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A study of strength fluctuations of Portland cement by FTIR spectroscopy

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Abstract. The study of daily tests (1-28 days) for compressive strength of ordinary Portland cement is presented. The daily strength curves were obtained for two water cement ratio (W/C=0.4 and W/C=0.5). On both curves, intervals with strength fluctuations were identified. For cement paste with W/C=0.4, one interval with fluctuations was detected (13th-18th days), for cement paste W/C=0.5, two intervals with fluctuations were detected (13th-17th days and 21st-25th days). The largest value of decrease in average strength for samples with W/C=0.4 is -7.1 MPa, for samples with W/C=0.5 the largest value of decrease in average strength is -2.7 MPa. For cement paste samples with W/C=0.4 for 15th-18th days, infrared spectra were obtained by Fourier-transform infrared spectroscopy. The analysis of the FTIR spectra showed that the strength fluctuation may be caused by the formation of a sulfate phase (ettringite) during hardening of the cement paste.

1. Introduction

Ordinary Portland cement (OPC) is one of the main hydraulic binders in the construction industry. On the basis of the OPC, concrete and reinforced concrete structures for buildings and structures are made. Compressive strength is the most important characteristic of the OPC due to the fact that concrete in building structures mainly works to compress from external power loads. Basically engineers and researchers are interested in the value of compressive strength on the 28th day under normal hardening conditions. On this day, class of OPC is assigned in accordance with regulatory standards. Also the compressive strength of OPC and other hydraulic binders based on OPC on intermediate days up to 28 days, for example, on days 1, 3 and 7 are of interest too [1-2].

Malinin *et al.* [3] showed that the daily compressive strength growth curve for 28 days for cement with higher alite content is not strictly increasing, specifically, the curve of the strength contains intervals with strength fluctuations. They a carried out studies using infrared spectroscopy and found that the cause of the fluctuations is silica polymerization, and they also did not exclude the effect on the strength fluctuations of cement with a higher alite content is formation of the sulfate phase in hydrated cement.

The aim of this work is to study of compressive strength fluctuations of the OPC by Fourier-transform infrared spectroscopy (FTIR).

2. Materials and methods

For the investigation, ordinary Portland cement of class CEM I 42.5 N was used in accordance with GOST standard 31108-2016 «Cements for general construction. Technical conditions». X-ray diffraction analysis of OPC showed the following phase composition: C_3S - 57%, C_2S - 23%, C_3A - 5%, C_4AF - 11%, Ca_2SO_4 - 3%. Two values of the water-cement ratio (W/C=0.4 and W/C=0.5) for cement paste were considered for research. Cement mixing was carried out with distilled water. OPC samples for testing were made in the form of cubes with dimensions of 2x2x2 cm^3. Hardening of the samples was carried out in distilled water under normal conditions for 28 days according to GOST standard 30744-2001 «Cement. Methods of testing with using polyfraction standard sand». On the required day, the samples were subjected to uniaxial compression strength tests, immediately before the tests, the samples were taken out of the water bath and wiped to remove water droplets, and then the geometric measurements of the samples were carried out. The number of samples for testing was taken at 5 pieces for each day.

Infrared spectra for hydrated samples were performed using FTIR spectroscopy. The FTIR spectrum was obtained using a KBr pressed disk containing 1.5 mg the test sample and 280 mg KBr. The manufacture of tablets for the FTIR spectrum and the obtaining of the spectrum were carried out immediately after uniaxial compression tests of the OPC samples. The number of spectra was taken at 3 pieces for the researched day and spectra were obtained from different samples.

3. Results

Curves of the dependence of the compressive strength of the OPC on the time (days) were performed according to the results of experimental tests. Experimental data for cement paste with W/C=0.4 are shown in figure 1, for cement paste with W/C=0.5 data are shown in figure 2. In addition to the curves showing the average values, curves are also shown that describe the minimum and maximum strength values in a series of 5 pieces. Figure 1 and figure 2 show that not one of the curves is not strictly increasing, there are strength fluctuations. It is also seen that the dependence of compressive strength over time has a logarithmic law. It should be noted that in the curve of daily compressive strengths for cement-sand mortar on the same OPC of the class CEM I 42.5 N there are no strength fluctuations [4].

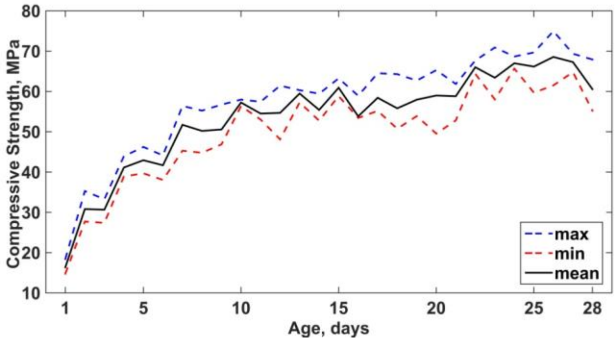

Figure 1. Experimental compressive strength data of cement paste with W/C=0.4.

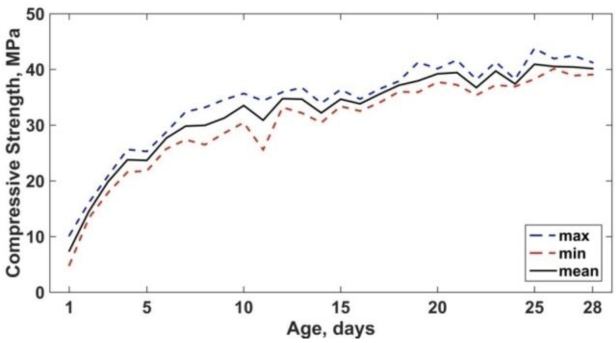

Figure 2. Experimental compressive strength data of cement paste with W/C=0.5.

In both figures (figure 1 and figure 2), two different curing intervals are visible: the first interval is characterized by an intensive increase in strength; the second interval is characterized by a moderate increase in strength. An interval with an intensive increase in strength is observed for 7 days for cement paste with W/C=0.4 and for 6 days for cement paste with W/C=0.5. In this case, with an intensive increase in strength, a decrease in strength is observed for cement paste W/C=0.4 on the 6th day and on the 5th day for cement paste with W/C=0.5. The most interesting points on the curves are the points at which the decreases and rises in strength values are corresponded with maximum, minimum and average values. These points are especially interesting at intervals with a moderate increase in strength. For cement paste with W/C=0.4 are such points located on interval from 13th to18th days. The largest value of decrease in average strength on this interval is -7.1 MPa (16th day), the largest value of increase in average strength is +5.5 MPa (15th day). For cement with W/C=0.5, two intervals with strength fluctuations are of interest: the first interval is located from 13th to17th days; the second interval with fluctuations is located from 21th to 25th days. For both intervals, the largest value of decrease in average strength is -2.7 MPa (22th day), the largest value of increase in average strength is +3.5 MPa (25th day). The difference in the more decrease strength between cement paste with W/C=0.4 compared to cement paste with W/C=0.5 can be explained by the greater brittleness of the samples with W/C=0.4.

To explain strength fluctuations FTIR spectra were obtained for cement paste with W/C = 0.4 for 15th-18th days (figure 3). The interpretation and peak position for the FTIR spectra in figure 3 is shown in table 1. According to the research of Malinin *et al.* [3], silica polymerization and formation of the sulfate phase in hydrated cement could influence on the strength fluctuations of cement with higher alite content. Therefore, mainly FTIR spectra in figure 3 were analyzed for peaks characterizing bond vibrations in silicate (Si-O) and sulfate (S-O) phases. Analysis of peaks characterizing bond vibrations in silicon-oxide compounds did not show a correlation with fluctuations in the compressive strength of the cement paste. An analysis of the peak located at 1113 cm^{-1} for various FTIR spectra in figure 3 showed changes that correlate with strength fluctuations. For this peak, a decrease in the area under the peak for day is observed, in which a decrease in strength. The peak area for 16th day less than the peak area for 15th day, while for 16th day there is a decrease in strength. On 17th day, the peak area increases, and on 18th day it decreases again, which correlates with the curve of compressive strength for cement paste with W/C=0.4. Horgnies *et al*. [5] shows that the peak at 1113 cm^{-1} corresponds to S-O vibrations in ettringite [6, 7]. Thus, the formation of ettringite during hardening of the cement paste influences on the strength fluctuations.

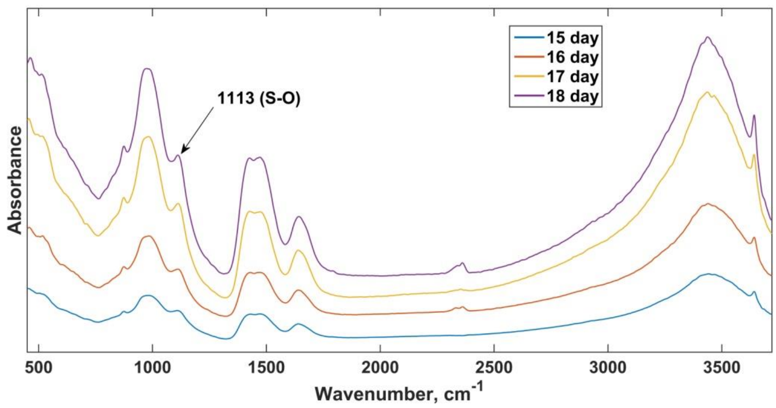

Figure 3. FTIR spectra of cement paste with W/C=0.4.

Table 1. Interpretation and peak position observed in figure 3.

Wavenumber, cm^{-1}	Bond	Phase	Reference
457	Si-O (in-plane vib.)	C-S-H	[5]
496	Si-O (out-of plane vib.)	Afwillite	[5]
518	Si-O (out-of plane vib.)	Afwillite	[5]

713	C-O (υ_4 of CO_3)	Vaterite	[8, 9]
875	C-O (υ_2 of CO_3)	Vaterite	[8, 10]
983	Si-O (asym. stretching vib.)	Afwillite	[5]
1113	S-O	Ettringite	[5]
1423, 1479	C-O (υ_3 of CO_3)	Vaterite	[9, 10]
1643	O-H (υ_2 of H_2O)	H_2O capil.	[5, 8]
3444	O-H	H_2O capil.	[5]
3643	O-H	Portlandite	[5, 8]

4. Conclusion

As a result of the studies, daily (1-28 days) values of compressive strength of cement paste from OPC were obtained. Two types of water cement ratios were considered: W/C=0.4 and W/C=0.5. On the curves of the dependence of compressive strength on time, intervals with strength fluctuations were noted. For samples with W/C=0.4 for 15th-18th days, which are part of the interval with strength fluctuations, infrared spectra were obtained by Fourier-transform infrared spectroscopy. Analysis of FTIR spectra showed that strength fluctuations arise due to the formation of a sulfate phase (ettringite) during hardening of the cement paste. We do not exclude the influence of other factors on strength fluctuation: change in another cement phases during hardening, for example, silica polymerization; formation of porosity of the cement paste, which causes stress fluctuations in material [11].

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