Modification of the cement stone active silt of urban treatment facilities

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Abstract. The use of recycled sludge from wastewater and products based on it in concrete is caused by an increase in the production of activated sludge in cities of millions and the restriction of free land for its disposal. The main components of activated sludge and fiber are SiO₂, CaO, Al₂O₃, Fe₂O₃, MgO and P₂O₅, which allow the pozzolanic mechanism to manifest itself in a hardening cement matrix. Thus, activated sludge and fiber can be used as a modifier of the structure of hardening concrete. The use of municipal wastewater treatment plants in the production of building materials reduces the cost of waste disposal, social tension and solve problems related to environmental protection.

Activated sludge is a dehydrated organic-mineral complex after the processing of municipal and industrial wastewater at sludge sites, containing some heavy metals, inorganic compounds (SiO₂, CaO, Al₂O₃, Fe₂O₃, MgO, P₂O₅), oil products, oils, fats, benzene compounds. The particle size reaches a size from a few microns to 10 mm [1-3]. A high phosphate content of 10 to 20 % by mass has an adverse effect on human health and the environment, especially leaching and soil contamination. The rapid expansion of sewage treatment plants leads to the production of large quantities of organic sludge, a by-product of the purification process. The resulting activated sludge accumulates toxic organic and inorganic substances due to multiple sources of pollution.

Activated sludge is generated at the sludge beds of the urban wastewater treatment plants, where household and industrial wastewater from enterprises is discharged. Sludge activated sludge has two main components, liquid and solid. The liquid part includes water and substances dissolved in it, containing both organic and inorganic. Organic solutes are carbohydrates and fatty acids, inorganic – dissolved salts. The solid portion of the sludge contains organic and inorganic solid. Organic matter includes living organisms and their decomposition products. The inorganic solid is in the form of metals and nutrients. Sludge components are not constant and vary depending on local conditions and the use of educational methods [4].

In general, more than 50 % of the dry mass of sludge components consists of organic materials [5]. Most of the organic material is soluble components such as amino acids, lipids, hydrocarbons and proteins. The nutrients in the sludge are inorganic substances found in various compounds of phosphorus, nitrogen, potassium and sulfur.

The method of stabilization of activated sludge by lime and Portland cement is widely used in the USA and Europe for mass burial [6], it is also used in the production of baked clay bricks in Germany.
The main purpose of this method is the disposal of activated sludge from urban wastewater treatment plants, and the reduction of environmental pollution with minimal financial investments.

A number of researchers have tried to use activated sludge in the form of sludge in the production of building mortars (Bhatty and Reid, 1989; Chen et al., 2006; Chiou et al., 2006; García-Alcocel et al., 2006; Lin and Lin, 2005; Monzó et al., 1996, 1999) and as an ingredient for modifying bitumen in the production of asphalt concrete compositions (Al-Sayed et al., 1995; Lin et al., 2006; Nishigaki, 2000).

Studies aimed at using in the production of lightweight concrete, have shown their failure due to the possible development in the construction of mold and fungi, which is unacceptable for rooms with the presence of people.

Foreign researchers (Lin et al., 2005, 2008; Weng and Lin, 2000) tried to apply active sludge in the production of glazed tiles [8]. When firing, emissions of gases were formed, which were classified as nervously paralytic gases.

Based on the data given in the open press, it can be assumed that the disposal of activated sludge in the production of building materials is hampered due to high humidity, the presence of a large amount of nutrients. At the same time, there is a decrease in the strength of building materials based on lime and Portland cement, and the formation of a nerve-paralytic gas during the firing of bricks, expanded clay gravel [5] and glazed tiles.

Activated sludge generated in urban sludge sites cannot be used as secondary raw materials in the production of construction materials without prior preparation. We have proposed the technological stages of raw sludge preparation. Stage 1 – the destruction of pathogenic organisms and the secretion of amino acids by stabilizing and preventing the re-growth of pathogenic microorganisms. Stage 2 – dehydration by drying in spray dryers, caused by 95 % water content by weight of sludge. As a result, cellulose is formed in the form of a fine, dry, gray-brown loose mass that does not possess any odor. Its bulk weight is 200 kg/m³. Long-term storage of the material obtained is possible without moisture in a well-ventilated dry room for up to a year.

Studies were conducted by us in order to maximize the utilization of fiber – a product of the processing of activated sludge from municipal sewage treatment plants. Concrete is a building material of mass use. Strength characteristics of concrete based on mineral binders and the maximum amount of recycled fiber were evaluated.

The following materials were used as materials: Portland cement type I CEM I 42,5H; slag binder on the basis of electrothermophosphoric slag of electrothermal phosphorus production of the Tolyatti production association Phosphor [10]. Slag meets the requirements of GOST 3476-74 "Domain and electrothermophosphoric granulated slag for the production of cements". The chemical composition of the slag is presented in Table 1.

| Table 1. The chemical composition of electrothermophosphoric slag. |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | $CaO$ | $MgO$ | $SO_3$ | $P_2O_5$ | $F$ |
| 42,71 | 3,42 | 0,17 | 42,66 | 3,72 | 0,52 | 2,11 | 2,67 |

Electrothermophosphoric slag was used in the form of ground powder with a specific surface $S_s = 3000 \div 3500$ $cm^2/g$. The modulus of basicity $Mb = 1,07$, the modulus of activity $Ma = 0,08$, the quality factor $K = 1,23$. The grinding of slag and mineral rocks was carried out using a laboratory ball mill of the ML type with ceramic grinding bodies and removable chambers of 10 and 20 liters. The dispersion of the grinding products was estimated using the PSC – 2 device. Weighing was carried out on a T – 1000 laboratory balance with an accuracy of 0,02 g.

As the alkaline component of the binder used liquid sodium glass (GOST 13078-81) and caustic soda technical (GOST 2263-79).
Large aggregate – crushed granite fraction 5-10 mm. Fine aggregate – Sursky sand with a modulus of particle size \( M_{ps} = 1,1-1,3 \) and sand Volsky with a modulus of grain size \( M_{ps} = 2,2 \div 2,4 \). Superplasticizer (SP) – MELFLUX 5581 liquid, concentration of 29 %. Water-drinking, plumbing.

The compaction was performed by vibration on a platform vibrator in the time interval from 2 to 5 seconds. Hardening of concrete samples occurred in the chamber of normal hardening at a temperature of 20-250°C and humidity of 90 %. Samples were tested for compressive strength. For testing the strength of the sample were made cubes with a size of 100 × 100 × 100 mm. The composition for testing was prepared in a laboratory mixer.

We studied the composition of concrete with a dosage of fiber 3, 10, 30 % by weight of cement; the value of W/C changed from 0,28 to 0,45; the effect of electrolytes was studied individually and in combination; the mixture manufacturing technology, technological parameters of mixing and compaction changed.

Fine-grained concrete used for the manufacture of paving slabs has a structure with characteristic properties: high uniformity, fine grain, high ductility of cement stone, the absence of a rigid stone skeleton. The test of fine-grained concrete for strength was carried out on samples of beams of 40×40×160 mm in size and cubes of 50×50×50 mm.

For high-quality compaction, the concrete mixture was vibrated with the load \( P = 2,4 \text{ kg/cm}^2 \). The results of testing samples on fine-grained concrete with fiber filling the structure in the amount of 30 % by weight of cements are presented in Table 2.

**Table 2.** Strength indicators of fine-grained concrete based on portland cement, modified fiber.

| №  | Composition | Material consumption per 1 m³, kg | W/C | Compressive strength (MPa) when stored under normal conditions 3 days 28 days | Densit \( y_{sd} \), kg/m³ | Notes |
|---|---|---|---|---|---|---|
| 1 | Cement CEM I 42,5H Cellulose Sand Water | 600 180 (30 %) 1000 360 | 0,6 0,25 2,0 | 2000 | | Self-destruction during storage in water for 30 days |
| 2 | Cement CEM I 42,5H Cellulose Sand CaCl₂ Water | 600 180 (30 %) 1000 3 % (18 kg) 360 | 0,6 3,05 3,5 | 2050 | | Self-destruction during storage in water for 30 days |
| 3 | Cement CEM I 42,5H Cellulose SandCaCl₂ Water | 600 180 (30 %) 1040 2,4 % (14,4 kg) 360 | 0,4 3,42 5,0 | 2100 | | Self-destruction during storage in water for 30 days |
| 4 | Cement CEM I 42,5H Cellulose Sand MELFLUX | 600 180 (30 %) 1040 0,383 1,02 2,0 | 2180 | | Self-destruction during storage in water for 30 days |
The data in Table 2 showed that fiber has a blocking effect on the curing of cement stone at a dosage of 30% by weight of portland cement. An aqueous solution of CaCl₂ electrolyte was used as an activator of hardening cement paste. The dosage corresponded to an optimum for usual concrete structures. When stored in water, samples with fiber swell and self-destruct, which is unacceptable for structures that are in a constant state of drying and moistening.

The use of electrolyte at a dosage of fiber 30% by weight of cement does not lead to positive results (Table 2). Thus, the use of fiber as a modifier of concrete based on Portland cement is not rational, because Concrete samples have a low value of compressive strength and self-destruct during water storage.

To identify the possibility of using rigid mixtures in the manufacture of paving slabs and side stones, the products were pressed at high pressure $P = 240$ kg/cm² and W/C = 0.34. The results of tests of concrete samples are presented in Table 3.

**Table 3. Strength indices of pressed concrete samples.**

| №  | Composition          | Material consumption per 1 m³, kg | W/C | Compressive strength, MPa | Density $(\gamma_{sd})$, kg/m³ | Hardening in water for 30 days  |
|----|----------------------|----------------------------------|-----|---------------------------|--------------------------------|---------------------------------|
| 1  | Cement I CEM I 42,5H| 204                              | 0.34| 0,8                       | 40,00                          | 2200                            |
|    | Sand                 | 1,2 %                            | 0.34| 0,8                       | 0,15                           | 2070                            |
|    | Water                | 204                              | 0.34| 9,6                       | 6,8                            | 2187                            |

Notes: heat and moisture treatment (HMT) $2 + (2 + 2) + 2$; Composition 1 had high plasticity and was molded under normal vibrations; N.H. – hardening in the chamber of normal hardening; Compounds 2 and 3 are pressed at $P = 240$ kg/cm².

As a result of pressing under load $P = 240$ kg/cm², noticeable pressing of concrete samples occurs, which is confirmed by an increase in their density, however, due to the lack of moisture, the processes of hydration of cement clinker are not fully implemented (Table 3). This once again confirms the ability of fiber in its developed porous structure to retain water, delaying it to hydrate the cement. During HMT, destructive changes occur in the samples, which lead to loss of concrete strength, and during storage of the samples in water for 30 days – to self-destruction.

Thus, the feature of cement-sand mixtures with fiber additives is revealed, which completely excludes the possibility of using traditional technologies for the preparation of mixtures for the manufacture of concrete products.

Analysis of the results of research and literature [3, 9], which took into account the pattern of redistribution of intercellular and intracellular moisture of cellulose in contact with mineral powders, indicates the need for a complete change in the technology for preparing cement-sand mixtures. In this regard, a separate cooking technology was proposed. The essence of which consists in mixing with a
high-speed mixer of fine aggregate and cellulose with the subsequent introduction of mixing water with SP, after 2 minutes binder is injected.

The concrete mix was compacted in forms by vibrating without weights. The results of the strength improvement of concrete are presented in Table 4.

**Table 4.** Strength of fine-grained concretes manufactured by separate technology.

| Composition          | Material consumption per 1 m³, kg | W/C  | Compressive strength, MPa | Density (γsd), kg/m³ |
|----------------------|----------------------------------|------|--------------------------|----------------------|
|                      |                                   |      | N.H. after | after HMT | after 28 | 12 | 28 | days | hours | days |
| Cement Volsky Sand   | 570                               | 0,45 | 3,2 | 21 | 26,1 | 2200 |
| Water                | 1450                              |      | 57  | 1325 | 68,4 | 256 |
| Cement Volsky Sand   | 570                               | 0,45 | 10,8 | 20,1 | 26,6 | 2190 |
| Cellulose 10 %       | 57                                |      | 1325 | 68,4 | 256 |
| Sand                 |                                   |      | 1325 | 68,4 | 256 |
| MELFLUX 5581- 1,2%   |                                   |      | 1325 | 68,4 | 256 |
| Water                |                                   |      | 1325 | 68,4 | 256 |

The use of separate technology allowed us to obtain plastic mixtures containing 10 % fiber with a higher kinetics of increase in strength under normal hardening conditions (as compared with control compositions that do not contain fiber). Self-destruction of concrete samples during storage in water conditions was absent.

Slag alkaline binder significantly differs from Portland cement in that it has a high alkalinity (pH13-15) at the time of its preparation. The test results of fine-grained concretes based on a slag binder are presented in Table 5.

**Table 5.** The kinetics of the strength of modified slag-alkaline concrete at electrothermophosphoric over time.

| Fiber dosage, % by weight of slag | Compressive strength, MPa |
|----------------------------------|----------------------------|
|                                  | Hardening in normal conditions, days | Hardening in normal conditions after HMT, days |
|                                  | 28  | 90  | 180 | 360 | 720 | 28  | 90  | 180 | 360 | 720 |
| –                                 | 75,5 | 80,6 | 86,2 | 90,0 | 93,1 | 71,9 | 76,6 | 84,0 | 89,3 | 91,1 |
| 3                                 | 80,8 | 84,7 | 90,3 | 93,6 | 96,0 | 79,8 | 82,3 | 87,8 | 91,1 | 95,5 |
| 10                                | 52,0 | 58,9 | 63,1 | 69,9 | 72,6 | 50,0 | 54,5 | 64,7 | 66,2 | 71,8 |

Notes: alkaline scavenger – NaOH + liquid glass (1: 1); slag consumption 475 kg/m³; the composition of the concrete mixture – slag: crushed stone: sand = 1: 2,52: 1,43; solution / slag = 0,34

Studies of changes in the strength of slag-concrete concretes over time were carried out on samples that were both solidified under normal conditions for 720 days, and on samples subjected to HMT and then solidified under normal conditions. The data obtained indicate that the strength of specimens subjected to HMT does not exceed the strength of specimens of similar compositions that were hard in normal conditions. The test results are presented in Table 5.

The data show that over time, the strength of the samples gradually increases, and the samples that have solidified under normal conditions exceed the strength of the samples subjected to HMT.
The most intensively the curing is observed in the range from 90 to 360 days both for those hardened in normal conditions and for the specimens subjected to HMT. For the control composition, the magnitude of the increase in strength to 720 days is 17.7-19.2 MPa, and with the addition of fiber – 15.2-15.7 MPa and 20.6-21.8 MPa, respectively.

Thus, the use of fiber and activated sludge in the production of building materials based on portland cement and clay is inefficient.

Receiving modified concretes on the basis of slag binder is possible as can be seen from the obtained results, the optimal dosage of fiber is 3 % by weight of slag. The introduction of a modifying additive contributes to the formation of a fine-crystalline structure (x-ray diffraction data and electron microscopic studies [10]), which leads to an increase in the strength and durability of products.

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