc. for defense.

In Zlín, December 2, 2016

prof. Ing. Lubomír Lapčík, Ph.D.

Professor for materials science and engineering
Tomas Bata University in Zlín