OPINION

on dissertation for obtaining the educational and scientific degree "Doctor" in the field of higher education 5. "Technical Sciences", professional field 5.13. "General Engineering", scientific speciality "Technology, Mechanization. and Automation of Woodworking and Furniture Industry"

Author of dissertation: Yasmina Georges Khalaf, MSc, part-time doctoral student at the Department of Mechanical Wood Technology, Faculty of Forest Industry, University of Forestry, Sofia, Bulgaria

Dissertation Topic: Utilization of Lignocellulosic Agricultural Residues for Obtaining Multifunctional Composite Materials, Scientific Supervisor: Assoc. Prof. Dr. Julia Mihajlova; scientific consultant: Prof. Dr. Roland El Hage

Reviewer: Prof. Dr. Petar Yordanov Antov, University of Forestry; professional field 5.13 "General Engineering", scientific speciality "Technology, Mechanization, and Automation of Woodworking and Furniture Industry" from the University of Forestry, Sofia, Faculty of Forest Industry, Department of Mechanical Wood Technology, appointed as a member of the scientific jury by Order No. ЗПС-405/20.06.2025 issued by the Rector of the University of Forestry.

1. Relevance of the problem

The development of sustainable multifunctional composite materials through the utilization of agricultural lignocellulosic residues and waste addresses key global challenges related to climate change, depletion of natural resources, environmental pollution, and the need for transition to a circular economy. Worldwide, more than 50 billion tonnes of such residues are generated annually; their composition and properties are comparable to those of wood, yet they often remain unused or are incinerated, leading to significant greenhouse gas emissions. The dissertation is in full alignment with the objectives of the European Green Deal and the strategies of the European Union aimed at reducing the use of formaldehyde-based resins through the implementation of bio-based binders with zero toxic emissions, such as chitosan. The scientific novelty lies in the integrated approach to the combined utilization of different types of agricultural residues (*Miscanthus x giganteus*, olive waste, rice husks, spent mushroom substrate, textile waste) for the production of multifunctional composite materials with and without the use of a binder. Particularly significant is the contribution in a previously unexplored area – the development of ultra-light bio-insulating boards and binder-free boards with enhanced fire resistance.

The results demonstrate an innovative combination of mechanical, thermal insulation, and fire-resistant properties of composite materials, achieved entirely from natural raw materials and environmentally friendly technologies. The obtained results have high application potential in the construction, furniture, and wood-processing industries and offer practical solutions for the efficient utilization of non-biodegradable agro-industrial wastes, thereby contributing to the reduction of the environmental footprint of materials produced.

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

The doctoral candidate demonstrates in-depth knowledge of the state of the art, reviewing a wide range of 285 up-to-date literature sources, including the latest developments in the utilization of agricultural lignocellulosic residues, the use of bio-based binders, and environmentally friendly methods for the preliminary treatment of raw materials. The literature review is systematically structured, with a clear distinction between issues that have already been resolved and those that remain open, and formulates specific scientific problems that the dissertation aims to address.

The review is distinguished by a high degree of analytical depth and creative interpretation, expressed through a critical analysis of existing technologies and the identification of opportunities for their improvement by combining different types of agricultural wastes and applying innovative treatments such as steam explosion and fire protection with phytic acid. The approach is integrative and original, substantiating the development of ultra-light bio-insulating boards and binder-free boards with improved mechanical, insulating, and fire-resistant properties, achieved entirely from natural raw materials.

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

The aim and objectives of the dissertation are clearly defined and focused on the utilization of agricultural lignocellulosic residues and waste (Miscanthus x giganteus, olive waste, rice husks, spent mushroom substrate, and textile waste) for the development of multifunctional composite materials – ultra-light bio-insulating boards, particleboards, and binderless fire-resistant boards. The formulated tasks are specific, logically structured, and encompass the entire research cycle – from the selection and preliminary treatment of raw materials, through the optimization of composition, to the investigation of the physical, mechanical, thermal insulation, and fire-resistant properties of the obtained composite materials.

The stated hypotheses are based on the assumption that combining different types of agricultural waste with bio-based binders (chitosan) and applying eco-friendly preliminary treatments, i.e., steam explosion, phosphorus-based fire protection with phytic acid and urea, will result in composites with improved performance characteristics comparable to or exceeding those of conventional wood-based materials.The selected methodology for conducting the experimental research is well chosen and applied in full accordance with the defined objectives and tasks. An integrated approach is used, including the mechanical preparation of raw materials and determination of their chemical composition, technological processes for particle modification (steam explosion, phosphorus-based fire protection), laboratory-scale production of multifunctional composite materials, and testing according to established international standards (EN, ASTM) to determine the physical-mechanical, thermal insulation, and fire-resistant properties of the resulting composites.

The applied research approach ensures the reliability, reproducibility, and high scientific value of the results, while allowing direct comparison with established technologies and materials in practice.

4. Visualization and presentation of the results

The results obtained are presented in a clear, systematic, and convincing manner, illustrated with a large number of tables, graphs, diagrams, and photographic materials that support understanding and analysis. The dissertation includes 16 tables and 67 figures, presenting both quantitative results and visual evidence of the morphological characteristics, technological processes, and behavior of the studied materials. The graphical layout is consistent and legible, which facilitates the perception of the information and the comparison of different experimental series.

The presentation of the results is accompanied by in-depth analysis and well-founded interpretations, with the doctoral candidate consistently comparing the obtained experimental values with available literature data. This critical comparison makes it possible to clearly highlight the achieved improvements and new scientific contributions in relation to already known solutions in the field.

Of particular note is the integration of visual materials that illustrate not only the results of the physical-mechanical, thermal insulation, and fire resistance tests, but also the stages of preliminary treatment of the raw materials and the laboratory-scale production of the composites.

5. Discussion of results and literature used

The discussion of the results in the dissertation is characterized by depth, logical consistency, and a critical approach. The doctoral candidate does not limit herself to merely presenting the experimental data but conducts a thorough analysis, tracing the relationships between the composition, structure, and technological processing of the raw materials and the resulting functional properties of the laboratory-produced composites. The conclusions are substantiated with clear arguments and supported by statistically processed data, which lends a high degree of credibility to the results.

The doctoral candidate demonstrates excellent scientific awareness, drawing skillful parallels between her own results and those available in the international literature, including the most recent publications in the field. The analysis includes a critical assessment of similarities and differences, as well as a reasoned explanation of the observed discrepancies related to the specific characteristics of the raw materials used and the technologies applied.

The literature used is up-to-date and includes 285 sources, among them publications in leading scientific journals, monographs, and proceedings from international scientific conferences. The selection is purposeful and covers the full thematic scope of the research – from the physicochemical characteristics of the agricultural lignocellulosic residues used, through current trends in the use of bio-based binders, to innovative, environmentally friendly technologies for enhancing the functionality of composites. The accurate citation and integration of literature data into the analysis highlight the author's high scientific culture.

6. Contributions of the thesis

The dissertation contains significant scientific-applied and applied contributions that expand the knowledge in the field of bio-based multifunctional composite materials and offer new technological solutions with high environmental and economic efficiency.

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 x 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 x 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 x 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. Assessment of the extent of the dissertator's personal involvement in the contributions

I am confident that the scientific-applied and applied contributions presented in the dissertation of MSc Yasmina Georges Khalaf have been achieved as a result of her personal work and research initiative. The doctoral candidate has demonstrated a high degree of research independence, having personally participated in all stages of the work – from formulating the aim, objectives, and hypotheses, through the preparation of raw materials and the development of technologies, to production, testing, analysis, and synthesis of the results. The contributions achieved are significant for the scientific field in which the competition was announced, namely Area of Higher Education 5. Technical Sciences, Professional Field 5.13. General Engineering, scientific specialty "Technology, Mechanization and Automation of the Woodworking and Furniture Industry," and have strong potential to be useful for the development of both science and industrial practice.

8. Critical comments and questions

In the dissertation and accompanying materials presented by the doctoral candidate, I did not find any significant omissions, inaccuracies, or errors in the interpretation of the obtained results. In view of the substantial volume of experimental work and the clearly demonstrated ability to conduct independent scientific research, I recommend that the doctoral candidate continue her academic development within the scholarly community. In addition, I consider it appropriate to publish a monograph abroad on the topic of the dissertation, which would further contribute to the wider recognition of MSc Yasmina Georges Khalaf's achievements in the international scientific community.

9. Published articles and citations

On the topic of the dissertation, the doctoral candidate is the author and co-author of five scientific publications, published in prestigious international journals indexed in Web of Science and Scopus, as well as in reputable specialized journals in the field. Among them are articles in journals with a high impact factor and strong citation metrics, including: *Polymers* (IF: 4.9, CiteScore: 9.7, Q1), *Construction and Building Materials* (IF: 8.0, CiteScore: 13.9, Q1), and *AppliedChem* (CiteScore: 2.9).

The doctoral candidate has also presented her research findings at four prestigious international scientific forums: *EuroFillers Polymer Blends* 2025 (Lyon, France), *CYSENI* 2022 (Kaunas, Lithuania), *ECOFRAM* 2022 (Alès, France), and *INNO* 2020 (Sofia, Bulgaria).

The publications related to the dissertation have already received 47 citations in prestigious international scientific journals, including high-impact factor publications and scholarly works by established researchers from leading universities and scientific institutions.

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

On the topic of the dissertation, the doctoral candidate has published five scientific articles in reputable international journals, peer-reviewed and indexed in the globally recognized databases Web of Science and Scopus. A significant portion of them have been published in high-impact journals, including those in the first and second quartiles (Q1 and Q2), which is indicative of the high quality and scientific significance of the conducted research. The publications are directly related to the main topics and results of the dissertation and reflect the key scientific-applied and applied contributions achieved.

The impact of these works within the scientific community is evident from the substantial number of citations recorded in authoritative international journals, which attests to the relevance of the research and to the interest of other authors in the results obtained. Data, approaches, and conclusions from the dissertation have been used in scientific publications dedicated to the development of environmentally friendly technologies for the production of bio-based composite materials. This fact is a clear indicator of the relevance and significance of the conducted research, as well as its influence in the scientific community.

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

Overall, the quantity and quality of the published works, together with the citations already obtained, demonstrate the doctoral candidate's strong scientific productivity and significant contribution to the researched field.

CONCLUSION:

On the basis of the different research methods learned and applied by the PhD student, the correctly derived experiments, the made generalizations and conclusions, I consider that the presented dissertation meets the requirements of the Law for the Development of the Academic Staff in the Republic of Bulgaria and the Regulations of the University of Forestry for its application, which gives me the reason to evaluate it as **POSITIVE**.

I take the liberty to propose that the esteemed scientific jury also vote in favour and award MSc Yasmina Georges Khalaf the educational and scientific degree of "Doctor" in the scientific speciality "Technology, Mechanization and Automation of the Woodworking and Furniture Industry."

06 August 2025
Sofia

Scientific jury member:
/Prof. Dr. Petar Antov/