Розроблено нові склади сировинних сумішей для виготовлення клінкеру з урахуванням фактору мінімізації вмісту барвних оксидів із використанням створеної комп'ютерної програми «КЛІНКЕР». Виявлено особливості структури та фазових перетворень дисперсних систем при варіюванні різновидів збагаченої карбонатної, алюмо- і кремнеземвмісної сировини України. Показано зв'язок оптико-фізичних властивостей кристалічних фаз з білизною клінкеру, в тому числі підвищення білизни при інтенсифікації синтезу $C_{12}A_7$, C_2AS, C_3A з відносно меншим коефіцієнтом заломлення світла

Ключові слова: цемент білий, комбінований спосіб, клінкер, суміш сировинна, каолін, склад хімічний, система дисперсна, структура коагуляційна, фази кристалічні, білизна цементу

Разработаны новые составы сырьевых смесей для изготовления клинкера с учетом фактора минимизации содержания красящих оксидов с использованием созданной компьютерной программы «КЛИНКЕР». Установлены особенности структуры и фазовых превращений дисперсных систем при варьировании разновидностей обогащенного карбонатного, алюмо- и кремнеземсодержащего сырья Украины. Показана связь оптико-физических свойств кристаллических фаз с белизной клинкера, в том числе повышение белизны при интенсификации синтеза $C_{12}A_7$, C_2AS, C_3A с относительно меньшим коэффициентом преломления света

Ключевые слова: цемент белый, комбинированный способ, клинкер, смесь сырьевая, каолин, состав химический, система дисперсная, структура коагуляционная, фазы кристаллические, белизна цемента

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ANALYSIS OF RAW MIXTURE COMPOSITION AS A FACTOR OF INCREASE OF PORTLAND CEMENT CLINKER WHITENESS

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1. Introduction

Owing to its high decorative properties, performance characteristics and numerous areas of practical application, white Portland cement is an advanced material of the 21st century. However, the cement production is impeded by the limited quantity of raw material and complicated technologies that include special clinker bleaching operations.

The development of non-metallic ore industry in Ukraine has ensured the production of new varieties of mineral raw materials with various enrichment rates. The efficiency of these varieties for the production of white Portland cement can be estimated after close study and analysis of differences in their chemical and mineralogical compositions. It is necessary to optimize compositions of the initial mixtures for clinker production as well as affect their structure formation and the properties of cement, including its whiteness.

In the production technology for white Portland cement, it is important to identify peculiarities of the phase composition of clinker as the main structural characteristic and to determine how the clinker whiteness depends on the optical and physical properties of largely varied individual phases.

The relevance of the study is determined by the possibility to enhance scientific understanding of the structure and properties of white Portland cement through the use of new varieties of raw materials with different enrichment rates, including potentially suitable for export deposits of Ukraine.

2. Literature review and problem statement

The development of cement production requires an expanded resource base, upgraded technologies and equipment, lower specific energy consumption, and an enhanced range of high quality products [1]. In addition, the production and use of white Portland cement is a special case [2, 3].

The world experience in producing white Portland cement is based on the technological requirements to minimize contamination of raw materials and clinker, increase the temperature of clinker burning up to over 1450 °C, use special operations for clinker bleaching at the final stage of its burning and cooling, and improve the final product's fineness [4]. These requirements considerably impede the development of white cement production.

The current technologies of producing silicate materials, including cement, are based on the scientific principles of physical chemistry, physicochemical mechanics of disperse structures, and modern material studies that examine the relationships in the system "composition-structure-properties". Under the provisions of physicochemical mechanics, the technological process of producing silicate materials is essentially the process of their formation and successive transformations of their structures. Thus, there are three basic types of structures: coagulated, condensed, and crystalline [5, 6]. Therefore, an improved quality of silicate materials (including cement) and an effective control of their properties require a comprehensive analysis of the structure formation and the involved production technologies. However, the existing studies of white cement technologies limit the value of feedstock composition to the general requirement of restricting the content of coloring oxides in it. Meanwhile, the researchers do not consider the effect of mineralogical compositions of aluminum- and silica-bearing components on the structure and properties of the final product.

It is proved that the properties of Portland cement are mainly determined by the composition and structure of clinker that is formed in burning the raw mixture [7, 8]. The scientific views of the technology of cement production are based on the analysis of the phase composition of clinker resulting from physical and chemical interaction of oxides – waste from burning the lattices of rock-forming minerals that constitute the feedstock [9, 10]. Numerous studies focus on crystal formation in calcium silicates such as $3CaO{\cdot}SiO_2$ and $2CaO{\cdot}SiO_2$, tricalcium aluminate $3CaO{\cdot}Al_2O_3$, and tetracalcium aluminoferrite $4CaO{\cdot}Al_2O_3{\cdot}Fe_2O_3$ [11, 12], whereas the properties of cement depend on the rate of its crystalline phases. Since the reviewed studies lack attention to the effect of optical and physical properties of clinker minerals on the degree of clinker and cement whiteness, it becomes a relevant aspect of this research.

3. The purpose and objectives of the study

The study was aimed at analyzing the effect of enriched raw material varieties on the cement structure and whiteness in combined cement production.

To achieve this goal, we set the following tasks:

– to calculate (by means of computer technologies) rational compositions of the raw mixtures;

– to identify the peculiarities of coagulation of aqueous systems of raw slurries;

– to identify the particular phase composition of Portland cement clinker as its whiteness factor;

– to determine the main technological parameters of making Portland cement clinker and cement whiter on the basis of enriched raw materials.

4. An optimal raw mixture composition

The objects of study were samples of carbonate, aluminum- and cilica-bearing raw materials, as well as mixtures thereof used in the manufacture of white Portland cement. The selected chalk came from the Vovcheyarivka, Novhorod Siversky and Zdolbuniv chalkpits, whereas kaolins came from the Volodymyrivka, Hlukhivtsi and Prosyana deposits (Ukraine). In addition, the alumina component was represented by aluminum hydroxide, whereas the silica one – by quartz sands from the Avdiyivka, Vyshneve (Starco sand) and Novoselivka (quartz dust) sand quarries (Ukraine).

The chemical composition of the Novhorod Siversky enriched chalk sample MMS-1 is characterized by the lowest content of SiO_2 and a higher amount of Al_2O_3 than the Vovcheyarivka and Zdolbuniv samples. The quantitative mineralogical compositions of the chalk samples show that although all of them have the same contents of basic rock-forming minerals, the Novhorod Siversky and Zdolbuniv samples are distinguished from the Vovcheyarivka sample by a slightly higher concentration of calcite and a significantly lower content of quartz.

Among the kaolin samples, the chemical composition of KV-3 is characterized by a higher content of coloring oxides $Fe_2O_3+TiO_2$ (2.06 to 0.64–1.58 wt. %). Enriched KS-1 and KVF-90 are characterized by higher concentrations of Al_2O_3 (36.2–36.8 wt. %) and lower ratios of SiO_2:Al_2O_3 (1.3 vs. 1.8 for KV-3 and 3.6 for KSSK).

The mineralogical composition distinguishes the above kaolin samples from the Kryvyn polymineral clay, since the former have significantly higher contents of kaolinite, smaller amounts of hydromica, calcite, and, which is important, hydroxides of iron and rutile.

Quartz dust is distinguished from the Avdiyivka and Starco sand varieties by its lowest content of coloring oxides, much higher dispersity, and developed specific surface of 2720 cm^2/g.

The study applies modern methods of physical and chemical analyses [13, 14], which complement each other, as well as standardized testing of the properties of raw materials, cement, and its compounds to determine the chemical composition, particle-size distribution, dispersity, and specific surface. The thermal analysis was made with the use of the Paulik-Paulik-Erdey derivatograph (OD-1000) at the heating rate of 10 deg/min, whereas the X-ray phase analysis – with use of diffractometer DRON-3M (Cu Kα radiation is 1–2, voltage – 40 kV, current – 20 mA, and rate – 2 deg/min), the electron microscopic analysis – with the help of the scanning electron microscope SELMI REM-106I, and the analysis of structural, mechanical and rheological properties – with the use of the Weiler-Rebinder apparatus and Reotest-2 (Ukraine).

According to modern technologies of producing white cement from raw mixtures of particular compositions, we prepared sample slurries that were dried, burnt, and subjected to fine grinding.

The raw mixtures' compositions were calculated with the use of the new computer program CLINKER in the programming language C# that can run on any PC with the operating system Windows, NT and later versions [15, 16].

The program was designed to determine the compositions of binary and multicomponent raw mixtures for the manufacture of clinker with specified characteristics, whereas the number of possible raw materials is unlimited.

This operating rate of calculation provides a significant amount of analytical information on how clinker characteristics, including the concentration of coloring oxides and the projected phase composition, depend on qualitative and quantitative contents of the feedstock components (Fig. 1).

Thus, the analysis of a ternary mixture based on the Novhorod-Siversky chalk with the use of aluminum hydroxide, SF values varied by 0.05 in the range of 0.80–0.95 and silica modulus number n varied by 0.50 in the range of 2.00–3.50 has proved the possibility to minimize the content of coloring oxides to 0.20–0.21 wt % and, thereby, make clinker whiter due to the projected reduced formation of iron-containing crystalline phases in the process of burning (Fig. 2). There have been significant differences in the calculated phase composition: when n=2.0, the C_3S content can grow from 33.26 to 64.7 wt % and C_2S can decrease from 37.6 to 8.6 wt %; when n=3.5, C_3S rises from 37.98 to 73.00 wt % and C_2S decreases from 42.98 to 9.72 wt %. When n=2.0, the projected C_3A

content equals to 26–28 wt %; when n=3.5 – 17–18 wt %. Meanwhile, the C_4AF content makes up 0.61–0.64 wt %.

It is found that the new computer program CLINKER provides rapid calculation of the raw mixture content to obtain Portland cement clinker with a minimum content of coloring oxides and probable formation of iron-containing phases in burning. Accuracy of the results depends solely on the source data error, i. e. accuracy of the chemical composition of possible raw materials. The calculation time virtually does not depend on the number of input raw materials.

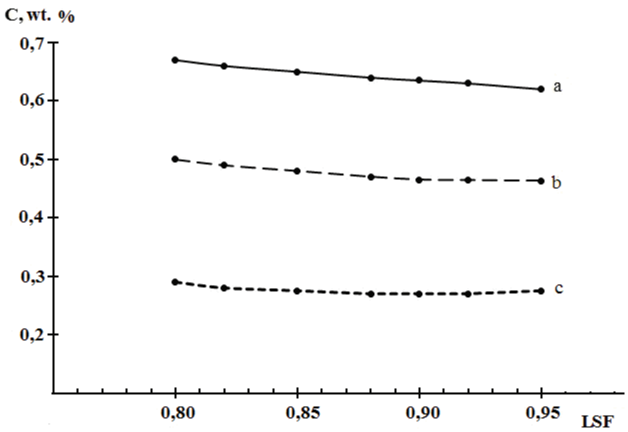


Fig. 1. Dependence of the content of coloring oxides (C) on the saturation factor (SF) of clinker made of binary mixtures with kaolins: *a* — KV, *b* — KS-1, and *c* — KSSK

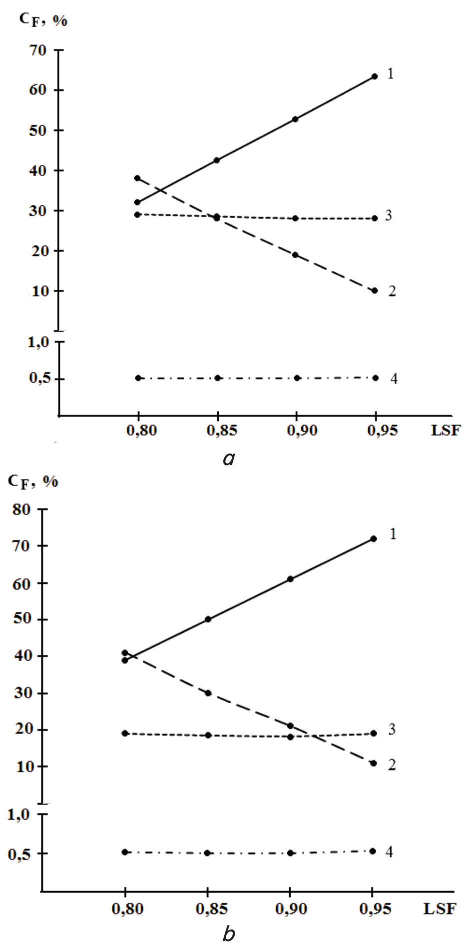


Fig. 2. Dependence of crystal phase formations C_3S(1), C_2S(2), C_3A(3) and C_4AF(4) on the clinker saturation factor: *a* — when n=2.0, *b* — when n=3.5

The new PC program has revealed binary and ternary mixtures for further study of their structure formation in the production technology of white Portland cement [17].

5. Peculiarities of the slurry coagulated structure

Coagulative characteristics of the aqueous systems of raw mixtures – slurries – were studied to determine the technological parameters of wet and combined production of white Portland cement, provided small amounts of mineralizing substances are added.

Analysis of the deformation processes has proved that the samples under study differ significantly in the character of deformations – quick elastic ε_0', slow elastic ε_2', and plastic $\varepsilon_1'\tau$ [18, 19]. In aqueous dispersions of ternary mixtures based on the Vovcheyarivka chalk with kaolin KV-3, Avdiyivka sand (sample E1) with kaolin KS-1, and Starco sand (sample E5) with kaolin KVF-90 and quartz dust (sample N24), due to the nature of deformations, sample E1 belongs to the second structural-mechanical type, when $\varepsilon_2'>\varepsilon_1'\tau>\varepsilon_0'$, whereas E5 and N24 – to the fifth type, when $\varepsilon_1'\tau>\varepsilon_2'>\varepsilon_0'$ (Table 1, Fig. 3). Meanwhile, there are significant differences in numerical values and the ratio of these kinds of deformation.

Table 1

The structural and mechanical properties of raw slurries

Sample code	$E_1 \cdot 10^{-4}$, Pa	$E_2 \cdot 10^{-4}$, Pa	P_{k1}, Pa	$\eta_1 \cdot 10^{-2}$, Pa·s	Λ	$\frac{P_{k1}}{\eta_1} \cdot 10^2 s^{-1}$	θ_1, s	$E_\varepsilon \cdot 10^{-10}$, J/cm^3
E1	12.06	1.95	2.1	78.44	0.86	0.01	4,163	1.35
E5	8.73	2.05	1.10	9.34	0.81	0.12	562	0.60
N24	8.88	5.30	0.39	6.87	0.63	0.06	207	0.60

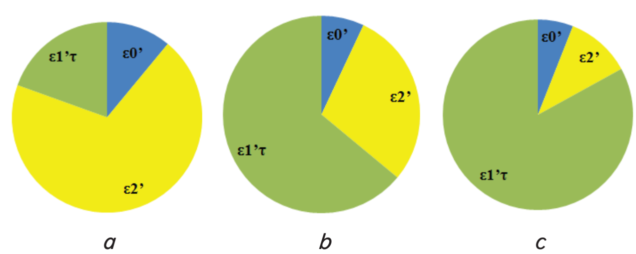


Fig. 3. The ratio of deformations and structural-mechanical types of aqueous systems of ternary mixtures based on the Vovcheyarivka chalk: *a* — E1 (type 1); *b* — E5 (type 5); *c* — N24 (type 5)

According to the concepts of physicomechanical properties of disperse structures, the prevailing development of plastic deformations $\varepsilon_1'\tau$ along with the lower values of the Shvedov viscosity η_1 and the Bingham plastic viscosity η_m^X reveal a better fluidity of slurry mixtures E5 and N24.

The overall research findings on the structural, mechanical and rheological properties of aqueous dispersions have confirmed their dependence on the chemical and mineralogical composition, particle size and concentration of the disperse phase, as well as chemical composition and properties of the dispersion medium [20, 21].

The research has found that varying the types of kaolin and quartz sand, with different degrees of concentration and dispersion in the raw mixture, is a positive factor for the structure coagulation and technological properties of white cement slurries. This allows controlling important parameters of production, such as viscosity, fluidity and kinetic stability of a slurry. In the aqueous systems of binary mixtures based on the Novhorod-Siversky chalk with unenriched kaolin KSSK, compared to a system with enriched kaolin KS-1, the Shvedov viscosity η_1 decreases 5.2 times, the Bingham plastic viscosity η_m^X – 5.1 times, and the conventional strain module E_ε – 2.4 times.

The use of aluminum hydroxide (instead of kaolin) as alumina-containing material at the same concentration of the disperse phase in a raw mixture also increases the slurry fluidity by reducing its viscosity and lowering the quantity and strength of the structural particles' contacts. In an aqueous system of a mixture with aluminum hydroxide, compared to a system with kaolin, the Shvedov viscosity η_1 decreases 2.9 times, the Bingham plastic viscosity η_m^X – 3.1 times, and the conventional strain module E_ε – 2.9 times.

The use of mineralizing substances, which are necessary to intensify clinker sintering, essentially affects the coagulation parameters of aqueous systems of the original raw mixture – the slurry – by changing the chemical composition and properties of the dispersion medium. The degree of effect depends on the kind and quantity of a mineralizing substance as well as mineralogical composition of the disperse phase.

The observed decrease in viscosity and better fluidity of the slurry mixtures under study reveal a possibility to reduce its their moisture as a resource saving factor in the technology of white cement production.

6. The phase composition of clinker as a factor of its whiteness

The potential tendencies and probability of the formation of crystalline phases in burning were assessed by the methods of thermodynamic analysis of silicates [22]. The calculation results indicate a thermodynamic possibility of obtaining mayenite $C_{12}A_7$ and gehlenite C_2AS in the phase composition of clinker via solid-phase reactions. Thus, mullite can interact with tricalcium aluminate to synthesize mayenite and gehlenite (reactions № 1–3), whereas interacting with dicalcium and tricalcium silicates it forms gehlenite and silica (reactions № 4–5):

$$40C_3A+9A_3S_2=18C_2AS+7C_{12}A_7, \quad (1)$$

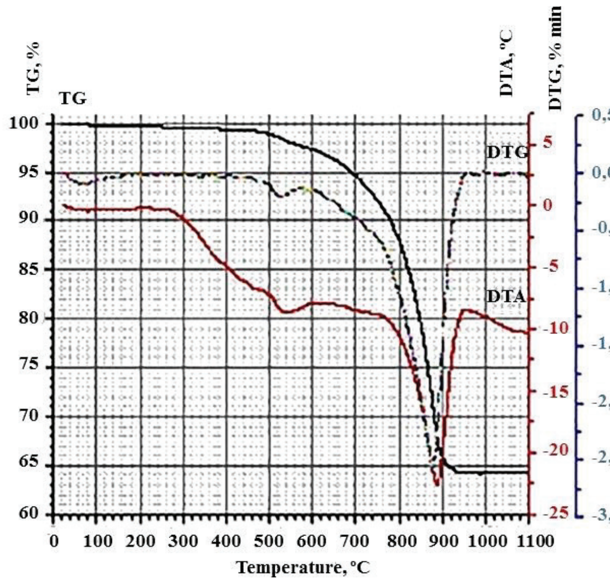
$$20C_3A+3A_3S_2=C_2AS+4C_{12}A_7+5C_2S, \quad (2)$$

$$33C_3A+6A_3S_3=9C_2AS+6C_{12}A_7+3C_3S, \quad (3)$$

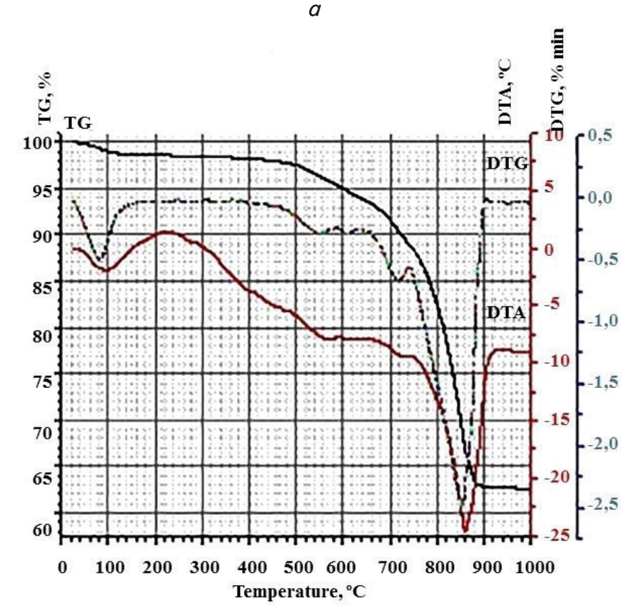
$$3C_2S+A_3S_2=3C_2AS+2S, \quad (4)$$

$$2C_3S+ A_3S_2= 3C_2AS+5S. \quad (5)$$

The obtained experimental data confirm the results of thermodynamic analysis and reveal some differences in the kinetics of physical and chemical processes that occur in clinker burning if the raw mixture composition is varied. According to the complex thermal analysis, if kaolin KV-3 and the Avdiyivka sand (E1) are replaced by the enriched KS-1 and Starco sand (E5), there appears a shift in the maximum endothermic effect associated with the destruction of kaolinite and calcite lattices to the area of 10–15 °C lower temperatures (Fig. 4). It is associated with an increase in the degree of deficiency of the basic rock-forming minerals and intensified sintering.



a



b

Fig. 4. The results of the thermal analysis of mixtures based on the Vovcheyarivska chalk with kaolin varieties: *a* — mixture E1; *b* — mixture E5

Therefore, the electron microscopy of the samples of clinker from raw mixtures shows that intensified sintering affected by mineralizing additives is accompanied by a perfected morphology of crystalline phases and the development of a glass phase.

The samples of clinker for obtaining white Portland cement are characterized by lack of iron-containing crystalline phases, intensified crystal formation of calcium silicates C_2S and C_3S, as well as the development of crystalline phases of gehlenite C_2AS and mayenite $C_{12}A_7$ (Fig. 5).

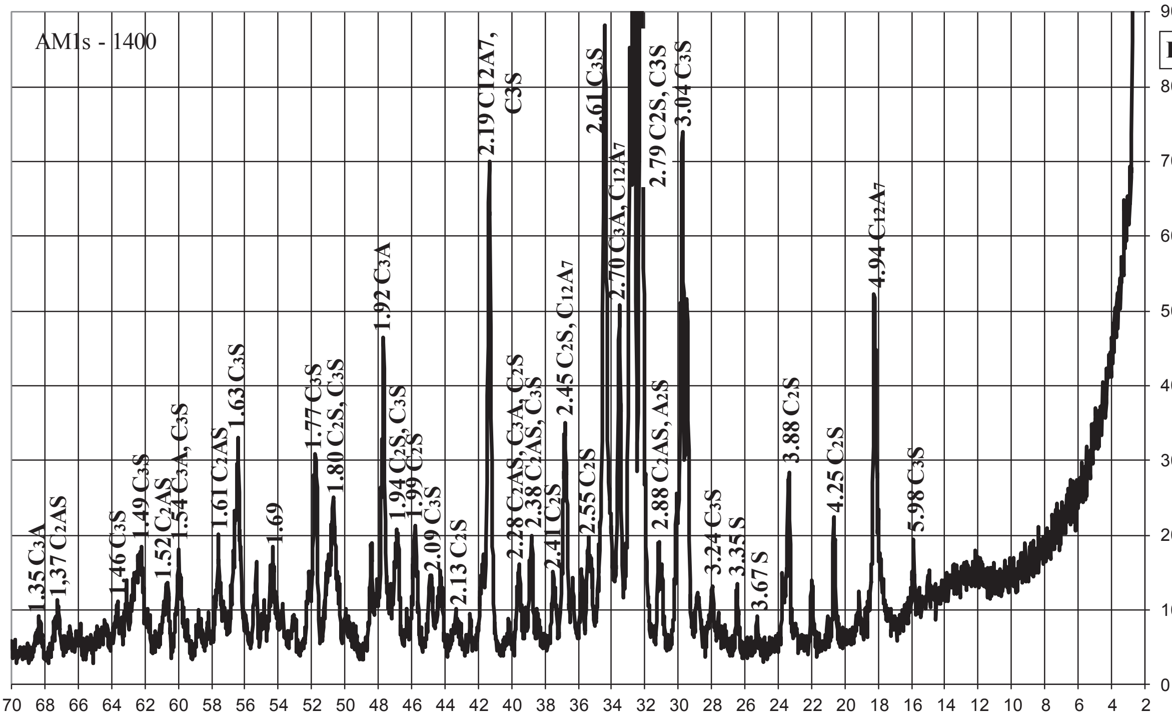


Fig. 5. A diffractogram of clinker AM1s after its burning at 1400 °C

The phase composition (Table 2) of the obtained clinker of white Portland cement is distinguished from ZdK clinker of chalk-based grey cement by a significantly lower (9 times) quantity of iron-containing phases C_4AF and CF (0.0–1.1 vs. 9.8 %), higher quantity of C_2S (30–36 vs. 19 %), lower proportion of C_3S:C_2S, as well as formation of mayenite and gehlenite with an overall growth of crystalline phases C_3A, C_2AS, and $C_{12}A_7$ (15.3–23.2 vs. 7.2 %).

The research reveals correlation between the whiteness of clinker and optical-physical properties of individual phases [23]. The presence of ferrohydroxides in the raw mixture of grey cement leads to the formation (in its clinker) of iron-containing phases characterized by optical anisotropy as well as higher values of density and refractive index: Ng=2.29 for $2CaO{\cdot}Fe_2O_3$ and Ng=2.46 for $CaO{\cdot}Fe_2O_3$ are, respectively, reddish brown and black. This fact predetermines recommendations to limit the content of coloring oxides, especially Fe_2O_3, in the raw material for producing white cement.

Table 2

The crystalline phases of clinker

Sample code	The content of minerals, wt. %							
	C_3S	C_2S	C_2AS	C_4AF	C_3A	$C_{12}A_7$	CF	Quartz
ZdK	62.3	19.2	–	9.1	7.2	–	0.7	–
E1	51.4	31.1	1.0	1.1	9.0	5.8	–	0.5
N30	40.0	36.0	2.1	0.2	15.0	6.1	–	0.5
N36	46.0	36.0	0.6	–	9.0	6.6	–	–
AM1s	47.0	36.0	2.8	0.2	7.3	5.3	–	1.0
N22	43.0	37.0	2.5	0.3	9.0	5.7	–	0.5
N24	49.5	34.5	1.1	–	9.5	4.7	–	0.2
M3	51.1	30.2	0.9	–	13.5	4.0	–	–

Among the main clinker minerals, $4CaO{\cdot}Al_2O_3{\cdot}Fe_2O_3$ is distinguished by the highest refractive index Ng, and there is revealed a sequence of $C_{12}A_7$<C_2AS<C_3A<C_2S<C_3S<C_4AF.

Clinker samples of white Portland cement produced of the developed raw mixtures are characterized by the absence of iron-containing crystalline phases, intensified formation of calcium silicates C_2S and C_3S, and the development of gehlenite C_2AS and mayenite $C_{12}A_7$ phases. The latter are characterized by a cubic crystal system, optical isotropy, transparency, and the lowest refractive index Ng among major clinker minerals.

The technology of white cement production has been tested in the laboratories of the National Technical University of Ukraine "Igor Sikorsky Kyiv Polytechnic Institute" and the I. M. Frantsevich Institute for Problems of Materials Science of the National Academy of Sciences of Ukraine (Kyiv). There have been tested the newly developed mixture compositions based on binary and ternary systems, such as: chalk – kaolin, chalk – kaolin – quartz sand, chalk – kaolin – aluminum hydroxide, and chalk – aluminum hydroxide – quartz sand with mineralizing additives.

The test findings show that Portland cement clinker produced of the new research-based raw mixtures is significantly whiter than clinker produced of the already known composition E1: 80–83 vs. 73 %.

White cement production included dosing clinker with additives and fine grinding of the components in a spherical mill to the fineness with a residue 10–11 wt. % on a sieve 008. Plaster stone (5 wt. %) and thermally activated kaolin (5–15 wt. %) were used as additives affecting the characteristics of the final product (setting time, strength, and whiteness). It is determined that the use of newly developed mixtures from raw materials of different regions of Ukraine at observing the main technological parameters ensures obtaining a white Portland cement whose main characteristics meet DSTU B V.2.7-257:2011, whereas the whiteness is not less than 82 % – the standard requirements of leading manufacturers (EN 197-1:2011 and ASTM C 150) (Table 3).

Table 3

Characteristics of white Portland cement produced of the newly developed mixtures

Characteristics	Sample code								
	E1	M3	AM1s	N22	N21	N24	N30	N36	N35
Residue on a sieve 008, %	10.0	10.5	11.0	10.5	10.5	11.0	10.0	10.0	10.0
Beginning of setting, min	62	58	56	58	52	48	50	50	49
End of setting, h	10.5	9.5	9.5	9.5	9.0	8.5	8.5	8.5	8.5
Standard compressive strength, MPa	37.5	38.3	37.9	38.0	39.1	38.2	38.5	38.3	38.4
Whiteness, %	75	87	85	85	83	82	82	82	82

7. Discussion of the findings on the structure and whiteness of cement clinker

The effectiveness of the new computer program CLINKER is proved by its ability to rapidly calculate the initial mixture composition, provided the quantity of potential raw materials for manufacturing white Portland cement is unlimited. This methodological improvement allows analyzing how the content of coloring oxides and the projected clinker whiteness depend on its specified characteristics, if the types and concentrations of raw materials are varied.

Varied types of chalk, aluminum- and silica-containing materials of different origins and degrees of enrichment as well as added mineralizers affect coagulation and crystallization of silicate dispersions in the process of white cement production.

The identified differences in the characteristics of coagulated structures of slurry suspensions based on the chalk-kaolin system, i. e. changes in the Shvedov viscosity and the Bigham plastic viscosity, fluidity, and kinetic stability are associated both with the concentration of kaolinite in the disperse phase and the influence of mineralizing additives on the composition of the dispersion medium.

The experimental research has revealed the peculiarities of phase formation in burning clinker from the designed raw mixtures that fully correlate with the results of projected thermodynamic calculations. Nucleation in burning raw mixtures is determined by qualitative and quantitative compositions of rock-forming minerals and mineralizing additives.

The use of mineralizing substances intensifies destruction of the lattices of major rock-forming minerals in the mixtures, causes a 10–15 °C decrease in the maximum temperature of the respective endoeffects, and increases the rate of defects in dispersed particles, thereby, accelerating physical and chemical processes in clinker burning.

The synthesized Portland cement clinker is whiter owing to minimized contents of coloring oxides and iron-containing phases, as well as developed aluminum-containing crystalline phases with lower rates of light absorption, including mayenite $C_{12}A_7$, that affect the optical properties and increase the product's whiteness to over 80 %.

The new compositions of mixtures are based on enriched raw materials that ensure obtaining white Portland cement whose main characteristics meet DSTU B V.2.7-257:2011, whereas whiteness meets the standards of leading manufacturers (EN 197-1:2011 and ASTM C 150).

8. Conclusions

1. The study has proved the effectiveness of the developed computer program CLINKER in a rapid specifying of the composition of a raw mixture for white Portland cement clinker and analyzing how the projected whiteness depends on the set characteristics of the material, provided the kinds and concentration of raw materials are varied. By implementing the designed program we have determined the versions of raw mixtures with minimized contents of coloring oxides in clinker – from 0.74–0.77 to 0.11–0.13 wt. %.

2. The study shows that variation of the types of chalk, aluminum- and silica-containing materials of various enrichment rates and addition of mineralizers affect coagulative and technological properties of the white cement slurry, as well as allow controlling the viscosity, fluidity and kinetic stability as important parameters in wet and combined production.

3. The found differences in chemical and mineralogical compositions of raw materials and new mixtures determine the peculiarities of physicochemical processes and phase formation in burning clinker. The potential trends and probability of crystalline phases in burning were assessed with the use of thermodynamic analysis of silicates with regard to the oxide system $CaO-SiO_2-Al_2O_3$, provided iron oxide additives are minimized and the maximum temperature is limited to 1400 °C. Along with the formation of clinker phases, such as C_2S, C_3S, and C_3A, we have determined the thermodynamic possibility of mayenite $C_{12}A_7$ and gehlenite C_2AS formation through solid-phase reactions between the decay products and modified transformations of rock-forming minerals in raw mixtures.

4. We have revealed the peculiarities of phase formation in burning clinker from the newly developed raw mixtures, analyzed correlation of the types and physical properties of crystalline phases and the product whiteness, as well as pointed out the importance of the effect of optical characteristics of certain crystalline phases.

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