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Evaluation of the effect of a complex nanomodifying additive "lignosulfonate / graphene oxide" on the non-autoclaved aerated concrete hydration process

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Abstract: The article evaluates the influence of various plasticizing and structure-forming additives on the hydration process of non-autoclaved aerated concrete (NAC). The authors have developed a method for the NAC formation with the introduction of the following modifiers: lignosulfonate (LS), graphene oxide (GO) (1 % aqueous suspension) and a complex additive – GO/LS. The formation of the structure and the study of new mineral formations in cement stone as a result of hydration were carried out by X-ray diffraction and differential thermal analysis. According to XRD-analysis, gas blocks of all compositions contain quartz, tobermorite, calcium hydrogarnets, xonotlite, C–S–H(I), and calcite. The diffraction pattern of the sample with the addition of GO/LS shows that NAC contains, first of all, high-intensity reflections of tobermorite, xonotlite, as well as C–S–H and calcite. All other NAC samples are characterized by a lower intensity of reflections of the indicated calcium hydrosilicates. TG- and DSC-curves for all studied gas blocks have a similar character – 3 stages of weight loss, except for the control sample. Aerated concrete without additives at temperatures up to 100 °C loses 0.96 % of its weight, with the addition of LS – 1.20 %, GO – 1.35 %, and complex additive – 1.72 %. In the temperature range of 400–500 °C, an endothermic effect appears, which indicates the dehydration of weakly crystallized gel-like hydrosilicates and calcium hydrogarnets. It is this peak that is absent in the control sample. Thus, based on the diagnostic results, it was established that the complex modifying additive allow to increase of the hydration product crystallinity of the hardened NAC. The results suggest that the modified NAC containing a complex additive is more stable and functional during operation than comparative samples of concrete of a traditional composition without this additive.

Keywords: non-autoclaved aerated concrete; lignosulfonate; graphene oxide; complex additive; X-ray diffraction; differential thermal analysis; calcium hydrosilicate; binding material hydration.

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Оценка влияния комплексной наномодифицирующей добавки «лигносульфонат / оксид графена» на процесс гидратации неавтоклавного газобетона

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Аннотация: Проведена оценка влияния различных пластифицирующих и структурообразующих добавок на процесс гидратации неавтоклавного газобетона (НГБ). Разработана методика формирования НГБ с введением следующих модификаторов: лигносульфоната (ЛС), оксида графена (ОГ) (1 % водная суспензия) и комплексной добавки – ОГ/ЛС. Формирование структуры и исследование минеральных новообразований в цементном камне в результате гидратации проводились методом рентгеноструктурного и дифференциально-термического анализа.

Согласно рентгеновской дифрактометрии, газоблоки всех составов содержат кварц, тоберморит, гидрогранаты кальция, ксонотлит, C–S–H(I), кальцит. Дифрактограмма образца с добавкой ОГ/ЛС показывает, что НГБ содержит, прежде всего, высокоинтенсивные отражения тоберморита, ксонотлита, а также C–S–H и кальцита. Для всех остальных образцов НГБ характерна более низкая интенсивность рефлексов указанных гидросиликатов кальция. ТГ- и ДСК-кривые для всех исследуемых газоблоков имеют схожий характер – 3 ступени потери массы, кроме контрольного образца. Газобетон без добавок при температуре около 100 °C теряет 0,96 % массы, с добавкой ЛС – 1,20 %, ОГ – 1,35 %, комплексной добавки – 1,72 %. В интервале температур 400…500 °C появляется эндотермический эффект, который говорит о дегидратации слабозакристаллизованных гелеобразных гидросиликатов и гидрогранатов кальция. Именно этот пик отсутствует у контрольного образца. Таким образом, по результатам диагностики установлено, что комплексная модифицирующая добавка увеличивает кристалличность продуктов гидратации затвердевшего НГБ. Результаты позволяют предположить, что модифицированный НГБ, содержащий комплексную добавку, является более стабильным и функциональным в процессе эксплуатации, чем сравнительные образцы бетона традиционного состава без такой добавки.

Ключевые слова: неавтоклавный газобетон; лигносульфонат; оксид графена; комплексная добавка; рентгеновская дифрактометрия; дифференциально-термический анализ; гидросиликат кальция; гидратация вяжущего.

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1. Introduction

The use of materials with thermal insulation properties is an urgent task and is of great importance for the development of the construction industry. For these purposes, so-called cellular concrete is successfully used, the category of which includes foam concrete, foam aerated concrete, autoclaved and non-autoclaved aerated concrete, etc. Aerated concrete is successfully used for wall construction – both external and internal (blocks, panels), floors (slabs), fences, etc., as well as for construction of frame structures [1, 2].

Autoclave gas blocks are produced only in factory conditions in accordance with Russian Standard 31360-2007 using high-pressure reactors – autoclaves. Non-autoclaved aerated concrete is produced in accordance with Russian Standard 25485-89 without the use of expensive equipment at the construction site. As is known, average density and strength are the main parameters by which the choice of thermal insulation building material is made. At the same density, non-autoclaved aerated concrete will exhibit lower mechanical characteristics than autoclaved one, which primarily affects its load-bearing capacity. Therefore, the urgent task is to develop the composition of a non-autoclaved aerated concrete mixture that provides high strength indicators along with good thermal insulation properties [3].

The mechanical properties of concrete materials are greatly influenced by micro- and nanoscale reactions occurring in the cement paste during the hydration process. A number of studies have proven that the addition of nanodispersed particles can improve the properties of concrete due to the fact that nanoparticles act as nucleation sites for the growth of the C–S–H gel framework. The nucleation of hydration products on nanoparticles promotes and accelerates the hydration of cement [4–8].

In [9], the authors obtained compositions of non-autoclaved aerated concrete with the addition of carbon nanomaterial (CNM) and a composite binder – fly ash. It has been proven that the introduction of CNM leads to an increase in the strength characteristics of aerated concrete by 15–20 % due to changes in the hydration process and the formation of additional amounts of calcium hydrosilicates according to IR spectroscopy.

The authors [10] studied autoclaved silicate aerated concrete modified with ultrafine mineral additives with a concentration of 5–10 % and a dispersion of multi-walled carbon nanotubes (MWCNTs) up to 0.001–0.005 % by weight of the binder. In this way, products were obtained from autoclaved aerated concrete with a density of 540–580 $kg \cdot m^{-3}$ with a compressive strength of 3.4–3.9 MPa, i.e. an improvement in the strength characteristics of products up to 30 % is achieved compared to the control sample.

The introduction of MWCNTs dispersion in an amount of 0.002 % by weight of Portland cement in the production of autoclaved aerated concrete [11] contributed to the production of samples with a density of 180–200 $kg \cdot m^{-3}$ with a compressive strength of 0.7–0.9 MPa, a thermal conductivity of 0.046 was 0.048 $W \cdot (m \cdot °C)^{-1}$. The authors of [11] state that the addition of nanoparticles leads to a change in the morphology of hydration products,

which are intertwined plate-like and needle-shaped crystals of low-basic calcium hydrosilicates. Thanks to this, a strong spatial frame of the composite is formed, less susceptible to cracking and destruction.

A team of authors [12] published a study of the effect of a 1–2 % MWCNTs suspension using carboxymethylcellulose as a surfactant on the quality and characteristics of autoclaved (AAC) and non-autoclaved aerated concrete (NAC). It has been established that modification makes it possible to increase the bending strength of NAC to 11.23 %, AAC – to 25.00 %; and compressive strength of NAC – up to 11.03 %, AAC– up to 24.50 %.

There are works aimed at modifying cement stone with graphene oxide (GO), graphene nanoplates, etc. [13–15]. Research results [16] show that the addition of multilayer graphene can achieve an increase in compressive strength by 54 % and flexural strength by 21 % for cementitious composites, respectively. The addition of 0.03 % GO [17] makes it possible to increase the flexural strength of cement stone by 77 % and compressive strength by 47.6 % for compositions aged 28 days. The results of modification with nanographene (multilayer graphene with a number of layers of 5–10) in an amount of 0.8 % of autoclaved aerated concrete [18] show an increase in compressive and tensile strength, as well as impact resistance by 45, 81 and 130 % compared to the control sample. At the same time, the water absorption of the samples was reduced to 61 %.

Thus, nanotechnologies and nanomaterials as modifying additives can be effectively used to improve the properties of concrete, increase their mechanical characteristics, thermal insulation capacity, and, as a result, increase the durability of building structures built on their basis.

The purpose of this work is to identify the influence of additives, in particular, based on GO and lignosulfonate, on non-autoclaved aerated concrete by studying cement stone using X-ray diffraction and differential thermal analysis.

2. Materials and Methods

2.1. Materials

To obtain NAC samples, Portland cement M500 (Paladium, Zhukovsky, Russia), dry sand Russian Standard 8736-2014, slaked construction carbonate-lime flour (LLC “StroyKomplekt”, Voronezh, Russia), aluminum powder, tap water (ratio water/cement was 0.4) were taken. To modify NAC, powdered lignosulfonate (AKVAKHIM LLC, Kazan, Russia) and a 1 % aqueous GO suspension (NanoTechCenter LLC, Tambov) were used.

2.2. Preparation of GO/LS non-autoclave aerated concrete specimens

Samples of aerated blocks were prepared with an GO/LS content of 0.0002 wt. % and 0.16 wt. % LS by weight of cement. NAC samples were also obtained only with the addition of GO at the indicated concentration and separately with the addition of 0.16 wt. % LS. To prepare the modifying additive, the following procedure was developed:

a) at the first stage, an aqueous solution of lignosulfonate was obtained: the required amount of lignosulfonate powder was added to distilled water heated to 80 °C and stirred continuously for 10–15 minutes;

b) the pH of the aqueous GO suspension was adjusted to 10 by adding 1 M NaOH solution. The pH was measured using a HI 2210 benchtop pH meter (HANNA Instruments, Woonsocket, USA);

c) a suspension of GO was added to LS solution cooled to room temperature and kept at 90 °C.

2.3. Test methods and specimens

For each cement composition, cubes measuring 70×70×70 mm were cast to determine compressive strength and 40×40×160 mm to assess flexural strength. The compaction of the aerated concrete mixture was carried out on a vibration platform in accordance with Russian Standard 17674-72 with the mandatory presence of a vertical component of vibrations. On the 7th day of hardening, the cubes were removed to continue strengthening (28 days). To determine the compressive strength, an IP-500 press with a maximum force of 50 tons (JSC ZIPO, Armavir) was used, and the bending strength was determined using a uniaxial testing machine with a power of 2000 kN and an applied load of 0.4 MPa·s^{-1}.

2.4. Analytic methods

The study of new mineral formations in cement stone was carried out by X-ray phase analysis using a Thermo Scientific ARL Equinox 1000 X-ray diffractometer (TechTrend Science Co., Ltd., Taiwan) (wavelength $\lambda = 0.1540562$ nm (copper anode). Determination of weight change under temperature influence were carried out using a NETZSCH STA 449 F3 Jupiter instrument (NETZSCH-Feinmahltechnik GmbH, Selb, Germany) with simultaneous thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) (measurement in the air atmosphere at a heating rate of 10 K·min^{-1}).

3. Results and Discussion

It is well known that the hydration process of Portland cement represents a series of chemical transformations [19]. When water interacts with cement grains, a chemical reaction occurs, due to which needle-shaped crystals appear on the surface of the grains and in the water. Over a short time, the volume of these new formations increases, and the cement grains form a developed spatial network among themselves. After 8–10 hours, the gelation process continues and the entire volume, in which cement grains gradually decrease, is filled with calcium hydrosilicates, aluminates, sulfoaluminates, ferrites, sulfoferrites and hydrogarnets (depending on hydration conditions, the composition of the concrete mixture, etc.). The remaining voids are filled, although not so intensively, with hydration products, i.e. clinker minerals. The resulting silicate structure turns into cement stone and after a day begins to displace the aluminate structure. After completion of the hydration process, the cement stone hardens and becomes strong [20].

The formation of the cellular structure of aerated concrete occurs as a result of a chemical reaction between the gas-forming agent and the component actively acting on it with the release of gas. The expansion of the forming micropores continues as a result of the pressure that the gas exerts on the pore walls and the concrete mixture in contact with them. In this work, aluminum powder was used as a gas generator; when interacting with calcium hydroxide, hydrogen is formed.

According to XRD-analysis (Fig. 1), NAC samples – both original and modified – contain the following compounds: quartz (β-SiO_2); tobermorite ($5CaO{\cdot}3SiO_2{\cdot}H_2O$); calcium hydrogarnets ($3CaO\times\times Al_2O_3{\cdot}xSiO_2{\cdot}(6–2x)H_2O$); xonotlite ($6CaO{\cdot}6SiO_2\times\times H_2O$); C–S–H(I); calcite $CaCO_3$.

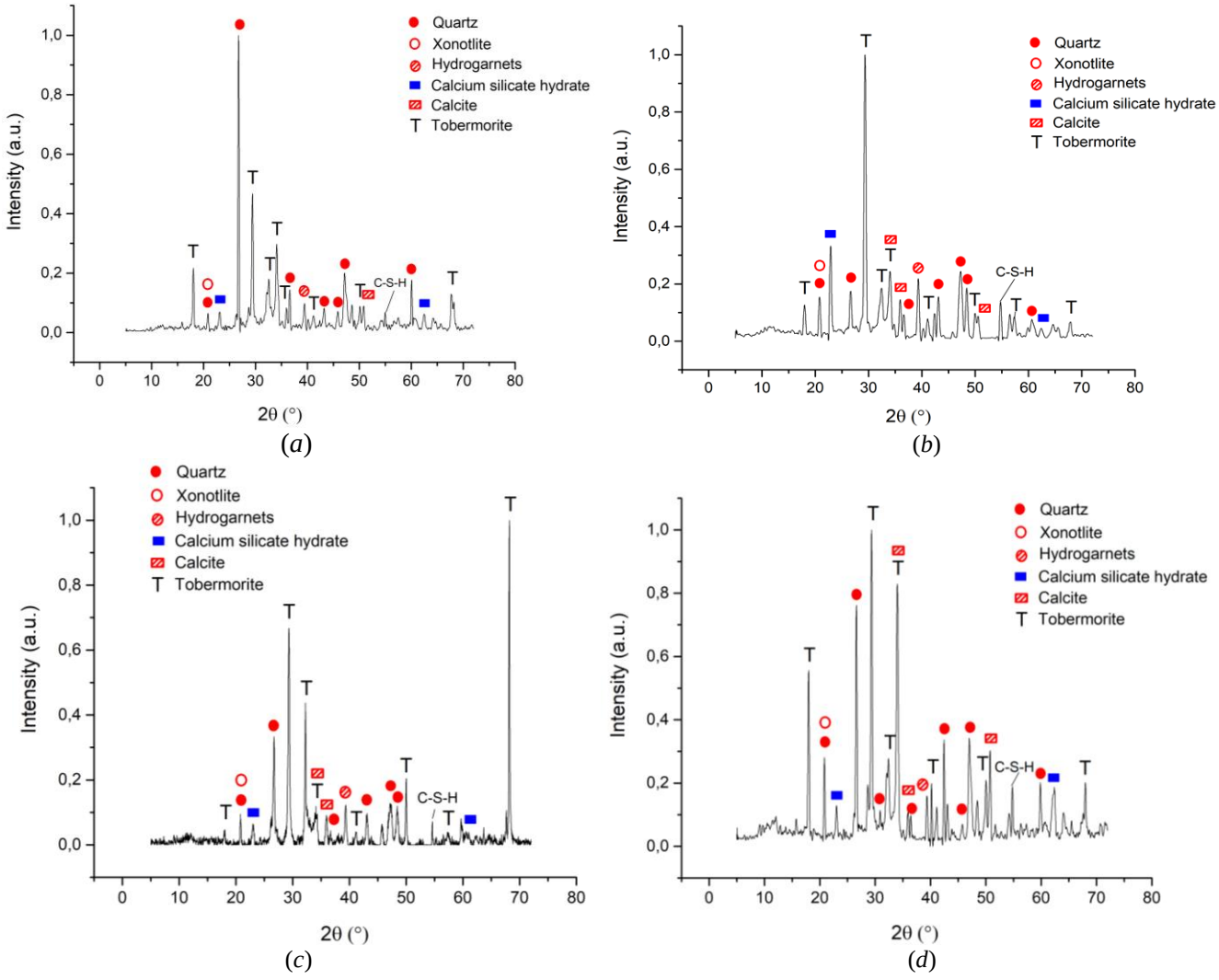

Fig. 1. X-ray diffraction patterns of NAC samples: *a* – control; *b* – modified LS; *c* – modified GO; *d* – modified with a complex additive

The X-ray spectrum of unmodified NAC shows reflections of quartz with high intensity peaks at 2θ = 26.5, 37, 46.5, 60º (Fig. 1, *a*). Peaks of calcium hydrosilicates, such as tobermorite and xonotlite, have a lower intensity value. The resulting diffraction pattern suggests an insufficient degree of crystallinity and heterogeneity of new formations that are formed as a result of hydration of cement stone. Also, the diffraction pattern of the control sample contains low-intensity reflections corresponding to hydrogarnets and calcite.

Aerated concrete samples modified with lignosulfonate (Fig. 1, *b*), according to XRD-analysis, contain large quantities of reflections related to low-basic calcium hydrosilicates, in particular, tobermorite, C–S–H, and xonotlite. It is noteworthy that the introduction of LS significantly increased the intensity of the tobermorite peaks, especially at 2θ = 29º, and the intensity of the quartz peak at 2θ = 26.5º decreased several times. The intensity of the peak corresponding to xonotlite also increased (at 2θ = 21º). The observed changes in the crystalline pattern in gas silicate are explained by the fact that heterogeneous reactions of calcium hydroxide and silicon-containing components in the aerated concrete mixture occur more fully [19].

The introduction of graphene oxide into the composition of the aerated concrete mixture (Fig. 1, *c*) also contributes to a change in the crystal structure of mineral new formations, as does the addition of drugs. Compared to the control sample, the intensity of diffraction reflections of tobermorite, xonotlite and hydrogarnets increases in NAC modified with GO. It is known that calcium hydrogarnets also participate in the structure formation of aerated concrete mixtures and increase the durability of products.

The X-ray diffraction pattern of NAC (Fig. 1, *d*), modified with a complex additive of GO/LS, shows a significant difference in the diffraction pattern of this sample from the previous ones. The difference lies in the high intensity of reflections primarily from tobermorite, xonotlite, as well as C–S–H and calcite. This fact suggests that the mineral new formations formed during the NAC hydration are well crystallized and form a strong framework of hydration products, thereby increasing the strength of aerated concrete due to the strengthening of interpore partitions [19].

The results obtained during the differential thermal analysis of the NAC samples studied in the article are presented in Fig. 2.

According to TG- and DSC-curves, at temperatures up to 100–150 °C, water is removed from low-basic calcium hydrosilicates of the tobermorite type, which is accompanied by heat absorption [12].

A pronounced endothermic effect in the temperature range of 400–450 °C indicates the dehydration of gelled hydrosilicates and calcium hydrogarnets. Small endo-effects at temperatures from 550–900 °C arise due to the decomposition of calcium bicarbonates formed during the carbonization of aerated concrete [21].

In general, the TG- and DSC-curves for all studied materials have a similar character as described above – 3 stages of weight loss, except for the control sample.

Aerated concrete without additives at a temperature of about 100 °C loses 0.96 % of its weight, with the addition of LS – 1.20 %, GO – 1.35 %, complex additive – 1.72 %. An increase in the percentage of residual weight in a given area indicates that the volume of water adsorbed by the sample is increasing. This fact allows us to assert that the introduction of a complex GO/LS additive increases the porosity of aerated concrete [12].

At the next stage in the temperature range of 400–450 °C, which is observed only in modified NAC samples, a slight weight loss of about 1 % is recorded for all samples. Recording of weight loss stops at 1098 °C. Up to this point, in the range of 550–700 °C, the weight of the modified samples decreases by an average of 4 %. The lowest value of the residual weight of NAC is observed for the control sample – 84.47 %, with the addition of LS – 86.40 %, GO – 87.10 %, complex additive – 87.57 %. This fact suggests that NAC with the addition of GO/LS is more durable and less susceptible to thermal influences.

Thus, as a result of X-ray diffraction and differential thermal analysis of cellular concrete samples, the effectiveness of introducing a complex GO/LS additive has been proven, which has a positive effect on the structure formation of cement stone and the morphology of hydration products – new formations of gas silicate.

It should also be noted that the patterns found as a result of the physical and structural analysis are correlated with the data of mechanical tests of the gas blocks under consideration (Table 1) [22].

It has been established that the complex additive allows to obtain the highest values of the mechanical characteristics of NAC: an increase in bending strength by 30 % and compressive strength by 35 % is achieved.

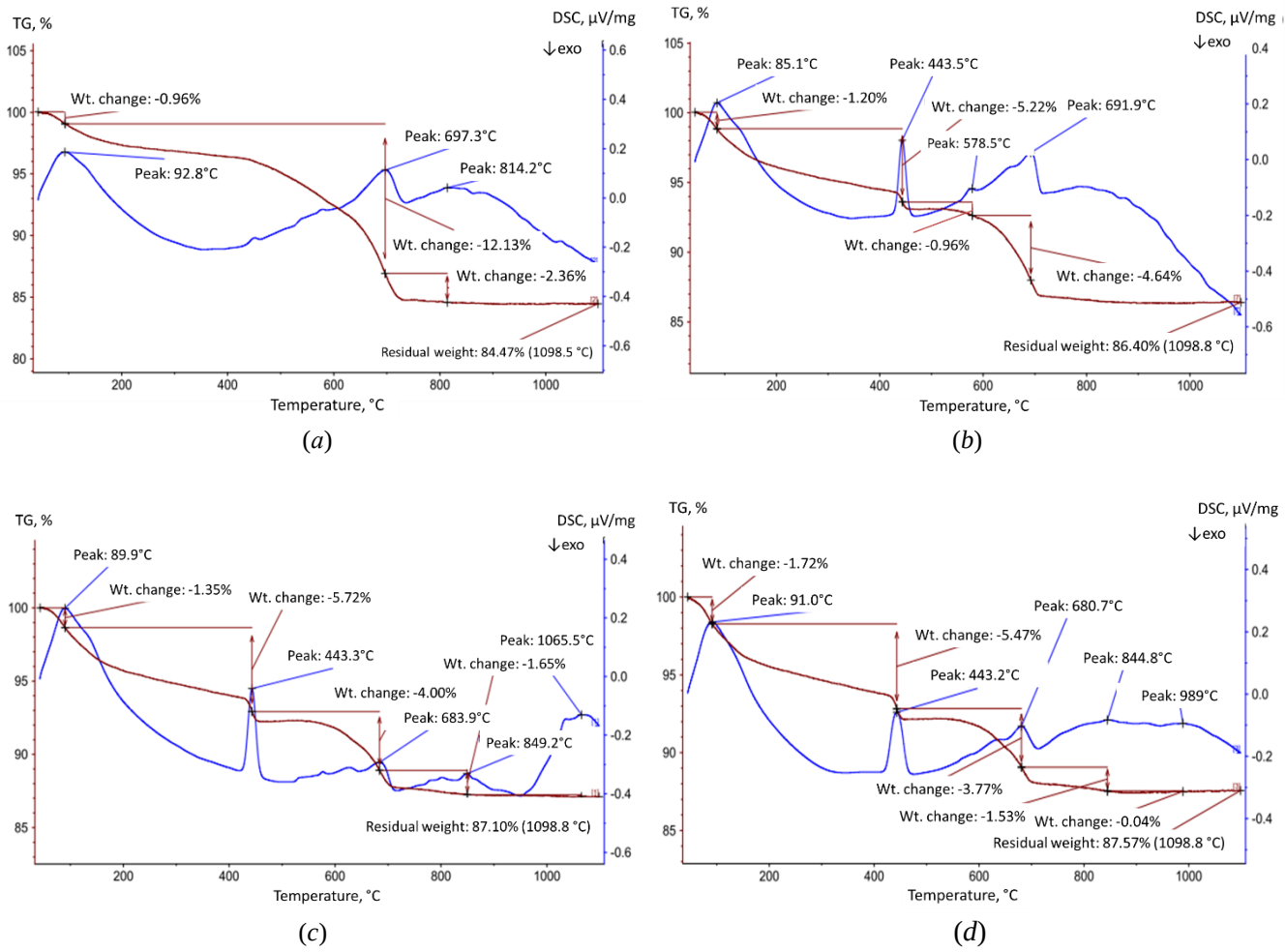

Fig. 2. TG- and DSC-curves of NAC samples: *a* – control; *b* – modified LS; *c* – modified GO; *d* – modified with a complex additive

Table 1. Flexural and compressive strength values of NAC

Samples	Control	LS additive	GO additive	GO/LS additive
Bending strength, MPa	1.15	1.38	1.27	1.67
Compressive strength, MPa	1.25	1.45	1.53	1.92

4. Conclusion

Using the methods of differential thermal and X-ray diffraction analysis, the quantitative and qualitative content of hydration products of cement stone of NAC samples modified with additives of LS, GO and the complex composition of GO/LS was assessed. XRD-analysis made it possible to determine the phase composition of new formations that arise during the hydration of the binder, as well as to evaluate the influence of various types of additives on the NAC crystallization. It was established that all the obtained gas blocks contain quartz, tobermorite-like calcium hydrosilicates, calcium hydrogarnets, xonotlite, C–S–H(I), calcite $CaCO_3$. When modifying NAC with a complex additive of GO/LS, an increase in the reflections of tobermorite, xonotlite and C–S–H(I) is observed, which allows to speak about an increase in the crystallinity degree of new mineral formations of cement stone. According to the results of TG- and DSC-analysis, endothermic effects caused by dehydration of calcium hydrosilicates and destruction of their structure were discovered. The thermograms of all samples revealed three main enfo-effects associated with the removal of adsorbed water from gel-like hydration products, dehydration of low-basic calcium hydrosilicates, and decomposition of calcium carbonate. The obtained XRD and TG/DSC analysis data are in good agreement with strength tests demonstrating the effectiveness of using the complex GO/LS additive for modifying NAC.

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7. Conflict of interests

The authors declare no conflict of interest.

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