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DOI: 10.18454/mca.2016.01.11

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## ЛАБОРАТОРНОЕ ФИЗИКО-СПЕКТРОСКОПИЧЕСКОЕ ИССЛЕДОВАНИЯ ЗОЛЫ УНОСА

#### Аннотация

Современное строительное материаловедение направлено на разработку экологически чистых материалов с оптимальными физико-механическими и другими характеристиками. Наиболее эффективно использовать для этой цели применять такие методы, как рентгеновскую дифракцию, дифференциально-термический анализ, термогравиметрию, спектрометрический анализ. В результате различных исследований определены характеристики золы уноса теплоэлектростанций. Выявлено, что золы двух электростанций Приморского края из четырех пригодны для использования в качестве компонента композиционного связующего.

**Ключевые слова:** зола уноса, рентгенофазовый анализ, дифференциально-термический анализ, термогравиметрия, спектроскопический анализ.

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## PHYSICAL EQUIPMENT SPECTROSCOPIC STUDY OF COAL ASH

#### Abstract

Modern construction materials study aims to develop eco-friendly materials with optimum physical and mechanical and other characteristics. The most effective techniques used for this purpose are applied techniques such as X-ray diffraction, differential thermal analysis, thermogravimetry, spectrometric analysis. As a result of various studies we have identified characteristics of fly ash. It has been revealed that the two power stations out of four in Primorsky Region are suitable for use as a component of the composite binder.

**Keywords:** coal ash, X-ray diffraction, differential thermal analysis, thermogravimetry, spectrometric analysis.

## Introduction

In the development of composite building materials, requires a comprehensive study of their components. In the modern building materials science applied techniques such as X-ray phase analysis, differential thermal analysis, thermogravimetry, spectrometric analysis.

Research has cast such additives in cement composites as fly ash thermal power plants.

## X-ray analysis

Investigation of the mineral composition and the structure was analyzed by X-ray analysis Powder X-ray diffractometer D8 Advance Company Bruker AXS (Fig. 1).

X-ray diffraction - a non-destructive method for the analysis of substances in powder form [1,2,3]. Provides the ability to conduct qualitative and quantitative analysis of crystalline phases of establishing the crystal structure of inorganic, organic, organometallic and metal complex compounds in polycrystalline form, the establishment of polycrystalline materials, the degree of crystallinity of polymers.

In the study used the database ICDD PDF-2 and Crystallography Open Database. Quantitative phase analysis is possible for 345 industrially important phases contained in the database TOPAS Structure Database, as well as for the phases of known structure.



Fig. 1 – X-ray powder D8 Advance diffractometer



Fig. 2 – Thermogravimetric analyzer Shimadzu DTG-60 H

The software package allows you to accurately determine the atomic structure of objects of inorganic, organic, organometallic and bioorganic origin. Using software DIFFRAC PLUS DQUANT can be carried out semi-quantitative analysis for the other phases. For polycrystalline materials can be calculated lattice parameters, deformation stresses the value of the crystallites in the powder, atomic structure, structural strain distribution. It is also possible for the structural analysis of the amorphous samples. The method of shooting: Bragg-Brentano. Options include high-speed position-sensitive detector VANTEC-1.

## Differential thermal analysis and thermogravimetry

Depending upon heating latched indicators are following methods of thermal analysis:

- differential thermal analysis (DTA) - shows the change in energy of the system (sample) [4,5,6];
- thermogravimetry (TG) - change in mass [7,8,9].

Derivatograms samples were obtained from thermogravimetric analyzer Shimadzu DTG-60H (Fig. 2). Program heating furnaces from 20 to 1500° C is carried out electronically heater at 20°C/min. Platinum thermocouple with an accuracy of 5°C temperature measurement (T), the scan speed at 2.5 mm/min a four-channel recorder signal is recorded on the paper. The temperature difference ( $\Delta T$ ) between the studied substance and a standard proportional to the thermal effect, can be written as the DTA curve (sensitivity of 500 mV). Simultaneously with the DTA curve is being recorded curve of weight loss (TG) and its derivative (DTG) (sensitivity of 500 mV). Sample weight of 113 mg. Weighing accuracy of 0.05 mg.

## Determination of radioactive materials

In the course of analysis of the literature, it was found that the ash has some TPP radioactivity. Due to the fact that the concept of modern construction is aimed at creating conditions for environmental safety of the home, it was necessary to quantitatively check the background radiation of these materials [10,11,12].

Specific (volume) activity of beta- and gamma-emitting nuclides in the sample is determined by counting spectrometric method using a universal spectrometric complex USC "Gamma Plus" (Fig. 3).



Fig. 3 – Universal spectrometric complex USC "Gamma Plus".

The study was conducted according to the requirements of the following normative documents: GOST 27451-87 "Means of measuring ionizing radiation. General specifications", GOST 26864-86 "Spectrometers of energies of ionizing radiation. Methods of measurement of basic parameters", TU 4362-002-46554900-06 "Universal complex spectrometric USC "Gamma Plus." Technical specifications".

The principle of operation is based on the complex transformation in the working volume of the detector energy gamma rays and beta particles in flashes of light (scintillation), the intensity of which is proportional to the energy lost by the gamma-quantum and beta-particle detector.

Flashes of light entering the photoelectric amplifier (PMT), converted into a stream of electrons that propagate under the influence of the applied potential difference, resulting in the formation of the photomultiplier output pulses of electric current whose amplitude is proportional to the particle energy is lost in the detector. This fact provides the possibility in principle of measuring the energy spectrum of the detected gamma - or beta radiation.

A signal detecting unit is amplified, shaped and converted into a pulse voltage. This pulse is applied to the input of analog-to-digital converter, where it is sorted in amplitude is converted into a digital code that allows you to record and store the information received in the computer's memory.

#### **Physical-spectroscopic study of CHP fly ash**

Object of study is the largest thermal power plant fly ash Primorye Territory: Vladivostok TPP-2, Artem CHP, Primorye SRPS and Partisansk Power Plant. An important factor is the possibility of individual selection of dry ash that is currently under way in these thermal power plants.

Given the focus of modern science to the development and use of environmentally friendly materials have been evaluated radioactivity ash. Ash as a function of the total effective specific activity of natural radionuclides  $A_{eff}$  to 370 Bq/kg is used for the production of materials, products and structures, residential and public buildings.

The results of determination of specific effective activity of fly ash on spectrometric complex "USK Gamma Plus" are given in tab. 1.

Ashes of Vladivostok TPP-2 and Artem TPP belong to the first class of materials (less than 370 Bq / kg) in accordance with GOST 30108-94 "Building materials and products. Determination of the specific effective activity of natural radionuclides." This material can be used for all kinds of construction work.

Ashes of Primorye SRPS and Partisansk Power Plant exceed the permissible parameters of radioactivity, so not suitable for use in construction.

Table 1 – Determination of specific effective activity of fly ash thermal power plants of Primorye Territory

Parameter	The measurement result A (Bq/kg)			
	Primorye SRPS	Vladivostok TPP-2	Artem CHP	Partisansk Power Plant
Activity $^{40}\text{K}$	$496,9 \pm 101$	$392 \pm 89$	$342 \pm 68$	$516,9 \pm 101$
Activity $^{232}\text{Th}$	$153,6 \pm 20,3$	$31,5 \pm 19,7$	$29,5 \pm 15,7$	$193,2 \pm 22,3$
Activity $^{226}\text{Ra}$	$163,1 \pm 9,36$	$37,63 \pm 6,32$	$27,23 \pm 5,93$	$113,1 \pm 6,37$
$A_{eff} = A_{Ra} + 1,31A_{Th} + 0,085A_K$	$>398$	$80 \pm 30$	$93 \pm 20$	$>410$

Thermal studies were performed on raw thermogravimetric analyzer Shimadzu DTG-60H at a rate of temperature rise of 20 K / min, in the range of 20-1100°C. The results of thermal analysis are presented graphically in Fig. 4.

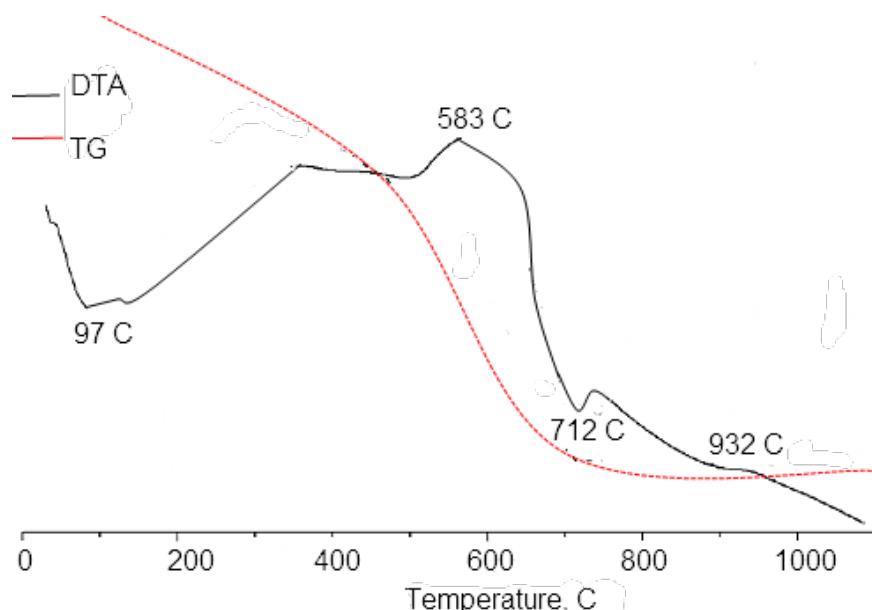


Fig. 4 – The results of DTA and TG of Vladivostok TPP-2 fly ash

During heat treatment the ash in the range of 40-200°C, the loss of water adsorbed surface area of the particles. Carbonate decomposition is observed at a temperature of 712°C. Burning residual fuel - 500-700°C. The nature and intensity of loss of mass during this indicates an amount of unburnt residues are particles of coal and coke and semi-coke residue. Relatively small exotherm with a maximum of 932°C reflects the crystallization of compounds in like mullite aluminosilicate phase.

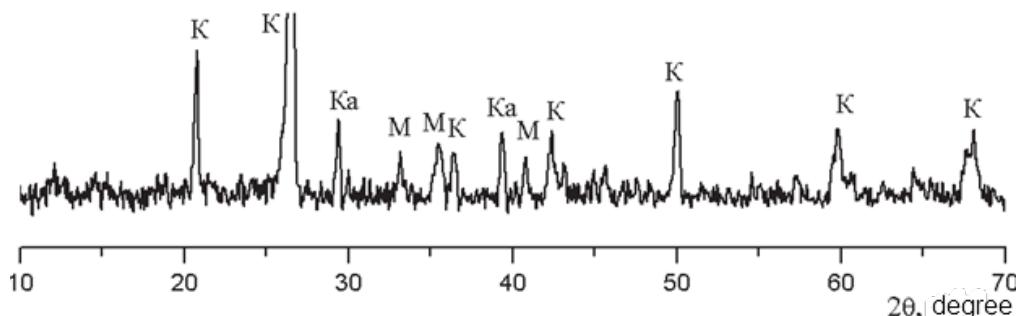


Fig. 5 – XRD results of Vladivostok TPP-2 fly ash. K - quartz, Ca - calcite, M - mullite

The test material is relatively homogeneous, multicomponent. According to the composition closest to aluminosilicates, due to the high content of silicon and aluminum oxides to 80-90%, of which about 2/3 of silicon oxide. At the same time, fly ash almost no unburned particles, which tend to concentrate harmful components. Ash consists of crystalline and amorphous phases. The crystalline phase contains quartz, feldspar, mullite, et al. The amorphous phase is represented primarily in the form of glass.

### Conclusion

Thus, the fly ash consists of minerals that are widely used separately as filler dispersed in composite binders. Therefore, we can assume that the ash Primorsky Region CHPs on the chemical composition suitable for use as a filler in the cement composition.

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