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Agreement number 2023-1-RO01-KA220-HED-000155412

European Network for Additive Manufacturing in Industrial Design for Ukrainian Context

Staff Training (STTE) – Edibon International S.A., Madrid, Spain, 7-10 May 2024

New materials and properties used in architectural design

«*Ultra-high strength composites*»

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AMAZE team from Yuriy Fedkovych Chernivtsi National University

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Yuriy Fedkovych
Chernivtsi National University

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The University was founded on 1875
by decree of Austro-Hungarian emperor Franz Joseph
The main building of the University – the previous Residence of the Orthodox Metropolitans of Bukovyna and Dalmatia – is the pearl of Bukovynian region, designed by the prominent Czech architect Josef Hlavka.

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Ultra-high strength composites are a material of high structural strength and density. They are made in the form of a special dispersed product with a compressive strength of 150 N/mm^2

Ultra-strength concretes, especially those with advanced additives and reinforcement, exhibit exceptional properties such as high strength, durability, and sometimes even self-healing capabilities. While they may not fit the traditional definition of "smart materials" like shape memory alloys or piezoelectric materials, they do possess certain characteristics that could be considered "smart" in the context of civil engineering and construction.

For instance, some ultra-strength concretes incorporate self-healing mechanisms, where cracks that form due to stress or other factors can be automatically repaired over time through chemical reactions within the material. This self-healing ability mimics some aspects of smart materials, which can autonomously respond to changes in their environment.

Furthermore, ultra-strength concretes can be engineered to have specific responses to external stimuli such as temperature variations, moisture levels, or mechanical stress. While these responses might not be as dynamic or sophisticated as those of traditional smart materials, they still demonstrate a degree of adaptability and functionality that aligns with the concept of smart materials

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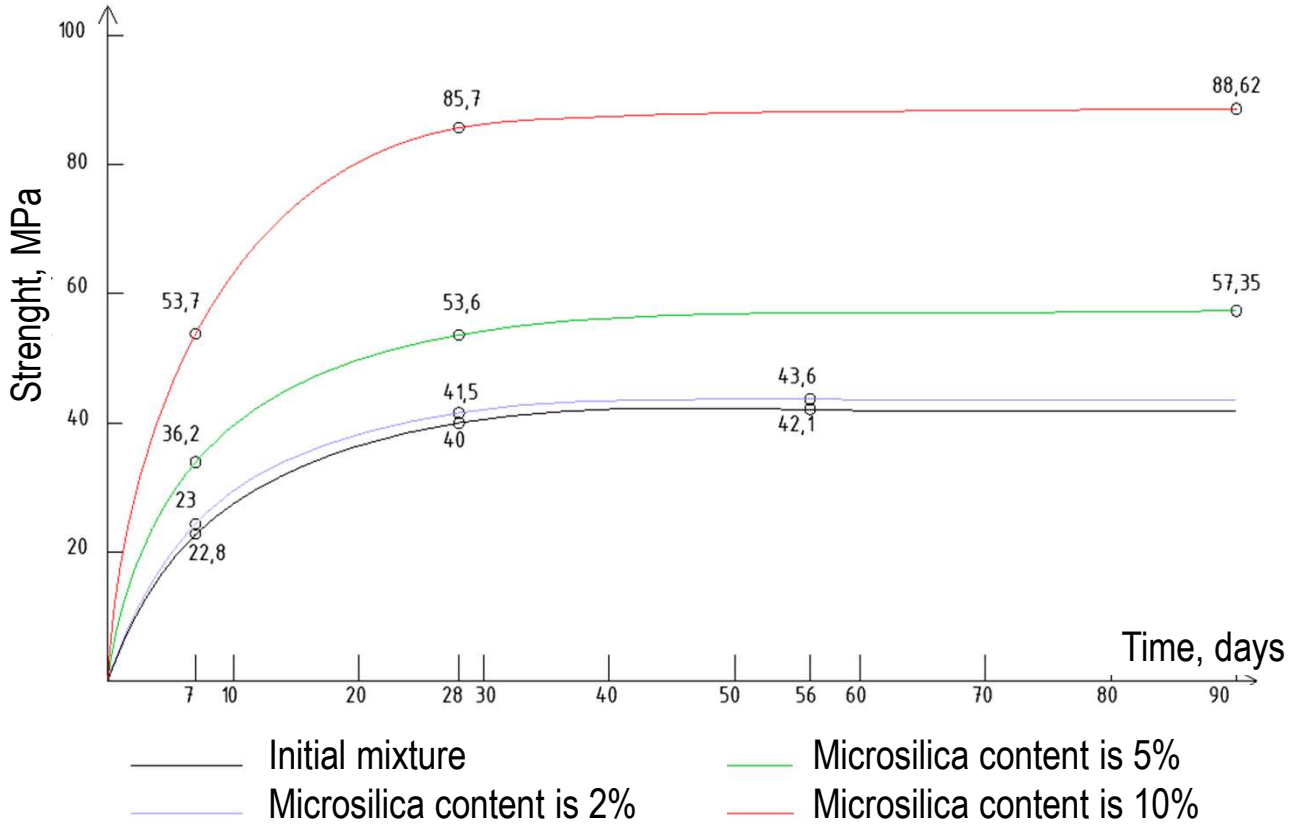
Results of strength testing of concrete mixtures



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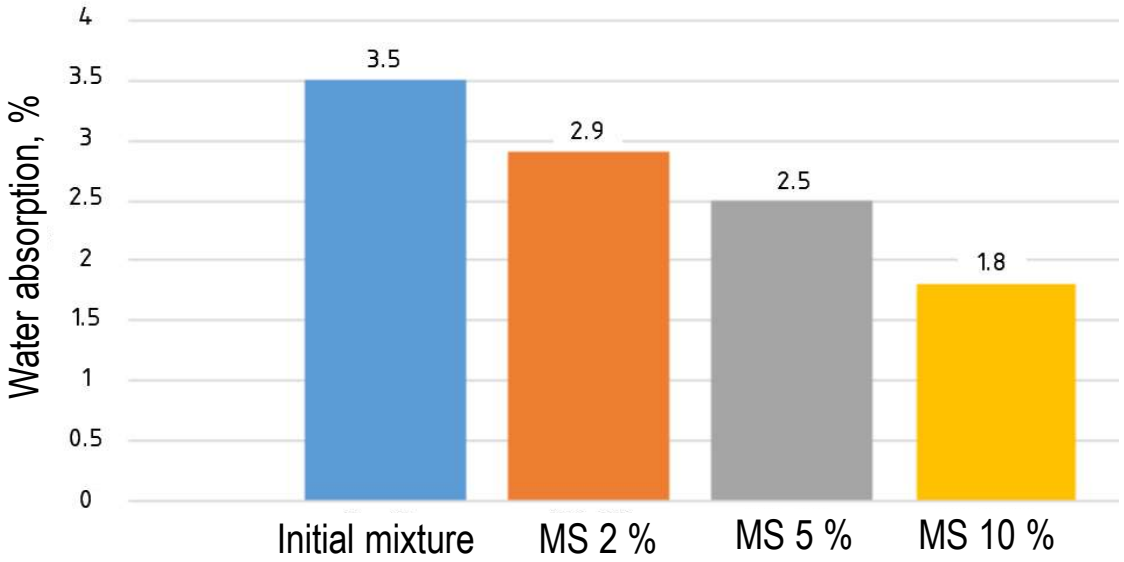

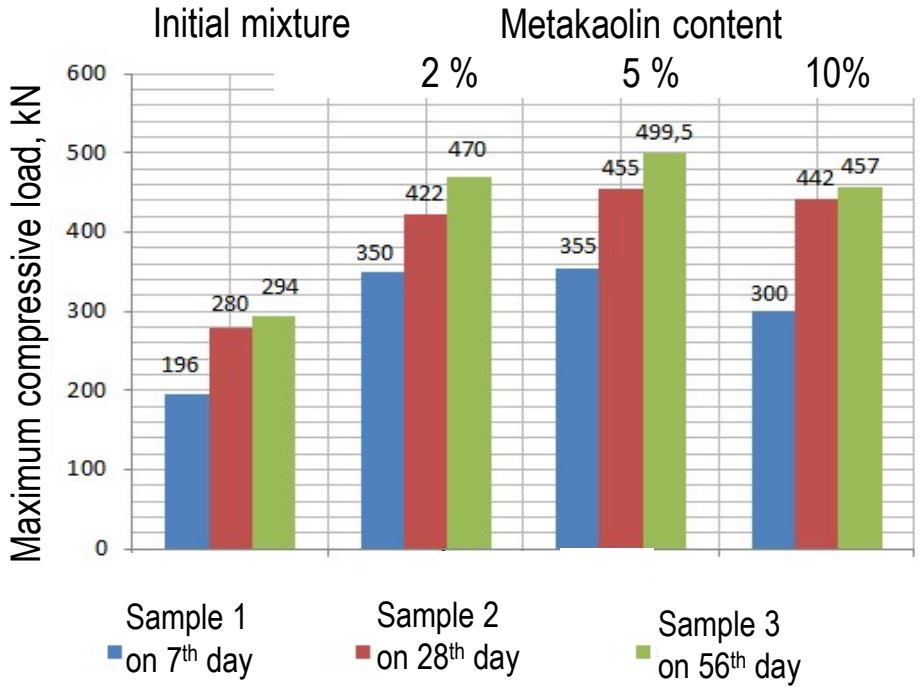

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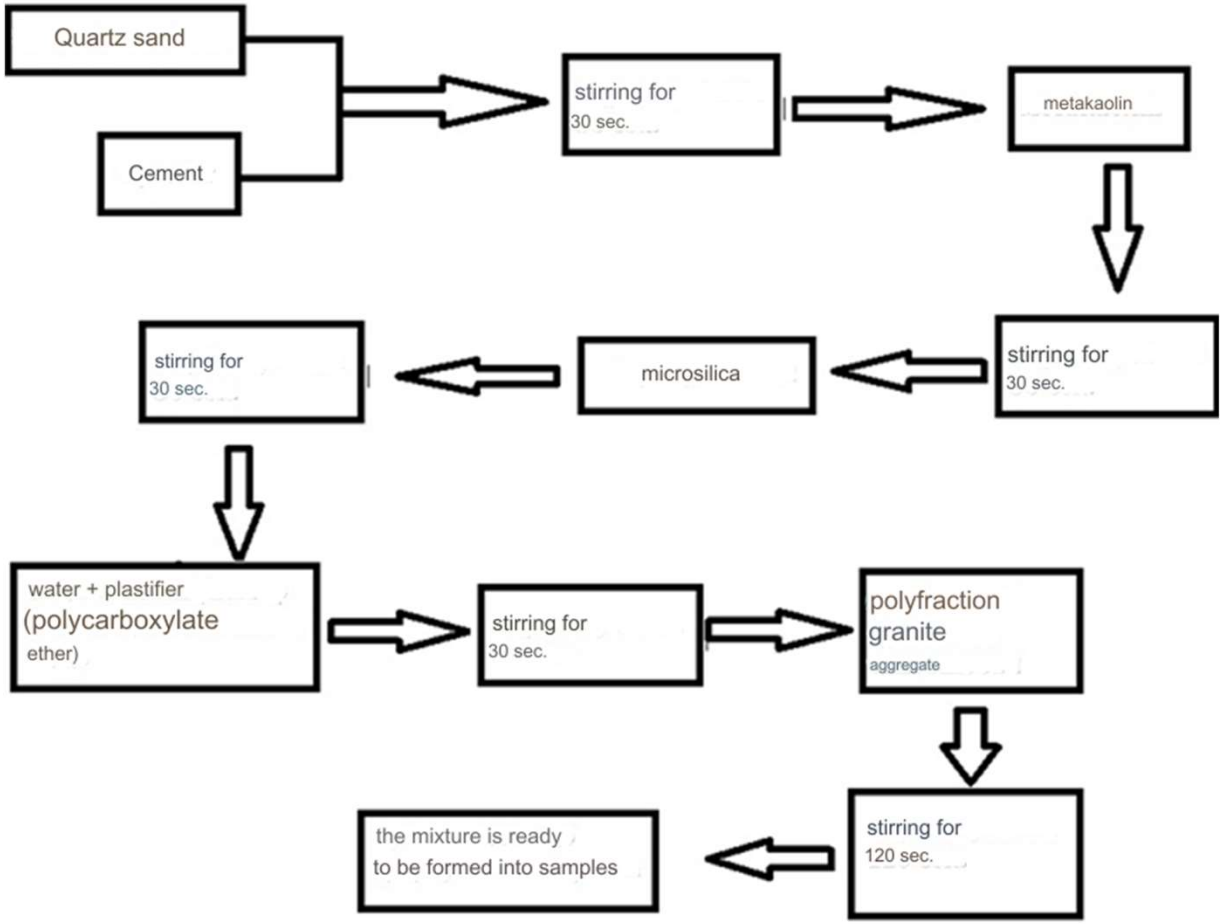

Technology of the mixture

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Research methods:

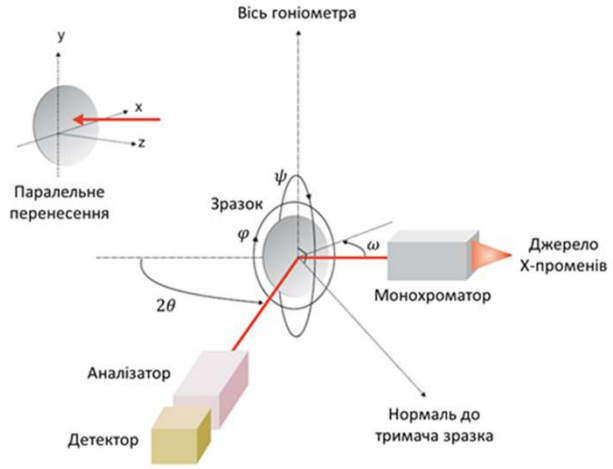
1.Scanning electron microscopy (SEM)

2. Energy dispersive X-ray spectroscopy, Hitach SU-70

3. High-resolution X-ray diffractometry

Zeiss EVO-50 scanning electron microscope with CCD detector

X'Pert PRO MRD diffractometer in a multicrystal diffraction scheme for CuKα1 radiation.



4. Determination of water resistance by the wet spot method in accordance with EN 12390-8

5. Determination of compressive strength on a hydraulic press in accordance with EN 12390-4

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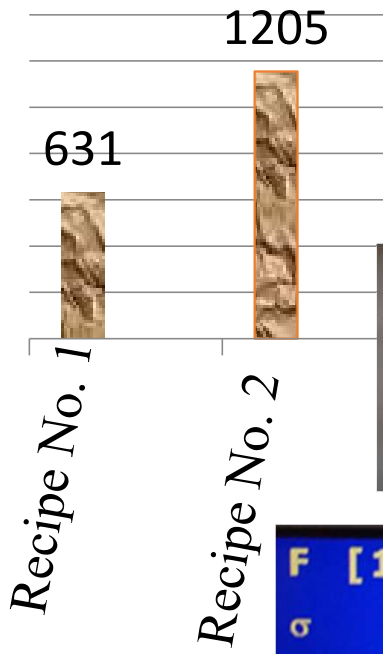
Development of cement composite formulation

	Recipe No. 1 kg/m3	Recipe No. 2 kg/m3
Cement PC-I 500 (EN 197-1:2011)	600	600
Quartz powder 50 microns.	-	30
Quartz sand, fraction 0.4-0.63 mm	584	520
Crushed stone diorite fraction 2/5 mm	315	315
Crushed stone diorite fraction 5/10 mm	315	315
Crushed stone diorite fraction 10/20 mm	660	660
Microsilica 0.1-0.3 microns.	-	60
Metakaolin 1-40 microns	-	30
Distilled water	160	160
Fiber	1%	1%
Plasticizer	5%	5%

Compressive strength in kN



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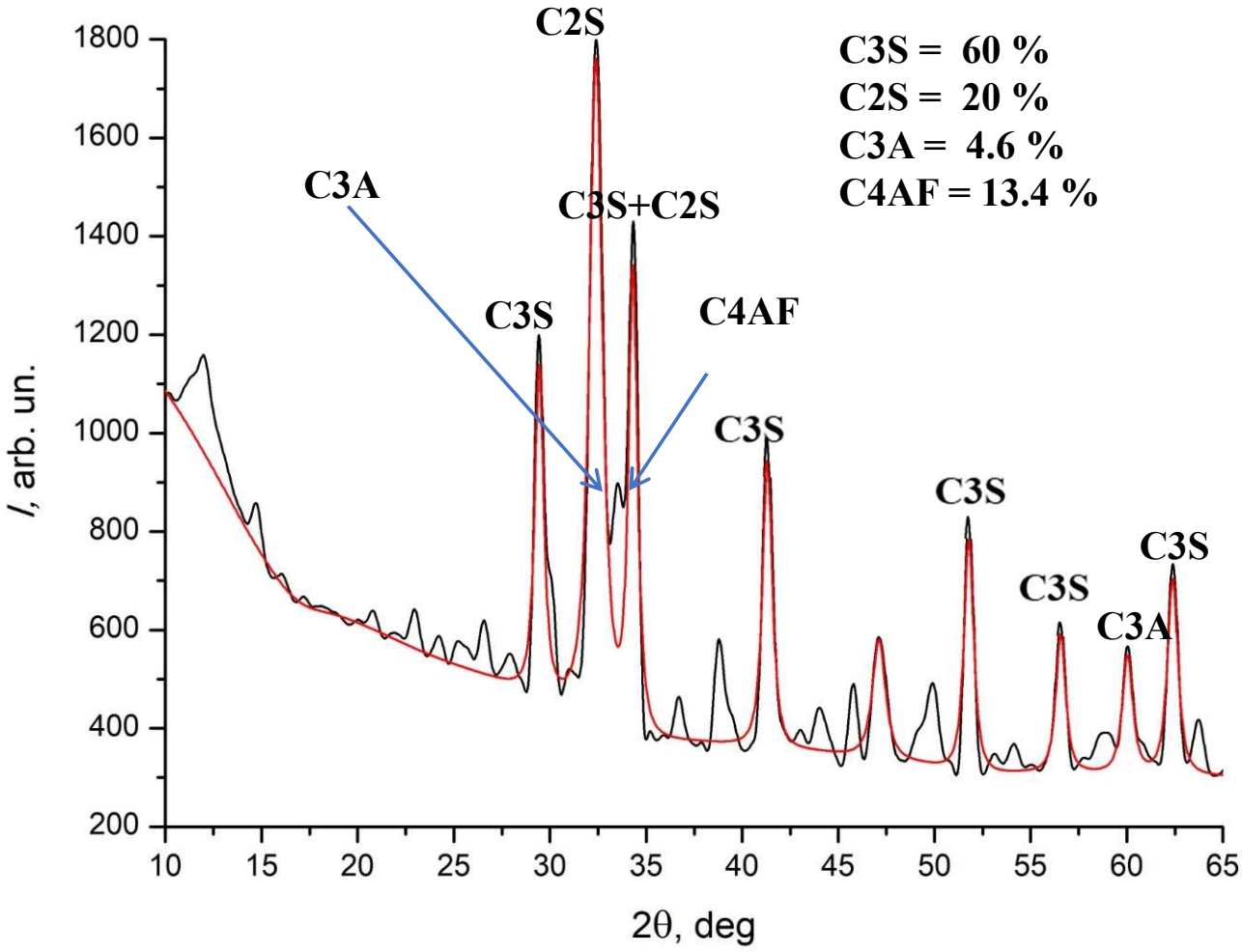

C3S = 60 %
C2S = 20 %
C3A = 4.6 %
C4AF = 13.4 %

The phase composition of cement containing various clinker minerals

from analysis of experimental X-ray diffractograms (by the Rietveld method)

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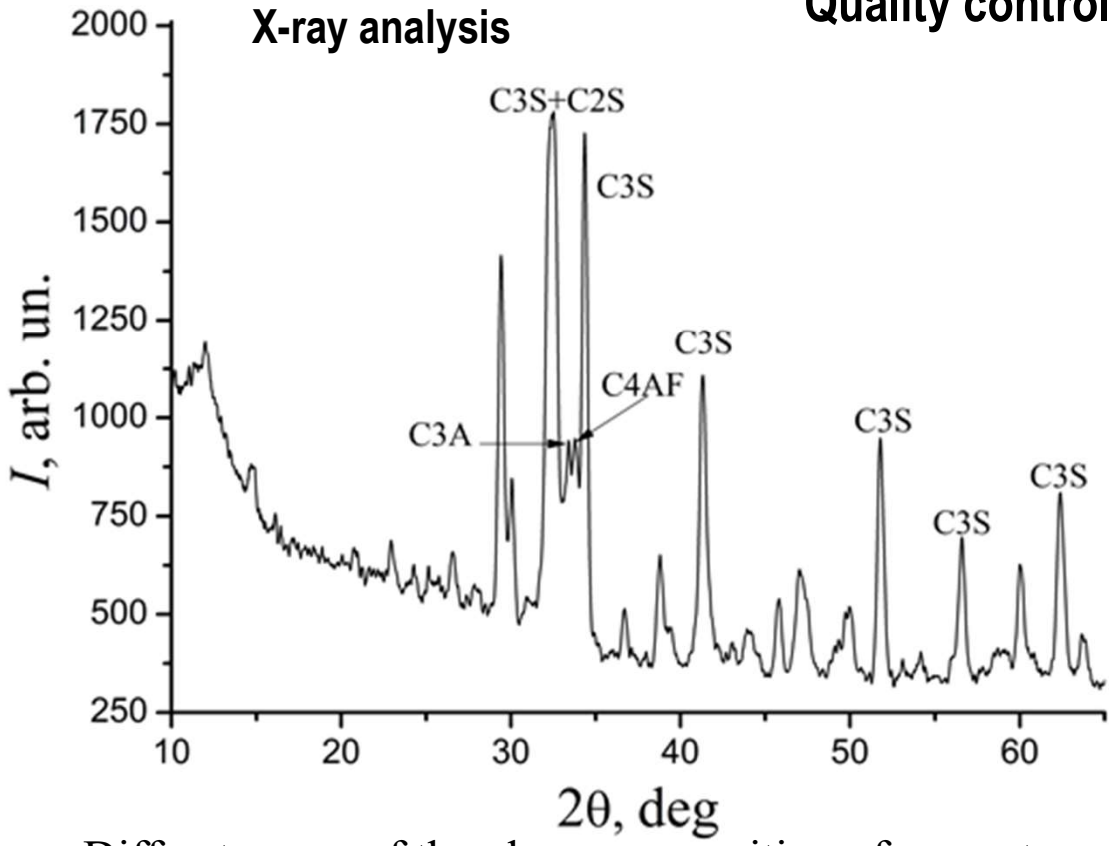
Quality control of cement CEM-I 42.5

X-ray analysis



№	Clinker minerals	Chemical formula	Contents, %.
1	C3S	Ca_3O_5Si	60.4
2	C2S	Ca_2O_4Si	22
3	C4FA	$Al_2Ca_4Fe_2O_{10}$	11,6
4	C3A	$Al_2Ca_3O_6$	6

Quantitative ratios (in %) between the main phases of cement

Diffractogram of the phase composition of cement containing various clinker minerals.

The use of cement with a low C3S content (less than 50%) significantly complicates the production of high-strength concrete, in particular, silica and metakaolin. The effectiveness of such additives requires the presence of excessive portlandite (CH) in the system, while a system with a low C3S content is characterized by a reduced content of calcium hydroxide Ca(OH)2

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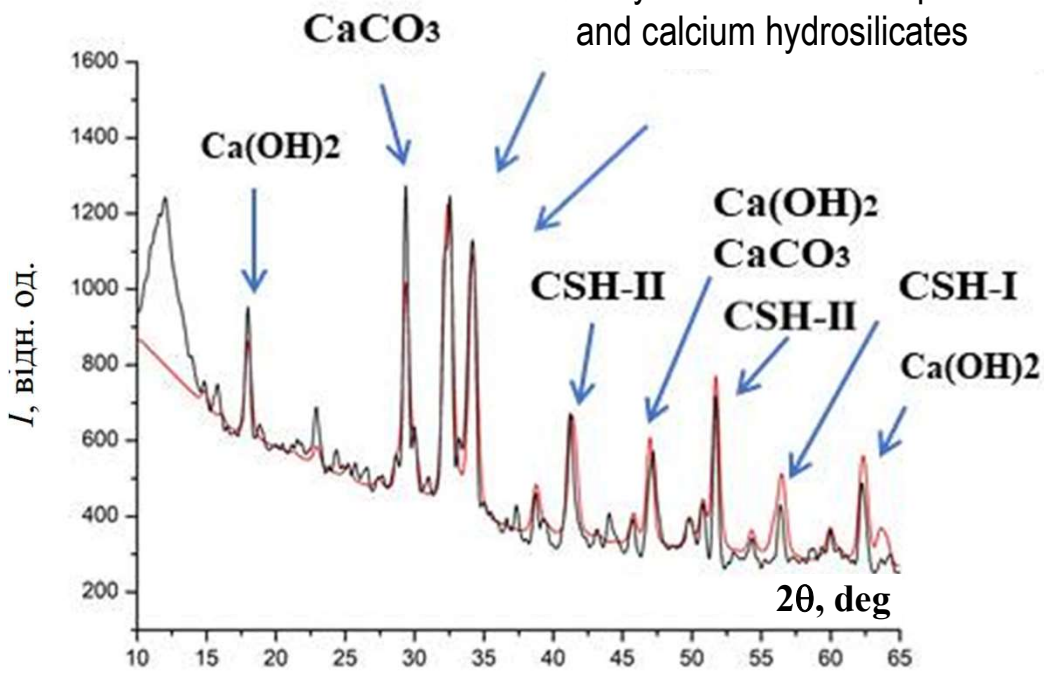
X-ray studies were performed at the collective use center in the Lashkarev Institute of Semiconductor Physics (Kyiv)

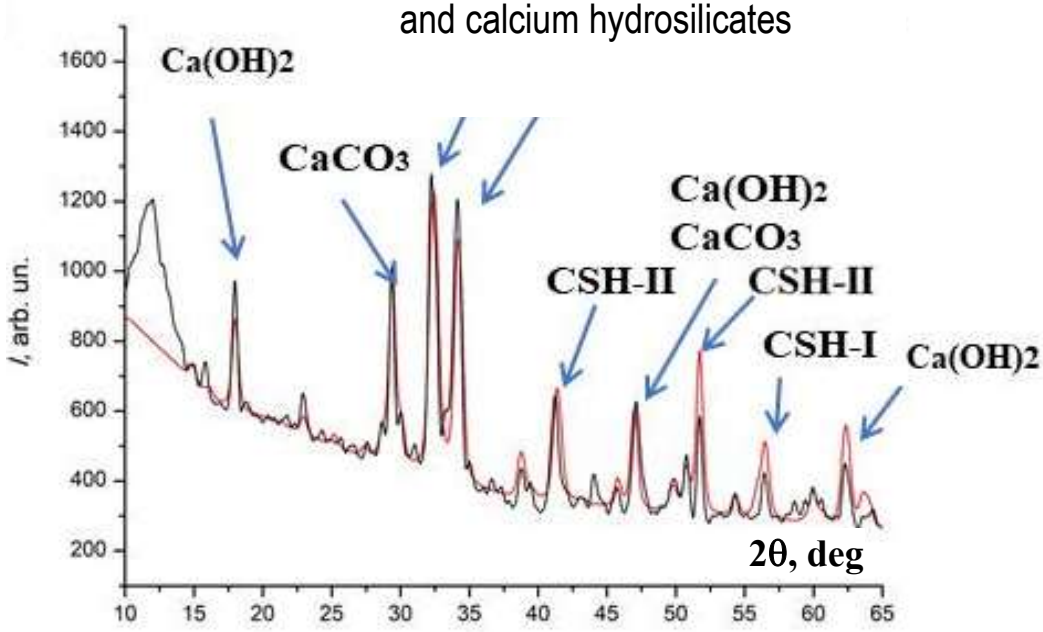
Recipe No. 1

Intensity peaks of of hydroaluminate compounds and calcium hydrosilicates



Recipe No. 2

Intensity peaks of of hydroaluminate compounds and calcium hydrosilicates



Analysis of theoretical (by the Rietveld method) and experimental diffractograms of hydration compounds

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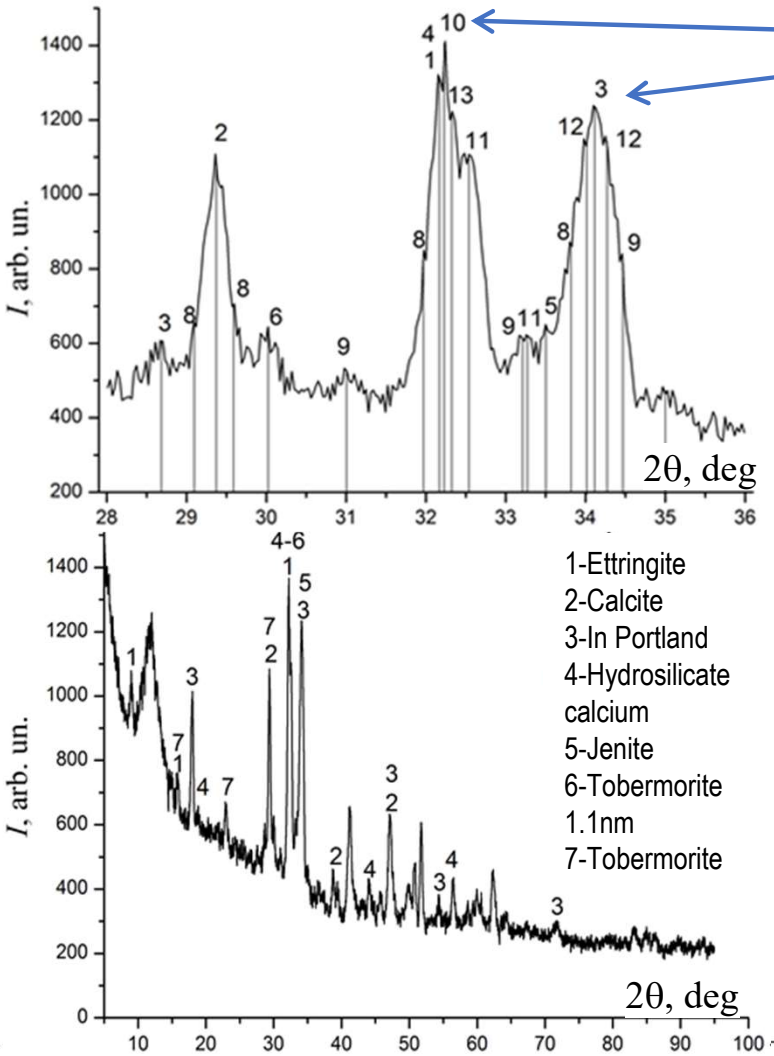

Peaks of intensity of of CHS, HAlC compounds

№	Chemical formula	*d*/*n*	Name of the compound
1	$Al_2Ca_6H_{66}O_{49.68}S_3$	0.974, 0.563, 0.388,	Ettringitis
2	$CaCO_3$	0.278, 0.303, 0.191	Calcite
3	$Ca(OH)_2$	0.491, 0.262, 0.192	Portlandite
4	$Ca_3H_2O_{7.5}Si_{1.5}$	0.278, 0.335, 0.181	Hydrosilicate calcium
5	$Ca_9H_{22}O_{32}Si_6$	1.049, 0.262, 0.278	Janite
6	$Ca_2H_3O_{11}Si_3$	0.308, 0.297, 0.351	Tobermoryt 1.1-nm
7	$Ca_{2.5}H_{11}O_{12.5}Si_3$	0.552, 0.310, 0.301	Tobermorite 1.4 nm
8	$Ca_5H_{10}O_{22}Si_6$	0.307, 0.301, 0.279	Wedge tobermorite
9	$Ca_2H_2O_5Si$	0.287, 0.269, 0.260	HSC
10	$Ca_5H_2O_{10}Si_2$	0.303, 0.277, 0.256	GSK
11	$Al_2CaH_{10}O_{21}Si_6$	0.305, 0.275, 0.268	CHS
12	$Al_2CaH_8O_{10}Si_{12}$	0.263, 0.262	CHAS
13	$Al_{3,5}Ca_3H_{9,7}O_{12}$	0.276, 0.309	CHA

X-ray diffractometry of cement containing ultradisperse modifiers

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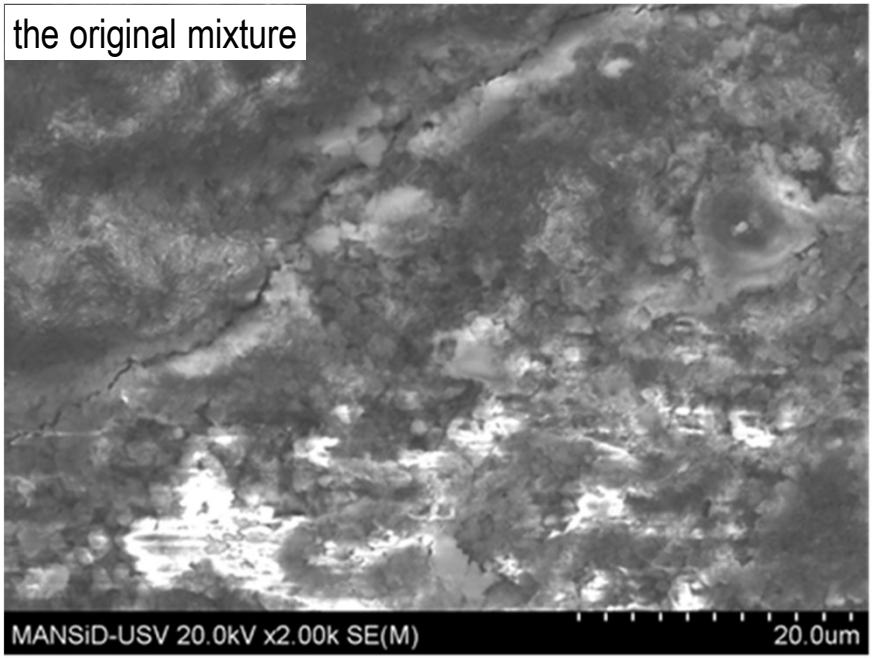
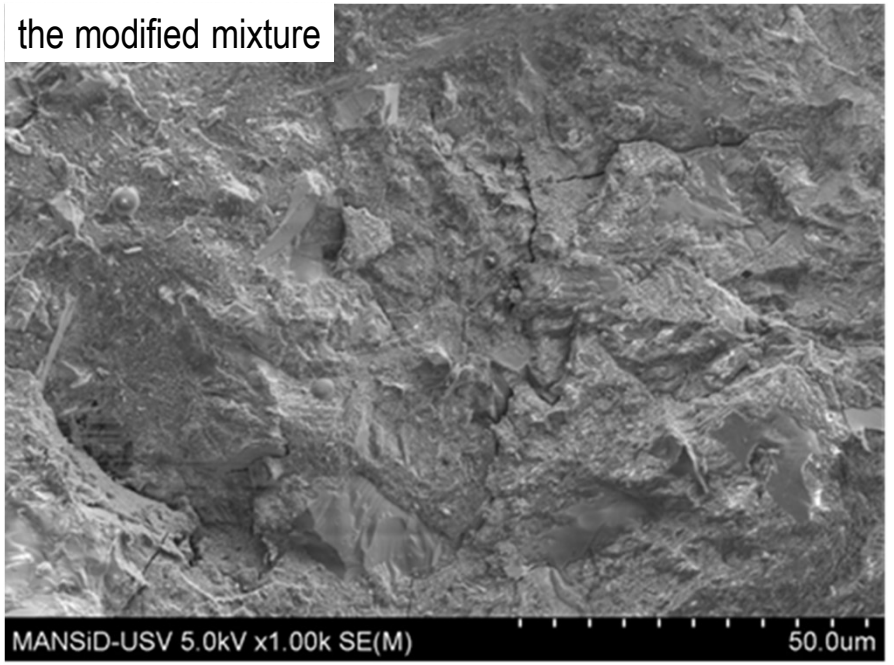
SEM images of the fracture surface of the cement matrix

To understand the formation of hydration phases in high-strength concrete composites and the effect of ultrafine modifiers, the microstructure of the fracture surface and their phase composition were obtained by scanning electron microscopy using energy dispersive x-ray spectroscopy

the original mixture



the modified mixture



Influence of ultrafine modifiers on the processes of cement stone structure formation

Experiments were carried out at the State University of Stefan cel Mare (Suceava, Romania)

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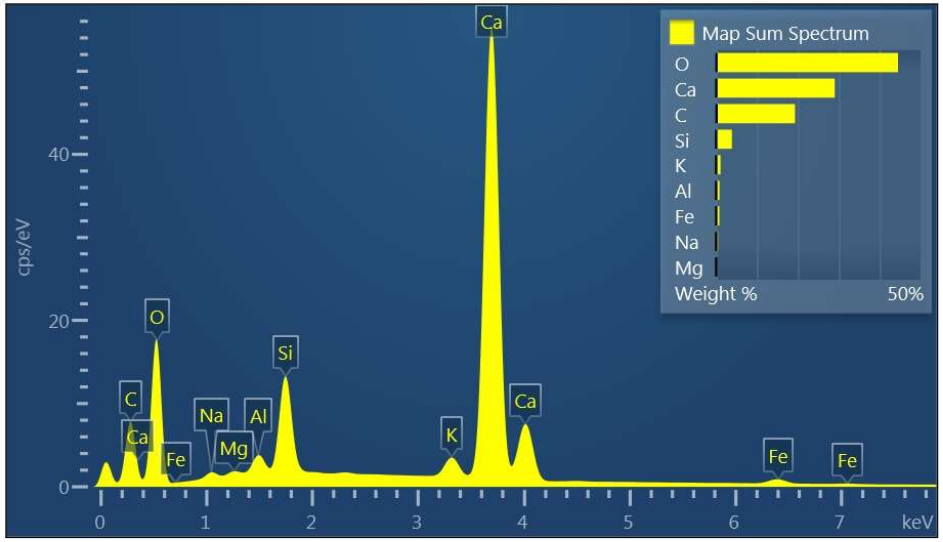
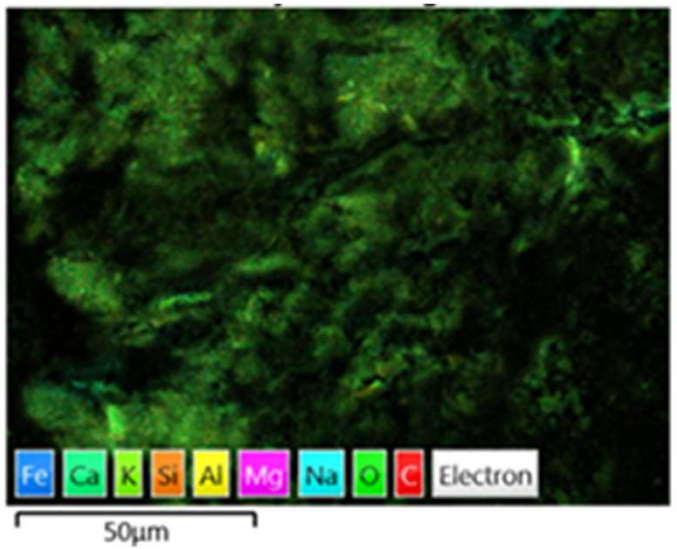


Element	Apparent Concentration	Wt%	Standard Label
C	108.28	**19.27**	C
O	285.15	**44.50**	SiO2
Na	5.13	0.45	Albite
Mg	2.27	0.21	MgO
Al	10.60	0.82	Al2O3
Si	55.79	3.86	SiO2
K	19.90	1.07	KBr
Ca	495.17	**29.03**	Wollastonite
Fe	10.54	0.79	Fe

Elemental composition of concrete samples of the initial mixture according to the results of EDX analysis, and the results of energy dispersive X-wave analysis, respectively, on maps



The list of elements in the table and their percentage content indicate the presence of the vast majority of calcite CaCO3 in concrete matrix No. 1. In the presence of moisture, as a result of the reaction of calcium oxide with atmospheric carbon dioxide, a layered structure with low adhesion and cohesion is usually formed. According to the EDX analysis, the fracture of concrete composite No. 1 mainly occurs in areas with high concentrations of calcite.

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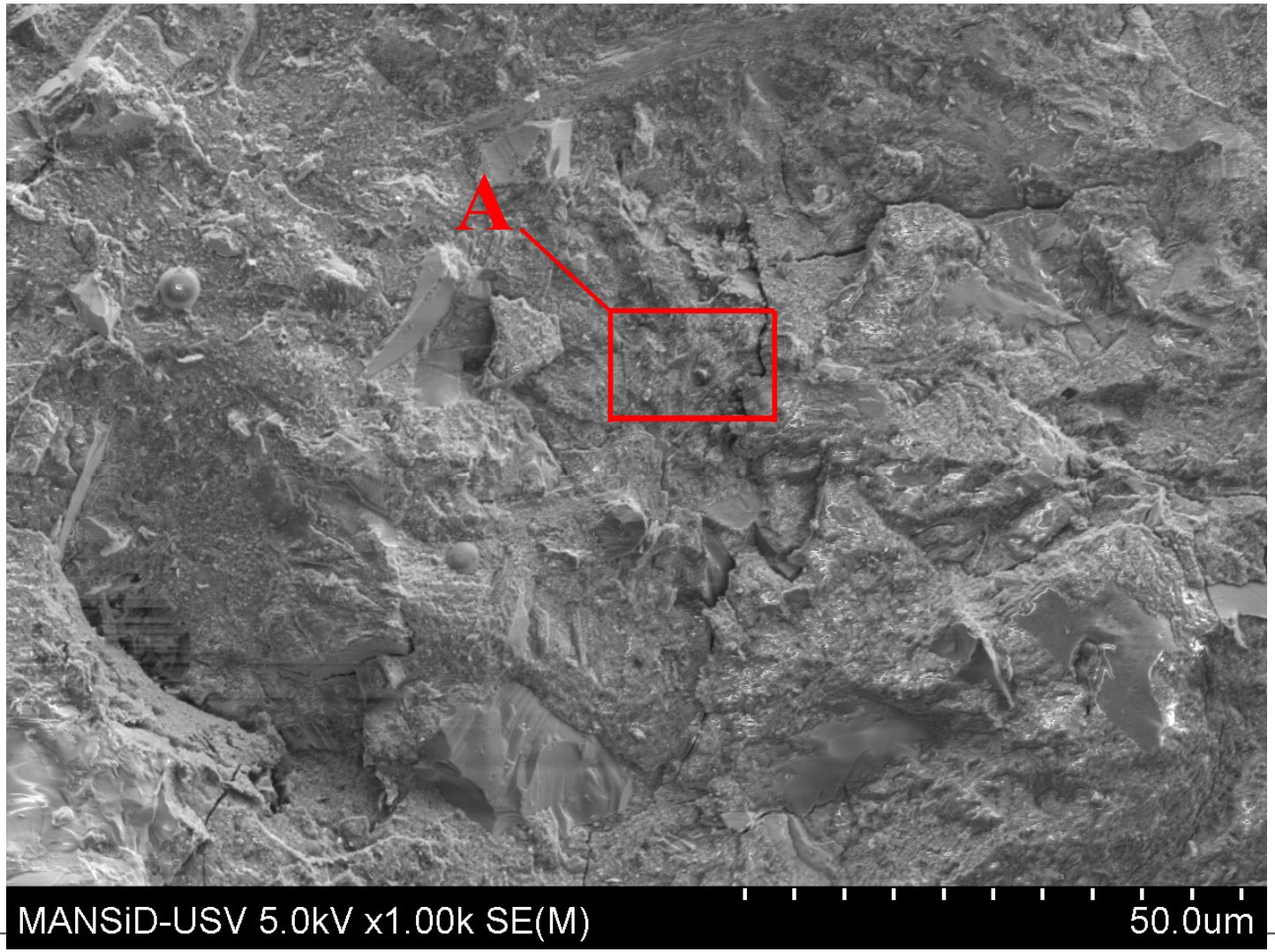

SEM image of the fracture surface of the cement matrix with the modified mixture

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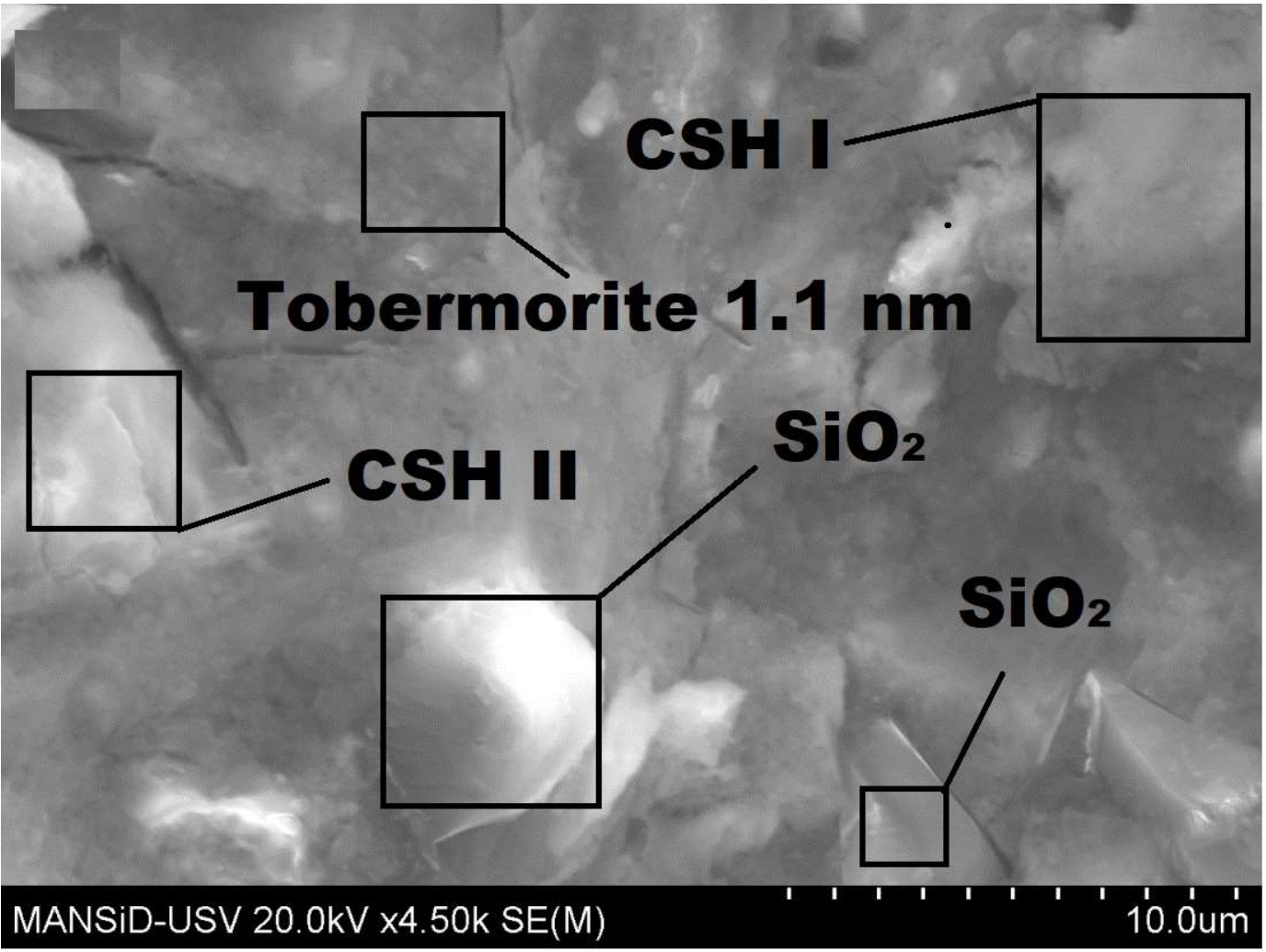

Enlarged SEM image of zone A of the fracture surface of the cement matrix of the modified mixture

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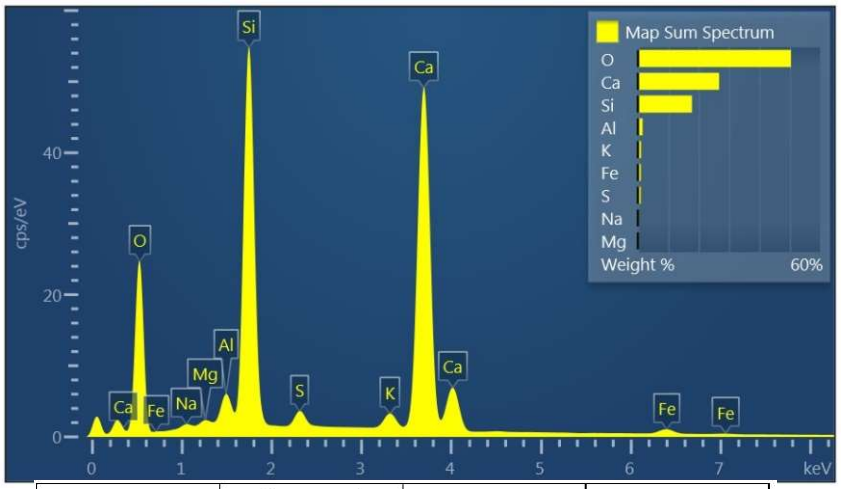
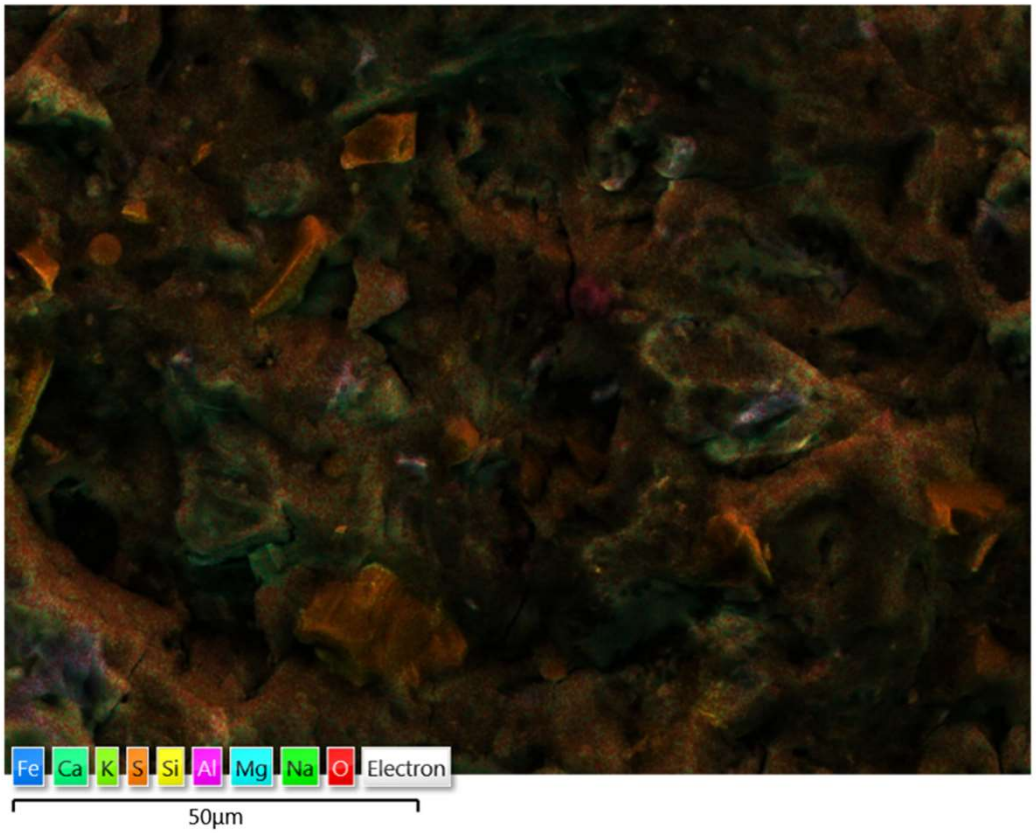
Elemental composition of concrete samples with the mixture modified by a complex based on microsilica and metakaolin EDX analysis and the results of energy dispersive X-ray analysis, respectively



Element	Apparent Concentration	Wt%	Standard Label
O	**393.92**	**50.40**	**SiO2**
Na	3.87	0.34	Albite
Mg	3.43	0.31	MgO
Al	19.98	1.52	Al2O3
Si	**257.48**	**17.81**	**SiO2**
S	12.53	0.90	FeS2
K	17.78	1.03	KBr
Ca	**439.97**	**26.75**	**Wollastonite**
Fe	12.70	0.94	Fe



The phase structure of the cement composite of formulation No. 2 is characterized by a large number of phases and their heterogeneity.The phase composition is dominated by compounds of low and high basicity HSCs, as well as unreacted microsilica particles. Probably, the significantly higher compressive strength of formulation No. 2 is associated with a more developed specific surface area of pozzolanic particles, which are able to react faster with Ca(OH)2, forming a dense microstructure.

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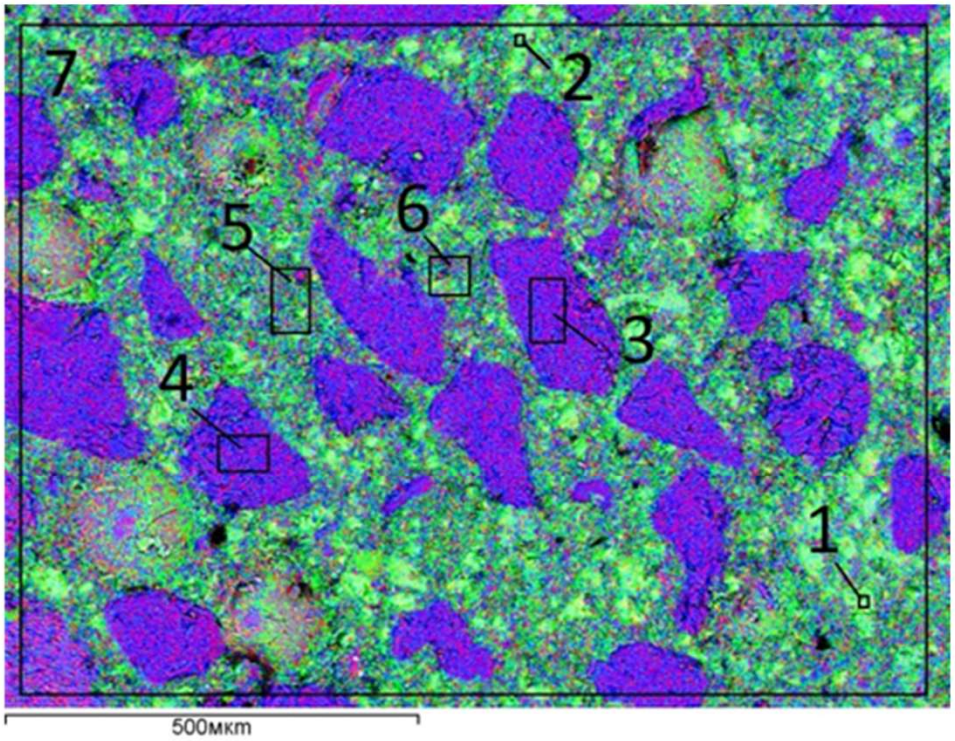
SEM images of the cement surface microstructure of the initial mixture

Experiments were carried out at the Bakul Institute of Superhard Materials, Kyiv. Ukraine



№ Region.	C	O	Mg	Al	Si	K	Fe	Ca
	%							
1	2.52	40,30	0,57	1,78	**13,38**	0,49	1,74	**39,23**
2	6,54	36,56	0,59	0,43	**12,34**	-	-	**43,54**
3	20,32	42,05	-	-	37,18	-	-	0,45
4	18,40	40,61	-	-	40,19	-	0,34	0,46
5	21,33	37,56	0,70	1,8	**17,81**	0,8	1,30	**18,69**
6	21,66	38,90	0,71	2,15	**18,44**	0,92	1,01	**16,20**
7	19,06	40,28	0,54	1,37	23,64	0,58	0,8	13,74

Intergranular zones 1 and 2 have a significantly lower carbon content (two times) and a higher calcium content than the corresponding zones 5 and 6, while the calcium to silicon ratio is almost the same.

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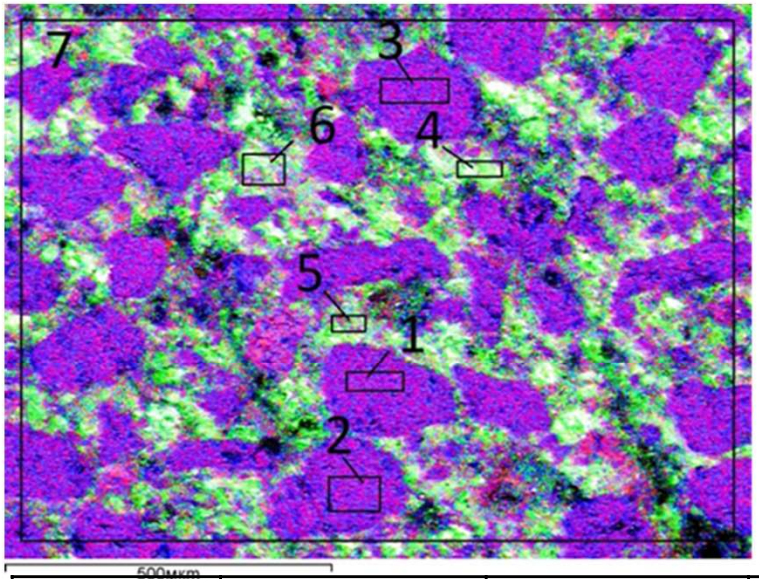

Fragments of SEM images of the microstructure of cement matrix surface with modified formulation

SEM images of the microstructure for formulation No. 2, are more developed and dense. This may be due to the fact that the modified samples are characterized by a higher content of hydration products, and the densified structure in this form gives a significant increase in strength.

Region №	C (%)	O (%)	Mg (%)	Al (%)	Si (%)	Fe (%)	Ca (%)
1	11,51	46,05	-	-	42,24	-	0,20
2	12,12	45,50	-	-	41,95	-	0,43
3	17,15	42,85	-	0,18	38,98	-	0,55
4	14,06	41,09	1,24	1,92	17,09	0,94	22,95
5	9,37	45,89	1,00	2,92	21,94	1,13	17,09
6	8,02	45,23	1,10	2,86	23,41	1,01	17,80
7	20,28	40,69	0,40	1,53	26,38	0,80	9,34

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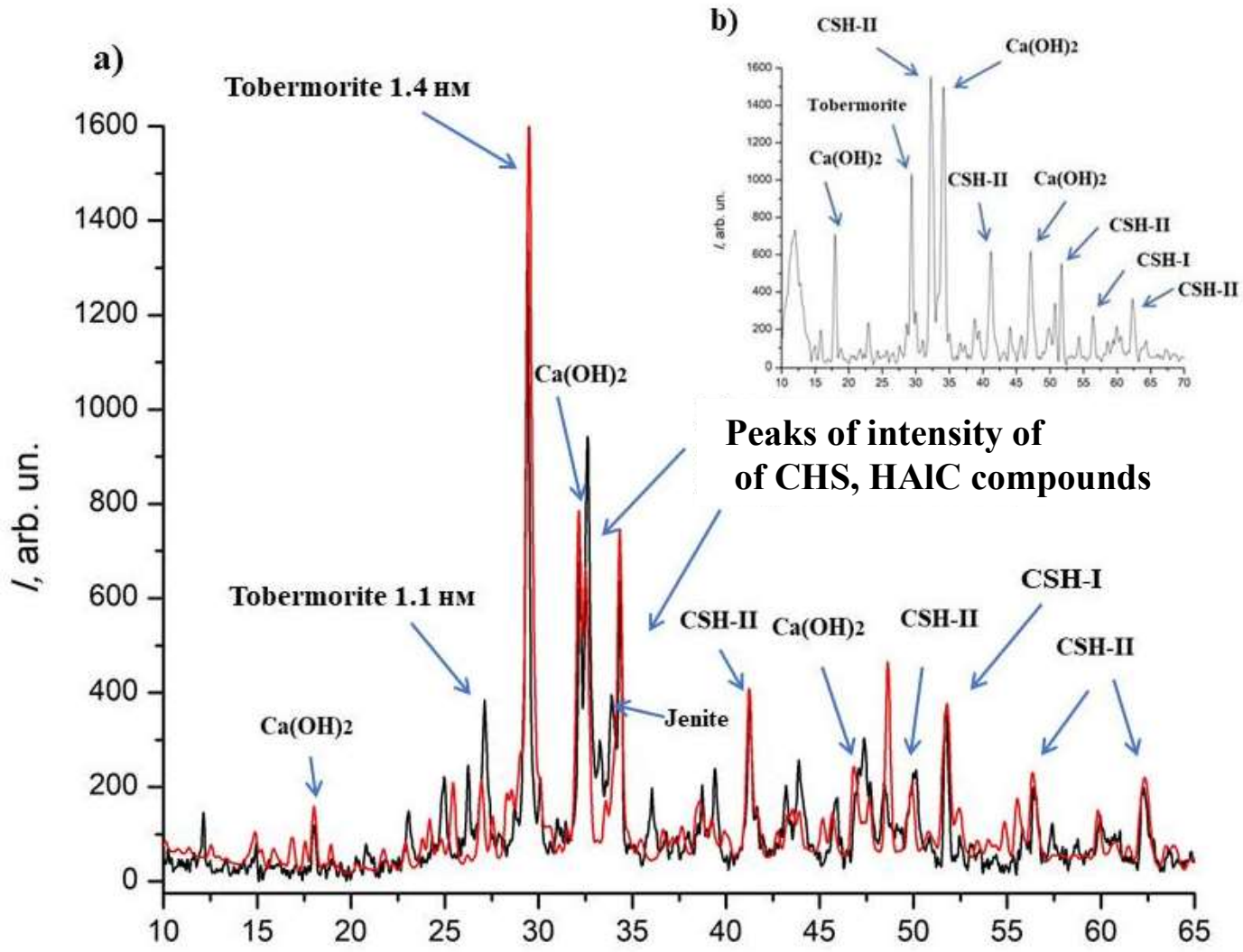

Peaks of intensity of of CHS, HAlC compounds

a) - experimental (dark line) and calculated (red) diffractograms for more than a year of hydration of the compounds of the modified composite;

b) - on the 28th day of hydration of the composite.

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To design concrete with a certain minimum durability, an understanding of the micro- and nanostructural-level processes that cause deterioration, including the rates at which these processes occur under the conditions in which the concrete will be exposed, is required. In the latter respect, a wide range of climatic, chemical and physical factors must be considered, which are the subject of further research.

- Modification of the cement matrix with a complex of finely dispersed silica and aluminosilicate compounds at a certain ratio leads mainly to the formation of low-base calcium hydrosilicates and such C-S-H structural models as jenite (d/n, nm: 1.049; 0.262; 0.278) and tobermorite (d/n, nm: 0.552; 0.310; 0.301; 0.308; 0.297; 0.351), which have a layered structure and are essentially nanomaterials. These phases were formed from Ca(OH)2 and active silica components with a Ca/Si ratio of 1.1-1.2.

- The adding of metakaolin cause the formation of stable calcium silicate hydroaluminates with crystalline structure which indicated by maxima on the X-ray diffractogram corresponding to d/n = 0.305; 0.275; 0.268; 0.263; 0.262 (nm) and hydroaluminates d/n=0,276; 0.309 (nm).

- It was found that in X-ray diffractograms, the main intensity maxima of hydration products are located in the same angular positions as the intensity maxima for clinker minerals, in particular, for alite (C3S) and belite (C2S). This indicates their crucial role in the formation of the cement matrix structure.
- The presence of compounds that include Al and Fe at the later stages of hydration (over 1 year) is a sign of the formation of secondary phases of hydroaluminates and calcium hydroferrites. The increase in strength by 36% is explained by the optimal use of free calcium hydroxide and amorphous silica, which is a sign of a progressive pozzolanic reaction in the cement matrix during one year of hydration
- During the year of hydration with the introduction of silica and aluminosilicate modifiers, a significant decrease in the content of Ca(OH)2 and high-base calcium hydrosilicates CSH-II is observed with a simultaneous increase in the content of jenite and tobermorite, which are likely to have been transformed from CSH, which is the reason for the increase in strength

THANK YOU !

GRACIAS !

MULȚUMESC !

DZIĘKUJĘ !

ДЯКУЮ !