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# **CONTENTS**



<span id="page-3-0"></span>

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#### *Article*

# **Testing of cement stone modified with an additive based on industrial waste**

[Daniyar Bazarbayev](https://orcid.org/0000-0001-8547-5440Daniyar%20Bazarbayev1)<sup>1,\*</sup>,DAdiya Zhumagulova<sup>1,</sup>, DDanagul Kadyrkhanova<sup>2</sup>

<sup>1</sup>Department of Technology of Industrial and Civil Engineering, L.N. Gumilyov Eurasian National University, Astana, Kazakhstan

<sup>2</sup>GoldConstructionGroup, LLP, Kosshy City, Kazakhstan \*Correspondence: [phdd84@mail.ru](mailto:phdd84@mail.ru)

**Abstract.** Nowadays under the conditions of intensively developing construction production, hardening time and strength of Portland cement are becoming more and more relevant research issues of this type of binder. The basic requirements to the cement stated in the normative industry documents are considered. The strength limits of Portland cement of normal and fast-hardening are given. Raw materials for the production of fast-hardening and high-strength cement must be homogeneous and provide a high degree of fineness of the binder to increase its surface area. The production of cement with the addition of microsilica will make it possible to obtain concretes with the given characteristics without additional costs. To describe the methodology of testing cement recommendations of the main regulatory documents operating in Kazakhstan are used. Necessary means and equipment used in the testing laboratory and satisfying the existing standards are listed in a sequential manner. The procedure of preparing standard samples of cement dough is described step by step. The prepared specimens were kept in water at the required temperature regime. Determination of bending and compressive strength was carried out on accredited equipment. Processing of test results of cement samples with and without additives is given. Setting times of cement samples of two compositions were carried out according to the requirements. The dependence of the activity of the cement setting process on its temperature has been determined. **Keywords:** fast-hardening cement, high-strength cement, active mineral additive, test of strength, cement setting time.

#### **1. Introduction**

For a number of construction needs, in particular, for the factory production of reinforced concrete building structures and parts, as well as for high-speed construction, fast-hardening cement is required, which is more intense than conventional Portland cement, characterized by increased strength at the initial stage of hardening. Over a long period of time, the growth of strength in it slows down and after a long period of time can reach the strength of ordinary Portland cement [1, 2].

After 3 days, fast-hardening Portland cement M400 and M500 in accordance with Interstate Standard GOST 31108-2020 [3], the tensile strength must be at least 25 and 28 MPa, respectively, and after 28 days – 40 and 50 MPa. For these brands, the bending strength limit should not be less than 4 and 4.5 MPa after 3 days. According to Interstate Standard GOST 31108-2020 [3], the state Quality Mark was issued for fast-hardening cement, the bending strength limit after 3 days should be at least 4.5 MPa, the tensile strength after 3 days – at least 28, and after 28 days – at least 40 MPa. Tensile strength when compressing samples from this cement after 1 Day 3+2+2 must be at least 26 MPa.

The use of fast-hardening cement in factories for the production of reinforced concrete structures and parts significantly speeds up the production process [4]. The use of fast-hardening portland cement for monolithic concrete reduces the cutting time and reduces the mass of the structure, since the high strength of the resulting concrete allows you to reduce the cross-section of the structure [4]. To obtain high-grade prestressed concrete, high-strength Portland сement is

required. According to Interstate Standard GOST 31108-2020 [3], when testing on solutions of plastic consistency, the tensile strength of this cement at the age of 28 days should be at least 60 MPa and bending strength limit – at least 6.5 MPa. This cement should not have signs of "false installation".

Raw materials to produce fast-hardening and high-strength portland cement should be as homogeneous as possible in chemical composition and contain the minimum number of undesirable impurities [5]. The atmospheric or small crystal structure of the material is preferred they enter chemical interactions more easily than materials of a large crystal structure. It is desirable that the raw material mixture does not contain thermally resistant aluminosilicate of magnesian and siliceous and calcareous feldspar of limestone. Fast-hardening cement and high-strength Portland cement as raw materials, marls and montmorillonite clays are easier to learn when they are easily blurred and have a high surface [6].

To fulfill the tasks and goals, we used materials that meet the requirements of state standards. The raw material, in turn, was adopted in accordance with the quality of the material provided for reinforced concrete structures or hydraulic structures [3, 7–9].

The main characteristics that we have taken for research are presented in Tables 1, 2 which are shown below.



Table $2$ – Mineral composition of cement									
Cement	Mineral composition, %				Alkali content, %		Gypsum	Active mineral	
designation	$C_3S$	$C_2S$	$C_3A$	$C_4AF$	$K_2O$ Na <sub>2</sub> O		content by	supplements,	
							$SO_3$ , %	$\%$	
	47.91	19.09	8.64	13.56			2.10		

Polyfractional sand was selected for testing of cement in accordance with the requirements of Interstate Standard GOST 6139-2020 [10].

Microsilica is formed in the melting process of ferrosilicon and silicon. After oxidation and condensation, the percentage of silicon monoxide is very small. The use of microsilica gives us a great opportunity to produce concretes with special characteristics for structures and products from simple materials. Concrete with microsilica has an excellent compressive strength that exceeds the strength of conventional concrete [11, 12].

In carrying out tests of cement were used standard methods of examination, which meet the requirements of the Standards of the Republic of Kazakhstan, Interstate standards.

#### **2. Methods**

In carrying out tests of cement were used standard methods of examination, which meet the requirements of the Standards of the Republic of Kazakhstan, Interstate standards.

To determine the high-strength cement, special controls and additional equipment were used, as well as a mixer equipped with a dosing device for sand.

Triple  $40 \times 40 \times 160$  mm mold for making samples. The design of their mold must provide the ability to remove finished samples from them without defects or damage. This is necessary for ease of use when forms are attached to each other and to the baseplate to prevent water leakage. The baseplate must be firm to prevent subsequent vibrations. Forms for the plates are 210×185 mm in size and 6 mm thick, they must be made of glass with smooth edges.

Trowel and ruler – for laying the cement mortar.

A vibrating table was used to compact the mortar in the formwork.

A device for determining the bending strength of finished beam samples of the structure, providing the application of additional load according to the required scheme. Limit load up to 10 kN and additional average rate of load growth should be  $50\pm10$  N/s.

Device for testing half specimens of any design, providing sample loading in pure compression mode only. Maximum load limit up to 500 kN. An error of  $\pm 1\%$  is allowed only in the upper ranges.

Finished specimen shall have movable balls to compensate for spatial disturbance of bearing surfaces:

− moisture storage chamber.

− water storage tub.

− scales (error not exceeding 2 g);

− measuring cylinder.

Preparation of the standard sample:

The sample was prepared from cement as well as from polyfractional sand in a W/C ratio of 1:3 by weight. Cement mortar is used to prepare one mix, which is further used in the sample beams. Initially, the sand was poured into the mixer measuring device. The container of the mixer was wiped with a damp rag, poured with water, added cement, after which the mixer was turned on low speed (Figure 1).



Figure 1 – The steps of preparing the cement dough

Before making the samples, the inside of the mold walls should be lubricated with a small layer of machine oil. The joints of the outer walls of the formwork with the plate are oiled.

The nozzle is placed in the already prepared mold, placed on the platform of the shaking table and secured with clamps. The ready CR was removed one at a time directly from the mixer bowl using a spatula, 3 portions of CR each weighing about 300 g. We used a trowel to flatten the surface and a shaking table to compact each layer of mortar, 60 times per cycle.

After compaction was completed, we grinded the surface of the specimens with a metal ruler. Three specimens were prepared for each test.

The mold with the samples was covered with a plate and placed in the humidity chamber.

Twenty-four hours after making, the sample molds were removed from the chamber and very carefully removed. Next, the samples are measured to check the quality of the work done.

Twenty minutes before the test, the specimens were removed and placed in a water bath in a supine position. The water temperature was  $(20\pm 1)$  °C. Every 2 weeks we changed half of the water in the bath with clean water.

# *2.1 Determination of bending strength*

The finished test specimen was mounted on the supports of the instrument so that its horizontal surfaces were in a vertical position and the facets with the numbered symbols were facing us. The specimen was tested according to the instructions for the device.

The load on the sample, that is, the average rate of loading, was  $50 \pm 10$  N/s (Figure 2).



Figure 2 – Bend strength test of the specimen

The halves that passed the bend test were immediately subjected to a compression test. The specimen halves were placed between the plates so that its horizontal surfaces were in a vertical position and the marked surface was facing us.

The ends of the patterns protruded from the clamping plates by about 10 mm. The samples were tested in the same way as described in the instructions for the instrument. The load on the sample, that is, the average loading rate was  $2400 \pm 200$  N/s.

# *2.2 Processing of results*

The bending strength of the sample of a single beam  $R_b$ , MPa, is calculated by the formula:

$$
R_b = \frac{1.5Fl}{b^3},\tag{1}
$$

 $F - load$  on the sample, N;

b – value of the side of the square section of the beam, mm;

l – distance between the axes of supports, mm.

For the bending strength, the arithmetic mean of the test results of three specimens is taken. The calculation result is rounded to 0.1 MPa.

The compression strength of the individual half of the beam specimen Rc, MPa is calculated by the formula:

$$
R_c = \frac{F}{s},\tag{2}
$$

 $F$  – breaking load, N;

 $b$  – working surface area of the pressure plate, mm<sup>2</sup>.

For compressive strength, the arithmetic mean of the test results of the six halves of the beam specimens is calculated. The calculation result is rounded to 0.1 MPa.

If one of the six results differs from the arithmetic mean by more than 10%, this result must be excluded and the arithmetic mean of the remaining five results must be calculated.

If another result differs from the arithmetic mean of the other five results by more than 10%, the tests are considered unsatisfactory, in which case all results are invalid.

# **3. Results and Discussion**

The setting time is the shortest curing period of the concrete mixture and occurs first.

The setting time depends on the composition of the concrete mixture and the ambient temperature. The higher the temperature, the more active the processes.

A sample without additives and a sample with an active mineral additive were prepared to compare the results (Tables 3 and 4).

Table 3 – Determination of cement setting time without additives

Parameter		Test results								
				4						
	60	70	80	90	100	110	l 20	130	160	
	min	min	mın	min	min	min	min	min	min	
Indicator of the device	$0 \text{ mm}$	$2 \text{ mm}$	$5 \text{ mm}$	mm						

Starting time of setting  $-130$  min, end of setting  $-160$  min.

						$1400C + 1200C$ minimation of coment setting three with additive		
Parameter	Test results							
	60	70	80	90	100	110	120	140
	min	min	mnn	min	min	mın	mın	min
Indicator of the device	$0 \text{ mm}$	$0 \text{ mm}$	$0 \text{ mm}$ $0 \text{ mm}$ $0 \text{ mm}$			$3 \text{ mm}$	$5 \text{ mm}$	1 mm

Table  $4$  – Determination of cement setting time with additive

Starting time of setting  $-100$  min, end of setting  $-140$  min.

Thus, in Table 3, you can observe the studied sample without adding the active mineral additive. The beginning of formation of normal density of the sample occurred at the 120<sup>th</sup> minute, and the completion at the 140<sup>th</sup> minute.

If we compare the sample with the additive, we can see that the setting process will start 10 minutes faster and at the same time will finish 10 minutes earlier.

#### *3.1 Determining the uniformity of volume change*

The hardening process of cements is accompanied by a change in volume of the material. Portland cement shows a slight decrease in volume during hardening. If there is free calcium oxide CaO (more than 1%) in the cement clinker, and if there is excess MgO magnesium oxide (more than 5%), the quenching process of CaO and MgO, accompanied by a local increase in volume, represents an irregular volume change of cement during hardening, which leads to deformation and cracking of cement stone. Therefore, cement paste specimens during hardening by boiling in water are tested for the uniformity of cement volume change, which increases the damping of CaO and MgO and accelerates the testing.



## **4. Conclusions**

This study was conducted as part of a project to develop a mixture to improve the strength of cement binder based on waste from a ferroalloy plant. The set tasks have been accomplished:

1. The composition and conditions of the samples were chosen.

2. The effect of dispersion of active mineral additive on cement binder has been proved. High strength characteristics as well as durability at work are provided.

3. Experience in demonstrating high grades of cement binders from M500 to M900 and peculiarities of their application.

4. Considered the possibility of using raw materials of the Republic of Kazakhstan, intended for the production of cement binder.

5. The optimal modes of production mixture and studied its physical and mechanical, construction and technical properties.

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# **Information about authors:**

*Daniyar Bazarbayev* – PhD, Acting Associate Professor, Department of Technology of Industrial and Civil Engineering, L.N. Gumilyov Eurasian National University, Astana, Kazakhstan, [phdd84@mail.ru](mailto:phdd84@mail.ru)

*Adiya Zhumagulova* – Candidate of Technical Sciences, Acting Associate Professor, Department of Technology of Industrial and Civil Engineering, L.N. Gumilyov Eurasian National University, Astana, Kazakhstan, [zaaskarovna@gmail.com](mailto:zaaskarovna@gmail.com)

*Danagul Kadyrkhanova* – MSc, Engineer, GoldConstructionGroup, LLP, Kosshy City, Kazakhstan, [kadyrkhanova\\_danagul@mail.ru](mailto:kadyrkhanova_danagul@mail.ru)

#### **Author Contributions:**

*Daniyar Bazarbayev* – concept, methodology, resources, editing, funding acquisition. *Adiya Zhumagulova* – visualization, interpretation, analysis, drafting. *Danagul Kadyrkhanova* – data collection, modeling, testing.

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# **Research on the effect of microsilica on the properties of the cement-sand mixture**

[R](https://orcid.org/0000-0003-0085-9934)auanLukpanov<sup>1</sup>, **DDuman Dyussembinov<sup>1</sup>, D[A](https://orcid.org/0000-0003-1010-9328)liya Altynbekova<sup>1,2,\*</sup> D[Z](https://orcid.org/0000-0002-2010-3715)hibek Zhantlesova<sup>1,2</sup>** 

<sup>1</sup>Solid Research Group, LLP, Astana, Kazakhstan <sup>2</sup>L.N. Gumilyov Eurasian National University, Astana, Kazakhstan \*Correspondence: [proyekt.2022@bk.ru](mailto:proyekt.2022@bk.ru)

**Abstract.** The article presents a study of changes in the mineralogical composition of cement when adding microsilica of different concentrations using diffractometer DRON-3 with СuCa-radiation, β-filter. X-ray phase analysis on a semiquantitative basis was carried out on powder sample diffractograms using the method of equal weights and artificial mixtures. The work examines the effects of microsilica on the mineralogical composition of cement at the microstructural level. The aim of the study was to improve the physico-chemical properties of the cement. Solutions with different percentages of microsilica additives (0 %, 10 %, 20 %, 30 %) were made for the analysis. The conducted research makes it possible to determine the effect of microsilica in different concentrations on the properties of the cement composition. The introduction of additive into products is introduced to increase mechanical properties, reduce porosity, increase water resistance and durability of products, possibly due to binding of calcium hydroxide in the structure of hardening cement stone by dispersions of microsilica. In general it can be concluded from the results of studies that the additive contributes to increasing the strength of the mixture, accelerating hardening. Consequently, the additive is one of the most effective additives to increase the strength.

**Keywords:** modified additive, microsilica, mineralogical composition, different concentrations, concrete.

#### **1. Introduction**

In the last few years, data have been obtained and published by many researchers showing that the combination of additives with cement provides a synergistic effect in concrete, allowing the best strength results to be obtained. The use of microsilica, a waste product from ferroalloys production, in different types of concrete has aroused great interest among both builders and researchers today. The positive effects of microsilica as a fine-grained active mineral admixture and the need for its use in concrete were described in publications as early as 40 years ago.

Microsilica is a by-product of the metallurgical process of melting ferrosilicon and its alloys, produced by reducing high-purity quartz with carbon in electric furnaces. In the process of melting silicon alloys some part of silicon monoxide SiO goes into gaseous state and undergoes oxidation and condensation to form an extremely fine product in the form of spherical particles with high content of amorphous silica [1].

One of modern trends in production of high-strength concretes is modification of binding structure of construction composites with additives of various composition and morphology. Particularly effective in this regard are ultradisperse additives based on microsilica.

Ultra-dispersible additives generally do not exist in a ready-to-use form, but must be synthesised. Ultradisperse additives generally do not exist in a ready-made form, but must be synthesised and the most important of which are the properties of the stone to be synthesised. application, stability in time, a similar time stability, similar crystallochemical structure with synthesised material, comparability of its particle size with the particles of cement gel and gel

pores, etc. The effectiveness of the additives is assessed by measuring the mechanical, chemical and mechanical properties of the cement paste. The effectiveness of the use of additives is assessed by means of the improvement in the mechanical and physico-chemical properties of the materials being modified properties of the modified materials [2].

The particles of microsilica, whose size is 0.5 ... 0.05 microns, can fill the gaps between the particles of cement and aggregates. The microsilica particles create a tightly bound shell of water around themselves, which prevents water migration to the surface of the concrete and consequently reduces the delamination of the concrete mix and the shrinkage of the concrete. The addition of microsilica increases water resistance by 25-50 % and sulphate resistance by 90-100 %. Adding only 6 % of microsilica provides concrete frost resistance grade F300 at  $B/C = 0.45$  [3].

The use of complex additives is now generally accepted as an effective way to improve the properties of cement concrete. In most cases additives are nowadays the obligatory component of concrete mixture. Analysis of scientific literature shows that additives that increase the rate of setting and hardening of cement are in demand, so the interest in developing new, competitively capable accelerating additives is not diminishing [4].

The creation and introduction into production of innovative materials is directly linked to research into their internal structure, which in turn determines the set of performance properties and their stability. Understanding the microstructure of a material is the key to understanding its properties and characteristics and how they relate to technological processes. Over the past few decades, tremendous progress has been made in methods for determining the microstructure of cement materials, especially in making these methods more quantitative. Quantification is very important because most cements have a roughly similar composition. Unfortunately, it is still not possible today to characterize the microstructure of a cement material with the same accuracy that can be obtained in mechanical characterization testing [5].

The aim of the study is to investigate the change in mineralogical composition of cement with the addition of microsilica in different concentrations.

In order to achieve the objective the following tasks were carried out:

1. The composition with optimum ratio of components was selected;

2. A comparative mineralogical analysis of samples with the addition of components in different percentages and without the addition of component;

3. Selection of the optimum sample with addition of component in percentage ratio;

Microstructural analysis of the mineralogical composition of each sample with the added component (microsilica):

Type-1: Control sample without additives;

Type-2: Sample with added microsilica (10 %)

Type-3: Sample with added microsilica (20 %);

Type-4: Sample with microsilica additive (30 %)

#### **2. Methods**

Four samples with the addition of (microsilica) of different percentages were designed. The cement content varied with the addition of microsilica, depending on the percentage content. Microsilica was added to the mixture in the amounts of 10, 20, and 30 % of the cement mass [6].

The raw materials produced on an industrial scale were mainly used in laboratory tests. Their main characteristics as defined by the standards in force are given below [7].

Portland cement M400 brand CEM I 42,5 H (Karaganda (Central Asian Cement), corresponding to the requirements of GOST 31108-2016 [8].

A waste condensed microsilica MK-85, corresponding to the requirements of GOST 10178 [9], was used as an additive.

Tap water corresponding to the requirements of GOST 23732-2011 [10] was used as mixing water for concrete mixture.



Table 1 presents technological compositions of compared types of cement-sand samples for X-ray diffraction and X-ray phase analysis.

As the samples for XRF and XRF analysis are relatively small in size, the control of the water-cement ratio becomes particularly important. Therefore, all tools and appliances used for mixing the samples were treated with a damp sponge before mixing (Figure 1). The components were weighed on a high-precision analytical scale in order to determine the weight of the samples (Figure 1). Figure 2 shows the finished samples.



Figure 1 – Sample preparation for XRF and XRF analysis



Figure 2 – Finished samples of different consistencies

#### **3. Results and Discussion**

The X-ray diffractometric analysis was carried out on an automated diffractometer DRON-3 with CuCa-radiation, β-filter. The diffractograms were taken with the following instrument settings: U=35 kV; I=20 mA; θ-2θ survey; detector 2 deg/min. X-ray phase analysis on a semi-quantitative basis was carried out on the basis of diffractograms of powder samples using the method of equal weights and artificial mixtures. Quantitative ratios of crystalline phases were determined. We interpreted diffractograms using PDF2 (Powder Diffraction File) Release 2022 from the ICDD Powder Diffractometer Database and clean mineral diffractograms. For the main phases a grade calculation was carried out. Possible impurities which cannot be identified unambiguously due to low grades and the presence of only 1-2 diffraction reflections or poor oxidation are listed in the table.

Table 2 – Results of semi-quantitative XRD analysis of crystalline phases

No	Type-1	$Type-2$	Type- $3$	Type-4
Hatrurite $(Ca_3SiO_5)$	62.1	64.7	56.3	41.90
Calcite $(Ca(CO_3))$	7.1	9.8	10.9	18.10
Portlandite $(Ca(OH)2)$	15.7	25.6	17.6	11.70
Laihunite (Fe1.58 $(SiO4)$ )				10.30
Ilvaite $(CaFe3Si2O8(OH))$	9.3		10.0	10.30
Quartz $(SiO2)$	5.8		5.10	7.80



Figure 3 – Graph of crystalline phases

Table 2 presents the chemical composition of the studied samples, characterizing which it is necessary to note insignificant variations in the content of all oxides.

Comparing the results of semi-quantitative X-ray diffraction analysis of crystalline phases with addition of microsilica we see the following results: the content of Hatrurite  $(Ca_3SiO_5)-(62.1 -$ 41.90) decreases significantly, the content of Calcite (Ca(CO3)-(7.1-18.10) increases, the content of Portlandite  $(Ca(OH)_2)$  – in all four cases has different value. Ilvaite  $(CaFe<sub>3</sub>Si<sub>2</sub>O<sub>8</sub>(OH))$  and Quartz  $(SiO<sub>2</sub>)$  – are characterized by close values (9.3-10.30) and (5.8-7.80).



Figure 4 – Diffractogram of sample No. 1



Figure 5 – Diffractogram of sample No. 2



Figure 6 – Diffractogram of sample No. 3



Figure 7 – Diffractogram of sample No. 4

#### **4. Conclusions**

The following conclusions can be drawn on the basis of the research carried out:

1. The mixture of microsilica additive with cement has been studied and the chemical and phase composition of the raw materials has been determined.

X-ray phase analysis revealed the presence of silicon oxide in the form of coesite or coesite in microsilica. This gives it a high chemical activity in aqueous medium. It is a highly baric modification of silica, chemical formula:  $SiO_2$ . Average density is 2.95...3 g/cm<sup>3</sup>, hardness 7.5...8 on Mohs scale. On lowering of pressure it transforms into quartz. Therefore, the presence of coesite in microsilica is unlikely.

2. The analysis of control samples showed higher amounts of calcium carbonate. This is probably due to the carbonation of free calcium hydroxide produced during cement hydration.

Increasing the content of microsilica to 30 % of the cement weight was noted to increase the total volume of calcium hydrosilicates  $(Ca(CO<sub>3</sub>)).$ 

3. Microsilica is introduced into products to increase mechanical properties, decrease porosity, increase water resistance and durability of products, possibly due to binding of calcium hydroxide in the structure of hardening cement stone by dispersions of microsilica. Microsilica in this case forms a structure with calcium hydrosilicates.

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## **Information about authors:**

*Rauan Lukpanov* – PhD, Professor, Scientific Supervisor, Solid Research Group, LLP, Astana, Kazakhstan, [rauan\\_82@mail.ru](mailto:rauan_82@mail.ru)

*Duman Dyussembinov* – Candidate of Technical Sciences, Associate Professor, Senior Researcher, Solid Research Group, LLP, Astana, Kazakhstan, [dusembinov@mail.ru](mailto:dusembinov@mail.ru) 

*Aliya Altynbekova* – Researcher, Solid Research Group, LLP, Astana, Kazakhstan; PhD Student, Department of Technology of Industrial and Civil Construction, L.N. Gumilyov Eurasian National University, Astana, Kazakhstan, [kleo-14@mail.ru](mailto:kleo-14@mail.ru)

*Zhibek Zhantlesova* – Junior Researcher, Solid Research Group, LLP, Astana, Kazakhstan; PhD Student, Department of Technology of Industrial and Civil Construction, L.N. Gumilyov Eurasian National University, Astana, Kazakhstan, [zhibek81@mail.ru](mailto:zhibek81@mail.ru)

# **Author Contributions:**

*Rauan Lukpanov* – concept, methodology, funding acquisition. *Duman Dyussembinov* – resources, interpretation, analysis. *Aliya Altynbekova* – data collection, modeling, testing. *Zhibek Zhantlesova* – visualization, drafting, editing.

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## *Article* **Determination of the softening temperature of bitumen with different modifiers**

ZhanarKusbergenova<sup>1,\*</sup>, <sup>ID</sup>Gulmira Baizakova<sup>2</sup>, IDAleksej Aniskin<sup>3</sup>

<sup>1</sup>Department of Civil Engineering, L.N. Gumilyov Eurasian National University, Astana, Kazakhstan <sup>2</sup>School of Architecture and Construction, Serikbayev East Kazakhstan Technical University, Ust-Kamenogorsk, Kazakhstan

> <sup>3</sup>Department of Civil Engineering, University North, Varaždin, Croatia \*Correspondence: [zh.kusbergenova@sapaortalygy.kz](mailto:zh.kusbergenova@sapaortalygy.kz)

**Abstract.** Road construction is an important part of economic development, and the most pressing problem is improving the quality of road surfaces. Road pavements are a combination of mineral fillers and bitumen, in which bitumen is used as a durable waterproof binder. Bitumen modification can improve the quality of bitumen, by increasing the plasticity interval, increasing adhesion to metal and mineral materials, increasing resistance to aging, and expanding the operating temperature range. Softening point characterizes the thermal resistance of bitumen: the transition from the elastic-plastic state to a viscous-fluid state, in which the bitumen loses its adhesive properties. And this paper presented the investigation of the properties of bitumen by modifying it and determining the softening temperature on the device Ring and ball. The analysis of the obtained results of the use of various modifiers, their comparative characteristics, and the formulation of conclusions about the possibility of involving them in the production of bitumen to improve their quality. **Keywords:** bitumen, modifier, property, softening temperature, investigation.

#### **1. Introduction**

#### *1.1 Bitumen as a construction material*

Currently, there is an increase in requirements for the operational and transportation characteristics of asphalt concrete pavements due to the annual increase in the number of vehicles in almost all regions of Kazakhstan, including heavy and extra heavy trucks, which reduces the service life of road asphalt concrete pavements [1-3]. Along with this, another cause of premature failure of road asphalt concrete pavement in the form of cracks, potholes, holes, ruts, etc. is a sharp drop in ambient temperature within a short period of time and severe weather conditions. At the same time, the service life of the pavement is affected by the quality of the bitumen used, which is one of the important components of the road surface in almost all categories of roads around the world [4-5].

Unlike natural bitumen, artificial bitumen is formed because of wastes of processed oil by releasing the following gases: butane, propane, ethylene. Bitumen has the following properties: density, flash point, viscosity, solubility, brittleness, plasticity, softening, adhesion, which affect the quality of bitumen [6].

However, to date, several the following problems are associated with bitumen in the road sector. One of them is the annual increase in the price of bitumen during the season of road construction and repair. Another problem is the low quality of proposed bitumen. Considering the above mentioned, one of the solutions of these problems is the modification of bitumen. First, it will solve the problem with bitumen storage, and secondly, it will reduce the amount of bitumen used in the construction and repair of roads of all categories, which will eventually lead to a reduction in demand for bitumen.

# *1.2 Application of various bitumen modifiers*

The research team [7] presented the results of tests conducted on bitumen modification of "polyethylene terephthalate (PET) beverage bottles and used rubber tires, which are major municipal solid waste that can lead to various environmental problems if they are not properly recycled.

The main purpose of this work was to study the mechanisms of PET waste recycling with triethylenetetramine (TETA) and ethanolamine (EA), describing the properties of the above additives with bitumen modified with old rubber products. Based on the results of the tests, "two additives from PET waste, PET-TETA, PET-EA, and their molecular structures, thermal properties and synthesis mechanisms have been characterized. At the same time, it was found that PET wastes are amenable to chemical recycling in the form of functional additives that allow increasing the operation of bitumen using rubber as a modifier.

A study by D.S. Mabui [8] studied polyethylene terephthalate (PET) and budded modified asphalt (BMA), and to solve the problem of heavy precipitation and temporary flooding considered the use of porous asphalt concrete in the surface layer of the roadway. The advantage of porous asphalt is that voids are created in the mixture of coarse aggregate, sand and filler, which absorb excess storm water in bad weather and do not allow moisture to collect on the road surface.

Another interesting study material was budstone asphalt, which contains 30% bitumen and 70% minerals. At the same time in the mixture for the study modified budstone asphalt was used in the amount of 6.0% of the total weight of the aggregate. The work of L. Desideri et al. [9] studied the issue of combining bitumen with polyethylene and Fischer-Tropsch waxes. During the study bitumen PG64-10, polyethylene in granules and powder and two commercial Fischer-Tropsch waxes were used. The tests confirmed the fact that the mechanical response of bitumen is very dependent on the chain length of the wax.

Thus, the reaction of bitumen with long-chain wax is similar to the reaction of polymers not organically modified with binders in the entire range of temperatures considered, while the shortchain bitumen changes sharply at temperatures above 50^0C in the form of melting and softening. At the same time, in view of the volatilizable of the components of the considered compositions and the resulting oxidation reaction in these mixtures because of high-temperature treatment, their mechanical properties change significantly. The patent "Asphalt-concrete mixture on nanomodified binder" [10] describes the composition of the asphalt-concrete mixture with the addition of rubber crumb of 0.25 mm in size as a bitumen modifier, which can improve the properties of asphalt-concrete on its basis.

However, each of the presented methods has its own characteristics and disadvantages, in this regard, the search for a solution of high-quality bitumen is open.

## **2. Methods**

The purpose of this study was to determine the softening temperature of bitumen modified with various additives and to determine their influence on this indicator.

The tests were conducted according to the requirements of EN 1427:2015 [11].

As modifiers were used:

1) pellet modifiers;

2) crushed plastic weighing  $1 g, 2 g, 4 g, 6 g;$ 

3) polyethylene weighing 50 mg, 100 mg, 150 mg.

In each recipe used road bitumen brand 70/100 examined on the device to establish the softening temperature of bitumen in the "pure" form, and then with the use of additives. Prior to the study, polyethylene was separated into small pieces, a plastic disposable cup with a lid was used as plastic, which was previously crushed into smaller pieces to dissolve them better in the material used.

The Figure1 shows the preparation of the composition of bitumen using additives, presented data on the amount of material used in the tests conducted.



Since the introduction of pellets already has a strict proportion, this modifier was chosen in one recipe. And the quantitative composition of the modifiers as plastic and polyethylene were tested in different ratios. Figure 2 shows samples of bitumen heated to a viscous state on the slab and mixed with additives.

Figure  $1 -$ Used additives





a) Molds before mixing with bitumen b) Molds after mixing with bitumen Figure 2 – Mixing bitumen with plastic and polyethylene samples

The molds were pre-lubricated with an ointment in the form of a mixture of glycerin and talc in a ratio of 1:2 to effectively remove the residual bitumen after the test. Figure 3 shows images of the ring molds before the bitumen was poured with the additives used and after.



a) Before filling b) After filling Figure 3 – Shapes for the "Ring and Ball" device

Properties of polymer bitumen depends on the peculiarities of the properties of the original bitumen, the compatibility of the injected polymer with bitumen, compliance with the technology of polymer bitumen preparation, considering the characteristics of the properties of polymer plasticizers [12].

The balls placed in the center of special round forms are pressed through the bitumen due to the increase in water temperature in the device (Figure 4) as well as due to the gradual softening of bitumen [8].





a) Start of the test b) Completion of the test Figure 4 – Conducting a test on the "Ring and Ball" device

# **3. Results and Discussion**

When introducing different ratios of modifiers to bitumen from the mass of the bitumen, the results of the study of the kinetics of change in the softening temperature are shown in Figure 5. Thus, the main results obtained are presented in Table 1, with the modifier of plastic and podiatylene selected as the best composition.



a) Plastic a) Polyethylene Figure 5 – Softening temperature depending on the amount of modifier

	Table 1 – Softening temperature regarding modification composition
Composition	Softening temperature, °C
Bitumen 70/100	52
Bitumen with granule	
modification	52.03
Bitumen with	
polyethylene	58.87
Bitumen with plastic	51.0

Table 1 – Softening temperature regarding modification composition

Analysis of the results compared with the properties of the original bitumen (Table 1) shows that the introduction of 50 mgr polyethylene into bitumen creates a spatial structure, resulting in an increase in the softening temperature (Figure 6).



Figure 6 – Comparison analysis

The effect of adding polyethylene occurs at its lower ratio to bitumen, which is the optimal modification between considered modifiers.

## **4. Conclusions**

Due to modern technologies, it is possible to improve the composition of asphalt concrete mixture in the following ways: by improving the production technology of bitumen and asphalt concrete mixture; by using modifying additives both in bitumen itself and in asphalt concrete mixture. As the analysis showed, the method of bitumen improvement by polymeric additives is more promising. Using it, it is possible to improve the quality of bitumen performance properties or to obtain a topical material with improved physical, mechanical and chemical properties.

The choice of materials presented in the study is since they are readily available, easy to use, and their volume is growing every year. The use of waste materials in the form of polyethylene and plastic is also an effective direction in obtaining polymer bitumen binders for asphalt concrete, as their combination with bitumen does not cause difficulties.

Based on the conducted study conclusions were obtained: the properties of polymer-bitumen binder depend on the properties of the initial bitumen, polymer properties and its compatibility with bitumen, the quality characteristics of the plasticizer; the required amount of polymer to obtain the polymer-bitumen binder to ensure reliable operation of waterproofing will depend on the structure of the initial bitumen.

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## **Information about authors:**

*Zhanar Kusbergenova* – Master Student, Department of Civil Engineering, L.N. Gumilyov Eurasian National University, Astana, Kazakhstan, [zh.kusbergenova@sapaortalygy.kz](mailto:zh.kusbergenova@sapaortalygy.kz)

*Gulmira Baizakova* – Senior Lecturer, School of Architecture and Construction, Serikbayev East Kazakhstan Technical University, Ust-Kamenogorsk, Kazakhstan, [gbaizakova@mail.ru](mailto:gbaizakova@mail.ru)

*Aleksej Aniskin* – Candidate of Technical Sciences, Assistant Professor, Department of Civil Engineering, University North, Varaždin, Croatia, [aaniskin@unin.hr](mailto:aaniskin@unin.hr)

## **Author Contributions:**

*Zhanar Kusbergenova* – concept, methodology, resources, data collection, funding acquisition. *Gulmira Baizakova* – visualization, interpretation, modeling, drafting, testing. *Aleksej Aniskin* – analysis, editing.

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*Article*

# **The impact of the temperature and humidity state of the road on heat and mass transfer in winter**

Assel[S](https://orcid.org/0000-0003-2249-7292)arsembayeva<sup>1,\*</sup>, **D**Philip Collins<sup>2</sup>, D[Z](https://orcid.org/0009-0009-9096-9766)amir Saginov<sup>3</sup>, DSaltanat Mussakhanova<sup>1</sup>

<sup>1</sup>Department of Civil Engineering, L.N. Gumilyov Eurasian National University, Astana, Kazakhstan <sup>2</sup>College of Engineering, Design & Physical Sciences, Brunel University London, London UB8 3PN, UK <sup>3</sup>RSE on REM "National Center for the Quality of Road Assets", Ministry of Industry and Infrastructure Development of the Republic of Kazakhstan, Astana, Kazakhstan

\*Correspondence: [assel\\_enu@mail.ru](mailto:assel_enu@mail.ru)

**Abstract.** Sharp diurnal temperature fluctuations in Astana, Kazakhstan, in winter, as well as freezing up to 2 or more meters leading to the destruction of the roadway, especially during the spring thaw, prompted a detailed study of the state of roads. In this work, the temperature and humidity of the highway structure layers were monitored in winter, and the mass transfer of water in the gaseous state due to the negative pressure of cryosuction in the frozen layers was also considered. It was determined that mass transfer of water in the form of steam 1.44  $10^{-4}$  g/h per 1 dm<sup>3</sup> of soil at temperature fluctuations of -5-8 °C. The rate of vapor passage towards the freezing front in the soil was 0.467 m/h. The freezing of the ground base continued for 132 days in the winter period of 2021-2022 in Astana with the formation of 456.72 g of ice due to the migration of water in a gaseous state in every 1  $\text{m}^3$  of soil, which increases the humidity by 40 % or more and significantly reduces the bearing road capacity during the spring thaw. As a solution to the problem of water migration in the form of steam, it is proposed to introduce an additional layer of vapour barrier over the soil base at a depth of -60 cm.

**Keywords:** highway structure, temperature and humidity monitoring, heat transfer, moisture transfer, soil freezing.

#### **1. Introduction**

The temperature and humidity state of the foundation of roads has a huge impact on their strength and deformation characteristics [1, 2]. This is especially true for regions with a continental climate where there are a large number of transitions through zero degrees [3]. At low temperatures, part of the water contained in the road subgrade soil passes from a liquid state to a solid state (ice). Freezing temporarily improves the deformation and strength characteristics of the subgrade soil [4, 5]. Sharp temperature fluctuations during the day in winter in Astana, Kazakhstan, as well as freezing up to 2 or more meters leading to the destruction of the roadway, especially during spring thaw, prompted a detailed study of the state of roads [6].

According to the results of previous studies, the cooling of pavement layers is more intense compared to unpaved roadside soils [7]. Highway construction materials with higher thermal conductivity are a zone of more intense cooling and thus, due to cryosuction forces, attract unfrozen water and, together with it, deicing chemicals used for surface treatment of highways and accumulated during cleaning in lateral reserves [8].

One of the unresolved problems is mass transfer from water in the gaseous state, which, unlike water in the liquid state, does not have surface tension forces, easily passes through the smallest pores, condenses, and sublimes when the vapour is saturated, and forms ice lenses in these places [9]. In order to study the state of roads and assess their bearing capacity under the influence of negative temperatures, a section of the road was studied in the conditions of the city of Astana.

To do this, the temperature of the pavement and subgrade was monitored during the period of temperatures below 0 degrees in winter 2021 - 2022, and soil samples were taken for a more detailed laboratory testing. To study frost heaving as well as the transfer of water from warm to cold layers of soil at the base of the highway, a method presented by Sarsembayeva et al. was used to determine the mass-heat transfer [9].

#### **2. Method**

#### *1.1 Experimental study of foundation soils*

The investigated section of the road is located near the airport of Astana, in the area of the Karkaraly highway, 8 km from the bypass road towards the town of Kosshy. All soils exposed at the site, according to the results of surveys, are water-bearing deposits. The groundwater regime is subject to seasonal fluctuations: the minimum standing is observed in February, the maximum falls on the end of May. The amplitude of groundwater level fluctuations is 1.0-2.0 m. Groundwater is fed mainly due to infiltration of precipitation. The feeding area is the distribution area of the aquifer.

In January 2022, soil samples were taken in the immediate vicinity of the highway in order to study their condition in winter. The density of the loam base soil was  $2.0 \text{ g/cm}^3$ , the moisture content in natural condition was 20.2%. By laboratory research methods, the soil filtration coefficient for Quaternary loams was determined - 0.01-0.13 m/day. Groundwater in this section of the road is characterized as sodium chloride, very hard, slightly alkaline, brackish. In relation to concrete grade W4 on Portland cement, groundwater is non-aggressive and slightly aggressive, in relation to reinforced concrete structures it is moderately aggressive. The corrosive aggressiveness of groundwater in relation to the lead and aluminum sheaths of the cable is high. Groundwater is corrosive in relation to steel structures (according to Shtabler). The physical and mechanical characteristics of the foundation soils are given in Table 1.



Table 1 – The physical and mechanical characteristics of foundation soils

#### *1.2 Monitoring the temperature of the roadway in winter*

The construction of the road is a non-rigid pavement made of an underlying layer of gravelsand mixture 15 cm thick on the compacted base soil (Figure 1). On top is the bottom layer of the base of the crushed stone-sand mixture with a thickness of 15 cm, then the top layer of the base of the hot highly porous asphalt mixture with a thickness of 12 cm. Above is a 10 cm hot mix asphalt pavement layer. And a 5 cm hot mix asphalt pavement surface layer.



The temperature was recorded by automatic measurement by sensors embedded in metal capsules installed at a depth of 0.05; 0.2; 0.4; 0.6; 0.8; 1.0; 1.4; 1.8; 2.2; 2.6 and 3.0 m from the asphalt concrete surface. The air temperature was also measured, taken here as the temperature of

#### *1.3 Calculation of the moisture transfer from the base layers upwards the freezing front*

the earth's surface 0 cm. Sampling frequency was 1 time per hour.

Figure 2 schematically shows the direction of water vapour migration to layers with higher thermal conductivity and, accordingly, lower temperatures in winter. Water mass transfer in a gaseous state from lower and warmer layers of the highway base towards the upper colder layers was calculated according to the method described in detail by [9].



Figure 2 – Moisture migration in the highway base

The vapour speed is calculated as a heat transfer over cumulative void channel dimensions, vapour flow density corresponding to the temperature and time:

$$
v_{vapour} = \frac{Q}{C \cdot \rho_{vapour} \cdot A_{air} \cdot \Delta T \cdot t}
$$
 (1)

where,  $v_{vapour}$  – average speed of vapour, cm/h;  $Q$  – heat transfer in a gaseous state,  $\Delta T$  – measured temperature difference,  ${}^{\circ}C$ ; *C* – specific heat of vapour passing through the cumulative air voids cross section, J/kg·°C;  $\rho_{vapour}$  – vapour density corresponding to the temperature, g/cm<sup>3</sup>;

 $A_{air \, voids}$  - cumulative cross section of the air voids'  $A_{air \, voids} = \frac{\pi \cdot d_a^2}{4}$  $\frac{u_a}{4}$ , cm<sup>2</sup>, corresponding to the porosity coefficient and moisture content;  $t -$  time, h.

The mass of ice built from the vapour passing over time period *t* with speed *v* over the cumulative air channel cross section  $A_{air}$  is calculated:

 $m_{ice} = \rho_{vapour} \cdot V_{air\,roids} = \rho_{vapour} \cdot v \cdot t \cdot A_{air}$  (2)

where,  $m_{ice}$  - mass of built ice in grams;  $\rho_{vapour}$  - is taken as an average density value of the vapour densities at the start and end time point,  $g/cm<sup>3</sup>$ .

#### **3. Results and Discussion**

The results of temperature monitoring (Figure 3) showed large amplitude jumps during the day in the upper layers of asphalt concrete. During November, eight 0°C crossings were recorded. In contrast to the asphalt concrete layers, which have sharp temperature fluctuations during the day, the temperature of the base soil slowly and steadily dropped over the course of a month. Soil freezing began on November 19, and then there were no strong jumps in the direction of thawing.





b) Temperature distribution for December 2021

*Technobius*, 2022, 2(4), 0029



c) Temperature distribution for January 2022



Figure 3 – Temperature distribution over time

In December, 8 transitions through  $0^{\circ}$ C were recorded, but the underlying layers of the gravel sand mixture remained frozen, apparently by compensating for the heat transfer of the underlying cooler layers. The relative stabilization of the foundation soils reached at a depth of -1.2 m at negative temperatures in the range of  $-5$   $-2$ °C. The freezing depth continues to steadily increase from 1.60 m to 2.0 m during the month. At the end of January, the freezing depth reaches 2.20 m and at the end of February 2.50 m.

Table 2 – Brief information on the surface temperature of highway surface in Astana during the winter months

WHILE HIUIHIIS								
Month	November	December	Januarv	February				
Transitions over $0^{\circ}C$								
Average temperature, $^{\circ}C$	$-0.74$	$-6.04$		-6.46				
Minimum temperature, $\mathrm{C}$		$-24.3$	$-20.9$	$-21.8$				

Figure 4 shows the change in the moisture content of the pore space in the soil base during the winter months. The difference from 21 to 11 % between the layers of 60 cm and 100 cm in November (Figure 3a) indicates a freezing front between these layers and an urgent solidification of moisture in the frozen part.

A decrease in saturated vapor pressure in the pore space in the frozen part causes the migration of moisture in the gaseous state towards the freezing front. The lack of pore pressure (negative pressure) is compensated by vapour migration from the warm part from below. In December, from 8 to 12, a freezing front occurs at a depth of 100 cm and is accompanied by a drop in humidity in the pore space from 19 to 15%. And in January, freezing occurs at a depth of 140 cm and 180 cm, as evidenced by a drop in humidity from 21 and 23%, respectively, to 17% in a frozen state. During February, there is a decrease in humidity at a depth of 2.20 m, which means freezing of this layer.



a) November 2021 humidity measurements



b) December 2021 humidity measurements



a) January 2022 humidity measurements



d) February 2022 humidity measurements Figure 4 – Moisture distribution over time

When calculating the amount of moisture migrating in 1 hour in 1  $\text{dm}^3$  of soil according to the method described by [9], we found that the water flow rate depended on the packing density of solid soil particles and the temperature distribution over the soil layers. The water mass transfer in a form of vapour  $1.44 \cdot 10^{-4}$  g/h per 1 dm<sup>3</sup> of soil at with temperature fluctuations -5-8 °C. The rate of passage of vapour towards the freezing front in the soil was 0.467 m/h.

Thus, 0.144 g of ice is formed per hour in 1 cubic meter at a temperature of -5-8 °C at a depth of -60-160 cm from the road surface, while in a day this amount is 3.46 g of ice formed by transferring moisture from warmer soil layers. Now, given that the freezing of the soil base lasted 132 days in 2021-2022 winter period in Astana city, we can assume that during this time, only due to the migration of water in the gaseous state under the influence of cryosuction negative pressure in 1 cubic meter of the road base, 456.72 g of ice formed, which will increase the soil moisture to 40 % and more, which will significantly reduce the bearing capacity of the road during spring melt time.

Thus, the effect of water transport in frozen ground is greatly underestimated and it is necessary to install an additional layer of vapor barrier in order to prevent the accumulation of ice lenses under the pavement structure.

# **4. Conclusions**

Based on the results of the study, the following outcomes were highlighted:

1. In the upper layers of asphalt concrete, from 8 to 14 transitions through 0 C per month were registered, while the temperature of the crushed sand-stone mixture at -37 cm depth remained in a stable frozen condition.

2. The freezing of the layers is accompanied by a sharp drop in the humidity of the pore space from 21-23 to 11-14 %, when part of the water in the gaseous state sublimates and forms a negative cryosuction pressure in these layers.

3. Base soil located at a depth of -60 cm consisting of light sandy sandy clayey soil is a frost susceptible and is in a stably frozen state at a temperature of -5-8 °C.

4. The water mass transfer in a form of vapour  $1.44 \cdot 10^{-4}$  g/h per 1 dm<sup>3</sup> of soil at with temperature fluctuations -5-8 °C. The rate of passage of vapour towards the freezing front in the soil was 0.467 m/h.

5. The freezing of the ground base continued for 132 days in the winter period of 2021-2022 in Astana with the formation of 456.72 g of ice due to the migration of water in a gaseous state in every 1  $m<sup>3</sup>$  of soil, which increases the humidity by 40 % or more and significantly reduces the bearing road capacity during the spring thaw.

6. As a solution to the problem of water migration in the form of vapour, it is proposed to lay an additional layer of vapor barrier over the soil base at a depth of -60 cm.

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# **Information about authors:**

*Assel Sarsembayeva* – Candidate of Technical Sciences, PhD (UK), Associate Professor, Department of Civil Engineering, L.N. Gumilyov Eurasian National University, Astana, Kazakhstan, [assel\\_enu@mail.ru](mailto:assel_enu@mail.ru)

*Philip Collins* – PhD, Vice Dean (Education), College of Engineering, Design & Physical Sciences, Brunel University London, London UB8 3PN, UK, [philip.collins@brunel.ac.uk](mailto:philip.collins@brunel.ac.uk)

*Zamir Saginov* – General Director, RSE on REM "National Center for the Quality of Road Assets" of the Ministry of Industry and Infrastructure Development of the Republic of Kazakhstan, Astana, Kazakhstan, [info@sapaortalygy.kz](mailto:info@sapaortalygy.kz)

*Saltanat Mussakhanova* – PhD Student, Department of Civil Engineering, L.N. Gumilyov Eurasian National University, Astana, Kazakhstan, [musaxanova.saltanat@mail.ru](mailto:musaxanova.saltanat@mail.ru)

# **Author Contributions:**

*Assel Sarsembayeva* – concept, methodology, funding acquisition. *Philip Collins – resources, interpretation, analysis. Zamir Saginov* – data collection, modeling, testing. *Saltanat Mussakhanova* – visualization, drafting, editing.

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## *Article* **Calculation of the stress-strain state of highways using the finite element method**

Zhanbolat Shakhmov<sup>1,\*</sup>, <sup>D</sup>Jakharkhan Kabdrashit<sup>2</sup>, DAru Kozhahmet<sup>3</sup>

<sup>1</sup>RSE on REM "National Center of Road Assets Quality", Astana, Kazakhstan

<sup>2</sup>Department of Algebra and Geometry, Faculty of Mechanics and Mathematics, L.N. Gumilyov Eurasian National University, Astana, Kazakhstan

<sup>3</sup>Department of Technology of Industrial and Civil Engineering, L.N. Gumilyov Eurasian National University, Astana,

Kazakhstan

\*Correspondence: E-mail: [zhanbolat8624@mail.ru](mailto:zhanbolat8624@mail.ru)

**Abstract.** The main purpose of the examination of roads is the timely identification of areas that require improvement of road conditions, as well as the evaluation of the state of all structural elements of roads. The study of the causes of road deformations, measures to eliminate and prevent warping are the main issue of road maintenance. The survey of roads was conducted in accordance with the approved methods using standard measuring tools. PLAXIS software was used to perform the finite element analysis of deformations and stability of soil, to calculate the interaction of the pavement with the ground, the effect of heat fluxes. Processing of data on the road Almaty-Taraz has shown that stresses and deformations arise directly in the place of contact of point loads with trapezoidal shape on the depth of influence. The authors conclude that when horizontal deformations occur, the greatest stresses occur on the road shoulders, which surrounds possible slope collapses. In addition, volumetric deformations of the foundation occur under direct dynamic loading and, under its prolonged effect, contribute to the appearance of volumetric bulging deformations. **Keywords:** cement concrete pavements, road defects, road survey, numerical modeling, PLAXIS.

#### **1. Introduction**

Concrete roads are not an invention of our day [1]. Concrete pavements on motorways in Europe in the 1930s were predominantly laid in two layers, with 20 to 25 cm thick freshly paved and paper lined [2]. Transverse joints, mostly as expansion joints, separated the concrete pavement at distances of 10 to 37.5 meters from each other [3]. A maximum of two transversal dummy joints were allowed between the expansion joints. The false joint was cut in the hardened concrete from 1938 onwards. Expansion dowel joints were used as longitudinal joints [4-5]. Concrete pavements were predominantly reinforced (minimum  $2.5 \text{ kg/m}^2$ ) and the cement content of the top concrete was approximately the same as today [6]. Dowels and anchors started to be used from around 1936 onwards [7].

Today, road construction has also aroused interest, and progressive technologies and maintenance processes for cement-bonded concrete roads are constantly being improved.

Road surfaces are heavily stressed due to dynamic influences and climatic conditions. In order to achieve high quality cement concrete roads, German road builders have defined the requirements for its paving, which include cement, aggregates, concrete strength, volume of air involved, slip resistance, surface treatment and concrete maintenance [8-9].

The total length of cement concrete paved roads in Kazakhstan is 1,628.48 km [10]. It is advisable to carry out comprehensive road inspections, serving also to accumulate a data bank on the condition of all road elements, at least once every 5 years. Road inspections are an integral part of all work aimed at ensuring high transport and operational performance of roads. Their nature is similar

to that of road surveys, involving the choice of dimensions for road elements in the light of traffic flow. The results of the surveys are used as input for traffic management projects, strengthening of the pavement, reconstruction of road sections, etc. [11].

One of the deformations of roads is subsidence. The comfort and safety of travel suffers greatly from uneven subsidence. Calculations and surveys are not always up to date, so one method is numerical modelling of the pavement. Since subsidence is caused by a compressible subgrade, it is advisable to use the Plaxis calculation program for the calculation.

#### **2. Methods**

PLAXIS is a finite element analysis software system used to solve geotechnical engineering and design problems. PLAXIS is a package of computational programs for finite element calculations of the stress-strain state of structures, foundations and foundations.

The programme is suitable for those who carry out calculations of structures in industrial, civil, hydraulic, transport, underground and other types of construction, as well as for surveyors:

- Surveyors - for assessing the stability of natural slopes and preliminary analysis of landslide control structures, as well as for assessing the effects of changing groundwater levels and calculating stresses from their own weight.

- Soil laboratories to be able to calibrate and issue complete sets of soil model parameters in accordance with current regulatory requirements for geotechnical calculations.

- Design organizations to provide design justification for design solutions, which is the basis for the design process. To obtain the parameters of structures and constructions in order to make a technical and economic comparison in case of variant design.

- To construction companies in order to check the correctness of design solutions and the possibility of effective evaluation of complicated geotechnical situations at a construction site and taking decisions.

- for checking the feasibility and reliability of design decisions.

This program was used to create a model of the road section for finite element calculations of the stress-strain state of the base and road pavement.

The calculation was carried out in PLAXIS numerical simulation software, stress and strain diagrams were obtained for the road pavement and subgrade; the calculation was carried out for the Taraz Almaty section 744-806 km, since source documents (geological surveys) were available for this section.

The roadway consists of the following layers: cement concrete (27 cm thick), polyethylene film (1 mm thick), crushed stone mix with 7% cement treatment (20 cm thick), gravel-sandy mix underlay (35 cm thick). The subgrade soil is silty-clay.

The container of the mixer was wiped with a damp rag, poured with water, added cement, after which the mixer was turned on low speed.

#### **3. Results and Discussion**

As a result of the calculations in the Plaxis software package, the stress and strain isofields were obtained as shown below. The physical and mechanical properties of each pavement were determined from reference materials and laboratory results according to the project documentation.

The stress mosaic (Figure 1) shows the results of stresses in the ground from a load of 160 kN with the obtained outputs where the maximum stress occurs in the silty-clay soil layers of the road and the pavement layers. The maximum stresses reach up to  $26 \text{ kN/m}^2$ .



Figure 1 – Effective average tangential stress

Relative tangential stress (Figure 2) occurs directly under the road, and is distributed over the gravel soils which, because of their greater strength, carry the main load from the road. It can be seen that the stresses are not created and distributed uniformly across the layers of the subsoil.



Figure 2 – Relative Tangential Stress

The Figure 3 shows that large stresses run completely through the entire pavement. The figure shows that large stresses run completely through the entire pavement. Each pavement itself distributes the load and consequently the materials withstand the load. But the important question is still that of the subgrade, which has to have a sufficient deformation modulus.



The vertical deformations (Figure 4) showed a negligible result, not more than 2 cm in total. According to the normative and technical documents it should not be more than 5 cm.



Figure 4 – Total Deformation

Horizontal deformations (Figure 5) showed a negligible result, not more than 0.5 cm in total. This result is not significant.



According to the results (Figure 6,7 and 8) obtained, it can be observed that at the top of the road slope and in the gravel part of the subgrade, there are 8 m depths.



Figure 6 – Cumulative strain increments



Volumetric and Tangential deformations (Figure 9 and 10) occur in the road slope areas and in the middle part in the road's median area. Maximum number is 1,4 %.



Figure 10 – Tangential Deformation

Stresses and strains occur directly at the point of contact of point loads with a trapezoidal shape along the depth of influence. The horizontal deformation isofields characterize the shear

#### **4. Conclusions**

According to the calculations performed, strain and stress mosaics were obtained.

- Tangential stress mosaics show that stresses propagate linearly along road pavement with maximum 26  $kN/m^2$  and loam subgrade, followed by non-linear stress concentration in hard gravel subgrade;

- Relative tangential stress mosaics show that the stress isofields spreads with a widening of the influence zone in the form of a trapezoid and spreading over the gravel bed;

- The mosaics of general deformation diagrams show that subsidence occurs linearly in pavement and subgrade layers, but soil bulging occurs in road slopes due to loading of the roadbed and settlement is no more 2 cm;

- According to the mosaics of volumetric deformation diagrams, volumetric deformations are found to occur directly under the roadbed with 1.4 % and volumetric bulging deformations occur in the asphalt surface between the roadbeds.

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#### **Information about authors:**

*Zhanbolat Shakhmov –* PhD, Associate Professor, RSE on REU «National Center for the Quality of Road Assets», Astana, Kazakhstan, [zhanbolat8624@mail.ru](mailto:zhanbolat8624@mail.ru)

*Jakharkhan Kabdrashit –* Master Student, Department of Algebra and Geometry, Faculty of Mechanics and Mathematics, L.N. Gumilyov Eurasian National University, Astana, Kazakhstan, [djakharkhan@gmail.com](mailto:djakharkhan@gmail.com)

*Aru Kozhakhmet –* Master Student, Department of Technology of Industrial and Civil Engineering, Faculty of Architecture and Civil Engineering, L.N. Gumilyov Eurasian National University, Astana, Kazakhstan, [arukozhahmet@mail.ru](mailto:arukozhahmet@mail.ru)

# **Author Contributions:**

*Zhanbolat Shakhmov* – concept, methodology, resources, funding acquisition. *Jakharkhan Kabdrashit* – interpretation, analysis, drafting, editing. *Aru Kozhakhmet* – data collection, modeling, testing, visualization.

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