R E V I E W

by
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Member of the Academic Jury set to render a decision on a procedure for the acquisition of the Academic Degree "Doctor of Philosophy" (PhD) in the Professional Field 4.2. Chemical Sciences according to the Classifier of the Areas of Higher Education and the Professional Fields (Scientific Specialty "Polymers and Polymer Materials")

Author of the dissertation: M.Eng. Selin Erdinch Kyuchyuk-Hyusein

Topic: Electrospun fibers with core-sheath architecture based on of poly(ethylene oxide), a biodegradable aliphatic polyester and beeswax

Scientific supervisors: Assoc. Prof. DSc Dilyana Paneva and Corresponding Member of the Bulgarian Academy of Sciences, Prof. DSc Iliya Rashkov

This Peer Review is prepared in response to Order № РД-09-125/01.10.2025 issued by the Director of the Institute of Polymers, Bulgarian Academy of Sciences, following the decision made by the Academic Jury that was held on 03.10.2025.

The Review complies with *the Development of Academic Staff in the Republic of Bulgaria Act (DASRB), the Rules for the Application of the Development of Academic Staff in the Republic of Bulgaria Act, the Rules of BAS and with the Rules set at the Institute of Polymers, Bulgarian Academy of Sciences, for applying the Act mentioned above.*

1. Biographical information about the candidate

M. Eng. Selin Erdinch Kyuchyuk-Hyusein is a graduate of the University of Chemical Technology and Metallurgy, Sofia. She received a bachelor's degree in 2021 in the speciality "Technology of Glass, Ceramics and Binders" and a master's degree in 2023 in the speciality "Fine Organic Synthesis". She also has an additional qualification in "Perfume and Cosmetic Products".

In 2019, she started working as a junior laboratory assistant at "Zlatna Panega Cement" AD, after which she has been working at the Institute of Polymers - BAS since 2020, where she is currently a chemist.

Since 01.11.2023, she has been enrolled as a PhD student in independent training in the Professional field: 4.2. Chemical Sciences (Polymers and Polymer Materials) at the Institute of Polymers - Bulgarian Academy of Sciences, Scientific field "Polymer Biomaterials", Laboratory of Biologically Active Polymers. The topic of her dissertation is “Electrospun fibers with core-sheath architecture based on of poly(ethylene oxide), a biodegradable aliphatic polyester and beeswax”, and her scientific supervisors are Assoc. Prof. DSc. Dilyana Paneva and Assoc. Prof. DSc. Iliya Rashkov, Corresponding Member of BAS.

2. Assessment of the scientific and research accomplishments of the candidate

2.1. Assessment to meet the minimal criteria in accordance with the Specific Rules for Granting Academic Degrees set at the Institute of Polymers, Bulgarian Academy of Sciences, Application 1

According to the Law on the Acquisition of Scientific Degrees and for Holding Academic Positions of the Bulgarian Academy of Sciences, the minimum total number of points from all required indicators must be 80 points in the defence of her dissertation, Eng. Selin Erdinch Kyuchyuk-Hyusein participated with 200 points, which exceeds this requirement many times. Under indicator A, which carries 50 points, she has presented a dissertation. Its results have been published in five scientific papers in indexed and referenced editions, as listed in global databases such as SCOPUS/Web of Science. Two of the publications are in the Q1 quartile and three are in the Q2 quartile, which brings 110 points on the G indicator, with 30 points required. The relevance and significance of the obtained and published scientific results are evidenced by the fact that each of these publications already has citations in sources referenced in SCOPUS/Web of Science. Their total number is 20, and the corresponding points are 40.

2.2. Assessment of the requirement for the doctoral thesis to contain valuable theoretical or applied science results that correspond to modern achievements and represent a significant and original contribution to polymer science

The research in this dissertation is focused on a current trend in the development of polymer science and textile materials, specifically the creation of new composite micro- and nanofibrous materials using electrohydrodynamic methods. Unique morphological characteristics, a specific surface, and the possibility of structural modification characterise these materials. These innovative materials have applications in biomedicine, pharmacy, the food industry, agriculture, environmental protection, and other fields.

As the doctoral candidate indicated in her dissertation, the presented results are part of the long-term systematic research of the Laboratory of Bioactive Polymers, in the Scientific Department Polymer Biomaterials, at the Institute of Polymers - BAS (LBAP), established in 1989 by one of its scientific supervisors, Corresponding Member Prof. DSc. Iliya Rashkov. This dissertation is based on the experience gained over the years in methodology, equipment, and the high competence and qualifications of the doctoral student's scientific supervisors, as well as the pursuit of innovation and the creation of new technologies.

The dissertation is presented on 148 pages, includes 70 figures, nine tables, three schemes and cites 123 literature sources, most of which are from the last 10-15 years. The dissertation consists of the following sections: Introduction, Overview of the state of the problem, Aim and objectives of the dissertation, Results and discussion, Experimental part, Conclusions, Contributions of the dissertation, which outline directions for future research, List of publications and scientific works reflecting the results of the dissertation, List of noted citations and References.

The literature review provides a brief overview of the properties of micro- and nanofibrous nonwovens, as well as the fundamentals of electrohydrodynamic spinning methods. However, electrospinning devices used to obtain monolithic fibres, especially those with complex architecture, which is related to the purpose of the dissertation, are examined in detail. The factors influencing the morphology of fibrous materials, the methods for their characterisation, the different polymers and the use of beeswax in the production of fibres by electrospinning have been considered. Based on a review of the literature sources, the goal and objectives of the dissertation

work have been formulated, specifically related to the production of composite fibres with core-sheath and core-double sheath architectures by single-nozzle electrospinning, without the use of a coaxial device. In response to modern requirements for newly created materials, biologically tolerable and biodegradable polymers with optimal molecular weights, as well as beeswax, have been utilised. Studies have also been conducted to evaluate the antibacterial, antifungal, and anticancer activities of new fibrous materials, which have proven their applicability in medicine, cosmetics, and agropharmacy. The information presented has gained international popularity and appreciation since it was published in a review article, "Core-Sheath Fibres via Single-Nozzle Electrospinning of Emulsions and Homogeneous Blend Solutions". This article, published in 2024, already has five citations.

The section "Results and Discussion" includes four chapters, the titles of which show the emergence of the idea of obtaining fibers with a "core-sheath" architecture, its development to obtain fibers with a "core-double sheath" architecture, proving the validity of the proposed approach for obtaining fibers with a "core-double sheath" architecture by using other biodegradable polyesters and studying the influence of the ratio of the molar masses of poly(ethylene oxide) PEO and polyester on the composition of the core and sheaths. The fourth chapter is related to proving the potential of new fibrous materials, with the inclusion of a biologically active substance for application in the field of medicine and "green" agriculture.

For the first time, an original approach for obtaining fibres with a "core-sheath" architecture is proposed, eliminating the need for an additional device in coaxial electrospinning. A suitable common solvent (chloroform) was found, through which homogeneous solutions of PEO, biodegradable aliphatic polyesters: polylactide (PLA), poly(ε-caprolactone) (PCL), polybutylene succinate (PBS), poly(D,L-lactide-co-glycolide) (PLAGA), poly(3-hydroxybutyrate) (PHB) and beeswax (BW) were obtained. The above finding enabled the application of a completely new method for obtaining fibres of the "core-sheath" and "core-double sheath" types, achieved through the self-organisation of the components during the electrospinning process. The inclusion of the natural component beeswax, which possesses antioxidant and barrier properties, provided the fibres containing PEO with a hydrophobic surface and a stable morphology. The inclusion of a biocompatible and biodegradable aliphatic polyester significantly improved the mechanical properties of the fibres compared to those obtained from PEO, PLA, and PEO/BW alone. The presence of PEO imparted the fibres with high elasticity. The optimal weight ratio was investigated initially between PEO and BW, then between PEO, PLA and BW, and by various methods such as scanning electron microscopy (SEM), differential scanning calorimetry (DSC) and X-ray diffraction (XRD), as well as by measuring the contact angle of wetting with water, and conclusions were made about the properties of the fibres. The structure of the fibres was confirmed by X-ray photoelectron spectroscopy (XPS), transmission electron microscopy (TEM), and by selective extraction with an appropriate solvent, followed by analysis with Fourier Transform Infrared Spectroscopy (FTIR).

The validity of the proposed approach for obtaining fibres with a "core-double sheath" architecture has been demonstrated by replacing PLA with one of the other biocompatible and biodegradable polyesters, PCL, PBS, PLAGA and PHB. It has been found that at a molecular weight of the polyester lower than that of PEO, fibres with a well-defined PEO core, an inner polyester sheath and an outer BW sheath are obtained. For polyesters with a molecular weight greater than that of PEO, it has been found that there is a presence of polyester macromolecules in the PEO core and of PEO chains in the inner polyester sheath. For the first time, sequential

selective extraction of the outer and inner sheaths has been applied using hexane and tetrahydrofuran (THF) as solvents, respectively. To prove the claim that directional deposition of hydrophilic and hydrophobic biologically active substances can be achieved in the electrospinning process, hydrophilic ZnO and hydrophobic ZnO(Si) were used as contrast agents and their distribution in the fibre structure was demonstrated using TEM.

The obtained fibres included the biologically active substances 5-chloro-7-iodo-8-hydroxyquinoline (CQ) and 5-nitro-8-hydroxyquinoline (NQ). The release kinetics of the compounds from the fibres were studied, as well as the antimicrobial and anticancer activities of the obtained materials.

The experimental part describes in detail the materials used, the preparation of the solutions, and the apparatus used to obtain the fibrous materials, as well as the methods by which they were characterised. The microbiological studies used, as well as the behaviour of the fibrous materials towards human pathogenic microorganisms, cancer and regular cell lines, and phytopathogenic and beneficial microorganisms for plants, are presented.

The conclusions drawn summarise the results obtained and emphasise the innovativeness of the proposed approaches not only in obtaining the new fibrous materials, but also in proving their structure, as well as in studying their properties and application possibilities.

2.3. Assessment of the scientific accomplishments of the candidates and contributions to the dissertation work

The dissertation work proposes several original ideas that have been successfully implemented and proven through various appropriately selected analysis methods. A new approach is presented for creating fibres with a "core-sheath" architecture from homogeneous solutions by single-nozzle electrospinning. In this way, for the first time, fibres with a "core-double sheath" architecture were also obtained through the self-organisation of the components, which renders the use of a coaxial electrospinning device unnecessary. It has been proven that this approach can be applied to various biodegradable polyesters, enabling the optimisation of the mechanical, surface, and biological properties of the fibres. The creation of fibres with a hydrophilic core and a hydrophobic sheath enables the inclusion of biologically active substances with varying properties, including hydrophilic, amphiphilic, and hydrophobic. The potential of the newly obtained materials in biomedicine and "green" agriculture is demonstrated.

2.4. Assessments of the scientific production of the candidate, as well as the reflection of the results in the works of other authors

The results of the dissertation work are summarised in five scientific articles, which have been published in journals with a high impact factor and quartile (two are in Q1 and three in Q2). Two of the articles have been published in the journals Macromol. Biosci. and J Appl Polym Sci. (publisher: Wiley), one in Polymers and two, one of which is a review in Materials (publisher: MDPI). In all publications, Eng. Kyuchyuk-Hyusein is the first author. By August 2025, according to Scopus data, each of the articles has already been cited, and the total number of citations in scientific periodicals is 20, which is a very high indicator of their relevance and interest from the scientific community.

The doctoral student has presented the results from the dissertation work at five scientific forums, including three oral and two poster presentations. For the presented research, she received two awards: For the best report at the Thirteenth Scientific Session "Young Scientists in the World

of Polymers", Institute of Polymers-BAS, 2022 and in the competition for young scientists "Academician Ivan Evstatiev Geshov", in the field of Nanosciences, New Materials and Technologies, BAS, 2023.

3. Assessment of the qualities of the extended abstract of the doctoral thesis, whether it correctly reflects the contributions of the doctoral thesis

The doctoral student has formulated her dissertation work and its abstract with precision, ensuring they meet the requirements. The abstract presents the relevance of the research conducted, the set goal and objectives of the dissertation work, the main results obtained and the conclusions drawn. The contributions achieved are noted, and directions for future research are outlined.

4. Opinions, notes and recommendations

M. Eng. Selin Erdinch Kyuchyuk-Hyusein has carried out a significant amount of experimental work, which has proven and further developed the initial idea of obtaining fibres with a "core-sheath" architecture by electrospinning, without the need for an additional coaxial device, to achieve fibres with an even more complex "core-double sheath" architecture. Several factors that could alter the properties of the fibres obtained in this manner have been analysed, and studies related to their application as antimicrobial and anticancer materials have been conducted. All ideas, as well as the conclusions drawn, have been proven with various modern methods of analysis. The results obtained have been skillfully discussed and supported by many schemes, figures, and tables.

The doctoral student has successfully completed the necessary exams and submitted all documents in a well-organised and formatted manner, meeting the requirements.

The doctoral student, together with her scientific supervisors, participated in two contracts, one utility model and one patent, and presented her results at three other scientific forums through three poster presentations, distinct from those in her dissertation work. All this is grounds for the claim that Eng. Kyuchyuk-Hyusein has received excellent training, which characterises her as a very well-prepared young researcher.

5. Conclusion

According to the grounds of the documentation presented by the candidate, as well as the review of her publications and the above assessment, I recommend to the Academic Jury that a positive decision be rendered for the acquisition of the Academic Degree PhD for M. Eng. Selin Erdinch Kyuchyuk-Hyusein in the professional field: 4.2. Chemical Sciences, scientific speciality: Polymers and Polymeric Materials.

12.11.2025 г.
Sofia

Reviewer:
Prof. Deisilava Staneva Grabcheva
Member of the Academic Jury