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CONCRETES WITH ADDITIVES PRODUCED BY SOL-GEL METHOD

Larisa A. Urkhanova [1], Solbon A. Lkhasaranov [2], Andrey A. Ivanov [2], Dmitry V. Dobrynin [2], Imad Jamal Moslemani [3]

[1] National Research Moscow State University of Civil Engineering, Moscow, RUSSIA
[2] East Siberia State University of Technology and Management, Ulan-Ude, RUSSIA
[3] Moscow Automobile and Road Construction State Technical University (MADI), Moscow, RUSSIA

Abstract: Additives with various functional purposes are used to modify concrete. Ultra- and nanodispersed additives produced using sol-gel technologies show great promise. The article examines different methods for producing modifying additives of silicon dioxide sol and metal hydroxides to enhance the properties of concrete for unique structures, civil engineering projects, and road construction.

The research utilized aluminosilicate materials—perlite rocks with varying degrees of crystallinity—as active mineral additives. Ultra-dispersed additives were obtained through the hydrolysis of a strong base and weak acid salt—sodium silicofluoride (Na_2SiF_6), hydrolysis of Portland cement, and hydrolysis of inorganic salts such as $CaCl_2$, $Ca(NO_3)_2$, and $CaSO_4$. Another method for producing a colloidal additive to modify cement stone and concrete involved mixing liquid colloidal fungicidal sulfur with water.

Modification with sulfur sol increases concrete strength by 10–25% for constructing fuel storage buildings and hydraulic structures of nuclear power plants.

Concrete for hydraulic structures using a complex additive containing silicic acid sol and superplasticizer is characterized by high physical-mechanical, hydrophysical, and operational performance.

When using an ultra-dispersed additive obtained through Portland cement hydrolysis, the strength of heavy concrete increases by 24% at the design age.

It has been established that modifying concrete for road pavements with micro-dosages of colloidal solutions of inorganic salts ($CaCl_2$, $Ca(NO_3)_2$, $CaSO_4$) increases the compressive and flexural strength of the composite. Ultra-dispersed additives have the greatest impact on accelerating the gain of concrete strength within the first 7 days of curing.

Keywords: heavy concrete, Portland cement, ultrafine additives, sol-gel method, silicic acid sol, superplasticizer, vitreous perlite, crystallized perlite, compressive strength, flexural strength, dense concrete structure

БЕТОНЫ С ДОБАВКАМИ, ПОЛУЧЕННЫМИ ЗОЛЬ-ГЕЛЬ МЕТОДОМ

Л.А. Урханова [1], С.А. Лхасаранов [2], А.А. Иванов [2], Д.В. Добрынин [2], Имад Жамаль Мослемани [3]

[1] Национальный исследовательский Московский государственный строительный университет, г. Москва, РОССИЯ
[2] Восточно-Сибирский государственный университет технологий и управления, г. Улан-Удэ, РОССИЯ
[3] Московский автомобильно-дорожный государственный технический университет (МАДИ), г. Москва, РОССИЯ

Аннотация: Для модификации бетона используются добавки, обладающие различным функциональным назначением. Перспективными являются ультра- и нанодисперсные добавки, полученные по золь-гель технологиям. В статье рассмотрены различные способы получения модифицирующих добавок золя диоксида кремния и гидроксидов металлов для улучшения свойств бетонов для уникальных сооружений, объектов гражданского и дорожного строительства.

В исследованиях в качестве активных минеральных добавок были использованы алюмосиликатные материалы - перлитовые породы с разной степенью кристалличности. Ультрадисперсные добавки получали путем гидролиза соли сильного основания и слабой кислоты - кремнефтористого натрия

Na_2SiF_6, гидролиза портландцемента, гидролиза неорганических солей, таких как $CaCl_2$, $Ca(NO_3)_2$, CaSO4. Еще одним способом получения коллоидной добавки для модификации цементного камня и бетона являлся процесс перемешивания с водой жидкой коллоидной фунгицидной серы.
Модифицирование золем серы позволяет увеличить прочность бетона на 10-25% для строительства зданий хранилищ топлива и гидротехнических сооружений атомных электростанций.
Бетоны для гидротехнических сооружений с применением комплексной добавки, содержащей золь кремниевой кислоты и суперпластификатор, характеризуются высокими физико-механическими, гидрофизическими и эксплуатационными показателями.
При использовании ультрадисперсной добавки, полученной при гидролизе портландцемента, происходит увеличение прочности тяжелого бетона на 24% в проектном возрасте.
Установлено, что при модификации бетона для покрытий автомобильных дорог микродозировками коллоидных растворов неорганических солей $CaCl_2$, $Ca(NO_3)_2$, $CaSO_4$ происходит увеличение прочности при сжатии и изгибе композита. Наибольшее влияние ультрадисперсные добавки оказывают на ускорение набора прочности бетона в возрасте до 7 суток твердения.

Ключевые слова: тяжелый бетон, портландцемент, ультрадисперсные добавки, золь-гель метод, золь кремниевой кислоты, суперпластификатор, стекловидный перлит, закристаллизованный перлит, прочность при сжатии, прочность при изгибе, плотная структура бетона

INTRODUCTION

The use of concrete in modern construction is unimaginable without various functional additives: plasticizing agents with water-reducing effects or providing rheological properties of the concrete mix [1-3], active mineral additives that increase the strength and density of the cement system [4, 5], modifying additives that improve the deformation and special properties of cement material, for example, elastic properties or corrosion resistance [6, 7].

Among the wide variety of used additives, the modification of concrete with ultra- and nanodispersed additives, especially SiO_2 nanoparticles as a basis for the pozzolanic reaction, is considered one of the most promising methods for improving the physicomechanical properties of the composite and is gradually being introduced into domestic construction practice. However, the low rate of implementation is associated with the lack of available industrial technologies for synthesizing additives and, as a consequence, limited practical experience in using them to modify the structure and properties of concrete [1].

Among all known methods for producing ultra- and nanodispersed particles, the sol-gel technology is particularly popular due to its accessibility and low-cost raw materials [8]. At the same time, sol-gel technologies for synthesizing additives differ in the type of precursor used, the composition of catalysts, the structure of the solvent, and the type of stabilizer [9].

The effectiveness of controlling the shape and size of particles and the nature of their surface during synthesis can be ensured by changing external conditions: pH of the medium, precursor concentration, solvent, aging temperature and time, nature of the catalyst, etc. Varying these parameters affects the reaction rate, the mechanism of the sol-gel transition, the nature of the polymerized particles formed in the sol, and the volumetric nature of the gel [9, 10].

The type of precursor used and the concentration of the aqueous solution determine the composition, shape, and size of the ultra- and nanodispersed particles of the obtained additives, resistance to external influences, aggregative and sedimentation stability, reactivity in the environment of hydrating cement, and cost.

Taking into account modern physicochemical concepts and approaches to producing ultra- and nanodispersed particles, the research team conducted studies on modifying the structure and properties of cement concrete for unique structures, civil and road construction facilities using additives of various compositions obtained using sol-gel technology.

Ultra-dispersed additives of silicon dioxide and metal hydroxides in the form of sol will help reduce porosity and improve the microstructure of cement stone, enhancing the physicomechanical properties of concrete when used.

MATERIALS AND METHODS

To improve the properties and enhance the physical-mechanical and operational characteristics of concrete based on ultrafine additives, Portland cement CEM I 42.5 N (GOST 31108-2020) was used as a binder. The cement was supplied by Timlyuicement LLC and Cementum Center LLC.

As active mineral additives (fillers) in the concrete mix composition, finely ground vitreous and crystallized perlite from the Mukhor-Tala deposit (Republic of Buryatia) were used, depending on the composition. The chemical compositions of vitreous and crystallized perlite are presented in Table 1.

Table 1-Chemical composition of glassy and crystallized perlite, mass.%

As surfactants (surface-active agents), superplasticizers based on modified polycarboxylates (from Poliplast-Siberia LLC) and naphthalene-formaldehyde superplasticizer S-3 were used. These were added to the mixing water in amounts of 0.2% and 0.7% of the cement mass, respectively.

The study proposed low-temperature, technologically simple, and accessible methods for producing ultrafine additives (UFA) through the hydrolysis of a salt of a strong base and a weak acid — sodium silicofluoride (Na_2SiF_6). The aim was to modify the properties of cement stone with colloidal particles of silicofluoride acid and hydrolysis salt products, as well as Portland cement (PC) [11, 12].

The multicomponent sol obtained during PC hydrolysis contains simultaneously silicic acid, aluminum hydroxide, iron hydroxide, and calcium hydroxide [12]. Additionally, UFA were obtained through the hydrolysis of inorganic salts such as $CaCl_2$, $Ca(NO_3)_2$, $CaSO_4$, and $CuSO_4$. As a result of the multistage salt hydrolysis process, hydroxide salts of the corresponding metals are formed [13,14].

Another method for producing a colloidal additive to modify cement stone and concrete was the process of mixing liquid colloidal fungicidal sulfur (LCFS) with water. LCFS was obtained through the processing of sulfur-containing industrial waste and contains 40% sulfur. When mixed with water, LCFS turns into a suspension due to the nanoscale size of sulfur particles — 15–35 nm [15].

The additive consumption, at mother liquor concentrations of 0.75% and 2.0%, ranged from 0.2–3.0% of the cement mass. This, when recalculated to dry matter, amounted to thousandths of a percent, depending on the type of additives and the water-cement ratio.

The concrete mix, with workability grade P1–P3 depending on the concrete application area, was prepared by mixing pre-dosed components in a laboratory concrete mixer for 1 minute. These components included Portland cement, finely dispersed perlite, sand with a fineness modulus M_f=2.1, crushed stone fractions of 5–10 and 10–20 mm, and water containing dosed additives obtained by sol-gel technology and superplasticizers.

From the concrete mix of equal mobility, cube samples measuring 100 × 100 × 100 mm were molded and tested using standard methods. For each type of test, samples were manufactured in accordance with the requirements of GOST 10181-2014 «Concrete mixtures. Testing methods», GOST 10180-2012 «Concretes. Methods for determining strength using control samples», GOST 12730.5-2018 «Concretes. Methods for determining water resistance», GOST 24452-2023 «Concretes. Methods for determining prismatic strength, elastic modulus, and Poisson's ratio».

RESULTS AND DISCUSSION

It is known that one of the main problems arising during the synthesis of additives using

the sol-gel method is the aggregative and sedimentation instability of their aqueous solutions, which subsequently transform into a gel. To stabilize the ultrafine additives (UFA), surfactants based on naphthalene-formaldehyde and polycarboxylates were added to the reaction solution. These surfactants tend to adsorb onto the hydrophilic surface of ultrafine particles, preventing their further aggregation and extending the storage life of the additives to 15–20 days without altering their properties.

All the studied additives, when used in rational quantities, ensure the improvement of the physical and mechanical properties of concrete for various functional purposes, accelerating the hardening process during the initial hardening period. The introduction of micro-dosages of sulfur sol allows for an increase in concrete strength by 10–25% for the construction of fuel storage buildings and hydraulic structures of nuclear power plants, or to reduce cement consumption when designing the composition of the concrete mix. Simultaneously, this accelerates the hardening processes and the achievement of design strength, while increasing the corrosion resistance of concrete, which leads to an extension of the service life of the structures (Table 2).

Table 1. Chemical composition of glassy and crystallized perlite, mass.%

Material	SiO_2	Al_2O_3	Fe_2O_3	FeO	CaO	MgO	TiO_2	SO_3	K_2O	Na_2O	R_2O	LOI
Vitreous perlite	68,40	13,75	0,50	0,06	0,50	0,30	0,15	0,10	3,91	3,37	-	5,97
Crystallized perlite	73,13	11,7	1,08	1,71	0,55	0,30	-	-	-	-	6,4	7,65

Table 2. The effect of the amount of liquid colloidal fungicidal sulfur additive on the rate of strength gain of concrete for nuclear power plant structures

A type of sulfur sol	Amount of additive, % of cement mass		Compressive strength, MPa, after hardening, days			Ratio of strength indicators, $R\tau$ / R_{28}	
	counting on the solution	based on dry weight	3	7	28	τ = 3 days	τ = 7 days
Liquid colloidal fungicidal sulfur	0.6	$4.8 \cdot 10^{-3}$	22	34	39	0.39	0.76
	1	$6.4 \cdot 10^{-3}$	21	35	40	0.56	0.87
	1.5	$8.0 \cdot 10^{-3}$	20	34	49	0.53	0.90
	2	$9.6 \cdot 10^{-3}$	19	31	46	0.52	0.87
	3	$14.4 \cdot 10^{-3}$	19	27	44	0.49	0.78
Concrete without additives	-	-	15	30	39	0.39	0.76

Taking into account the modern requirements of the construction industry, it is necessary to ensure an appropriate level of safety for massive concrete and reinforced concrete hydraulic structures, primarily those that have been in long-term operation and require repair or reinforcement.

One of the main problems of hydraulic concrete is dangerous cracking, which leads to the rapid decommissioning of hydraulic structures. Obtaining hydraulic concrete with enhanced operational properties can be achieved through multi-level regulation of the structure and properties of the binder using a complex additive containing silicic acid sol and superplasticizer, which interacts with cement hydration products at the nano-, meso- and macro-levels, and also affects the formation of the structure and properties of cement stone.

As research results have shown, concretes using a complex additive are characterized by high physical-mechanical, hydro physical and operational indicators, which confirms the creation of a dense material structure and the strengthening effect of the additive.

The use of vitreous perlite in concrete improves the technological properties of the concrete mix, reduces the content of Portland cement in the mix composition, and together with the complex additive leads to an improvement in the basic properties of hydraulic concrete with composite binder (Table 3).

One of the promising methods for producing multicomponent silicic acid sol is the hydrolysis of Portland cement. The process of Portland cement hydrolysis occurs in several stages, with the formation of hydroxide salts containing the corresponding metallic elements at one of the intermediate stages.

A heavy concrete was developed for modification for monolithic construction having the following composition: Portland cement — 420 kg, sand — 760 kg, crushed stone with a fraction of 5–20 mm — 930 kg, water — 215 liters. The consumption of the ultrafine additive in the form of a 2% solution amounted to 5% of the binder mass (Table 4).

Table 3. Effect of a complex additive containing silicic acid sol (sodium fluorosilicate hydrolysis) and surfactants on the properties of hydraulic concrete

Composition	Compressive strength, MPa		Flexural strength, MPa		Crack resistance index	Water absorption, %	Water resistance	Frost resistance, cycles, F_2	Corrosion resistance coefficient	Modulus of elasticity E_b, MPa×10^3	Prism strength, MPa	Poisson's ratio	Total porosity, cm^3/g
	3 days	28 days	7 days	28 days									
PC	24.6	38.7	4.1	7.1	0.18	4.90	W10	150	1.32	32.362	34.6	0.216	0.096
PC+20% perlite + superplasticizer	28.8	48.1	4.3	7.5	0.19	3.66	W12	200	1.62	35.680	40.2	0.202	0.090
PC+ complex additive	30.2	45.5	6.9	10.6	0.21	3.05	W14	300	1.79	34.493	39.6	0.191	0.089
PC+ 20% perlite + complex additive	35.2	53.4	7.8	10.9	0.22	2.43	W16	300	1.86	37.345	46.3	0.199	0.081

Table 4. Effect of ultrafine additive obtained by hydrolysis of Portland cement on the properties of heavy concrete

Properties	Indicators of concrete	
	control	with additives
Concrete mix mobility (slump), cm	13	12
Volume of entrained air, %	4.3	4,5
Average density of concrete, kg/m^3	2320	2335
Compressive strength, MPa, at age		
3 days	14.5	18.5
7 days	21.3	29.3
28 days	32.1	39.8

The introduction of a multicomponent sol in the form of an ultrafine additive provides a significant improvement in the strength characteristics of concrete: the compressive strength of concrete increases by 37% compared to the control composition at an age of up to 7 days of hardening and by 24% at the design age. This effect is achieved due to the active interaction of the ultrafine additive with the hydration products of Portland cement clinker minerals.

During the hydrolysis of Portland cement, active dissolution is observed not only of exchange ions, but also of hydrated forms of ions $Ca(OH)^{+}$ and $(H_2SiO_4)^{2-}$, which sequentially saturate the aqueous solution. After that, the formation of new crystalline phases occurs in the volume and on the surface of the particles (Fig. 1).

Concrete mix for road pavements must comply with the requirements and conditions of regulatory documents [16].

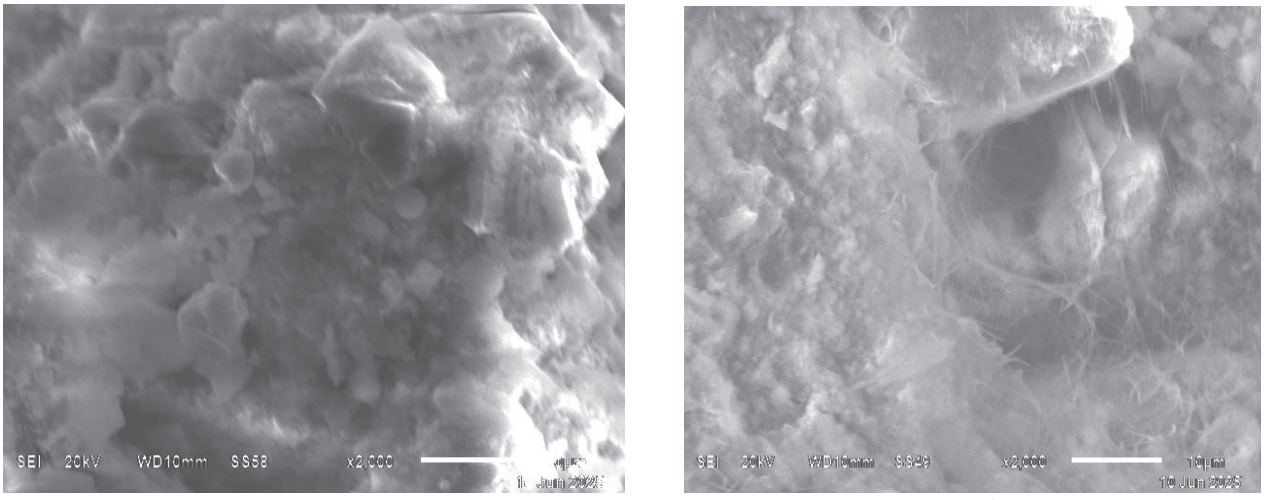
The production of high-quality concrete for road construction requires the use of quality components: cement, mineral additives, sand, crushed stone, chemical additives. It is also necessary to create optimal conditions for concrete hardening, which will increase the durability and reliability of cement-concrete structures by regulating the properties of the composite [16, 17].

When producing concrete mix, it is important to ensure uniformity, workability, and a rational water-cement ratio.

According to GOST R 59300, the concrete mix for road pavements constructed in sliding formwork must meet the P1 grade (cone slump from 1 to 4 cm) in terms of workability and the required volume of entrained air from 5% to 7%. This mobility class was adopted to ensure better placement and compaction of the concrete mix without surface defects, in combination with other properties of the concrete mix.

For research purposes, a concrete mix design calculation was carried out for road pavements of technical category I with a compressive strength class of B30. The dosage of additives in the form of a 2% solution in the concrete mix was 0.1–0.2% of the cement mass, calculated on dry salt.

The analysis of the results obtained showed that $CaCl_2$ and $CaSO_4$ additives increase the strength of concrete by an average of 22–42% during the design hardening time, effectively accelerating the concrete hardening process in the early stages of hardening and increasing its strength by 40–90% on the 3rd day of hardening (Fig. 2).



a *b*

Figure 1. Microstructure of cement stone modified with an ultrafine additive obtained by hydrolysis of Portland cement (x2000): a – control, b – with an ultrafine additive

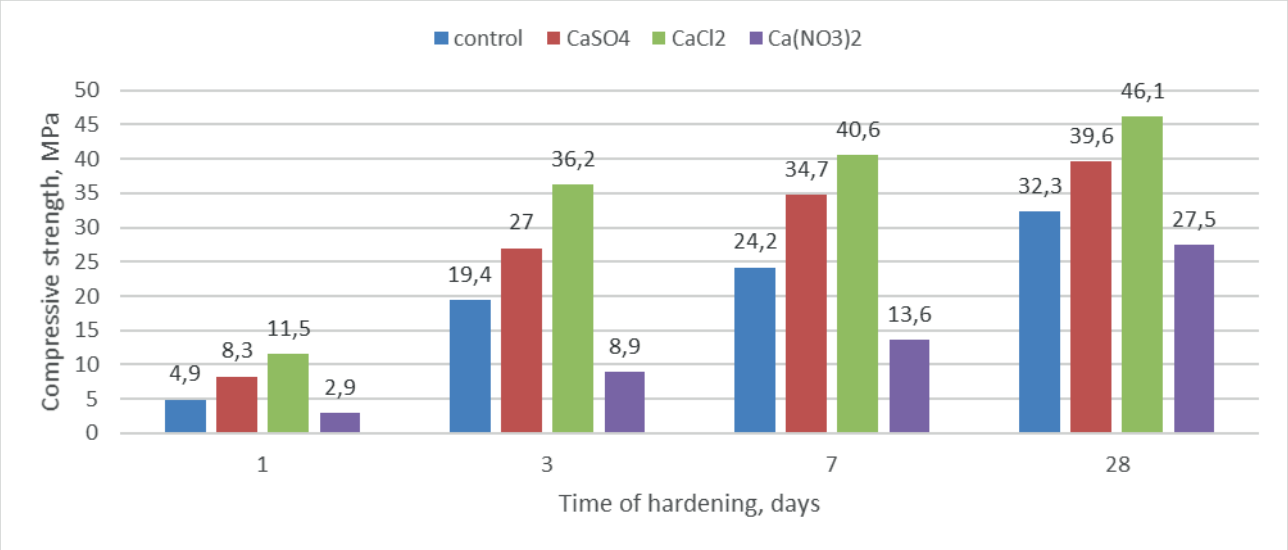


Figure 2. Kinetics of hardening of concrete for road surfaces with modifying additives obtained using sol-gel technology

The increase in strength of Portland cement and concrete with micro-dosages of additives is associated with changes in the porosity of the hydrate stone, its phase composition, and microstructure.

The tensile strength index in bending of concrete is important for ensuring the durability and safety of road surfaces. Road surfaces are constantly exposed to dynamic loads, including bending loads that occur under vehicle loads and temperature deformations.

Various methods are known to increase the tensile strength in bending of concretes for road pavements — mainly micro-reinforcement with fiber [18]. However, it is also possible to increase the strength characteristics of concrete by modifying the structure of the cement stone in concrete using chemical or electrophysical methods [18–21].

A study of the tensile strength in bending of concrete samples using micro-dosing of $CaCl_2$ salt (Table 4) showed that the use of an additive in the form of a 2% solution in an amount of 0.2%, calculated on dry salt from the mass of cement, increases the tensile strength in bending by 45% in 28 days of hardening.

Table 4. Results of tensile testing of cement concrete under bending

Composition	Flexural tensile strength of concrete, MPa, at age, days			
	1	3	7	28
PC	0.6	1.1	1.5	2.9
PC+$CaCl_2$	1.6	1.9	2.7	4.2

The hydroxides formed by multivalent metals in the form of colloidal solutions with varying time-dependent stability immobilize part of the water into the hydration shell. This leads to a decrease in the water-to-cement ratio while maintaining the rheological characteristics of the cement paste and the formation of a denser structure of the cement stone.

Such constrained conditions promote the growth of fibrous calcium hydrosilicates, thereby increasing the tensile strength in bending of concrete.

CONCLUSION

Here are the results of the authors' research on improving the physical-mechanical and operational properties of concrete for various functional purposes in civil, hydraulic, and road construction using modifying additives produced by sol-gel technologies.

The processes of producing additives using various precursors and their concentrations have been studied. It has been substantiated that the selection of stabilizers and optimization of production conditions allows for the targeted regulation of the synthesis of modifying additives with the required particle morphology and size, as well as physicochemical properties.

It has been proven that the use of additives accelerates the hardening processes and the rate of strength gain in concrete, which is associated with changes in the structure and phase composition of cement composites. The developed methods and approaches for producing modifying additives must meet the technical and economic criteria for the effective use of concrete for its intended purpose, taking into account the requirements of regulatory documents and design documentation.

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Larisa Alekseevna Urkhanova, Doctor of Technical Sciences, Professor, Professor of the Department of Urban Planning, National Research Moscow State University of Civil Engineering, 26 Yaroslavskoe Highway, Moscow, 129337, Russia, urkhanova@mail.ru

Solbon Alexandrovich Lkhasaranov, Candidate of Technical Sciences, Associate Professor, Head of the Department of Construction Materials, Highways and Woodworking, East Siberian State University of Technology and Management, 40V Klyuchevskaya Street, Ulan-Ude, 670013, Russia, solbon230187@mail.ru

Andrey Andreevich Ivanov, Lecturer of the Department of Construction Materials, Highways and Woodworking, East Siberian State University of Technology and Management, 40V Klyuchevskaya Street, Ulan-Ude, 670013, Russia, vadim290607@gmail.com

Dmitry Vyacheslavovich Dobrynin, Postgraduate Student of the Department of Construction Materials, Highways and Woodworking, East Siberian State University of Technology and Management, 40V Klyuchevskaya Street, Ulan-Ude, 670013, Russia

Imad Jamal Moslemany, Postgraduate Student of the Department of Road Construction Materials and Chemical Technologies, Moscow Automobile and Road Construction State Technical University (MADI), 64 Leningradsky Prospekt, Moscow, 125319, Russia

Лариса Алексеевна Урханова, д-р техн. наук, профессор, профессор кафедры «Градостроительство», Национальный исследовательский Московский государственный строительный университет, дом 26, Ярославское шоссе, г. Москва, 129337, Россия, urkhanova@mail.ru

Солбон Александрович Лхасаранов, канд. техн. наук, доцент, зав. кафедрой «Строительные материалы, автомобильные дороги и деревообработка», Восточно-Сибирский государственный университет технологий и управления, дом 40В, ул. Ключевская, г. Улан-Удэ, 670013, Россия, solbon230187@mail.ru

Андрей Андреевич Иванов, преподаватель кафедры «Строительные материалы, автомобильные дороги и деревообработка», Восточно-Сибирский государственный университет технологий и управления, дом 40В, ул. Ключевская, г. Улан-Удэ, 670013, Россия, vadim290607@gmail.com

Дмитрий Вячеславович Добрынин, аспирант кафедры «Строительные материалы, автомобильные дороги и деревообработка», Восточно-Сибирский государственный университет технологий и управления, дом 40В, ул. Ключевская, г. Улан-Удэ, 670013, Россия

Имад Жамаль Мослемани, аспирант кафедры «Дорожно-строительные материалы и химические технологии» Московский автомобильно-дорожный государственный технический университет (МАДИ), дом 64, Ленинградский проспект, г. Москва, 125319, Россия