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MIRYUK O. A.

Rudny Industrial Institute, Rudny, Kazakhstan, e-mail: psm58@mail.ru

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A CEMENT CLINKER FORMATION WITH THE USE OF MAGNETITE SCARN ORES WASHERY REFUSES

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Abstract. The clinker formation processes results of studies during the raw mixtures burning containing magnetite scarn ores washery wastes are submitted. Chemical and mineral compound, thermal transformations of ore washery wastes are provided. The clinker phase formation features involving the man-made component of the raw mixture, which consist in the low-temperature interactions activity, the variety of intermediate phases are revealed. A belite formation stages with the participation of actinolite, albite, andradite, grossular, diopside, chlorite, epidote is established to determine by gradual transformations of minerals into intermediate phases of C_2AS, C_3MS_2, out of which C_2S is formed at 1000 – 1300 °C. An alite formation is intensified due to high-temperature belite formation and doping impurities. The possibility of cement clinkers basicity reducing due to the chemical and mineral characteristics of man-made raw materials is substantiated. The influence of rough ore washery refuses on the alite crystallization nature is investigated, which is manifested in the microvolumes formation differing in the amount and viscosity of the liquid phase. The use of polyfractional refuses gristing was revealed to increase the proportion of highly active fine-crystalline alite, contributing to the cement stone strengthening. The thermal calculations results show 18 – 34 % decrease of heat costs to clinkers formation of different basicity when using ore refuses.

Keywords: magnetite scarn ores, man-made materials, thermal transformations, clinker burning, clinker formation processes, belite, alite

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Information about authors: Ministry of Education and Science of Kazakhstan. Republican state enterprise on the right of business «Rudny Industrial Institute». Head of the department of building construction materials. Rudny, Kostanay region, Kazakhstan
Miryuk Olga Aleksandrovna - Doctor of Technical Sciences, Professor. https://orcid.org/0000-0001-6892-2763 e-mail: psm58@mail.ru

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МИРЮК О.А.

Рудный индустриялық институты, Рудный, Қазақстан, e-mail: psm58@mail.ru

СКАРЛЫ-МАГНЕТИТТІ БАЙЫТЫЛҒАН КЕНДЕР ҚАЛДЫҚТАРЫН ПАЙДАЛАНА ОТЫРЫП ЦЕМЕНТТІ КЛИНКЕРДІҢ ҚАЛЫПТАСУЫ

Түйіндеме. Скарлы-магнетитті кендерді байыту қалдықтары бар шикізат қоспаларын күйдіру кезінде клинкер түзілу үдерістерін зерттеу нәтижелері ұсынылған. Кенді байыту қалдықтарының химиялық және минералды құрамы, термиялық түрленуі туралы мәліметтер келтірілген. Шикізат қоспасының техногенді компонентінің қатысуымен клинкерлік фазаларды қалыптастыру ерекшеліктері анықталды, яғни олардың төмен температуралы өзара әрекеттесуі белсенділігі мен аралық фазалардың алуан түрлілігінен байланысты. Актинолит, альбит, андрадит, гроссуляр, диопсид, хлорит, эпидоттың қатысуымен белиттің түзілу сатылары минералдардың біртіндеп C_2AS, C_3MS_2, аралық фазаларына айналуымен байланысты, яғни олардан 1000 – 1300 ⁰C температураларында C_2S қалыптасады. Алиттің қалыптасуы, жоғары температуралы белиттің түзілуі мен легірлеуші қоспалар арқылы қарқындыланады. Техногенді шикізаттың химиялық-минералды ерекшеліктеріне байланысты цемент клинкерлердің негізділігін төмендету мүмкіндігі сипатталған. Байыту кендерінің өрескел ұнтақталған қалдықтарының алиттің кристалдануына әсері зерттелген, яғни микроөлшемдердің пайда болу кезіндегі сұйық фазаның тұтқырлығы мен санына байланысты. Қалдықтардың полифракциялық әжіктерін пайдаланған кезде цемент тасының беріктігін арттыруға бағытталған белсендігі жоғары ұсақ кристалды алиттің үлесі арта бастайтыны анықталған. Жылу техникалық есептеулердің нәтижелері бойынша байыту кендерінің қалдықтарын пайдалану кезінде award кликерлердің әр-түрлі негізділерінің пайда болуына жылудың шығыны 18 – 34 % – ға төмендейтіні көрсетілген.

Түйін сөздер: скарлы-магнетитті кендер, техногенді материалдар, термиялық айналымдар, клинкерді күйдіру, клинкерқұрылу үрдістері, белит, алит.

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МИРЮК О. А.
Рудненский индустриальный институт, Рудный, Казахстан, e-mail: psm58@mail.ru

ФОРМИРОВАНИЕ ЦЕМЕНТНОГО КЛИНКЕРА С ИСПОЛЬЗОВАНИЕМ ОТХОДОВ ОБОГАЩЕНИЯ СКАРНОВО-МАГНЕТИТОВЫХ РУД

Резюме: Представлены результаты исследований процессов клинкерообразования при обжиге сырьевых смесей, содержащих отходы обогащения скарново-магнетитовых руд. Приведены сведения о химическом и минеральном составе, термических превращениях отходов обогащения руд. Выявлены особенности формирования клинкерных фаз с участием техногенного компонента сырьевой смеси, которые заключаются в активности низкотемпературных взаимодействий, многообразии промежуточных фаз. Установлено, что стадийность белитообразования с участием актинолита, альбита, андрадита, гроссуляра, диопсида, хлорита, эпидота обусловлена постепенными превращениями минералов в промежуточные фазы C_2AS, C_3MS_2, из которых при температурах 1000 – 1300 °С формируется C_2S. Формирование алита интенсифицируется за счет высокотемпературного белитообразования и легирующих примесей. Обоснована возможность снижения основности цементных клинкеров благодаря химико-минеральным особенностям техногенного сырья. Исследовано влияние грубомолотых отходов обогащения руд на характер кристаллизации алита, которое проявляется в образовании микрообъемов, отличающихся количеством и вязкостью жидкой фазы. Выявлено, что при использовании полифракционной крупки отходов увеличивается доля высокоактивного мелкокристаллического алита, способствующего повышению прочности цементного камня. Результаты теплотехнических расчетов свидетельствуют о снижении на 18–34 % затрат тепла на образование клинкеров различной основности при использовании отходов обогащения руд.
Ключевые слова: скарново-магнетитовые руды, техногенные материалы, термические превращения, обжиг клинкера, процессы клинкерооообразования, белит, алит

Introduction. A cement industry is characterized by a high resource capacity, which is why the products output increase is accompanied by the significant growth of raw materials, fuel and energy spend. The mineral and raw materials base of the contemporary cement industry is represented by natural and man-made materials. The standard reserves of the traditional raw material sources are under exhaust. An output and industrial wastes accumulation are observed as the natural resources deficit increase. The cement production becomes a huge wastes consumer of various branches. [1-15]. An extensive group of man-made materials is intended for use as an iron-containing component of the cement raw mix. The overburden rocks, mining and enrichment wastes of various ores and coal, ferrous and nonferrous metallurgy slags, and ashes of thermal power plants serve as an important source of aluminosilicate raw materials [7, 10, 12–15]. A lack of pure limestone deposits as well as their production when the longtime open-pit mining induce to use another available more often low-grade raw materials. A list of materials are wholly replacing traditional carbonaceous component. In this regard, the multifunctional man-made raw materials suitable to replace clay serving as an iron-containing component and simultaneously allowing to reduce a carbonaceous content is more efficient. It is necessary to further expand the resource base due to little-used natural and man-made sources. It is important to preserve or improve the quality of cement when change-over to unconventional raw materials.

Having the man-made components make the chemical and mineral compound of the raw material mixes complicated and change the standard nature of different interactions at burning. The clinker phases are produced using the other schemes by means of untraditional materials. It is necessary to develop ideas about individual interactions in the synthesis of clinkers from the new raw materials. Having notion of the patterns of clinker formation processes will serve as a theoretical basis for the development of rational ways of using man-made materials in the cement technology.

The paper is aimed to research the influence of the man-made component on the process of formation of the cement clinker and its features.

Experiments. The study objects were raw mixes with various content of skarn-magnetite ores washery refuses and a clinker based on them.

The tails of dry and wet magnetic separation (Dry Magnetic Separation and Wet Magnetic Separation) are formed at various stages of processing of skarn-magnetite ores (Sarbayskoe and Sokolovskoe deposits, Kostanay region). Fine-dispersed wastes of WMS differ from the coarse-grained tailings of DMS with an increased proportion of ferrous compounds, by a lower content of aluminosilicates and the highest concentration of pyrite. At the same time, the substantial proximity of the physical and chemical properties of the wastes at various stages of enrichment remains. We used DMS tails here.

DMS tails represent break stones-like mass (particle size is up to 25 mm) with a low humidity (1–2 %). The chemical composition of the washery refuses of skarn-magnetite ores is characterized as stable and is presented, wt%: SiO_2 40–45; Al_2O_3 10–12; Fe_2O_3 (total) 16-18; FeO 6–8; CaO 12–13; MgO 5–6; mass loss ignition 3–6. Multivalent iron ions have predominantly octahedral coordination. The wastes contain a number of catalytic and modifying elements, wt.%: S 2–5; R_2O 2–4; TiO_2 0.50–0.53; P_2O_5 0.25–0.30; MnO 0.35–0.40; V_2O_5 0.04–0.06; Cl 0.09–0.12; Cu 0.04–0.05; Ni 0.007–0.008.

The mineral base of the DMS tails is made of silicates differing in their genesis, composition, structure, physical properties, chemical activity and thermal stability, wt%: pyroxenes (diopside) 20–25; epidote 10–13; feldspar (albite) 8–12; chlorites 7–10; scapolite 8–11; garnets (andradite, grossular) 7–12; amphiboles (actinolite) 7–14. The wastes consist of, wt.%: calcite 4-7; pyrite 4–8; quartz 2–4; magnetite 3-4.

The carbonate, aluminosilicate and ferrous components are included into a composition of the raw mixes to produce cement clinker. A limestone is the carbonate component of the raw materials under investigation, containing, wt.%: SiO_2–2.3; Al_2O_3–0.5; Fe_2O_3–0.3; CaO–51.5; MgO–2.4; others - 0.1; mass loss ignition – 42.9. The blank raw mix is composed of standard raw materials: a clay loam composition was used as the aluminosilicate component, wt%: SiO_2–60.5; Al_2O_3–14.6; Fe_2O_3–6.2; CaO – 7.9; MgO – 1.3; others – 0,2; mass loss ignition – 9.3; the pyrite butts containing 69.5 % of Fe_2O_3 served as the ferruginous component. The washery refuses ores in the studied mixtures of the M type, performed a function of iron-containing and aluminosilicate components. In C types raw mixes, only pyrite cinder replaced the DMS tails.

The raw mixes were prepared by thorough mixing of preliminary grained components. The mixtures compound were calculated according to the generally accepted method and provided comparable values of the saturation coefficient SC=0.77–0.95, the silicate modulus p=2.5; alumina modulus p=0.9–1.2. The raw mixes were molded into the briquettes with a diameter and a height of 2 cm. The samples were fired in the temperature range of 900–1450 °C. The materials composition was determined by X-ray phase analysis on a DRON-3 x-ray diffractometer with a copper cathode and a nickel filter. Thermal and chemical processes were studied by the differential thermal analysis method, which was carried out using a derivatograph of the IOM company by the system of F. Paulik, I. Paulik and L. Erdey. The crystal optical studies of clinkers were performed on an MMP – 2P microscope in reflected light.

Discussing the results. A polymineral composition of washery refuses of the skarn-magnetite ores has stipulated a step by step nature of the thermal transformation of the man-made component: 400–600 °C is of pyrite and magnetite oxidation; 600–800 °C is decarbonization of calcite, intensified by pyrite decomposition products; anhydrite formation; dehydration of actinolite, chlorite, epidote; 800–1000 °C is the destruction of the crystal structures of the listed silicates, as well as scapolite, accompanied by an increase in the proportion of pyroxenes, garnets, and feldspar; 1000–1200 °C is melting, amorphization [7].

The thermal analysis of the raw mixes has showed the exothermic heat of transformation in the DMS tails at 480–500 °C temperature (Figure 1), the catalytic effect of the decomposition products of pyrite accelerates decarbonization, helps to reduce the process completion temperature.

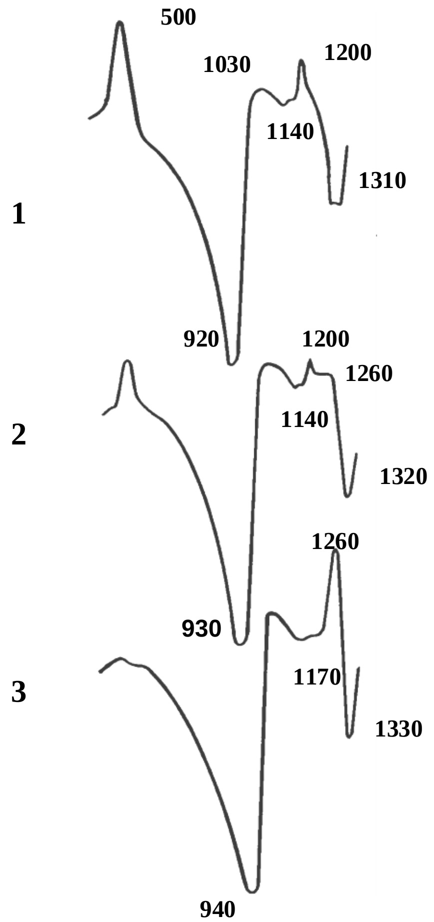


Figure 1 – The DTA curves of raw mixes with various DMS tails content: 1-M type; 2-C type, 3- testing

This is also caused by a reduction in the carbonate component in the tail-containing feedstock. The decrease in the area of endo-effects of the calcium carbonate decomposition for mixtures with wastes is associated with the simultaneous occurrence of decarbonization reactions and the clinker phases formation of.

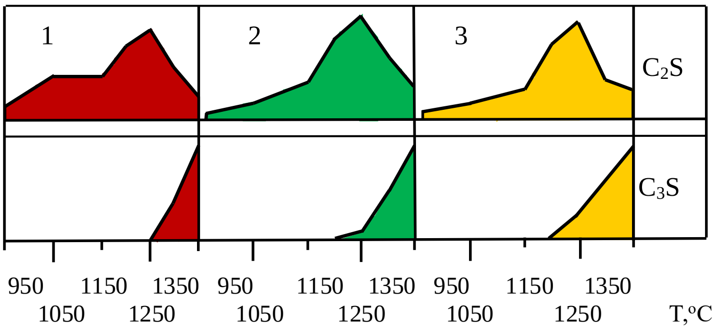


Figure 2 – The clinker phases formation in the raw mixes with various DMS tails content: 1-M type; 2-C type; 3-testing mix

The phase composition of portland cement clinker is represented by C_3S alite, C_2S belite, C_3A aluminate, and C_4AF calcium aluminoferrites (the silicate technology has the abbreviations C–CaO, S–SiO_2, A–Al_2O_3, F–F_2O_3). Calcium silicates form the basis of clinker; therefore, the formation of belite and alita determine the nature of the clinker formation (Figure 2).

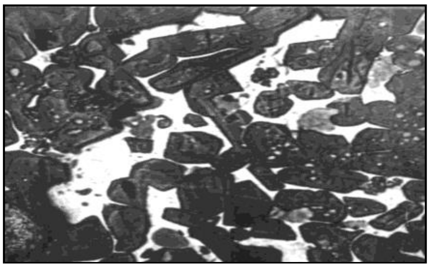
The exothermic effects at 1200 and 1260 ºC specify the belite formation C_2S. The DMS tails in the raw mix determine multistage of belite formation, which is stipulated by gradual transformation of man-made minerals into the intermediate phases C_2AS, C_3MS_2, out of which C_2S is formed.

The onset of belite formation is determined by the complexity of the silicon-oxygen grounds reorganization of the skarn minerals into an orthosilicate structure and increases in the following order: insular like (garnets, epidote) → chain like (diopside) → strips like (actinolite) and layered like (chlorite) → frame like (albit, scapolite).

The belite formation intensity depends on intermediate silicate phases stability. A low temperature of molten appearance (endoeffect at 1140 ºC), the belite formation speaks of extra ability of reaction of tails containing mixture in the 1050 ºC – 1150 ºC temperature interval. The terminate stages of clinker formation in the mixes of M and C types take place with the extra molten quantity involvement. Owing to this, the baking outset in the tails containing feedstock is provided at such temperatures when in the standard mix the reactions are still going in the tough phase.

The interaction intensity with liquid phase availability is provided by a lower molten velocity owing to the favorable ratio of magnesium oxide, sulfur trioxide, alkali content; catalytic impurities of titanium, manganese, nickel, copper compositions. The formation of the highly basic phase C_3S occurs in a very narrow interval, often bordering on the melting point (destruction) of the structure of minerals or intermediate phases formed from them.

As a result the clinker synthesis temperature out of tails containing stocks reduces on 30-50 ºC, a length of isothermal sintering gets lower. The alite formation features predetermine the crystallizing nature of this phase. The presence of local melt zones with different concentrations of sulfur trioxide causes uneven growth of alite grains in the clinker of the M type (Figure 3).



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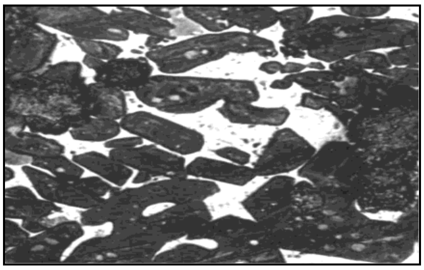

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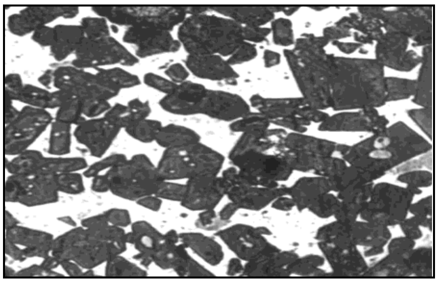

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Figure 3 – A clinker microstructure (zooming x 300) from raw mix with various DMS tails content: 1 – M type; 2 – C type; 3 – testing mix

Therefore, along with clearly defined prism like (size 20–30 µm), the individual alita crystals have amoeba-like shape (size 60–120 µm), contain inclusions.

C_2S crystals enlargement was supposed to be expected when lowering the temperature of belite formation at DMS tails availability. But having easily decomposed calcic silicate in the synthesis stipulates a “finely-dispersed” belite formation.

Lowering a basic capacity of clinker without reducing its hydraulic activity is an essential saving store of fuel and power resources. Such as a phase content alteration allows to lower the burning temperature to 1350 ºC and heat quantity to 125 – 250 kJ/kg of the clinker [1, 8].

The increased temperature of belite formation, the presence of doping impurities in the mixtures with washery refuses ores provide the activity of low-base clinkers, characterized by SC=0.73-0.83 (Figure 4).

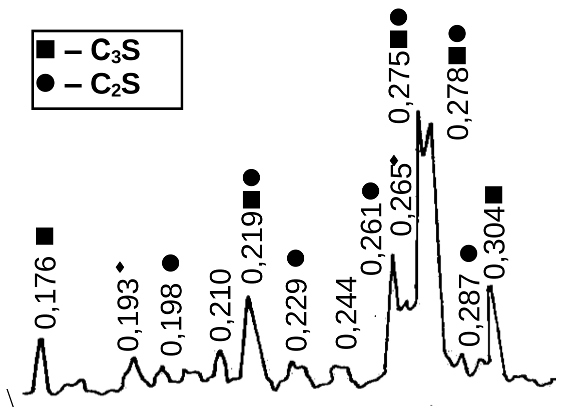

Figure 4 – X-ray pattern of low-basic clinker from the raw mix of M type

A later belite formation, excluding its combination with decarbonization and C_2S passivation, is aimed at increasing the disequilibrium of the clinker phases. In man-made raw material clinker, the phases inherit a feature of the crystalline structures of natural minerals, which consists in close contact of basic and isomorphic elements. This increases the deformation of the lattice and the doping of the clinker phases.

The low-base clinker activity depends on alite content, with intensive hydration of which the necessary amount of portlandite is released to form a crystalline intergrowth in a hardening stone. The low alite clinker in terms of refuse ores providing high cement grade strength is characterized by a delayed hydration at the initial hardening stage. The clinker activation exclusively by means of belite phase is technologically complicated and frequently poorly effective. On the other side, alite hydration capability is stipulated by modification composition and C_3S crystal state, greatly sensitive to the alteration of raw materials reacting capacity. It seems that the complex chemical and mineral composition of the enrichment waste of skarn-magnetite ores will provide the ability to control the rate of clinker formation, structure and activity of the phases, especially alite.

Producing of C_2S substantial amount in the narrow range of high temperatures increases the level of thermal influence on the material owing to localized exotherm reaction and enhance alite production. The convergence of the C_2S and C_3S synthesis processes contributes to the enhancement of the disequilibrium of the phases and an increase in their activity. The stepwise nature of the minerals saturation of skarn-magnetite wastes causes a portion of the heat when they interact with CaO. The calculations have shown that the thermal effect of the reactions decreases as the basic capacity of the initial silicates increases. Consequently, the multistep process of belite formation is accompanied by a smooth heat release in an extended temperature range. To localize the exothermic effect, the stages merge is advisable. This is achieved by removing part of the CaO from the sphere of low-temperature interactions, which will limit the solid-phase saturation of the natural silicates and increase the intensity of heat release at elevated temperatures.

The influence of coarse ground waste of different fractional composition (particle size up to 200 microns) on the clinker formation was investigated. With an increase in the grains concentration, the contact surface of the reacting particles is reduced and, as a result, low-temperature interactions are inhibited, the stability of natural silicates increases. High-silica coarse-ground man-made component melts at a temperature of 1150 - 1170 °C, which is lower than the temperature of a eutectic melt appearance formed by the clinker components (1200 - 1250 °C). The liquid phase arises in the form of discrete areas from which it spreads, forming impregnation zones in the lime component of the cake. With the advent of the melt, the processes of formation of highly saturated intermediate compounds (C_2AS, C_3MS_2) are activated. The major belite part is formed in a narrow range of the high temperatures (1200–1250 °C). A localized heat generation provides a “thermal shock”, an increase in thermal stress in the material, and contributes to the acceleration of clinker formation (Figure 5).

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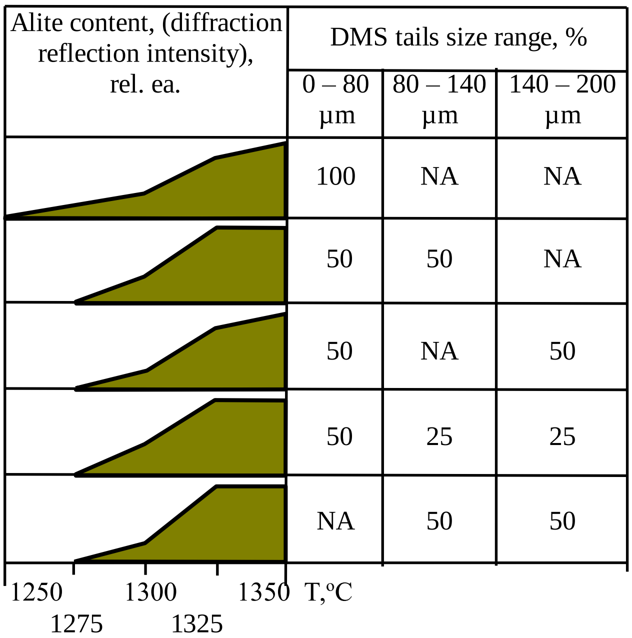

Figure 5 – DMS tails dispersion impact on the clinker phases formation

Hemical, mineral, and fractional heterogeneity of the coarse-ground particles of anthropogenic component causes differences in the melt properties and the rate of alite formation in the individual areas of the calcined material. The spreadability of the liquid phase is considered a factor is limiting alite formation.

Obviously, the melt penetration rate is determined not only by the rheology of the liquid phase, but also by the volume of the impregnated material. The smallest grain hollowness is achieved with limited particle size and polyfraction activity of the grains.

The molten zone change and the rate of clinker formation determine an evident poly-grain of clinker microstructure, lead to the phases formation of nonuniform crystal structure. In the areas enriched with the liquid phase, C_3S crystals become larger when combined into blocks, the process is intensified with an increase in the molten mobility. A fine-crystalline structure is formed in areas with increased viscosity of the liquid phase, where the convergence of C_3S crystals is difficult. The different nature of the molten spreadability from polyfractional grains contributes to the enrichment of the microstructure with fine-crystalline zones. A variety of alite crystalline forms ensures its high hydration activity during various periods of hardening. The increase in the small C_3S crystals proportion (20–40 µm) contributes to the growth of early stone strength by 50 % (Table 1).

The efficiency of using DMS tails can be estimated by the heat consumption in theory upon the physical and chemical transformations in a mixture. The heat effect of clinker formation (CHE) was defined as the algebraic sum of the heat values of endothermic processes (limestone decarbonization, clay minerals dehydration, a liquid phase formation) and exothermic reactions (pyrite oxidation introduced by DMS tails; clinker phases formation). The decrease in the carbonate oxides content of calcium and magnesium, hydrated water in mixtures with wastes is taken into account at calculations.

A decarbonization process differs by its biggest energy intensity, 1780 kJ is required for $CaCO_3$ 1 kg dissolution.

Table 1 – DMS tails dispersion impact on the clinker properties

DMS tails size range, %			Alite crystals content, %, by range, µm			Cement samples resistance 2x2x2 cm, MPa	
0–80 µm	80–140 µm	140–200 µm	20–40	40–70	80–100	1 d	28 d
100	not	not	12	58	30	28	83
50	50	not	17	45	38	31	88
50	not	50	10	46	44	24	91
50	25	25	39	26	35	42	87
not	50	50	42	38	20	44	89

Calcium and magnesium silicates existence in DMS tails contributes to a decrease in the limestone proportion and to a reduction in the heat cost for decarbonization in mixtures of an equal basic capacity by 4–11 %. Saving heat on the carbonates dissociation is also achieved by changing the SC from 0.95 to 0.77; for M, C type mixtures and the testing, this indicator is 165, 151 and 116 kJ/kg of clinker, respectively. Remarkable is the estimated heat consumption for highly basic tail-containing mixtures of the M and C types is less or comparable to the value for a low basic capacity mixture from standard raw materials.

As the clay component is eliminated from the mixture, the thermal costs of dehydration of minerals are reduced, and the savings reach 47–150 kJ/kg of clinker. Low-temperature interactions in the feedstock with waste are associated with pyrite and magnetite transformations. The heat generated during the pyrite oxidation compensates a large part of the heat consumption in the tail-containing mixtures. The

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energy benefit from the presence of pyrite in the tail-bearing feedstock is comparable to or exceeds that of the reduction of basic capacity for the blank mixtures.

The economic efficiency of mixtures with wastes is illustrated by the change in the specific fuel equivalent consumption for the clinker formation (Figure 6). With the introduction of DMS tailings into a mixture, fuel economy in the Fuel and Energy Complex is 6–19 kg/ton of clinkers of equal basic capacity.

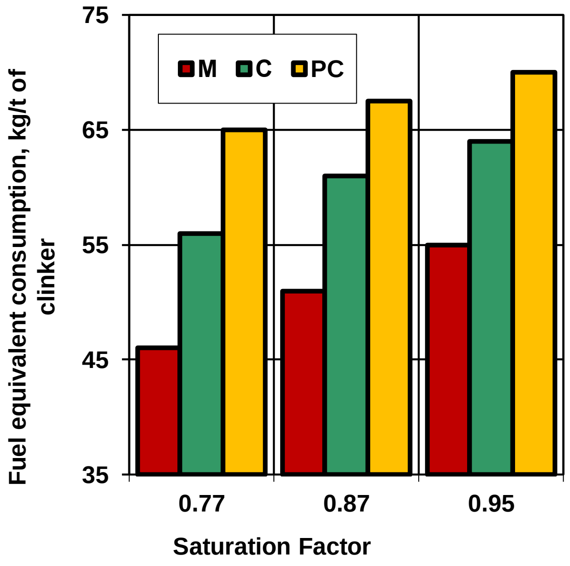

Figure 6 – Specific fuel consumption to the clinker formation for raw mixtures of various content

The transition from the standard mixture to the low-base tail-containing feedstock ensures a reduction in the unit cost of reference fuel by 18–34 %.

The environmental aspects that determine the benefits of using washery refuses of skarn-magnetite ores are the efficient use of natural resources, reducing fuel and energy costs, obtaining a scavenging from the man-made materials. A serious environmental problem in the process of clinker burning is the emission of various gaseous substances. The reduction of the carbonate component in the tail-containing feed stock, especially of the low-basic onesaccording to the heat engineering calculations, leads to a decrease in carbon dioxide emissions by 6–15 %. Moreover, the CO_2 emission in the combustion products of the fuel is reduced by decrease of fuel consumption.

Conclusions. The summary of the study results allows distinguishing the following preferences of using the washery refuses of skarn-magnetite ores in the cement clinker production:

- production effectiveness of the man-made aspect: DMS tails - disintegrated mass with a low humidity, stable chemical composition; does not require special sealing of the equipment during transportation and application; does not affect the grindability of the raw material feed stock;

- radiation and environmental safety of the raw materials: DMS tails are specified by the low values of the specific effective activity of the radioactive nuclides, the absence of toxic emissions; heavy metals are mostly low volatile and are able to concentrate in the clinker during calcination;

- reactive capacity of the raw material mixture: the activity of phase transformations, oxidative exothermic reactions, decarbonization temperature decrease, acceleration of the liquid-phase interactions;

- the cement clinker quality: the phase composition is represented by active modifications of alite and belite; high hydraulic activity of the clinkers of various basic capacity;

- fuel and energy costs reduction: achieved by reducing the thermal effect of the clinker formation due to the non-carbonate lime availability in the wastes; exothermic processes in the tailing raw mix; catalytic and dopant impurities availability in the DMS tailings; at the transition to the low-base clinkers;

- saving of the raw materials: provided as a result of the reduction of the clinker basic capacity; lowering of the specific consumption of the raw mix; a decrease in the raw feed stock of part of the natural carbonate and aluminosilicate raw materials;

- the environmental problems solution: large-tonnage wastes disposal; reduction of gaseous and dust emissions during the clinker production.

Consequently, washery refuses of the skarn-magnetite ores are an effective cement raw material, providing high-quality portland cement clinker using low-energy and resource-saving technology.

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