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RESEARCH ARTICLE

INVESTIGATION OF THE PROPERTIES OF A WATERBORNE THERMAL INSULATION COMPOSITE BASED ON SHELL LIMESTONE-SHELL ROCK AND HOLLOW MICROSPHERES

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Abstract. *The aim of this study is to develop a technology for producing a water-dispersion thermal insulation composition based on limestone shell rock mining waste and an acrylate dispersion containing hollow microspheres, as well as to investigate the influence of filled binders on the physical, mechanical, and thermophysical properties of thermal insulation coatings incorporating limestone shell rock from the Zhetybay deposit, Karakiya District, Mangystau Region, Kazakhstan. The microstructure of shell limestone was investigated using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) with a JEOL JSM-7000F scanning electron microscope. The determination of the weight and atomic percentages of the major elements confirmed the carbonate nature of the studied raw material and its consistency with the mineral composition of shell limestone mining waste (wt.%/at.%: O – 50.25/59.34; Ca – 34.14/16.09; C – 15.61/24.56). It was established that, to obtain a water-dispersion composition with low density, it is advisable to use finely dispersed fillers in the form of powder, as well as hollow glass and aluminosilicate microspheres. The particle size of shell limestone powder was 5.67×10^{-6} m, while the contents of calcium carbonate ($CaCO_3$) and silica (SiO_2) were 71.0 wt.% and 22.5 wt.%, respectively. A relationship between the proportion and content of shell limestone and microspheres in the filled binder system was identified, demonstrating a significant variation in thermal conductivity within the range of 0.010–0.015 W/(m·°C), as well as changes in the density and adhesive strength of the thermal-insulating coating, reaching $\rho_{(dry)}$ = 0.410–0.415 g/cm³ and $\sigma_{(adh)} \geq$ 2.05–2.08 MPa, respectively. The optimal composition of the water-dispersion thermal-insulating coating was determined. The content of the main components is as follows (wt.%): styrene–acrylic or acrylic dispersion, 20–25; shell limestone, 20–25; glass microspheres, 3–9; and aluminosilicate microspheres, 20–30.*

Keywords: *hollow microspheres, shell limestone, water-dispersion thermal insulation composition, acrylate dispersion, thermal conductivity.*

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ҒЫЛЫМИ МАҚАЛА

ӘКТАС-ҰЛУТАС ЖӘНЕ ҚУЫС МИКРОСФЕРАЛАР НЕГІЗІНДЕГІ СУ-ДИСПЕРСТІ ЖЫЛУОҚШАУЛАҒЫШ КОМПОЗИЦИЯНЫҢ ҚАСИЕТТЕРІН ЗЕРТТЕУ

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Аңдатпа. *Осы жұмыстың мақсаты – әктас-ұлутас өндіру қалдықтары, акрилаттық дисперсия және қуыс микросфералар негізінде су-дисперсиялық жылуоқшаулағыш композицияны алу технологиясын әзірлеу, сондай-ақ Маңғыстау облысы Қарақия ауданының Жетібай кен орнының әктас-ұлутасы негізіндегі толтырылған байланыстырғыштардың жылуоқшаулағыш жабынның физика-техникалық, механикалық және жылутехникалық қасиеттеріне әсерін зерттеу болып табылады. Әктас-ұлутастың микроқұрылымы сканерлеуші электрондық микроскопия (СЭМ) және энергодисперсиялық спектралдық талдау (EDS) әдістерімен JEOL JSM-7000F сканерлеуші электрондық микроскопын пайдалану арқылы зерттелді. Негізгі элементтердің массалық және атомдық үлестерін анықтау зерттеліп отырған шикізаттың карбонатты табиғатын және оның әктас-ұлутас кенін өндіру қалдықтарының минералдық құрамына сәйкестігін растады (масс.үл./атом.үл., %: O – 50.25/59.34; Ca – 34.14/16.09; C – 15.61/24.56). Төмен тығыздықтағы су-дисперсиялық композицияны алу үшін ұнтақ тәрізді жұқа дисперсті толтырғыштарды, сондай-ақ қуыс шыны және алюмосиликатты микросфераларды пайдалану орынды екендігі анықталды. Әктас-ұлутас ұны бөлшектерінің өлшемі $5.67 \cdot 10^{-6}$ м, кальций карбонатының ($CaCO_3$) мөлшері – 71.0 %, кремнеземнің (SiO_2) мөлшері – 22.5 % құрайды. Толтырылған байланыстырғыш құрамындағы әктас-ұлутас пен микросфералардың арақатынасы мен үлестік мөлшерінің өзара тәуелділігі анықталды. Зерттеу нәтижелері жылуөткізгіштік коэффициентінің 0.010 ÷ 0.015 Вт/(м·°С) аралығында елеулі өзгеретінін, сондай-ақ жылуоқшаулағыш жабынның тығыздығы мен адгезиялық беріктігінің өзгеретінін көрсетті: $\rho_{құрғ}$ = 0.410 ÷ 0.415 г/см³ және $\sigma_{адг} \geq$ 2.05 ÷ 2.08 МПа. Су-дисперсиялы жылуоқшаулағыш композицияның оңтайлы құрамы анықталды, онда негізгі компоненттердің мөлшері, масс. % бойынша, келесідей: стирол-акрилді немесе акрилді дисперсия – 20 ÷ 25; әктас-ұлутас – 20 ÷ 25; шыны микросфералар – 3 ÷ 9; алюмосиликатты микросфералар – 20 ÷ 30.*

Түйінді сөздер: *қуыс микросфералар, әктасты ұлутас, су-дисперсиялық жылуоқшаулағыш композиция, акрилаттық дисперсия, жылуөткізгіштік коэффициенті.*

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НАУЧНАЯ СТАТЬЯ

ИССЛЕДОВАНИЕ СВОЙСТВ ВОДНО-ДИСПЕРСИОННОЙ ТЕПЛОИЗОЛЯЦОННОЙ КОМПОЗИЦИИ НА ОСНОВЕ ИЗВЕСТНЯКА-РАКУШЕЧНИКА И ПОЛЫХ МИКРОСФЕР

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Аннотация. *Целью данной работы является разработка технологии получения водно-дисперсионной теплоизоляционной композиции на основе отходов добычи известняка-ракушечника и акрилатной дисперсии с содержанием полых микросфер, изучение влияния наполненных связующих на физико-технические, механические и теплотехнические свойства теплоизоляционного покрытия с содержанием известняка-ракушечника Жетыбайского месторождения Каракиянского района Мангистауской области. Исследование микроструктуры известняка-ракушечника проводилось методом сканирующей электронной микроскопии (СЭМ) и энерго-дисперсионного спектрального анализа (EDS) с использованием сканирующего электронного микроскопа JEOL JSM-7000F. Определение массовых и атомных долей содержания основных элементов подтвердило карбонатную природу исследуемого сырья и соответствие минеральному составу отходов добычи известняка-ракушечника (масс.д./атом.д., %: O – 50.25/59.34; Ca – 34.14/16.09; C – 15.61/24.56). Установлено, что для получения водно-дисперсионной композиции с низкой плотностью целесообразно использовать тонкодисперсные наполнители в виде муки, а также полые стеклянные и алюмосиликатные микросферы. Размер частиц муки известняка-ракушечника – $5.67 \cdot 10^{-6}$ м, содержание карбоната кальция ($CaCO_3$) – 71.0 %, кремнезёма (SiO_2) – 22.5 %. Выявлена зависимость соотношения и доли в составе наполненного связующего известняка-ракушечника и микросфер, которая показала существенное изменение теплопроводности в пределах 0.010 ÷ 0.015 Вт/(м^2 ^{0}С), а также изменение плотности и адгезионной прочности теплоизоляционного покрытия - $\rho_{сух}$ = 0.410 ÷ 0.415 г/см^3 и $\sigma_{адг} \geq$ 2.05 ÷ 2.08 МПа. Определен оптимальный состав водно-дисперсионной теплоизоляционной композиции, в которой содержание основных компонентов следующее, масс. %: стирол-акриловой или акриловой дисперсии 20 ÷ 25; известняка-ракушечника – 20 ÷ 25; стеклянных микросфер – 3 ÷ 9; алюмосиликатных микросфер – 20 ÷ 30.*

Ключевые слова: *полые микросферы, известняк-ракушечник, водно-дисперсионная теплоизоляционная композиция, акрилатная дисперсия, коэффициент теплопроводности.*

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1 INTRODUCTION

At present, thermal-insulating paint and coating materials are gaining increasing popularity in the construction materials market. The advantages of thermal-insulating coatings include the stability of their thermal insulation properties throughout their service life, ease of application on hard-to-reach areas of structural surfaces, fire safety, environmental friendliness, resistance to atmospheric influences, and other beneficial characteristics [1; 2]. Thermal-insulating paint and coating materials can be applied to virtually any type of substrate, including concrete, ceramic, wooden, and metal surfaces [3].

Issues related to energy conservation in the construction industry are becoming increasingly important due to the continuous rise in energy costs. Heat losses during transportation through district heating and heat supply systems can reach 20% or more of the total heat energy consumption. Therefore, reducing thermal losses through the use of energy-efficient materials and technologies represents an important direction for the development of the construction sector [4; 5].

In recent years, Kazakhstan has experienced significant growth in both new construction projects and the renovation of existing residential buildings, which has stimulated the active development of the construction and paint-and-coating materials market. At the same time, the share of paint and coating materials produced for construction applications has been steadily increasing, accounting for approximately 30% of total production.

The principal component of any paint and coating material is the film-forming binder, which constitutes the continuous phase of the system. Film-forming agents of various origins are capable of forming a continuous film on a solid substrate with sufficient strength, hardness, elasticity, adhesion, and other essential performance characteristics. The selection of a film-forming binder is primarily determined by its operational and service properties [6].

The author [7] notes that, as a binder in thermal insulation coatings intended for the insulation of district heating pipelines, the most effective paint and coating systems are organosilicate, epoxy, silicone-based, polyurethane, and silicate-enamel coatings.

It is well known that mineral surfaces absorb moisture when humidity increases and release it in the form of water vapor when humidity decreases. According to the author [8], coatings applied to such surfaces should possess a structure that prevents the penetration of water in its condensed phase while allowing the diffusion of moisture in its gaseous (vapor) state. If the façade is unable to «breathe» moisture will accumulate within the wall structure, leading to the deterioration of the mineral substrate.

Due to their hygroscopic nature, wooden surfaces absorb moisture, resulting in dimensional changes in both tangential and radial directions until their hygroscopic limit is reached. During the drying process, the substrate undergoes shrinkage. These cyclic dimensional changes adversely affect the durability of the coating, eventually causing cracking and delamination. Therefore, binders intended for wooden substrates should exhibit high elasticity and water resistance [9].

In recent years, water-dispersion paints have gained widespread acceptance, as their production and application do not require the use of flammable and toxic organic solvents. These paints are environmentally friendly, provide high coating quality, and are economically advantageous because they eliminate losses associated with evaporating organic solvents and reduce expenditures on ventilation systems and safety measures. Additional advantages of water-dispersion paints include rapid drying, the ability to be applied to damp surfaces, and the possibility of carrying out painting operations under conditions of elevated air humidity [10].

According to the type of film-forming binder, water-dispersion paints for construction applications are classified into four categories:

- acrylic;
- acrylic-styrene;
- butadiene-styrene;
- polyvinyl acetate (PVA)-based paints [11].

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Among all types of water-dispersed paints, formulations based on acrylic aqueous dispersions offer the greatest advantages, as they form coatings characterized by enhanced weather resistance, water resistance, high durability against aging, and excellent resistance to alkaline environments [11].

Particular interest is associated with limestone-shell rock quarrying waste from the Zhetybai deposit located in the Karakiyan District of the Mangystau Region. The chemical composition of the material confirmed its carbonate nature and corresponded to the mineral composition of limestone-shell rock quarrying waste (wt. % / at. %: O – 50.25/59.34; Ca – 34.14/16.09; C – 15.61/24.56). A review of the available literature revealed no previous studies on the development of water-dispersion thermal-insulating compositions based on this waste material in combination with an acrylate dispersion containing hollow microspheres.

The utilization of technogenic formations accumulated in mineral deposits and tailing storage facilities remains an important scientific and practical challenge. The development of waste-free technologies for the extraction and processing of natural stone has been addressed in a number of studies [12–16], which propose various technical solutions for the recycling and valorization of mining and mineral-processing waste.

Therefore, the aim of the present study was to develop a scientifically substantiated technology for producing a water-dispersion thermal-insulating composition based on finely dispersed fillers, possessing enhanced thermal-insulating and эксплуатационные properties.

The following objectives were accomplished:

1. Investigation of the surface morphology and structural changes of shell limestone waste using scanning electron microscopy (SEM);
2. Determination of the chemical and elemental composition of shell limestone waste by energy-dispersive X-ray spectroscopy (EDS) using a JEOL JSM-7000F scanning electron microscope;
3. Establishment of the optimum content of shell limestone waste (up to 30 wt.%) and evaluation of its influence on the properties of the thermal insulation coating;
4. Development of a technology for producing a water-dispersion thermal insulation composition based on shell limestone waste and hollow microspheres, as well as assessment of the applicability of the developed coatings for industrial use.

Based on a comprehensive literature review, the following hypothesis was formulated: the incorporation of mineral raw material wastes, namely diatomite and hollow microspheres, promotes the formation of a porous microstructure and enhances the thermal insulation performance of the coating without compromising its physicomechanical properties [17; 18].

One of the promising approaches to improving the efficiency of shell limestone deposit exploitation is the utilization of waste generated during the extraction of dimension stone [19]. Although these wastes have found applications in the chemical industry, cement production, and cosmetic products, their accumulation continues to exceed the rate of utilization due to the insufficient exploitation of this valuable mineral resource.

Studies conducted by the author [20] demonstrated that construction materials produced from limestone waste meet the requirements of the construction industry and can be manufactured directly at quarry sites where building stone is extracted. This approach can reduce production costs for enterprises and create significant opportunities for industrial and construction applications, thereby contributing to a more sustainable environment and mitigating the environmental impact of shell limestone quarrying operations.

A thermal-insulating coating was developed based on the commercially produced acrylic dispersion Acrémos-101 and K20 hollow glass microspheres. The coating exhibited the following performance characteristics: tensile strength (σ_p) of 1.07 MPa, elongation at break ($\mathcal{E}_p$) of 15%, and thermal conductivity (λ) of 0.063 W/(m·K). The developed thermal-insulating protective coating, Acr-101-30, was applied in the housing and public utilities sector for the thermal protection of hot-water supply and heating pipelines. The calculated reduction in heat losses per unit surface area reached 41% [21].

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The authors of [22] investigated the physical and mechanical properties of shell limestone sawing waste and demonstrated its suitability for use in construction applications.

In study [23], the feasibility of utilizing waste products from titanium dioxide production, vinyl chloride copolymers, and bleaching powder as raw materials for the manufacture of paint and coating compositions was theoretically substantiated. The developed coatings exhibited enhanced adhesion, coating elasticity, and resistance to gasoline, mineral oils, salts, and water, while also demonstrating faster drying rates compared to the conventional XS-510 paint and coating material.

The study reported in [24] established the feasibility and effectiveness of using hollow fly ash microspheres as a microspherical filler in the formulation of thermal-insulating paint and coating materials.

A water-dispersion thermal-insulating, anti-corrosion, and anti-condensation coating composition for metal surfaces is described in [25-35]. The formulation contains a film-forming binder, hydrophobic dispersant, polyurethane and cellulose thickeners, defoamer, anti-corrosion pigments, fillers, coalescing agents, neutralizing agent, film fungicide, and solvent. However, the disadvantages of this coating include the complexity of preparation, relatively low physicomechanical properties, and limited functional performance during service.

Thus, the results obtained in the development of a water-dispersion thermal-insulating composition based on shell limestone waste confirm the relevance of the present research and complement existing studies. The findings indicate that the incorporation of 20–25 wt.% shell limestone is optimal due to the spherical morphology of the microsphere particles and the nanoscale particle-size distribution, which ensure a high packing density of the hollow filler particles within the thermal-insulating coating matrix and contribute to reduced thermal conductivity.

2 MATERIALS AND METHODS

Particular attention in this study was devoted to the development of an effective preparation method for water-dispersion thermal-insulating coatings based on dispersed fillers, including shell limestone powder, limestone powder, and diatomite powder, modified with hollow microspheres (glass and aluminosilicate).

Zhetybay deposit located in the Karakiya district of the Mangystau region was used as the mineral filler, while an acrylic dispersion served as the polymer binder.

Shell limestone is a waste product generated during the extraction of natural shell rock from the Zhetybay deposit. It belongs to sedimentary rocks of organogenic origin and consists of small shell fragments cemented by a heterogeneous fine-crystalline carbonate binder, forming a layered structure.

Amorphous aluminosilicates are commonly used to expand the raw material base and reduce the production cost of thermal insulation materials. In the present study, they were utilized as a modifying additive.

Hollow glass microspheres are a micropowder produced through grinding and classification of synthesized glass. The production method of hollow glass microspheres involves creating conditions for the preliminary dissolution of a certain amount of gases during the preparation of the intermediate product, followed by gas release during thermal dissociation. The high heating temperature, which causes glass softening, together with gas evolution, promotes bubble growth within the particles and the formation of microspheres. Rapid cooling conditions ensure the stabilization of the dimensions and morphology of the formed structure.

The characteristic diameters of hollow glass microspheres depend on their specific application and typically range from 10 to 200 μm, while the shell wall thickness varies from 0.5 to 2.0 μm [26].

The microstructure of shell limestone was investigated using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) employing a JEOL JSM-7000F scanning electron microscope.

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The particle size distribution of fine fillers, including limestone shell rock sawing waste (limestone flour) and limestone powder, was determined using a Microtrac S3500 particle size analyzer. The measurement range of the instrument is 0.021 – 2000 µm. For particles within the upper size range, a dry measurement method was employed, in which the sample was dispersed by an air stream. The particle size distribution of finely dispersed materials was determined using a wet measurement technique with water as the dispersing medium. To ensure uniform particle dispersion and prevent agglomeration, ultrasonic treatment was applied for 3 min. Measurements were performed using the laser diffraction method with a laser wavelength of 780 nm. The measurement accuracy was characterized by a relative deviation not exceeding 0.7%.

The required amount of additive was introduced into a 200 mL glass beaker and mixed at room temperature using a magnetic stirrer for 5 min. Plate specimens made of St3 carbon steel with dimensions of 100 × 50 × 1 mm were used as substrates. The coating was applied to the steel specimens in two layers by the dip-coating method at room temperature, followed by drying in a laboratory drying oven at 40°C until tack-free. Subsequently, the coated specimens were conditioned at room temperature for 24 h. The main performance characteristics of the paint coatings, including porosity, adhesion strength, flexibility, hiding power, and thermal conductivity, were evaluated using standard test methods.

3 RESULTS AND DISCUSSION

The development of formulations utilizing locally available raw materials and industrial waste will make it possible to obtain a product that is comparable in quality to foreign counterparts while being more economically attractive. To address this objective, based on the results of the conducted studies, a list of key factors governing the structure formation of a water-dispersion thermal insulation composition is presented in Table 1.

Table 1 – List of controlling factors affecting the structure formation of a water-dispersion thermal insulation composition [Authors' material]

Level	Factors			
	Composition-Related Factors		Technological Factors	
	№	Factor Name	№	Factor Name
I	1	Content of components (mineral additive, filler, functional filler, plasticizer, modifier, and water)	1	Mixing regime
			2	Mixing regime
II	1	Fineness of the mineral additive	1	Duration and intensity of component mixing
	2	Fineness of the filler		
	3	Fineness of the functional filler	2	Duration and intensity of component mixin
	4	Interfacial bond strength	3	Hardening/drying duration and strength development
	5	Modifier activity		
III	1	Chemical activity of cement/lime	1	Surface roughness of the filler
	2	Chemical activity of the mineral additive		
		Chemical activity of the modifier		
	3	Chemical activity of the functional filler	2	Modifier incorporation procedure

Mining waste of shell limestone obtained from a section of the Zhetybai deposit located in the Karakiya District of the Mangystau Region, Kazakhstan, was used in this study.

SEM image analysis revealed that the shell limestone mining waste is characterized by a heterogeneous dispersed structure with a highly developed rough surface morphology. The micrographs show irregular angular particles, agglomerated formations, and intergranular voids of various sizes (Figure 1 a, b.).

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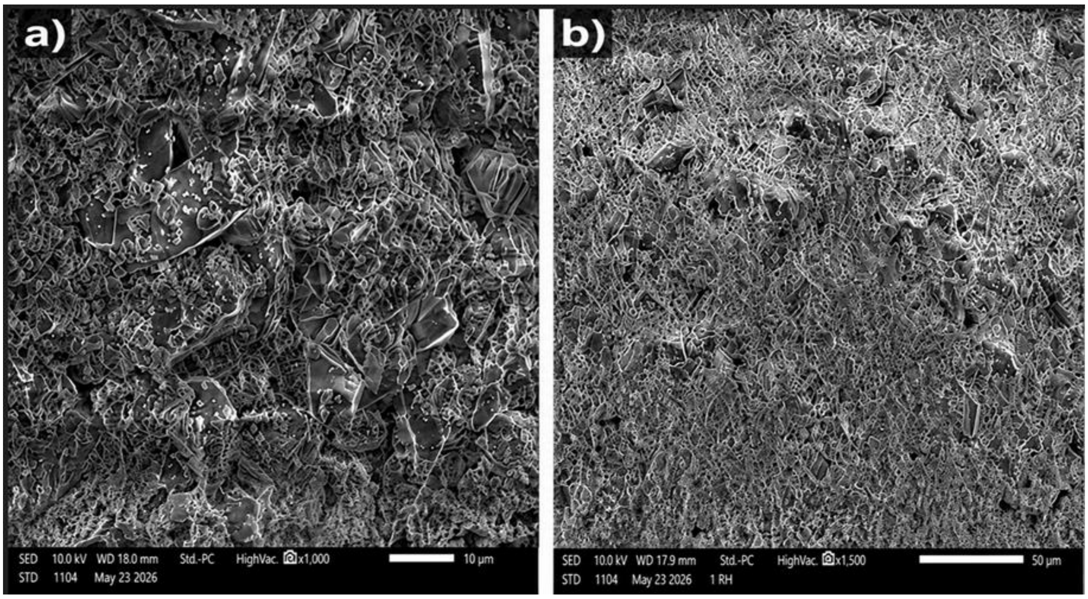

Figure 1 – SEM images of waste from the Zhetybai shell limestone quarry [Authors' material]

At a magnification of ×500, the overall heterogeneity of the structure and the distribution of particles with different shapes and sizes can be observed. At a magnification of ×1000, lamellar structures, a rough surface microrelief, and finely dispersed particles become more clearly distinguishable.

The presence of a porous structure and intergranular voids may contribute to a reduction in the thermal conductivity of the investigated raw material due to an increase in the volume of entrapped air. To determine the elemental composition of the investigated raw material, energy-dispersive X-ray spectroscopy (EDS) analysis was performed (Figure 2 a, b and Table 2).

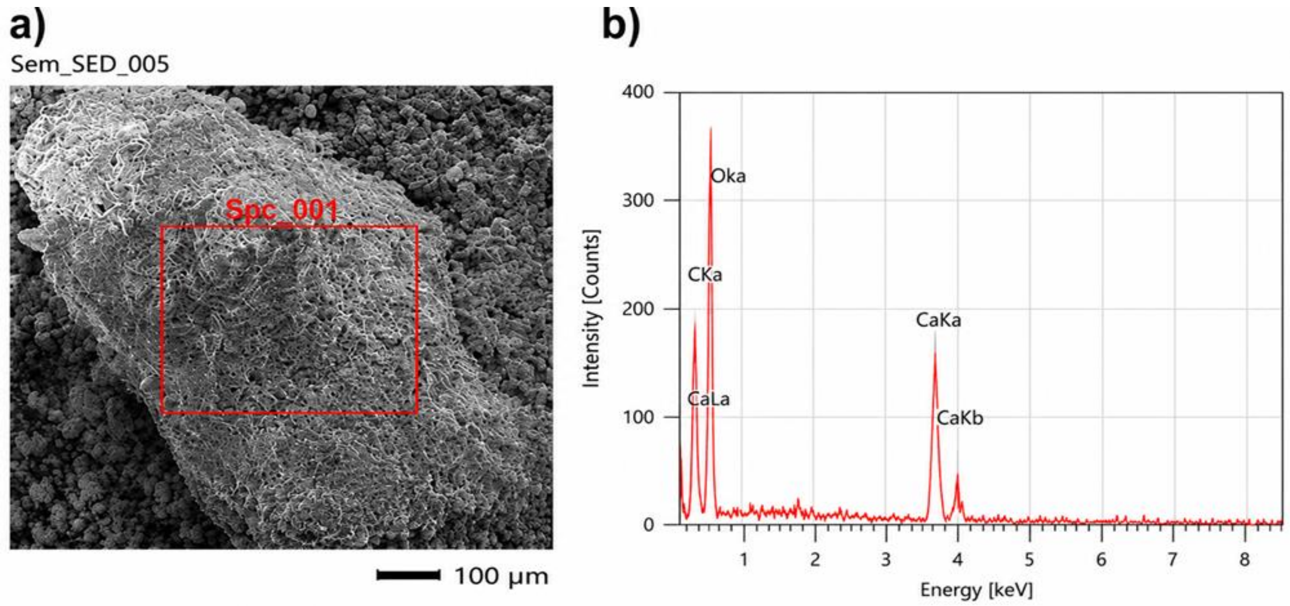

Figure 2 – EDS spectrum of shell limestone quarry waste (Spc_001 area) [Authors' material]

Table 2 – Results of the EDS analysis of shell limestone quarry waste from the Zhetybai deposit [Authors' material]

№	Element	Weight Fraction, %	Atomic Fraction, %
1	C	15.61	24.56
2	O	50.25	59.34

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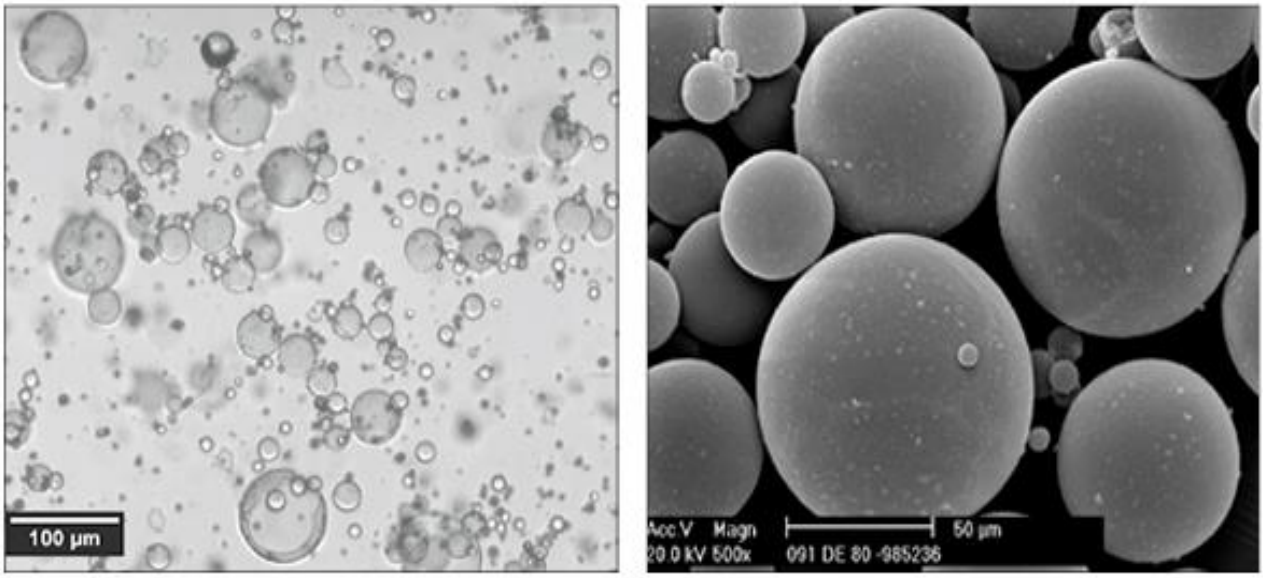
3	Ca	34.14	16.09

The EDS analysis results revealed that the principal elements of the investigated raw material are oxygen (O), calcium (Ca), and carbon (C). The elevated contents of calcium and oxygen indicate the predominance of carbonate compounds, which are characteristic of shell limestone. The determination of the weight and atomic fractions of the major elements confirmed the carbonate nature of the investigated material and its consistency with the mineral composition of shell limestone quarrying waste (wt.%/at. %: O – 50.25/59.34; Ca – 34.14/16.09; C – 15.61/24.56).

The hollow glass microspheres used in this study were separated from defective particles by water flotation. This procedure yielded microspheres with particle sizes ranging from 20 to 100 µm, an average diameter of 75 µm, and a wall thickness of 1–2 µm (Figure 3 a, b.).



a) 20-100 µm b) 75 µm

Figure 3. Scanning electron micrograph of hollow glass microspheres [Authors' material]

The bulk density of the dried microspheres was 0.107 g/cm³, while their thermal conductivity coefficient was 0.05 W/(m·K). Hollow glass microspheres act as highly opaque light-reflective pigments, making them suitable for use in the formulation of water-dispersed paint and coating compositions.

The incorporation of microspheres into composite materials contributes to enhanced thermal insulation and sound absorption performance. The main technical characteristics and chemical composition of the glass microspheres are presented in Tables 3 and 4.

Table 3 – Main characteristics of 3M™ K15 hollow glass microspheres [Authors' material]

Grade	Wall thickness, µm	Density, g/cm³	Ratio V_{inner}/V_{total}	Crushing strength of 90% intact particles, MPa (bar)	Thermal conductivity, W/(m·K)	Mean diameter, µm
K15	0.61	0.154	0.942	2.14 (214)	0.0511	61

Table 4 – Chemical composition of glass microspheres [Authors' material]

Oxides	Si_2O	Na_2O	CaO	B_2O_3
Content, wt.%	78	5	10	4

The mineral phase of the water-dispersion composition consisted of finely dispersed fillers of different origins and particle size distributions.

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The following mineral additives were used - shell limestone powder is a finely ground sedimentary porous limestone rock composed predominantly of marine shell fragments and whole shells. The average particle size was 5.67 × 10^{-6} m, while the average contents of calcium carbonate ($CaCO_3$) and silica (SiO_2) were 71.0% and 22.5%, respectively. The particle size distribution curve of the shell limestone powder is presented in Figure 4.

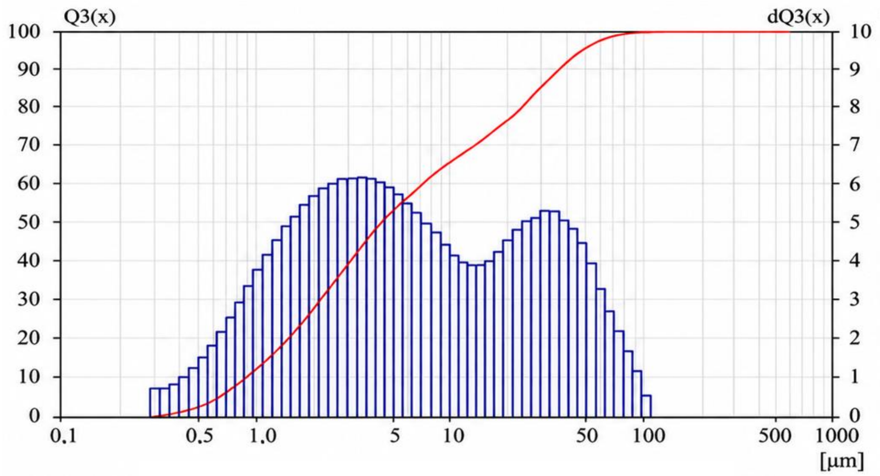

Figure 4. Particle size distribution curve of shell limestone powder [Authors' material]

- limestone powder was produced by grinding waste generated during the crushing of carbonate rocks or coarse screening fractions obtained in crushed stone production. The physicochemical characteristics of the limestone powder are presented in Table 5. Figure 5 - illustrates the particle size distribution of the limestone powder.

Table 5 – Physicochemical properties of limestone powder [Authors' material]

№	Parameter	Value
1	Appearance	White powder
2	Total mass fraction of $CaCO_3$ + $MgCO_3$ (on a dry basis), %	89.9
3	Moisture content, %	0.31
4	Mass fraction of ferromagnetic impurities, %, not more than	8.9
5	Mass fraction of non-hazardous impurities, %	1.25
6	Total content of toxic substances, %, not more than	0.01

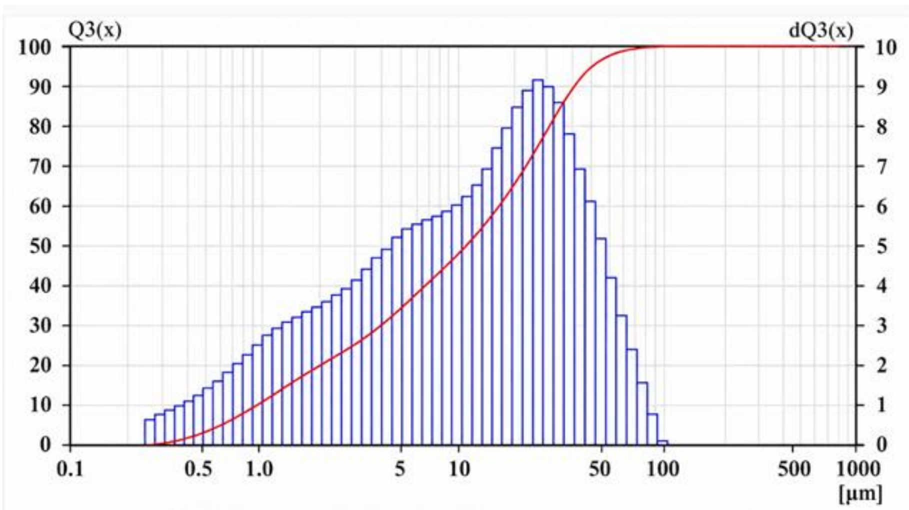

Figure 5. Particle size distribution curve of limestone powder [Authors' material]

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Aluminosilicate microspheres are characterized by a bulk density of 370–450 kg/m³, an average particle size of 110 μm, a shell hardness of 5–6 on the Mohs scale, and a thermal conductivity coefficient of 0.11 W/(m·°C).

The amorphous aluminosilicate additive was synthesized by precipitating aluminosilicates from liquid sodium silicate with a silicate modulus of 2.81 through the introduction of a 15 wt.% solution of technical-grade aluminum sulfate, $Al_2(SO_4)_3$, followed by washing the resulting precipitate with distilled water. The obtained precipitate was dried at 100°C to constant mass and subsequently ground to a specific surface area of 688.8 m²/kg.

The physicochemical properties of the mineral additive based on amorphous aluminosilicates are presented in Table 6.

Table 6 – Physicochemical characteristics of the mineral additive [Authors' material]

№	Parameter	Value
1	Appearance	White powder
2	Activity, mg/g	351.0
3	Specific surface area, m²/kg	688.8
4	Bulk density, kg/m³	456.0
5	True density, kg/m³	2130.0

During the course of the research, the optimal formulations of the water-dispersion thermal-insulating composition were determined. The resulting compositions are presented in Table 7.

Table 7 – Optimal formulations of the waterborne thermal insulating coating composition [Authors' material]

№	Component	Component content, wt.%		
		Composition		
		No.1	No.2	No.3
1	Styrene-acrylic or acrylic dispersion	20	22.5	25
2	Shell limestone powder	25	22.5	20
3	Hollow glass microspheres	9	6	3
4	Amorphous aluminosilicates	20	25	30
5	Hydrophobic dispersing agent	0.4	0.5	0.6
6	Defoamer	0.05	0.07	0.09
7	Sodium nitrite	0.15	0.10	0.05
8	Talc	3	2	1
9	Titanium dioxide pigment (TiO_2)	2	2,5	3
10	Cerium oxide (CeO_2)	4	3	2
11	Water	Other		

The incorporation of a specific combination of components into the composition made it possible to achieve the required coating properties, including early water resistance, elasticity, hydrophobicity, and low thermal conductivity.

The developed composition is based on filled binder systems comprising aqueous styrene-acrylic or acrylic dispersions, shell limestone, and hollow microspheres. The resulting thermal insulation coating, characterized by the high porosity of finely dispersed fillers, exhibits a low bulk density, which contributes to a reduction in thermal conductivity. The lowest thermal conductivity values ranged from 0.010 to 0.015 W/(m·°C), which was achieved through the use of filled binder systems. Comparative test results of the developed composition are presented in Table 8.

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Table 8 – Test results of the optimal formulations of the developed water-dispersion thermal insulation coating [Authors' material]

№	Parameter	Example 1	Example 2	Example 3	Control sample
1	Hiding power, g/m²	76	78	76	80
2	Drying time of the coating (to degree 3) at T = 20°C, h	2	2	2	2
3	Fineness of grind, µm	50	50	50	50
4	Resistance of the coating to temperature fluctuations from −40°C to +60°C, cycles	25	25	25	25
5	Adhesion, score	1	1	1	1
6	Flexibility on bending, mm	1	1	1	1
7	Resistance to static water exposure at T = (20 ± 2)°C, h	760	780	800	720
8	Resistance to elevated humidity and temperature, h, not less than	760	780	800	720
9	Tensile strength after application, MPa	3.2	3.2	3.2	3.2
10	Frost resistance of the coating after 10 freeze–thaw cycles, appearance	No changes	No changes	No changes	No changes
11	Thermal conductivity coefficient, W/(m·°C)	0.010	0.013	0.015	0.020

Initially, a premix is prepared by loading the components into a mixing vessel under continuous stirring in the following sequence: water, hydrophobic dispersant, defoamer, sodium nitrite, fillers (talc and cerium oxide, CeO_2), titanium dioxide pigment (TiO_2), followed by the addition of amorphous aluminosilicates. After thorough mixing, the premix is fed into a bead mill, where the fillers and pigments are ground to a fine particle size of approximately 10 µm. Subsequently, a filled binder system is introduced into the premix, consisting of a styrene-acrylic or acrylic dispersion, shell limestone, and hollow glass microspheres.

During the dispersion and mixing process, continuous temperature monitoring is carried out to maintain the temperature below 40 °C by cooling both the mixing vessel and the bead mill. The mixing duration is determined based on the fineness of grind and coating application properties of the product until a viscosity of 1500–5000 cP (Brookfield viscosity) is achieved. The comparative test results of the developed composition are presented in Table 9. During the experimental investigations, the thermal conductivity of filled binder compositions and thermal insulation coatings based on these binders was evaluated. It is well known that dry air is one of the most effective thermal insulators, exhibiting a thermal conductivity coefficient of 0.023 W/(m·K), particularly when it is trapped within the pores of a material and remains immobile. When the pores are large and interconnected with each other and the external environment, convective air movement occurs, resulting in an increase in the thermal conductivity of the material. Fine-dispersed mineral powders possess high porosity, which significantly reduces the density and, consequently, the thermal conductivity of the developed compositions. As a result, the obtained materials exhibited relatively low thermal conductivity values.

According to the Barrett–Joyner–Halenda (BJH) method, the pore volume and pore size of aluminosilicate microspheres were determined to be 3.84×10^{-6} m³/kg and 40.9 nm, respectively. This can be explained by the absence of a well-developed pore structure within the microsphere wall that could facilitate capillary water absorption into the microsphere interior. Hollow aluminosilicate microspheres contain defects in the form of hemispherical formations, the occurrence of which is associated with both the microsphere formation process and the physicochemical processes taking place within the structure of the thermal insulation coating.

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Table 9 – Test Results of the optimal formulations of the developed water-dispersed thermal insulation composition [Authors' material]

Parameter	Example 1	Example 2	Example 3	Control sample
Hiding power, g/m²	76	78	76	80
Drying time of coating (to degree 3), h, at T = 20°C	2	2	2	2
Fineness of grind, µm	50	50	50	50
Resistance of coating to temperature fluctuations from −40°C to +60°C, cycles	25	25	25	25
Adhesion, score	1	1	1	1
Flexibility in bending, mm	1	1	1	1
Resistance to static water exposure at T = (20 ± 2)°C, h	760	780	800	720
Resistance to elevated humidity and temperature, h, not less than	760	780	800	720
Tensile strength after application, MPa	3.2	3.2	3.2	3.2
Frost resistance of coating (10 cycles), appearance	No changes	No changes	No changes	No changes
Thermal conductivity coefficient, W/(m·°C)	0.010	0.013	0.015	0.020

Thus, it can be concluded that aluminosilicate and glass hollow microspheres do not possess a well-developed porous structure. Their adverse effect on the flowability of the water-dispersed composition, including the disruption of the natural relationship between the specific surface area of mineral components and the rheological properties of the system, is primarily associated with the pronounced surface roughness of the hollow filler particles.

As a result of the study, a relationship between the proportion and content of shell limestone and microspheres in the filled binder composition was established. This relationship demonstrated a significant variation in thermal conductivity within the range of 0.010–0.015 W/(m·°C), as well as changes in the density of the thermal insulation coating (ρ_{dry} = 0.410–0.415 g/cm³) and adhesion strength ($\sigma_{adh} \geq$ 2.05–2.08 MPa).

The spherical morphology of the microsphere particles and their particle-size distribution in the nanometer range provide a high packing density of the hollow filler within the thermal insulation coating matrix. The high mechanical strength of the microsphere shell, combined with strong interfacial adhesion between the finely dispersed mineral filler and the hollow filler, contributes to the formation of high-strength composite materials.

The incorporation of finely dispersed fillers resulted in a significant increase in the viscosity of the composition, which is attributed to their high adsorption capacity. Prior to the introduction of hollow microspheres into the filled binder system, the compositions were adjusted by adding an additional amount of water. The total water consumption at all stages of preparing the filled binders and other components, calculated per 100 g of mixture, is presented in **Figure 6.**

In the present study, the values corresponding to the characteristics of the thermal insulation coating were adopted as reference parameters for all optimized properties (thermal conductivity, density, and adhesive strength): $\lambda \leq 0.074$ W/(m·K); $\rho_{dry} \leq 0.414$ g/cm³; and $\sigma_{adh} \geq 1.04$ MPa.

Hollow microspheres were incorporated into the filled binder compositions until the required workability of the mixture was achieved. The content of K15 3M™ hollow glass microspheres as a function of composition is presented in Figure 7. Considering the low density of the glass microspheres (0.154 g/cm³), their total content in the compositions ranged from 3 to 9 wt. %.

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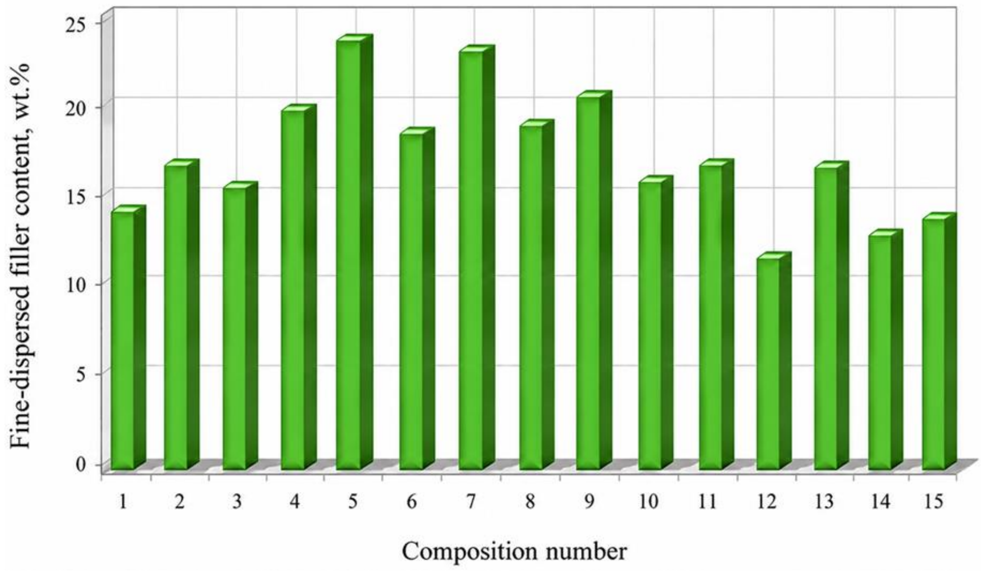

Figure 6. Variation in the acrylic dispersion content as a function of the thermal insulation coating mass [Authors' material]

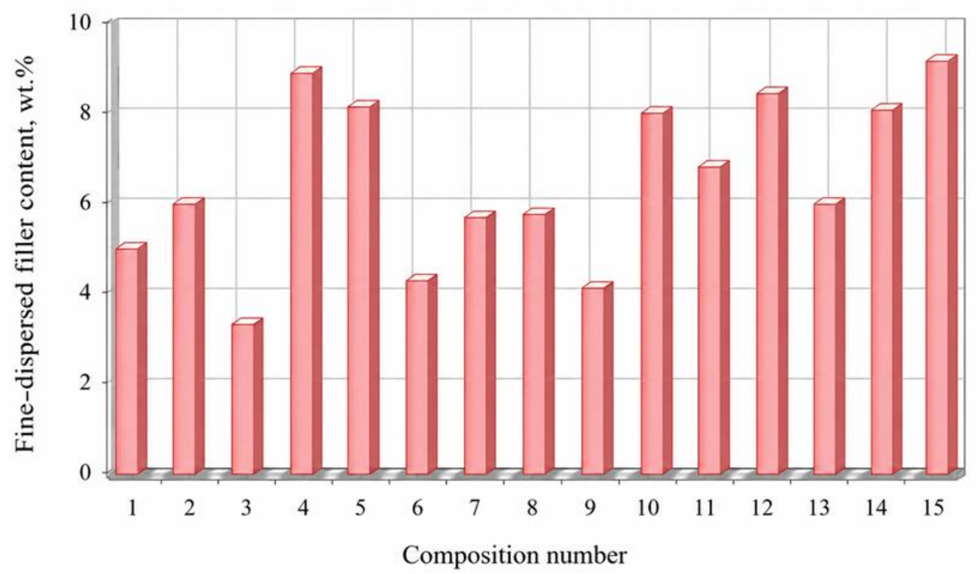

Figure 7. Variation in the content of K15 3M™ hollow glass microspheres as a function of the mass of the thermal insulation coating [Authors' material]

The feasibility of incorporating hollow glass and aluminosilicate microspheres is justified by their advantageous physicomechanical properties, low thermal conductivity, and their potential to reduce the production cost of water-dispersed thermal insulation compositions.

The use of styrene-acrylic or acrylic dispersions makes it possible to obtain coatings with enhanced weather resistance, water resistance, high durability against aging, and excellent alkali resistance. An important advantage of acrylic-based coatings is their long service life. In particular, coatings formulated with acrylic binders can maintain their performance characteristics for 8–10 years and, in certain applications, up to 30 years.

Thus, the developed method for producing a water-dispersed thermal insulation composition and the optimized formulations based on shell limestone modified with hollow microspheres enable the formation of a durable thermal insulation coating capable of maintaining its performance under atmospheric exposure and adverse climatic conditions. The resulting coatings exhibit improved

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thermophysical properties, enhanced adhesion and operational performance, extended service life, and improved storage stability of the paint composition.

4 CONCLUSIONS

The research findings confirm the feasibility and effectiveness of utilizing limestone-shell rock waste from the Zhetybay deposit (Mangystau Region, Kazakhstan) as a finely dispersed filler in waterborne thermal-insulating coatings in combination with hollow glass and aluminosilicate microspheres. The incorporation of limestone-shell rock positively affects the overall performance characteristics of the coating.

Based on the experimental results, the following conclusions can be drawn:

1. A waterborne thermal-insulating composition was developed using filled binders based on aqueous styrene–acrylic or acrylic dispersions, limestone-shell rock, and hollow microspheres. The resulting thermal-insulating coating is characterized by a highly porous structure of finely dispersed fillers and low density, which contributes to a reduction in the thermal conductivity coefficient;

2. An effective method for producing the thermal-insulating composition was established. The process involves the preparation of a premix in a mixing vessel under continuous stirring according to the following sequence: water → hydrophobic dispersant → defoamer → fillers → pigment, followed by the addition of amorphous aluminosilicates. Subsequently, the filled binder consisting of styrene–acrylic or acrylic dispersion, limestone-shell rock, and hollow glass microspheres is introduced into the premix;

3. The selection of the main components and nanoscale fillers for producing a thermal insulation coating with low thermal conductivity was substantiated. It was established that, to obtain a water-dispersed composition with low density, it is advisable to use finely dispersed fillers in the form of mineral flour, as well as hollow glass and aluminosilicate microspheres. The spherical morphology of the microsphere particles and their particle size distribution within the nanoscale range ensure a high packing density of the hollow filler particles within the matrix volume of the thermal insulation coating;

4. The relationship between the proportion and content of shell limestone and microspheres in the filled binder composition was determined. The results demonstrated a significant variation in thermal conductivity within the range of 0.010–0.015 W/(m·°C), accompanied by changes in the density and adhesive strength of the thermal insulation coating, namely ρ_{dry} = 0.410–0.415 g/cm³ and $\sigma_{adh} \geq$ 2.05–2.08 MPa;

5. The most effective composition of the water-dispersed thermal insulation material was identified. The optimal formulation contains the following components (wt. %): styrene-acrylic or acrylic dispersion – 20–25; shell limestone – 20–25; glass microspheres – 3–9; aluminosilicate microspheres – 20–30.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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During the preparation of this manuscript, the authors used artificial intelligence tools (ChatGPT) solely for editorial assistance, such as improving phrasing and checking grammar, spelling, and punctuation. All ideas, interpretations, and conclusions are the responsibility of the authors, who take full accountability for the content of the article.

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