## **REVIEW**

## of the official reviewer for the thesis work

of Bergaliyeva Saltanat Amangeldinovna on the topic «Standardization of Recycled Plastic Materials for Additive Manufacturing», provided for the degree of Doctor of Philosophy (PhD) in the educational program «8D07502 – Standardization and Certification (by industry)».

No	Criteria	Compliance with the criteria (one of the options must be marked)	Justification of the position of the official reviewer
1.	The topic of the thesis (as of the	1.1 Compliance with priority directions of science development or state programs:	
*	date of its approval) corresponds to the directions of development of science and/or state programs	1) The thesis was carried out within the framework of a project or target program funded from the state budget (specify the name and number of the project or program)  2) The thesis was carried out within the framework of another state program (specify the name of the program)  3) The thesis corresponds to the priority direction of science development, approved by the Higher Scientific and Technical Commission under the Government of the Republic of Kazakhstan (specify the direction)	The thesis work was conducted within the framework of the project "Improvement in resource efficiency and sustainability through the implementation of additive manufacturing methodologies for maintenance of facilities in the chemical industry". Funding: Fundación Campus Tecnológico. Participating entities: Universidad de Cádiz and Indorama Ventures Química SLU. Period from 1 January 2020 to 31 June 2021. Granted amount: 6058.48€.
2.	Importance for science	The work <u>makes</u> /does not make a significant contribution to science, and its importance is <u>well disclosed</u> /not disclosed	Coordinator: Sales Lérida, David.  The widespread of additive technologies into the production process is hindered by the lack of regulatory documents regulating the requirements for raw materials for 3D printing, especially those obtained from a secondary polymer. Therefore, the results obtained within the framework of this dissertation are valuable for the dissemination of additive technologies, as well as for the effective recycling of polymer waste. The importance of the topic of the dissertation is sufficiently fully disclosed in the section Courset state of the graphless.
3.	Principle of independence	Level of independence: 1) High; 2) Medium; 3) Low; 4) There is no independence	in the section Current state of the problem.  The level of independence of the doctoral student was confirmed by publications based on the materials of the dissertation work, in which the author is the first (main) author and the author for correspondence
4.	Principle of internal unity	<ul><li>4.1 Justification of the relevance of the thesis:</li><li>1) <u>Justified;</u></li></ul>	The relevance of the scientific data presented in the dissertation is justified. Nanocomposite materials based

		<ul> <li>2) Partially justified;</li> <li>3) Not justified</li> <li>4.2 The content of the thesis reflects the topic of the thesis:</li> <li>1) Reflects;</li> <li>2) Partially reflects;</li> <li>3) Does not reflect</li> </ul>	on pure and secondary polylactide and titanium dioxide nanoparticles were obtained, and their thermomechanical properties were standardized for further certification.  The content of the dissertation reflects the topic of the dissertation. The third, fourth and fifth sections present the thermomechanical properties of recycled and modified secondary polylactide for additive technologies. The sixth section presents the results of standardization of the material obtained in the fifth section based on polylactide debris.
		<ul><li>4.3 The purpose and the tasks correspond to the topic of the thesis:</li><li>1) correspond;</li><li>2) partially correspond;</li><li>3) do not correspond</li></ul>	The goals and objectives of the dissertation work correspond to the research topic, reflect the content and reveal the main aspects of the dissertation work.
*		<ul> <li>4.4 All sections and provisions of the thesis are logically interconnected:</li> <li>1) completely interconnected;</li> <li>2) the interconnection is partial;</li> <li>3) there is no interconnection</li> </ul>	The introduction, six main sections and conclusion presented in the dissertation are logically interrelated.
		<ul> <li>4.5 New solutions (principles, methods) proposed by the author are reasoned and evaluated in comparison with known solutions:</li> <li>1) there is a critical analysis;</li> <li>2) the analysis is partial;</li> <li>3) the analysis does not represent the one's own opinions, but quotes from other authors</li> </ul>	The results obtained by the author are substantiated and proved by means of a critical analysis conducted on the basis of ideas about the thermo-mechanical properties of samples and an up-to-date review of available literature data. The author uses standardized methods for studying the thermal and mechanical characteristics of materials.
5.	Principle of scientific novelty	<ul> <li>5.1 Are the scientific results and provisions new?</li> <li>1) completely new;</li> <li>2) partially new (25-75% are new);</li> <li>3) not new (less than 25% are new)</li> </ul>	The scientific results and provisions obtained in the framework of the dissertation work are new. It was found that 3D printed polylactide samples aged up to 1.5 years are suitable for mechanical recycling, and nanocomposites based on them with the addition of 7 % titanium dioxide nanoparticles and 18 % pure polylactide have thermomechanical properties comparable to a standard sample.
		<ul> <li>5.2 Are the conclusions of the thesis new?</li> <li>1) completely new;</li> <li>2) partially new (25-75% are new);</li> <li>3) not new (less than 25% are new)</li> </ul>	The conclusions of the dissertation are completely new and have not been previously obtained in other works. The conclusions are confirmed by the results of a detailed comparative analysis of experimental data.

		<ul> <li>5.3 Technical, technological, economic or management decisions are new and reasonable:</li> <li>1) completely new;</li> <li>2) partially new (25-75% are new);</li> <li>3) not new (less than 25% are new)</li> </ul>	Technical, technological, economic solutions are new and justified and can be used in the production and certification of nanocomposites based on secondary polylactide for 3D printing.
6.	Validity of the main conclusions	All the main conclusions are <u>based</u> /not based on scientifically sound evidence or well grounded (for qualitative research and areas of study in the arts and humanities)	All the main results of the dissertation work are based on scientifically significant evidence and independently conducted experimental studies obtained using standardized techniques and certified equipment.
7.	The main provisions for the defense	It is necessary to answer the following questions for each provision separately: 7.1 Is the provision proven?  1) proven; 2) rather proven; 3) rather not proven; 4) not proven	Statement #1. Standardized process of accelerated hydrothermal aging in standard of organization St JSC 002-2023 "Polylactide for additive manufacturing. Accelerated hydrothermal aging test" of 3D printed polylactide samples at 50 °C and 70 % humidity for 1344 hours results in a 33% reduction in tensile strength.  After 1.5 years of operation, the strength characteristics of 3D printed polylactide products deteriorate.
		7.2 Is it trivial? 1) yes; 2) no	The dynamics of polylactide aging are of interest to specialists in the field of materials science, recycling of polylactide waste, additive technologies and standardization.
		7.3 Is it new?  1) yes; 2) no	The result of the study is new, since the author has carried out a complex of scientific and analytical developments to create a methodology for conducting hydrothermal aging of 3D printed polylactide.
		7.4 Level for application: 1) narrow; 2) medium; 3) wide	The effect of the application of the provision can be observed in materials science, polylactide waste recycling, additive technologies and standardization.
		7.5 Is it proven in the article?  1) yes; 2) no	Statement #1 is proved in the article <b>Bergaliyeva S.A.</b> , Sales D. L., Delgado F., Bolegenova S., Molina S.I. Manufacture and Characterization of Polylactic Acid Filaments Recycled from Real Waste for 3D Printing // Polymers (Basel). – 2023. – Vol. 15, №9. P. 2165. https://doi.org/10.3390/polym15092165 (IF=5.0, Q1,

1	Percentile= 82 %)
7.1 Is the provision proven?	Statement #2. Increasing the proportion of secondary
1) proven;	polylactide from 0 to 75 % in a mixture with pure
2) rather proven;	polylactide increases the tensile strength of FFF printed
3) rather not proven;	samples from $44.20 \pm 2.18$ MPa to $52.61 \pm 2.28$ MPa.
4) not proven	2.20 Mil d.
	A nanocomposite for 3D printing based on polylactide
	waste with the addition of pure polylactide and titanium
	dioxide nanoparticles may have thermo-mechanical
	properties similar to those of a sample of pure polylactid
7.2 Is it trivial?	The scientific justification for the recycling of polylactid
1) yes;	waste as a raw material for additive technologies is given
2) no	waste as a raw material for additive technologies is give
7.3 Is it new?	For the first time, studies of the printability and quality
1) yes;	indicators of mixtures based on polylactide debris and
2) no	pure polylactide for additive technologies were conducted
	in percentages of 0 to 100, 25 to 75, 50 to 50, 75 to 25.
	respectively.
7.4 Level for application:	The effect of the application of the statement can be
1) narrow;	observed in materials science, polylactide waste recyclin
2) medium:	additive technologies and standardization.
3) wide	additive technologies and standardization.
7.5 Is it proven in the article?	Statement #2 is proved in the article Bergaliyeva S.A
1) yes;	Sales D. L., Delgado F., Bolegenova S., Molina S.I.
2) no	Manufacture and Characterization of Polylactic Acid
	Filaments Recycled from Real Waste for 3D Printing //
	Polymers (Basel). – 2023. – Vol. 15, №9. P. 2165.
	https://doi.org/10.3390/polym15092165 (IF=5.0, Q1.
	Percentile= 82 %)
7.1 Is the provision proven?	Statement #3. Adding 18 % of pure polymer and 7 % of
1) proven;	titanium dioxide nanoparticles to secondary polylactide
2) rather proven;	increases the tensile strength and fluidity of FGF sample
3) rather not proven;	to match those of a standard sample during 3D printing.
4) not proven	printing.
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	dioxide nanoparticles may have thermo-mechanical properties similar to those of a sample of pure polylactide.
7.2 Is it trivial?	The scientific justification for the recycling of polylactide
1) yes;	waste as a raw material for additive technologies is given.
<u>2) no</u>	
7.3 Is it new?	For the first time, studies have been conducted on the
1) yes; 2) no	suitability and quality indicators of nanocomposites based on polylactide debris for additive technologies.
7.4 Level for application:	The effect of the application of the statement can be
1) narrow;	observed in materials science, polylactide waste recycling,
2) medium;	additive technologies and standardization.
<u>3) wide</u>	
7.5 Is it proven in the article?	Statement #3 is proved in the article Bergaliyeva S.A.,
<u>1) yes;</u>	Sales D.L., Cabello J.M.J., Pintos P.B., Delgado N.F.,
2) no	Gago P.M., Zammit A., Molina S.I. Thermal and
	mechanical properties of recycled-polylactide
	acid/titanium dioxide nanocomposites for material
	extrusion additive manufacturing // Polymers (Basel). –
	2023. – Vol. 15, №16. P. 3458.
	https://doi.org/10.3390/polym15163458 (IF=5.0, Q1,
*	Percentile=82 %)
7.1 Is the provision proven?	Statement #4. Standard of organization St JCS 001-2023
1) proven;	"Nanocomposites based on polylactide and its waste with
2) rather proven;	titanium dioxide nanoparticles for additive manufacturing.
3) rather not proven;	Technical specifications" establishes the percentage of
4) not proven	pure and recycled polylactide and titanium dioxide
	nanoparticles in proportions 25/75/0, 22/75/3. 18/75/7 for
	subsequent certification.
7.2 Is it trivial?	Standardization of the results of scientific research is an
1) yes;	important component for the subsequent
2) no	commercialization of the results of scientific work.
7.3 Is it new?	For the first time, an improved nanocomposite material
1) yes;	based on polylactide waste, which can be used in 3D
2) no	printing, has been standardized.
7.4 Level for application:	The effect of the application of the statement can be
1) narrow;	observed in materials science, polylactide waste

		2) medium; 3) wide	processing, additive technologies and standardization.
		7.5 Is it proven in the article?  1) yes; 2) no	Statement #4 is proved in the article Bergaliyeva S.A., Sales D.L., Cabello J.M.J., Pintos P.B., Delgado N.F., Gago P.M., Zammit A., Molina S.I. Thermal and mechanical properties of recycled-polylactide acid/titanium dioxide nanocomposites for material extrusion additive manufacturing // Polymers (Basel). —
			2023. – Vol. 15, No. 16. P. 3458. https://doi.org/10.3390/polym15163458 (IF=5.0, Q1, Percentile=82 %)
8.	Principle of reliability Reliability of sources and information provided	8.1 Choice of methodology —is justified or methodology is described in sufficient detail:  1) yes;  2) no  8.2 The results of the thesis work were obtained using modern methods of scientific research and methods of processing and interpreting data using computer technologies:  1) yes;  2) no  8.3 Theoretical conclusions, models, identified relationships and regularities have been proved and confirmed by experimental research (for areas of training in the pedagogical sciences the results have been proved on the basis of a pedagogical experiment):  1) yes;	The choice of methodology is reasonable. To obtain the main results of the dissertation research, methods of 3D printing with fused filaments and granules, scanning electron microscopy, differential scanning calorimetry, thermogravimetric analysis and tensile testing were used. The results were obtained using modern methods of research, processing and interpretation of data using computer technology. The research methodology is described with a detailed description of the methods of sample preparation, experimental equipment and approaches to analyzing the results.  The results obtained in this dissertation work do not contradict the research of other authors and are fully confirmed by experimental data.
		2) no 8.4 Important statements are <b>confirmed/partially</b> confirmed/ not confirmed by references to relevant and reliable scientific literature	Important statements of the work in all sections of the dissertation, if necessary, are confirmed by references to relevant and reliable scientific literature.
)	Dringinla of	8.5 Used literature sources are <u>sufficient</u> /not sufficient for a literature review	The literature sources used are sufficient for a literary review and consist of 162 titles of scientific papers.
,	Principle of practical value	9.1 The thesis has a theoretical value: 1) <b>yes</b> ; 2) no	The dissertation has theoretical significance, since the normative documents on standardization of hydrothermal aging of polylactide and nanocomposites based on

	8		recycled polylactide can be used by any manufacturer without re-testing these normative documents.
		<ul> <li>9.2 The thesis is of practical importance and there is a high probability of applying the results obtained in practice:</li> <li>1) yes;</li> <li>2) no</li> </ul>	The dissertation has practical significance. During the work, the percentage of nanocomposites based on recycled polylactide with thermo-mechanical properties similar to those of a standard sample was determined. The developed and approved standard of the organization St JSC 001-2023 is sufficient to carry out the certification procedure of the obtained nanocomposite materials for additive manufacturing.
		9.3 Are the suggestions for practice new?  1) <u>completely new;</u> 2) partially new (25-75% are new);  3) not new (less than 25% are new)	The suggestions for practice are completely new.
10.	Quality of writing and design	The quality of academic writing is:  1) high; 2) average; 3) below average: 4) low.	The dissertation work is performed at a fairly high level and represents a completed research work. The text of the dissertation is written in a clear scientific language, using the terminology accepted in this field of research. The style of presentation of the letter corresponds to scientific works.

## Conclusion on the possibility of awarding the degree of Doctor of Philosophy (PhD)

Thesis work performed on the topic: «Standardization of Recycled Plastic Materials for Additive Manufacturing» is a completed research work. I believe that the thesis on the relevance of the problems solved and the quality of the results obtained, in terms of their scientific and practical significance, is a serious scientific research and meets all the requirements of the rules of awarding the degree of Doctor of Philosophy (PhD) of the Committee for Quality Assurance in the Field of Science and Higher Education of the Ministry of Science and Higher Education of the Republic of Kazakhstan, and its author Bergaliyeva Saltanat Amangeldinovna deserves to be awarded the degree of Doctor of Philosophy (PhD) in the educational program «8D07502 – Standardization and Certification (by industry)».

Специалист по кадрам БГТУ

## Official reviewer:

Professor of the Department of Physical and Chemical Methods and Quality Assurance, Belarusian State Technological University (Minst Candidate of Physical and Mathematical Sciences. Associate Professor

Vetohin S.S.