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Переработка отходов кремнегеля и известняка в иракский экологически безопасный («зеленый») бетон и сравнение с микро- и нанокремнеземом

Работа связана с использованием материалов для «зеленого» (экологически безопасного) бетона. Она представляет целесообразность использования побочных материалов типа порошков кремнегеля и известняка в качестве частичной замены цемента. Порошки вводились в четырех концентрациях: 1, 2, 3 и 4 мас. % цементирующего материала в бетонную смесь. Экспериментальные исследования модифицированного бетона были проведены через 28 сут выдерживания бетона во влажных условиях (водное твердение бетона) для определения механических свойств, таких как прочность при сжатии, прочность при изгибе и прочность на растяжение при разрыве. Также было проведено тестирование на водопоглощение для получения свойств стойкости бетонных образцов. Бинарное сочетание кремнегеля и известняка также рассматривалось для изучения комбинированного действия переработанных порошков. Эффект добавок был очевиден в улучшении механических свойств и прочности бетона. Также было проведено сравнение пуццолановой активности среди нанокремнезема, микрокремнезема и кремнегеля, при этом смеси с отходами кремнегеля показали сопоставимый показатель прочности.

Ключевые слова: «зеленый» (экологически безопасный) бетон, переработка материала, известняк в бетоне, прочность бетона.

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Recycling of Waste Silica Gel And Limestone in Iraqi Green Concrete and Comparisons with Micro and Nanosilica

The paper covers the aspect on using material for green concrete. It presents the feasibility of the usage of by product materials like silica gel and limestone powder as partial replacement of cement. powders were added in four different dosages of 1, 2, 3 and 4% of weight of the cementitious material into the concrete mixture. Experimental investigations on modified concrete were conducted after 28 days of water curing to obtain the mechanical properties such as compressive strength, flexural strength and split tensile strength of specimen. Also, water absorption test was investigated for obtaining the durability properties of concrete specimen. Binary combination of silica gel and limestone also considered to study the combined effect of the recycled powders. The effect of additives was obvious by enhancement the mechanical properties and durability of concrete. Also pozzolanic activity comparison was made among nanosilica, micro silica and silica gel in which the mixes with waste silica gel showed comparable strength index.

Keywords: green concrete, recycling material, limestone in concrete, durability of concrete, nanotechnology, nanosilica, micro silica.

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Concrete is one of the most widely used man-made building materials in the world. Compared to other building material concrete has numerous advantages such as abundant resources, easy operation, steady mechanical properties, durability, of production. The aim of green concrete is to meet three requirements i. e. very low energy and resource consumption, no environmental pollution and sustainable development [1]. It has been observed that 0.9 tons of CO_2 is produced per ton of cement production. Also, the composition of cement is 10% by weight in a cubic yard of concrete. Thus, by the use of green concrete it is possible to reduce the CO_2 emission in atmosphere towards eco-friendly construction technique [2].

The paper covers the aspect on how to choose a material for green concrete. It presents the feasibility of the usage of by product materials like limestone and silica gel as replacement of cement in concrete.

Qing Ye et al. have been studied the pozzolanic activity of nano silica and micro silica with content 3% of the weight of cement. Results indicate that the bond strength at the interface between aggregate and hardened cement paste, the compressive strength and the bonding strength of concrete incorporated with 3% nanosilica increased more than those with silica fume. The pozzolanic activity of nanosilica was much greater than that of microsilica [3]. Camiletti J. et al. [4] shown that $CaCO_3$ accelerates the hydration process by acting as a nucleation site on which cement hydration product form. This micro-physical influence leads to higher improvement rate of mechanical properties, the gain in strength can be due to the formation of calcium silicate hydrate CSH gel which is stronger than the normal calcium hydrate CH gel. Bentz Dale P. et al. [5] examined the performance of limestone in cement-based materials at multiple scales and found that physical and chemical interaction of limestone with the cement hydrates also likely contributes to the superior mechanical properties of concretes containing limestone aggregates in comparison to similar ones based on siliceous aggregates. Yusuf M.O. et al. [6] studied the performance of infused nano-SiO_2 gel (NSG) on the developed alkaline activated binary blending of ground blast furnace slag and ultrafine palm oil fuel ash based mortar. The finding revealed that the compressive strength of the mortar increased with NSG and the maximum strength achieved was 57.3 MPa

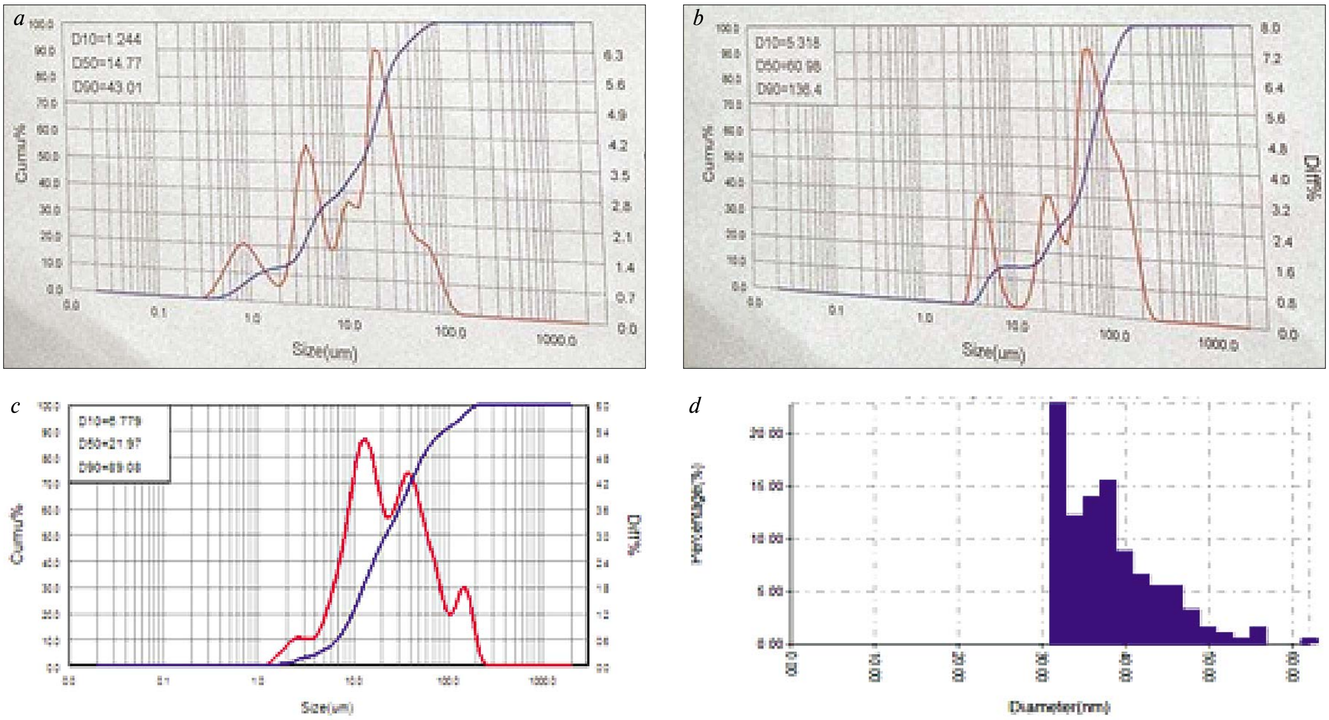

Рис. 1. Распределение частиц: *a* – микроизвестняка; *b* – кремнегеля; *c* – микрокремнезема; *d* – распределение по размерам частиц нанокремнезема
Fig. 1. Particle size analysis: *a* – of micro limestone; *b* – of silica gel; *c* – of micro silica; *d* – AFM analysis of nano silica

signifying 21% strength gain compared to NSG-free sample. The NSG also played the role of micro cracks and pore fillings, increased the product homogeneity and reduced its amorphousity due to silicate re-organization.

Исследования / Experimental

Commercially available ordinary Portland cement Type I, named Karasta, is used in this study. Chemical and physical properties of cement, which are indicated that the cement is conformed to Iraqi specifications (I.Q.S.) No. 5/1984 [7], are shown in Table 1 and Table 2 respectively. Natural sand (from Al-Akhedher in Karbala) was used. Table 3 shows the grading of the fine aggregate and sulfate content according to the limits of the Iraqi Specification No. 45/1984 [8]. To obtained (HSC) crushed gravel used throughout this work of maximum size 14 mm. Table 4 shows the grading of the fine aggregate and sulfate content according to the limits of the Iraqi Specification No. 45/1984 [8].

Physical and chemical properties using materials are shown in Table 5. Silica gel (waste material used as desiccator for saving goods purchased from Fluka Company is used as admixture. Limestone powder from debris of building). Micro silica is used as pozzolanic admixture.

Nano silica is used as concrete admixture in this research Table 5 which includes as received properties of nano silica. Fig. 1 gives the particles analysis for both silica gel and limestone powders, Fig. 2 shows the XRD spectra. Superplasticizer Glenium 54 (G54) high range water reducing admixture, purchased form BASF Company, is used as workability adjusting material for concrete mixtures. Water Tap water is used for mixing and curing of all concrete mixes and specimens.

Таблица 1
Table 1

Химический состав цемента
Chemical analysis of the cement test

Oxide / Оксиды	%
CaO	66.11
SiO_2	21.93
Al_2O_3	4.98
Fe_2O_3	3.10
MgO	2.0
K_2O	0.75
Na_2O	0.35
SO_3	2.25
Loss On Ignition (L.O.I)	2.39
Lime Saturation Factor (L.S.F)	0.93
Insoluble residue (I.R)	1.29
Free lime (F.L)	0.67
Compound Composition / Минералогический состав	%
C_3S	50
C_2S	20.48
C_3A	4
C_4AF	13.17

Технологический процесс / Procedure

Mix proportion. Mortar mixes details for nano silica (NS), micro silica (MS), silica gel (SG) and silica gel crushed (SGC) pozzolanic activity investigation, are shown in Table 6. The proportion of admixture is 10%, as replacement of cement weight was depended for this test as in ASTM C 1240 [9] for testing pozzolanic activity index of silica fume. In absorption specimen's mixes, preparation the target design

Таблица 2
Table 2

Физические свойства цемента
Physical Properties of Cement

Physical Properties / Физические свойства	Test Results / Значение
Fineness, Blaine, cm^2/gm	3300
Setting Time:	
Initial hrs.; min	2;05
Final hrs.; min	4;00
Compressive Strength, MPa	
3-days	20
7-days	25

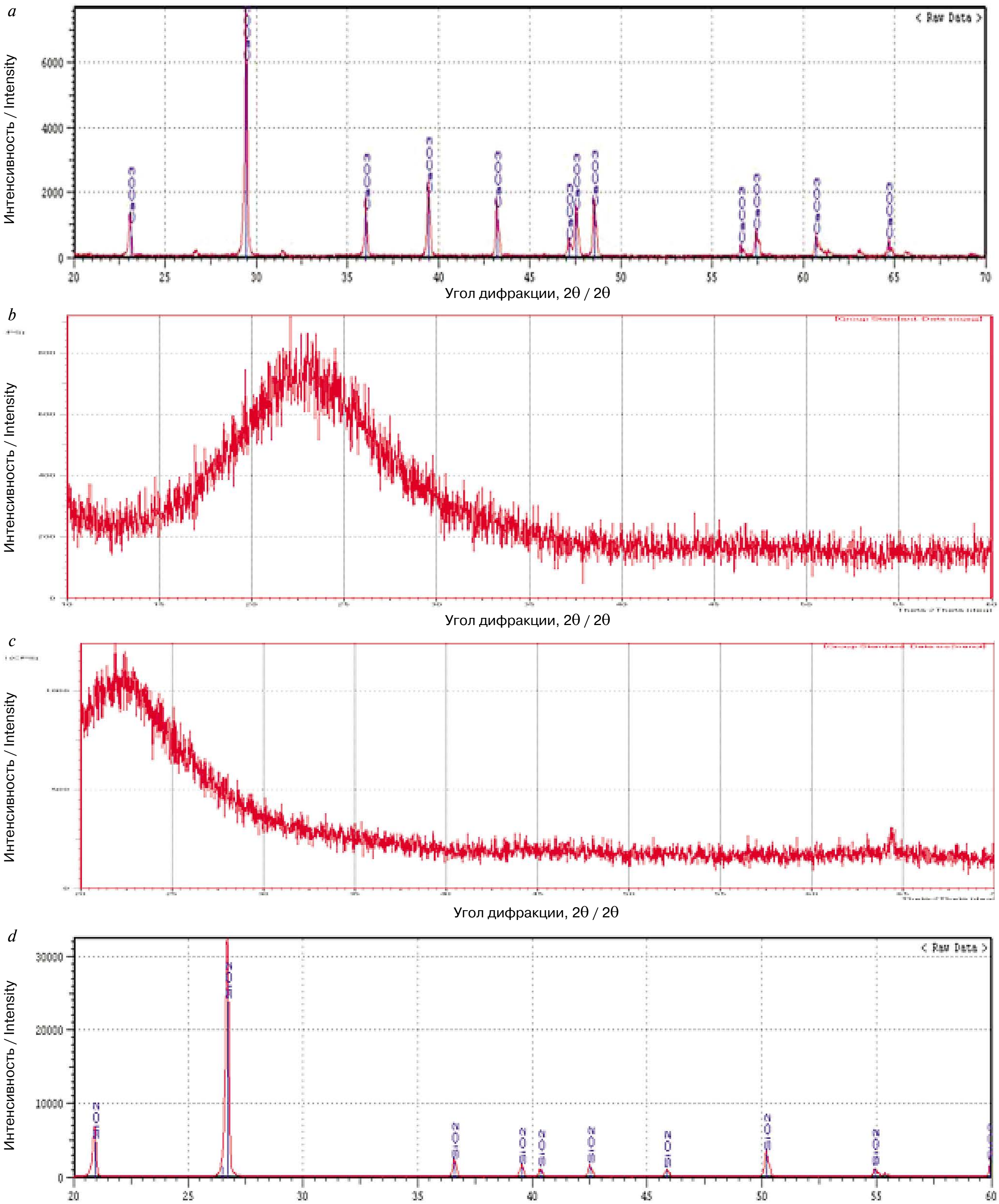

Рис. 2. Дифрактограммы: *a* – порошка известняка; *b* – порошка кремнегеля; *c* – порошка микрокремнезема; *d* – нанокремнезема
Fig. 2. XRD spectra of: *a* – limestone powder; *b* – crushed silica gel powder; *c* – nano silica powder; *d* – micro silica

strength of 50, 70 MPa were designed according to British mix design method BS5328. Part 2:1991 [10], seventeen types of concrete mixes is implemented in this study. The fixed parameters for all mixes are: water/cementitious, coarse and fine aggregate fractions, and superplasticizer contents. Mixes details and symbols can be seen in Table 7.

Испытания / Tests

Compressive strength test. This test is carried out according to BS 1881-Part 116 [11]. The studied age is 28 days. Three cubes are made for each mix at the specified age.

Split tensile strength test. This test is carried out according to ASTM C 496/C 496M – 04 [12]. The studied age is

Таблица 3
Table 3

Ситовой анализ и содержание сульфатов в мелком заполнителе
Sieve analysis and sulfate content of fine aggregate

Размер ячейки сита, мм / Sieve opening (mm)	Остаток на сите, %/ Accumulative passing, %
10	100
4.75	94
2.36	85.6
1.18	76.9
0.60	46.3
0.3	10.8
0.15	1.1
0.075	0.5
SO_3	0.4

Таблица 4
Table 4

Ситовой анализ и содержание сульфатов в гравии
Sieve analysis and sulfate content of gravel

Размер ячейки сита, мм / Sieve opening (mm)	Остаток на сите, %/ Accumulative passing, %
14	97
10	62
5	10
0.075	0.037
SO_3	0.09

Таблица 5
Table 5

Свойства материалов / Material properties

Свойство / Property	Значение или описание / Value or description
Кремнегель / Silica gel	
Material structure	Transparent beads
Color	Violet
M.Wieght	60.08 g/mol
Particle size	≈2.5–4 mm
Abs. capacity	30% of its weight
Source	Fluka company – Switzerland
Известняк / Limestone	
Material structure	micro $CaCO_3$
Color	Light brown
Particle size	100–200 µm
Микрокремнезем / Micro silica	
Material structure	Densified micro silica
Color	Gray
Density	1300 kg/m^3
M. Weight	60.08 g/mol
Particle size	≈40 µm
Нанокремнезем / Nano silica	
Material structure	Hydrophilic Water-soluble SiO_2
Color	White
Density	1300 kg/m^3
Purity	99.8%
Particle size**	30–60 nm
Source	Hwnanomaterial china

Таблица 6
Table 6

Соотношение ингредиентов в бетонной смеси / Mortar mixes proportion for pozzolanic activity index test

Обозначение / Mix symbol	Цемент, г / Cement, g	Песок, г / Sand, g	Нанокремнезем, г / NS, g	Микрокремнезем, г / MS, g	Кремнегель, г / SG, g	Кремнегель измельченный, г / SGC, g	В/Ц, % W/B, %	Суперпластификатор G54/ цемент, % / Superplasticizer G54/cement, %	Подвижность, мм / Flow, mm
Control	500	1375				–	0.485	0.5	160
NS	450	1375	50	–	–	–	0.485	0.75	153
MS	450	1375	–	50	–	–	0.485	0.5	155
SG	450	1375	–	–	50	–	0.485	0.6	160
SGC	450	1375	–	–	–	50	0.485	0.7	157

28 days. Three cylinders are made for each mix at the specified age. The splitting tensile strength of the specimen calculated as follows:

$$\sigma_{sp} = 2P/\pi dL, \qquad (1)$$

where: σ_{sp} – splitting tensile strength, MPa; P – maximum applied load indicated by the testing machine, N; L – length, mm; d = diameter, mm, [12].

Flexural strength. This test is carried out according to ASTM C293-02 [13]. Duplicate beam specimens were tested and the average results were considered. The flexural strength is then calculated by using equation:

$$\sigma_{fl} = 3PL/2bd^2, \qquad (2)$$

where: σ_{fl} – flexural strength, MPa; P – maximum applied load, N; L –span length, mm; d – depth of the specimens, mm; b – width of the specimens, mm [13].

Water absorption test. The water absorption was conducted according to ASTM C642 [14], the water absorption test is carried out using (50×100 mm) cylinder specimens, and the average water absorption of two samples was recorded and considered.

$$\text{Absorption after immersion, }\% = \frac{B-A}{A}\times 100, \qquad (3)$$

where: A – mass of oven-/dried sample in air, gm; B – mass of saturated surface-dry sample in air after immersion, gm.

Pozzolanic activity index test. For testing pozzolanic activity index mortar cubes of 50 mm side length are used for

Таблица 7
Table 7

Mixes symbols, content and quantity

Обозначение / Mix symbol	Цемент, кг/м^3 / Cement, kg/m^3	Песок, кг/м^3 / Sand, kg/m^3	Гравий, кг/м^3 / Gravel, kg/m^3	В/Ц, % W/B, %	G54 kg/m^3	L, kg/m^3 (rep. %)	SG, kg/m^3 (rep. %)	SGC, kg/m^3 (rep. %)
Control	515	721	1030	0.32	6.43	–	–	–
1L	509.85	721	1030	0.32	6.43	5.15(1%)		
2L	504.7	721	1030	0.32	6.43	10.3(2%)		
3L	499.55	721	1030	0.32	6.43	15.45(3%)		
4L	494.4	721	1030	0.32	6.43	20.6(4%)		
1SG	509.85	721	1030	0.32	6.43		5.15(1%)	
2SG	504.7	721	1030	0.32	6.43		10.3(2%)	
3SG	499.55	721	1030	0.32	6.43		15.45(3%)	
4SG	494.4	721	1030	0.32	6.43		20.6(4%)	
1SGC	509.85	721	1030	0.32	6.43			5.15(1%)
2SGC	504.7	721	1030	0.32	6.43			10.3(2%)
3SGC	499.55	721	1030	0.32	6.43			15.45(3%)
4SGC	494.4	721	1030	0.32	6.43			20.6(4%)
1(L+SGC)	509.85	721	1030	0.32	6.43	2.575(0.5%)		2.575(0.5%)
2(L+SGC)	504.7	721	1030	0.32	6.43	5.15(1%)		5.15(1%)
3(L+SGC)	499.55	721	1030	0.32	6.43	7.725(1.5%)		7.725(1.5%)
4(L+SGC)	494.4	721	1030	0.32	6.43	10.3(2%)		10.3(2%)

Примечание. SG – бетонные смеси с шариками из кремнегеля; SGC – бетонные смеси с порошками измельченного кремнегеля; L – бетонные смеси с известняком.
Note. SG – concrete mixes with silica gel beads; SGC – concrete mixes with crushed silica gel powder; L – concrete mixes with limestone.

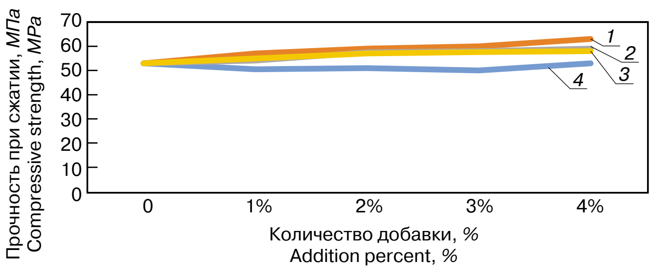

Рис. 3. Результаты испытаний прочности при сжатии: *1* – измельченный кремнегель; *2* – измельченный кремнегель+известняк; *3* – кремнегель шарики; *4* – известняк
Fig. 3. The results of compressive strength: *1* – crushed silica gel; *2* – crushed silica gel+limestone; *3* – silica gel beads; *4* – limestone

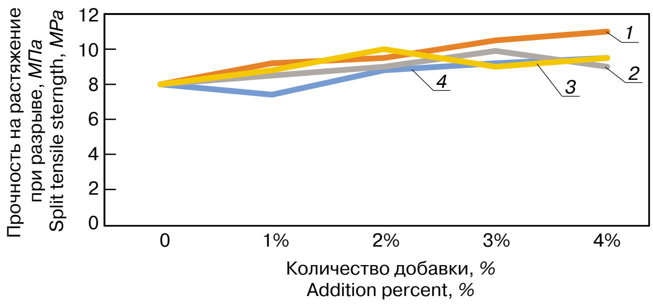

Рис. 4. Результаты испытаний прочности на растяжение при разрыве: *1* – измельченный кремнегель; *2* – измельченный кремнегель+известняк; *3* – кремнегель шарики; *4* – известняк
Fig. 4. The results of splitting tensile strength: *1* – crushed silica gel; *2* – crushed silica gel+limestone; *3* – silica gel beads; *4* – limestone

this test. One proportions of nano silica, micro silica, silica gel and crushed silica gel 10%, as replacement of cement weight, are depended for this test. The ratio is as used in ASTM C 1240 [9] for testing pozzolanic activity index of silica fume. A Pozzolanic activity index can be determined using the following equation [9]:

$$\text{Strength Activity Index} = A_p / B_p \times 100,$$

where: A_p – average compressive strength of mortar cubes of mixes incorporated admixture; B_p – average compressive strength of mortar cubes of without-adding (control) mixes.

Результаты / Results

Results of Mechanical Properties Tests. All mechanical properties showed the same trend, concrete mixes with crushed silica gel give higher enhancement while mixes with limestone showed the lower effect. In fig. 3 the results of compressive strength test explained that maximum enhancements were 18.8%, 11.3%, 9.4 % and 0% for concrete mixes with crushed silica gel, crushed silica gel+limestone, silica gel beads and limestone respectively. Fig. 4 results of splitting tensile strength maximum enhancement founded are 37.5%,12.5%, 18.7% and 18.7% for concrete mixes with crushed silica gel, crushed silica gel+limestone, silica gel beads and limestone respectively. Fig. 5 the flexural strength showed remarakable enhancement for all mixes compared with compressive strength and splitting tensile strength results where the enhancements

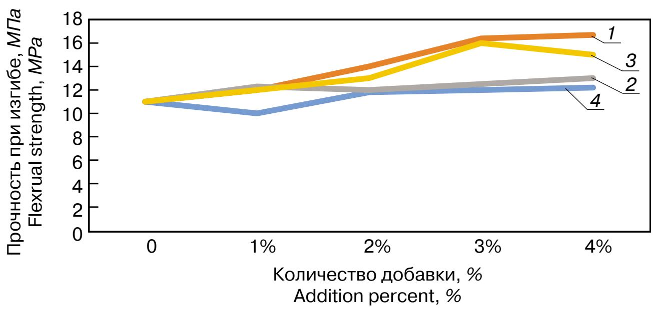

Рис. 5. Результаты испытаний прочности при изгибе: *1* – измельченный кремнегель; *2* – измельченный кремнегель+известняк; *3* – кремнегель шарики; *4* – известняк
Fig. 5. The results of flexural strength: *1* – crushed silica gel; *2* – crushed silica gel+limestone; *3* – silica gel beads; *4* – limestone

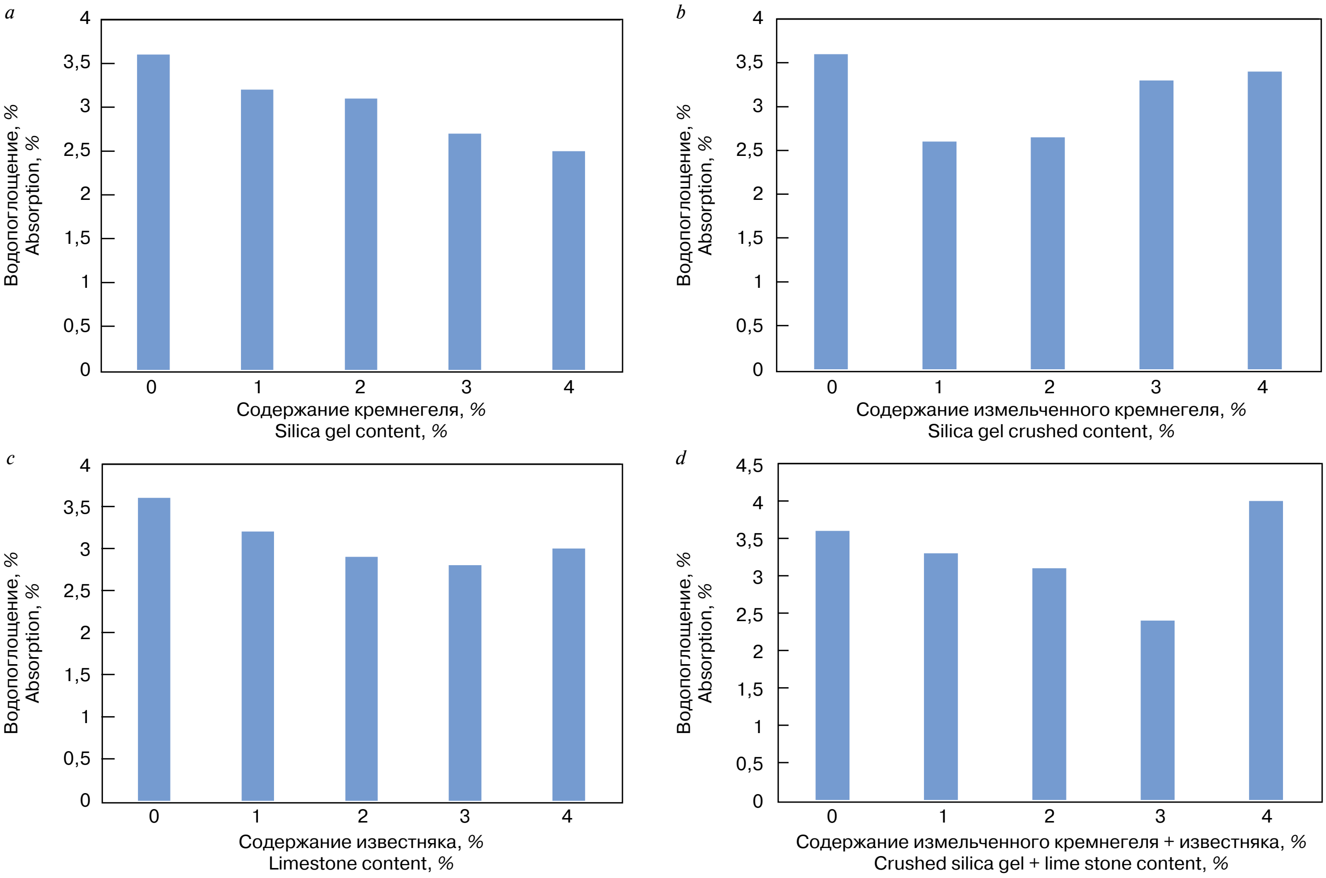

Рис. 6. Результаты испытаний образцов на водопоглощение: *a* – кремнегель; *b* – измельченный кремнегель; *c* – известняк; *d* – измельченный кремнегель+известняк
Fig. 6. The results of water absorption test: *a* – silica gel; *b* – crushed silica gel; *c* – limestone; *d* – crushed silica gel+limestone

were 51.8%, 18.8%, 36.3% and 10.9% for concrete mixes with crushed silica gel, crushed silica gel+limestone, silica gel beads and limestone respectively. The development in mechanical properties in the presence of silica gel can be attributed to the pozzolinic effect of silica when water is added to cement hydration occurs forming two products, CSH gel and CH, in the presence of micro silica, SiO_2 will react with the calcium hydroxide to produce more aggregate binding CSH gel [15]. Studies have shown that $CaCO_3$ accelerates the hydration process by acting as a nucleation site on which cement hydration products form, this micro-physical effect results in a higher enhancement rate of mechanical properties. A higher accelerating effect occurs when more $CaCO_3$ is added, [4, 16] this interpreted the little effect on mechanical properties of concrete when added limestone powder to their mixes.

Results of water absorption test. The results of absorption test showed remarkable improvement by reduction of water absorption for all concrete mixes. As shown in fig. 6, silica gel (beads and crushed powder) implemented in this work generally exhibits a reduction in the water absorption potential of concrete this reduction, recognized to be increased with the content of silica gel (beads and crushed powder) introduced to the mixes. The reduction in water absorption values in silica gel mixes were attributed due to their higher pozzolanic effect which made the concrete more compact and denser than conventional concrete. In mixes with limestone less reduction in water absorption cab be recognized when comprised with silica gel mixes. Moderate enhancement was found in binary mixes due to the dual effect of silica and limestone by accelerate the hydration process and formation additional amount of CSH gel which develop the pore structure and mechanical properties of concrete [17–19].

Pozzolanic activity index test results. The results are reported as averages of 3 replicates. The control mortar strength was 26 MPa, when replaced 10% of cement by nano silica, crushed silica gel, micro silica and silica gel the strength became 47, 42, 40.8 and 36.4 MPa, respectively, so the pozzolnic activity indexes obtained from these results were 181, 161, 157 and 130 respectively as indicated in fig (3). Nano silica showed higher pozzolanic activity because nano silica reacts with the CH produced during cement hydration and results in more strength carrying C–S–H into the paste. As a more pozzolanic reaction occurs in the mix, more strength-carrying C–S–H is produced, which ultimately leads to a higher overall strength [20] these results were agree with Madhuwanthi Rupasinghe et.al 2017[20], and Wengui Li et.al. 2015[44], also YE Qing 2006 [3] found that the pozzolanic activity of nano silica was much greater than that of micro silica. The reaction rate of $Ca(OH)_2$ with nano silica and the velocity of C–S–H gel formation from $Ca(OH)_2$ with nano-SiO_2 were much quicker than that of $Ca(OH)_2$ with micro silica, in this work micro silica showed lower pozzolanic activity by about 13% with respect to that of nano silica. Crushed silica gel showed good pozzolanic activity, it was experimentally confirmed by Daunte Vaičiukynienėa et.al 2012 that the thermally activated silica gel could be used as an additive in hardened cement paste. This amorphous SiO_2 reacted with $Ca(OH)_2$ and form (C–S–H) type calcium silicate hydrates that additionally strengthened the hardened cement paste [21], so this quantity of silica gel additive enables to decrease the quantity of used cement and improved the mechanical properties of concrete. Silica gel also improved the pozzolan reactivity compared to control mix, but not as good as crushed silica gel's improvement.

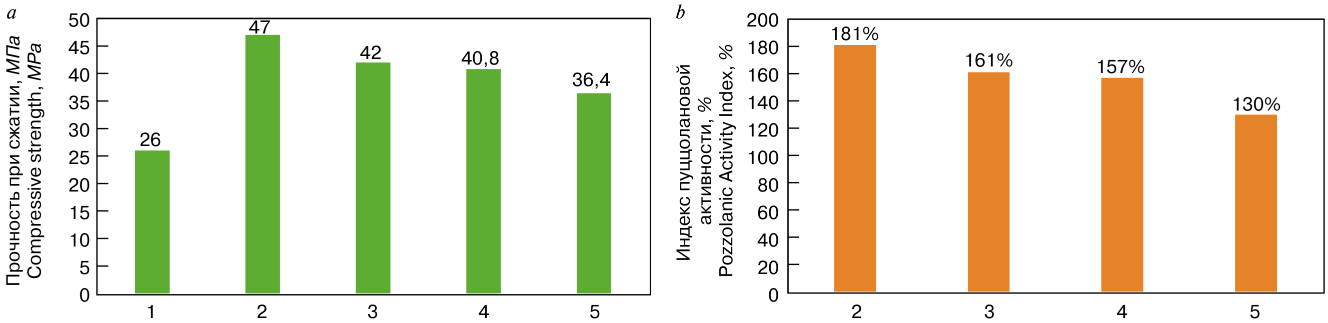

Рис. 7. Результаты испытаний прочности при сжатии для растворов с различными добавками (*a*); индекс пуццолановой активности цементного раствора с различными добавками (*b*); *1* – контрольный; *2* – нанокремнезем; *3* – измельченный кремнегель; *4* – микрокремнезем; *5* – кремнегель
Fig. 7. Results of compressive strength for mortar with different admixtures (*a*); Pozzolanic activity index for cement mortar with different admixtures (*b*). *1* – Control; *2* – nano silica; *3* – crushed silica gel; *4* – micro silica; *5* – silica gel

Выводы / Conclusions

Recycled silica gel showed good improvement in mechanical properties when added to concrete mixes and marked reduction in water absorption.

Pozzolanic activity results showed comparable activity between nano silica and crushed silica gel, but at the same time beads of silica showed low activity that belonged to the effect of surface area factor which increase the reactivity of silica gel after crushing.

Limestone powder didn't show remarkable improvement in the mechanical properties of concrete, this behaviour attributed to small amount of addition.

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