# INFLUENȚA ARGILEI UȘOARE CALCINATE ASUPRA PROPRIETĂȚILOR PASTEI DE CIMENT ÎNTĂRITE

# INFLUENCE OF THE CALCINED LIGHT LOAM ON THE PROPERTIES OF THE HARDENED PORTLAND CEMENT PASTE

**NAILIA R. RAKHIMOVA[1]*, RAVIL Z. RAKHIMOV[2], ALBERT R. GAIFULLIN[2], VLADIMIR P. MOROZOV[3]**

[1]*Faculty of Civil Engineering,Ton Duc Thang University (Ho Chi Minh City, Vietnam)*

[2]*Department of Building Materials, Kazan State University of Architecture and Engineering (Kazan, Russian Federation)*

[3]*Department of mineralogy and lithology, Kazan Federal University (Kazan, Russian Federation)*

***The increasing worldwide production of Portland cement and demand to reduce $CO_2$ emissions has resulted in the need to increase the volume and varieties of supplementary cementitious materials. The most promising source of raw materials for the production of supplementary cementitious materials is ubiquitous and unlimited reserves of polymineral clays. In this article the effect of calcined loam clays depending on its concentration, calcination temperature (400-800$^0$C), and specific surface area (250-800 $m^2$/kg) on the properties of Portland cement is studied. It is found out the calcined loam clays increase the compressive strength of Portland cement hardened paste up to 35%, density up to 1.4%, water resistance from 0.92 to 0.93-0.97, and decrease the water adsorption from 1 to 0.9%. The reasonability of production and application of calcined loam clays, which aren't lower in efficiency than high-priced metakaolin, is stated.***

***Keywords**: metakaolin, light loam, calcination, Portland cement paste, pozzolana, compressive strength, density*

## 1. Introduction

The acceptance in 1992 by UN World Summit in Rio de Janeiro in the framework of the "Agenda for the XXI century on" the concept of "sustainable development", focused on resource, energy conservation and improving environmental safety of the Earth's civilization, has led to a revision of the strategy of further development of all sectors industry to solve these problems [1, 2]. The global cement industry is responsible for about 5% of the global $CO_2$ emissions [3] which decrease is one of the actual problems nowadays. One of the priorities of its decision is to reduce the Portland clinker content in cements by introducing supplementary cementitious materials. Currently, blended Portland cements with mineral additions (CEM II -V) are already the main volumes of Portland cement production.

By 2050 the world cement production is expected to be 5-6 bln.t. [3] and increasing demand to reduce the volume of $CO_2$ emissions is linked with demand to expand the volume and range of applied supplementary cementitious materials (SCM). Currently, a wide range of mineral supplementary materials of natural and technogenic origin is being known and applied, the most effective of which varieties which have binding properties and pozzolanic activity. From natural pozzolans the flask, diatomite, tripoli, volcanic ash, tuff, pumice and tracks are used. However, deposits and reserves are unevenly distributed across countries and regions. In particular, almost 25% of the pozzolanic materials production in 2013 accounted for only two federal districts in Russia - the Central and Volga regions [4]. A similar problem - the uneven distribution across countries and regions of the sources and amounts of the most used mineral additives of technogeny origin – granulated blast furnace slag, fly ash, rice husk ash, silica fume. They also are characterized by significant economic and seasonal variations. Nowadays in some countries the produced Portland cements are incorporated 5 to 20% of limestone, which is inert that limits the expanding of its use [3].

Obviously, due to the above noted circumstances in recent decades in many countries the research activity was increased for study the feasibility and efficiency of widely spread clays for production of pozzolans, given the experience of mankind in the effective use of calcined clays as pozzolans from ancient times.

In the present time metakaolin which is calcined kaolin clay is being widely used as supplementary cementitious material [3, 5]. However, even in the nineteen forties in USSR as a result of research of 207 different clays from different regions, was found that only 11% of them turned out to be unsuitable for obtaining a pozzolan material with sufficient hydraulic activity. American company «Eddyston Cement Corporation» widely advertised in the US and Western Europe "New hydraulic cements", which are Portland cements in

* Autor corespondent/*Corresponding author*,
E-mail: nailia.rakhimova@tdt.edu.vn

which calcined clay is incorporated [6].

Metakaolin and calcined kaolin clays, even though high efficiency confirmed by numerous studies and application experience, can not be produced in sufficient amount as a pozzolanic material for Portland cement due to limited deposits and reserves in many countries and regions, high demand in them in other industries and high cost [7].

On this background, the polymineral clays including various clay minerals, relic minerals, and impurities, represents an unlimited source of raw materials for the production of pozzolans. Such "impure" clays are very widespread and the cement industry will not compete in the expensive fight for kaolin clay with other industries such as the paper or ceramic industries [3].

In this regard, systematic studies of pozzolanic activity and efficiency in the blended binder systems of other than kaolinite calcined minerals [8-15], calcined polymineral natural and man-made formulations based on different clay minerals [16-22], clay with different contents of kaolinite and its complete lack of [23-25], clays with predominant content of one of the clay minerals [26-28] are of high scientific and practical interest.

The 1stInternational Conference on Calcined Clays for Sustainable Concrete which was held in Lausanne (Switzerland) in 2015 proved the relevance of the research regarding the efficiency of pozzolans based on the calcined wide-spread polymineral clays [29].

In this paper the results of research regarding the effect of the calcined and ground light loam on the properties of the Portland cement paste in comparison with the properties of that containing metakaolin are presented.

## 2. Experimental

### *2.1. Materials*

The following materials were used as starting materials:

1) Light loam with the following mineral composition (wt. %) (Fig. 1): kaolin-3.25, montmorillonite-5.78, chlorite-1.02, calcite-0.66, quartz-51.70, albite-18.51, mica-3.08, microcline - 8.44, dolomite-7.58.

2) CEM I - Portland cement of 52.5 strength class and the following chemical composition and mineralogical composition (wt. %): CaO-63.0, $SiO_2$-20.5, $Al_2O_3$-4.5, $SO_3$-3.0; $C_3S$-67.0, $C_2S$-11.0, $C_3A$-4.0, and $C_4AF$-15.0. The specific surface area ($S_{sp}$) was 345 $m^2$/kg, apparent density was 1000 kg/$m^3$, water for normal consistency was 27%, initial setting time, and final setting time of the cement were 2 h 50 min, and 4 h 10 min, respectively.

3) Metakaolin (MK) had the following chemical composition (wt %): $SiO_2$-86.0–92.0% and $Al_2O_3$-43.2%. The apparent density, $S_{sp}$, and pozzolanic activity of MK were: 170–200 kg/$m^3$, 1200 $m^2$/kg and >1000 mgCa$(OH)_2$/g, respectively.

### *2.2. Methods*

The light loam was calcined at 400, 600, and 800 °C and the heating rate was 1.7, 2.5, and 3.3 °C/min, respectively. An exposure time at each temperature of 3 h was used. The calcined light loam was ground to 250, 500, and 800 $m^2$/kg.

XRD data were obtained using a D2 phase X-ray diffractometer in a θ-2θ configuration using the Bragg-Brentano method. The diffractometer used a Cu Kα radiation and operated at 40 kV and 30 mA. Data handling was performed by the DIFFRACplus Evaluation Package with the EVA search/match module. The PDF-2 ICDD database was used.

The powders were dry-blended using a hand mixer at low speed prior to adding the water.

The Portland cement pastes samples with admixtures of calcined light loam were prepared in cubic molds (2 cm × 2 cm × 2 cm). The compressive strengths of the cubes were tested after steam curing. Prior to steam curing, the cubes were stored 24 h at room temperature. It took 4 h to reach the desired temperature, and a dwell time at 90-95$^0$C of 6 h was used. The cubes were cooled over a period of 3 h. The average compressive strength for each mixture during each testing stage was calculated by carrying out six measurements.

A reference sample was used for comparison with the samples with MK and calcined light loam admixtures.

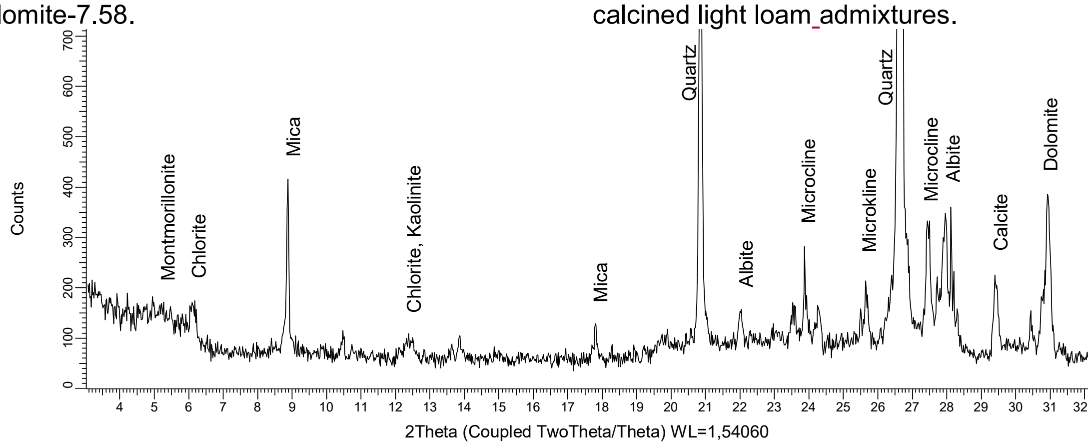


Fig.1. X-ray diffractogram of light loam.

**Table 1**

Properties of the Portland cement pastes with calcined light loam depending of calcination temperature and fineness

| Calcination temperature (°C) | Specific surface area ($m^2$/kg) | Properties | | | | |
|---|---|---|---|---|---|---|
| | | Water for normal consistency (%) | Density ($kg/m^3$)/(%) | Compressive strength, (MPa)/(%) | Water adsorption, (%) | Water resistance |
| 1 | 2 | 3 | 4 | 5 | 6 | 7 |
| Admixture of calcined light loam 5% | | | | | | |
| 400 | 250 | 27.0 | 2265/-0.2 | 54.2/-5 | 1.30 | **0.925** |
| | 500 | 27.3 | **2275/+0.2** | **58.2/+2** | 1.20 | **0.935** |
| | 800 | 27.6 | **2283/+0.6** | **77.1/+35** | 1.10 | **0.960** |
| 600 | 250 | 27.2 | 2245/-1.1 | **61.5/+7** | 2.20 | **0.940** |
| | 500 | 27.6 | **2281/+0.5** | **67.1/+17** | 1.10 | **0.970** |
| | 800 | 27.9 | **2271/+0.0** | 56.5/-1 | 1.70 | **0.975** |
| 800 | 250 | 27.5 | 2245/-1.1 | 52.3/-9 | 1.94 | **0.930** |
| | 500 | 27.8 | 2235/-1.5 | 50.2/-12 | 2.00 | **0.930** |
| | 800 | 28.2 | **2303/+1.4** | **74.5/+30** | 1.24 | **0.965** |
| Admixture of calcined light loam 10% | | | | | | |
| 400 | 250 | 27.3 | 2241/-1.3 | 53.2/-7 | 1.60 | **0.925** |
| | 500 | 27.6 | **2255/+0.7** | 57.9/-1 | 1.20 | **0.930** |
| | 800 | 27.9 | **2292/+0.9** | **69.9/+22** | **0.90** | **0.945** |
| 600 | 250 | 27.4 | 2235/-1.5 | 55.5/-3 | 2.40 | **0.930** |
| | 500 | 27.8 | 2255/-0.7 | 58.5/-2 | 2.10 | **0.955** |
| | 800 | 28.1 | **2272/+0.0** | **63.2/+10** | 1.15 | **0.945** |
| 800 | 250 | 27.6 | 2229/-1.8 | 50.0/-13 | 2.40 | **0.945** |
| | 500 | 27.9 | 2212/-2.5 | 53.9/-6 | 2.20 | **0.930** |
| | 800 | 28.4 | 2245/-1.1 | **65.2/+14** | 2.00 | **0.955** |
| Admixture of calcined light loam 15% | | | | | | |
| 400 | 250 | 28.0 | 2110/-7.0 | 49.9/-13 | 1.40 | **0.925** |
| | 500 | 28.5 | 2231/-1.7 | 53.2/-7 | 1.15 | **0.930** |
| | 800 | 28.9 | 2255/-0.7 | **68.2/+19** | **0.90** | **0.940** |
| 600 | 250 | 28.2 | 2200/-3.0 | 52.4/-9 | 2.60 | **0.930** |
| | 500 | 28.5 | 2255/-0.7 | **58.5/+2** | 2.10 | **0.955** |
| | 800 | 28.8 | 2272/+0.0 | **65.2/+14** | 1.90 | **0.970** |
| 800 | 250 | 28.3 | 2209/-2.7 | 49.5/-14 | 2.25 | **0.935** |
| | 500 | 28.7 | 2230/-1.7 | **60.0/+5** | 1.70 | **0.940** |
| | 800 | 30.0 | 2265/-0.2 | **64.5/+13** | 1.50 | **0.950** |
| Admixture of calcined light loam 20% | | | | | | |
| 400 | 250 | 28.5 | 2200/-3.0 | 45.2/-21 | 1.90 | 0.920 |
| | 500 | 28.9 | 2211/-2.6 | 55.1/-4 | 1.70 | **0.925** |
| | 800 | 29.2 | 2232/-1.7 | **67.2/+17** | 1.20 | **0.935** |
| 600 | 250 | 28.8 | 2185/-3.7 | 50.0/-13 | 2.75 | **0.925** |
| | 500 | 29.0 | 2231/-1.7 | 51.0/-11 | 2.40 | **0.930** |
| | 800 | 29.4 | 2242/-1.2 | 52.5/-8 | 2.10 | **0.960** |
| 800 | 250 | 29.1 | 2200/-3.0 | 45.9/-20 | 2.30 | 0.920 |
| | 500 | 29.2 | 2260/-0.4 | **58.0/+1** | 1.90 | **0.930** |
| | 800 | 30.2 | **2275/+0.2** | **60.0/+5** | 1.60 | **0.935** |

The water resistance of the hardened Portland pastes was determined after steam curing as from ratio of the compressive strength of the samples after saturation with water at a temperature 20–22 °C to that of the samples before saturation.

## 3. Results and discussions

### *3.1. Effect of calcined light loam on the physical-mechanical properties of fresh and hardened Portland cement paste*

Table 1 shows the results of the measurements of the water for normal consistency, density, compressive strength, water adsorption, and water resistance of the Portland cement pastes with calcined light loam at different temperatures and ground to different $S_{sp}$ values. The mentioned properties for the reference sample were: density = 2270 $kg/m^3$, compressive strength = 57.3 MPa, water adsorption = 1.0% and water resistance = 0.92, respectively.

In columns 4 and 5 of Table 1, the numerators represent the density and compressive strength of the hardened Portland cement pastes with calcined light loam admixture and the denominators represent the change in their density (in per cent) with respect to the reference sample. The parameters that were found to be higher for the hardened Portland cement paste samples with calcined light loam than for the reference paste are indicated in boldface.

From an analysis of the data shown in Table 1, the following conclusions can be made.

The water for normal consistency of the fresh Portland cement pastes increased from 27 to 27.4–31.2% as the content of loam increased from 5 to 20% , calcination temperature increased from , 400 to 800 °C, and fineness of the calcined light loam, increased from 250 to 800 $m^2$/kg, respectively.

Of the 36 varieties of hardened Portland cement paste with different content of light loam calcined at different temperatures and grinding to different $S_{sp}$, 27.7% have higher density, 50% have higher compressive strength, up to 94.4% have higher water resistance and 11.1% have lower water absorption compared to reference sample.

The hardened Portland cement pastes with 5 and 10% of light loam calcined at 400 and 800°C and ground at 800 $m^2$/kg showed the largest increase of the density (up to 1.4 and 0.9%). The hardened Portland cement pastes with 10-15% of light loam calcined at 400 and ground at 800 $m^2$/kg showed the largest increase of the water adsorption. The hardened Portland cement pastes with 5-20% of light loam calcined at 600 and ground at 500 and 800 $m^2$/kg showed the largest increase of the water resistance.

The hardened Portland cement pastes with 5-10% of light loam calcined at 400-800°C and ground to 800 $m^2$/kg showed the highest increase of the compressive strength (up to 35%). The hardened Portland cement pastes with 15-20% of light loam calcined at certain temperatures and ground at 500-800 $m^2$/kg showed the highest increase of the compressive strength (up to 14-19%).

### *3.2. Comparison of the properties of Portland cement pastes with calcined light loam - and MK admixtures*

Table 2 highlights the effect of MK admixture on the properties of Portland cement paste. It was found that the Portland cement pastes with 5 and 10% MK showed improved compressive strengths of up to 29.3 and 9.9%, respectively. Incorporation of 5–20% MK increased the water adsorption of the Portland cement pastes. As the MK content increased from 15 to 20%, the compressive strengths and water resistance of the blended cement pastes decreased; however, water resistance was higher than that of the reference sample.

Figure 2 shows the results of the compressive strength measurements of the Portland cement pastes with light loam calcined at 400-800$^0$C and ground to 250-800 $m^2$/kg in

**Table 2**

Properties of the Portland cement pastes with MK content

| The content of MK (%) | Properties | | | |
|---|---|---|---|---|
| | Density ( kg)/(m)$^3$ | Compressive strength (MPa) | Water adsorption (%) | Water resistance |
| 0 | 2270 | 57,3 | 1,00 | 0,920 |
| 5 | 2298 | 74,0 | 1,70 | 0,925 |
| 10 | 2254 | 62,9 | 1,80 | 0,960 |
| 15 | 2222 | 52,3 | 1,95 | 0,930 |
| 20 | 2221 | 50,7 | 3,00 | 0,920 |

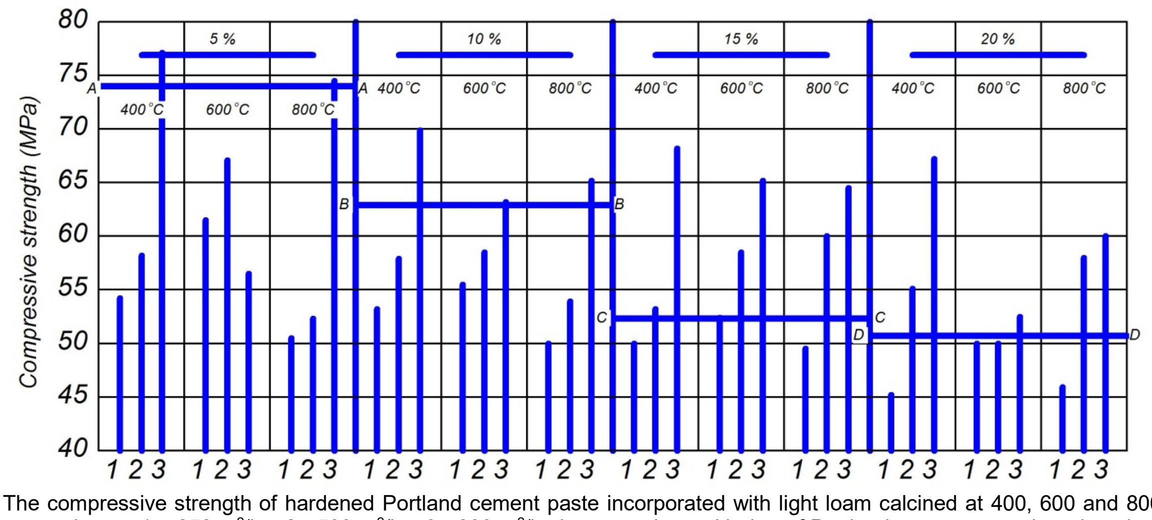


Fig.2 - The compressive strength of hardened Portland cement paste incorporated with light loam calcined at 400, 600 and 800$^0$C and ground up to 1 – 250 $m^2$/kg, 2 - 500 $m^2$/kg, 3 - 800 $m^2$/kg in comparison with that of Portland cement paste introduced with MK - 5 % (A-A), 10 % (B-B), 15% (C-C) and 20 % (D-D).

**Table 3**

The differences in the properties of the Portland cement pastes with MK and calcined light loam depending of admixtures content, calcination temperature and their fineness

| Calcination temperature (°C) | Specific surface area, ($m^2$/kg) | Properties | | |
|---|---|---|---|---|
| | | Density, (kg/$m^3$)/(%) | Water adsorption (%) | Water resistance |
| 1 | 2 | 3 | 4 | 5 |
| Admixture of calcined light loam 5% | | | | |
| 400 | 250 | -33/1.40 | -0.40 | +0.00 |
| | 500 | -23/1.00 | -0.60 | +0.10 |
| | 800 | -15/0.60 | -0.60 | +0.35 |
| 600 | 250 | -27/1.20 | +0.40 | +0.15 |
| | 500 | -17/0.70 | +0.50 | +0.45 |
| | 800 | -53/2.30 | +0.60 | +0.10 |
| 800 | 250 | -53/2.30 | +0.70 | +0.05 |
| | 500 | -63/2.70 | +0.30 | +0.05 |
| | 800 | -45/1.90 | -0.20 | +0.40 |
| Admixture of calcined light loam 10% | | | | |
| 400 | 250 | +7/0.30 | -0.20 | -0.35 |
| | 500 | +21/0.90 | -0.60 | -0.30 |
| | 800 | +38/1.70 | -0.90 | -0.15 |
| 600 | 250 | +1/0.04 | +0.60 | -0.30 |
| | 500 | +38/1.70 | +0.30 | -0.05 |
| | 800 | +21/0.90 | -0.70 | -0.15 |
| 800 | 250 | -22/0.90 | +0.60 | -0.15 |
| | 500 | -5/0.20 | +0.40 | -0.30 |
| | 800 | +11/0.50 | +0.20 | -0.10 |
| Admixture of calcined light loam 15% | | | | |
| 400 | 250 | -23/1.00 | -0.55 | -0.05 |
| | 500 | -3/0.15 | -0.85 | +0.00 |
| | 800 | +21/0.90 | -1.05 | +0.10 |
| 600 | 250 | -34/1.50 | +0.65 | +0.00 |
| | 500 | -12/0.50 | +0.11 | +0.25 |
| | 800 | +48/1.70 | -0.05 | +0.40 |
| 800 | 250 | -25/1.10 | +0.25 | +0.05 |
| | 500 | -34/1.50 | +0.15 | +0.10 |
| | 800 | +1/0.04 | -0.05 | +0.20 |
| Admixture of calcined light loam 20% | | | | |
| 400 | 250 | -21/0.90 | -1.10 | -0.15 |
| | 500 | -10/0.40 | -1.30 | -0.10 |
| | 800 | +11/0.50 | -1.80 | +0.08 |
| 600 | 250 | -36/1.60 | -0.40 | -0.10 |
| | 500 | -9/0.40 | -0.60 | -0.05 |
| | 800 | +11/0.50 | -0.90 | +0.25 |
| 800 | 250 | -21/0.90 | -0.40 | -0.15 |
| | 500 | -6/0.30 | -0.50 | -0.05 |
| | 800 | +14/0.60 | -0.70 | +0.00 |

comparison with samples containing MK in the quantities 5-20%. Table 3 compares the properties of the hardened Portland cement pastes with calcined light loam and MK admixtures at the content 5-20%.

In the column 3 of Table 3 the numerator and denominators on columns 3 and 4 show the differences in the properties in natural value and per cent, respectively. The positive values represent the increase of property of the hardened Portland cement paste with calcined light loam related to that of the paste with MK.

From the data shown in Table 3, the following conclusions can be made. Of the 36 hardened Portland cement paste samples obtained by mixing of Portland cement with different amounts of light loam calcined at different temperatures and ground to different $S_{sp}$ values, 36.1% showed a higher density, 44.4% higher compressive strength, 44.4% higher water resistance and 63.9% higher water adsorption than the Portland cement pastes with the same content of MK.

The Portland cement paste with 5% MK showed a higher compressive strength than those with 5% of calcined light loam. Only 5% of light loam calcined at 400 and 800$^0$C and ground to 800 $m^2$/kg slightly increased the compressive strength of hardened blended Portland cement paste than that with the same amount of MK.

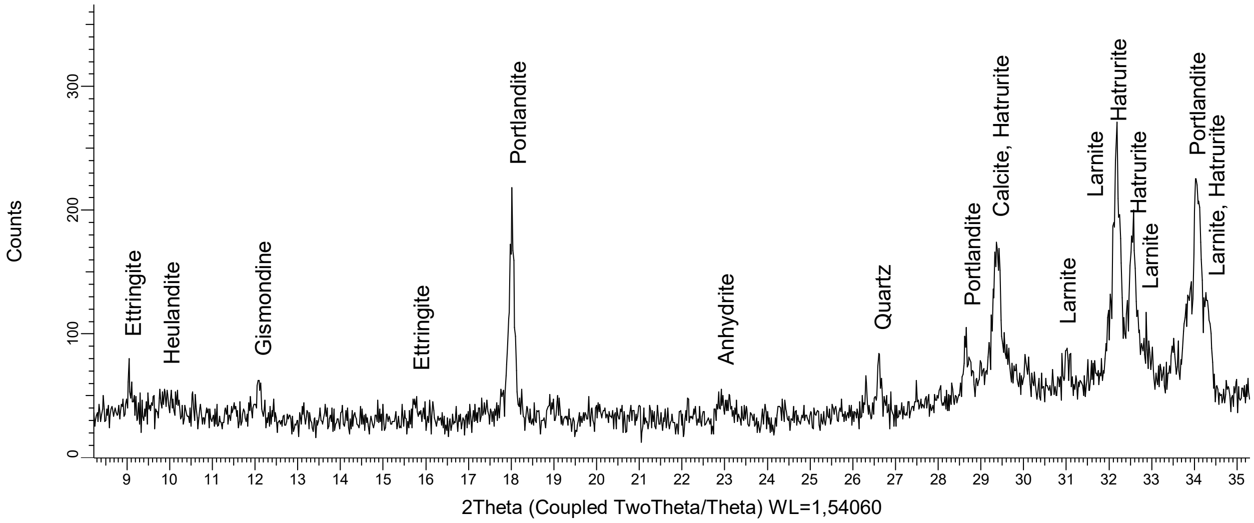


Fig.3- X-ray diffractogram of hardened Portland cement paste (reference sample)

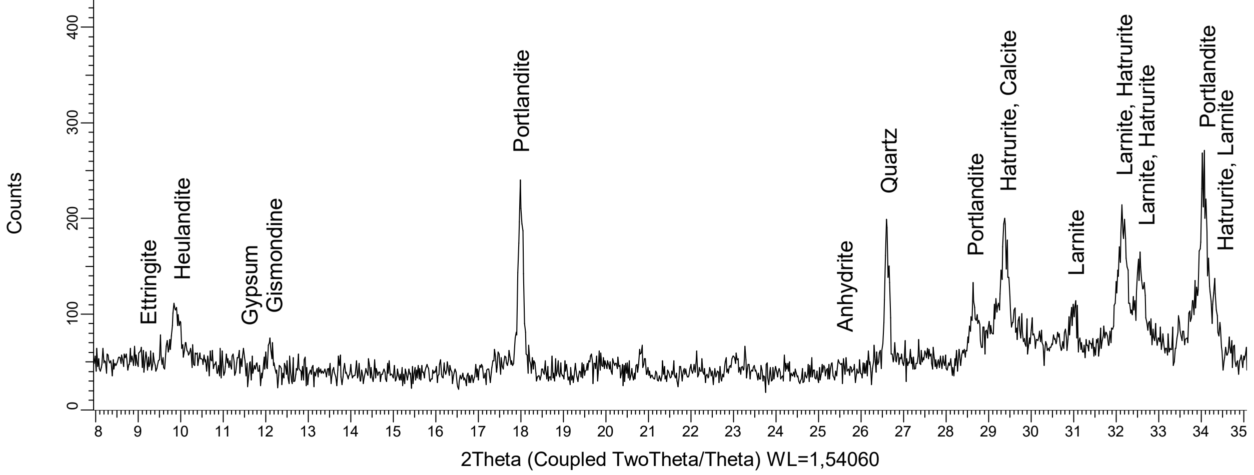


Fig.4 - X-ray diffractogram of hardened Portland cement paste with 15% of light loam calcined at $400^0$C

However, the increase of the calcined light loam content up to 10-20% is more effective than the same admixture of MK in terms of the compressive strength improvement of the hardened Portland cement paste. For example, the compressive strength of hardened Portland cement paste mixed with 15-20% of loam admixture calcined at $400^0$C and ground to 800 $m^2$/kg is higher up to 30-33% than that of the sample with the same content on of MK. Revealed improvement of compressive strength with calcined light loam admixture at Portland cement in comparison with those with MK is caused not only by pozzolanic activity of calcined light loam but by its filling effect [30] and physical activity also [31].

### *3.3. Influence of the calcined light loam on the mineral composition of hardened Portland cement paste*

As can be concluded from the Tables 1 and 2 the hardened Portland cement pastes incorporating l5% of light loam calcined at 400°C and ground to 800 $m^2$/kg showed better compressive strength and lower water absorption than that with 15% of MK. Figures. 3 and 4 show the X-ray diffractograms of the reference hardened Portland cement paste and hardened Portland cement paste containing mentioned calcined light loam, respectively.

A comparison of the X-ray patterns presented in Figures 3 and 4 shows that mineral composition of Portland cement paste and that with clacined light loam are slightly differ in intensity of peaks and presence of calcite. The difference in the intensity of the XRD peaks of these samples indicates the qualitative changes in the mineral content of these samples. This change explains to some extent the changes in the properties of the pastes with calcined light loam.

Along with the properties, the formation process of the blended Portland cement pastes was also influenced by the formation of calcium carboaluminate hydrate and probably by the formation of solid solutions of calcium carboaluminate hydrates and calcium hydroxoaluminate ($3CaO{\cdot}Al_2O_3{\cdot}Ca(OH)_2{\cdot}11H_2O$) [32].

## 4.Conclusion

The additions of 5–10% light loam calcined at temperatures ranging from 400–800°C and ground to 250–800 $m^2$/kg at Portland cement improved its compressive strengths up to 35%, increased density up to 1.4%, water resistance (from 0.92 to 0.93–0.98), and lowered water adsorption from 1 to 0.9%.

The hardened Portland cement paste with 5-20% light loam calcined at 400 and 800$^0$C and ground to 800 $m^2$/kg has highest compressive strength characteristics and water resistance.

The admixtures of 10-20% of calcined light loam increased the improvement of compressive strength up to 30-33% depending on specific surface area and calcination temperature.

Improvement of physical-mechanical properties could be probably explained by:

- formation of calcium carboaluminate hydrate and of solid solutions of this with calcium hydroxoaluminate;
- a filling effect.

Light loams are more available by more reserves than kaolin clays. The reasonability of production and application of calcined loam clay, which aren't lower in efficiency than high-priced metakaolin, was stated.

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