REVIEW

on PhD thesis for obtaining the educational and scientific degree "doctor", in the field of higher education: 5. "Technical sciences", professional field 5.13. "General Engineering", scientific specialty "Technology, mechanization and automation of the woodworking and furniture industry".

Author of the PhD thesis:
Yasmina Georges Khalaf, an external PhD student at the department of "Mechanical Wood Technology" at LTU, Sofia.

Dissertation topic:
"Utilization of Lignocellulosic Agricultural Residues for Obtaining Multifunctional Composite Materials" with scientific advisor prof. Dr. Julia Mihajlova from the Department of "Mechanical Wood Technology", Faculty of Forest Industry at LTU - Sofia.

Reviewer:
Prof. Dr. Ivo Vladimirov Valchev, UCTM - Sofia, 1756, St. Blvd. Kliment Ohridski 8, department of "Pulp, paper and printing arts", scientific area 5. "Technical sciences", professional field 5.10. "Chemical technologies", scientific specialty "Technology, mechanization and automation of the pulp and paper industry". The review was prepared in response to order № ЗПС-405 20.06.2025.

1. Brief introduction of the PhD student

Yasmina Georges Khalaf was born on 17.09.1993 in Lebanon. She was graduated master's degree of Physical Chemistry of Materials at Lebanese University, Beirut, in 2016.

Yasmina Khalaf was enrolled as an external PhD student at the department of "Mechanical Wood Technology" from 15.01.2020 in professional field 5.13 "General Engineering", scientific specialty "Technology, mechanization and automation of the pulp and paper industry". The PhD work was reviewed at an extended scientific council of the "Mechanical Wood Technology" department. The meeting was attended by five professors and two associate professors with competence in scientific specialty. With 9 votes "in favor" out of 9 voters, a unanimous decision was made that the dissertation work presented by Yasmina Khalaf meets the requirements of ZRASRB and the Regulations for its

implementation for opening a procedure for awarding the educational and scientific degree "doctor".

2. Relevance of the problem

The PhD thesis is in the relevant in recent years area of research into the possibilities for obtaining composite materials from agricultural waste. Residual plant raw materials are in significant quantities in Bulgaria and in this aspect the dissertation is extremely promising for our country. As a result of the research conducted, conclusions have been drawn that can serve as a basis for future research.

3. Scope of knowledge of the state of the problem and creative interpretation of the literature review

The chapter "State of Art" is spread over 50 pages, citing 285 literary sources, of which over 45% were published in the last 10 years. The overview is based on four subsections, based on which general conclusions are drawn with resolved and unresolved issues. The experience of obtaining composite materials from agricultural waste and the replacement of resins with bio-based binders is analyzed. The emphasis is on the unresolved problems and prospects of applying steam explosion treatment and fireproofing in the production of multifunctional composite materials.

4. Purpose, tasks, hypotheses and research methods. Corespondence of the chosen research methodology with the set goal and tasks of the dissertation.

The aim of the PhD thesis is clearly formulated and is aimed at investigating and establishing the possibilities for the utilization of Miscanthus x giganteus, olive waste, spent mushroom substrate, rice husks and textile waste with and without chitosan as a binder, for the development of low-density boards, particle boards and fireproof particle boards. To achieve the set goal, three main tasks have been formulated.

In the chapter "Materials and Methods" the initial lignocellulosic raw materials and chemical products are characterized. The processes of physical treatment of lignocellulosic materials, carrying out steam explosion treatment, the process of imparting fire resistance to particles and the preparation of a solution of chitosan binder are described. The conditions for obtaining ultra-light insulation boards, particle boards, as well as particle boards without a binder are presented.

The methodologies for characterizing the treated lignocellulosic materials and the performance of the resulting composite materials according to the requirements of EN standards are described in detail.

5. Visualization and presentation of the obtained results.

The chapter "Results and Discussion" of the conducted experimental research and analysis covers 52 pages and is based on six main subsections with 18 figures and 9 tables included.

6. Discussion of the results and the used literature.

The obtained comprehensive experimental results were analyzed and after the discussion, the conclusion was made that the preliminary steam explosion treatment of miscanthus particles significantly reduces the hemicellulose content and changes the length and width of the particles, which leads to a mass with a more homogeneous size and a higher content of lignin and cellulose.

Ultralight insulation boards were obtained from Miscanthus x giganteus, rice husks and textile waste as reinforcement with chitosan binder. These boards showed high porosity, good thermal insulation and mechanical properties and slightly higher than typical thermal conductivity than that of wood-based insulation materials according to EN 13171.

Eco-friendly boards with a density between 685 and 907 kg/m^3 have also been obtained from Miscanthus x giganteus, olive waste, spent mushroom substrate and textile waste with a chitosan binder. The boards based on defatted olive pomace and miscanthus (MOF) meet the standard requirements of EN 312 in terms of flexural strength MOR and IB, while those based on spent mushroom (SMSOF) show better compressive characteristics and meet the standard requirements of EN 312 for IB strength, making them suitable for general use in the dry state.

Binderless boards made from steam-exploded Miscanthus particles, with and without refractory treatment and cake, have been successfully developed, with Miscanthus (Mse) boards showing the best flexural properties. Moisture resistance is improved with the use of refractory Miscanthus particles, but decreases with and without olive pomace.

7. Contributions of the dissertation.

Contributions can be summarized in two groups: scientific-applied and applied which can be reduced to four.

Scientific-applied contributions

1. The optimal conditions of steam explosion treatment of miscanthus particles for the production of binderless boards have been established.

2. The possibility of obtaining ecological fireproofing boards from miscanthus particles after a 2-hour treatment with a combination of phytic acid and urea has been proven.

3. Ultralight insulation boards with medium insulation values were obtained using miscanthus, rice husks and textile waste in various formulations with chitosan as a biobased adhesive.

3. Experimental-statistical models have been derived for the influence of the content of recycled fibers and urea-formaldehyde resin on the physical and mechanical indicators of medium-density fiberboard, and the optimal ratio of the content of recycled fibers and urea-formaldehyde resin regarding the indicators of medium-density fiberboard has been established.

4. Ecological particle boards with a density between 685 and 907 kg.m-3 were obtained from miscanthus, olive waste, substrate from used mushrooms and textile waste in various formulations and chitosan adhesive.

5. Particle boards with a density of about 700 kg.m-3 have been successfully produced using miscanthus after steam explosion treatment, treated with phytic acid and urea solution or untreated, with or without olive pomace.

Applied contributions

1. It has been proven that the use of chitosan as a biobased adhesive in the production of composites improves their internal bonding and makes it competitive with other types of adhesives.

2. Ultralight insulation boards have been proven to meet the mechanical compression requirements of the relevant European standard EN 13171, and insulation boards containing small miscanthus particles are well suited for applications requiring improved load-bearing capacity.

3. It has been found that ecological particleboards based on chitosan, produced with miscanthus and defatted olive pomace, as well as those produced with spent mushroom substrate and defatted olive pomace, meet the requirements of the European standard EN 312 for general purpose boards in dry environments.

4. Fireproof particle boards have been successfully developed using phosphorus-grafted miscanthus particles without binders, showing significant fire and moisture resistance and retaining their structure when burned.

8. Assessment of the degree of personal participation of the PhD student in the contributions.

The design of the dissertation, the presentation and interpretation of the results, as well as the summary of the conclusions and contributions are an indicator of the personal participation of the PhD student.

9. Critical remarks and questions.

In connection with the clarification of the results obtained and further practical orientation of the research, I have the following questions:

During steam explosion treatment, a significant amount of hemicelluloses is released, which reduces the yield by 25–30%. What is proposed for the utilization of this hydrolysate?

The obtained after steam explosion treatment mass is as close to fibers as possible. With minimal beating, fibers for MDF production can be obtained, and shouldn't future research be in this direction (taking into account the significantly lower yield, of course)?

10. Published articles and citations.

The PhD student has 5 publications on the thesis and 47 citation are observed.
1) Khalaf, Y., Sonnier, R., Brosse, N. and El Hage, R., 2025. An extensive study of an eco-friendly fireproofing process of lignocellulosic Miscanthus x Giganteus particles and their application in flame retardant panels. Polymers, 17(2), pp. 241. https://doi.org/10.3390/polym17020241. Impact Factor: 5.0 (2025). Cite Score: 8.0; Quartile: Q1 (Chemistry) and Q1 (Polymers and Plastics).
2) Khalaf, Y., El Hage, P., Mansour, S., Brosse, N., Mihajlova, J.D., Bergeret, A., Lacroix, P. and El Hage, R., 2024. Eco-Friendly Chitosan Composites: Transforming Miscanthus, Mushroom, Textile and Olive Waste into Sustainable Materials. AppliedChem, 4, pp.302-319. https://doi.org/10.3390/appliedchem4030019. ISSN 2673-9623. Indexed in Scopus.
3) El Hage, R., Khalaf, Y., Abou Fayssal, S., Hammoud, M., El Sebaaly, Z. and Sassine, Y.N., 2021. Harvest and postharvest technologies. Mushrooms: Agaricus bisporus, pp.357-426, CABI. ISBNs :1800620411 and 9781800620414.
4) Khalaf, Y., El Hage, P., Mihajlova, J.D., Bergeret, A., Lacroix, P. and El Hage, R., 2021. Influence of agricultural fibres size on mechanical and insulating properties of innovative chitosan-based insulators. Construction and Building Materials, 287, p.123071.https://doi.org/10.1016/j.conbuildmat.2021.123071. Impact Factor: 7.4; Cite Score: 13.8; Quartile: Q1 (Building and Construction); Q1 (Civil and structural Engineering) and Q1 (Materials Science).
5) Khalaf, Y., Hajj, P., Mihaylova, J., Lacroix, P. and El Hage, R., 2021. Innovative fireproof insulating panels from agricultural waste. Innovations in Woodworking and Engineering Design, 19, pp. 24-28. ISSN 1314-6149.

11. Evaluation of publications on the dissertation work.

The PhD student has submitted 5 publications, of which the calculated points by group of indicator D are 36.4 with a minimum requirement of 30 points and exceed the minimum national requirements of Art. 2b. of the Low on the development of academic staff in Bulgaria and Art. 2a. of the Rules of the development of academic staff of Forestry University for the award the scientific degree "doctor" in scientific field 5. Technical sciences, PN 5.13. General

engineering. The abstract corresponds to the content of the PhD thesis, and the topic fully corresponds to the scientific specialty "Technology, mechanization and automation of the woodworking and furniture industry".

Conclusion

In conclusion I consider that the presented PhD thesis fully meets by volume, methodical level and publications in the scientific literature the requirements of the Rules of the development of academic staff of Forestry University for the award the scientific degree "doctor".

Based on the above and based primarily on the scientific-applied level of the PhD thesis and the results obtained, I recommend the members of the Scientific Jury to vote "positive" and to award to Yasmina Georges Khalaf the educational and scientific degree "Doctor" in professional field 5.13 "General engineering", scientific specialty "Wood science and wood materials" - (Technology, mechanization and automation of the woodworking and furniture industry).

Date: 04. 08. 2025 **Revi**

Prof. Ivo Valchev, PhD