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Activation of cement clinker with high content of belite

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Abstract: The results of studies of cement clinkers with a high content of belite are presented. To obtain clinkers polymineral wastes of enrichment of skarn-magnetite ores were used. To intensify the hardening of cements, a method for activating the silicate phases of clinker (alite and belite) is proposed. Active phases are formed during the synthesis of clinker using a dual-blend technology, which provides an increase in the reactivity of the components of the raw material mixture. Thermodynamic calculations have been carried out, confirming the likelihood of belite formation reactions based on minerals of the raw material mixture. The composition of raw materials blends is justified. To assess the low-melting charge, a basicity coefficient (BC) was introduced and the preferred values of BC = 0.25 – 1.50 were shown. An increase in the activity of alite and stabilization of the active modification of belite provide hardening of the cement stone. The work is aimed at creating resource-saving technologies for Portland cement.
Keywords: technogenic materials, alite, belite clinker, activation.

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Introduction

A substantial savings in fuel and energy resources are achieved by reducing the basic capacity of Portland cement clinker. Low-base (belite) cements are low-energy binders with a high content of C_2S belite. The consumption of calcium carbonate in the raw material mixture is reduced, clinker firing costs and carbon dioxide emissions are decreased with a fall in the proportion of C_3S alite in belite clinker [1 - 10]. Belitic cements have benefits as low heat during hydration, increased service life of concrete in a severe environment. However, when using cements with a low alite content, difficulties associated with slow hardening arise [2, 4, 8, 9].

An increase in low-alumina clinker hydraulic activity is provided by a directed change in the reactive capacity of the raw material [4 - 8]. The chemical and mineral features of man-made materials determine their preference for active low-base clinkers [5,6,10 –16].

Clinker activation solely due to the belite phase is technologically complex and often becomes ineffective [17–19]. The curing rate of low-base cements shows an increased dependence on alite activity. With the intensive hydration of alite, the necessary amount of portlandite is released to form the crystalline framework of the stone. The hydration ability of alite, due to the modification composition and crystalline state of the phase, is very sensitive to changes in the reactive capacity of raw materials.

The possibility of obtaining cements using tailings of skarn-magnetite ores has been proved [5, 15, 20]. The complex chemical and mineral composition of the man-made material seems to provide the opportunity to control the rate of clinker formation, the structure and activity of the phases, particularly alite.

The alite formation kinetics is determined by the ratio of the dissolution rates of C_2S and CaO, and the melt composition. Technological methods are used to intensify the process such as raw material mixture modification due to additives, grinding coarsening of the carbonate component, its feeding into the sintering zone to prevent CaO recrystallization; high-temperature belito formation, excluding the C_2S passivation of [1, 9, 10, 13, 18]. The dual-charge technology developed by V.D. Barbanyagre [9] considers the composition of one of the blends provides an increased amount of melt at a low temperature.

The study is aimed to investigate the low-base clinkers synthesized by the dual-charge technology using skarn-magnetite ore tailings.

Testing

Limestone and wastes of skarn-magnetite ores enrichment were used for the cement clinkers synthesis, quartz sand was introduced as a correcting siliceous component. The chemical composition of limestone, wt.% as follows SiO_2 0,84; Al_2O_3 0,51; Fe_2O_3 0,63; CaO 53,73; MgO 1,08; SO_3 0,24; R_2O 0,10; LOI 42.87.

Tails of dry and wet magnetic separation are made after processing skarn-magnetite ores. In the study, gravel-like tails of dry magnetic separation (tails of DMS) were used a disintegrated mass with a particle size of up to 25 mm; humidity was 1–2%. Tails of DMS surpass cement clinker and quartz sand in grinding ability.

The chemical composition of tails of DMS is presented, wt.% as SiO_2 40,82; Al_2O_3 11,86; Fe_2O_3 (overall) SiO_2 40,82; Al_2O_3 11,86; Fe_2O_3; LOI 3.55. Modified impurities are found in the wastes, wt.% as S 2.79; R_2O 3,21; TiO_2 0,50; P_2O_5 0,26; MnO 0,38; V_2O_5 0,04; Cl 0,11; Cu 0,04; Ni 0,008.

The mineral base of tails of DMS is composed of silicates that differ in genesis, composition, structure, physical properties, chemical activity and thermal stability, wt.% as Pyroxenes (diopside) 20 - 25; epidote 10 - 13; feldspars 8 - 12; chlorites 7–10; Scapolite 8 - 11; garnets (andradite, grossular) 7–12; amphiboles (actinolite) 7-14. The wastes have, wt.% as Calcite 4-7; pyrite 4-8; quartz 2-4; magnetite 3-4.

Raw mixtures were prepared by co-grinding of pre-ground starting materials. The grinding fineness of the raw mix was evaluated by the residue left on the sieve No. 008 which was 2-5%. Clinkers synthesis was provided by burning samples of the raw material mixture in a laboratory furnace with silica heaters. Cement was obtained by grinding clinker with two-water gypsum.

The materials composition was studied using differential thermal, x-ray phase analysis and microscopy. Strength characteristics of cements were determined on samples of small size (2x2x2 cm) made from paste of normal density.

The study is based on the limited CaO content in one of the two parts of the mixture will contribute to the formation of intermediate compounds (aluminosilicates and magnesium calcium silicates) hypothesis. Intensive heating of another part of the mixture (limestone) in the sintering zone is accompanied by almost instant dissociation of calcite and the occurrence of highly reactive calcium oxide. Calcium oxide responds with the intermediate phases to form belite when two heat-treated feed streams are combined. Discharge of a significant amount of C_2S in a narrow temperature range increases the degree of thermal effect on the material due to exothermy of the reaction brought under control and activates alitogenesis.

Reducing the temperature and time intervals between the synthesis of C_2S and C_3S promotes the active crystalline structure formation, enhances the phase imbalance.

The ΔH enthalpy reactions of feed mixture silicates interaction with calcium oxide was calculated, which result in the formation of C_2S. The reference thermodynamic characteristics were used in calculations. The initial data are provided in Table 1, the calculation results in Table 2.

The systematic nature of the minerals saturation determines the heat release by portions during their interaction with CaO (Table 2). As the basicity of the starting silicates increases, the reaction thermal effect is remarkable decreases. Consequently, the extension of the multi-stage process will provide smooth heat release in a wide temperature range. To contain the exothermic effect, the stages merge is advisable by reducing the gaps between the individual reactions. This can be achieved by removing part of CaO from the sphere of low-temperature interactions, which will limit the low-temperature saturation of natural silicates and will allow one to control the intensity of heat release.

Results and discussion

Various options for preparing a low-basic mixture with a saturation coefficient SC = 0.75 were studied to test initiated proposals. The first part of the raw material mixture, an easily melted furnace feed, contains tailings of skarn-magnetite ores, an adjusted siliceous additive (silica sand), and part of the carbonate component (limestone). The second part of the mixture is the remaining part of the limestone.

Table 1 Thermodynamic characteristics of the compounds

Compound	The heat of formation of the elements «–»ΔH^0_{298}, kJ / mol	Avg = a + vT + cT $^{-2}$, J / (mol · deg)		
		a	*в·10* $^{-3}$	*c·10*^{5}
CMS_2	3208,30	221,53	32,85	– 65,95
CAS_2	4238,11	269,66	57,35	– 70,70
NAS_6	3934,00	274,14	43,86	– 79,29
C_2S	2309,58	151,67	36,94	– 30,29
C_3A	3558,10	260,70	19,17	– 50,11
CaO	635,85	48,85	4,53	– 6,53
MgO	602,11	42,61	7,28	– 6,20
Na_2O	416,09	77,11	19,33	– 12,59
SiO_2	908,30	46,94	34,31	– 11,30

Table 2 The calculated values of the reactions thermal effects

Ser.No.	Equation of reaction	Enthalpy of reaction, «–»H^0_{298}, kJ / mol
1	$CMS_2 + 3C \rightarrow 2C_2S + M$	105,42
1.1	$CMS_2 + C \rightarrow C_2MS_2$	35,75
1.2	$C_2MS_2 + C \rightarrow C_3MS_2$	54,95
1.3	$C_3MS_2 + C \rightarrow 2C_2S + M$	14,72
2	$CAS_2 + 6C \rightarrow 2C_2S + C_3A$	124,05
2.1	$CAS_2 + 2C \rightarrow C_2AS + CS$	85,73
2.2	$C_2AS + 3C \rightarrow C_2S + C_3A$	1,11
2.3	$CS + C \rightarrow C_2S$	37,21
3	$NAS_6 + 15C \rightarrow 6C_2S + C_3A + N$	4359,92
3.1	$NAS_6 \rightarrow NAS_2 + 4S$	1808,90
3.2	$NAS_2 + 7C \rightarrow 2C_2S + C_3A + N$	2032,70
3.3	$2C + S \rightarrow C_2S$	129,58

The raw material mixture and the first furnace feed are characterized by a silicate module 2.0 and an alumina module 0.7. The composition of the raw material furnace feed (Table 3) provides a lower melting temperature of the calcined mass, the formation of a modified silicate melt.

An additional indicator - the basicity ratio (BR) is introduced to assess the composition of tail-containing mixtures, characterized by extremely low values of the saturation coefficient, which allows you to take into account the complex composition of the phases of the burnt furnace feed. It is customary Ternary compounds are acceptable to consider as a conditional combination of simple phases: C_2AS = CS + CA and C_2MS_2 = 2CS + M. The basicity ratio characterizes the degree of saturation of silica with calcium oxide to CS metasilicate. When calculating BR according to formula (1), the possibility of CA, C_2F, $CaSO_4$ formation is taken into account:

$$BR = \frac{C - 0{,}55A - 0{,}18F - 0{,}70\hat{S}}{0{,}93S}, \qquad (1)$$

wherein 0,55 – the ratio of CaO to Al_2O_3 at CA;
0,18 – the ratio of CaO to Fe_2O_3 at C_2F;
0,70 – the ratio of CaO to SO_3 at $CaSO_4$;
0,93 – the ratio of CaO to SiO_2 at CS.

The whole silica (and the intermediate phases conditionally including it) is assumed to be bound to calcium metasilicate at BR = 1, with an increase in BR values, the availability of dicalcium silicate is possible.

At the first stage, the furnace feed was calcined at a slow rise in temperature (10 °C / min) with a holding time of 20 min at a temperature of 1200 °C. Then, the calcined material was finely ground, minced with the required amount of limestone to obtain a mixture with SC = 0.75 (Table 3), placed in a preheated furnace, and burned at temperatures of 1250 °C and 1300 °C, maintaining a high heating rate (200 °C / min).

The composition of the easily melted furnace feed determines their state after the initial firing and the nature of the interactions during subsequent sintering of the total mixture. Unlike melted materials (furnace feed with BR = 0.25 - 0.50) and dense cakes (furnace feed with BR = 1.0–1.85), burnt furnace feed D6 (BR = 2.25) containing an increased proportion of limestone, acquired the state of low-caking mass.

Furnace feed firing is accompanied (Figure 1) by melting tails of DMS (endoeffect at 1120 °C), the formation of complex silicates, calcium aluminosilicates and solid solutions based on them (exo-effects at 1000 °C and 1180 °C), which availability is fixed on the XRD pattern (Figure 2). The fraction of free CaO in the calcined materials is insignificant (up to 1%), which indicates the completeness of the interaction processes. The synthesis of clinkers based on tails of DMS is characterized by inhibited alitogenesis, which is due to sulfate-alkaline segregation [5]. During sintering of a double furnace feed mixture, its easily melted component forms a high-siliceous melt of low viscosity, increased homogeneity, and uniformly saturated with modifying elements.

C_2S high-temperature synthesis determines the intensive dissolution of belite in a clinker melt. A defective microstructure of CaO is formed during thermal activation of the carbonate component, which ensures its accelerated dispersion and dissolution in the melt. As a result, crystallization of alite is intensified. The rapid growth of C_3S crystals is accompanied by the introduction of modifying elements present in the melt into their structure. Alite crystals formed in this way are distinguished by a distorted crystal lattice and increased hydration activity [3, 9, 19].

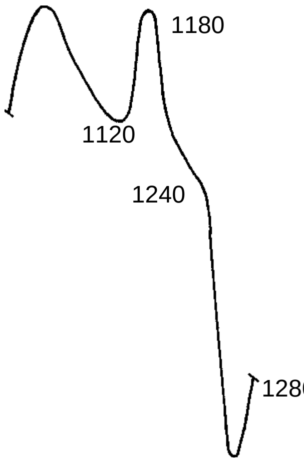


Figure 1 DTA curve of D4 furnace feed

Table 3 Preparation of the raw mix using easily melted furnace feed options

Mixture code	1 and 2 parts content in the mixture, %		The material composition of the easily melted furnace feed 1,%			Furnace feed 1 parameters	
	1	*2*	*limestone*	*Tails of DMS*	*Silica sand*	*BR*	*SC*
D1	38,52	61,48	19,67	68,23	12,10	0,25	0,01
D2	44,48	55,52	31,12	57,46	11,42	0,50	0,09
D3	59,17	40,83	47,76	44,08	8,16	1,00	0,26
D4	72,46	27,54	57,21	36,44	6,35	1,50	0,42
D5	82,17	17,83	62,91	30,88	6,21	1,85	0,54
D6	93,46	6,54	67,48	26,79	5,73	2,25	0,67
K	100	нет	69,59	24,91	5,50	2,47	0,75

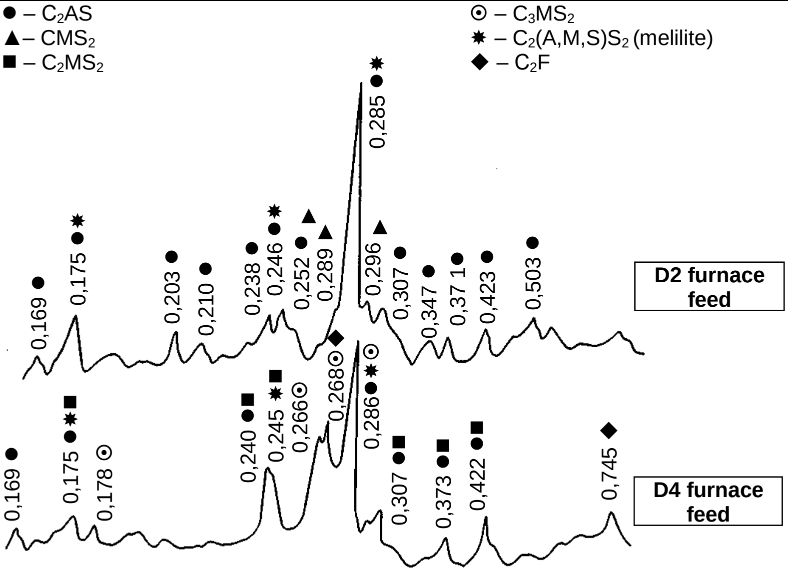


Figure 2 X-ray diffraction patterns of easily melted furnace feed primary firing

The intensity of clinker formation during firing of double furnace feed mixtures is confirmed by a more uniform crystalline structure of clinkers. Alite crystals of hexagonal crystals with good faceting and a size of 60 - 80 μm prevail (Fig. 3). According to petrography and X-ray diffraction analysis (Figure 4), an increase in the alite content of 5–8% in clinkers synthesized using double furnace feed technology was revealed. This is ensured by the intensification of the final stages of clinker formation, the presence of modifying elements in the man-made raw materials, and the formation of a highly iron aluminoferrite phase less saturated with calcium oxide (shift of the diffraction maximum of 0.265 nm to lower angles).

When firing double furnace feed mixtures, the α'_m – C_2S modification is stabilized (Figure 4), the largest amount of which is noted for D1 and D2 clinkers. The formation of the reactive form α'_m – C_2S is achieved due to the complex effect of doping impurities during the high-temperature phase formation with the participation of the modified melt.

Preliminary heat treatment of raw materials positively affects the hydration activity of synthesized clinkers (Table 4). An increase in the strength of cement stone by 12–68% is caused by an increase in the amount and degree of hydration of C_3S, and the formation of the hydraulically active modification of α'_m – C_2S belite.

Maximum strength indicators are achieved when using the most easily melted tail-containing D1 furnace feed.

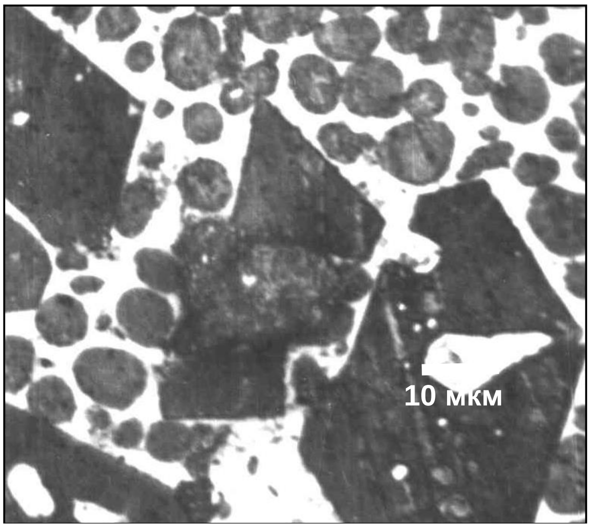
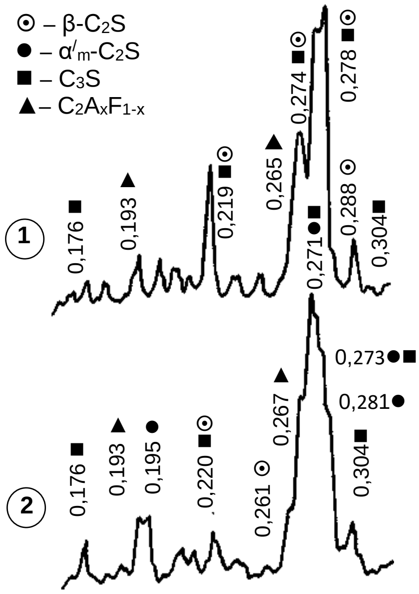


Figure 3 The clinker microstructure synthesized from a double furnace feed mixture



1– traditional; 2– double furnace feed

Figure 4 Clinkers X-ray patterns synthesized by various technologies

Table 4 The influence of synthesis conditions on hydration activity of clinkers

Clinker code	Alita hydration degree ,%, Aged, days		Maximum strength, MPa, samples 2x2x2cm aged, days	
	1	***28***	***1***	***28***
D1	32	96	42	112
D2	31	92	38	110
D3	31	94	38	107
D4	29	93	33	105
D5	27	90	29	99
D6	28	89	28	97
K	23	80	25	82

However, this option involves feeding into the sintering zone almost half of all the raw materials; therefore, it is technically complicated and requires deep engineering study. This technology implementation is possible in furnace units of a different configuration. Compiling options of D4 - D6 mixtures providing a limited input of the carbonate component into the sintering zone are of the greatest practical interest for the existing clinker firing technology.

Findings

The possibility of activating belite clinkers based on skarn-magnetite ore tailings using the double furnace feed technology is given.

The synthesis efficiency of the low-base clinkers based on double furnace feed is determined by the thermal activation of the man-made component in combination with a given amount of carbonate component and the subsequent interaction of the calcined furnace feed with thermally activated calcium oxide.

A well-aimed change in the state of raw materials and the nature of their interactions enhances the modifying effect of skarn-magnetite ore tailings on the properties of the melt, the formation of C_2S and CaO reactive, the intensification of alite formation and stabilization of hydraulically active forms of belite and alite.

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Құрамында белиті жоғары цемент клинкерлерін белсендіру

Мирюк О.А.

Түйіндеме. Құрамындағы белиттің мөлшері жоғары цемент клинкерлерін зерттеу нәтижелері келтірілген. Клинкерлерді алу үшін скарн-магнетит кендерін байытудың полиминералды қалдықтары пайдаланылды. Цементтердің қатаюын қарқындату үшін клинкердің (алит және белит) силикатты фазаларын белсендіру (активтендіру) әдісі ұсынылған. Белсенді фазалар клинкерді синтездеу кезінде екі шихта технологиясы арқылы қалыптасады, ол шикізат қоспасы компоненттерінің реакциялық қабілетін арттыруды қамтамасыз етеді. Шикізаттық қоспа минералдарының негізінде белит түзілу ықтималдығын растайтын термодинамикалық есептер орындалды. Шикізат шихталарының құрамы дәлелденген. Жеңіл балқитын шихтаны бағалау үшін негізділік коэффициенті енгізілген және КО = 0,25 – 1,50 мәндерінің артықшылығы көрсетілген. Алит белсенділігінің артуы және белиттің белсенді модификациясының тұрақтануы цемент тасының нығыздалуын қамтамасыз етеді. Жұмыс портланд-цементтің ресурс үнемдеуші технологияларын құруға бағытталған.
Түйін сөздер: техногенді материалдар, алит, белит клинкері, белсендіру.

Активизация цементных клинкеров с повышенным содержанием белита

Мирюк О.А.

Аннотация. Приведены результаты исследований цементных клинкеров с высоким содержанием белита. Для получения клинкеров использованы полиминеральные отходы обогащения скарново-магнетитовых руд. Для интенсификации твердения цементов предложен способ активизации силикатных фаз клинкера (алита и белита). Активные фазы формируются при синтезе клинкера по двухшихтовой технологии, которая обеспечивает повышение реакционной способности компонентов сырьевой смеси. Выполнены термодинамические расчеты, подтверждающие вероятность реакций образования белита на основе минералов сырьевой смеси. Обоснован состав сырьевых шихт. Для оценки легкоплавкой шихты введен коэффициент основности и показана предпочтительность значений КО = 0,25 – 1,50. Увеличение активности алита и стабилизация активной модификации белита обеспечивают упрочнение цементного камня. Работа направлена на создание ресурсосберегающих технологий портландцемента.
Ключевые слова: техногенные материалы, белитовый клинкер, алит, активизация.

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