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# METHODOLOGICAL BASES OF STUDYING THE DISPERSION COMPOSITION OF MINE COAL DUST

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# МЕТОДОЛОГИЧЕСКИЕ ОСНОВЫ ИЗУЧЕНИЯ ДИСПЕРСИОННОГО СОСТАВА ШАХТНОЙ КАМЕННОУГОЛЬНОЙ ПЫЛИ

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*Key words:*
coal dust, scanning electron microscopy, dust dispersion, optical microscopy, laser diffraction analysis, sieve granulometric analysis, mine dust, self-dispersing of coal dust.

The methodology of investigating the dispersion composition of mine dust is presented. The methodology is based on modern science-intensive methods such as the method of scanning microscopy and simple methods such as the method of sieve granulometric analysis. Today, the granulometric analysis is given great attention aimed to study the dimensions and aerodynamic characteristics of mine dust particles. The methods are applied separately from each other. Therefore, the paper discusses the most popular science methods gives recommendations on their joint application on the basis of the author's research on the dispersion composition of mine dust. Methods of scanning ectronic and optical microscopy are briefly described. Laser diffraction and sieve granulometric analyzes used to study the composition of coal mine dust are considered. That is chosen to use mine coal dust collected from the surface of hydraulic support racks of cleaning faces and coal dust obtained by the method of forced grinding of hard coal samples of different grades and anthracite as the samples for study. Samples of coal are taken from the working space of the mines of the Pechora, Kuznetsk and Donbass coal basins.
Based on a comprehensive study of methods and analysis of results of studying the dispersion composition, their main advantages and disadvantages are given.
The research methodology is based on physical methods for studying the dispersion characteristics of mine dust. In connection with the fact that one or another method is implemented in one device (installation), then in order to obtain complex data it is proposed to combine them into knowledge-intensive pairs.
The combination in pairs of the equipment allows to study thoroughly both the dispersion composition and morphology of the dust particles, including the surface structure of dust particles if such a task is posed. Selection of samples and processes of preparing samples for research are in the basis of all methods used for obtaining qualitative and reliable scientific results.
As a result of the experimental work carried out to prevent endogenous fires and explosions in the coal mine area, the author proposes to use a comprehensive approach consisting in applying synchronous thermal analysis methods together with methods for studying the dispersion composition of coal dust.

*Ключевые слова:*
угольная пыль, растровая электронная микроскопия, дисперсность пыли, оптическая микроскопия, лазерный дифракционный анализ, ситовой гранулометрический анализ, шахтная пыль, самодиспергация угольной пыли.

Приведена методология исследования дисперсионного состава шахтной пыли, в основе которой лежат как современные наукоемкие методы, например метод растровой микроскопии, так и наиболее простые методы, например метод ситового гранулометрического анализа. В настоящее время гранулометрическому анализу уделяется большое внимание, направленное на изучение размеров и аэродинамических характеристик частиц шахтной пыли, причем методы применяются раздельно друг от друга. Поэтому в настоящей статье рассмотрены наиболее востребованные наукой методы и даны рекомендации по их совместному применению на основе полученных автором работ по изучению дисперсионного состава шахтной пыли. Кратко описаны методы растровой электронной и оптической микроскопии, рассмотрен лазерный дифракционный и ситовой гранулометрический анализы, применяемые для изучения состава каменноугольной шахтной пыли. Образцами для исследований выбрана шахтная угольная пыль, отобранная с поверхности стоек гидравлической крепи очистных забоев, а также угольная пыль, полученная методом принудительного размола образцов каменного угля различных марок и антрацита. Образцы каменного угля отобраны из рабочего пространства лавы шахт Печорского, Кузнецкого и Донбасского угольных бассейнов.
На основании комплексного всестороннего исследования методик и анализа результатов изучения дисперсионного состава приведены их основные достоинства и недостатки.
Методология исследования основана на физических методах изучения дисперсионных характеристик шахтной пыли. В связи с тем что тот или иной метод реализован в одном приборе (установке), то для получения комплексных данных предложено их объединить в наукоемкие пары.
Сочетание в парах указанного оборудования позволит всесторонне изучить не только дисперсионный состав, но и морфологию частиц пыли, в том числе, если будет поставлена такая задача, и структуру поверхности частиц пыли. В основе всех методов для получения качественных и достоверных научных результатов лежат отбор пробы и процессы подготовки образцов для исследования.
В результате проведённых экспериментальных работ для профилактики и предотвращения эндогенных пожаров и взрывов в пространстве угольных шахт автор предлагает применить комплексный подход, заключающийся в применении методов синхронного термического анализа совместно с методами изучения дисперсионного состава угольной пыли.

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## Introduction

An analysis of modern studies in the field of industrial safety showed that emergencies in Russian mining (primarily in coal mines), continue to occupy the main place among the most dangerous phenomena in underground mining [1, 2]. In addition, despite the existing and developed in the industry regulatory and technical measures to improve the safety of production, emergencies related to the death of miners and suspension/termination of the operation of mines occur every five years. Dynamics show that the number of large accidents compared to the previous decade has increased twice [3–8].

In order to prevent emergencies in the industry, a number of coal companies are developing new standards dedicated to ensure the safety. There is an example of Siberian Coal Energy Company JSC who developed the "Charter of Combat with Hazardous Industrial Situations" [9]. However, in case of constant development of mining equipment to increase its productivity, mining and roadheading of deeper seams and as a result, geological and mining conditions that changes for the worse, taking actions are insufficient. In our opinion, due to the growing pressure on the face and increasing depth of production, there is an increase in accidents for such dangerous factors as methane and coal dust. The contribution of methane and coal dust to the emergencies can not be ignored. It is necessary to study some times the physical, chemical and explosive-fire hazard properties of both hybrid methane-coal mixtures and coal dust. We believe that due to explosive and fire hazard properties of coal and coal dust that constantly change (especially while mining down to underlying horizons) they should be given by special attention. In addition, a dispersion composition change of mine dust can lead to the risk of safety of respiratory organs of miners by traditional means of protection. The facts mentioned are confirmed in the papars [10–14]. In addition, it was established in [15] that coal dust tends to self-disperse. It is also established that changes in dust dispersion composition toward decreasing particle size plays an important role in the autocatalytic spontaneous combustion which can lead to a fire or explosion of the methane-coal mixture.

Taking into account the above mentioned, we believe that in order to ensure the fire and explosion safety of production in general and coal mine in particular it is impossible not to use the methods of prevention and control of coal dust explosions based on the laws of chemical kinetics and thermodynamics and a number of other physical and chemical characteristics of mine dust. The assertion we made is based on the analysis of results of research given in [1, 9, 11, 12, 16–22]. It should be noted that the main efforts should be focused on a comprehensive study of chemical processes occurring both on the surface and inside each particle. That kind approach continues and deepens the research of mine dust, carried out by S.B. Romanchenko, who studied the dimensions and aerodynamic properties of dust aerosols. The main results are given in works [15, 23 and 24].

## Objective

The purpose of the paper is to develop a methodology for studying the dispersion composition of coal dust taking into account the modern (innovative) achievements of science and technology that allow to study both the geometric, physical and chemical characteristics of dust particles in comples and reveal the most informative and effective express methods for its analysis.

## Object of study

The objects of the study were:

1) mine dust samples taken from the surface of hydraulic support rails of a longwall (according to recommendations [23]);

2) samples of coal from the Pechorskiy coal basin (Zh-type coal), Kuznetskiy coal basin (coal of Zh, G and DG types) and Donetskiy coal basin (anthracite), taken from the longwal lspace;

3) dispersed coal samples of the types mentioned above.

## Methods and techniques of research

The information given in [22–29] was considered in the research work. Taking into account the scientific and technical information given in references, the following methods for investigating the dispersion composition of mine dust were used:

1) method of scanning electron microscopy (SEM);

2) microscopic method of granulometric analysis;

3) sieve method of granulometric analysis;

4) laser diffraction analysis of dispersed materials.

A scanning electron microscope TESCAN (SEM) with an X-ray spectral analysis system was used in the study. Work on a SEM was guided by the technique of the equipment manufacturer and data stated in papers [15, 30].

Analysis of dust dispersion compositions was carried out using a LEICA DM 4000 series microscope. For visualization of different dust samples on dispersion appropriate techniques of work with the complex were used in accordance with recommendations (microscope LEICA DM 4000 + information processing application software Image Scope Color), experience of other researchers was considered as well [15, 23, 24, 31–33].

Dispersion analysis of hard coal and coal mine dust was carried out using an AS 200 RETSCH screen analyzer and set of RETSCH sieves. For laser diffraction analysis, a Malvern Mastersizer 2000 diffraction analyzer was used according to the method described in the papers [23, 30 и 33–39].

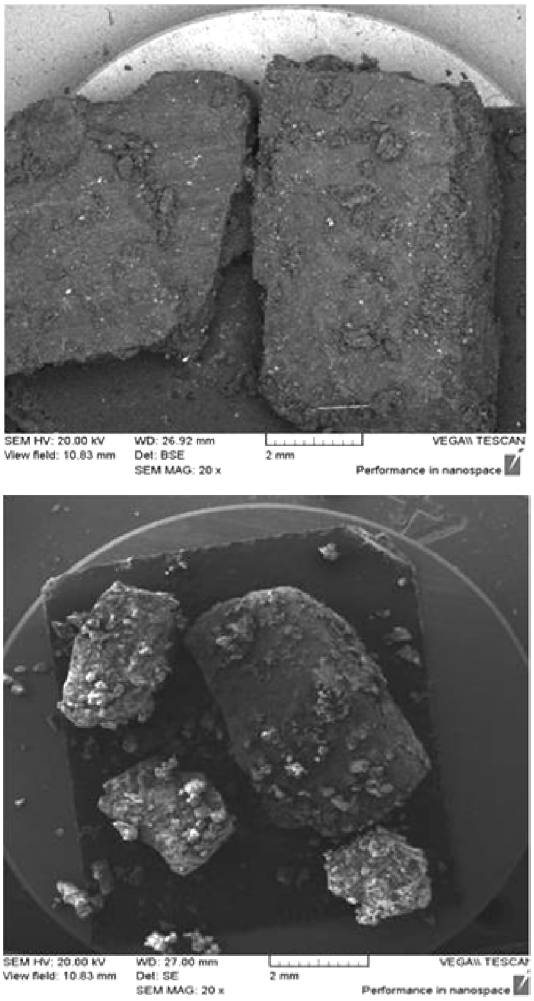
## Results and discussion

Images of the studied coal dust samples obtained by scanning electron microscopy are given in Fig. 1-3. Results were visualized using a TESCAN VEGA microscope with an X-ray microanalysis system.

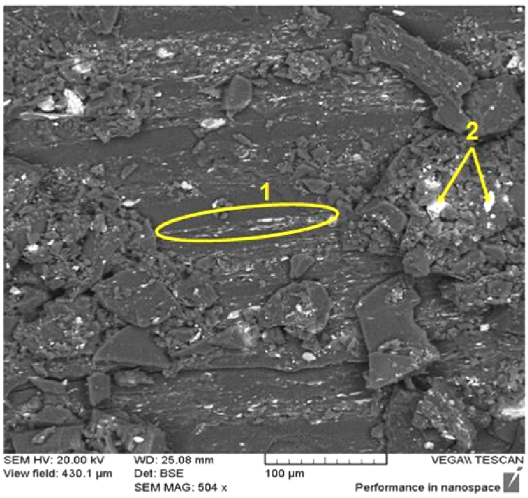
Application software allows processing received images in automatic mode in accordance with the tasks assigned by the operator. The X-ray spectral analysis system allows determining the chemical elements of the focusing zone. In our case, that option allowed finding the location of the silicon in the coal (inner space of region 1 in Fig. 1). We also found that visually distinguished white zones are inclusions and interlayers of silicon in the coal (see Fig. 1*b*), and the areas indicated by the arrows are rock dust (see Fig. 1*b* zone 2).

The main feature of the research methodology is sampling and sample preparation, conducted by well-known standard methods, for example, sampling for analysis was performed by the quartization method.

Fig. 1 shows the particles of G-type coal, glued to a substrate fixed on working tables of a microscope. Smaller dust particles located on the sample surface are clearly visible even at increase in 20 times. Fig. 1*b* shows a zoomed in 504 times fragment of the surface of one of the samples. A structure of the sample being studied is layered




*a*




*b*

Fig. 1. Samples of coal with hard rock dust on the surface: *a* – increase in 20 times; *b* – the increased in 504-times area of one of the samples of Zh-type coal: in the selected area *1* – interlayer/impregnations of silicon; *2* – glowing particles – rock

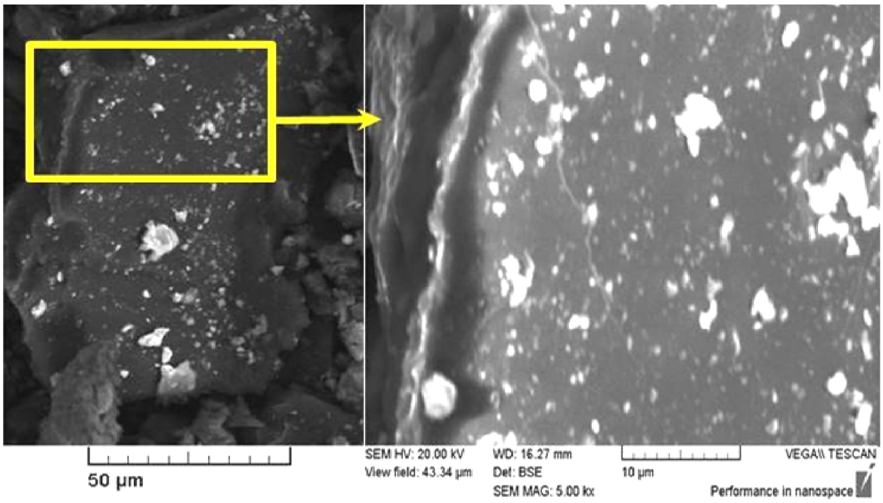
and heterogeneous. The zone 1 shows silicon interlayers, detected and established by X-ray diffraction analysis, and the light sections 2 indicated by the arrows are rock dust.

There are samples of rock dust particles zoomed in 1000 times visible in Fig. 2. Such a zoom is insufficient for analyzing the particle surface. It is clearly seen at 5000 times zoom that there is a crack passes through the surface of the particle. We can assume that, due to the presence of such cracks, the oxidation process can take place practically throughout the entire volume of the coal particle.

We believe that the processes of self-dispersion and all-round accelerating oxidation in conditions of unsatisfactory heat-exchange will ultimately lead to spontaneous combustion. It is confirmed by the authors of [11–13, 15, 22–24, 32, 38, 39] that due to complexity of detailed study of physical and chemical processes leading to smoldering and self-ignition of mine dust it is important to study the coal dust particles by various methods.

It can be seen from visualization of dust samples given in Fig. 1–3 that zoom of 700 times is sufficient to study the appearance, structure and shape of particles.

Such a zoom helps to analyze the sample surface region interesting for us by elemetns, i.e. to determine the chemical element in the structure of the sample. Thus, the analysis of images, some of which are presented in the article, showed that the zoom of more than 700 times is necessary for a more detailed study of the structure and morphology of the sample. However, in order to create a representative (evidence) base, indicating a significant amount of dusty carbon-rock particles with a size of less than 5 microns, for example, as shown in Fig. 3, it is needed to zoom



*a* *b*

Fig. 2. Particles of coal dust with zoom of 1000 times (*a*) and 5000 times (*b*)

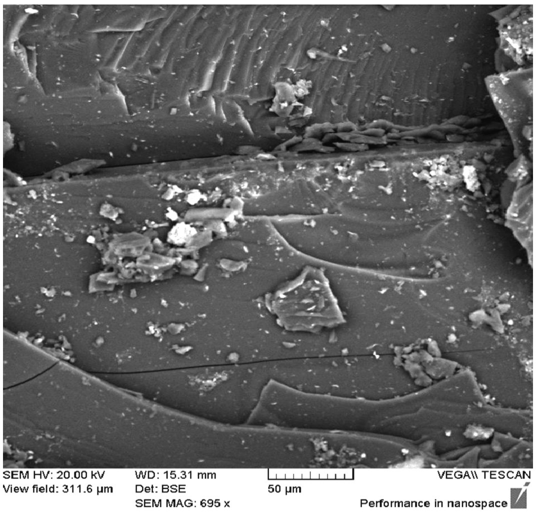


Fig. 3. Surface of a D-type coal sample zoomed in 695 times

in 1000 and 5000 times. The zoom of more than 5000 times is only necessary for studying the internal structure, i.e. study of the structure of dust particles and inclusions.

Based on our studies with scanning electron microscopy, the conclusions below can be drawn.

Advantages: the method is innovative and informative, meets almost all requirements to study both the dispersion composition of coal and hard coal particles and their morphology as well.

Disadvantages:

1) high cost of equipment and its maintenance are disadvantages of the method. The equipment exists only in large and/or advanced institutions of higher education, research institutes and laboratories;

2) high requirements for the qualification of personnel working on equipment;

3) in order to obtain qualitative results, it is necessary to carry out studies that require careful preparation of samples and strict adherence to the methods of operation at SEM.

Fulfillment of this condition is necessary to make any changes to an analysis process depending on the results obtained. Microscopic method for studying the granulometric composition of mine dust samples is an addition to the method of scanning electron microscopy only for the purposes of dispersion analysis.

Study of the dispersion composition of coal and mine dust was carried out on a LEICA DM 4000 microscope equipped with a digital camera. The images obtained were processed in the Image Scope Color application software. Some of the results obtained by us are shown in Fig. 4 and 6.

The figures show examples of the optical image of a microscope processed by software methods. Such a processing is necessary for statistical and geometric analysis of dust particles. An example of results of data processing is shown in Fig. 5.

A picture of coal dust particles taken from the hydraulic rack of the mechanical support of the mining section of the coal mine and processed in a special software, after which a particle size analysis was carried out is given below.

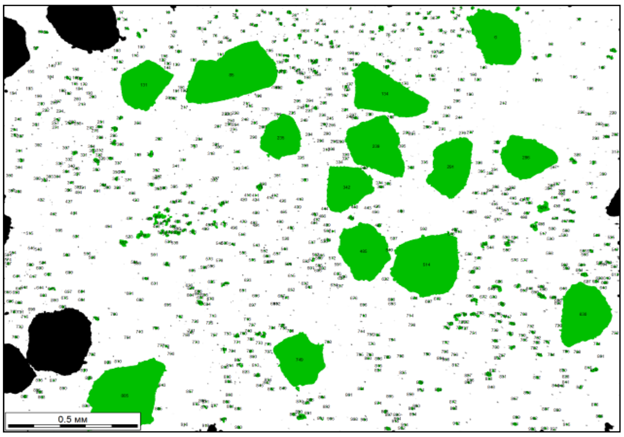



Fig. 4. Example of image analysis in the software Image Scope Color (the picture shows a scale of 0.5 mm)

In this case Fig. 4 shows the intermediate result of the analysis of visible field image of the sample with fractional composition of 140–200 μm. Despite the fact that during the sieve analysis static electricity was removed and sample of 100 g was taken, numerous dust particles much smaller in size than the particles of the studied fractional composition are observed on the photo.

The particles located at the image boundary and particles (see Fig. 4) in the lower left corner, which are in contact with them, were not subjected to analysis. In this case, when analyzing and comparing the image data with the image in the microscope eyepieces, it is possible to determine the separation of particles among themselves. The researcher can directly check in the eyepieces of the microscope and on the monitor screen whether the image is a particle conglomerate or single particle. The microscopic method of study is more labor-intensive in processing than other methods (SEM and laser diffraction analysis). The optical method of microscopic analysis should be applied, depending on specific problems, as the main or auxiliary method for studying mine dust particles. The result of software processing of a single shot of coal dust is given below as an example. Application software for the optical microscope LEICA DM 4000, Image Scope Color, allows to obtain complex characteristics of coal dust in the form of data presented in the table. Using the software complex, it is possible to measure not only the equivalent diameter, but also other geometric characteristics of the samples (length, width, area and others). The analysis of the data obtained is carried out in an automatic mode. In other words there was mathematical processing of the received data array performed. Results of determining the minimum and maximum particle size, dispersion and root-mean-square deviation are grouped to a table, an example of which is shown below (Fig. 5).

| Parameter measured | Diameter of eq. |
|---|---|
| Number of measurements | 869 |
| Minimum value | 0.0228 mm |
| Maximum value | 0.923 mm |
| Average value | 0.0787 mm |
| Dispersion | 0.00346 mm$^2$ |
| Mean-square deviations | 0.0588 mm |

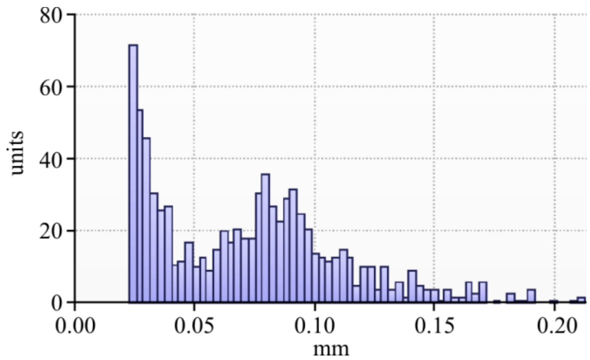



Fig. 5. Example of the variance report table

In order to obtain reliable data, depending on the particle size, it is recommended to investigate at least 500 particles. A sufficient number is the number of particles equal to 2500 pieces [23, 26, 34, 38]. Results of the variance analysis for the same fraction, but with the calculation of a different number of particles are shown in Fig. 6.

On the basis of our studies performed with optical microscopy, the following conclusions can be drawn.

Advantages:

1) the method is sufficiently informative, meet almost all the needs for studying both the dispersion composition of coal and hard rock particles and their morphology. The advantages mentioned above are achieved by the presence of special lens allow to increase the particle in 50, 100, 200, 400 and 600 times; to study a particle in a different light; high-resolution digital video camera and capabilities of a specialized image processing software Image Scope Color;

2) availability of training in working with techniques on equipment and using software;

3) practical experience of studying dust particles showed that in some cases, in particular the comprehensive study of particles larger than 63 μm (for Zh, GZh-types of coal of 100 microns), instead of using the microscope, a high-resolution digital camera equipped with a macro lens can be implemented. Using the Image Scope Color for image processing software allow in some case to get even better results, than on a microscope. In addition to the optical microscope LEICA DM 4000, a digital camera Sony alfa-5000, macro lens Sony 30 mm f/3.5, macro- and LED lighting Aputure Amaran AHL-N60 were applied by the autor during the study of coal dust particles. Besides, a digital microscope Digital Microscope with a 5-megapixel camera and 300-fold magnification was used above equipment mentioned to study fractions over 100 microns.

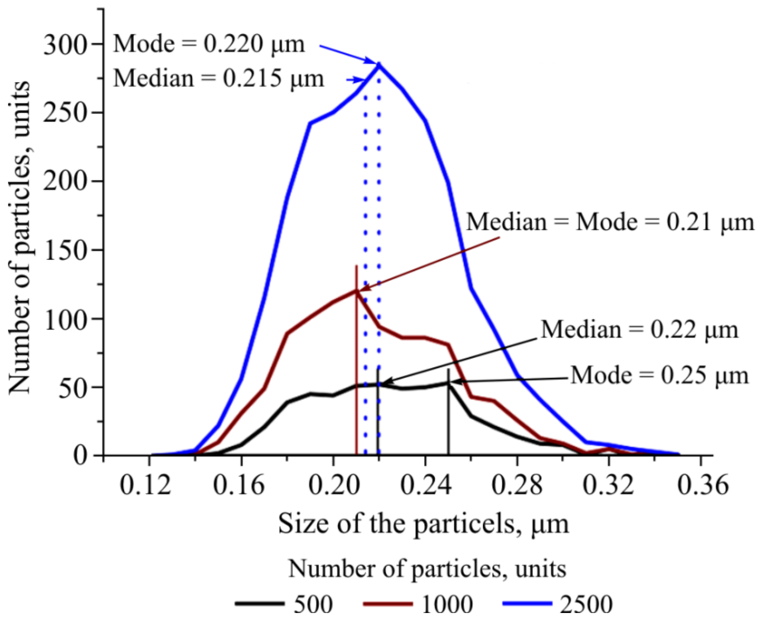



Fig. 6. Dynamics of change in the dispersion analysis as a function of number of particles

Disadvantages:

1) existing limitations on the study of fractions less than 63 μm;

2) it takes time to get skills and work algorithm in order to get high-quality results;

3) a lot of labor, it is required to receive and process up to 2500 pieces of dust, for which it is necessary to make about 200 shots.

To our mine, third place for informativeness is taken by the sieve method of granulometric analysis. The method is somehow often used as a preparatory sample before the studies with use of electronic or optical microscopes.

In particular, using this method we managed to establish that for coal types of various deposits considered in this article fractions of less than 140 μm account for more than 50 % of the sieving mass. The main results are shown in Fig. 7.

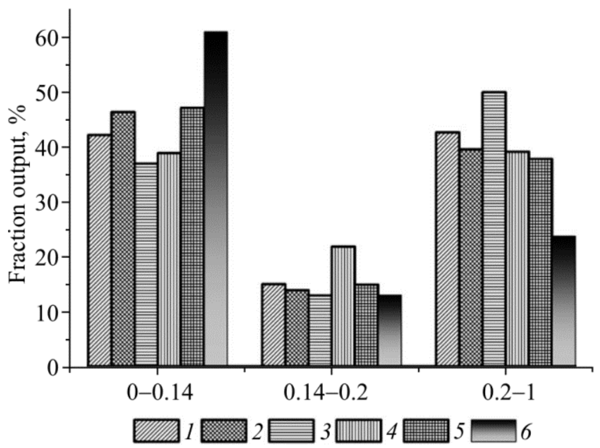



Fig. 7. Results of sieving granulometric analysis of coal dust with a dispersion of less than 1 μm

It can be seen from the data given in Fig. 7 that depending on the grindability of coal, the proportion of large fractions with a dispersion of 0.2–1.0 mm is from 26 to 50 %, fractions of 0.14–0.2 μm – from 12 to 20 %, and the rest is accounted for fraction 0-0.14 μm, i.e. 37–60 %.

The granulometric analysis performed in the study allowed making the following conclusions.

Advantages:

1) availability of equipment and ease of use;

2) the possibility of obtaining the result only with help of a set of sieves, i.e. manually;

3) dry and wet granulometric analysis.

Disadvantages:

1) a qualitative dispersion analysis is impossible, sieving for certain fractions is only possible;

2) granulometric analysis by a dry method is applicable only for fractions with a dispersion of 63 microns, carrying out a granulometric analysis by the method of dry screening with a dispersion of less than 63 becomes inexpedient in terms of labor costs and reliability of the results obtained.

The fourth place in terms of largest number of solved problems is taken by laser diffraction analysis, which was performed with the help of the Malvern Mastersizer 2000 laser diffraction analyzer and software made for it. The fraction with dispersion of 0–200 μm was the first subject to laser diffraction analysis. Then a sieve granulometric analysis was performed with obtaied fractions 0–45, 45–63, 63–94, 94–125, 125–140 and 140–200 microns. Each fraction was analyzed separately. The results are presentedin Fig. 8.

The method implemented with help of Mastersizer 2000 is necessary at the stage of testing the modes of the sieve granulometric analysis. It serves as a kind of passport for the quality of sieve screening of the sample. With help of that it is necessary to determine the maximum mass of the sample to be sieved and select the optimal mode of operation of the sieve analyzer. In the first case, this will reduce the amount of fine fractions. In the second case it reduces the number of reoriented larger particles that have slipped through the holes of the screenand in the second case if the vibrator and shock forces of the analyzer are properly selected.

Based on the experience of work at the Malvern Mastersizer 2000 following conclusions can be made.

Advantages:

1) any researcher can master the work on the equipment, i.e. we note the extreme simplicity of the operations necessary for performing the analysis;

2) highly informative and reliable method, since not single particles are studied, but the volume of a sample in which such particles can be more than $10^{12}$ pieces.;

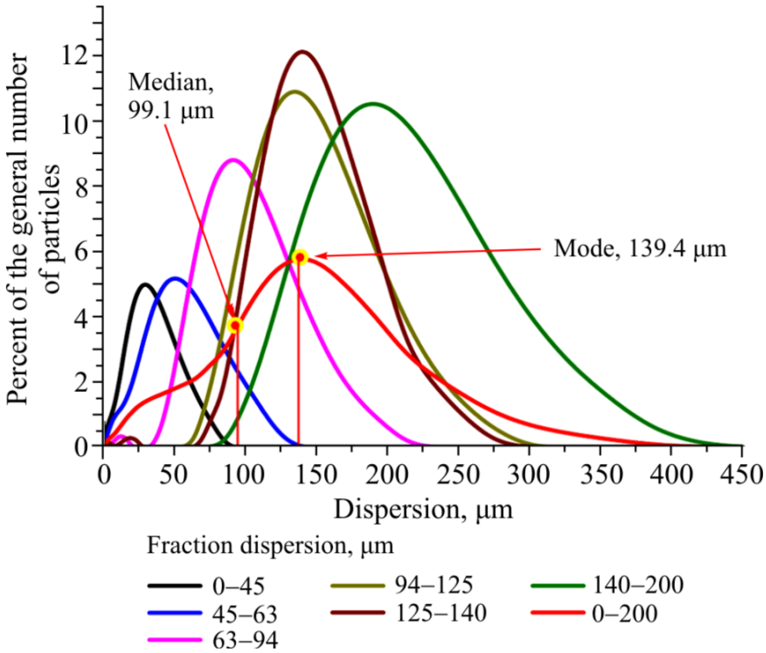



Fig. 8. Graphical display of results of analysis of anthracite coal dust, performed using the Malvern Mastersizer 2000

3) dry and sol (particles in an water medium) matter analysis. Application of "wet" analysis is especially important when studying the dispersion composition of mine or coal dust fractions with a size of less than 63 µm;

4) the simplest way to assess the quality and drill the methodology for implementation of a sieve granulometric analysis, which allows to reduce the labor costs for carrying out the dispersion analysis of dust particles significantly, in comparison with the microscopic study method.

Drawbacks of the method are as follows: it can be used to study only the dispersion composition of particles (size), and therefore it servs as an alternative method for determining the dispersion composition. A granulometric analysis can be used as a control method for particle size analysis.

For each of the analysis methods discussed above, there is a technique described in the equipment operation manual, but the initial stage of the determination of dispersion and other characteristics of mine (coal-rock) hard coal dust for all methods is the same.

Initial stage of any research work is sampling and sample preparation.

Extraction of mine dust samples was carried out according to the recommendations set forth in [23], in particular from the surfaces of hydraulic stands of mechanical longwall support. Taken samples were loaded on site (in the working space of a longwall) in sealed containers. After a container was closed, the contact with the external environment is impossible, i.e. there is no access of air inside the container. Sample preparation included an external examination of the sample after opening the container, drying in an autoclave to an equilibrium state and sieve sieving. Samples passed through a sieve with a hole diameter of 1 mm were analized. Since in our case it was not necessary, the samples were not sieved when conducting studies with a scanning electron microscope.

## Conclusions

1. Base on the research work carried out with help of various methods of studying the dispersion composition of mine dust of scientific research, we believe that the compilation of two mutually complementary methods should be based on the methodology for the comprehensive study of mine dust. In our opinion, such pairs can be presented by:

– laser diffraction analysis + microscopic method of investigation; in this case both the dispersed composition and structure of grains are reliably studied;

– sieve granulometric method + microscopic study method. The combination of these methods, just like in the previous case, will allow to comprehensively study mine dust. The difference between them is only in a larger number of operations to obtain reliable information (up to 250 images may be analyzed), i.e. labor intensity.

2. Studies of the dispersion composition of dust showed that in order to prevent and control both the occurrence of endogenous foci and explosions of methane-coal aerosol dispersions, in addition to methods for studying the dispersion composition and properties of mine dust, an integrated approach should be used. Such the approach as compilation of possibilities of disperse analysis and synchronous thermal analysis allows to answer the questions of chemical kinetics and thermodynamics (responsible for combustion and explosion processes that arise, including the space of coal mines).

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Просьба ссылаться на эту статью в русскоязычных источниках следующим образом:

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