The study of the phase composition and microstructure of composite binders using industrial waste Transbaikalia

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Abstract. The further development of industry is decelerated by lack of energy resources. This is especially significant for the construction industry. Considerable energy savings can be achieved by using specially prepared raw materials. The modern complex approach to solving problems in civil engineering requires additional development of resource-saving technologies, which imply the widespread use of associated industrial products, designed to significantly reduce the energy intensity of cement and concrete production without a fundamental change in technology. The article presents the results of studies on the possibility of obtaining a composite binder using technogenic waste - fly ash and polycarboxylate type superplasticizers. It was investigated that natural perlite and fly ash can be used as part of a multi-component fine cement. With the introduction of fly ash and superplasticizers, the technological and mechanical properties of cement change. High mechanical properties of the composite binder were obtained when the content of fly ash in the amount of 30 wt. % This is due to the structure-forming role of fly ash during Portland cement hydration. Using the method of differential thermal analysis and IR spectroscopy, it is shown that the combined use of fly ash and superplasticizer leads to a change in hydration processes, the binding of portlandite with the formation of an additional amount of calcium hydrosilicates.

1. Introduction

Recently, environmental aspects of the development of engineering and technology have been given closer attention, and therefore, work related to the use of technogenic materials in the construction industry has been actively resumed [1-8].

It is known that cement is the most expensive component of concrete mix, the cost of which depends on the cost of concrete itself. The issue of saving cement for the production of concrete and reinforced concrete products and structures is one of the important issues of modern construction. Improving the quality of concrete requires the use of new composite binders instead of conventional Portland cement, with improved physical and mechanical properties [1, 3, 6, 7].

At present, more than 1.5 billion tons of ash and slag waste have accumulated in ash dumps in the Russian Federation, the amount of which increases by 25 million tons annually. At the same time, no more than 14% of the generated waste is disposed of. At the same time, ash and slag waste is a valuable anthropogenic resource: in the Russian market there is a demand in the cement industry, civil and road construction for certified secondary products from ash and slag waste.

2. Materials
In current research to obtain composite binders were used Portland cement CEM I 32,5, fly ash from Gusinozersk thermal power plant, super plasticizers Neolite 303 and Sika Viscocrete®. Composite binders were obtained with consistent milling of fly ash (0-50 weight %) with OPC. Specific surface area of composite binders were 450-480 m²/kg. From these binders were prepared samples with dimensions 40*40*40 mm. Samples were stored in molds at t = 20-22 °C, relative humidity of 90-95%, and without the molds above the water for a period of 28 days.

3. Results and Discussion

Studies have shown that the use of fly ash as part of composite binders leads to a change in normal consistency, setting time and physical and mechanical properties (Table 1, Fig. 1).

| Parameter                  | with Neolite when ash content, wt.% | with Sika Viscocrete when ash content, wt.% | OPC |
|---------------------------|------------------------------------|---------------------------------------------|-----|
| Normal consistency, %     | 10 30 50                           | 10 30 50                                    | 28  |
| Setting time, min:        |                                    |                                             |     |
| Start of setting          | 130 140 150                        | 140 150 160                                 | 90  |
| End of setting            | 290 300 310                        | 300 320 330                                 | 245 |

Figure 1. Compression strength of Portland cement with fly ash at 28 days

The introduction of Neolite 303 and SikaViscocrete additives increases the compressive strength by 10-15%. In addition to the water-reducing effect, which helps to increase physical and mechanical characteristics, superplasticizers significantly affect the processes occurring in the cement system.

A change in the mechanical properties of composite binders is associated with a change in the structure of cement stone. Fly ash when mixed with Portland cement acts as an active mineral additive, is involved in the structure formation of cement.

To assess the effect of fly ash on Portland cement, a set of research methods was carried out, including differential thermal and IR spectral analyzes, and proving a change in the phase composition of the cement stone.
The results of the differential thermal analysis (Fig. 2) of the cement matrix indicate a change in the endoeffect in the temperature range 515-520 °C in ordinary Portland cement to an exoeffect in Portland cement with fly ash.

![Figure 2. Curves of differential scanning calorimetry of Portland cement (a) and Portland cement with fly ash - 30% (b)](image)

This indicates the binding of calcium hydroxide to the active components contained in fly ash. When comparing the DTA curves, a change in the basicity of the formed calcium hydrosilicates is observed: the endothermic effect in the temperature range of 815–820 °C shifts to the right, towards the temperature increase to 853 °C. A wide temperature range is associated with the fact that the introduction of fly ash leads to the formation of an additional amount of calcium hydrosilicates that differ from traditional ones. In Portland cement with fly ash, additional low-basic calcium hydrosilicates are formed, which are structured along the surface of the solid phase, with the formation of a denser composite with improved physical and mechanical properties.
The change in the phase composition of the cement stone is confirmed by IR spectral analysis on an IRAffinity-1 IR Fourier spectrometer (Fig. 3).

Figure 3. IR spectra of Portland cement (a) and Portland cement with 30% fly ash (b)

An analysis of the IR spectra showed that with the introduction of fly ash there is a change in the intensity of the absorption band in the frequency range 1000-1100 cm\(^{-1}\), which correspond to vibrations of Si-O bonds, which is associated with the formation of calcium hydrosilicates. This indicates a change in the process of hydration of Portland cement with the introduction of fly ash and the formation of an additional amount of calcium hydrosilicates. The shift in the frequencies of the absorption bands corresponding to calcium hydrosilicates also suggests that the structures formed differ from traditional ones.

4. Conclusion
The results showed that the introduction of fly ash in an amount of 10-50% does not lead to a decrease in the strength of the composite binder compared to conventional Portland cement. The greatest result was shown by a composition with 30% fly ash. Nevertheless, an increase in fly ash content up to 50% can significantly save material costs for obtaining a binder and concrete based on it, and may be appropriate for a feasibility study.
With the introduction of superplasticizers in the composition of composite binders, the W / C ratio decreases, the rheological properties of the binders improve, and their strength increases. The addition of SikaViscoCrete and Neolite to the binder in an amount of 0.6% leads to an increase in the strength of the composite binder.

The analysis (DTA and IR spectroscopy) allows us to conclude that the introduction of fly ash contributes to the directional formation of a high-strength stone structure from low-basic calcium hydrosilicates and a decrease in the content of calcium hydroxide.

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