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THE CONDITIONS OF FORMATION AND STABILITY OF THE CARBONATE COMPLEX ($C_3A \cdot CaCO_3 \cdot nH_2O$) UNDER HYDROTHERMAL CONDITIONS

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The carbonate complex ($3\,CaO \cdot Al_2O_3 \cdot CaCO_3 \cdot nH_2O$) *was found to be formed in the optimum way under hydrothermal conditions (at 181 °C) in the presence of* $Ca(OH)_2$ *in a paste mix consistency. The presence of β-quartz, power station flyash, cement or 11 Å-tobermorite affects negatively formation of the complex, and when this has already existed in the system, the components even show a decomposing effect. The results obtained have confirmed the earlier assumption in the sense that the carbonate complex cannot be formed under the conditions of the manufacturing technology of high-pressure cured lime-silicate materials including asbestos cement.*

INTRODUCTION

In the production of high-pressure cured building materials, use is made of some raw-material components, above all lime or quartz sand, which usually contain variable amounts of calcite. The present study had the aim to determine whether in the course of the autoclave process in the production of cellular concrete or asbestos cement or other materials, the $CaCO_3$ present may react with C_3A producing the carbonate complex $C_3A \cdot CaCO_3 \cdot nH_2O$. It was also thought possible that formation of this complex may preclude the occurence of the undesirable hydrogarnate component ($C_3AS_xH_{6-2x}$), which has been proved [1], [2] to have no substantial effect on the physico-mechanical properties of high-pressure cured products. Dosch and H. zur Strassen [3] reported that the complex formed under hydrothermal conditions and designated as "monocarbonate" has the probable composition $C_3A \cdot CaCO_3 \cdot 11\,H_2O$. Spohn and Lieber [4] have proved that $CaCO_3$ combines with $C_3A(C_4AF)$ in the form of suspension or paste producing the component $C_3A \cdot CaCO_3 \cdot 11\,H_2O$. The phase of this composition was studied in particular by Turriziani and Schippa [5], Seligman and Greening [6], [7], Budnikov et al. [8], Greene [9] and Feldman et al. [10]. Ludwig and Schwiete [11] have summarized the results of the works published so far on the subject. This summary indicates that most of the authors assume the carbonate complex to contain $10-11\,H_2O$ molecules. On the other hand, Jones and Roberts [12] report that the phase contains $12\,H_2O$ molecules. Šauman et al. [13] have synthetized the complex and found $11\,H_2O$ molecules; however, they have not confirmed the presence of this phase in quartz sand-cement mixes with stepped up $CaCO_3$ addition, which have been autoclaved at 181 °C. Jambor [14] has pointed out the superior binding properties of the carbonate complex compared to those of the hydrogarnate phase or calcium hydroaluminates under cement hydration conditions.

EXPERIMENTAL

Specimen preparation and the methods employed

The carbonate complex (C_3A . $CaCO_3$. 11 H_2O) was prepared from high purity C_3A and precipitated $CaCO_3$ A.R. (Merck). The basic equimolar mix prepared from finely dispersed components in the weight ratio C_3A : $CaCO_3$ = 2.7 : 1 was subjected to hydration in the form of suspension and in that of paste in high-pressure cylinders. The suspension was composed of 1 part of solids and 10 parts of the liquid phase (reboiled distilled water). The cylinders containing the suspension were revolved in the course of isothermal hydration, and the hydration products were dried in flowing nitrogen at 105 °C for 8 hours.

In some instances the suspension was allowed to stand for a longer period of time at 25 °C, and after filtration the precipitate was washed with aceton and ether. The specimens were investigated by X-ray diffraction (Mikro 111, Philips, Netherlands), differential and gravimetric thermal analysis (Derivatograph, Orion, Budapest), infrared spectral analysis (Perkin Elmer, Infracord 337) and scanning electron microscopy (Stereoscan, type 2A, Cambridge, England).

ANALYSIS OF THE RESULTS OBTAINED

Determination of the optimum reaction conditions

Hydration of the equimolar mixture of C_3A and $CaCO_3$ in the form of paste and suspension at 181 °C for 24 hours of isothermal heating yielded products, the X-ray analysis of which is shown in Fig. 1.

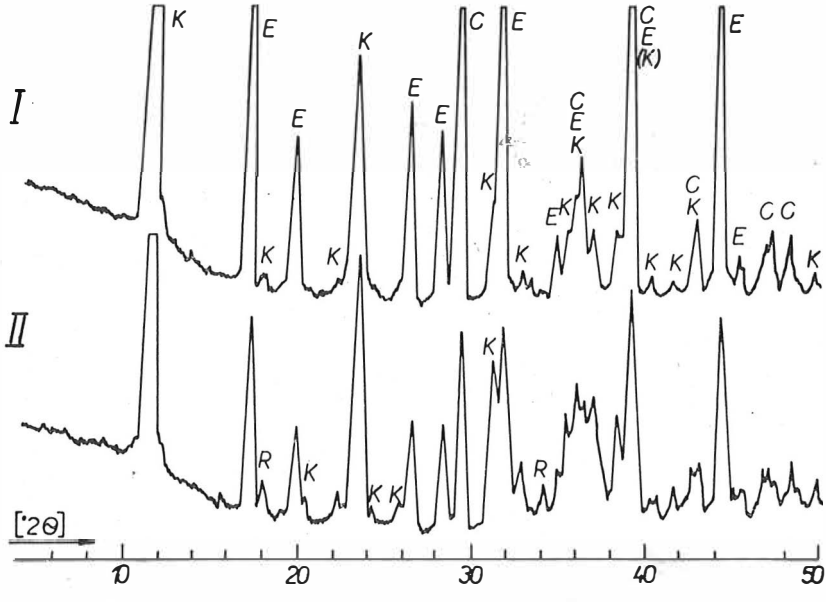

Fig. 1. X-ray diffraction patterns of equimolar mixtures of C_3A *and* $CaCO_3$, *24 hrs/181° C, I — suspension, II — paste. Symbols employed: C — calcite, E —* C_3AH_6, *K —* C_3A . $CaCO_3$. 11 H_2O, *Q — ß — quartz, R —* $Ca(OH)_2$.

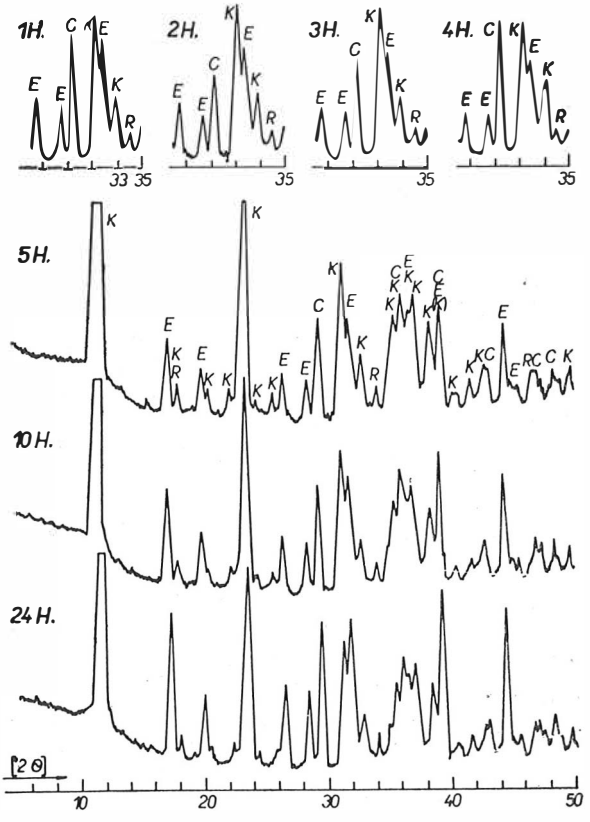

Fig. 2. X-ray diffraction patterns of the equimolar mixture of C_3A *and* $CaCO_3$ *hydrated at 181°C with various lengths of isothermal dwell.*

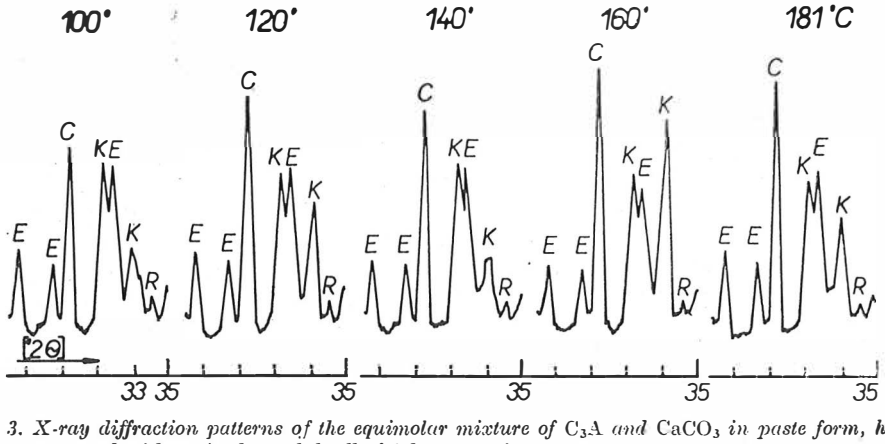

Fig. 3. X-ray diffraction patterns of the equimolar mixture of C_3A *and* $CaCO_3$ *in paste form, hydrated with an isothermal dwell of 2 hrs at various temperatures.*

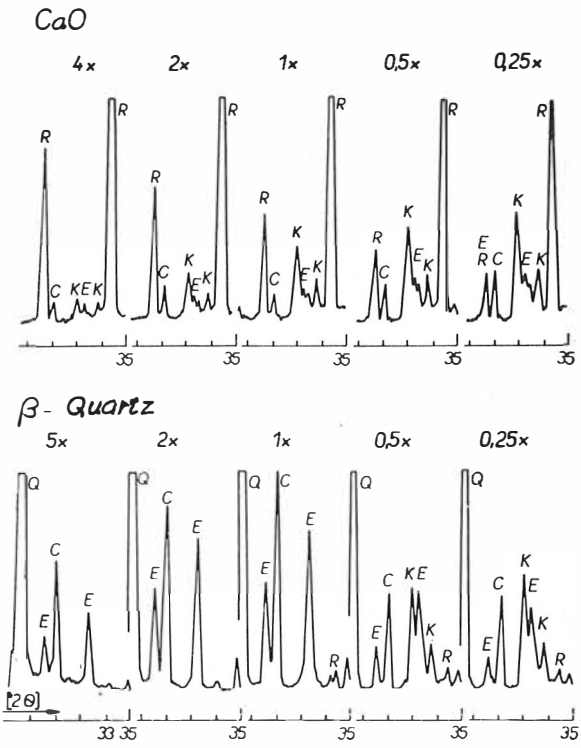

Fig. 4 X-ray diffraction pattern of the equimolar mixture of C_3A *and* $CaCO_3$ *in paste form with an addition of* CaO *or* β*-quartz, hydrated for 5 hrs at 181 °C. Admixture* = X . C_3A.

The reaction mixture contained the complex C_3A . $CaCO_3$. 11 H_2O beside the initial components C_3AH_6 and $CaCO_3$. The paste consistency specimen contained a higher amount of the C_3A . $CaCO_3$. 11 H_2O phase and a lower content of the initial components.

The optimum length of hydrothermal heating was determined on a series of paste-form specimens of the basic equimolar mixture, hydrated at 181 °C with isothermal heating for 1, 2, 3, 4, 5, 10 and 24 hours respectively.

The X-ray evaluation (Fig. 2) indicates that the complex C_3A . $CaCO_3$. 11 H_2O is already formed within one hour of the reaction and that its amount does not significantly change for the further 4 hours. Longer hydration results in a partial decrease of the carbonate complex content, obviously as a result of its decomposition into the initial components C_3AH_6 and $CaCO_3$.

The optimum hydration temperature was determined in a similar way. The specimens of paste consistency were subjected to hydrothermal reaction with an isothermal dwell of 2 hours at 100, 120, 140, 160 and 181 °C respectively. X-ray analysis revealed (Fig. 3) that the complex C_3A . $CaCO_3$. 11 H_2O is formed already at 100 °C in the same amounts as at the higher temperatures.

The effect of medium on the formation of $C_3A \cdot CaCO_3 \cdot 11\ H_2O$

Utilization of the carbonate complex $C_3A \cdot CaCO_3 \cdot 11\ H_2O$ for binding the aluminate components of hydrothermally cured cements depends on its formation in systems containing also other components, above all $Ca(OH)_2$, β-quartz (low) and 11 Å-tobermorite.

The approximate theoretical ratio $CaO : C_3A = 4 : 1$ corresponds to the cement employed. The amount of CaO added to the basic equimolar mixture of C_3A and $CaCO_3$ corresponded to 4-, 2-, 1-, 0.5- and 0.25-fold of the weight of C_3A. The specimens were hydrated in paste for 5 hours at 181 °C, dried and subjected to X-ray analysis, the results of which are summarized in Fig. 4.

X-ray analysis has shown that the complex $C_3A \cdot CaCO_3 \cdot 11\ H_2O$ is formed even at a four-fold excess of calcium hydroxide. From the intensity ratio of the diffraction lines for the carbonate complex, C_3AH_6 and calcite it may be assumed that the reaction equilibrium in the system with a high $Ca(OH)_2$ content is shifted towards formation of the complex $C_3A \cdot CaCO_3 \cdot 11\ H_2O$.

The same procedure was used for studying the effect of quartz on the formation of the carbonate complex. The basic equimolar mixture of C_3A and $CaCO_3$ was mixed with pure β-quartz in amounts corresponding to 5-, 2-, 1-, 0.5- and 0.25-fold of the C_3A content; the specimens in paste form were subjected to hydrothermal reaction at 181 °C with a 5-hour isothermal dwell. The X-ray analysis of the reaction products is shown in Fig. 4.

The reaction mixture with a 5-fold excess of quartz does not contain any $C_3A \cdot CaCO_3 \cdot 11\ H_2O$ detectable by X-ray analysis. Only minute amounts of the complex were determined in the specimens containing 2-fold and 1-fold amounts of quartz, while the addition of 0.5 to 0.25-fold amount of quartz will not significantly affect the reaction equilibrium so that the complex $C_3A \cdot CaCO_3 \cdot 11\ H_2O$ will be formed in amounts of the same order as had been produced from the basic components C_3A and $CaCO_3$ in the absence of β-quartz.

The effect of β-quartz on the complex $C_3A \cdot CaCO_3 \cdot 11\ H_2O$ is so intensive as to bring about its decomposition, when mixed with quartz and subjected to hydrothermal conditions. The complex prepared by hydration of the equimolar mixture of $C_3A + CaCO_3$ was mixed with five-, two- and one-fold of quartz and the mixture subjected to further hydrothermal reaction (5 hours at 181 °C). X-ray analysis showed that with the five-fold amount of quartz all the complex compound has decomposed, with the two-fold amount only a slight proportion of the carbonate complex remained intact, while only the treatment with the one-fold amount has resulted in no discernible decomposition, as indicated by the phase composition of the mixture shown in Fig. 5. The possibility of affecting formation of the complex $C_3A \cdot CaCO_3 \cdot 11\ H_2O$ by other forms of SiO_2, and their decomposing effect were studied with the use of silicic acid gel.

The basic equimolar mixture $C_3A + CaCO_3$ was mixed with SiO_2-gel in amounts corresponding to 4-, 2- and 1-fold of the C_3A content; the specimens in paste form were hydrated for 5 hours at 181 °C. X-ray analysis of the reaction product has shown that the SiO_2-gel does not significantly influence formation of the carbonate complex, as indicated by Fig. 5.

Further study of the complex $C_3A \cdot CaCO_3 \cdot 11\ H_2O$ has shown that its formation and stability is strongly negatively affected by the presence of 11 Å-tobermorite in the reaction system. The amount of the crystalline tobermoritic phase equivalent

to the content of C_3A will completely suppress formation of the complex, or will completely decompose all the C_3A . $CaCO_3$. 11 H_2O present.

The same negative effect on the carbonate complex is exhibited by Portland cement. The amount of cement corresponding to 0.5-fold of the C_3A content, added to the basic equimolar mixture of C_3A and $CaCO_3$, will influence the reaction system

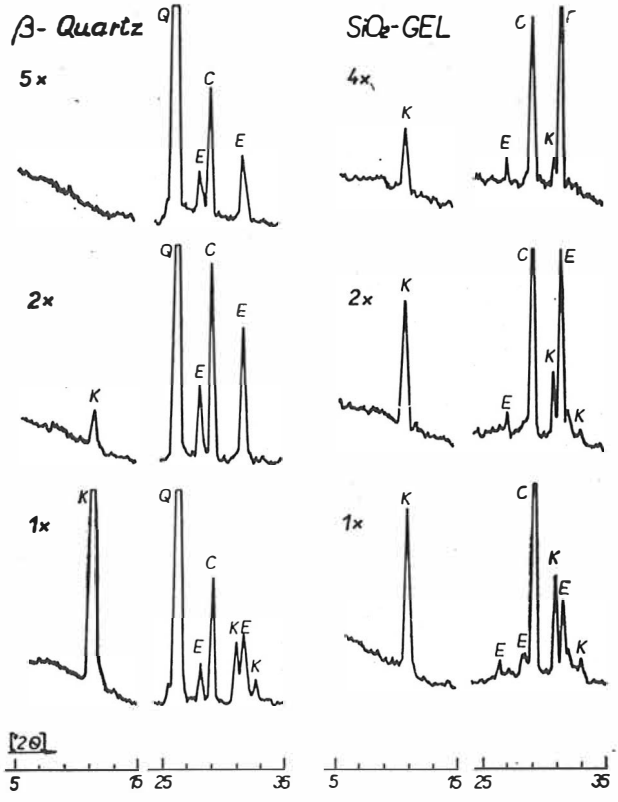

Fig. 5. X-ray diffraction patterns of C_3A . $CaCO_3$. 11 H_2O *with an addition of ß-quartz after hydrothermal treatment for 5 hrs at 181 °C, and of an equimolar mixture of* C_3A *and* $CaCO_3$ *with an addition of* SiO_2 *gel in paste form hydrated for 5 hrs at 181 °C. Admixture* = X . C_3A . $CaCO_3$. 11 H_2O; = X . C_3A.

so that hydrothermal reaction for 5 hours at 181 °C in the paste specimen will yield only minute amounts of the carbonate complex.

The experiments mentioned above were supplemented with those using mixtures with quartz sand or fly-ash with CaO or cement and stepped-up amounts of $CaCO_3$ (0—20 %). No carbonate complex has been determined in the respective high-pressure cured mixtures (181 °C).

The properties of the carbonate complex $C_3A \cdot CaCO_3 \cdot 11\ H_2O$

All the mixtures treated under hydrothermal conditions were subjected to microstructural X-ray analysis, as shown by the Figures.

Infrared spectral analysis was used for analysing the specimens of the carbonate complex prepared under various hydration conditions. The absorption spectra are shown in Fig. 6.

The complex $C_3A \cdot CaCO_3 \cdot 11\ H_2O$ was found to be characterized by the absorption band with a maximum at 3380 cm^{-1}. The latter is considerably greater for the specimen on the last record (suspension, 168 hrs at 25 °C) than with the paste specimen in pattern II. The lowest band intensity is exhibited by spectrum I of the suspension specimen (24 hrs/181 °C). The carbonate complex is obviously also characterized by the absorption band with the maximum at 1380 cm^{-1}.

The properties of the carbonate complex were further studied by differential thermal analysis on the same specimens employed in the IR analysis. The DTA

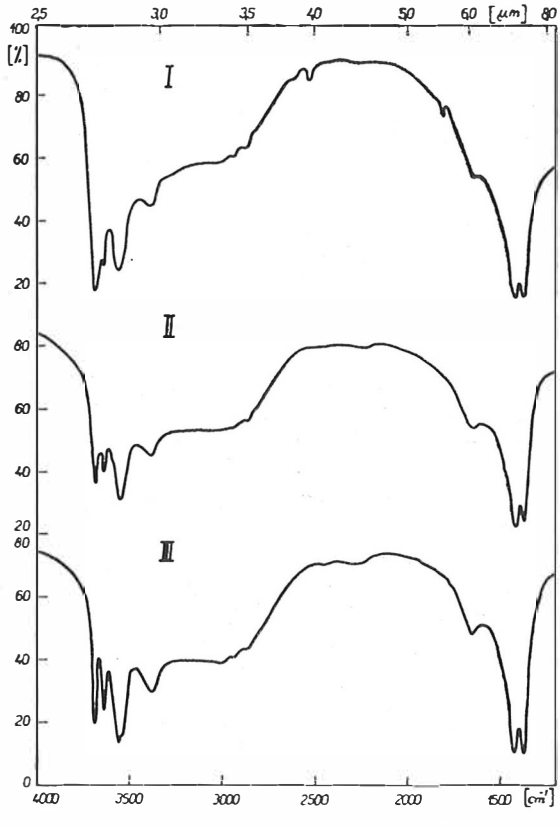

Fig. 6. Infrared spectra of $C_3A + CaCO_3$ *mixtures in molar ratios after hydrothermal treatment:*
I — $C_3A + CaCO_3$ *(1 : 1) 24 hrs/181 °C — suspension*
II — $C_3A + CaCO_3$ *(1 : 1) 5 hrs/181 °C — paste.*
III — $C_3A + CaCO_3$ *(1 : 1.01) 168 hrs/25 °C — suspension.*

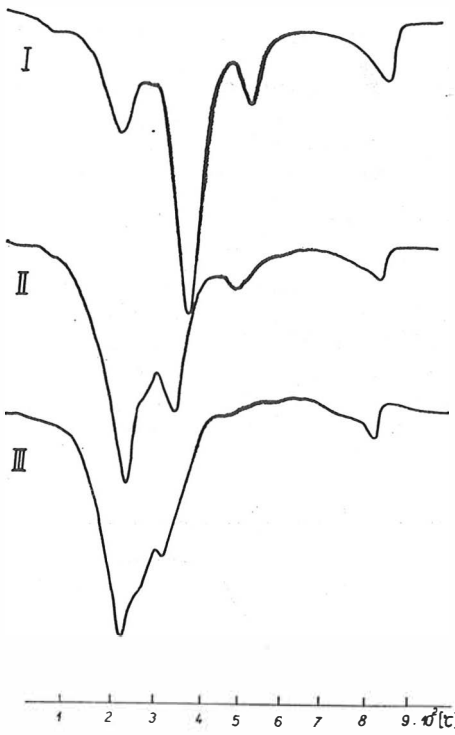

Fig. 7. DTA curves of the mixture $C_3A + CaCO_3$ *in a molar ratio after hydrothermal treatment:*
I — $C_3A + CaCO_3$ *(1 : 1) 24 hrs/181 °C — suspension.*
II — $C_3A + CaCO_3$ *(1 : 1) 5 hrs/181 °C — paste.*
III — $C_3A + CaCO_3$ *(1 : 1.01) 168 hrs/25 °C — suspension.*

curves shown in Fig. 7 confirm the conclusions reached on the basis of the methods dealt with above. All the curves show identical endothermal peaks characterizing decomposition of the carbonate complex (220—240 °C), of C_3AH_6 (330—370 °C), $Ca(OH)_2$ (500—530 °C) and decomposition of $CaCO_3$ (820—850 °C).

The scanning electron microscope was found to be a very valuable tool in the study of the morphology of the carbonate complex particles. The investigation was concerned with specimens treated at room temperature, as well as under hydrothermal conditions. The typical scaly or plate-shaped character of the crystals is well discernible on the stereoelectron micrographs in Figs. 8 and 9. Among the prevailing crystals of the complex, there are a few individual sharp-edged crystals of the phase C_3AH_6, characteristic by their hexagonal shapes.

CONCLUSION

The study was concerned with the formation of the carbonate complex $C_3A \cdot CaCO_3 \cdot 11\ H_2O$ from the equimolar mixture of C_3A and $CaCO_3$ under hydrothermal conditions (100 °C—181 °C) using isothermal periods of 1—5 hours and specimens of paste consistency.

Formation of the complex $C_3A \cdot CaCO_3 \cdot 11\ H_2O$ is promoted by excess $Ca(OH)_2$ in the reaction system.

Creation of $C_3A \cdot CaCO_3 \cdot 11\ H_2O$ is considerably suppressed by the presence of β-quartz. The carbonate complex may even be decomposed to its original components by an addition of quartz. No negative effect has been observed even at higher additions of silica gel.

Portland cement and 11 Å-tobermorite was likewise found to have a strongly negative effect on the formation of $C_3A \cdot CaCO_3 \cdot 11\ H_2O$ which is also decomposed in their presence.

On the basis of the findings mentioned above and on that of further experiments with mixtures of quartz sand or fly-ash and cement or lime with stepped-up $CaCO_3$ contents it has been explicitely proved that the carbonate complex cannot be formed during technological production of high-pressure cured materials made from these raw materials.

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PODMÍNKY VZNIKU A STABILITY KARBONÁTOVÉHO KOMPLEXU ($C_3A \cdot CaCO_3 \cdot nH_2O$) ZA HYDROTERMÁLNÍCH PODMÍNEK

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Studován vznik karbonátového komplexu ($C_3A \cdot CaCO_3 \cdot 11\ H_2O$) z ekvimolární směsi C_3A a $CaCO_3$ za hydrotermálních podmínek (100—181 °C) při délce izotermní prodlevy 1—5 h a pastovité konzistenci vzorku.

Tvorba komplexu $C_3A \cdot CaCO_3 \cdot 11\ H_2O$ je podporována nadbytkem $Ca(OH)_2$ v reakční soustavě.

Vznik $C_3A \cdot CaCO_3 \cdot 11\ H_2O$ je značně potlačován přítomností β-křemene. Vzniklý komplex může být přísadou křemene dokonce rozložen na původní složky. Negativní vliv nebyl pozorován i při zvýšeném přídavku SiO_2-gelu.

Negativní vliv na tvorbu $C_3A \cdot CaCO_3 \cdot 11\ H_2O$ byl prokázán též za přítomnosti 11 Å-tobermoritu a portlandského cementu.

Na základě výše uvedeného a provedených pokusů se směsmi křemičitého písku nebo elektrárenského popílku a cementu nebo vápna s odstupňovaným obsahem $CaCO_3$, jež byly hydrotermálně zpracovány, bylo jednoznačně prokázáno, že při technologické výrobě autoklávovaných hmot z těchto surovin nemůže dojít ke tvorbě karbonátového komplexu.

Obr. 1. Rentgenové difrakční záznamy ekvimolární směsi C_3A *a* $CaCO_3$, *24 h/181 °C, I — suspenze, II — pasta. Použité symboly: C — kalcit; E —* C_3AH_6; *K —* $C_3A \cdot CaCO_3 \cdot 11H_2O$; *Q — β-křemen; R —* $Ca(OH)_2$.

Obr. 2. Rentgenové difrakční záznamy pasty ekvimolární směsi C_3A *a* $CaCO_3$ *hydratované při 181 °C s různou délkou izotermní prodlevy.*

Obr. 3. Rentgenové difrakční záznamy pasty ekvimolární směsi C_3A *a* $CaCO_3$ *hydratované s izotermní prodlevou 2 h při různých teplotách.*

Obr. 4. Rentgenové difrakční záznamy pasty ekvimolární směsi C_3A *a* $CaCO_3$ *s přísadou* CaO *nebo β-křemene, hydratované 5 h/181 °C. Přísada =* $X \cdot C_3A$.

Obr. 5. Rentgenové difrakční záznamy $C_3A \cdot CaCO_3 \cdot 11H_2O$ *s přísadou β-křemene po hydrotermální reakci 5 h/181 °C a pasta ekvimolární směsi* C_3A *a* $CaCO_3$ *s přísadou* SiO_2*-gelu, hydratovaná 5 h/181 °C.*
Přísada = $X \cdot (C_3A \cdot CaCO_3 \cdot 11\,H_2O)$; $= X \cdot (C_3A)$.

Obr. 6. Infračervená spektra směsi $C_3A + CaCO_3$ *v molárním poměru — po hydrotermální reakci:*
I — $C_3A + CaCO_3$ (*1 : 1*) *24 h/181 °C — suspenze,*
II — $C_3A + CaCO_3$ (*1 : 1*) *5 h/181 °C — pasta,*
III — $C_3A + CaCO_3$ (1 : 1,01) *168 h/ 25 °C — suspenze.*

Obr. 7. Diferenční termické křivky směsi $C_3A + CaCO_3$ *v molárním poměru — po hydrotermální reakci:*
I — $C_3A + CaCO_3$ (1 : 1) *24 h/181 °C — suspenze,*
II — $C_3A + CaCO_3$ (1 : 1) *5 h/181 °C — pasta,*
III — $C_3A + CaCO_3$ (*1 : 1,01*) *168 h/ 25 °C — suspenze.*

Obr. 8. Stereoelektronogram směsi $C_3A + CaCO_3$ (*1 : 1*) — *2 h/181 °C.*

Obr. 9. Stereoelektronogram směsi $C_3A + CaCO_3$ (*1 : 1*) — *168 h/25 °C.*

УСЛОВИЯ ОБРАЗОВАНИЯ И УСТОЙЧИВОСТИ КОМПЛЕКСА КАРБОНАТОВ ($C_3A \cdot CaCO_3 \cdot nH_2O$) В ГИДРОТЕРМАЛЬНЫХ УСЛОВИЯХ

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Изучено образование комплекса карбонатов ($C_3A \cdot CaCO_3 \cdot 11\,H_2O$) из эквимолярной смеси C_3A и $CaCO_3$ в гидротермальных условиях (100—181 °C), в зависимости от продолжительности изотермической выдержки 1—5 часов и пастообразной консистенции образца.

На образование комплекса $C_3A \cdot CaCO_3 \cdot 11\,H_2O$ имеет положительное влияние избыток $Ca(OH)_2$ в реакционной системе. Возникновение $C_3A \cdot CaCO_3 \cdot 11\,H_2O$ в значительной степени подавляется присутствием β-кварца. Уже возникший комплекс можно даже разложить при помощи добавки кварца на первоначальные составляющие. Отрицательное влияние не наблюдалось даже при повышенном добавлении SiO_2-геля.

Деструктивное влияние на образования $C_3A \cdot CaCO_3 \cdot 11\,H_2O$ проявилось также у 11 Å — тоберморита и портландского цемента.

На основании выше изложенного и по результатам осуществленных опытов со смесями кремневого песка или золы-уноса электростанций и цемента или извести с разделенным по степеням содержанием $CaCO_3$, которые подвергались гидротермальной орбаботке, было однозначно доказано, что при технологическом производстве автоклавных материалов из этого сырья не может быть образован карбонатный комплекс.

Рис. 1. Рентгеновские дифракционные записи эквимолярной смеси C_3A *и* $CaCO_3$, *сутки/181 °C, I — взвесь, II — паста; C — кальцит, E —* C_3AH_6, *K —* $C_3A \cdot CaCO_3 \cdot 11\,H_2O$, *Q —* β*-кварц, R —* $Ca(OH)_2$.

Рис. 2. Рентгеновские дифракционные записи пасты эквимолярной смеси C_3A *и* $CaCO_3$, *гидратированной при 181 ° C с разной величиной изотермической выдержки.*

Рис. 3. Рентгеновские дифракционные записи пасты эквимолярной смеси C_3A *и* $CaCO_3$ *гидратированной с изотермической выдержкой 2 часа при разных температурах.*

Рис. 4. Рентгеновские дифракционные записи пасты эквимолярной смеси C_3A *и* $CaCO_3$ *с добавкой* CaO *или β-кварца, гидратированной 5 ч/181 °C. Добавка* = X . C_3A.

Рис. 5. Рентгеновские дифракционные записи C_3A . $CaCO_3$. 11 H_2O *с добавкой β-кварца после гидротермической реакции 5 ч/181 °C и пасты эквимолярной смеси* C_3A *и* $CaCO_3$ *с добавкой* SiO_2*-геля гидратированной 5 ч/181 °C. Добавка* = X . (C_3A . $CaCO_3$. 11 H_2O); = X . (C_3A).

Рис. 6. Инфракрасные спектры смесей C_3A + $CaCO_3$ *в молярном отношении — после гидротермической реакции: I —* C_3A + $CaCO_3$ *(1 : 1) сутки/181 °C — взвесь, II —* C_3A + $CaCO_3$ *(1 : 1) 5 ч/181 °C — паста, III —* C_3A + $CaCO_3$ *(1 : 1,01) 168 ч/25 °C — взвесь.*

Рис. 7. Дифференциальные термические кривые смесей C_3A + $CaCO_3$ *в молярном отношении — после гидротермической реакции: I —* C_3A + $CaCO_3$ *(1 : 1) сутки/181 °C — взвесь, II —* C_3A + $CaCO_3$ *(1 : 1) 5 ч/181 °C — паста, III —* C_3A + $CaCO_3$ *(1 : 1,01) 168 ч/25 °C — взвесь.*

Рис. 8. Стереоэлектронограмма смеси C_3A + $CaCO_3$ *(1 : 1) — 2 ч/181 °C.*

Рис. 9. Стереоэлектронограмма смеси C_3A + $CaCO_3$ *(1 : 1) — 168 ч/25 °C.*