

## Review of the PhD. Thesis

**PhD. candidate:** Md. Mostafizur Rahman

**Title of the thesis:** Displacement washing of spruce pulp with different delignification degree

The topic of the doctoral thesis focused on displacement washing of spruce kraft pulp is up-to-date due to the possibility of using the acquired theoretical knowledge in practice in increasing the efficiency of pulp washing. The displacement washing is currently the most widely used process, so it is very important for the practice to optimize it for each type of pulp. By optimizing the washing process of unbleached spruce kraft pulp, a reduction in production costs can be achieved due to increased production the thermal energy from spent pulping liquor burning and reducing the consumption of bleaching chemicals.

The scientific objective of the PhD. thesis was to investigate the influence of different parameters on washing efficiency of the unbleached spruce kraft pulp with the various delignification degree and comparison of the displacement washing of kraft pulp and sulphite pulp. In evaluating the chemical composition and geometrical properties of pulps, chemical and physical properties of spent pulping liquors and characteristics of pulp fibre beds were used relevant rigorous methods. The mathematical model of fluid flow through the pulp bed was described by a dispersion flow model with Péclet number, the wash flow in pores of the pulp by Reynolds number. The displacement washing was further characterized by washing efficiency, axial dispersion coefficient, the mean residence time of lignin in the pulp bed. The author presents in detail the original experimental data in a number of tables and figures. This arrangement increases the clarity of work. The results were mainly expressed graphically and in the form of correlation equations.

The contribution of the doctoral thesis are new knowledge about the influence of individual parameters on the displacement washing process of spruce kraft pulp. The obtained experimental data and developed mathematical model are a good starting point for optimizing the displacement washing process in pulp mills. Wash yield and the bed efficiency increased with Péclet and kappa number.

Interesting results were obtained when monitoring the effect of pulp bed consistency and wash liquid temperature on the washing efficiency of mixed softwood kraft pulp. The pulp bed consistency greatly affected the bed porosity and permeability, however, no notable effect was observed on the washing efficiency. The washing efficiency of kraft pulp has changed very

little with wash liquid temperature, but the hydrodynamic properties of the pulp bed and specific volume and surface of the pulp fibres changed with the temperature. Justification for the difference in the shape of displacement curves between kraft and sulfite pulp is logical and correct. The sulphite pulp bed has a higher average porosity compared with kraft pulp.

The results of the doctoral thesis are a valuable contribution to the further development of science and technology. The main benefit is in the experimental part of determining the impact of Péclet and kappa numbers on the wash yield. The doctoral thesis does not contain serious mistakes and thus it can be regarded as a scientific work in general.

### Questions

1. Why did you use unbleached spruce kraft pulp with kappa numbers from 18.1 to 50.1 at thesis?
2. Why did you use sulphite spruce pulp after oxygen delignification for comparison with unbleached spruce kraft pulp?

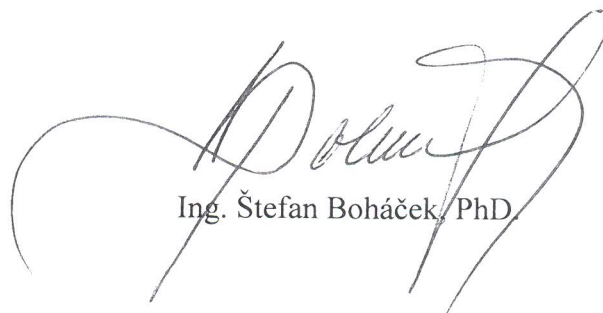
### Conclusion

Md. Mostafizur Rahman has proved to be capable of solving difficult research problems. His doctoral thesis presents new results that can be used in practice to increase the washing efficiency of the pulp and in the development of washing equipment.

In doctoral thesis Md. Mostafizur Rahman clearly demonstrated the ability of independent scientific work. The results of his doctoral thesis have been published in prestigious scientific international journals and presented on conferences. Proposed thesis achieved all important criteria and thus

### I recommend

proposed doctoral thesis to state Ph.D. for dissertation defense and after a successful defense award the doctoral degree.



Ing. Štefan Boháček, PhD.

In Bratislava, 14/05/2019

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**doc. Ing. Michal  
Jablonský, PhD.**

**May 7th, 2019**

**Opponent Review of Doctoral Thesis of Md. Mostafizur Rahman**

**“Displacement washing of spruce pulp with different delignification degree”**

Doctoral thesis is involved in the topical theme, being one of the key subjects of pulping and bleaching. Choice of the subject can be evaluated as unambiguously correct.

The submitted dissertation is elaborated in 153 pages, contains 82 figures, 28 tables, 20 tables in appendix and a total of 80 citations. Extent of thesis - from the point of number of pages - can be considered as adequate. It is well-arranged, with a logical structure, on a high formal and grammatical level (with minimal formal errors). Formal features of the thesis correspond with standard requirements.

I give the following statement to the presented Doctoral Thesis:

- a. Washing is a key task in the process of delignification and bleaching of pulp. It has a practical significance for the field of pulping, both from an economic and ecological point of view.
- b. To achieve the goals of the theses the methods were chosen well - spruce kraft pulps with a different lignin content (kappa from 18.1 to 50.1) have been prepared. Additionally also a sulphite pulp with a kappa number 12.5 have been analyzed. Specific hydraulic resistance, permeability, porosity and diameter were determined for characteristic of pulp fibre beds. The main evaluated parameters were the wash yield, bed efficiency of the pulp washing, axial dispersion coefficient for different Reynolds and Péclet number.



- c. The submitted doctoral thesis brought new knowledge and information about this topic, mostly:
- a detailed overview of the current state of knowledge in the field of washing have been documented. The list of references contains 80 citations.
  - a detailed description of washing process and its effect on the lignin removal and content of hexenuronic acid has been provided.
- d. For the benefit of the submitted doctoral thesis work to the development of science and technology I consider the following:
- a detail overview of the current state of washing processes,
  - determination of selected physical characteristics for the washing,
  - description of the washing process for spruce kraft pulp with different lignin content

Presented work fulfils the goal of obtaining up-to-date information on the state of art of knowledge in the area of washing for various delignification degrees. Secondary objectives are oriented on the relationship between kappa number and lignin, the effect of pulp consistency on bed characteristics, and there was investigated effect of temperature on washing efficiency. It can be stated that the presented work fulfilled the set goals in full range.

**Comments:**

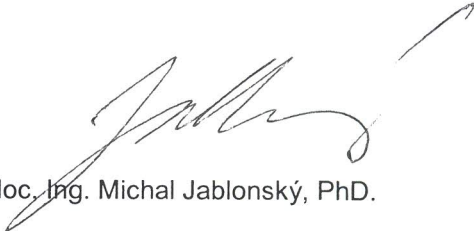
- p. 22 – absorbance and cross sectional area have the same abbreviation
- p.39: what is the meaning of expression “sulphate or kraft process”, please explain the difference between sulphate and kraft process if any.
- p. 92-95 the same ranges on the figures should be used
- p. 86 table 6.1 missing link to the reference for spruce pulp
- a brief information about hexenuronic acid should be added to the theoretical part

**Questions:**

1. How do you explain the increase of hexenuronic acid content after washing - p.125?
2. Fig. 6.61 p. 131. How do you explain the decrease of dispersion coefficient for washing at 50 °C?
3. Fig. 6.65 p. 133. How do you explain the decrease bed permeability for washing at 50 °C?
4. Fig. 6.69, 6.70, p. 137 Why do you compare the displacement washing and wash yield for pulps with different kappa numbers? (sulphite 12.5, kraft spruce pulp 18.1). Wouldn't be more beneficial to compare the samples with the same kappa number?

The work as a whole satisfies basic requirements set down for dissertations in the particular area. The author has demonstrated his abilities to focus on topical important subject and, in principle, to correctly address scientific problem and possible method of its solution. I recommend mentioned dissertation thesis for defence procedure, during which its author should clearly answer raised questions and should defend conclusions presented in his thesis before the committee.

In Bratislava, 07.05.2019



doc. Ing. Michal Jablonský, PhD.

## **Review of the doctoral thesis of Md. Mastafizur Rahman “Displacement washing of spruce pulp with different delignification degree”**

The doctoral thesis consists of nine chapters divided into subchapters. It contains 189 pages including the tables summarized functionally in the Appendix given on CD. The results and their discussion are included into subchapters in the sixth chapter. The structure of doctoral thesis corresponds to principles and claims on the structure of scientific thesis.

The thesis is written in English. The text is accompanied by figures that well document and illustrate the substantiality of the thesis. The word processing of the thesis is adequate. The author has studied and used appropriate number of bibliography sources cited in the thesis.

The thesis meets formal requirements on a good level.

Brown stock washing is a critical step in processing fibrous material for papermaking and related paper products. With increased capacity requirements in growing markets it is increasingly more important to maintain adequate washing efficiencies while optimizing capital investment in equipment.

Inefficient washing results in higher chemical and biochemical oxygen demand in waters of kraft pulp mills. If brown stock washing is carried out insufficiently, all areas of the pulp mill are negatively affected.

Hence, the topic of this dissertation work concerning the description of the displacement washing process of fiber pulp from the point of view of chemical engineering analysis is very actual and relevant.

The main objective of the doctoral thesis was to investigate the displacement washing efficiency for kraft spruce pulp delignified to various degrees. Besides, specific aims, namely the effect of wash liquid temperature and pulp bed consistency on the wash yield, as well as the displacement washing of sulphite pulp, were solved as well.

The objectives of the thesis described on page 32 may be considered fulfilled.

The appropriateness of the methodology to solve the set of declared aims complies with high requirements of pulp washing investigation. The scientific level and the adequacy of the used methodology are well characterized.

The dispersion flow model using one dimensionless criterion known as the Péclet number was used to describe the displacement washing process. Step function input signal has been widely used in the analysis of the flow through porous media such as the pulp fiber bed.

The equations for predicting the displacement wash yield should include all the properties of the pulp and wash liquor. Therefore, the distribution of fiber length along with fiber coarseness were measured using a Kajaani fiber length analyzer. The chemical analysis of spruce wood and pulps was performed according to the Tappi test methods. In order to describe the washing curve given as the time dependence of the tracer in the liquor stream leaving the pulp bed, the lignin concentration was measured spectrophotometrically.

The methods of research work were appropriate to the aims and hypothesis formulated in the thesis.

A lot of original results can be found in the thesis submitted by the author. With respect to supervisor's results published in the scientific journals earlier, I would like to emphasize mainly the relationships (equations (6.10) and (6.45) derived between the wash yield and/or bed efficiency, on the one hand, and the Péclet number and kappa number, on the other hand, evaluation of the effect of washing upon the kappa number, residual lignin and hexenuronic acid contents, and comparison of results obtained for sulphite pulp washing with those for kraft spruce pulp.

The results obtained are of benefit to existing knowledge in the research of pulp washing, as well as to pulp mills producing kraft and sulphite pulps.

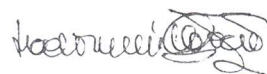
#### Comments

- (i) In my opinion, the chemical analysis of wood and pulp was mentioned too in detail with respect to accessibility of the Tappi Test Methods describing most of procedures. On the contrary, the author might pay more attention to the treatment and discussion of the results.
- (ii) I think that the influence of the pulp bed consistency on the hydraulic resistance, as well as the specific resistance of the pulp bed could be illustrated in the sub-chapter 6.9.
- (iii) It can be expected that due to the character of the pulp fiber bed, the wash water temperature will not have an effect on the washing efficiency, just because the displacement is the dominant mechanism. But the temperature can affect the rate of leaching. I believe the author could also examine at least the kappa number of the pulp fibers washed at different temperatures.



In my opinion, Mostafizur Rahman has demonstrated his creative capabilities and his work meet all the requirements for the dissertation in the given field. His doctoral thesis presents new results in the area of displacement washing of pulp. Therefore, I recommend the thesis submitted by Mostafizur Rahman for the defense.

Pardubice, May 09, 2019



Ing. Vladimír Špaček, CSc.