# ABSTRACT

for the thesis on the topic

**«Plasma-chemical synthesis of fullerenes in an arc discharge and their purification from impurities»**

submitted for the degree of Doctor of Philosophy (PhD) in the educational program 8D07112 – «Nanomaterials and Nanotechnologies»

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**General characterization of the work.** This dissertation reports experimental investigations on the plasma-chemical synthesis of fullerenes in an arc discharge under optimized technological conditions, their purification from impurities, and detailed structural characterization. Furthermore, the study examines the influence of the synthesized fullerenes on the performance and properties of pyrotechnic composites, as well as their effect on the tribological behavior of lubricating formulations.

**Relevance of the study.**

With the development of nanotechnology, nanomaterials—particularly carbon-based nanostructured materials—are becoming increasingly demanded for various practical applications and across multiple industrial sectors. These materials play a crucial role in medicine, electronics, energy, the oil and gas industry, construction, aerospace, mechanical engineering, and other fields. Carbon nanostructures represent a promising class of materials with a unique combination of physicochemical properties, including high mechanical strength, large specific surface area, electrical conductivity, and chemical stability. These properties underpin their wide-ranging applications in modern high-tech technologies.

In medicine, carbon nanomaterials are employed in the design of targeted drug delivery systems, highly sensitive biosensor platforms, and biocompatible scaffolds for tissue engineering. Their structural characteristics enhance diagnostic accuracy and improve the efficacy of therapeutic interventions.

In electronics, carbon nanotubes and graphene serve as the basis for the development of miniaturized and energy-efficient electronic components. They are used to fabricate nanotransistors, sensors, and flexible electronic elements, facilitating the advancement of high-performance, compact devices.

In the energy sector, these materials are applied in the development of high-efficiency solar cells, batteries, and supercapacitors. Due to their high conductivity and large specific surface area, carbon nanostructures enhance energy storage and conversion processes.

In construction technologies, carbon nanomaterials are incorporated into composites to improve their strength, deformation resistance, and thermal conductivity. This enables the creation of next-generation materials with superior performance characteristics.

Among the wide variety of nanostructured carbon materials, fullerenes occupy a special place. Recent analyses of scientific studies and publications indicate that researchers continue to focus on optimizing fullerene synthesis technologies, investigating their physicochemical properties, and exploring new application areas. This demonstrates the sustained interest in fullerenes. These research directions are critical both for fundamental studies aimed at understanding the underlying principles of nanotechnology and nanomaterials science, and for applied research, where the potential of fullerenes can be harnessed to develop novel materials and technologies. Advancements in these areas contribute to further progress in nanomaterials and nanotechnology, opening new opportunities for innovation and improving quality of life.

Currently, various methods exist for fullerene synthesis, including laser ablation, gas-phase synthesis, graphite arc evaporation, electron-beam evaporation, and ion-beam sputtering. However, challenges such as low yield, suboptimal product quality, and the need for purification remain significant.

One of the most widely used and effective methods for fullerene production is plasma-chemical synthesis employing high-temperature arc discharges. This method facilitates efficient fullerene formation due to the high energy levels, which promote the decomposition of carbon-containing materials and the formation of the fullerene structure. Additionally, this approach allows the production of fullerenes in macroscopic quantities and is more cost-effective compared to other methods.

Therefore, research aimed at improving the efficiency of plasma-chemical synthesis of fullerenes in high-temperature arc discharges, as well as expanding their practical applications, remains highly relevant and significant in the field of nanomaterials and nanotechnology.

**The aim of the thesis research dissertation** is to obtain fullerenes via plasma-chemical synthesis under optimized arc-discharge parameters, to purify them from impurities, and to investigate the effects of fullerenes on the performance and characteristics of pyrotechnic composites and tribological lubricating formulations.

**Objects of study.**

The objective of this dissertation is to synthesize fullerenes via plasma-chemical methods under optimized arc discharge parameters, purify them from impurities, and investigate their effects on the performance and characteristics of pyrotechnic composites and tribological lubricating formulations.

**To achieve this objective, the following tasks were set:**

- To develop and optimize the arc discharge method for the plasma-chemical synthesis of fullerenes and the subsequent heat-treatment process, in order to produce fullerene soot at a production scale; and to establish a method for purifying the fullerene soot obtained after arc synthesis.
- To study the influence of fullerenes on the burning time, flame intensity, and energy release characteristics by incorporating them into pyrotechnic composites composed of potassium nitrate, aluminum, and charcoal; and to elucidate the mechanism by which fullerenes affect the combustion kinetics of pyrotechnic systems, with particular emphasis on their thermal conductivity.
- To experimentally investigate the effect of fullerenes on the tribological

properties of lubricating formulations; to evaluate the evolution of surface microrelief at bearing joints, the degree of surface damage, and the effectiveness of fullerenes in reducing wear.

**Research Objects and Subjects.**

The objects of this study are fullerenes and composite materials based on them, obtained via plasma-chemical arc synthesis.

**Objects of study.**

The objective of this dissertation is to synthesize fullerenes via plasma-chemical methods under optimized arc discharge parameters, purify them from impurities, and investigate their effects on the performance and characteristics of pyrotechnic composites and tribological lubricating formulations.

**The subject of the study is the technological** conditions of the arc synthesis process for producing fullerene soot at a production scale, as well as the parameters and characteristics of pyrotechnic composites and lubricating formulations modified with fullerenes.

**Research Methods**

A variety of experimental methods were employed to achieve the objectives of this dissertation. In particular, the morphological characteristics of the materials were analyzed using scanning electron microscopes (Quanta 3D 200i, FEI Company; Hitachi TM4000 Plus) and an optical microscope (DM 6000M, Leica). Structural characterization was carried out using Raman spectroscopy (Ntegra Spectra C3M, NT-MDT). Thermal conductivity measurements were performed using a setup based on the steady-state heat flow method equipped with a temperature control system (Lake Shore Model 336 Temperature Controller).

**Scientific novelty of the research:**

1. Comprehensive investigations were conducted on the influence of arc discharge parameters and the preliminary thermal treatment of carbon rods on the plasma-chemical synthesis of carbon nanostructures. The optimal technological parameters for the plasma-chemical synthesis of thermodynamically stable, spherically closed fullerene structures were established.
2. The effect of fullerene-containing soot (FCS) on the pyrolitic parameters and characteristics of the heterogeneous $KNO_3$/Al/charcoal system was studied. For the first time, it was demonstrated that the main pyrolitic parameters, such as the duration of steady-state diffusion combustion and the integral flame luminosity, can be effectively controlled by varying the concentration of FCS.
3. The influence of fullerene-containing soot and $Al_2O_3$ nanoparticles on the tribological properties of industrial lubricating materials (ILMs) was investigated. For the first time, it was shown that the combined introduction of FCS and $Al_2O_3$ into ILMs leads to the adsorption of a nanostructured tribofilm on the surface of components. This results in a reduction of the contact wear area and an increase in the time during which surface microrelief homogeneity is maintained under frictional conditions.

**Statements submitted for defense:**

1. Using plasma-chemical synthesis with optimized arc discharge

parameters—working gas: He, P = 300 Torr, U = 30 V, I = 300 A—and preliminary thermal treatment of the graphite rod, it is possible to obtain thermodynamically stable, spherically closed fullerene structures. After chemical purification, fullerene soot consisting of a $C_{60}$ and $C_{70}$ mixture is obtained with a product yield of up to 18%, featuring an increased fraction of $C_{70}$.

2. The introduction of fullerene-containing soot at concentrations of 5–15 wt.% into the heterogeneous pyrotechnic system $KNO_3$/Al/charcoal increases the duration of steady-state diffusion combustion by 2.2 times (from 5 to 11 s) and enhances the integral flame luminosity by 33%. This effect is attributed to the high heat-storage capacity of fullerene molecules, their catalytic influence on the thermal decomposition kinetics of potassium nitrate, and additional $CO_2$ release during controlled oxidation of fullerenes.

3. The combined addition of fullerene-containing soot (1 g) and $Al_2O_3$ nanoparticles (1 g, particle size 32–190 nm) into industrial lubricating materials results in the formation of an adsorbed nanostructured tribofilm on the sliding bearing surfaces. This leads to a reduction of the contact wear area by approximately 20% and preserves the homogeneity of the surface microrelief after 200,000 cycles under an axial load of 980 N and a rotational speed of 750 rpm.

**Scientific and practical significance of the study**

The results obtained in this work are important for advancing the physics of fullerenes, their synthesis technologies, and practical applications. The established optimal technological parameters for plasma-chemical synthesis of fullerenes, yielding a high proportion of the final product, open prospects for the industrial implementation of this technology. The identified patterns of improved performance and characteristics of pyrotechnic composites and industrial lubricating materials upon modification with fullerene-containing soot significantly expand the potential practical applications of fullerenes.

**The reliability and validity of the study results** are ensured by the use of modern equipment and research methods. Furthermore, the credibility of the findings is supported by publications in high-impact international journals, as well as in journals recommended by the Committee on Coordination of Scientific Research of the Ministry of Education and Science of the Republic of Kazakhstan, and by presentations at international scientific conferences both near and far abroad.

**Author's Personal Contribution**

All samples investigated in this dissertation were personally prepared by the author. The development of methodologies and the selection of anodization parameters were carried out independently by the author. The study of morphology, phase composition, elemental analysis, and Raman spectroscopy of the synthesized samples was conducted jointly with the research team of the Open National Nanotechnology Laboratory at Al-Farabi Kazakh National University.

The research results presented in the dissertation, including the participation in the creation of the experimental setup for arc synthesis of fullerenes, the development of a plasma-chemical arc discharge-based method for fullerene synthesis with automatic desorption of graphite rods, experiments on arc synthesis of fullerene soot, optimization of synthesis parameters, extraction of the fullerene

phase from fullerene-containing soot, chromatographic separation of fullerenes, and the study and analysis of the properties of the obtained samples, were all conducted by the author.

Experiments on the influence of fullerenes on the tribological properties of lubricating formulations were carried out with the assistance of the laboratory supervisor, PhD Bakhytuly Nauryzbek, using a specialized laboratory setup at the Metallurgy and Enrichment Institute of the Kazakh National Research Technical University named after K.I. Satpayev. The formulation of research tasks and discussion of the experimental results were conducted jointly with scientific consultants.

**Publications**

Based on the results of the dissertation, 12 publications have been produced: 3 articles in journals listed by the Committee on Coordination of Scientific Research of the Ministry of Education and Science of the Republic of Kazakhstan (for publication of the main results of the dissertation for the PhD degree), 1 article in a high-impact international journal indexed in Scopus, and 8 papers presented at international scientific conferences.

**Dissemination of Research Results**

The results obtained during the dissertation research were presented and discussed at the following international conferences:

International Scientific Conference for Students and Young Scientists “GYLYM JANE BILIM - 2022” (2022, Astana, Kazakhstan);

International Scientific Conference “Interdisciplinary Science Studies” (2023, Dublin, Ireland);

XXVII International Conference “EUROPEAN ACADEMY OF SCIENCES & RESEARCH” (2022, Germany).

**Articles Published in Journals Indexed in the International Scientific Databases Thomson Reuters and Scopus:**

**1.Aimasheva Zh.,** Coruh A., Ismailov D., Partizan G., Ibraimov M.K., Khaniyev B. Synthesis of Fullerenes by Electric Arc Discharge and their Applications in Pyrotechnic and Lubricants // ES Materials and Manufacturing. – 2025. – V. 28. – P. 1444. **[Q1, 91%]**

**Articles Published in Journals Recommended by the Committee for Quality Assurance in Science and Higher Education of the Ministry of Science and Higher Education of the Republic of Kazakhstan:**

1. Aimasheva Zh., Ismailov D.V., Oman Z., Orynbai B. Synthesis of Fullerenes in Arc Discharge and Their Purification from Impurities // Reports of the National Academy of Sciences of the Republic of Kazakhstan. – 2023. – No.1. – Vol.345. – P.96–100.
2. Aimasheva Zh.K., Ismailov D.V., Ksenofontov D.A., Muratov M.M., Vorobyeva K.A. Application of Nano- and Micromaterials in Military Science // Scientific Works of VIIR&E. – 2024. – No.4. – Vol.58. – P.111–117.
3. Aimasheva Zh.K., Ismailov D.V., Grishchenko V.F., Stefano B., Partizan G., Koshtybayev T.B. Application of Micro- and Nano-Materials in Pyrotechnics // Bulletin of KBTU. – 2024. – No.2. – Vol.69. – P.229–237.

**Publications in the Proceedings of International Conference Abstracts:**

1. Aimasheva Zh.K. Synthesis of Fullerenes by the Arc Discharge Method // Proceedings of the International Conference of Students and Young Scientists "Farabi Alemi". – 2022. – P.346.

2. Aimasheva Zh.K. Plasma-Chemical Synthesis of Fullerenes in Arc Discharge and Their Purification from Impurities // Proceedings of the International Scientific Conference "GYLYM JANE BILIM – 2022" for Students and Young Scientists. – 2022. – P.459–463.

3. Aimasheva Zh., Ismailov D.V. Synthesis of Fullerenes by the Electric Arc Discharge Method // Proceedings of the Scholarly Abstract "European Academic Science and Research". – 2022. – P.12.

4. Aimasheva Zh. Synthesis of Carbon Nanomaterials and Their Purification from Impurities // Global Science and Innovation 2023: Central Asia, "Technical Physics" Series. – 2023. – No.1. – Vol.19. – P.46–51.

5. Aimasheva Zh.K., Ismailov D.V. Synthesis of Fullerenes by the Electric Arc Discharge Method // International Scientific Conference "Interdisciplinary Science Studies". – 2023. – P.5–7.

6. Aimasheva Zh.K. Micro- and Nanomaterials in Pyrotechnics // Proceedings of the International Conference of Students and Young Scientists "Farabi Alemi". – 2023. – P.59.

7. Aimasheva Zh.K. Application of Fullerene Soot Synthesized in Arc Discharge in Bearings // Proceedings of the International Scientific Conference "Farabi Alemi". – 2024. – P.343–345.

8. Aimasheva Zh.K., Ismailov D.V. Synthesis of Nano- and Micromaterials and Their Applications // International Scientific-Research Forum Dedicated to the 90th Anniversary of al-Farabi Kazakh National University. – 2024. – P.81–83.

**Relation of the dissertation topic to research plans**

The dissertation was carried out within the framework of grant funding (GF) provided by the Science Committee of the Ministry of Science and Higher Education (MoSHE) of the Republic of Kazakhstan, Grant No. AP19678266.

**Scope and structure of the dissertation**

The dissertation includes a list of abbreviations, an introduction, a main part consisting of three chapters, a conclusion, and a list of references. The work comprises 99 pages and contains 57 figures, 8 tables, and 139 bibliographic references.