REPUBLIC OF AZERBAIJAN

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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

PHYSICAL-TECHNOLOGICAL FEATURES AND APPLICATION ASPECTS OF CARBON NANOSTRUCTURES SYNTHESIS

Specialty:	3311.02 – Control devices and methods over the natural environment, substances, materials and products
Field of science:	Technique
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GENERAL CHARACTERISTICS OF THE WORK

Relevance of the case. In modern world, a number of significant results have been achieved in the development of technologies, equipment and devices for the synthesis and application of carbon nanomaterials (CNM) in world scientific centers. One of the main stimulating factors in the development of nanotechnology was the creation of semiconductor nanostructures based on this technology and new electronic devices capable of performing tasks such as genetic engineering, materials science (surface reinforcement, fullerenes, catalysts and computer chips), information storage and transmission. Recently, fundamental researches on the achievements in nanoscale systems physics and structures has led to the conclusion that new ideas about the nature of matter can be formed. This will mean the creation of quantum devices in the near future.

However, the issue of obtaining high-yield, high-quality and low-cost CNM remains mainly unresolved.

The creation of technologies and testing facilities for the nanomaterials synthesis, the organization of the getting and application of these materials is extremely relevant in the context of the rapid development strategy in the nanosphere.

The purpose of the study and the issues to be resolved. The purpose of the dissertation work is to study the physical, chemical and technological properties of carbon nanomaterials and their application in various scientific and technological spheres.

In order to achieve this goal, the following issues were set and resolved in the dissertation:

1. Comparative analysis of methods for obtaining, determining, separating and purifying carbon nanostructures and creating a theoretical and technological basis for further research.
2. Improving the process of high-yield synthesis of nanomaterials by studying the physical, chemical and technological properties of their existing obtaining methods.

3. Creation of pilot experimental devices for production of carbon nanostructures and the acquisition of various nanostructures in these devices , determination of their presence by instrumental and chemical methods, separation, purification and sorting of synthesis products.

4. Development of new, multifunctional urethane-type polymers and forpolymers synthesis technology, which can play a very important role in the application of the obtained nanomaterials, development of methods of their synthesis and application.

5. Synthesis of nanocomposite materials based on the obtained carbon-based nanomaterials (fullerene, nanotubes, nanolyphs, etc.), ferromagnetic nanomaterials, urethane-type polymers and forpolymers, the study of their application in various fields of science and technology.

Research methods. Problems of analysis and synthesis, necessary sections of physical and chemical analysis, spectral analysis. Carbon nanostructur chlorination of low molecular weight hydrocarbons in the "hot" catalyst layer in the production of new generation polymers and forpolymers such as chloromethanes, urethane, individual carbohydrogen pyrolytic synthesis of sludges and polycondensation and polybirdation methods in the polymer-monomer system were used.

Scientific innovations.

1. The possibility of obtaining nanomaterials, synthesis of fullerenes and nanotubes in the ethylene pyrolysis process with high yield, as well as by chlorination of natural gas has been confirmed.
2. The yield of nanomaterials was optimized to 20-24% due to the improvement of technological factors in catalytic chlorination and pyrolysis processes.
3. Development of a new for the synthesis technology of new, multifunctional urethane-type polymers and forpolymers that can play a very important role in the application of the obtained nanomaterials development of their synthesis and application methods.

4. During the chlorination of natural gas and other hydrocarbons in the "hot layer" reactor, it was possible to obtain fullerenes and carbon nanotubes in the same mode.
5. After separation and purification of nanotubes and fullerenes obtained in catalytic chlorination and pyrolysis processes, they are involved in various polymer compositions as components and their high efficiency can be applied in mechanical engineering, oil extraction, oil refining, aerospace, medicine and other fields.

The main provisions of the defense.

1. Theoretical and technological developments for the acquisition, determination, separation and purification of carbon nanostructures.
2. Obtaining nanomaterials, synthesis of fullerenes and nanotubes in the process of pyrolysis of ethylene with high yield, as well as by chlorination of natural gas.
3. Methods developed for the improvement of new technologies for the synthesis of multifunctional urethane-type polymers and forpolymers.
4. Involvement of the obtained nanotubes and fullerenes into various polymer compositions as components and methods of their application in various fields of science and technology.

Theoretical and practical significance of the work.

1. Theoretical and technological bases for obtaining nanostructures by chlorination and pyrolysis processes of natural gas and other low-molecular hydrocarbons with high yield have been created.
2. Pilot-experimental devices have been created for the synthesis of carbon nanostructures, urethane-type polymers and for-polymers, and their nanocomposites.
3. The obtained nanomaterials were tested with high efficiency in mechanical engineering, oil extraction, oil refining, aviation and instrument making, and the application possibilities were defined.

4. The possibility of mass application of multifunctional, fully transparent, non-toxic odorless, urethane-type new generation polymer coatings, which play the matrix role in the production of nanocomposite materials, have been identified for various fields of the industry.
5. A solid scientific and technical base for the production of new generation urethane-type polymers, composite materials and numerous carbon and other nanomaterials on an experimental and experimental scale had been established for the first time in Azerbaijan.
6.

Approbation of research work and application of results. The main results of the research were presented and discussed at the following scientific and technical conferences:

- 1st International scientific conference on "Nanostructured materials-2008" folk. Minsk, April 22-25, 2008.
- 5th International Conference on Special High Technologies. Ukraine, Yalta, September 22-26, 2008.
- International conference on nanomaterials and hydrogen energy, held within the NATO's "International Cooperation for Peace" program. Georgia, Batumi, October 7-10, 2012.
- "February Readings - 2018: Creative potential of youth in solving aerospace problems" Proceedings of the III International Semi-Practical Youth Conference. Baku, National Aviation Academy, February 12-14, 2018.

According to the results of the wide research, experiments held in IOGP named after N.Narimanov under Azneft PU, in OGPD (2009), in oil production wells of “GARASU” Operating Company (2016), in “AZMOL” OJSC located in Berdyansk, Ukraine (2008), in Russian “Aviation Materials” Research Institute (2013) and the Russian Federal State Civil Aviation Research Institute (2014). The gained results were applied and necessary application acts were approved.

Published works. 30 scientific works on the topic of the dissertation were published. These works include 14 scientific articles (including 6 articles in periodicals published in international summary and indexing systems), conference material and thesis - 9, monograph (published abroad) - 2, Patent-invention - 5 (1 Eurasian patent).

The structure and scope of the work. The dissertation consists of total 163 pages, including a list of glossary and abbreviations, introduction, four chapters, 14 tables, 46 figures, main results, a list of 118 bibliography and 225510 characters, without tables, pictures, bibliography.

Summary.

The introduction substantiates the urgency of the research work, provides information on the research purpose and issues of research, scientific modernity of the work, the main provisions of the defense, the practical significance and approbation of the results obtained.

The **first chapter of the dissertation** examines the physical and technological properties of carbon nanostructures, their production methods, existing synthesis technologies and the operation principles of devices based on these technologies.

It was noted that in 1973, on the basis of quantum chemical calculations of D.A.Bochvar and Y.G.Galperin in Russia, and before them in Japan, Y.Osawa, suggested the idea of closed electron coating and aromaticity, they are defined in a form with a stable character and chemical stability. 1985 N.V.Kroto, R.F.Kerl, R.Y-.Smolli modeled the existence of "carbon stars" by evaporating graphite under the influence of laser beams in a stream of helium at a temperature of 10000°C.

The end of the XX century and the beginning of the XXI century scientifically and practically perspective nanomaterials and nanotechnologies, were produced in various assortments and application directions and successfully applied in various fields of science and technology.

At the same time, in the second chapter, different obtaining methods of nanomaterials were compared, their advantages and disadvantages in terms of yield and quality indicators were evaluated, and other characteristics of the obtained materials were worked out.

The synthesis properties of carbon-based fullerene, nanotubes and other nanoclusters were investigated.

X-ray, spectroscopic and other basic instrumental methods which is used to study the physical characteristics of nanomaterials of different nature, technologies for the separation and purification of carbon nanomaterials are also considered.

The second chapter studies various methods, systems and technological devices for the carbon nanomaterials production and reflects their main features. Some advantages of these technological methods and prospects of the obtained materials as hydrogen sorbents have been evaluated.

It was shown that one of the synthesis methods of fullerons is incomplete burning of hydrorogens. For example, fullerons are gained from the flame formed during the combustion of benzene/oxygen or ethylene (acetylene/oxygen) mixture. The scheme of the device created for the synthesis of fullerenes by incomplete burning method of carbohydrogenes was analyzed, as well. The device consists of a low-pressure camera, a viewing window for visual and optical diagnostics, and the device for combustion of mixture and obtain gained samples. The combustion of fossil fuels and oxygen takes place on a horizontal copper plate with holes, through which fuel and oxygen enter through holes into the plate. The output of the fullerene C_{60} -C_{70} mixture in this device is about 8%.

This chapter also provides extensive information on other methods and devices used to obtain CNMs. However, it woths mentioning that, in all cases, both the output and the quality indicators of the CNMs in the experimental facilities are low. In the best case, in some devices, for example, in a multifunctional electric arc device, the output is incomplete 15%. The synthesized carbon nanotubes and nanofibers are obtained dirty and defective. The gained nanomaterials cost expensive, which does not allow the widespread use of these materials.

Thus, the acquisition of high-cost and clean CNMs remains relevant, and the next third chapter addresses the issue of creating the proposed technology and facilities.

In the third chapter, the author underlines the technology of production, separation, purification and sorting of carbon nanomaterials through the processes of catalytic chlorination and pyrolysis of light hydrocarbons. In this chapter, considerable space is devoted to the analysis of the characteristics of the production of light hydrocarbons by chlorination, the catalyst selection and the experimental device.

The carbon nanomaterials getting characterizes from ethylene in the pyrolysis process were also viewed in this chapter. The acquisition features of the study of technological features of these processes were analyzed from the obtaining high-quality fullerene and nanotubes with high yield point of view.

The reactor (Figure 1), developed and exploited by us for the chlorination process, consists of two parts, a hollow, column-type apparatus mounted vertically: a reaction zone and a precipitation zone.

1 - reaction zone
2 - sedimentation zone
3 - two-stage cyclone
4 - explosion disc
5 - nozzle for the catalyst
6 - catalyst
7 - distributor
8 - outlet valve
9 - meter for chlorine
10 - meter for methane
11-15 - control measurement devices

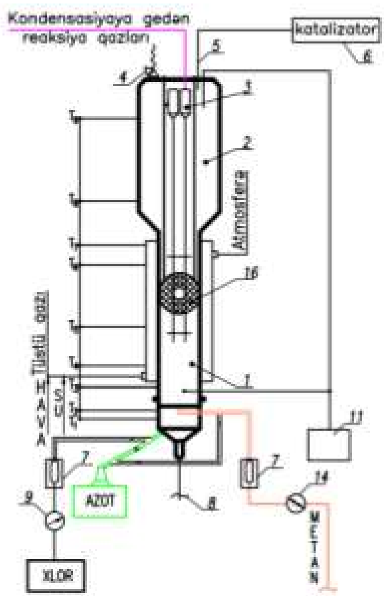


Figure 1. Reactor used for chlorination process

From the outside the reaction zone of the reactor is covered with a jacket and a nichrome bandage for heating. In the circular area of this shirt, either cold air or cold water can be given to eliminate the heat generated by the reaction.

The system works as follows: after the catalyst is fed to the reactor to create a "hot layer", a certain amount of methane or chlorine is distributed. It takes 4-5 hours to heat the reactor. When the required temperature is reached, chlorine is started. After that, the required amount of methane enters by barbate passing through the meter.

To control the chlorination process in the reactor, 9 temperature points are installed pulse tubes to measure the level and concentration of the catalyst, the pressure of the reaction gas on the hot bed. The purple color of the solvent (toluene) as the flue gases pass from the receiver to the condensate confirms the presence of fullerene C_{60} . Upon completion of the experiment, the amount, purity and yield of fullerene and nanotubes obtained on the basis of current technologies can be determined. Chlorination products are determined after cooling.

The method of obtaining carbon modifications is to supply gaseous methane and chlorine to the injector ducts installed in the reactor chamber, before which the catalyst is loaded into the reactor, small dispersed activated carbon, iron, perlite or quartz sand is used as catalyst, and nitrogen is added to form a "hot layer" after chlorine. Figure 2 shows the characteristic Raman spectrum confirming the multilayer nanotubes existence obtained during the experiments, and Figure 3 shows the output of the fullerene C_{60} - C_{70} mixture obtained by the finished chlorination of low molecular weight hydrocarbons in the hot layer reactor.

The presence of carbon nanotubes in the contest of duda which is obtained during the pyrolysis process with the help of the Raman spectrum had been proven. No other carbon nanostructures were found in the duda (Figure 4).

In addition to the Raman spectra, the presence of nanotubes obtained in the process of complete chlorination of methane in the “Hot bed” reactor was confirmed by X-ray photographs (Figure 5).

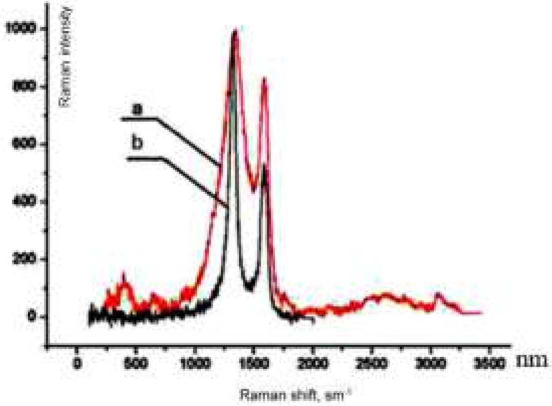


Figure 2. The characteristic Raman spectrum confirming the availability of multilayer nanotubes obtained during experiments: a- obtained carbon nanotube spectrum and b - reference nanotube spectrum.

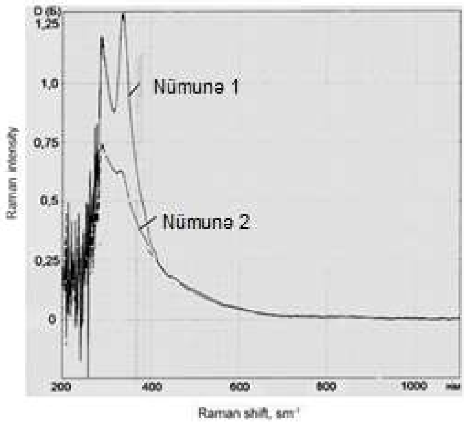


Figure 3. Yield of Fulleren C_{60} - C_{70} mixture on the given samples: Sample 1 - 23.6%, Sample 2 - 13.5%.

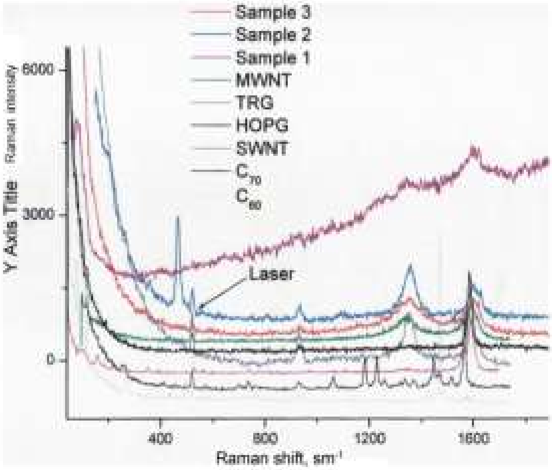


Figure 4. Raman spectrum of carbon nanotubes obtained during the pyrolysis process

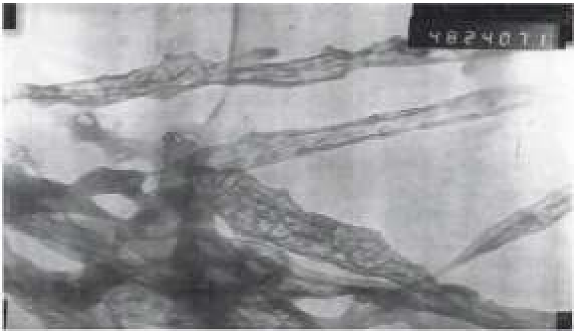
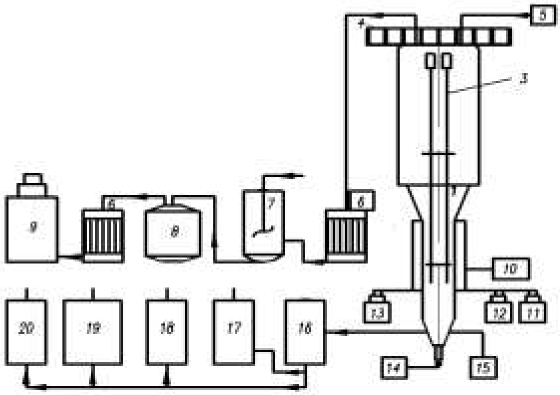


Figure 5. X-ray photo of carbon nanotubes obtained in the "Hot layer" reactor

The process of obtaining nanomaterials from chlorinated hydrocarbons, as well as chlorinated hydrocarbons C1-C4 in the finished chlorination reaction experiments was carried out in a special laboratory facility below. Based on the physical and technical parameters obtained in the laboratory facility by the finished chlorination method of chlorine carbons and carbon nanostructures, a technological scheme of the experimental facility had been developed (Figure 6).

When the dimensions and structure of the reactor unit were changed and other improvements were made better, i.e. satisfactory and different results were obtained in the chlorination process (for example, pre-drying of gas streams entering the reactor, more efficient introduction into the reactor, high temperature resistance in the contact due to the use or addition of a carrier-catalyst, a gradual increase in the share of catalyst, etc.). It should be noted that at this stage, the efficient use of gases from the pyrolysis process is also possible.



1 - reaction zone
2 - sedimentation zone
3 - two-stage cyclone
4 - explosion disc
5 - nozzle for the catalyst
6 - refrigerator
7 - mixing reactor
8 - intermediate capacity
9 - capacity for toluene
10 - methane meter
11-13 - control measuring devices
14 - outlet valve
15 - chlorine meter
16-20 - chlorocarbon capacities

Figure 6. Preliminary scheme of the experimental device developed by the author for the production of chlorine carbon and carbon nanostructures by the finished chlorination method

The method of obtaining quaternary carbon and other chloromethanes, as well as carbon nanostructures (fullerene C_{60} , C_{70}, endometofullerenes, high molecular weight fullerenes, single-layer and multilayer nanotubes, various nanoclusters) on the "hot layer" catalyst has a number of advantages: e.g. low cost of natural gas, hydrocarbon emission, use of industrial hydrocarbons mixtures and chlorine production wastes instead of methane, high quality and high yield of products (20-24%), increase of carbon nanoparticles by changing the heat balance, recipe, catalyst and other chemical and technical parameters opportunities to increase, the use of special, rare and expensive catalysts in this technology and the process is environmentally friendly and waste-free. However, the continuous synthesis of fullerene and its chlorinated derivatives, the creation of an experimental-industrial facility with risk-free and low capital investment in a short time is also possible.

The held research showed that the dispersed state of the catalyst plays an important role in the methane chlorination reaction. If the size of the catalyst is 50-100 microns, then 25-30% of the chlorine is used for destructive chlorination. These leads to the formation of hydrogen chloride and carbon. If the size of the catalyst is reduced from 100 mkm to 10 mkm, the rate of the destruction is gently reduced. The effect of the nature of chloromethanes and catalysts applied to the release of nanostructured smoke during the inspection of the finished chlorination process was also studied.

According to these results, it should be noted that the yields of 4-chlorinated carbon and nanostructured experiments conducted under similar conditions of the tested catalysts are different.

Thus, while the yield of 4-chlorinated carbon is between 83.3-98%, the yield of nanostructured duda varies between 17.2-36.4%. It can be concluded that, the constituent materials of the heat carriers of the applied catalysts during the chlorination of methane in the "Hot layer" have a different degree of catalytic effect on the reaction course.

In the course of the experiments, pumice in different sizes was selected as the most active catalyst to study the effect of catalyst sizes, duration of chlorination process in the hot bed reactor and yield of obtained chlorocarbons and nanostructured smoke, and its participation as a catalyst was tested.

It was found out that when changing the size of pemza, for example, in tests up to 60 microns, the reaction time is slightly reduced, and the yield of products increases significantly. While holding the experiments, natural gas, mainly consists of methane, was used as a hydrocarbon. When using methane directly, a sharp increase in carbon nanomaterials and nanoclusters was observed in the dude.

Many researchers for the synthesis of acetylene and ethylene had used carbon nanomaterials, Fe, Co, Ni powders or their mixtures as catalysts graphite, SiO_2 , Al_2O_3 or zeolite as carriers and Pyrolysis is usually carried out at a temperature of 500-800°C and a partial pressure of 0.1-0.5 atomic hydrocarbons, using N_2 or H_2 as a diluent gas.

The scheme of the proposed laboratory device for pyrolysis of hydrocarbons is shown in Figure 8.

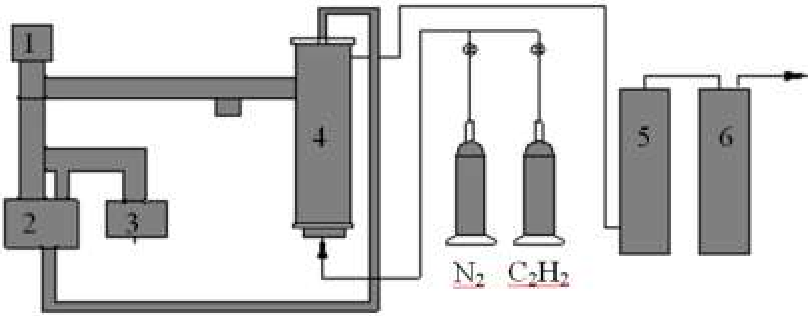


Figure 8. Scheme of the device for pyrolytic synthesis of carbon nanostructures from ethylene: 1 - automatic connector, 2 - thermal regulator, 3 - magnetic release, 4 - reactor, 5 - trap, 6 - trap holder

Increasing the process temperature usually has little effect on the length of the nanotube, but it influences on the rise of the amount of amorphous carbon. Increasing the pyrolysis time leads to an development in the yield, length and diameter of the nanotube. The composition of the diluent gas has almost no effect on the development of the nanotube, although the amount of amorphous carbon decreases during the use of H_2 . Many studies state that the size of the catalyst particles determines the diameter of the nanotube or nanofiber, and the duration of the process - their length.

The size of the catalyst particles depends on the diameter of the nanotube or nanofiber, and the duration of the process – is defined by its length.

The fourth chapter is devoted to the creation and application of some polymer compositions based on the obtained carbon nanomaterials. The issues of obtaining quality composite materials based on carbon nanostructures and polyester polyurethane varnish were also studied in the Chapter.

This chapter covers the acquisition of polymer nanocomposites based on carbon nanostructures, urethane and forpolymer-type compositions, their application in many fields of science and technology, including aviation, production of plastic lubricants, oil and gas extraction and other fields. The application of toxic odorless and transparent forpolymer composition is considered separately in the Chapter. The scheme of acquisition and application of forpolymer varnish composition, the scheme and the table of possible application areas of the experimental device and fullerenes for production of chlorinated carbons and carbon nanostructures by the finished chlorination method are given.

Acquisition of modern, high-performance plastic lubricants for high-capacity vehicles and mechanisms, the addition of C_{60} - C_{70} fullerenes and fullerene-containing smoke is one of the most promising, interesting and practical areas.

In the laboratory, a new composition of the flora of mineral lubricant containing fullerene C_{60} has been developed, which is based on the method of catalytic chlorination of low molecular weight hydrocarbons.

Some high-quality materials, corrosion and erosion-resistant coatings have been successfully tested for aviation applications by using various compositions.

The mode of operation of modern aircraft construction products requires a constant increase in the performance of materials. Polymer compositions, which are the basis of protective coatings has significant importance.

Polymer-based compositions consist of two or more components, the high quality of which depends on the binder, which is their main component - the correct choice of polymer.

It should be noted that one of the greatest achievements of the last century was the synthesis and application of composite materials (CM) based on glass, hydrocarbons and chemical elevators. In the 21st century, the synthesis and application of new types of composites - polymer nano-coatings - has become a new phenomenon in the World science and technology.

In 2004, we developed and patented a completely new and unique polyester-polyurethane (PEU) sealing compound synthesis technology in the field of polymer coatings. This new generation PEU is the first patented invention for lacquer coating.

The difference between PEU lacquer coating and other urethane coatings is due to the technology of its production. Thus, for the first time, the hardener acts as an activator in this process.

The reacting polymer (polyester brand MEF-1002) acts as a monomer and is attached to the polymer to be obtained by other manomer-styrene involved in the polymerization process. The result is a PEU-based polymer coating with a heavy molecular weight and a macromolecule arranged in a straight line. This molecular structure has been proven in tests and applications by giving the polymer high quality properties.

One of the advantages of forpolymer varnish composition is that it opens up a wide range of opportunities for the production of new thermostable polymers of flame retardant type, based on the appropriate technology and methods.

Either the PEU or the forpolymer composition based on it surpass their existing analogues in physical, mechanical, chemical and biological properties in all respects. The total number of these indicators is more than 33. From the mentioned indicators, adhesion by continuous cutting method (1 point), impact resistance (50 kg/cm^2), water solubility (no), biological resistance by all indicators (6 points system is 0), working temperature (-70°C + 350°C), chemical resistance in the hardest acid and alkaline solutions (lasts for a month according to GOST), resistance to seaweeds, algae, mussels (does not stick), molecular weight (1600), speed of sound (2000 m/sec), etc.

Laboratory, stand and other tests conducted at the All-Russian Research Institute of Aviation Materials (VIAM) for six months in 2013 are known as PINS-AT (Russia), DINITROL AV-40 (France) and CORBAN-35 (USA) surpassed the highly accepted foreign analogues on almost all indicators.

Similar and similar tests were conducted by the Civil Aviation Institute (Russia) over a six-month period (Table 1). PINS-AT (Russia), ADROX AV-8, AV-25, AV-30, AV-100D, CHEMETALL (Germany) and SOCOPAL (France) analogues were taken for comparison in 2014. Both institutes, after all inspections and tests, noted the high protective properties of PEU varnish composition and offered it for application.

Table 1

	Brand of ingredients

Indicators	*PINSAT*	*AV-40*	*PEUK*	*SORVAN-35*
Cover appearance on the metal surface	Gray, poorly painted coating, dry	Gray, poorly painted coating, with fractures	High transparency weakly painted, dried	Highly bright pink, transparent dry
Cover thickness, mkm	50-60	40	30	35
Initial drying duration, 20 ° C, hours	40 minutes	1 hour	40 minutes	15 minutes
Penetration capacity, mm	30	43,5	40	40
Water repellent capacity, mm	60	90	90	100
Applicable temperature	40 ° - 100° Up to C.	-40 ° - up to 240 ° C	-40 ° - up to 100 ° C	-40 ° - up to 170 ° C

MAIN RESULTS

1. The prospects for obtaining nanomaterials from natural gas and other low-molecular hydrocarbons in finished chlorination and pyrolysis processes are scientifically, technically and technologically substantiated [2,4-6,9,10,12,15,20,22,25].
2. The technology of obtaining fullerenes and nanotubes by finished chlorination and pyrolysis methods has been simplified and their continuous and waste-free production method has been developed. The yield of fullerenes (C_{60} , C_{70}) in the first time finished completed chlorination and pyrolysis processes reached 20-24% [4,5,15,20,21].
3. The structure of fullerenes (C_{60}, C_{70}), endofullerenes and nanotubes obtained in finished chlorination and pyrolysis processes has been confirmed by X-ray and spectroscopic instrumental analysis methods [2,4,5,15,20,21].
4. A composition consisting of a new generation urethane-type polymer coating and fullerene was obtained and patented for the first time [7,10,16,30].
5. Laboratory, stand and flight tests of the polyester-based polymer nanocomposite have confirmed the possibility of its application as a protective substrate in aviation. At the same time, the obtained composition was tested as an anti-erosion, corrosion and paraffin deposition material in oil wells of various oil companies and appropriate application acts were obtained [1-4, 6-8,14,18,19,22,23].
6. For certifying the wide application of the obtained nanomaterials, a technological process for getting a new forpolymer coating of urethane type was created, synthesis of forpolymer nanocomposites based on the achieved forpolymer lacquer coating and carbon nanomaterials (C60, C70 fullerene, singlelayer nanotubes, etc.) was insured, and experimental samples were led [23,25-29].
7. In order to ensure the wider application of the obtained nanomaterials in aviation, a technological process for the production of a new urethane-type forpolymer coating was

created and the synthesis of nanocomposites was organized. At the same time, for the first time, a technology for obtaining a new type of aviation polymer nanocomposite against icing, lightning and pollution has been developed and relevant application acts have been obtained [2,4,7,9,29].

8. For the first time in Azerbaijan, a real scientific and technical base has been created for the production of more than 20 types of carbon, natural minerals and other basic nanomaterials [2,4,5,11,15,20,21,25,26].

List of published scientific works on the dissertation topic.

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5. Н.Ф.Джавадов, З.А.Матысина, С.Ю.Загинайченко, Д.В.Щур, О.В.Мильто. Растворимость фуллерита C_{60} в органической матрице // Наносистеми, наноматеріали, нанотехнології. Институт Металлофизики имени Г.В. Курдюмова НАН Украины. Киев, 2009, т. 7, № 3, с. 671-681.
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