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# DETERMINATION OF PROGRESSIVE RESEARCH METHODOLOGY OF USING MODERN MEASURING DEVICES TO DETERMINE PHYSICAL, CHEMICAL AND MINERALOGICAL PROPERTIES OF RAW MATERIALS AND MINERAL WASTES

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Currently, in science, rapid development in the use of new research methods takes place. Examples of these methods are scanning electron microscopy, x-ray diffraction analysis, thermal analysis performed using derivatograph, grain size analysis performed using laser particulate measuring instruments, density measurements using gas pycnometers and measurements of specific surface area carried out using the Blaine detector. These methods can be used in almost any field of science dealing with solids. Modern methods are also used in studies of properties of raw materials and mineral wastes. Further studies, using the above and other advanced testing methods will create comprehensive testing procedures for mineral raw materials and waste.

The paper presents progressive research methodology of using modern measuring devices to determine physical, chemical and mineralogical properties of fly ashes. In the theoretical part the characteristics of different research methods and the principles of operation of test equipment were described. In the practical part the methodology and exemplary results were presented.

keywords: fly ash, mineralogical properties, x-ray diffraction, x-ray microanalysis; thermal analysis, laser size analysis; gas pycnometery, surface area

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#### 1. INTRODUCTION

So far in the laboratory studies of physical and physicochemical properties of raw materials, mining waste and power industry waste traditional research methods were used (such as petrographic and mineralogical studies, sieve analysis, enrichment analysis, determination of magnetic and electrostatic properties). In addition, to determinate coal properties extra research methods were used (such as determination of moisture, ash content, sulphur content, volatile matter content, analysis of heating values).

Scientific development, which took place in the last century in many fields of science dealing with solids, allowed to create more accurate methods of research. These methods can be used in studies of properties of raw materials and mineral waste. The results of such tests are used to assess feasibility of enrichment of raw minerals, technological processes and feasibility of utilization and treatment of waste.

Among these methods of measurement, the greatest potential for use has X-ray diffraction analysis, scanning electron microscopy, X-ray microanalysis, thermal analysis, grain size analysis, gas pycnometery and Blaine analysis. These methods have many applications. A few of them are presented below.

Scanning Electron Microscopy (SEM) and Energy Dispersive Spectrometers (EDS) were, for instances, used to analyze the solid reaction products of reduction of ilmenite pellets under a  $CO-N_2$  gas mixture. With these methods metallic Fe with small amount of Ti dissolved in it and titanium oxides were found in the solid reaction products. However, no formation of nitride phases was found under most conditions (Zhao et al., 2008).

X-ray diffraction (XRD) and differential thermal analysis (DTA) for instance were used to identify physical properties and mineral and chemical composition of bentonite from different calcium bentonite deposits in Turkey. The bentonite was used to produce desiccant clay (Bulut et al., 2008).

Laser particle size analysis was used for high resolution on-line measurement of particle size distribution in highly concentrated ore slurries (Jenny et al., 2008).

Blaine specific surface area tester, laser size analyzer, scanning electron microscope (SEM) and X-ray diffraction analyzer (XRD) were used to determinate the working mechanism of High Pressure Grinding Rolls (HPGR) and to optimize grinding, balling and firing processes parameters (Pan et al., 2008).

A wide range of publications point to the fact that modern research methods are widely applicable, not only in mineralogy and geology, but also in raw materials and mineral waste properties studies (Mo and Fournier, 2007; Erol et al., 2007; Kutchko and Kim, 2006; Maenami et al., 2004; Chaowasakoo and Sombatsompop, 2007; Styszko-Grochowiak et al., 2004; Bouzoubaâ et al., 1998; Sarbak et al., 2004; Blaha et al., 2008; Inaba and Matsumoto, 1999; Zaenia et al. 2010). Further studies, using these

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methods, will create comprehensive testing procedures for mineral raw materials and waste on laboratory scale. Then, these procedures can be applied for in situ measurements of variable properties of raw materials, products and waste on industrial scale. The test procedure will be helpful in continuous evaluation of the properties of raw materials, products and wastes in processing plants. This will allow to improve enrichment technology, to increase the yield of useful components and to find the most economic means of disposal of waste.

#### 2. METHODS AND MATERIALS

In this paper, systematics of modern research methods and opportunities of using these methods to determinate physical and chemical properties of raw materials and mineral waste is presented. Characteristics of these methods as well as the possibility of their use to measure and quantify properties of mineral waste materials are shown on the example of fly ashes.

#### 2.1. METHODS

#### 2.1.1. STRUCTURAL AND PHASE X-RAY DIFFRACTION ANALYSIS (XRD)

In this method, X-ray of known wavelength is used to test unknown crystalline phases. In X-ray phase analysis the most frequently used technique is the powder method, performed using diffractograms (X-ray apparatus). Polycrystalline samples in the form of powdered mineral or rock and monolithic samples of fine-grained and fine-crystalline minerals (i.e. fly ash), are tested by this method. This method is based on an assumption that a sample consists of grains oriented in random way. A limited number of these grains is located relative to a monochromatic X-ray beam that falls on the sample, in such a way that the specified crystal planes (hkl) satisfy the Bragg condition for wave interference. Each crystalline phase is attributed to crystal planes that are filled in a specified way with atoms or ions forming that phase. Each substance is characterized by a corresponding deviation from the original direction of incidence of the interference beam. The interference beam is recorded on a film (film technique) or by using a Geiger or scintillation counter (diffractometer, counter technique). The result of the test, using the powder method, is a record of spectra in form of diffraction pattern. The diffraction pattern plots the diffractive extremes. It is the basis for the identification of the crystalline phases contained in the sample. The diffraction pattern is obtained in numerical and graphical forms. The mineral composition of the sample and the percentage of individual phases are identified

automatically using appropriate software that includes directories of characteristic values for each phase. The result of testing is a complete qualitative and quantitative analysis of the mineral composition of tested samples (Kędzior et al., 2003; Ahuja and Jespersen, 2009; Chancey et al., 2010; Jiang et al., 2007; Koukouzas et al., 2009).

#### 2.1.2. SCANNING ELECTRON MICROSCOPY (SEM)

It is currently the most common modern method used for observing and testing microstructures. In the scanning microscope, an electron spot beam bombards the sample. The beam produces emission of secondary electrons through a linear sweep of the sample's surface. Thus, the sample emits a variety of signals. The signals are recorded by detectors and converted sequentially into an image of the test samples or X-ray spectrum (Goldstein et al., 2003).

Signals that generate information about the properties of the samples can be divided into groups: secondary electron emissions (topography, morphology andcrystalline structure of the sample, distribution of potential and intensity of electric and magnetic fields in the sample); backscattered electron emissions (topography and morphology of the sample, the distribution of magnetic domains in the sample, composition contrast); auger electron emissions (chemical analysis of surface layers of the sample, measurement of local potentials in the sample); cathodoluminescence (recombination processes in material, identification of impurities, additives and structural heterogeneity) and characteristic X-ray emission (qualitative and quantitative chemical analysis of the material) (Goldstein et al., 2003; Krawczykowska, 2007; Gomes, et al., 1999; Hsieh and Tsai, 2003; O'Keefe et al., 2000; Sarbak et al., 2004; Słówko et al., 2002).

#### 2.1.3. COMPLEX THERMAL ANALYSIS

This is a two step method. The analysis is performed using derivatograph. In the first step, changes in mass of the sample during the cooling or heating are measured as a function of time. As a result of this measurement graphs showing the curves of DTG (Differential Thermogravimetric Curve) and TG (Thermogravimetric Curve) are plotted. The second step is called Differential Thermal Analysis (DTA Curve). In this step the difference between temperature of the test sample and a reference substance, in relation to time or temperature, is measured. The samples and the reference substance are in the same conditions, and their heating or cooling is strictly controlled. Comprehensive thermal analysis methods allow to trace the reactions taking place at pre-selected temperature intervals. The type of the reaction (dehydration, oxidation, degradation, polymorphic transformation, sintering, melting, etc.) can be specified as a result of the interpretation and comparison the DTA, DTG and TG curves. The

analysis of the TG and DTG curves allows for accurate quantitative determination of this reactions and processes (qualitative analysis and quantitative assessment of processes). This method provides information on crystallization of phases, recrystallization of minerals and compounds, amount of heat needed for the synthesis of products, quality and quantity of newly formed compounds or loss in weight of the material (Nocuń-Wczelik, 2003; Guo et al., 1998; Moreno et al., 2005; Wyrwicki, 2004).

#### 2.1.4. GAS PYCNOMETERY DENSITY ANALYSIS

This method is used to determinate accurate density of the test materials. Density is determined by the measurement of the weight of the sample (carried out by a analytical balance) and the measurement of the sample volume (performed by a gas pycnometer). In gas pycnometry, the volume of solids is determined by measuring volume of gas, which is repressed by the test sample, from a calibrated measure. The substances used as a medium have properties similar to the properties of perfect gas. These substances neither react with the test material nor adsorb on the material surface (e.g. helium) (Ahuja and Jespersen, 2006; Thipse et al., 2002).

#### 2.1.5. BLAINE SPECIFIC SURFACE AREA ANALYSIS

Measurements of the specific surface is carried out by the Blaine detector. In this device the gas flow through a layer of compacted formulation is measured (method of permeability). This method is based on the properties of laminar (viscous) flow (Poiseuille-type) through a porous layer. Measurement is performed for two substances - the standard sample and the test sample. The results are relative and depend on the standard sample used in the experiment. To obtain the absolute value of the specific surface area it is necessary to make additional calculations (Rymon-Lipiński and Zborowski, 1978; Kordek et al., 2005; Tosun, 2006).

#### 2.1.6. LASER PARTICLE SIZE ANALYSIS

Grain size analysis, performed using a laser particulate measuring instrument, gives a full assessment of the composition of the fine-grained sample. Particle size analysis, in the device of this type, is based on measuring the intensity of coherent laser light scattering by the grains in suspension. When the laser light encounters the population of grains, the volumetric distribution of grain sizes is expressed by the intensity of light scattered on them. Refraction angle of the laser beam is higher on smaller grains than on larger grains. Diffraction pattern, which is formed in that way, is identified by D.K. Szponder, K. Trybalski

a system of sensors, while the received signals are used to calculate the particle size distribution (Trybalski et al., 2004). During particle size analysis, the frequency of occurrence of selected size grades is determined. The results of particle size analysis are presented as tables or graphs (e.g. histogram - the percentage of the grains which sizes are contained in the selected size grades; cumulative curve - is a continuous function, that shows the contents of particles with diameters smaller or larger than the selected diameter D in the tested material; particle size distribution curve – differentiate cumulative curve, equivalent of probability density). In order to characterize the size distribution of grains (similar to Gaussian distribution) such parameters as average value (median), standard deviation and modal value (mode) are defined. When the particle size distribution is different than Gaussian distribution, additional parameters such as asymmetry factor and flattening coefficient are determined (Bolewski et al., 1981; Jones et al., 2006; Styszko-Grochowiak et al., 2004).

# 2.2. MATERIALS

Fly ash is an artificial puzzolana that is produced as a result of combustion of coal. It leaves a pulverized, fuel-fired furnace with flue gases. It takes the form of fine mineral dust from light to dark grey in colour and consists primarily of silicon oxides, aluminium oxides and iron oxides. In addition, it contains a variety of trace elements and small amount of unburned carbon (Kasprzyk and Pietrykowski, 2007).

Fly ashes from Power Plant BOT Opole SA (sample designated as Sample O) and from Thermal-Electric Power Station "Cracow" SA (sample designated as Sample L) were used in the studies. Samples were collected from the fly ash retention tanks. Samples (approximately 1 kg) were collected with a probe (with an inside diameter of 80 mm and a length of 1,500 mm), from the hopper of retention tank, at the time of gravitational movement of material. The collected samples were an averaged mixture of ash produced in these plants.

#### 3. RESULTS AND DISCUSSION

This publication aims to systematize the knowledge on modern research methods and measuring equipment that is used to determine the physical, chemical and mineral composition of solids. It also shows their applicability to measure properties of various types of raw materials and mineral waste. Therefore, the samples of different types of the fly ashes were examined. Based on laboratory studies, usability of these research methods and measuring equipment to determine the properties of raw materials and mineral waste was evaluated.

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#### 3.1. CHARACTERISTICS OF MINERAL COMPOSITION

#### 3.1.1. STRUCTURAL AND PHASE ANALYSIS - X-RAY DIFFRACTION ANALYSIS (XRD)

To determine the mineral composition of the samples the X-ray phase analysis was used. The powder method was used because sample contained only small particles. The results of X-ray analysis of Sample L and Sample O are shown in Table 1 and Table 2.

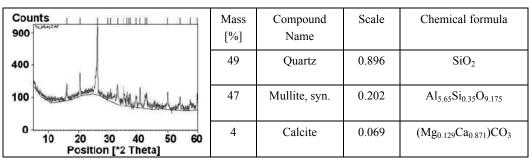


Table 1. Mineralogical composition of fly ash by X-ray analysis - Sample L

Table 2. Mineralogical composition of fly ash by X-ray analysis - Sample O

Counts 400	Mass [%]	Compound Name	Scale	Chemical formula
L i had i i	25	Quartz	0.906	SiO <sub>2</sub>
100 Manufactor Contraction of the second second second	6	Hematite	0.199	Fe <sub>2</sub> O <sub>3</sub>
	62	Mullite, syn.	0.501	Al <sub>4.984</sub> Si <sub>1.016</sub> O <sub>9</sub>
0 10 20 30 40 50 60 Position [*2 Theta ]	7	Dolomite	0.195	CaMg(CO <sub>3</sub> ) <sub>2</sub>

It can be concluded that the fly ash from Sample L belongs to group of siliceous fly ashes containing a significant amount of aluminium compounds. The main component of this fly ash is silicate that occurs in the form of quartz (gangue) and mullite (aluminosilicate, carbonaceous shale and refractory clay. Quartz in fly ash has similar use as natural mineral aggregates of similar composition, while mullite makes fly ash highly flame resistant. In addition, mineralogical composition of this fly ash is supplemented by calcium carbonate and magnesium carbonate, that affect its binding properties. Sample O belongs to the siliceous-aluminum ashes group, which contains small quantities of calcium in the form of dolomite. It consists of a large quantity of synthetic mullite, which was formed as a result of burning coal that contains mainly aluminosiliacates, and slate and clay as the waste rock. Another component of the sample that is present in large quantities is quartz. It is a component of many types of gangue, with different characteristics and origins. The test sample contained also dolomite, which affects its binding properties. In addition, the composition of the fly ash is supplemented by a certain amount of iron in the form of hematite, which is a valuable component of the supplemental material for the production of clinker, cement and ceramics.

As it was demonstrated in this studies, X-ray analysis, carried out by diffraction patterns, can be used to accurate determine mineralogical composition of almost any raw mineral or mineral waste. However, tested substance must be in form of powder. The result of testing is a complete qualitative and quantitative analysis of the mineral composition of samples.

#### 3.2. CHARACTERISTICS OF CHEMICAL COMPOSITION

Chemical properties of samples were determined by using two research methods – scanning electron microscopy and thermal analysis.

# 3.2.1. ELEMENTAL COMPOSITION AND SURFACE MORPHOLOGY - SCANNING ELECTRON MICROSCOPY

To determine the elemental composition and morphology of samples scanning electron microscopy (SEM) was used. Figures 1 to 8 present the scanning microscopic images and diffraction patterns of samples of fly ashes.

The scanning microscopy proved that Sample L and Sample O are fine-grained fly ashes (grain 1 - 100  $\mu$ m) (Figs 1 and 2). These fly ashes contained grains of various sizes and shapes. The shape and size of grains depend on its chemical composition. Both samples are dominated by spherical grains with a smooth surface (pirospherical (Fig. 3), cenospherical (Fig. 4), and plerospherical (Fig. 5)), single (Fig. 4 and 5) or combined into agglomerates (Fig. 3). In Figures 3a and 3b, the results of point microanalysis performed on the surface of this kind of grains (No. 1 and No. 2 in Fig. 3) are plotted. They consist mainly of silicon, aluminium and oxygen (mineralogical composition – silicate glass, quartz - SiO<sub>2</sub>, mullite -  $3Al_2O_3$ ·2SiO<sub>2</sub>).

Other grains can be divided into three groups. First contains grains of irregular shape, which cement spherical grains or form a crust on their surface. This grains are composed of magnesium carbonate, calcium carbonate, sodium carbonate, potassium carbonate (calcite and dolomite) (Figs 6 and 6a). The second group of grains contains spherical grains made of crystals with a structural fabric. It contains iron, titanium and other metals (hematite) (Figs 7 and 7a). In the last group irregular in shape, very porous with rounded or sharp-edged borders, grains can be found. Such grains contain unburned coal (Figs 8 and 8a).

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These studies show that the scanning microscope with an X-ray microanalyzer can be used to obtain information about sample morphology and grains amount, type, shape and elemental composition. These factors have a significant impact on properties of the sample. These measurements can be used for all raw materials and mineral wastes, not only in the powder forms, but also in the form of microsections or cuts. Due to this analysis, the elemental composition, the chemical and physical properties of almost all minerals, can be determined.

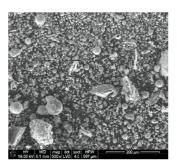


Fig. 1. General view of Sample L

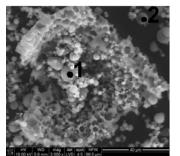


Fig. 3. Agglomerate- Sample O

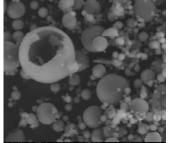


Fig. 4. Cenospherical grain – Sample L

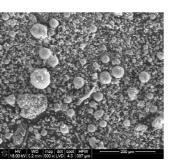


Fig. 2. General view of Sample O

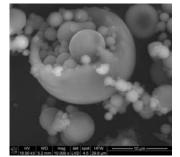


Fig. 5. Plerospherical grain – Sample O

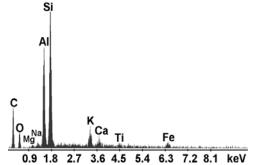
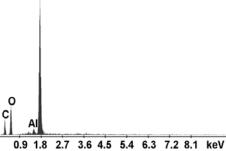
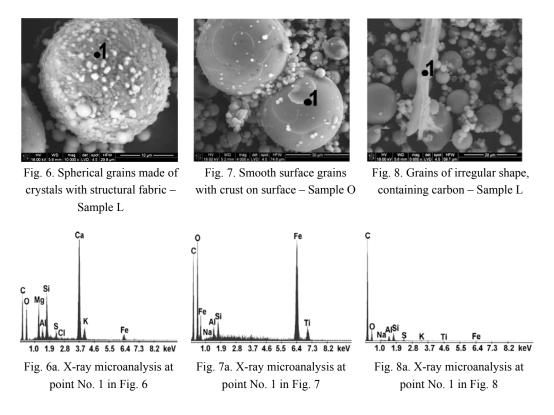


Fig. 3a. X-ray microanalysis at point No. 1 in Fig. 3



Si

Fig. 3b. X-ray microanalysis at point No. 2 in Fig. 3



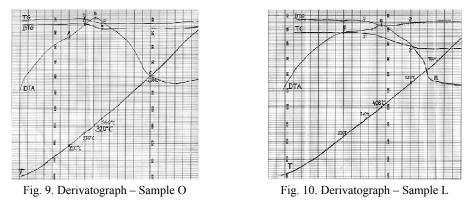
# 3.2.2. QUANTITATIVE AND QUALITATIVE DETERMINATION OF CHEMICAL PROCESSES -THERMAL ANALYSIS

Thermal analysis was used to determine chemical reactions taking place during cooling or heating of the sample. The results of thermal analysis of Sample L and Sample O are shown in Figs 9 and 10.

Based on the analysis of the results obtained in studies using derivatograph, during heating of the test sample, chemical reaction of oxidation of residual carbon occurred producing carbon dioxide ( $CO_2$ ). The percentage of carbon remaining in Sample L, after combustion in power plants, is 3.5 wt% of fly ash. The percentage of carbon remaining in Sample O is 3 wt% of fly ash.

Research, carried out by this method, allows tracing the reactions taking place in the material during cooling or heating. A comparison and interpretation of DTA, DTG and TG curves provided information about the type of reaction that takes place. The analysis of the TG and DTG curves was used to determine quantitatively this reaction. The behaviour of raw materials and mineral wastes under different conditions of temperature, their frost resistance and their behaviour during combustion or burning,

can be specified through this analysis. These factors are very important and they are used on a wide scale in selection of ways and methods of utilization of raw materials and mineral wastes.



3.3. PHYSICAL PROPERTIES

Basic physical properties of the samples were determined by using research methods such as density analysis (gas pyctometer), specific surface area analysis (Blaine detector) and grain size analysis (laser particle size analyzer).

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3.3.1. DENSITY ANALYSIS - GAS PYCTOMETER
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The density of the samples was determined by measuring weight of the sample (carried out by analytical balance) and the measurement of sample volume (performed by helium pycnometer). The results of density analysis of Sample L and Sample O are presented in Table 3.

Sample	Density, g/cm <sup>3</sup>	Specific surface area, cm <sup>2</sup> /g
Sample L	2.2529	3233.7
Sample O	2.1847	3523.5

Table 3. Density and specific surface

Both samples have similar densities. The density results from chemical and mineralogical compositions of the tested samples. The value is closed to the average densities of minerals that build the investigated ashes.

The measurement of density is a basic test carried out for all the waste and raw materials used in building industry, mining industry as well as in mineral processing. This test is often performed and it allows to select an appropriate composition of the mixtures used in manufacture of building materials, fillings etc.

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### 3.3.2. SPECIFIC SURFACE AREA ANALYSIS – BLAINE DETECTOR

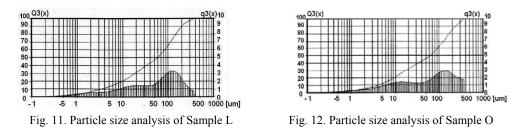
The Blaine detector was used to determine specific surface area. The results of specific surface area analysis of Sample L and Sample O are shown in Table 3.

Specific surface areas are similar for both samples. Literature specific surface area of fly ashes is  $2500 - 6000 \text{ cm}^2/\text{g}$  (Giergiczny, 2002; Małolepszy and Tkaczewska, 2007). Both tested fly ashes have specific surface area that classifies them into the group of fly ashes with developed specific surface area (fine-grained, porous and spherical grain).

Measurement of specific surface area is a basic study carried out for all waste and raw materials that are used in mining and mineral processing. The specific surface area is particularly important in determining the properties of cements. It is also important in the case of use of fly ash in the manufacture of ceramics, concrete, filling, road embankments, and even in agriculture.

#### 3.3.3. GRAIN SIZE ANALYSIS – LASER PARTICLE SIZE ANALYZER

Laser particle size analyzer was used to determine grain size distribution. The results of the grain size analysis of Sample L and Sample O are shown on Figs 11 and 12.



The results of grain size distribution of Sample L present size of grains that is in the range of 0 to 420  $\mu$ m. Only about 3% of grains are smaller than 1  $\mu$ m while 99% of grains are smaller than 340  $\mu$ m, and 80% are smaller than 150  $\mu$ m. It may be said that the fly ash is considered as fine-grained material. The grain size distribution of Sample L has a maximum of approximately 126  $\mu$ m. It has average value of 57  $\mu$ m and the standard deviation of 9  $\mu$ m. Sample O has similar grain size distribution. The volume of grains is in the range of 0 to 360  $\mu$ m. Only about 2.5% of grains are smaller than 1  $\mu$ m, while 99% of grains are smaller than 350  $\mu$ m, and 80% of them are smaller than 170  $\mu$ m (fine-grained material). The grain size distribution of Sample O has one maximum approximately from 125 to 138  $\mu$ m. It has average value from 58 to 62  $\mu$ m and the standard deviation of 10  $\mu$ m. Both fly ashes can be used as a raw material for the manufacture of many ceramic materials, cements, concretes, fillings, plastics without further fragmentation, but its use in the manufacture of artificial aggregates or in agriculture is limited.

An accurate determination of the grain size distribution in loose raw materials and mineral wastes has extraordinary meaning in the selection of methods for their utilization in industry in which they can be used. It is particularly important when these materials are used in the mining, building, roads, ceramic and agriculture industry.

#### 4. CONCLUSION

An article presents progressive research methodology of using modern measuring devices to determine physical, chemical and mineralogical properties of fly ash. It also shows that this methodology allows to describe most important properties of tested material. The results allow to determine the direction of utilization of fly ash.

Although, the article discusses the application of different methods and measuring devices for analyzing samples of fly ash (a mineral substance with a very fine particles), most of these methods can be also used in studies of physical and chemical properties of other minerals.

The tests show that each of methods taken separately give only a part of the characteristics of investigated materials. These methods in parallel studies complement and confirm each other and allow for full characterization of the physical, chemical and mineralogical properties of various raw materials, waste and products. The results obtained by various methods overlap or act as input for other, e.g. elemental composition of the sample obtained in the point X-ray microanalysis (scanning microscope) corresponds to elemental composition determined by X-ray apparatus (elements, in the form of specific minerals).

Further studies, using the above and other advanced testing methods, will create comprehensive testing procedures for mineral raw materials and waste on a laboratory scale. Then, these procedures can be applied for in situ measurements of various properties of raw materials, products and waste on an industrial scale.

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W ostatnich latach obserwuje się dynamiczny rozwój różnorodnych dziedzin naukowych związany z wprowadzeniem nowych, doskonalszych urządzeń badawczych. Należą do nich między innymi: mikroskop elektronowy skaningowy, mikroanalizator rentgenowski, derywatograf, dyfraktometr rentgenowski, granulometr laserowy, piknometr helowy, aparat Blaine'a. Mogą być one wykorzystane przez praktycznie każdą dziedzinę nauki zajmującą się ciałami stałymi. Te nowoczesne metody znalazły również zastosowanie w badaniach właściwości surowców i odpadów mineralnych. Wykonywanie takich pomiarów może przyczynić się do stworzenie kompleksowych procedur badania surowców i odpadów mineralnych w skali laboratoryjnej. W publikacji podjęty został problem określenia metodyki badań właściwości zarówno fizycznych jak i chemicznych surowców i odpadów mineralnych na przykładzie popiołów lotnych, z zastosowaniem nowoczesnych metod i urządzeń pomiarowych. W części teoretycznej została przedstawiona charakterystyka poszczególnych metod badawczych, a także zasada działania urządzeń badawczych. Natomiast w części praktycznej omówiono metodykę wykonywania poszczególnych badań, a także przedstawiono przykładowe wyniki..

słowa kluczowe: popioły lotne, właściwości mineralogiczne, rentgenowska analiza dyfrakcyjna, mikroanaliza rentgenowska, analiza termiczna, analiza granulometryczna, pomiar powierzchni właściwej

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