Methods of sample preparation for experimental registration of the size of soot particles in ICE

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Abstract. When analyzing the content of harmful substances in diesel exhaust gases, in particular soot particles, it is necessary to determine the sampling methodology. In addition, this is a determining parameter in the further study of the obtained samples using microscopes. In this paper, we consider options for sampling exhaust gases and methods for preparing the obtained samples for further research. The results of these studies are presented.

1. Introduction
Reducing harmful emissions by reciprocating internal combustion engines (ICE) is an important problem, the solution of which is associated with the improvement of working processes, maintenance systems, the development of methods for neutralizing exhaust gases [1-5]. To date, the composition of the products of combustion of hydrocarbon fuels, with more than 1200 components, has been studied in sufficient detail [2-8].

During the combustion of hydrocarbon fuels, the exhaust gas of an internal combustion engine always contains solid carbon in a dispersed state (soot). Diesel soot is not pure carbon and contains hydrogen, oxygen, volatile, ash, coke compounds. The specific surface area of soot is significant and can reach 300 m²/g. Therefore, the combustion of hydrocarbon fuels leads to an increase in the fraction of radiation in the total heat transfer, which significantly increases the heat stress of the walls of the combustion chamber of a diesel engine and reduces their resource. Sizing of the soot particles contained in the combustion products is necessary not only from the point of view of economic efficiency, but also from the point of view of environmental safety of exhaust gases.

On the basis of the Department of Heat Engines, Automobiles and Tractors of the Vyatka State Agricultural Academy, soot formation processes in ICE cylinders are studied both during the combustion of diesel fuel and alternative fuels. This paper presents the studies conducted for the D-21A1 diesel manufactured by VMTZ. This is a two-cylinder air-cooled diesel engine with a hemispherical combustion chamber with a radius of R=36 mm. Engine displacement of 2,08 liters. Sampling of exhaust gases and soot particles was carried out at nominal conditions with a crankshaft rotational speed n=1800 min⁻¹ and a load pₑ = 0.585 MPa. Two locations for sampling soot particles were selected: at the exit of the cylinder and at the exit of the exhaust pipe. The obtained samples were powdery soot layers on glass plates [9-15].

Further studies were carried out on the basis of the laboratory of modern methods of physicochemical analysis [3], which is part of the Nano-technology research and educational center of Vyatka State University [4].
2. Experimental
To detect the microstructure of soot particles, we used a scanning (scanning) electron microscope JSM-6510LV from JEOL (Japan), based on the principle of interaction of the electron beam with the test substance. An electron gun is a cathode (has a negative potential of up to 30 kV, called an accelerating voltage) that emits electrons. A beam of electrons passes through a lens system of electronic optics. A deflecting system deploys the probe over a given area at the facility. As a result of the interaction between the electron probe and the sample, low-energy secondary electrons are generated, which are collected by the secondary electron detector. Each collision event is accompanied by the appearance of an electrical signal at the detector output. The intensity of the electric signal depends both on the nature of the sample (to a lesser extent) and on the topography (to a greater extent) of the sample in the interaction region. After analog-to-digital conversion and amplification, these signals are visualized using a personal computer. Thus, scanning the surface of the object with an electron beam, a relief map of the analyzed area is obtained. For example, Fig. 1 presents micrographs of soot particles obtained with an increase from ×50 to ×20 000. As can be seen from the photographs, soot is an irregular cloud-shaped formation. Diesel soot is prone to the formation of conglomerates containing from several hundred to several thousand particles. The concentration of soot particles located on glass plates is high, which leads to mutual adhesion and superposition of particles on each other. A further increase in the resolution of the microscope is useless for the prepared samples. Therefore, under the described conditions it was not possible to isolate individual particles and determine their sizes [16-21].

The next stage of research involves the use of a JEM-2100 transmission electron microscope from JEOL (Japan). The device consists of the following main parts: a vacuum system, an electron source, a series of electromagnetic lenses, imaging devices, and also devices for inputting, outputting, and moving a sample under an electron beam. This microscope has high performance and high-contrast electronic optics with a maximum accelerating voltage of 200 kV. The microscope is equipped with a LaB6 cathode of increased brightness, a digital scanning device, a device for changing the angle of convergence of the electron beam to perform studies using the convergent beam method, and a goniometer with piezocontrol of the position of the object at the atomic level. The design of the microscope has increased vibration resistance. Image output is carried out both on the fluorescent screen and on the monitor using a high-resolution CCD camera with an increased field of view. WINDOWS operating system is used to control the multifunctional electronic-optical system of the device and image output [22, 23].

Preparations for research on an electron transmission microscope were prepared according to the following procedure. The initial carbon black samples were cleaned off glass plates, placed in a solvent, and shaken with a mechanical mixer. A formvar substrate was applied to copper grids with a diameter of 3 mm (inner diameter of about 2.5 mm), which was strengthened by carbon deposition. About 0.5 mkl of a suspension of soot particles dissolved in a specific liquid was applied to the prepared grids. After 5 minutes exposure, excess moisture was removed with filter paper. The mesh with the sample was fixed on the table in the holder, which allowed it to be moved along several axes. After drying, the soot preparations were analyzed using a microscope at magnifications of 30 to 120 thousand times and an accelerating voltage of 200 kV.

Thus, the JEM-2100 microscope allows you to study only specially prepared small samples of small size specially prepared for placement in a vacuum.

The study was carried out using four solvents: AI-92 gasoline, carbon tetrachloride, butyl alcohol and acetone.

One of the main objectives of this study was to analyze different types of solvents and identify the most suitable soot for the preparation of samples. In the analysis of each sample obtained with the participation of a particular solvent, photographs of individual fields of view were taken. As a result of one experiment, 17...20 photos with different magnifications were obtained. Taking into account all four investigated samples and four solvent liquids, the total number of photographs was 298. For an example, figures 1–3 show photographs of soot particles obtained for sample № 1 using all of these solvents.
Figure 1. Micrographs of the shape and particle size of diesel soot particles (sample 1) obtained with different magnifications of the JEOL JSM-6510 microscope: a. ×50; b. ×100; c. ×1,000; d. ×5,000; e. ×10,000; f. ×20,000.

By analyzing the obtained photographs, as well as conducting a comparative assessment of solvents, the following conclusions can be drawn. Carbon tetrachloride (CCl₄) has the highest density, so the
photos are cloudy, the individual particles are almost indistinguishable (figure 3). The cost of this solvent is high. Butyl alcohol is the most harmful on the basis of MPC, has the highest hazard class (explosion and fire hazard, toxicity, human effects, danger during transportation, etc. are taken into account), has an increased viscosity, as a result of which particles often overlap each other. The photographs taken for gasoline and acetone are clear, individual spherical particles are clearly visible (figures 2 and 3). Based on other indicators (density, viscosity, cost, etc.), these solvents are the most optimal for studies of the shape and size of soot particles [24-33].

![Micrographs of the shape and particle size of diesel soot particles for sample 1 (solvent - AI-92 gasoline) obtained using a JEM-2100 microscope.](image1)

![Micrographs of the shape and particle size of diesel soot particles for sample 3 (solvent - gasoline) obtained using a JEM-2100 microscope.](image2)

An analysis of photographs taken with a JEM-2100 microscope with low-density solvents (gasoline and acetone) allows us to consider the primary structures that make up soot. They are spherical particles (spheroids) with predominant sizes of 30...40 nm, which are formed in the combustion chamber of a diesel engine. Moreover, some particles can reach sizes up to 80...90 nm.

3. Conclusion
Particle size dispersion allows statistical processing of the results using a JEM-2100 microscope (using the built-in Olimpus statistical analysis program) and the construction of a particle size distribution function formed in the combustion engine of an internal combustion engine. Further movement of soot particles with combustion products along the exhaust path allows you to track the change in particle size of soot due to adhesion and agglomeration [24, 26].

Even in the combustion process, coagulation of soot particles occurs, leading to the formation of secondary and tertiary structures. Soot in the exhaust gases of diesel engines is an irregularly shaped
formation with linear dimensions up to 100 microns (figure 1b). The concentration of soot in the exhaust gas of a diesel engine depends on both the formation process and the burnout process. The study of soot formation processes, including the determination of particle sizes in combustion products, is the main component in determining the effect of various types of alternative fuels on the economy and efficiency of internal combustion engines. The initial information on the dispersion of the particles of the combustion products allows us to calculate the radiation and complex heat transfer, as well as to determine the thermal stress of the walls of the combustion chamber and to predict the wear resistance of engine structural elements.

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