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«ҚАЗАҚСТАН РЕСПУБЛИКАСЫ
ҰЛТТЫҚ ҒЫЛЫМ АКАДЕМИЯСЫ» РҚБ

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ИЗВЕСТИЯ

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STUDY OF CHEMICAL-MINERALOGICAL COMPOSITION OF LIMESTONE-SHELL FROM THE ZHETIBAI FIELD

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Abstract. Construction is a vital sector where the use of concrete materials is steadily increasing. In Kazakhstan, these technologies are employed by more than 100 enterprises. However, due to intensive mineral extraction, more than 20 billion tons of waste have accumulated, including overburden rocks and slags, which contribute to the pollution of water, air, and soil. Global experience shows that an effective solution is the recycling of such waste into construction materials. This approach reduces environmental impact, conserves natural resources, and decreases energy consumption. *Results.* This study presents a method for evaluating the quality of limestone-shell rock from the Zhetybai deposit. Derivatographic analysis (DTA) was conducted using the "Derivatograph Q-1500D" device (MOM), which integrates DTA, TG, and DTG methods. It was determined that heat release is associated with the oxidation of magnesium carbonate ($MgCO_3$) and calcium carbonate ($CaCO_3$). Experimental data allowed the determination of optimal concrete compositions (35 seconds setting time), including the addition of sulfite-alcohol bard (SAB) and polyacrylamide (PAA) at 0.2% of the cement weight. These additives improved compressive strength by 24–33% in conventional concretes and by 1.5–11% in lightweight concretes. *Scientific novelty.* A new polymer-cement composition has

been developed based on limestone-shell rock sawing waste. The composition includes a 0.15% polyacrylamide solution combined with white cement (7–8%), filler (68–73%), white spirit (0.5–1.0%), and a hardening accelerator (0.7–1.2%). *Practical Value.* The proposed composition offers an efficient method for recycling mining waste into environmentally safe and durable construction materials.

Keywords: limestone-shell rock, polymer-cement composition, sample preparation, thermogram, derivatographic analysis, thermogravimetry

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ЖЕТІБАЙ КЕН ОРНЫНЫҢ ӘКТАС-ҰЛУТАСЫНЫҢ ХИМИЯ-МИНЕРАЛОГИЯЛЫҚ ҚҰРАМЫН ЗЕРТТЕУ

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Аннотация. Құрылыс – бетон материалдарын қолдану үнемі өсіп келе жатқан маңызды сала. Қазақстанда бұл технологиялар 100-ден астам кәсіпорында қолданылады. Алайда, пайдалы қазбаларды қарқынды өндіру нәтижесінде 20 миллиард тоннадан астам қалдық жиналып, су, ауа және топырақтың ластануына себеп болып отыр. Дүниежүзілік тәжірибе бұл қалдықтарды құрылыс материалдары ретінде қайта өңдеу – тиімді шешім екенін көрсетеді. Мұндай тәсіл экологиялық жүктемені азайтып, табиғи ресурстар мен энергия шығынын үнемдеуге мүмкіндік береді. *Нәтижелері.* Жұмыста Жетыбай кен орнының ұлутас жынысының сапалық қасиеттерін бағалау әдісі қарастырылған. Зерттеуде «Derivatograph Q-1500D» (MOM фирмасы) қондырғысында дериватографиялық талдау (DTA) қолданылды.

Онда DTA, TG және DTG әдістері біріктірілген. Зерттеу нәтижесінде жылу бөлу процесі магний карбонаты ($MgCO_3$) мен кальций карбонатының ($CaCO_3$) тотығуымен байланысты екені анықталды. Эксперименттік деректер негізінде ұлутас толтырғыштары бар бетонның оңтайлы құрамы (қаттылық уақыты – 35 секунд) анықталды. Цемент массасының 0,2%-ын құрайтын сульфит-спирт бардасы (ССБ) мен полиакриламид (ПАА) қоспаларын енгізу нәтижесінде кәдімгі бетонның қысу беріктігі 24–33%-ға, ал жеңіл бетонда 1,5–11%-ға артты. *Ғылыми жаңалығы.* Ұлутас қалдықтары негізінде жаңа полимерцементтік композиция әзірленді. Оның құрамына 0,15% полиакриламид ерітіндісі, ақ цемент (7–8%), толтырғыш (68–73%), уайт-спирит (0,5–1,0%) және қатайтқыш үдеткіш (0,7–1,2%) кіреді. *Практикалық құндылық.* Ұсынылған композиция тау-кен өндірісі қалдықтарын экологиялық қауіпсіз әрі берік құрылыс материалына тиімді айналдыруға мүмкіндік береді. Бұл шешім құрылыс индустриясында қолданылып, шикізат шығынын және қоршаған ортаға түсетін жүктемені азайта алады.

Түйін сөздер: әктас-ұлутас, полимерцементті композиция, сынама дайындау, термограмма, дериватографиялық талдау, термогравиметрия

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ИССЛЕДОВАНИЕ ХИМИКО-МИНЕРАЛОГИЧЕСКОГО СОСТАВА ИЗВЕСТНЯКА-РАКУШЕЧНИКА МЕСТОРОЖДЕНИЯ ЖЕТЫБАЙ

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Аннотация. Строительство — важная отрасль, где растёт использование бетонных материалов. В Казахстане эти технологии применяются на более чем 100 предприятиях. Однако из-за активной добычи полезных ископаемых накопилось свыше 20 млрд тонн отходов, загрязняющих воду, воздух и почву. Мировой опыт показывает, что эффективным решением является переработка этих отходов в строительные материалы. Это снижает экологическую нагрузку, экономит природные ресурсы и уменьшает энергозатраты. Результаты. В работе

рассмотрен метод оценки качества известняка-ракушечника Жетыбайского месторождения. Применён дериватографический анализ (DTA), выполненный на установке «Derivatograph Q-1500D» (фирма MOM), с объединением DTA, TG и DTG-методов. Установлено, что тепловыделение связано с окислением карбонатов $MgCO_3$ и $CaCO_3$. На основе экспериментальных данных определены оптимальные составы бетона (жесткость 35 сек), включающие добавки сульфитно-спиртовой барды (ССБ) и полиакриламида (ПАА) в количестве 0,2% от массы цемента. Это привело к увеличению прочности на сжатие у обычных бетонов на 24–33%, у лёгких — на 1,5–11%. Научная новизна. Разработана новая полимерцементная композиция на основе отходов от распиловки известняка-ракушечника. Раствор полиакриламида (0,15%) используется в сочетании с белым цементом (7–8%), наполнителем (68–73%), уайт-спиритом (0,5–1,0%) и ускорителем твердения (0,7–1,2%). Практическая ценность. Предложенный состав позволяет эффективно перерабатывать отходы горных работ, получая экологически безопасные и прочные строительные материалы. Разработка может быть внедрена на предприятиях строительной индустрии, снижая экологическую нагрузку и затраты на сырьё.

Ключевые слова: известняк-ракушечник, полимерцементная композиция, пробоподготовка, термограмма, дериватографический анализ, термогравиметрия

Introduction. Creation of polymer-cement compositions from waste saw limestone as fillers in concrete were first put forward by the authors (Duran-Herrera, et al., 2019; Busari, et al., 2019; Aruova, et al., 2017; Yessentay, et al., 2021; Alzhanova, et al., 2016), they proved that using low-strength limestone-shell rock, it is possible to obtain, at normal rates of cement, structural concrete strength to 300 kg/cm^2 and volume weight lower than heavy concretes. Using limestone with a strength of 50-100 kg/cm^2 in a piece can be obtained concrete grades “15”-”30”, the consumption of cement 120-160 kg/m^3. Such material, with its high-performance qualities, is quite suitable for the construction of monolithic walls of buildings with a number of floors from one to three.

Taking into account that most of developed fields of saw-toothed limestone give stones of low durability, the new method of usage of wastes of low durability limestone which allows to receive a wall material which durability considerably exceeds initial durability of an aggregate (Bukayev, et al., 2020) is developed. Mangyshlak limestone-shell rock meets the requirements of GOST 9479-2011 as a rock for obtaining high-quality sawn products (GOST 9479-2011, 2021).

Materials and methods. In purpose of determining the qualitative and quantitative composition of limestone-shell rock sample at Site-3 in the area of Zhetybai field, used in the work, was carried out derivatographic analysis (DTA). The method is based on registration by the device changes in the thermochemical

and physical parameters of the substance, which may be caused by its heating. Derivatographic analysis (DTA) of limestone-shell rock was carried out on an upgraded installation “Derivatograph Q-1500D” by IOM, which combined the various options of thermal analysis, such as DTA (differential thermal analysis), TG (thermogravimetric) and DTG (differential thermogravimetric) for two hours.

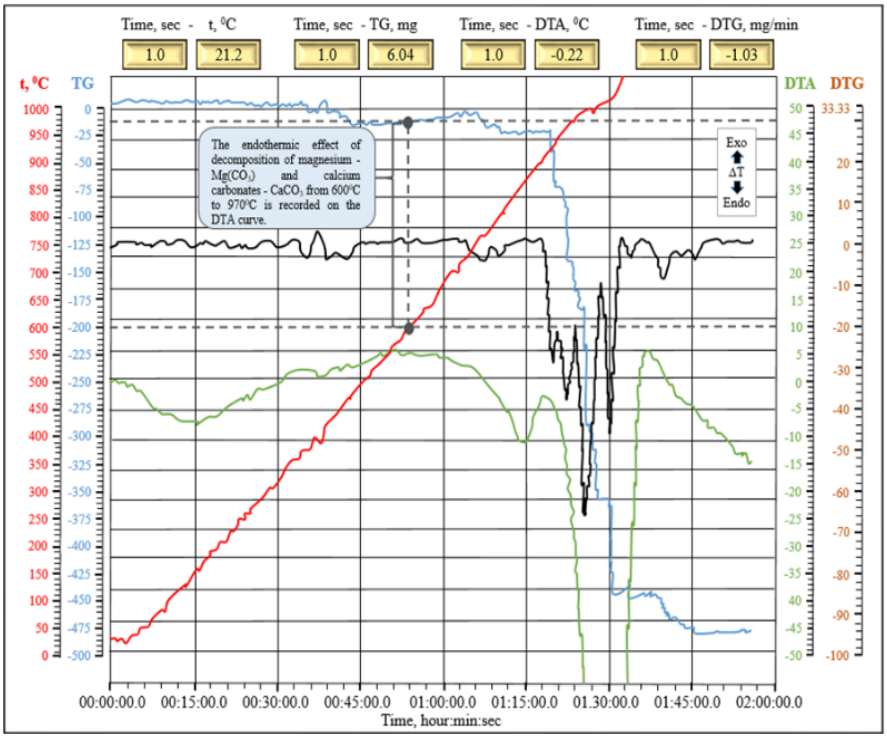

Fig. 1. - Derivatographic analysis (DTA) of a sample of limestone-shell rock of Zhetybai fields

For the analysis, the sample preparation was as follows:

- The test substance was ground in an agate mortar to a powder state;
- a sample of the substance under study was poured into the crucible;
- taking thermograms was carried out up to 1000^{0}C with the heating rate of 10^{0}C per minute.

Al_2O_3 powder was used as a reference in thermocouple. The thermograms were taken up to 1000^{0}C with the heating rate of 10^{0}C per minute.

Figure 1 shows the results of derivatographic analysis of limestone-shell rock sample.

Results. According to the results of derivatographic analysis of limestone-shell rock sample on the DTA curve recorded endothermic decomposition of magnesium carbonates $MgCO_3$ and calcium $CaCO_3$ from 600^{0}C to 970^{0}C.

The thermogram’s the calcination loss rate (CLR) is 43.74 %.

Figure 2 shows a graph of thermogravimetry of limestone-shell rock sample.

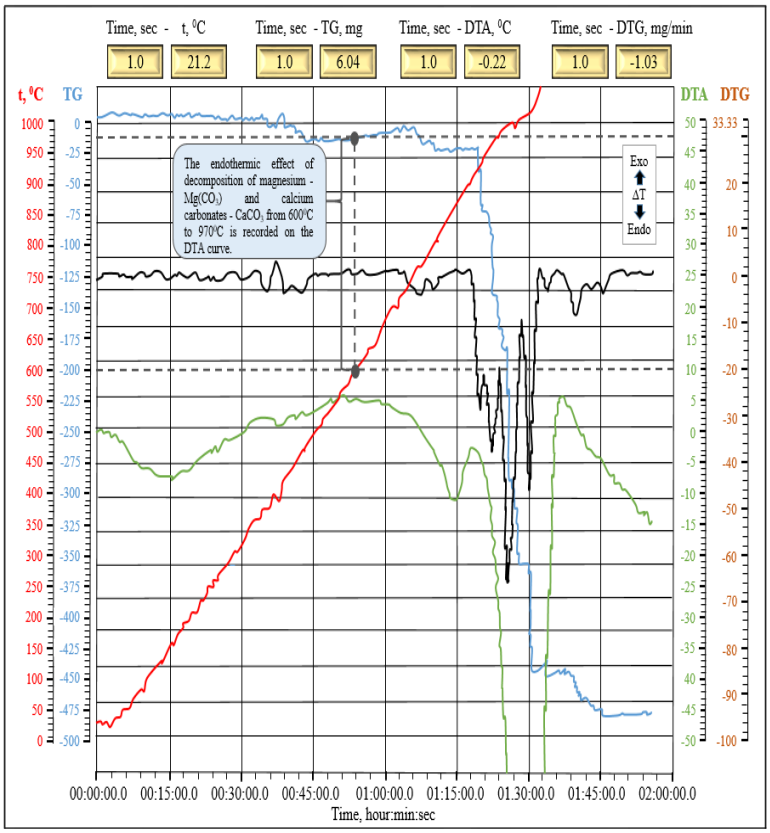

Fig. 2. - Thermogravimetry of limestone-shell rock sample

Identification of components of powder samples was carried out by morphologies of thermal curves and numerical values of intensities of endo- and exothermic effects using coupled with them thermogravimetric indications of TG-lines.

On the basis of the curve TG it is possible to judge how the mass of the sample of limestone-shell rock of Zhetybai fields changed during heating. By removing the difficulty of assessing the TG curve of the limestone-shell rock sample is the following method of differential thermal analysis (DTA).

The component composition of the sample was determined by differentiating its thermochemical readings of their belonging to any known (by reference data) formations.

Deciphering of thermal analysis diagrams, mineralogical interpretation of their curves and explanation of the thermal behavior of the studied model systems were carried out using the following literature (Wagner, 2018; Aimbetova, et al., 2021; Shambilova, et al., 2015).

In the interval from 750^{0}C to 720^{0}C (time - 8 min) begins intensive development of endothermic process, then in the interval from 720^{0}C to 740^{0}C (time - 5 min) shows the transition to exothermia. At temperature from 740^{0}C to 725^{0}C (time -

3 min) endothermic process is manifested, further in the interval from 725^{0}C to 740^{0}C (time - 10 min) exothermic process is manifested.

At a temperature of 590^{0}C to 470^{0}C (time - 10 min) endothermic process is manifested, further in the interval from 470^{0}C to 600^{0}C (time - 4 min) intensive development of exothermic process is manifested. By time increase at 600^{0}C to 260^{0}C (time - 5 min) clearly pronounced endothermic process, further in the interval from 260^{0}C to 680^{0}C (time - 10 min) clearly pronounced exothermic process is manifested. In the interval from 680^{0}C to 410^{0}C (time - 5 min) an intensive development of endothermic process starts, further in the interval from 410^{0}C to 765^{0}C (time - 5 min) an obviously expressed exothermic process. At temperature from 765^{0}C to 720^{0}C (time - 4 min) development of endothermic process, further in the interval 720^{0}C to 765^{0}C (time - 7 min) development of exothermic process. At temperature from 765^{0}C to 680^{0}C (time - 9 min) the endothermic process appears, then in the interval from 680^{0}C to 755^{0}C (time - 10 min) the exothermic process appears. As the time increases at 755^{0}C (time - 9 min) DTG curve goes straight.

DTA in differential thermal analysis DTA curve of limestone-shell rock sample, the introduction of heat into the system at the initial stages of endothermic and exothermic process proceeds smoothly. In the interval from 500^{0}C to 440^{0}C (time - 13 min) shows the smooth development of endothermic process, then in the interval from 440^{0}C to 545^{0}C (time - 39 min) shows the smooth development of exothermic process. At rise in time from 545^{0}C to 400^{0}C (time - 22 min) an intensive development of endothermic process, further in a range from 400^{0}C to 470^{0}C (time - 6 min) an intensive development of exothermic process and at temperature from 470^{0}C to 0^{0}C (time - 7 min) an obviously expressed endothermic process is shown. At increasing time from 0^{0}C to 555^{0}C (time - 5 min) an intensive development of exothermic process, further in the interval from 555^{0}C to 470^{0}C (time - 6 min) an intensive development of exothermic process and at temperature from 470^{0}C to 360^{0}C (time - 18 min) an evident endothermic process is manifested.

The entire process of heat energy release is caused by the oxidation of magnesium and calcium carbonates. Thereby, according to the results of thermal analysis diagrams on the curve recorded endothermic decomposition of magnesium and calcium carbonates from 600^{0}C to 970^{0}C.

X-ray phase analysis (XRF). X-ray phase analysis of limestone-shell rock sample was carried out on an upgraded diffractometer DRON-3M on CuK α-radiation with software.

The sample preparation was carried out according to the following stages:

- the substance under study was grinded in an agate mortar to a powder, then the powder was poured into a Plexiglas cuvette pre-lubricated with vaseline and slightly pressed.

- to eliminate the texture, the excess powder was cut off with a blade.

Table 1 shows the parameters set for the X-ray phase analysis of the sample of limestone-shell rock of Zhetybai fields.

Table 1 - Parameters set for X-ray phase analysis of the limestone-shell sample from Zhetybai field

Parameters set for X-ray phase analysis	
Range of shooting angles	Start angle 100
	Final angle 700
Scanning step	0.05
Scanning speed	2 degree/min
Exposition	1.5
Maximum pulse number	2125

Figure 3 shows the X-ray phase analysis of a sample of limestone-shell rock.

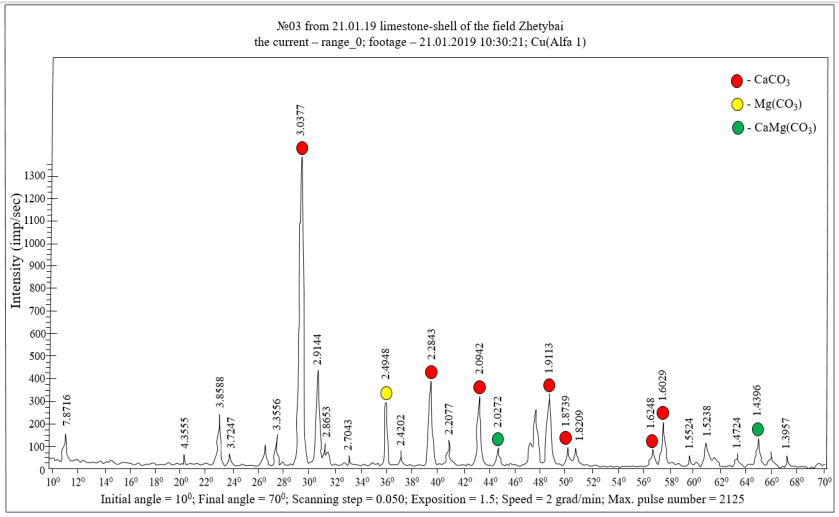

Fig. 3. - X-ray phase analysis of the sample limestone-shell of the Zhetybai field

Designing the composition of concrete mixtures is based on the basic parameters of strength, stiffness and curing regimes. When designing the composition of concrete based on the main parameters: the stiffness of the concrete mix, assigned in accordance with the characteristics of the product forming a concrete mix of the mechanism and the achievement of the required strength of concrete with the curing regime.

On the basis of experiments for the optimal composition of concrete (at 35 sec stiffness) on the fillers from the limestone fields studied the recommended cement consumption and water-cement ratio (W/C) for concretes of various brands to facilitate the composition of concrete on the filler from Zhetybai limestone field (waste rock sawing from it is currently used in experimental construction) schedules were made, which depend on the specified hardness of the concrete mixture and the strength of the limestone concrete, you can determine the consumption of cement (kg/m^3) composition of the concrete mixture by weight to set its water-cement ratio (figure 4).

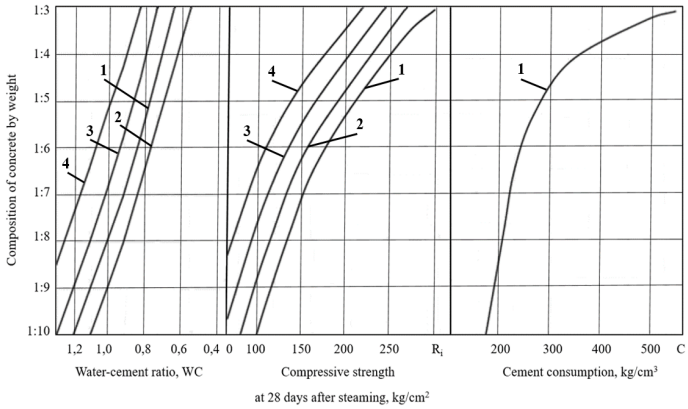

Fig. 4. - Selection of compositions of limestone concrete on filler of Zhetybai field
1 - stiffness 30-40 sec; 2 - stiffness 60-80 sec; 3 - stiffness 20-10 sec; 4 - standard cone settling 0-8 cm.

Discussion. When designing compositions of limestone concretes in the scheduling it is necessary to consider the basic properties and characteristics of the concrete mixture and concrete on the limestone filler (the dependence between stiffness and water content of the concrete mixture, the rule of constant water content, the dependence between the strength of the filler and the strength of the concrete). Rather high strength of concrete allows you to make from it a wide range of building products: road slabs, partition boards, curbs, pedestrian slabs, etc.

Another direction is the use of limestone as filler for concrete. Replacement of dense and strong crushed stone (gravel) in concrete with local limestone allows you to get concrete with less bulk weight and increased thermal and sound insulation qualities.

Development of new technologies for the production of cement stone on the basis of waste wall stone made with various mineral additives.

The bonding strength of cement stone with ground mineral plates of different chemical composition for limestone is several times higher than for granite, labrador, quartz, etc. The study of processes taking place in the contact zone of tuff and peplopemza fillers with cement stone under a microscope showed that the structure-forming role of porous filler is manifested in the formation of mechanical bonding with the hardening cement dough (binder) by filling the surface irregularities of grains, products of its hydration in the pores of the filler and in the change of cementitious mass density as a result of absorption of the liquid phase from it by porous filler with a decrease in the water-cement ratio.

For concrete with a strength of 7.5 to 15 MPa, used for massive structures, foundations, retaining walls, the maximum grain size of natural porous fillers, with appropriate justification can be increased up to 40 mm or more. Experiments have shown that with the introduction of PAA the stiffness of the concrete mixture on the filler of porous limestone is significantly reduced. Thus, at addition of 0.2% PAA it

decreases more than 2 times as compared with the stiffness of the mixture without additive (at s = 40s).

The introduction of polyacrylamide PAA can increase the strength of concrete up to 20% (table 2). The increase in mobility of concrete mixture at a dosage of PAA 0.2% of the weight of cement reaches 35-40%.

Table 2 – The effect of PAA additive on concrete strength and change in cement consumption: a) C – cement; b) S – sand; c) G – gravel (crushed stone); d) W - water

Расход материалов, кг, на 1 м^3 бетона				PAA, % by mass of cement	Concrete strength at the age of 28 days, MPa	Cement savings, %
C	*S*	*G*	*W*			
1	692	1014	230	0	27,3	0
2	706	1034	217	0,1	31,2	5,65
3	711	1045	206	0,15	27,9	10,17
4	714	1046	209	0,2	30,3	9,9

The nearest to the invention in terms of technical essence and the achieved result is polymer-cement composition including the following composition in mass %: white cement 17-20, ground silica sand 10-12, butadiene styrene latex 5.5-6.0, pyrite cinders 1.7-2.0, ground clay brick 54-61 and water 2 (A.S. 1004304. USSR., 1983).

The main disadvantage of this composition is the low strength of the finished products at a significant consumption of cement and polymer.

The purpose of the present invention is to reduce the consumption of cement and improve the quality and physical and mechanical properties of the product.

Table 3 - Contains of polymer-cement composition

Source material	Mass, %
White cement	7–8
The mentioned filler	68–73
The mentioned water solution of polymer	15–18
White spirit	0.5–1.0
The mentioned curing accelerator	0.7–1.2

The purpose is achieved by the fact that polymer-cement composition including white cement, filler and aqueous polymer solution contains additionally white spirit and hardening gas pedal - duralumin solution in hydrochloric acid in the ratio 1:10, diluted with water to 1% concentration. As a filler - ground limestone sift of shell rock, and as an aqueous polymer solution - 0.15% solution of polyacrylamide at the following ratio of components, mass % (table 3) (Patent №4370 RK, 2019).

Conclusion.

1. It is established that in the time interval from 0^0C to 555^0C (time - 5 min) the intensive development of exothermic process, further in the interval from 555^0C to 470^0C (time - 6 min) the intensive development of exothermic process and at 470^0C

to 360^0C (time - 18 min) the clearly pronounced endothermic process, therefore, the whole process of heat energy release is caused by oxidation of magnesium $Mg(CO_3)$ and calcium carbonates $CaCO_3$. Thereby, according to the results of thermal analysis diagrams, endothermic effect of decomposition of magnesium $Mg(CO_3)$ and calcium carbonates $CaCO_3$ from 600^0C to 970^0C is fixed on the curve;

2. According to the results of processing of experimental data of limestone-shell rock sample, eight peaks of calcite $CaCO_3$, two peaks of dolomite $CaMg(CO_3)$ and one peak of magnesite $Mg(CO_3)$, which indicates a large amount of limestone-shell rock carbonate calcite $CaCO_3$;

On the basis of experiments for the optimum composition of concrete (at 35 sec stiffness) on fillers from the limestone fields studied, given the recommended cement consumption and water-cement ratio (W/C) for concrete grades 100, 150, 200, 250, 300 and 400;

3. Introduction of optimal amounts of sulfite-alcoholic bard (SAB) and polyacrylamide (PAA) additives in conventional and lightweight concretes (0.2% of cement weight) leads to an increase in the compressive strength limit for conventional concretes from 24 to 33% and for lightweight - from 1.5 to 11%;

4. A new polymer-cement composition for the manufacture of wall blocks from sawtooth rock mining wastes from limestone-shell rock: and as an aqueous polymer solution - 0.15% solution of polyacrylamide with the following ratio of components, mass. % cement white - 7-8%, filler - 68-73%, aqueous polymer solution - 15-18, white spirit – 0.5-1.0, hardening gas pedal - duralumin solution in hydrochloric acid – 0.7-1.2.

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