RESEARCH OF A CONCRETE STONE BY METHODS OF X-RAY PHASE AND SPECTRAL ANALYSIS

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Summary: *Currently, there are a variety of test methods for products and constructions made of concrete. X-ray phase and spectral analysis are qualitative characteristics of the material, which allows to know the structure of the sample. This versatile and rapid methods of the analysis.*

Keywords: *concrete, X-ray phase method, spectral method.*

1. INTRODUCTION

In recent years, the annual production of concrete for construction in the world exceeds 1.5 billion cubic meters, which indicates its superiority over other building materials.
Currently, there is a variety of test methods in products and structures made of concrete. One of these is X-ray phase analysis.
The purpose of the X-ray phase analysis is the identification of the substance in the mixture by the set of its interplanar distances (*d*) and the relative intensities (*I*) of the corresponding lines on the X-ray diffraction pattern. For this, according to the Bragg-Wolf law, it is necessary to determine the reflection angles θ.

$$2d\sin\theta = n\lambda, \tag{1}$$

where d - spacings, θ - glancing angle (Bragg angle), n - order diffraction peak, λ - wavelength.
The main task of XFA is the identification of different phases from the mixture on the basis of the analysis of the diffraction pattern given by the sample under study.
At the heart of the RFA are the following principles:

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- the powder diffraction pattern is, so-called, a peculiar characteristic of crystalline matter;

- each crystalline phase always gives identical diffraction spectrum which is characterized by a set of interplanar distances *d* (hkl) lines and relative intensities *I* (hkl), inherent in a particular crystalline phase;

- X-ray diffraction spectrum from a mixture of individual phases is a superposition of their diffraction spectra;

- from the diffraction spectrum of the mixture, a quantitative estimate of the ratio of the crystalline phases that are present in the sample under study is permissible.

The ratio of the intensities of crystalline phases present in a particular sample is proportional to the content of the phases in it [1].

Qualitatively and quantitatively, any radiation is characterized by the wavelength (with different radiation energy) and its intensity. To characterize any inhomogeneous radiation, one must consider its spectrum, that is, the dependence of the intensity on the wavelength of the radiation.

X-ray emission occurs as a result of the collision of electrons flying at high velocities with the anode material of the X-ray tube.

Electron flying with velocity *U*, when hitting the tube anode is inhibited, the energy (*p*) spent on interaction with the anode material, and the remaining energy is converted into electromagnetic radiation energy dependence of the wavelength of the electron energy is expressed by the Einstein equation:

$$\frac{hc}{\lambda} = \frac{mU^2}{2} - p \qquad (2)$$

If the p - value is small compared to the energy electron and it can be neglected, then the resulting radiation will have a maximum energy, i.e., the minimum wavelength. If the p-value is non-zero, then the image of the radiation has a longer wavelength. Of all the possible wavelengths, a continuous emission spectrum consists.

The X-ray method of phase analysis is based on the fact that the crystal lattice is diffractive for x-rays.

If the object under investigation consists of several phases, then each phase will have its own diffraction pattern. In this case, the diffractogram is the imposition of diffractograms of all phases in the sample under study. The intensity of the reflexes of each phase will depend on its amount in the test mixture. Thus, from Fig. shows that the most intense reflections are Cu and weakest - Cu_2O. Therefore, in the sample contained significantly more copper than CuO and Cu_2O [3].

X-ray diffraction method is often used in mineralogy. This is a universal and fast method of analysis.

The main feature of the crystalline state of a substance is the emphasis I ordered arrangement of its constituent elements in the entire volume of the body.

For the study of a large aggregate, 10 samples of a large aggregate, a multifunctional complex under construction were selected from grids Karl Marx in the city of Kursk, which was carried out X-ray phase analysis of the composition of minerals used as a major s and filler.

Figure 1. Samples of a large aggregate from the body of grillage for X-ray phase analysis.

For the radiographic analysis, samples were prepared, the samples were ground into a powdered state in a laboratory porcelain mortar, and then sieved through a sieve. Analysis of the X-ray diffraction data were compared with the Americans and the Kan filing PDF software minerals [4].

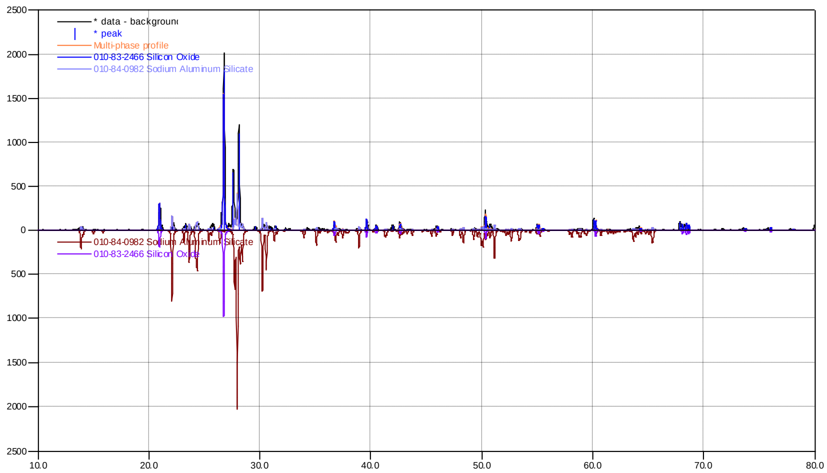

Figure 2. XRD pattern of the test samples of concrete blocks

Samples diffractogram testifies a large number of peaks of high intensity Na(AlSi3O8) and Ca(AlSi3O8) and indicates that the samples are represented mainly

$Na(AlSi_3O_8)$ and $Ca(AlSi_3O_8)$, the chemical formula of which corresponds to feldspar minerals.

To investigate the cement stone ten concrete samples were selected, which was carried out X-ray analysis of the samples, selected from a body of grillages of the under construction "Multifunctional complex on the street Karl Marx in the city of Kursk". The main feature of the crystalline state of a substance is the emphasis I ordered arrangement of its constituent elements in the entire volume of the body [2, 3].

Analysis of the X-ray diffraction data were compared with the Americans and the Kan filing PDF software minerals.

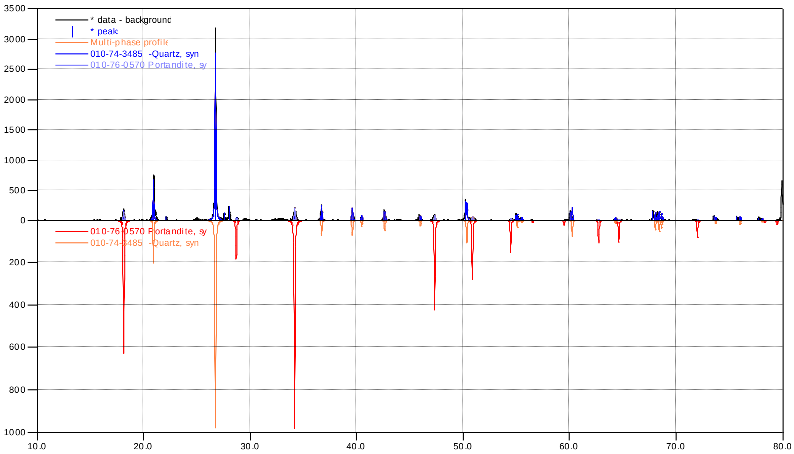

Figure Diffraction pattern of the test cement stone samples

Diffractogram samples indicating high Pixel Count peaks of high intensity SiO_2 and $Ca(OH)_2$ and indicates that the samples are represented mainly SiO_2 - 58% and $Ca(OH)_2$ - 40%, the chemical formula which corresponds to SiO_2 - sand and $Ca(OH)_2$ - portlandit.

According to the research it can be concluded discrepancy concrete mix to requirements of technical standards GOST 26633-2012 Concrete hard and fine-grained.

Spectral analysis occupies a special place and is widely used in practice for more than 100 years. It is based on the analysis of the emission and absorption of atoms, molecules and a solid. Such an analysis uses a wide range of wavelengths from X-ray to radio waves, but the greatest application is analysis within the optical wavelength range from 750 μm to 10 nm [1]. Depending on the physical phenomena distinguish the emission, absorption, Raman and luminescent spectral analysis.

Emissive spectral analysis is carried out on the emission spectra. It involves the emission of a certain amount of a sample in an electric arc of direct or alternating current, in a gas flame. In this case the sample evaporates molecular compounds are usually broken down into atoms, which are excited in the same source of energy and provide illumination in the form of a line spectrum.

At the heart of the emission method are two main provisions:

1) The atoms of each element and different molecules are characterized by a certain set of spectral lines and there are sufficiently complete tables of these lines

the intensity of each spectral line depends on the concentration of atoms and molecules in the plasma. These provisions are obvious, but their use in practice meets a number of difficulties.

Absorption analysis is carried out on the absorption spectra of the sample in any aggregate state. In contrast to the emission analysis, the substance does not decompose into atoms in it, therefore, by means of an absorption analysis, the problems of determining the elemental and molecular composition of the sample, as well as the structural analysis of molecules and a solid are solved [3]. The basis for the absorption analysis is the dependence of the absorption coefficient of the medium not only on the wavelength of the optical radiation incident on the substance, but also on the concentration of the particles absorbing this radiation.

$$\chi(\lambda, C) = \alpha(\lambda) \cdot C \qquad (3)$$

Here $\alpha(\lambda)$ the light absorption coefficient, calculated per unit concentration of absorbing substance and per unit thickness of its layer. It does not depend on the concentration of matter and the intensity of the incident light, but depends only on its wavelength (frequency). With such a recording of the absorption coefficient, Bouguer law takes the form:

$$\mathrm{I}(\lambda, C) = \mathrm{I}_0(\lambda) e^{-\alpha(\lambda) \cdot C \cdot \mathrm{I}} \qquad (4)$$

And is called the Bouguer-Lambert-Beer law, it should be noted that the assumption of proportionality of the absorption coefficient of the concentration is approximate.

Combination analysis is based on a study of Raman spectra and measuring the intensity of its component spectrum of Raman scattering is a vibrational-rotational spectrum of the molecule or the vibrational spectrum of the solid, but not located in the IR region, and in the wavelength of the excitation light [1]. Its appearance is due to a change in the dipole moment of the molecule or the luminescence center induced by the exciting light. The magnitude of this moment is

$$|P| = \alpha |E| \qquad (5)$$

Where $|E| = |E_0| \cos(\omega t + \varphi)$ module of the electric vector of the exciting light cycle frequency ω and the initial phase of oscillation φ, α - is the polarizability of the molecule (cents luminescence). The polarizability depends on the internuclear distances. Therefore, the vibrations of molecules and luminescence centers polarizability and dipole moment will change, ie the polarizability can be represented as a function of configuration (normal) coordinates of q, the atoms in a molecule or solid

$$q_i = c_i \cos(\Omega_i t + \Phi_i) \qquad (6)$$

c_i, Ω_i and Φ_i - amplitude, cycle frequency and the initial phase of oscillation of normal fluctuations. Expanding this function in a Taylor series in the normal coordinates and only the first two terms of the expansion, we spend trigonometric transformation and obtain an expression for the induced dipole moment of the module, depending on the parameters of the normal vibrations:

$$|P| = \alpha_0 |E_0| \cos(\omega t + \varphi) + \frac{1}{2}|E_0| \sum_{i=1}^{n} \frac{d\alpha}{dq_i} c_i \{\cos[(\omega + \Omega_i)\, t + (\varphi + \Phi_i)] + \cos[(\omega - \Omega_i) t + (\varphi - \Phi_i)]\} \quad (7)$$

Since the induced moment varies with time, scattered light must be observed. The first member (7) describes the Rayleigh scattering of light by the frequency of the exciting radiation and the remaining terms describe the scattering by the total frequency of the incident radiation and the normal vibrations - Raman scattering. The account of the subsequent terms of the expansion yields overtones and composite frequencies. However, their contribution to the intensity is much less than the contribution of the fundamental frequencies. These two spectral lines are arranged symmetrically with respect to the spectral line of the exciting light [4]. Measurement of the intensity and shift of these lines relative to the exciting line makes it possible to obtain qualitative and quantitative information on the type and concentration of molecules in gas, liquid, and in a solid.

Molecular analysis from Raman spectra is in many ways analogous to emission analysis, in principle it is even simpler, since the complex question of the influence of excitation conditions in emission analysis does not arise for Raman analysis. The combination analysis is simpler and the absorption analysis performed on the vibrational-rotational spectra in the IR region. As the Raman spectrum can be excited in any region that is determined only by the wavelength of the exciting light. The use of laser radiation to excite the Raman spectrum largely eliminates these difficulties [5]. The main measured characteristics are the frequencies of Raman lines, counted from the excitation line, the width and intensity of the individual components of the spectrum.

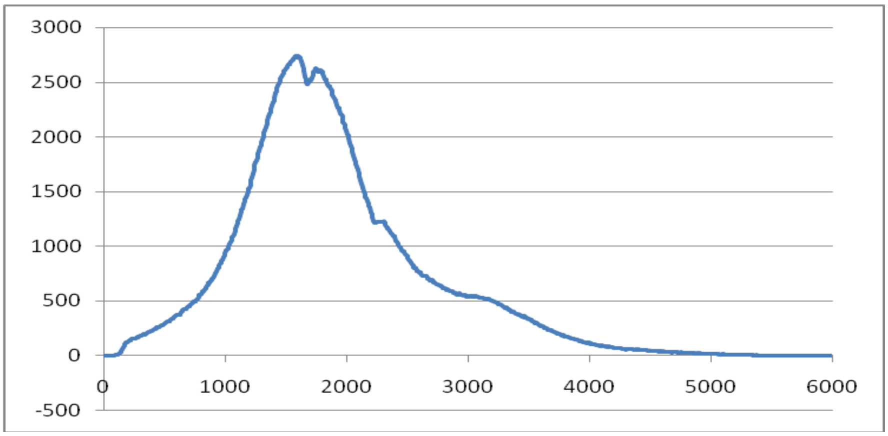

Figure 4. Graph about the hours of concrete when saturated with water samples taken from the body grillages under construction "Multipurpose center on Karl Marx street in the city of Kursk"

Spectral analysis of the water was carried out on the device "RAMAN spectrometer". After standing in the water for 48 hours concrete specimens from the body of grillages under construction "Multipurpose center on Karl Marx street in the city of Kursk" was observed turbidity, established Ph = 12, which is typical for a strongly alkaline medium.

2. CONCLUSION

The results of the spectral analysis instrument "RAMAN spectrometer" has shown that in the water, which was kept for 48 hours concrete samples taken from the body grillages under construction " Multipurpose center on the street Karl Marx in Kursk " is the high concentration in the dissolved minerals feldspar $Na(AlSi_3O_8)$. This indicates the washing out of the concrete samples of minerals belonging to the feldspar and their dissolution in water.

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СТУДИЈА БЕТОНА КАМЕНА МЕТОДА РЕНДГЕНСКЕ ФАЗНЕ И СПЕКТРАЛНЕ АНАЛИЗЕ

Резиме: *У овом тренутку постоји разноврсност метода испитивања у производима и конструкције од бетона. Рентгенофазовый и спектралне анализе су квалитетним карактеристикама испитиваног материјала, које вам омогућавају да науче структуру узорка. То је универзални и брзе методе анализе.*

Кључне речи: *бетон, рентгенофазовый метод, спектрална метод.*