

# FORMAREA $\alpha$ -C<sub>2</sub>S HIDRATAT ÎN AMESTECURI CU CaO/SiO<sub>2</sub> = 1,75 PRIN TRATAMENT HIDROTERMAL LA 200°C

## FORMATION OF $\alpha$ -C<sub>2</sub>S HYDRATE IN THE MIXTURES WITH CaO/SiO<sub>2</sub> = 1.75 BY HYDROTHERMAL TREATMENT AT 200 °C

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*The parameters of  $\alpha$ -C<sub>2</sub>SH hydrothermal synthesis from a non-stoichiometric composition of CaO and SiO<sub>2</sub>·nH<sub>2</sub>O mixtures (the molar ratio CaO/SiO<sub>2</sub> = 1.75) in unstirred suspensions were determined and a sequence of intermediary compounds formation was examined and explained. It was evidenced that  $\alpha$ -C<sub>2</sub>SH is formed already after 4 h of hydrothermal treatment at 200 °C. However, together with this compound a fair amount of some intermediary compounds (C-S-H(I) and C-S-H(II)) are formed. It was determined that after 24 h of hydrothermal treatment,  $\alpha$ -C<sub>2</sub>SH starts to recrystallized into kilchoanite which dominates in the products after extending the duration (48-72 h) of synthesis.*

**Keywords:**  $\alpha$ -C<sub>2</sub>S hydrate; calcium silicate hydrate (C-S-H); hydrothermal synthesis; kilchoanite

### 1. Introduction

Recently, synthesis, properties and structure of  $\alpha$ -C<sub>2</sub>S hydrate ( $\alpha$ -C<sub>2</sub>SH) – a mostly common compound formed during a hardening of autoclaved calcium silicate products, have obtained a new interest, because on its basis a new hydraulic cementitious materials family has been created [1,2].

$\alpha$ -C<sub>2</sub>S hydrate ( $\alpha$ -C<sub>2</sub>SH) was first discovered in steam-cured Portland cement mortars by Thorvaldson and Shelton (1929) [3], who also described its optical properties. It has since been prepared under hydrothermal conditions either from calcium oxide and silica gel or from anhydrous dicalcium silicates [4]. L. Heller (1952) [5] found that under the conditions studied (100–200 °C; up to 7 days) the initial product was usually calcium silicate hydrate (I). Further treatment gave  $\alpha$ -C<sub>2</sub>SH and finally hillebrandite and/or afwillite.

G. L. Kalousek et al. (1952) [6] studied the effects of two forms of silica, quartz and silicic acid, the methods of mixing, and autoclaving conditions on the formation of the lime-rich crystalline calcium silicate hydrates designated as alpha-, beta-, and gamma-type hydrates.  $\alpha$ -C<sub>2</sub>SH was synthesized from mixtures of lime slurry and quartz in compositions with 1.8 to 2.4 CaO/SiO<sub>2</sub> molar ratios. However, this series of compounds, free of all contaminants, showed X-ray patterns containing many or nearly all lines of crystalline Ca(OH)<sub>2</sub>.

Many studies have been made regarding the synthesis of  $\alpha$ -C<sub>2</sub>SH [7-9]. Synthesis is also possible from lime and quartz [9,10] or from lime and colloidal silica [11]. According to several

sources [12,13],  $\alpha$ -C<sub>2</sub>SH forms quite easily under hydrothermal conditions from CaO and silicic acid as starting materials in the temperature range 150–200 °C. Crystallization from quartz and Ca(OH)<sub>2</sub> is very slowly. The best results were obtained using C<sub>2</sub>S polymorphs as the starting material [8,14]. As indicated by H. F. W. Taylor [4] the rudiments of crystals aids the formation of  $\alpha$ -C<sub>2</sub>SH under hydrothermal conditions.

$\alpha$ -C<sub>2</sub>SH is produced primarily in the process used to form 1.1 nm tobermorite when a suspension of calcium hydroxide and fairly large size quartz is hydrothermally treated at about 433 K. The thermal and spectroscopic properties of this phase have been reported in [15,16]. Whereas the reassignment of powder diffraction peaks has been reported, the crystal structure has not been refined since Heller's original study [5].

L. Black et al. (2003) [17] have prepared by hydrothermal treatment, a number of crystalline calcium-silicate-hydrate (C-S-H) phases, with calcium–silicon ratios varying from approximately 0.5 (K-phase) to 2.0 (hillebrandite and  $\alpha$ -dicalcium silicate hydrate). They shown that X-ray photoelectron spectroscopy (XPS) can be used to obtain valuable structural information for C-S-H phases.

Thus, the formation of  $\alpha$ -C<sub>2</sub>SH from different mixtures (with molar ratio C/S = 2) is a complex and long-term process because of a low solubility of CaO-containing compounds and/or reactivity of raw materials. In order to accelerate the interreaction between intermediary compounds in the CaO–amorphous SiO<sub>2</sub>–H<sub>2</sub>O system and to avoid unreacted portlandite in the products, the

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molar ratio (C/S) of initial mixture was reduced till 1.75.

The main objective of the present work was to determine the parameters of  $\alpha$ -C<sub>2</sub>SH hydrothermal synthesis as well as to analyze and explain the sequence of intermediary compounds formation.

## 2. Materials and methods

In this paper the following reagents were used: - fine ground SiO<sub>2</sub>·nH<sub>2</sub>O („Reaktiv“, Russia), having a specific surface area  $S_{sp} = 1309 \text{ m}^2/\text{kg}$  determined with CILAS LD 1090 granulometer; the loss of ignition – 5.19%; - calcium oxide which has been produced by burning of calcium hydroxide („Reaktiv“, Russia) at 550 °C for 1 hour and ground to a specific surface area  $S_a = 1673 \text{ m}^2/\text{kg}$  determined with CILAS LD 1090 granulometer; the quantity of free CaO was equal to 98.7%.

The molar ratio C/S (CaO/SiO<sub>2</sub>) of initial mixture was equal to 1.75. Homogenized raw materials were mixed with distilled water to obtain water/solid ratio of suspension equal to 10. The hydrothermal synthesis has been carried out in unstirred suspensions in 25 ml volume PTFE cells, which were placed in „Parr instruments“ (Germany) - autoclave, under saturated steam pressure at 200 °C temperature, for 8; 16; 24; 48 and 72 hours. Temperature of 200 °C was reached in two hours' time. The resulted product was filtered, rinsed with acetone to prevent carbonation process of materials, dried at  $50 \pm 5 \text{ °C}$  for 24 h and sieved through a sieve with a mesh width of 80  $\mu\text{m}$ .

The X-ray powder diffraction (XRD) data were obtained with a DRON-6 X-ray diffractometer with Bragg-Brentano geometry using Ni-filtered Cu K <sub>$\alpha$</sub>  radiation and graphite monochromator, operating with the voltage of 30 kV and emission current of 20 mA. The step-scan covered the angular range 2–60° ( $2\theta$ ) in steps of  $2\theta = 0.02^\circ$ .

Simultaneous thermal analysis (STA: differential scanning calorimetry–DSC and thermogravimetry–TG) was also used for measuring the thermal stability and phase transformation of samples at a heating rate of 15°C/min; the temperature ranged from 30 °C up to 1000 °C under air atmosphere. The test was carried out on a Netzsch instrument STA 409 PC Luxx. The ceramic sample handlers and crucibles of Pt-Rh were used.

FT-IR spectra have been carried out with the help of a Perkin Elmer FT-IR Spectrum X system. Specimens were prepared by mixing 1 mg of the synthesised product with 200 mg of KBr. The spectral analysis was performed in the range of 4000–400  $\text{cm}^{-1}$  with spectral resolution of 1  $\text{cm}^{-1}$ .

## 3. Results and discussions

It was determined that by using unstirred CaO–SiO<sub>2</sub>·nH<sub>2</sub>O–H<sub>2</sub>O suspensions, cured for 4

hours under saturated steam pressure at 200 °C, dicalcium silicate hydrate –  $\alpha$ -C<sub>2</sub>SH (2CaO·SiO<sub>2</sub>·H<sub>2</sub>O ( $d$ -spacing–0.425; 0.321; 0.268; 0.245 nm), C-S-H(II) ( $d$ -spacing–0.303; 0.280; 0.184 nm) and a low basic semi-crystalline C-S-H(I) type ( $d$ -spacing–0.303; 0.280; 0.184 nm) were formed (Fig. 1, curve 1). It should be noted that under these synthesis conditions, the basic reflections ( $d$ -spacing–0.493; 0.193; 0.179 nm) of partially unreacted portlandite was observed on X-ray diffraction pattern. Meanwhile, alongside mentioned maximums a broad basal reflection, without peaks, in 26 – 37° diffraction angles range was noticed and can be assigned to amorphous structure compounds. It should be noted that diffraction peaks ( $d$ -spacing – 0.304, 0.191 nm) typical to calcium carbonate are also identified on XRD curve, indicating that carbonization of synthesis products proceeded.

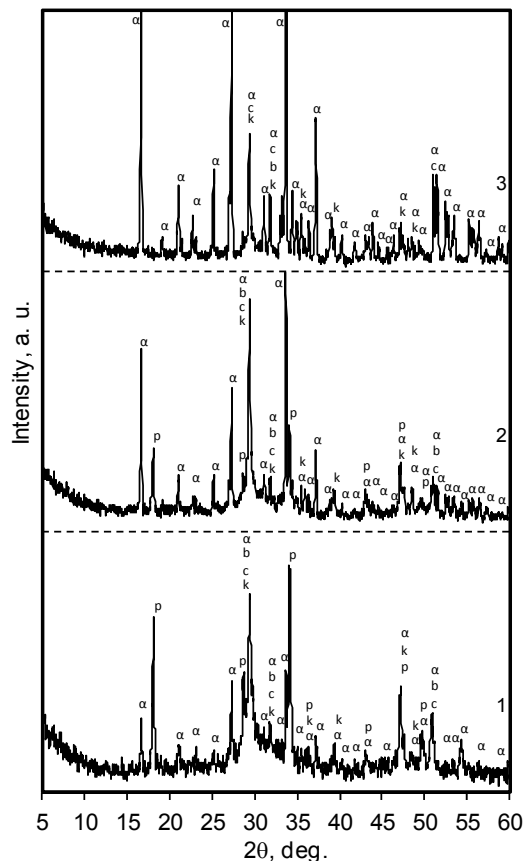


Fig. 1 - XRD patterns of synthesis products in the mixtures with C/S molar ratio of 1.75, when the duration of hydrothermal treatment at 200 °C temperature is, h: 1 – 4; 2 – 8; 3 – 16. Indices: c – C-S-H(I);  $\alpha$  –  $\alpha$ -C<sub>2</sub>S hydrate; p – Ca(OH)<sub>2</sub>; k – CaCO<sub>3</sub>, b – C-S-H(II)

The previous results were confirmed by STA analysis data. On DSC curve, an endothermic effect at  $\sim 448 \text{ °C}$  can be attributed to the dehydration of  $\alpha$ -C<sub>2</sub>S hydrate as well as to the decomposition of portlandite. During these reactions, a large amount of heat (70.59 J/g) is absorbed, which proves that dibasic calcium silicate hydrate and portlandite were obtained in

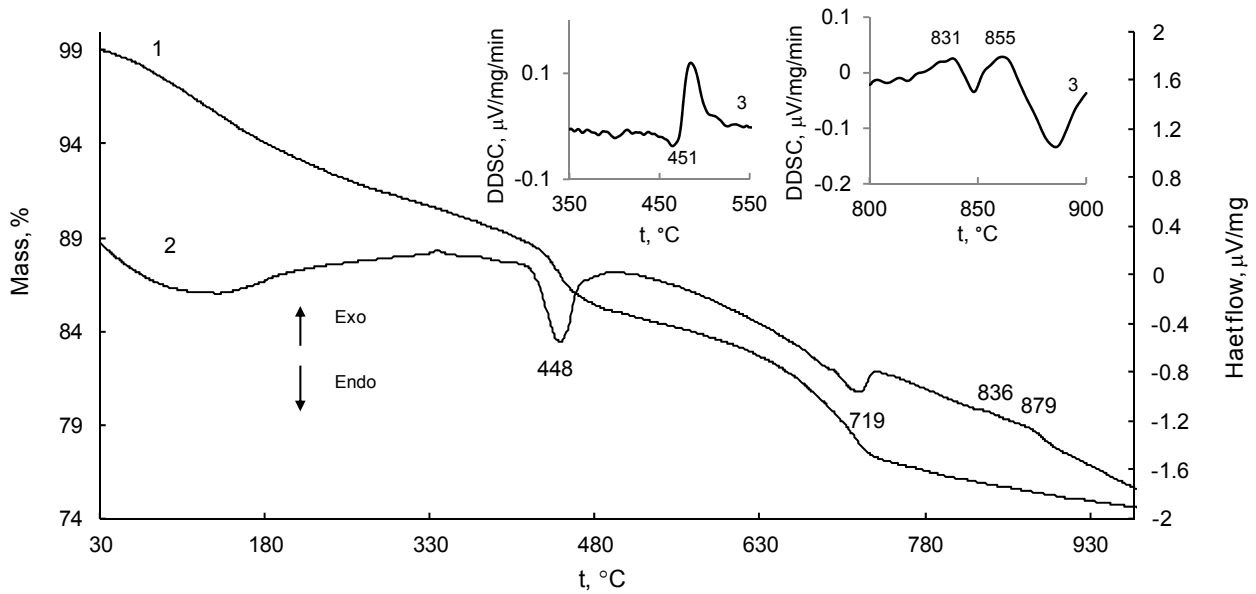


Fig. 2 - TGA (curve 1), DSC (curve 2), DDSC (curve 3) curves of synthesis products, when the duration of hydrothermal treatment at 200°C temperature is 4 h.

the synthesis products (Fig. 2, curves 2 and 3). Also, two very small exothermic effects at ~836 and ~879 °C, characteristic to some crystallisation processes (semi-crystalline C-S-H(I) and C-S-H(II) respectively), were identified (Fig. 2, curve 2). It was calculated that, during mentioned process, the quantity of the heat flow was equal to: 1.01 J/g – for C-S-H(I) and 3.34 J/g – for C-S-H(II) (Fig. 2, curve 2, Table 1). Also, an endothermic effect at ~719 °C was assigned to the decomposition of calcium carbonate (Fig. 2, curve 2). According to TGA analysis data, it was calculated that 6.3 % of the latter compound is present in the synthesis products (Fig. 2, curve 1).

Table 1

The heat of thermal effects typical for  $\alpha$ -C<sub>2</sub>SH, C-S-H(I) and C-S-H(II)

Hydrothermal synthesis duration, h	Heat of thermal effect, J/g		
	$\alpha$ -C <sub>2</sub> SH	C-S-H(I)	C-S-H(II)
4	70.59	1.01	3.34
8	83.75	1.48	4.31
16	157.20	2.78	0.24
24	117.00	4.35	-
48	75.86	1.88	-
72	-	0.98	-

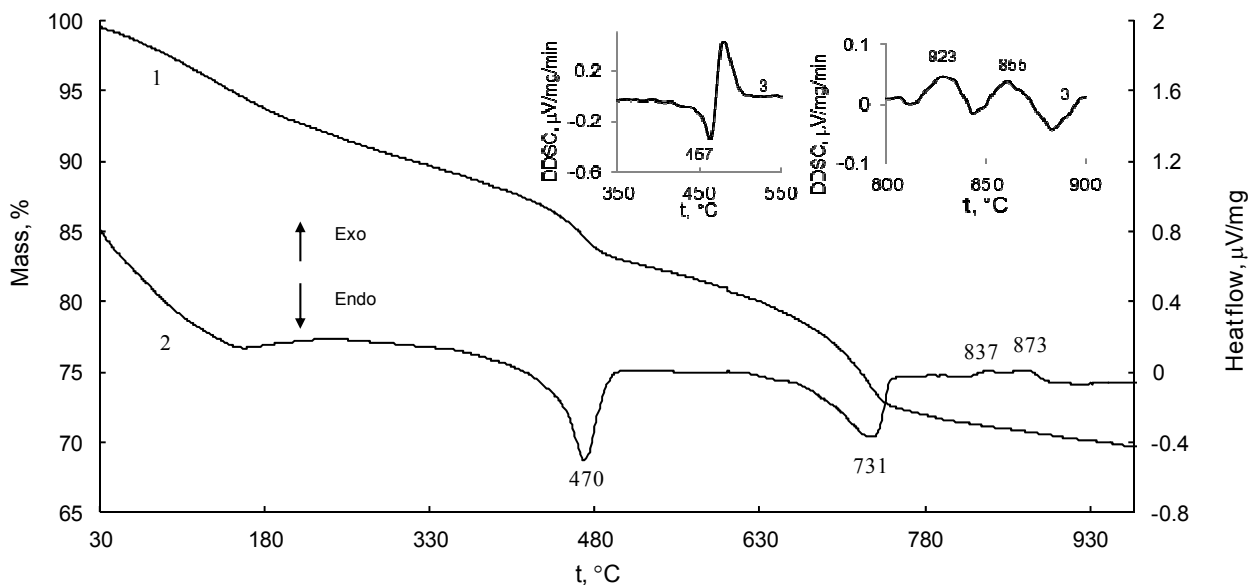


Fig. 3 - TGA (curve 1), DSC (curve 2), DDSC (curve 3) curves of synthesis products, when the duration of hydrothermal treatment at 200°C temperature is 8 h.

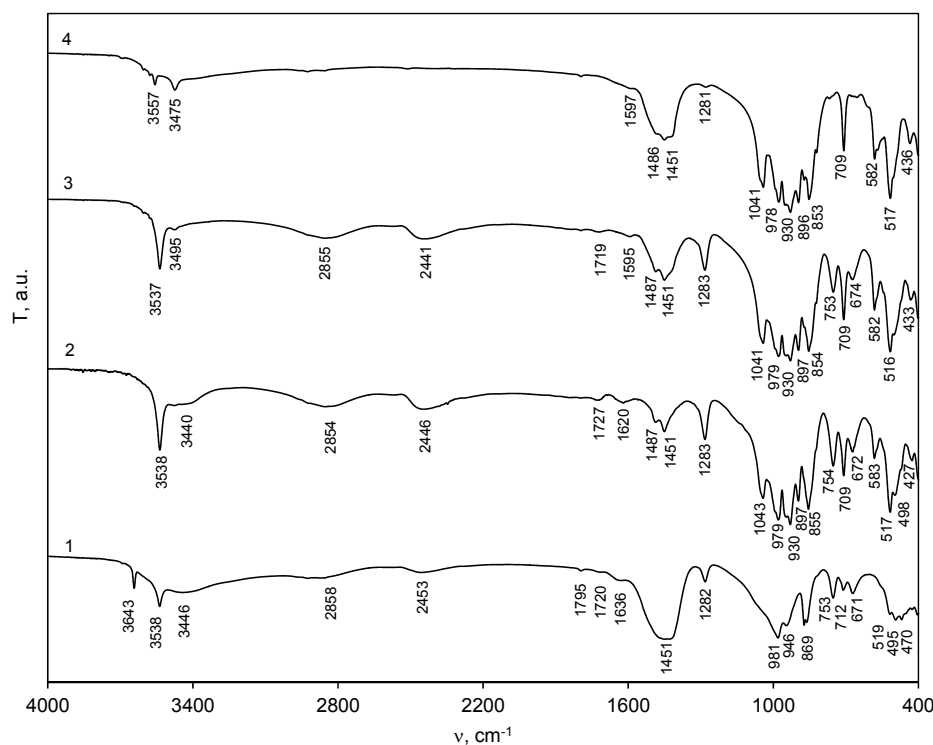


Fig. 4 - FT-IR spectra of synthesis products, when the duration of hydrothermal treatment at 200 °C was, h: 1 – 8; 2 – 24; 3 – 48; 4 – 72.

After 8 h of hydrothermal treatment, STA analysis data showed that, at ~470 °C, the amount of adsorbed heat attributed to dehydration of  $\alpha$ -C<sub>2</sub>S hydrate and portlandite was increased to 83.75 J/g (Fig. 3, curve 2, Table 1). This fact allows to state that a greater quantity of  $\alpha$ -C<sub>2</sub>S hydrate was formed in the synthesis products. It was also supported by XRD results, which indicated that the amount of formed portlandite is significantly lower. Moreover, at higher temperatures, the trivial quantity of released heat assigned to C-S-H(I) (~837 °C) and C-S-H(II) (~873 °C) recrystallization processes is observed (Fig. 3, curve 2). A sharp endothermic effect at ~730 °C temperature characteristic to the decomposition of calcium carbonate is detected and according to TGA analysis data, it was estimated, that 15.5 % of the latter compound is formed in the synthesis products (Fig. 3, curves 1 and 2).

Furthermore, the FT-IR spectrum of synthesis products after 8 h of hydrothermal treatment shows that in symmetrical  $\nu_s(\text{O-SiO})$  stretching vibration range the doublet at 946 and 981 cm<sup>-1</sup> is clearly seen (Fig. 4, curve 1). Meanwhile, the identified absorption band at 3643 cm<sup>-1</sup> belonging to HO<sup>-</sup> groups supports the XRD analysis data, which indicate that portlandite is not fully reacted.

The interaction degree between raw materials is greater when the duration of isothermal treatment is prolonged to 16 h: the initial formed compound – portlandite – is fully reacted, so that typical diffraction peaks for this compound are not detected on XRD pattern. It should be noted that a

significant increase in the intensity of  $\alpha$ -C<sub>2</sub>S hydrate diffraction maximums is observed; while a broad basal reflection, without peaks, in 26 – 37° diffraction angles range disappeared (Fig. 1, curve 3). STA analysis data showed that the amount of heat at ~475 °C, which can be assigned to the dehydration of  $\alpha$ -C<sub>2</sub>S hydrate, increased in two times (Fig. 5, curve 2). It should be underlined that semi-crystalline C-S-H(II) becomes metastable, because only a small exothermic effect at ~866 °C is observed (Fig. 5, curve 1 and 2; Table 1). Moreover, the thermal effect intensity of C-S-H(I) recrystallization to wollastonite at ~843 °C increased in 2 times (from 1.48 till 2.78 J/g), while the carbonation degree is smaller, so that only 7.5 % of calcium carbonate is formed (Fig. 5, curve 1 and 2; Table 1).

The FT-IR spectrum showed no significant changes, only the multiplicity of absorption maximums is shifted and the absorption band at 3630-3660 cm<sup>-1</sup> attributed to HO<sup>-</sup> groups in portlandite disappeared.

Furthermore, after 24 h of hydrothermal treatment,  $\alpha$ -C<sub>2</sub>S hydrate recrystallization to anhydrous kilchoanite proceeded: typical to the latter compound diffraction maximums (*d*-spacing – 0.355; 0.304; 0.288; 0.267 nm) are identified (Fig. 6, curve 1). It should be noted that diffraction peaks of  $\alpha$ -C<sub>2</sub>S hydrate are still very intensely.

XRD results are supported by STA analysis data. The heat flow of the latter compound dehydration is decreased from 157.2 to 117 J/g (Table 1). Moreover, a trivial increase in the heat flow of C-S-H(I) recrystallization (4.35 J/g)

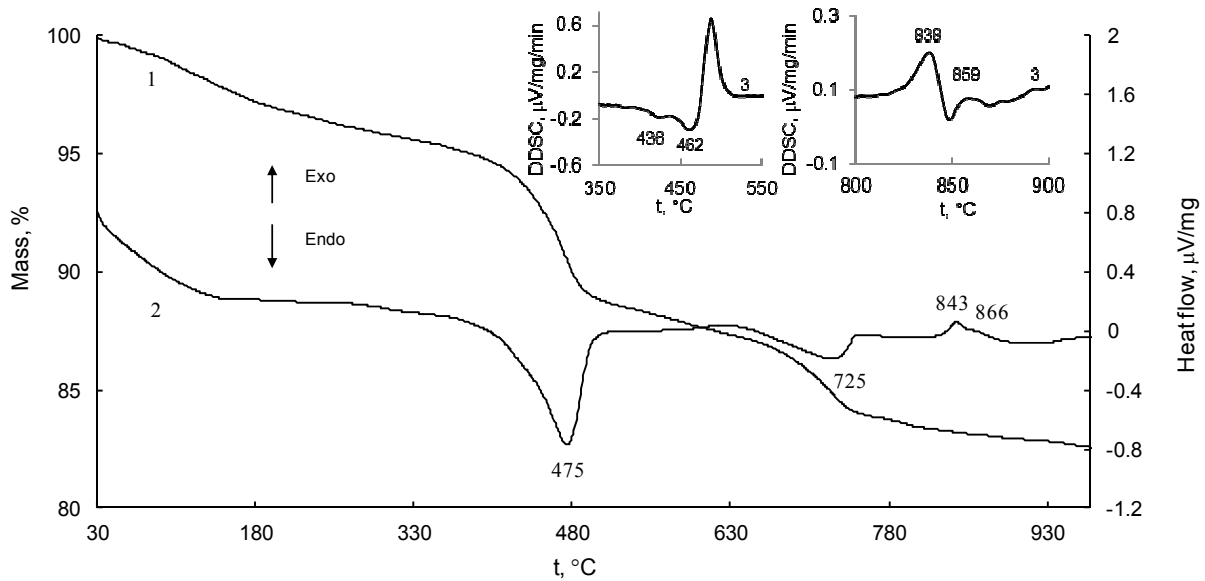


Fig. 5 - TGA (curve 1), DSC (curve 2), DDSC (curve 3) curves of synthesis products, when the duration of hydrothermal treatment at 200°C temperature is 16 h.

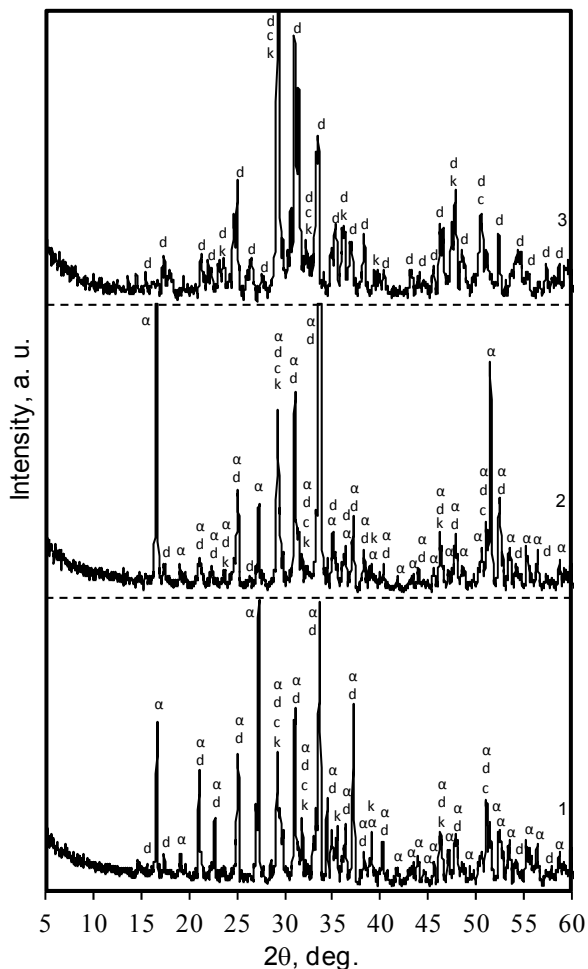


Fig. 6 - XRD patterns of synthesis products in the mixtures with C/S molar ratio of 1.75, when the duration of hydrothermal treatment at 200 °C temperature is, h: 1 - 24; 2 - 48; 3 -72. Indices: c - C-S-H(I);  $\alpha$  -  $\alpha$ -C<sub>2</sub>S hydrate; d - kilchoanite; k - CaCO<sub>3</sub>, b - C-S-H(II)

is observed, while an exothermic effect typical to C-S-H(II) is not identified. It should be underlined

that the amount of calcium carbonate was decreased in 3 times (till 2.6 %) as shows the mass loss equal to 1.14 % (Fig. 7, curve 1).

It can notice that after 24 h of hydrothermal treatment FT-IR spectrum of synthesis products is significantly changed, concerning Si-O vibrations (Fig. 4, curve 2), in comparison with absorption bands obtained after 8 h of treatment (Fig. 4, curve 1). In bending  $\delta$ (Si-O-Si) and  $\delta$ (O-Si-O) signals range, two absorption bands at ~427 and ~583 cm<sup>-1</sup> as well as a duoblet at ~498, ~517 cm<sup>-1</sup> are identified. In higher frequency interval (890-1000 cm<sup>-1</sup>), the absorption maximums are assigned to symmetrical  $\nu_s$ (O-SiO-) stretching vibrations; in this particular region a triplet at ~897, ~930 and ~979 cm<sup>-1</sup> is detected. The absorption band at ~1043 cm<sup>-1</sup>, which can be attributed to asymmetrical  $\nu_{as}$ (Si-O-Si) stretching signals, indicates that [SiO<sub>4</sub>]<sup>4-</sup> tetrahedron forms a line in the structure of synthesis products. Due to a large amount of formed  $\alpha$ -C<sub>2</sub>S hydrate, the absorption maximums, corresponding to OH<sup>-</sup> and H<sub>2</sub>O vibrations, did not appear to change. It should be noted that absorption bands in 1400-1500 cm<sup>-1</sup> frequency range, assigned to  $\nu$ (C - O<sub>3</sub><sup>2-</sup>) link form a doublet, meanwhile the absorption maximum at 855 cm<sup>-1</sup> is not even changed. Moreover, the intensity of a doublet at ~1451 and ~1487 cm<sup>-1</sup>, which was mentioned before, decreases in 2-3 times.

It was determined that after 48 h of hydrothermal treatment, there are still a large amount of  $\alpha$ -C<sub>2</sub>S hydrate in the system: the intensive diffraction peaks characteristic to this compound are identified on XRD pattern (Fig. 6, curve 2). Moreover, the intensity of diffraction maximums, corresponding to *d*-spacing - 0.532 and 0.266 nm, respectively (16.68° and 33.65°) increases disproportionately; this can be explained by the reorientation of the plates, during  $\alpha$ -C<sub>2</sub>S hydrate recrystallization to kilchoanite. The

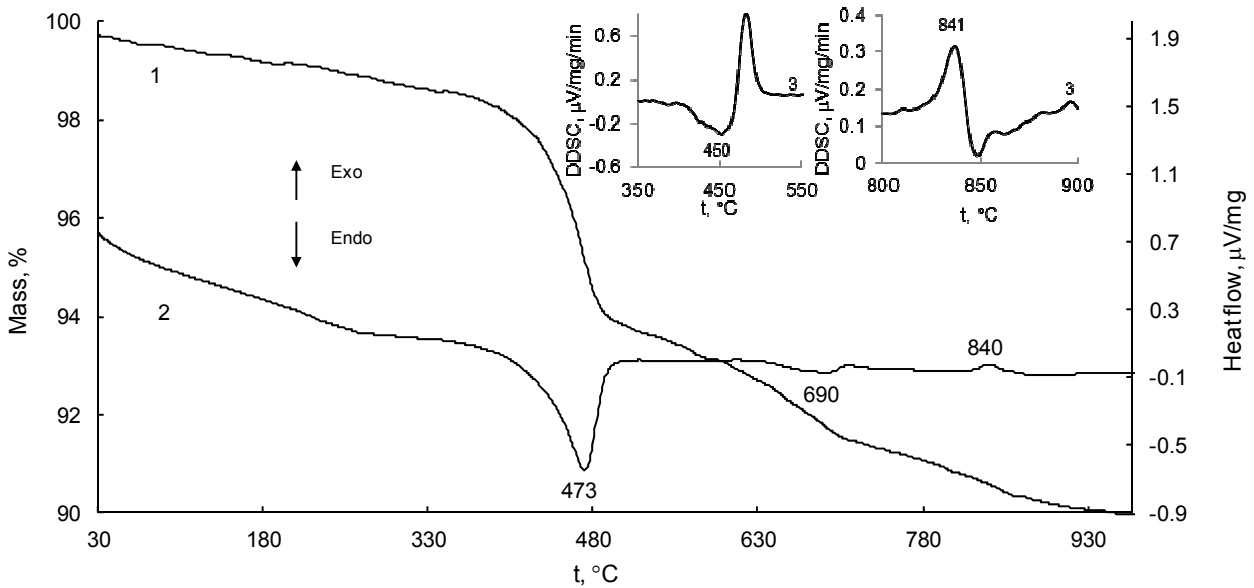
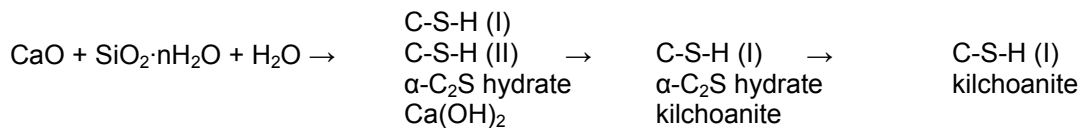


Fig. 7 - TGA (curve 1), DSC (curve 2), DDSC (curve 3) curves of synthesis products, when the duration of hydrothermal treatment at 200°C temperature is 24 h



investigation of FT-IR spectrum showed that the intensity of absorption bands attributed to OH<sup>-</sup> and H<sub>2</sub>O vibrations is decreased, meanwhile only the amplitude of absorption maximums assigned to Si-O signals is changed (Fig. 4, curve 3).

When the duration of synthesis is extended to 72 h, a full  $\alpha$ -C<sub>2</sub>S hydrate recrystallization to kilchoanite could be noticed: diffraction peaks characteristic to  $\alpha$ -C<sub>2</sub>S hydrate disappeared in XRD curves (Fig. 6, curve 3). Meanwhile, in FT-IR spectrum, adsorption bands, reflecting HO<sup>-</sup> and H<sub>2</sub>O vibrations (2855 cm<sup>-1</sup> and 2441 cm<sup>-1</sup>) disappeared (Fig. 4, curve 4).

It is possible to assume that when the mixture of CaO and amorphous SiO<sub>2</sub> is hydrothermal treated at 200 °C under saturated steam pressure, the reactions of a new compounds formation occur in the sequence above.

Thus,  $\alpha$ -C<sub>2</sub>S hydrate can be synthesized from a non-stoichiometric composition (the molar ratio C/S = 1.75) of CaO and SiO<sub>2</sub>·nH<sub>2</sub>O in unstirred suspensions.  $\alpha$ -C<sub>2</sub>SH without impurities of portlandite is formed after 16 h at 200 °C. It starts to recrystallize into kilchoanite after 24 h of treatment. In the future work, the synthesized  $\alpha$ -C<sub>2</sub>S hydrate will be used for prepare of some hydraulic cementitious materials.

#### 4. Conclusions

1. It was determined that in unstirred CaO–SiO<sub>2</sub>·nH<sub>2</sub>O–H<sub>2</sub>O suspensions when the molar ratios CaO/SiO<sub>2</sub> of primary mixture was equal to 1.75, for 4 hours of isothermal curing at 200 °C dicalcium silicate hydrates –  $\alpha$ -C<sub>2</sub>SH, C-S-H(II) and a low basic semi-crystalline C-S-H(I) were formed.
2.  $\alpha$ -C<sub>2</sub>SH without impurities of portlandite is formed after 16 h. It starts to recrystallize into kilchoanite after 24 h of treatment. For this reason, the heat flow of  $\alpha$ -C<sub>2</sub>SH dehydration decreased from 157.2 J/g to 117 J/g. Furthermore, after extending the duration of isothermal treatment to 72 h,  $\alpha$ -C<sub>2</sub>S hydrate is fully recrystallized into kilchoanite. As a result, the thermal conversions attributed to the removal of crystallization water as well as a new formed synthesis product – kilchoanite are not observed in DSC curve.

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## MANIFESTĂRI ȘTIINȚIFICE / SCIENTIFIC EVENTS

### ECRA Seminar on Alternative Fuels:

### Quality and Environmental Control

**Date: 08/08/2014 - 09/08/2014**

**Location: Retznei (Austria)**



The environmentally safe and sound recovery of alternative materials has been taking place in the international cement industry for many years. On the one hand there is the necessity to substitute fossil fuels and natural raw materials with suitable alternative materials. On the other hand, the environmentally safe and sound operation of the process must be safeguarded. Moreover, the product quality as such has to be taken into account. The amount of alternative fuels has been continually increasing over the past few years and maintaining quality surveillance for these materials is of great importance. The seminar will begin with an overview of the current European legislative requirements. In this context, the existing international standards will also be examined. Furthermore, the seminar will summarise how good product quality as well as a good environmental performance of the production process can be assured when alternative materials are used. Aspects of occupational health and safety will also be covered. The seminar includes a visit to the Lafarge cement plant in Retznei, Austria.

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