STATEMENT

On a dissertation for the acquisition of 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 dissertation: **Yasmina Georges Khalaf**, part-time doctoral student at the Department of Mechanical Wood Technology, Faculty of Forest Industry, University of Forestry – Sofia.
The dissertation has been developed in collaboration with the Lebanese University and the University of Lorraine.

Dissertation topic: Utilization of Lignocellulosic Agricultural Residues for Obtaining Multifunctional Composite Materials

Member of the scientific jury: Prof. DSc Sanchi Konstantinova Nenkova, University of Chemical Technology and Metallurgy (UCTM)
5. Technical Sciences, 5.10. Chemical Technologies (Technology, Mechanization and Automation of Forest Chemical Industries),
appointed as a member of the scientific jury by Order № ZPS-263/09.05.2025 issued by the Rector of the University of Forestry – Sofia.

1. Relevance of the research problem

The topic of the dissertation – Utilization of lignocellulosic agricultural residues for obtaining multifunctional composite materials – is highly relevant. It addresses the utilization of certain agricultural plant and textile residues in the production of wood-based panels, either without a binder or using chitosan as a binding agent.

2. Degree of knowledge of the state of the problem and creative interpretation of the literature review

The dissertation is structured in accordance with academic standards and includes an introduction, literature review, methodology, experimental section, and conclusions. It comprises a total of 205 pages.

The literature review is presented over 49 pages, and a total of 303 bibliographic sources are cited throughout the dissertation.

A comprehensive overview is provided of plant-based raw materials used in the production of fiberboard panels. Traditional raw materials, as well as the main formaldehyde-based binding agents, are described in detail.

The author examines potential alternative sources for fiberboard production, including agricultural residues and waste, textile waste, spent mushroom substrate, olive waste, rice husks, and combinations thereof. Chitosan is proposed as a promising natural binder.

In addition, a method has been developed to enhance the fire-retardant treatment of lignocellulosic materials. The use of phosphorus is recommended as an environmentally friendly flame retardant for reducing the flammability of these materials.

Unresolved issues in the production of fiberboard panels using non-traditional raw materials have been clearly identified, including:

- The use of chitosan as a bio-based binder in the production of fiberboard panels from plant waste materials has not been previously studied.
- The mechanical and thermal insulation properties of *Miscanthus x giganteus*, textile waste, and rice husks have not been determined in relation to the production of eco-friendly, ultra-light insulation panels.
- The potential for utilizing olive pomace in combination with *Miscanthus x giganteus* particles for the development of new eco-friendly panels has not been explored.
- The effect of a novel fire-retardant treatment through phosphorus-based thermal grafting of phytic acid particles onto *Miscanthus x giganteus* fibers for improved fire resistance has not been investigated.
- The impact of steam explosion treatment on *Miscanthus x giganteus* particles has not been studied.
- No available studies exist on the production of fiberboard panels from *Miscanthus x giganteus* without the use of binding agents.
- The production of panels from mixed plant raw materials without any binder has not been examined.

At the end of the literature review, 11 resolved and 7 unresolved research questions are formulated, which serve as the foundation for defining the main research objective of the dissertation.

3. Aim, objectives, hypotheses and research methods. Relevance of the chosen research methodology to the stated aim and objectives of the dissertation

The objective of the dissertation is:

Utilization of many agricultural lignocellulosic residues and waste (*Miscanthus* x *giganteus*, olive waste, spent mushroom substrate, rice husks, and textile waste) in different formulations, with and without chitosan adhesive, for the conception of fully bio-sourced ultra-light insulating boards, particleboards, and fireproof binderless particleboards.

Key performance indicators of the developed panels — including density, moisture content, water absorption, thickness swelling, modulus of elasticity, mechanical strength, and fire resistance — are systematically evaluated. The applied methods enable a comprehensive assessment of the extent to which the research objective has been achieved.

4. Visualization and presentation of the results

To achieve the defined objective, the following experimental tasks have been undertaken and successfully completed:

1. The production and characterization of ultra-light insulation panels utilizing chitosan as a bio-based binder and incorporating particles derived from *Miscanthus* × *giganteus*, rice husks, and textile waste in varying proportions.
2. The development and analysis of particleboards composed of *Miscanthus* × *giganteus*, olive residues, spent mushroom substrate, and textile waste in diverse formulations, with chitosan applied as a binding agent.
3. The pre-treatment of *Miscanthus* × *giganteus* through steam explosion and phosphorus grafting (a combined application of phytic acid and urea) to modify the particle surface and enhance fire resistance prior to their inclusion in particleboard formulations, both with and without olive pomace.
4. The selection of optimal hydrothermal hydrolysis conditions aimed at improving the structural and performance characteristics of the fibers.

The experimental results have been comprehensively presented through **67 figures and 16 tables**, which contribute to the clear and systematic illustration of the research findings.

5. Discussion of results and the references used

This dissertation focuses on the development of sustainable composite materials composed entirely of bio-based components, primarily derived from agricultural by-products and textile waste. A comprehensive investigation has been conducted into the use of *Miscanthus* × *giganteus*, olive processing residues, spent mushroom substrate, rice husks, and textile waste in various formulations, with and without the addition of chitosan as a natural binder.

The impregnation of lignocellulosic fibers with a 20% phytic acid solution combined with a 10% urea solution has resulted in the formation of fire-retardant particles characterized by minimal phosphorus and nitrogen content.

Ultra-light insulation panels have been successfully produced from *Miscanthus* × *giganteus*, rice husks, and textile waste, employing chitosan as the adhesive component. Additionally, particleboards with a density of approximately 700 kg/m^3 have been developed without the use of any synthetic or bio-based binder.

The study convincingly demonstrates the potential of agricultural lignocellulosic residues and wastes as sustainable alternative raw materials for the production of fiberboards and particleboards.

A total of 303 scientific sources are cited in the dissertation.

6. Contributions of the thesis

The dissertation undoubtedly includes both scientific-applied and applied contributions, which can be summarized as follows:

Scientific-Applied Contributions

1. It has been proven that steam explosion treatment at 210 °C for 8 minutes after water impregnation is necessary to obtain Miscanthus particles with a high lignin content (36%) and uniform dimensions (7 mm in length and 0.4 mm in width), making them ideal for binderless particleboard production.
2. The feasibility of eco-friendly fireproofing *Miscanthus* × *giganteus* particles using a combination of phytic acid (20 wt %) and urea (10 wt %) was demonstrated. Optimal fireproofing was achieved with a 2-hour cooking time.
3. The production of ultra-light insulating boards of moderate insulation and densities between 350 and 400 $kg.m^{-3}$ using *Miscanthus* × *giganteus*, rice husks, and textile waste in various formulations with chitosan as a biobased adhesive has been successfully achieved.
4. Ecofriendly particleboards with densities between 685 and 907 $kg.m^{-3}$ have been successfully manufactured using *Miscanthus* × *giganteus*, olive waste, spent mushroom substrate and textile waste in different formulations and chitosan adhesive.
5. Binderless particleboards with densities around 700 $kg.m^{-3}$ have been successfully manufactured using steam exploded Miscanthus particles either treated with phytic acid and urea solution or untreated, with or without olive pomace.

Applied Contributions

1. It has been found that increasing the phytic acid levels and the duration of cooking during the fireproofing process leads to an increase in the phosphorus and nitrogen grafted on Miscanthus particles.
2. It has been proven that the use of chitosan as a biobased adhesive in the production of composites improves their internal bonding making it competitive with other adhesives.
3. It has been proven that the ultra-light insulating boards meet the mechanical compressive requirements of the relevant European standard EN 13171.
4. It has been found that due to their exceptional mechanical strength, insulating boards that contain small size miscanthus particles (Ms and Ms60T40 boards) are very suitable for applications requiring improved load-bearing capacities.
5. It has been found that the eco-friendly chitosan-based particleboards manufactured using Miscanthus and oil free pomace, as well as those produced with spent mushroom substrate and oil free pomace, meet the requirements of the European standard EN 312 and are suitable for general purposes in dry conditions.
6. Fireproof binderless particleboards displaying significant fire and humidity resistance and that retain their structure when burned have been successfully developed using phosphorus grafted Miscanthus particles.

7. Assesment of the degree of personal involvement of the PhD candidate in the contributions

Yasmina Georges Khalaf is a responsible and conscientious young researcher. She possesses strong theoretical knowledge, which has enabled her to successfully conduct laboratory investigations, as well as to effectively analyze and interpret the obtained results.

8. Critical comments and questions

No critical remarks.

9. Published articles and citations

Publications and Communications

Journal articles:

1. Khalaf, Y., Sonnier, R., Brosse, N., and El Hage, R., 2025. An extensive study of eco-friendly fireproofing process of lignocellulosic *Miscanthus* × *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). Cited: 1 time.
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. Cited: 4 times.
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. Cited: 11 times.
4. Khalaf, Y., El Hage, P., Mihajlova, J.D., Bergeret, A., Lacroix, P., and El Hage, R., 2021. Influence of agricultural fibers 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.
5. Khalaf, Y., Hajj, P., Mihaylova, J., Lacroix, P., and El Hage, R., 2021. Innovative fireproof insulating panels from agricultural wastes. *Innovations in Woodworking and Engineering Design*, 19, pp. 24-28.

Oral and Poster Presentations:

1. Khalaf, Y., El Hage, P., Mansour, S., Brosse, N., Bergeret, A., Lacroix, P., & El Hage, R. Eco-friendly chitosan-based composites from olive waste, *Miscanthus*, spent mushroom substrate, and recycled textile: meeting standards for sustainable wood alternatives. Presented at Eurofillers Polymer Blends, January 27-30, 2025; Lyon, France.

2. Khalaf, Y., El Hage, P., El Hage, R., Mihajlova, J.D., Brosse, N., Bergeret, A., & Lacroix, P. New formaldehyde-free particle panels made from agricultural wastes and chitosan. Presented at CYSENI, The 18th International Conference of Young Scientists on Energy and Natural Sciences Issues, May 24-27, 2022; Kaunas, Lithuania.
3. Khalaf, Y., Brosse, N., Sonnier, R., Mihajlova, J.D., & El Hage, R. Flame retardancy of Phytic acid/Urea grafted steam-exploded *Miscanthus*-based materials. Presented at ECOFRAM, The 3rd International Conference on ECO-friendly Flame Retardant Additives and Materials, May 17-18, 2022; Ales, France.
4. Khalaf, Y., Hajj, P., Mihaylova, Y., Lacroix, P., & El Hage, R. Innovative fireproof insulating panels from agricultural wastes. Poster presentation at INNO, The 10th Scientific and Technical Conference 'Innovations in Forest Industry and Engineering Design,' October 1-3, 2020; Sofia, Bulgaria.

10. Assessment of the publications on the dissertation: number, nature of the publications in which they are printed. Reflections in science – use and citation by other authors

The PhD student Yasmina Khalaf has 5 publications related to the dissertation, which have been cited a total of 54 times.

The submitted abstract objectively reflects the structure and content of the dissertation.

CONCLUSION

Based on the diverse research methods acquired and applied by the PhD student, the properly conducted experiments, and the well-founded evaluations and conclusions, I consider that the presented dissertation meets the requirements of the Law and the Regulations for the Development of Academic Staff at the University of Forestry. Therefore, I have grounds to assess it as **POSITIVE**.

I would like to propose to the esteemed Scientific Jury to also vote positively and award Yasmina Georges Khalaf, the educational and scientific degree "Doctor" in the scientific specialty "Technology, mechanization, and automation of the woodworking and furniture Industry".

Date: .....

Sofia

PREPARED BY

/Prof. DSc Sanchi Nenkova/