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# Study of the Behavior of Cement Composites in the Conditions of Cyclic Exposure Positive and Negative Temperatures

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**Abstract**. The paper presents the results of tests of cement composites under the conditions of cyclic exposure to negative and positive temperatures for 45 days. The stability factor (durability) compares the compositions with various fillers and additives. Resistance (long-term preservation of the structure and properties) of composites was estimated from the change in the relative hardness of the material, characterized by the area of the polygon, obtained as a result of a piecewise linear approximation of the exposure points of the test materials.

## 1. Introduction
One of the most important properties of building materials and, in particular, cement composites is durability under the influence of various operational factors. Quite a lot of work has been devoted to the study of the resistance of building composites to various environments (physical, chemical, biological), including works [1–21]. From literary sources, it follows that for composites with various additives in their composition, the values of resistance coefficients, as a rule, differ markedly.

## 2. Materials and methods
This article analyzes the results of exhibiting cement composites under the cyclic effects of positive and negative temperatures. As objects of research, cement composites of the following type were considered: cement stone based on a test of normal density (Composition No. 1); cement stone with high water content (Composition No. 2); normal density cement stone with a Melflux hyperplasticizer (Composition No. 3); Cement composite with Melflux hyperplasticizer filled with sand of fractions 0–0.63 and 0.63–2.5 mm with the addition of microquartz (Composition No. 4); Cement composite with Melflux hyperplasticizer filled with sand of fractions 0–0.63 and 0.63–2.5 mm with the addition of microquartz and silica fume (Composition No. 5); Cement composite with Melflux hyperplasticizer filled with sand of fractions 0–0.63 and 0.63–2.5 mm (Composition No. 6). Samples before testing

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hardness for several days hardened in normal-humidity conditions. Samples were tested according to the following mode.

1. Cooling samples from room temperature (+ 23 °C) to minus 50 °C for about an hour (50–55 minutes).
2. Exposure of samples at a temperature of minus 50 °C – 9 hours.
3. With the camera turned off, the natural heating of the samples to room temperature (+ 23 °C) is at least 5 hours.
4. Exposure of samples at room temperature (+ 23 °C) – 9 hours.

The coefficient of stability (durability) was established by changing the hardness on the surface. As is known, hardness is directly related to the strength index [11, 14, 17]. Composite materials initially have different hardness, because they contain components with initially sharply differing hardness, which does not allow to judge about their resistance to cyclic temperature variation in terms of absolute hardness. Determination of the hardness of composite porous materials has its own characteristics as compared with dense mono-materials. The hardness of porous materials is greatly influenced by the structure of their pore space, which is characterized by the value of the total porosity and the specific surface area of the pore space of the material. The resistance of composite materials to cyclic temperature changes (thermal resistance) compared to mono-material is also influenced by the difference in coefficients of linear temperature expansion of the material components, their Poisson's ratio and elastic modulus. The test points for the time changes in the properties of the composites were recorded after 15 and 45 days of testing. The starting point zero corresponds to the benchmark property. In this regard, all the results obtained are reduced to relative values of hardness relative to the control sample before the start of exposure. As in [16], the concept of a polygon is introduced in the paper, which is formed through piecewise linear interpolation of test data. In addition, assuming that the strength of the cement composites may decrease with the exposure time with cyclical changes in negative and positive temperatures, the assessment of such a change can be characterized by the ratio of the area of the polygon to the area of the rectangle. A rectangle characterizes the immutability of a particular property of cement composites. The explanation for the input ratio (K) of the ratio is shown in Figure 1.

$$K = \frac{S_{AEFCD}}{S_{ABCD}}$$

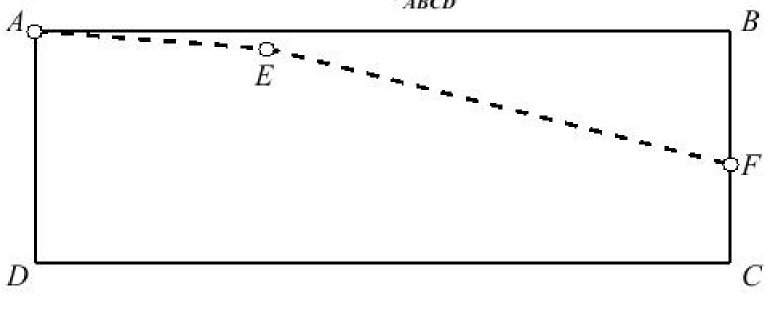


**Figure 1.** Example of calculating the ratio.

In Figure 1 points A, E, F correspond to the characteristic values of the tested property at the points of exposure. The area of the rectangle ABCD corresponds to an idealized composition, the properties of which do not change during exposure in any adverse conditions. The ratio is a dimensionless quantity, which remains unchanged both for the absolute values of the property under investigation and for their relative values.

For a larger number of composites under study, the composites can be sorted by the method given in [18], when either the area value or the ratio is taken as the key, and the name of the composite is taken as the value. The case of identical keys is also considered in [18].

## 3. Results and discussion

The control sample of the composite is designated as composition 1. Comparable samples will be designated as composition 2, composition 3 and elc. The studies were carried out in 4 stages: the results of the composition of cement stone with different water content and with the introduction of a hyperplasticizer were compared (stage No. 1); cement composites, including sandy fractions, as well as the addition of silica fume with stone flour (stage number 2); cement composites incorporating pigments (stage number 3); cement composites, including biocidal additives in their composition, as well as biocidal additives in combination with a hyperplasticizer (stage No. 4). The compositions and test results are given in Table 1.

**Table 1.** The investigated compositions of cement composites and their initial indicators of hardness.

| № composition | Initial components of the composite material | The amount of substance in relation to the cement content (the ratio of the number) | Hardness index (MPa) |
|---|---|---|---|
| 1 | Cement Ulyanovsk CEM I 42.5 N<br>Water | 1<br>0.267 | 4010.17 |
| 2 | Cement Ulyanovsk CEM I 42.5 N<br>Water | 1<br>0.35 | 2065.24 |
| 3 | Cement Ulyanovsk CEM I 42.5 N<br>GP "Melflux 1641 F"<br>Water | 1<br>0.009<br>0.171 | 7016.08 |
| 4 | Cement Ulyanovsk CEM I 42.5 N<br>Ground sand (microquartz)<br>Sand fr. 0-0.63 mm<br>Sand fr. 0.63-2.5 mm<br>GP "Melflux 1641 F"<br>Water | 1<br>1.1<br>2.753<br>2.347<br>0.009<br>0.6 | 5476.90 |
| 5 | Cement Ulyanovsk CEM I 42.5 N<br>Ground sand (microquartz)<br>Silica fume<br>Sand fr. 0-0.63 mm<br>Sand fr. 0.63-2.5 mm<br>GP "Melflux 1641 F"<br>Water | 1<br>0.75<br>0.1<br>1.775<br>1.975<br>0.009<br>0.475 | 9746.86 |
| 6 | Cement Ulyanovsk CEM I 42.5 N<br>Sand fr. 0-0.63 mm<br>Sand fr. 0.63-2.5 mm<br>GP "Melflux 1641 F"<br>Water | 1<br>2.065<br>1.76<br>0.009<br>0.525 | 4089.57 |
| M3 | Cement Ulyanovsk CEM I 42.5 N<br>Activated water on mode (3-3) | 1<br>0.26 | 5168.04 |
| T3 | Cement Ulyanovsk CEM I 42.5 N<br>Biocidal additive "Teflex Universal"<br>Water | 1<br>0.03<br>0.267 | 1174.46 |
| P1 | Cement Ulyanovsk CEM I 42.5 N<br>Pigment "iron oxide red"<br>Water | 1<br>0.0333<br>0.292 | 3600.11 |
| P3 | Cement Ulyanovsk CEM I 42.5 N<br>Pigment "iron oxide yellow"<br>Water | 1<br>0.0571<br>0.331 | 3074.22 |

Consider first the composites, which are based on cement stone. The results in absolute and relative values of the coefficient of resistance with different water content are given in Table 2–3, which also includes the values of the areas of polygons and the coefficients of the ratio. The values of the ratio factors are enclosed in brackets. The diagram of the change in the hardness coefficient of the compositions 1, 2 is shown in Figure 1.

**Table 2.** The results of the exposure of cement composites of compositions 1, 2 at positive and negative temperatures.

| Duration of exposure (day) | Hardness indicators (MPa) | | Relative hardness values | | Polygon area (ratio) | |
|---|---|---|---|---|---|---|
| | 1 | 2 | 1 | 2 | 1 | 2 |
| 0 | 4010.17 | 2065.24 | 1.00000 | 1.00000 | 23.9767 (0.5328) | 29.1196 (0.6471) |
| 15 | 2037.07 | 1274.53 | 0.50798 | 0.61713 | | |
| 45 | 1349.36 | 1064.85 | 0.33648 | 0.51561 | | |

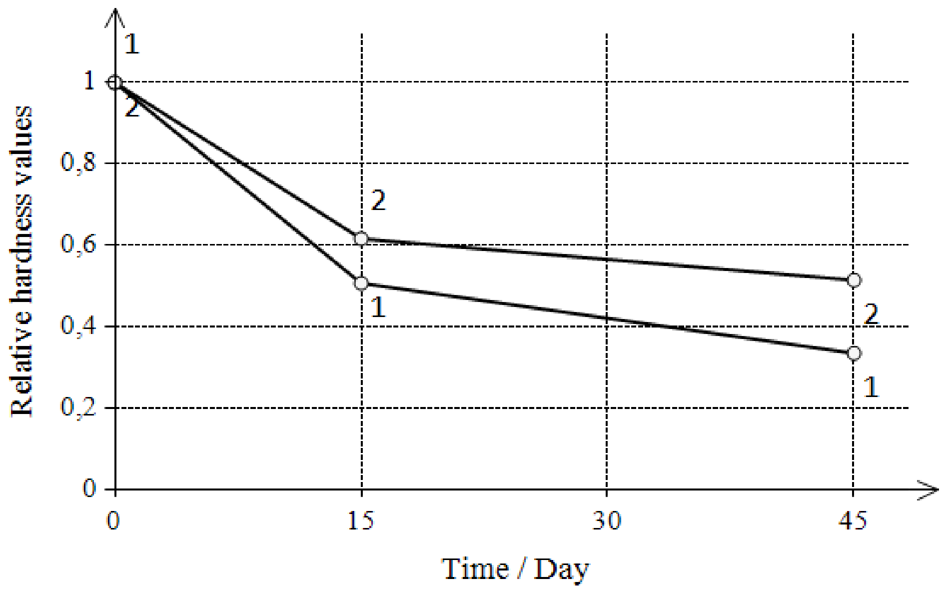


**Figure 2.** The change in the hardness coefficient of the compositions 1, 2 depending on the duration of exposure.

The thermal stability of porous composite materials, to which all studied compositions belong, essentially depends on their total porosity and average pore diameter, since they are hygroscopic and there is always moisture in their pore space under conditions of natural humidity. Freezing of this moisture leads to the formation of crystallization pressure of the formed ice, hydrostatic and hydrodynamic pressure of the moving fluid on the pore walls, which leads to the destruction of the structure of materials [17]. According to our data (see Table 1), the cement stone of composition 1 has, compared to stone 2, 1.94 times the initial hardness index, and judging by W / C, the total porosity is much less. At the same time, the more preferable initial indicators of the material did not lead to an increase in its durability under the conditions of cyclic exposure to positive temperatures.

It is known that the frost resistance of cement composites decreases with an increase in their porosity. However, in this case, the effect of a different type of cement composites takes place and an increase in the number of large pores is not yet a factor of a more intensive decrease in the hardness of the material.

In general, the following factors can affect the change in surface hardness under conditions of cyclic exposure to positive and negative temperatures: material homogeneity, coefficients of linear and volumetric expansion of components, structural components (total porosity, pore sizes and their nature). Since the first two factors in the cement stone of compositions 1 and 2 have no differences, it is obvious in this case that the intensity of the decrease in the hardness coefficients was affected by the difference in structural components.

From Figure 2 shows that the hardness indicator of cement stone composition 1, under the conditions of cyclic impact of positive and negative temperatures, decreases more intensively than the same characteristic of stone composition 2, although its water-cement ratio is 1.3 times greater, and, accordingly, more porosity than material of the first composition.

According to many studies, in these cases the pores of the stone will differ in their size and character. It can be assumed that a material with a smaller W / C has in its volume, including on the surface, more micropores, in which moisture will condense when the material is cooled.

Filling the pores with moisture followed by freezing will contribute to the reduction of surface hardness. Since the filling of pores with condensate is greater for a stone that has smaller pores, i.e. in this case, for a material of composition 1, then it should be expected to have a more pronounced loss of property index. In addition, the rate of decrease in the property index is influenced by its initial values [18]. The greater the initial indicator of the property, with equal resistance of the composites to aggressive media, the greater its decline.

The study of compositions 1, 3, M3 and T3 are given in Table. 3. Composition 3 with a cement kam-nem of self-compacting cement slurry including a hyperplasticizer. Composition M3 is a cement stone on the mixing water activated in the E + M mode (3-3). Composition T3 is a biostable composite with the additive Teflex Universal. The diagram with changes in hardness coefficients with time for compounds 1, 3, M3 and T3 is shown in Figure 3.

**Table 3.** The results of exposure of cement composites of compositions 1, 3, M3, T3 at positive and negative temperatures.

| Duration of exposure (day) | Hardness indicators (MPa) | | | | Relative hardness values | | | | Polygon area (ratio) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | 3 | M3 | T3 | 1 | 3 | M3 | T3 | 1 | 3 | M3 | T3 |
| 0 | 4010.17 | 7016.08 | 5168.04 | 1174.46 | 1.00000 | 1.00000 | 1.00000 | 1.00000 | (0.5328) 23.9767 | (0.5282) 23.7697 | (0.6714) 30.2148 | (0.6602) 29.7082 |
| 15 | 2037.07 | 3629.11 | 3366.30 | 714.39 | 0.50798 | 0.51726 | 0.65137 | 0.60827 | | | | |
| 45 | 1349.36 | 2166.32 | 2776.63 | 667.26 | 0.33648 | 0.30877 | 0.53727 | 0.56814 | | | | |

The change in the relative value of the hardness ratio of composition 3 almost coincides with the change in the relative coefficient of hardness of composition 1. It is obvious that the resistance of cement stone obtained from the mixture with the lowest water-cement ratio (see part 3, Table 1) with a stone of composition 1. From the research results it follows that the use of activated mixing water (the technology is described in [20]) in obtaining a test of normal thickness contributed to an increase in hardness on the surface of the cement stone and its more High resistance to the environment. The introduction of a biocidal additive led to the same effect. However, compositions of such a composition have the lowest initial hardness (hardness index is 1174.46 MPa, see Table. 1) and the lowest property index after cyclic exposure to positive and negative temperatures (hardness index 667. 26 MPa, see Table 3). Since the composite has a water / cement ratio equal to that of composite 1, the porosity of the materials can be assumed to be equal. Then, with a uniform resistance of composites to aggressive effects, the smaller the initial property index, the slower its decrease [19]. The results of an experimental study applied to composites of compositions 1 and T3, shown in Figure 3, confirm this pattern.

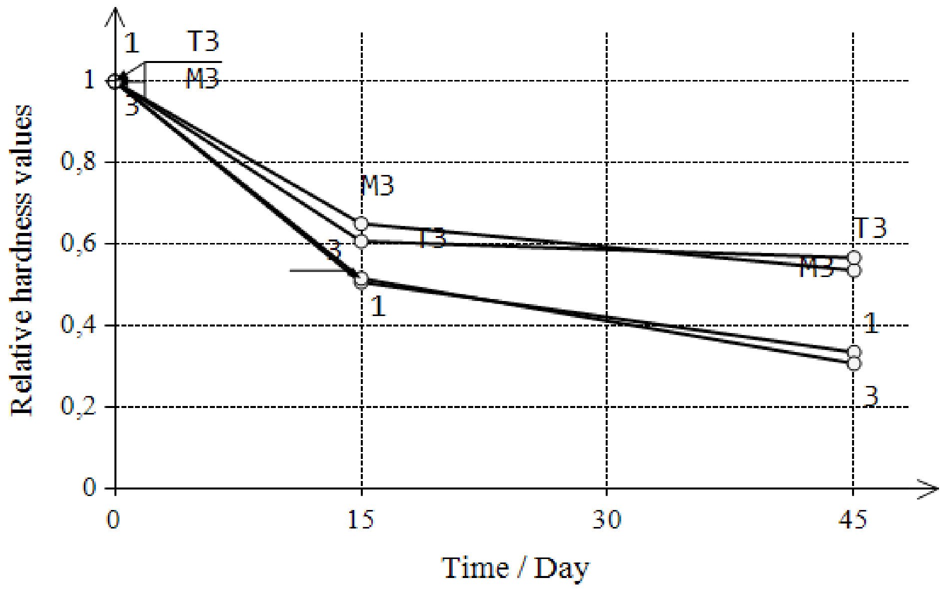


**Figure 3.** The change in the coefficient of hardness of compositions 1, 3, M3, T3 depending on the duration of exposure.

The study of compositions 1, 3, 4, 5 and 6 are given in Table 4. Composition 4 is a sandy be-tone of a new generation with HP and KM (stone flour) with low cement consumption. Composition 5 is a sandy concrete of the new generation with HP and CM and silica fume (MK). The technology for obtaining concrete of a new generation is given in [20, 21]. Composition 6 is a sandy concrete of transitional generation with GP. The diagram with changes in hardness coefficients over time for compounds 1, 3, 4, 5 and 6 is shown in Figure 4.

**Table 4.** The results of exposure of cement composites of compositions 1, 3, 4, 5, 6 with positive and negative temperatures.

| Duration of exposure (day) | Hardness indicators (MPa) | | | | | Relative hardness values | | | | | Polygon area (ratio) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | 3 | 4 | 5 | 6 | 1 | 3 | 4 | 5 | 6 | 1 | 3 | 4 | 5 | 6 |
| 0 | 4010.17 | 7016.08 | 5476.90 | 9746.86 | 4089.57 | 1.00000 | 1.00000 | 1.00000 | 1.00000 | 1.00000 | (0.5328) 23.9767 | (0.5282) 23.7697 | (0.7232) 32.5418 | (0.4460) 20.0714 | (0.7513) 33.8093 |
| 15 | 2037.07 | 3629.11 | 3966.10 | 3786.73 | 1893.79 | 0.50798 | 0.51726 | 0.72415 | 0.38851 | 0.46308 | | | | | |
| 45 | 1349.36 | 2166.32 | 3194.28 | 2488.71 | 4332.25 | 0.33648 | 0.30877 | 0.58323 | 0.25533 | 1.05934 | | | | | |

Of all the compositions of composites listed in Table 1 composite of composition 5 has the largest number of components and the highest initial hardness index (9746.86 MPa) and a low cement ratio (0.475) compared to other filled composites, i.e. according to the reasoning below, there are more intensive factors decrease in property, as evidenced by Figure 4. The final value of the hardness index is 0.255, which is the lowest value we have obtained in the experiment. At the same time, a high initial hardness coefficient indicates that the presence of finely ground components leads to filling the pore space of the composite with their constituent substances and interaction products and reduces the average pore size, but does not lead to their total clogging, since the material's resistance to aggressive action is negligible.

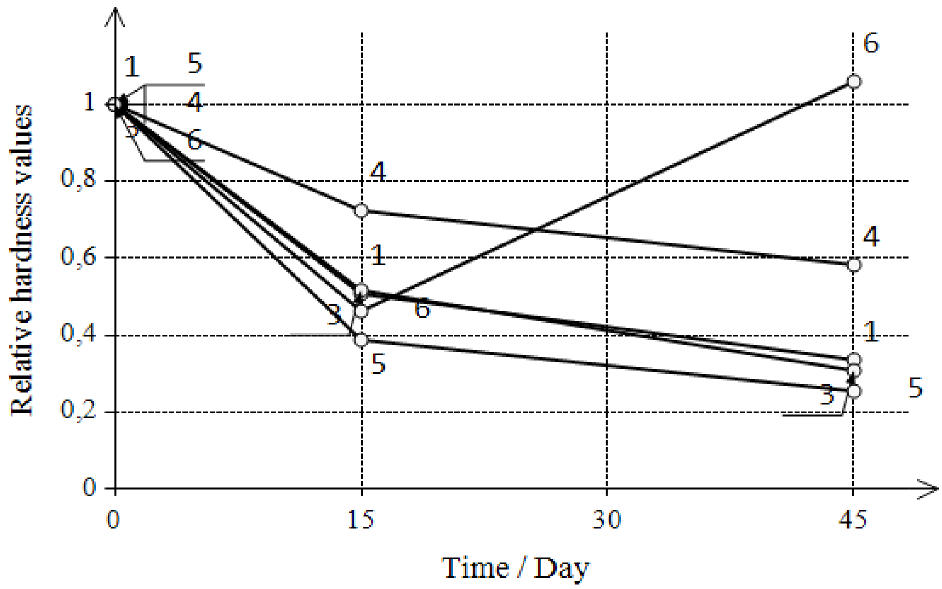


**Figure 4.** The change in the coefficient of hardness of compositions 1, 3, 4, 5, 6, depending on the duration of exposure.

The composite of composition 4 also contains a microfiller and can therefore be compacted. However, it was obtained from the mixture with the highest water-cement ratio in the experiment (w / c = 0.6). High water / cement ratio leads to a decrease in the number of micropores. In addition, the initial indicator of its hardness is about 1.8 times less than a similar indicator of composition 5. As a result, we have the characteristics of the material, leading to a slower decrease in the indicator of hardness in comparison with the composite of composition 5. The final value of the hardness coefficient is 0.58, which is significantly higher than the composite of composition 5, which is equal to 0.255. The sandy composite of composition 6 does not contain microfiller, its water-cement ratio is intermediate between the same indicators of composites of compositions 4 and 5. The initial microhardness index is about the same as that of the composite (see Table 1). It is obvious that the change in the hardness index of the composite during the cyclic effects of positive and negative temperatures should correspond to these factors. After 15 cycles of exposure to the environment is observed. The composite loses its hardness by about 54% (see Figure 4). However, as further testing is underway, a recovery and even a 6% excess of the initial hardness indicator occurs. Since the regularity of changes in the hardness of this composite under the conditions of cyclic exposure to positive and negative temperatures is an exception (see Table 2–5), it (the regularity) requires independent consideration and confirmation, if necessary, with additional studies. In any case, it is necessary to find out which factor after 15 cycles of exposure to a variable environment has affected the additional solidification of the composite.

The study of compounds 1, P1, P3 are given in Table 5. Composition P1 – decorative composite with iron oxide red pigment. Composition P3 is a decorative composite with the iron oxide yellow pigment. The diagram with changes in the hardness ratio of the compositions 1, P1, P3 is shown in Figure 5.

**Table 5.** The results of exposure of cement composites of compositions 1, P1, P3 with positive and negative temperatures.

| Duration of exposure (day) | Hardness indicators (MPa) | | | Relative hardness values | | | Polygon area (ratio) | | |
|---|---|---|---|---|---|---|---|---|---|
| | 1 | P1 | P3 | 1 | P1 | P3 | 1 | P1 | P3 |
| 0 | 4010.17 | 3600.11 | 3074.22 | 1.00000 | 1.00000 | 1.00000 | (0.5328) 23.9767 | (0.9796) 44.0817 | (1.0412) 46.8531 |
| 15 | 2037.07 | 4117.03 | 3630.86 | 0.50798 | 1.14358 | 1.18107 | | | |
| 45 | 1349.36 | 2604.36 | 2619.05 | 0.33648 | 0.72341 | 0.85194 | | | |

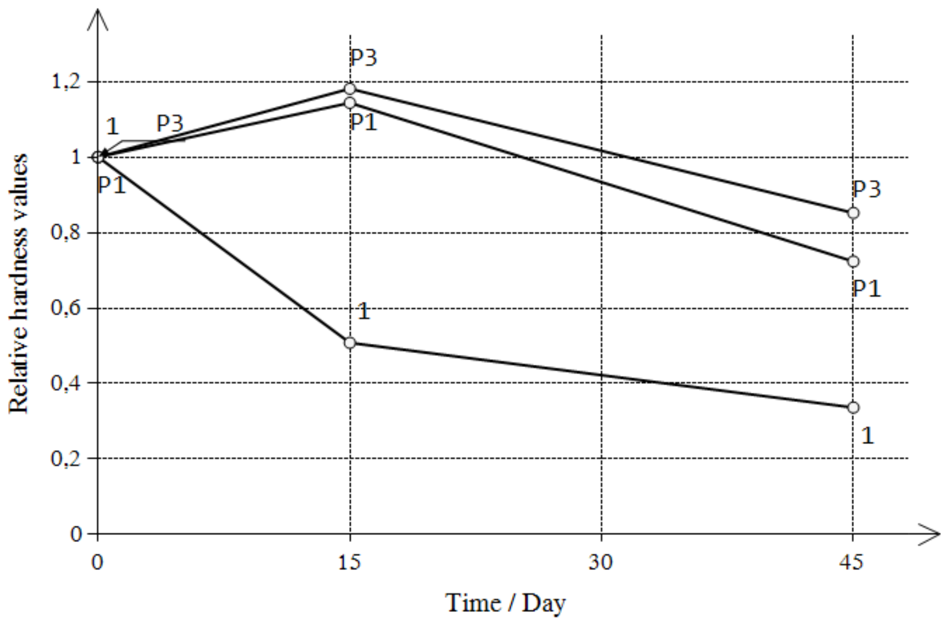


**Figure 5.** The change in the coefficient of hardness of the compositions 1, P1, P3, depending on the duration of exposure.

It is seen that composites of compositions P1 and P3 under the conditions of cyclic exposure of positive and negative temperatures for 45 days retain high hardness coefficients, which are respectively equal to 0.72 and 0.85 from the initial indicators of 3600.11 MPa and 3074.22 MPa. Water-cement ratios were 0.292 and 0.331. The indicators indicate that the change in the hardness of the composites with the impact should be slow. It can also be assumed that part of the pores that condense moisture when the composite is cooled will be clogged with pigments. It is also impossible to exclude the flow, along with destructive processes, of positive effects, which can lead to the temporary strengthening of composites [19]. In this regard, changes in the hardness indices of composites of compositions P1 and P3 are quite understandable.

## 4. Conclusions

The conducted studies confirm the regularity given in [18]. Under impacts, the greater the possible change in the characteristic of a property and the less resistance to this change, the greater it is in any suitable period of time, and vice versa. Studies have also established that homogeneous in composition and coefficients of linear and volumetric expansion of cement composites, in which during cooling as a result of condensation of moisture does not occur critical filling of micropores are more stable under the conditions of cyclic exposure to positive and negative temperatures. In addition, research suggests that using the areas of polygons that are under the lines of change under the influence of the characteristics of the property environment, you can estimate the remaining life of the durability of materials and products.

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