Physico-chemical structure of expanded clay concrete properties with complex chemical additive KJ-3 of the "Relaxol" series

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ABSTRACT--*The article presents the results of a physico-chemical analysis of the cement stone microstructure and processes of hydration changes, crystallization in cement systems, with the complex chemical additive KJ-3 of the "Relaxol" series using infrared spectroscopy, X-ray phase analysis and electron microscopy methodsю It has been established that these additives in cement systems contribute to the creation of new formations crystallizing in a finely dispersed medium, clog capillaries and pores of portland cement stone, compacting and strengthening its structural construction. The content of KJ-3 additive in the composition of expanded clay concrete leads to an increase in its density by 8-10% and prismatic strength by 60% compared with the performance of control samples. At the same time, the complex chemical additive KJ-3 of the "Relaxol" series increases the mobility and frost resistance of expanded clay concrete.*

Keywords-- *Portland cement, complex additive KJ-3, expanded clay concrete, X-ray phase analysis, electron microscopy, IR spectroscopy, physical and mechanical properties of concrete.*

I. INTRODUCTION

Currently, the technological compositions of almost all types and varieties of concrete used abroad are being developed with complex chemical additives. However, in Uzbekistan, complex chemical additives are introduced into a very limited range of structural materials and products. Complex chemical additives increase the mobility, reduce the water demand of the concrete mixture, which leads to an increase in the density and strength of expanded clay concrete, at the same time, it accelerates hardening in the first day and positively affects the increase in frost resistance, durability and other operational qualities of concrete.

II. SYSTEM VALUE

The scientific significance of the research results is due to the development and application of optimal quantities of complex chemical additive KJ-3 of the "Relaxol" series in expanded clay concrete compositions, the desire to maximize the use of positive priority qualities of additives and eliminate the negative properties inherent in the additives of this series, mainly hardening accelerators and surfactants. By correctly combining the types and

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quantitative ratios of additives, the structure and physical and mechanical properties of cement stone and expanded clay concrete can be purposefully regulated.

III. LITERARY RESEARCH

The basis of Relaxol system additives is formed by specially prepared products of coke-chemical industry that have undergone multi-stage purification of rhodanides and sodium thiosulphates, which are well known in the world practice as additives and reagents that intensify hardening of cement and concrete. At present, the base for production of additives is being developed, which includes the most diverse components in terms of functional technological effects, that is, plasticizing, water reducing and regulating the rate of concrete curing in summer and winter conditions, increasing frost resistance, waterproofing and resistance to aggressive environments.

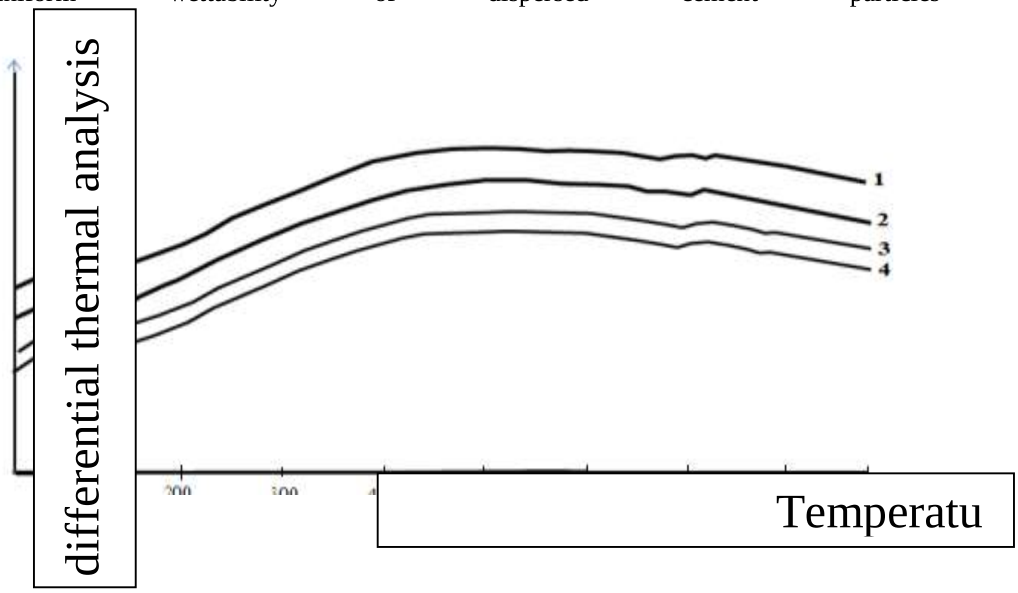
Physical and chemical research of cement composition microstructure with polymeric modifying additives was carried out in accordance with additives classification according to ISO 30459-2008 "Complex additives". Complex chemical additive KJ-3 of "Relaxol" series refers to additives regulating properties of concrete mixtures.

IV. METHODOLOGY

Physical and chemical methods of analysis are aimed at studying hydration, crystallization and structural formation of cement stone in cement systems which contains complex chemical additive KJ-3 of "Relaxol" series[1, 2].

The infrared spectroscopy method, X-ray phase analysis, differential thermal analysis and electron microscopy were used to study the structure formation process of the composition.

Hydration of portland cement substances is carried out in the presence of a small amount of water, for example, in natural conditions, resulting in the formation of a large number of small crystals. With mechanical stirring, it is difficult to evenly provide binder water. This task is performed by a complex additive, improving the uniform wettability of dispersed cement particles [3, 4,5].



1 - without additives; with the addition of a complex additive Kj-3 in the amount of:

2 - 0.5% of a complex additive; 3 - 0.8% of a complex additive; 4 - 1% of a complex additive.

Figure 1: Curves of differential thermal analysis of cement stones.

Table 1: The results of differential thermal analysis

Temperature intervals of endo-effects, K	The duration of heating, min.	Amount of volatile substances, %	The rate of weight loss, % / min	Total weight loss %
Cement stone without additives				
391-431	14.44	2.17	0, 15	11.59
433-452	16.78	2.32	0.138	
1043-1071	85.55	11.6	0.135	
With a composition of 0.5% complex additive KJ-3				
391-415	12.67	2	0.158	10.4
423-468	18.55	2.3	0.124	
1038-1068	85.22	10.4	0.122	
With a composition of 0.8 % complex additive KJ-3				
395-413	12.44	1.91	0.153	10.2
433-448	16.33	2.23	0.136	
1023-1053	83.55	10.2	0.122	
With a composition of 1 % complex additive KJ-3				
323-411	12.22	1.2	0,098	8.38
418-463	18	1.8	0.1	
1018-1071	85.55	8.38	0.1	

When studying thermogravimetric curves of samples, we can observe a sharp change in weight loss in the first two endo effects of cement stone with complex additive KJ-3. These effects are negligible or do not appear at all in cement systems without additives. On the IR spectra of the CA and CA_2 minerals formed during hydration, an intense band appears with an absorption maximum at 520 cm^{-1}, which refers to stretching vibrations of the Al – O in AlO_6 - octahedron bonds. The bands with maximums at 1150, 1020, 970, and 920 cm^{-1} in the IR spectra of hydrated CA and CA_3 are caused by deformation oscillations of –OH gibbsite bonds. In the area of valence oscillations of –OH groups, the band at 3400 cm^{-1} belongs to C_3AN_6, and the rest belong to –OH groups of gibbsite molecular water.

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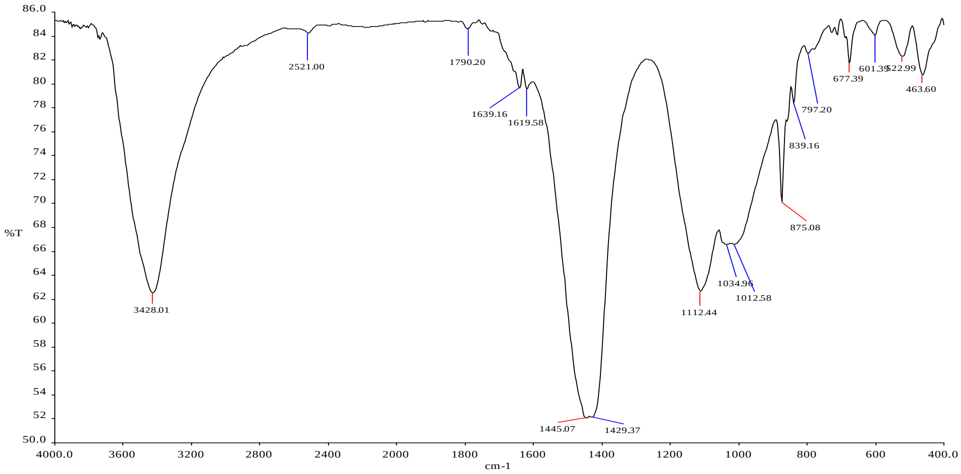


Figure 2: IR spectrum of cement stone without complex additive

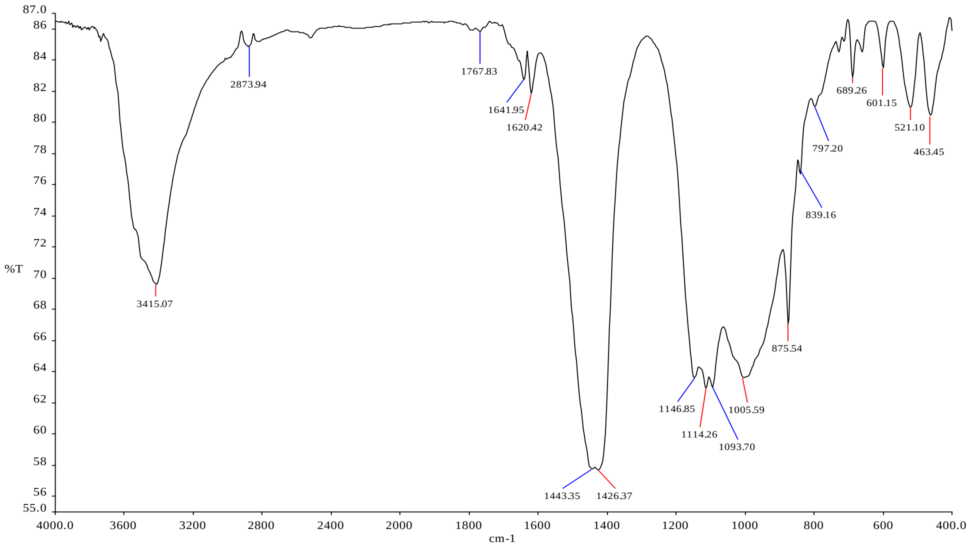
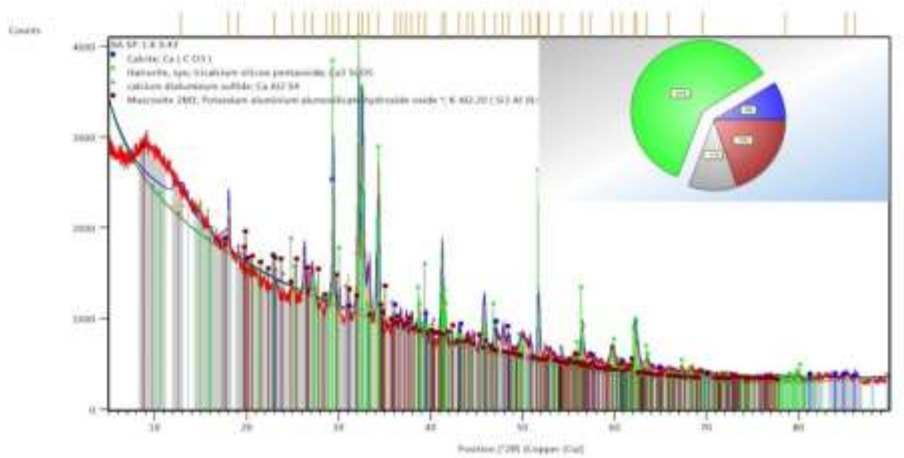


Figure 3: IR-spectrum of cement stone with complex additive KJ-3, in the amount of 1.0% by weight of cement

Consequently, IR spectroscopy shows a change in the coordination of aluminum atoms in the process of the hydration of calcium aluminates, for example transitions from tetrahedral coordination (non-hydrated minerals) to octahedral (hydrated phases) with the parallel formation of –OH groups instead. Absorption bands in areas 463, 513, 661, 727, 863, 1000, 3546 cm^{-1} show the formation of calcium hydrosilicate minerals. As can be seen in the figures, when adding complex additive KJ-3, one can observe high intensity of absorption bands typical for minerals, providing high strength and durability of cement stones.

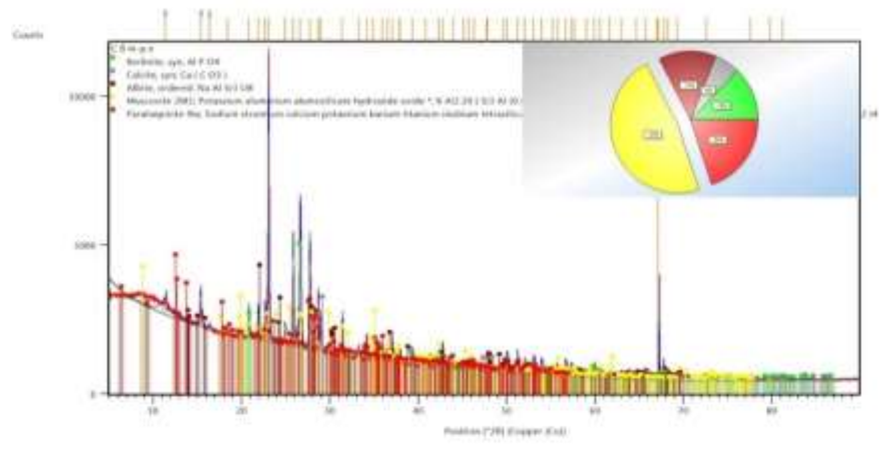
Figure 4 shows that the spectrograms of cement stone are characterized by the presence of several specific maximums. The presence of a maximum of absorption bands at 900-1000 cm^{-1} characterizes the presence of calcium hydrosulfoaluminate. In this case, a clearer rarefaction of the spectrum with a maximum in the region of 1000 cm^{-1} indicates a better crystallization of GSAC in the presence of the complex additive KJ-3. Maximum absorption at 1400-1600 cm^{-1}, as well as a wide spectral band in the area of 3300-3500 cm^{-1}, indicate the presence of submicro crystals of tobermorite group hydrosilicates, the content of which in samples with the complex KJ-3 additive is greater than in the composition without additives . Good spectral resolution in these areas indicates a higher degree of crystallization of the calcium hydrosilicates mentioned above in the presence of a complex additive. A narrow, well-resolved absorption spectrum band with a maximum of 3590-3650 cm^{-1} characterizes the presence of hydrosilicates of the xonotlite group.Figure 4 shows that the control sample, which solidified under natural conditions, has diffraction reflections of the unhydrated minerals of portland cement clinker, namely C_3S - alite (3.034; 2.745; 2.609 Å), C_2S - belita (4.426; 2.745; 2.609; 2, 17 Å), C_3A - tricalcium aluminate (2.694 Å) and hydrated neoplasms of Ca $(OH)_2$ - calcium oxide hydrate (3.113; 2.456 Å) and 3CaO • $A1_2O_3$ • $3CaSO_4$ • $31H_2O$ - calcium hydrosulfoaluminate (9.69; 5.492; 2.629; 2.456 Å).

The diffraction pattern of the sample with the complex additive KJ-3 (Fig. 4. b) shows reduced peaks of alite (3.00; 2.30 Å), belite (4.501 Å), celite (7.317 Å) and hydration products — a reduced peak of calcium hydroxide (4.921; 3.107, 2.627 Å), an increased peak of calcium hydrosulfoaluminate (9.414 Å) and a peak of calcium hydrosilicates (8.224 Å). There is a disappearance of the tricalcium aluminate peak. The decrease in the peak of calcium hydroxide is explained by its binding to sulfate components and its transition to gypsum and hydrosulfoaluminate. Crystallization occurs from a solution of calcium hydrosulfoaluminate in the liquid phase, as can be seen from electronic images filling the pores of the cement stone.



a)

b)



a) control without additive; b) with complex additive KJ-3, in the amount of 1.0% by weight of cement

Figure 4: X-ray curves of cement stone samples hardened in natural conditions

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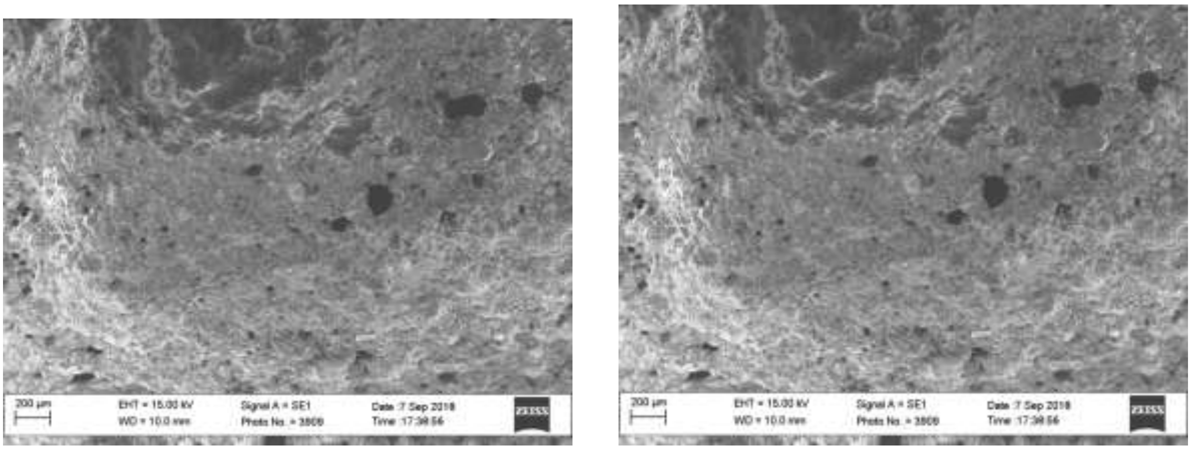
When the content of calcium hydroxide decreases, the possibility of formation and existence of multibasic calcium hydroaluminates decrease. This circumstance prevents the formation of GSAC in the later stages of hardening.

The resulting neoplasms crystallize in the presence of a these additives in cement systems contribute to the creation of new formations crystallizing in a finely dispersed medium, clog capillaries and pores of portland cement stone, compacting and strengthening its structural construction. The phase composition of hydrate neoplasms of cement stone, made of normal density dough on cement Kuvasai factory mark PC400 D20 with different content of complex additive, was studied by methods of electron microscopy. Figure 5.



a) control without additives; b) with a complex additive KJ-3 in the amount of 1.0%

Figure 5: Electron-microscopic images of cement stone samples

Figure 4 shows the complex structure of the cement substance. In the main gel-like mass of neoplasms, there are needle-shaped crystals of ettringite filling the free cavities. Ettringite neoplasms are formed in free volumes. On electron micrographs of cement stone samples with the addition of the complex additive KJ-3, there are pores filling with both gypsum and calcium hydrosulfoaluminate. Moreover, when a complex additive is added, the amount of hydrosulfoaluminate becomes predominant. Increasing the concentration of calcium hydrosulfoaluminate and increasing the specific surface of hydrate phases, both in the general structure of the cement stone and in defective areas of the spatial skeleton, leads to hardening of the material. Thickening and strengthening of the structure of porland cement compositions at the initial stages of hardening is a consequence of the fact that both gypsum and calcium hydrosulfoaluminate, with the addition of complex additive, crystallize with increasing volume.

V. EXPERIMENTAL RESULTS

For experimental researches, Portland cement whose mark is "Kuvasaycement" PC400 D20, expanded clay concrete whose mark is M100, mobility of expanded clay concrete mixture corresponded to mark P3 were used. KJ-3, synthesized at Tashkent Research Institute of Chemical Technology of Uzkimyosanoat State Joint Stock Company, was used as a complex chemical additive. Studying the various compositions of expanded clay concrete

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with the addition of KJ-3 experimentally, with the content of additive 0.6 and 1.0% of cement mass, it was found that high performance indicators of the components were observed with an additive content of 1.0%. The physic-mechanical properties of expanded clay concrete with KJ-3 have been investigated by manufacturing 3 series of prisms of twin samples with dimensions 4x4x16 cm. The first series of control samples are without additive, the second are with the content of KJ-3 additive in the amount of 1.0%. Tests were carried out in 1, 3, 7, 14 and 28 daily hardening periods. The test results are presented in table 1 and in figure 6. Studies have established that the density of expanded clay concrete with the introduction of complex chemical additive KJ-3 increases by 8-10%. The introduction of KJ-3 additive into the composition of expanded clay concrete increases the strength of expanded clay concrete at all hardening periods [7.8].

Table 2 : Results of the study of compressive strength of expanded clay concrete with complex chemical additive KJ-3

№	Name of samples	The content of additives in % by weight of cement	Average density, kg / m^3	Cone draft, cm	The compressive strength of expanded clay concrete (MPa) in age and its growth (%), day				
					1	3	7	14	28
1	Control	0	1289	5	6,1	8,6	9,8	11,3	12,5
2	Hardening under normal temperature conditions	0,6	1367	5	6,3 3	10,9 27	16,4 67	18,1 60	18,3 46
3	Hardening under normal temperature conditions	1,0	1386	5	6,5 6	12,8 48	18,4 87	20,7 83	20,9 67

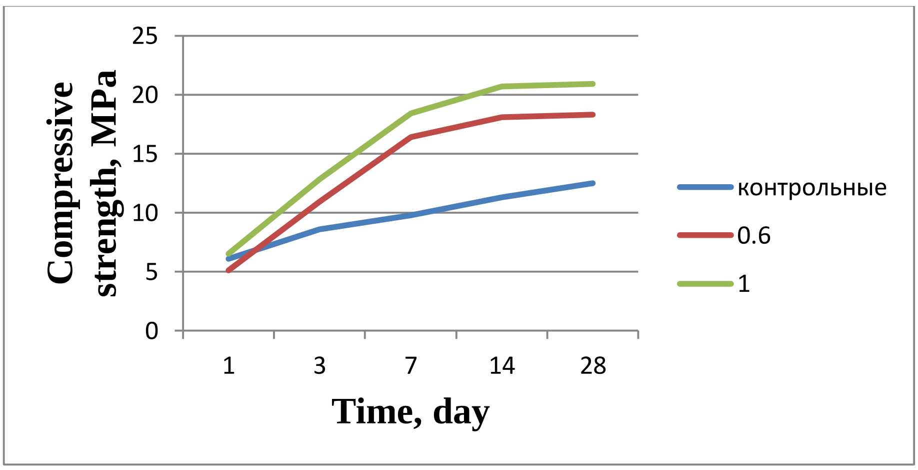

Figure 6: Effect of complex chemical additive KJ-3 on the compressive strength of expanded clay concrete

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1 - the strength of expanded clay concrete without additives; 2 - strength of expanded clay concrete with KJ-3 additive in the amount of 0.6% and 1.0% by the weight of cement hardened under normal temperature conditions, respectively.

Thus, experimentally examining various compositions of expanded clay concrete with the addition of KJ-3, with the amount of 0.6 and 1.0% by weight of cement, it was found that high performance indicators of the components were observed with an additive content of 1.0%.

VI. CONCLUSION

The development and application of the multifunctional complex chemical additive KJ-3 is relevant in the development of modern technology of expanded clay concrete. It is also an effective basis for obtaining high-quality and durable cement systems. Compositions of complex chemical additive KJ-3, which were synthesized on the basis of local raw materials, also gave positive experimental benefit. It has been experimentally established that the use of the complex chemical additive KJ-3 in the amount of 0.6-1.0% by weight of cement is considered optimal in terms of economic efficiency and resource conservation.

High mobility, density and strength of cement compositions with KJ-3 were established. In expanded clay concrete compositions, it is proposed to take the optimal consumption of KJ-3 in the amount of 1.0% by weight of cement. At the same time, the consumption of water for mixing is reduced by 15-20%, which leads to an increase in concrete strength by 50-60% in 28th days. With the content of KJ-3 additive about 1.0%, the mobility of composites is 12 - 15 cm, when the precipitation of the control cone is 3-5 cm.

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