Sergejus GAIDUČIS

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SUMMARY OF DOCTORAL DISSERTATION

TECHNOLOGICAL SCIENCES,
MATERIALS ENGINEERING (08T)

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VILNIUS LEIDYKLA TECHNIKA 2009

VILNIUS GEDIMINAS TECHNICAL UNIVERSITY

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Introduction

Topicality of the problem. Reprocessing of phosphogypsum (PG) is becoming more and more relevant issue not only in Lithuania, but also in the rest of the World. Both, Lithuanian and foreign scientists are trying to solve PG recycling problem for a long time. However, PG is still utilised by pouring it to the water, unexploited mines or by transporting to the dumps.

Currently PG is not reprocessed in Lithuania, but transported to the dumps. Although a lot of work is done to tackle the PG reprocessing problem, there is a weakness of the past scientific investigations. During the application of the reprocessing methods developed so far (such as rinsing, neutralising of the impurities by lime milk), the chemical energy accumulated in PG is lost (approximately 160 kWh/t). In addition, the properties of the produced product depend very much on the amount of acidic impurities in waste material, i.e. the properties of the samples, produced from the neutralised PG, depend on the orthophosphorous acid processing parameters as well as effectiveness of the filtration of waste materials. Additionally, the applied neutralisation technologies of harmful acidic impurities are based on the complicated technological processes and larger power consumption, comparing to the one for the production of alabaster from the natural raw material.

Scientists seek to implement wider investigations and obtain summarised data on the relations between the amount of harmful impurities existing in the PG, technological parameters of the processing, products' hardening conditions and properties, as well as to define the tendencies of these relations for a long time. However, the relation between the properties of particular PG samples and amount of acidic impurities in PG, as well as parameters of the processes of technological processing, is still uncertain.

Subject of research

The subject of research of current work is phosphogypsum and influence on its properties mechanical activation and additives.

Aim and tasks of the work. Aim of the work is to propose new, effective additives neutralising acidic extractive hemihydrate impurities of phosphogypsum and effective mechanical activation technology for phosphogypsum processing, which would allow to reprocess this largest amount of mineral technogenic waste materials in Lithuania to the high quality composite gypsum binding material and its construction products. Additional aim is to analyse the influence of mechanical activation methods, including two-stage mechanical activation, which was not employed so far, on the physico-mechanical properties of industrially produced PG and its samples. The following tasks must be solved in order to achieve aims of the work:

1. Analysis of the properties of two types of extractive hemihydrate PG (produced from the apatites of Kovdor deposit and from the mixture of Morocco phosphorites and apatites of Kovdor deposit) freshly taken from waste disposal conveyor, uncooled and unhydrated, analysis of the variations of these properties due to the mechanical activation and influence of the neutralising additives and determination of the possibilities of their usage to produce composite gypsum binding material and high quality products.
2. Analysis, by using a mechanical activation disintegrator and a muller, of the influence of various parameters on the properties of mechanically activated extractive hemihydrate PG with the neutralising additives and determine the most suitable activation methods. Analysis of the influence of two-stage mechanical activation on the structure and properties of the structure of material produced by the disintegration method.
3. Analysis of the influence of the drying on the physico-mechanical properties of composite gypsum binding material produced from the extractive hemihydrate PG and determine the possibilities to use it for the production of dry mix.
4. Determination of the possibilities to use low energy consumption additives (carbonaceous opoka, granular slag glass (GSG), monoethanolamine) to neutralise the acidic impurities of the PG and analysis of its influence on the processes of hydration and hardening of composite gypsum binding material, as well as on the properties of the hardened samples.
5. Formation through the production process of the masonry products from the mechanically activated composition gypsum binding materials and analysis of the properties of the samples cut from these products.

Scientific novelty. During the preparation of the dissertation, the following results, that are new for the material engineering science, were obtained:

1. It was determined that the mechanical activation of extractive hemihydrate PG lowers the concentration of free CaO in liquid medium of the composition of the hardening PG, cement and pozzolanic additives. Therefore, the hardened material is resistant to the formation of minerals (ettringite, thaumasite) that could cause fragmentation.
2. The influence of various neutralising additives (Portland cement, opoka, GSG, monoethanoamine) on the properties of mechanically activated PG, freshly taken from waste disposal conveyor, was analysed. It was determined, that if the contemporary parameters were used during the

production of orthophosphorous acid, the amount of acidic impurities in PG has no influence on the properties of the composite gypsum binding material and its samples when these impurities are neutralised during the mechanical activation. It was found, that when additives of mineral origin are used to neutralise the acidic impurities, pH of binding material influences the strength and water resistance of the samples.

3. The two-stage mechanical activation method was developed (at the beginning – by using disintegrator at the intensity of 3000 rpm, and then by using a muller for 3 min.). This method allows us to improve the structure of the hardened composite binding material produced by implementing the disintegration method, to lower the porosity and water impregnation, to increase the density, strength and softening coefficient, as well as to shorten the formation time of the products.

Practical value. The main application area of the binding material produced during the research is the production of the masonry products. By applying the mechanical activation technology (suggested in the research) on the PG with additives, the binding material with stable properties is produced. The production process of this materials requires simple technology (it consists of just few technological operations) where fuel is not used, there is no need of large amount of energy, almost no waste materials are created. These factors allow to offer cheaper products for the market of masonry products, comparing to other similar purpose products existing in the market. In future, the products from PG could replace the ceramic, silicate and other low energy consumption products used for the partitions of the buildings.

Defended propositions

1. When the mechanical activation is applied, the amount of the deliquescent acidic impurities in the phosphogypsum has no influence on the strength properties of the formation masses and their samples.
2. The properties of the binding material of phosphogypsum with additives (produced by mechanical activation) and samples from this material are influenced by the type of the additive and composition's pH.
3. The application of two-stage mechanical activation allows us to decrease the products' formation period and improve the structure and properties of the hardened material.

Scope of scientific work. The scientific work consists of a general characteristic of dissertation, an introduction, 5 chapters, conclusions, a list of literature and a list of publications. The total scope of dissertation makes 152 pages, 13 numbered formulas, 36 pictures and 23 tables. 164 source of literature was used while preparing the dissertation.

Structure of the work. In preface, the analysed problem, research topicality is discussed, research object is described, research aim and the tasks are formulated, scientific novelty is described, practical value of the research results, defended propositions are specified.

First chapter is dedicated to the summary of the references. At the end of the section the summary and conclusions are formulated, and tasks of the dissertation are detailed. Second chapter deals with the description of the characteristics and properties of the materials used, research methodologies and equipment, preparation of the experimental samples. In third chapter the influence of the mechanical activation methods on the properties of PG and its compositions is analysed. In fourth chapter the influence of low energy consumption additives on the properties of mechanically activated extractive hemihydrate PG is analysed. Fifth chapter is dedicated to the experiments of the usage of PG in the production of masonry products.

1. Reprocessing problem of phosphogypsum

After the analysis of reference data it was concluded that, although a lot of work done to tackle the reprocessing problem of the PG, its global reprocessing amounts are very low and in Lithuania PG is simply stored in dumps. Such situation is related to the complicated reprocessing technologies used most often and high costs. Therefore, it is necessary to increase the investigations in the areas of neutralisation of acidic impurities, mechanical activation and search for the compositions, in order to improve the physical, mechanical and structural characteristics of the samples and products produced from these compositions, to extend the applications of the usage of these products for the construction, as well as to lower their production costs.

2. Materials and research methodology

The properties of two types of PG were analysed in the research: produced from the apatites of Kovdor deposit and produced from the mixture of Morocco phosphorites and apatites of Kovdor deposit. The following additives were used to neutralise acidic impurities of PG: Portland cement, GSG, carbonaceous opoka, monoethanolamine. Carbonaceous opoka and microsilica were used as pozzolanic additives.

Mechanical PG activation was implemented by using disintegrator DIA–01 (3000, 4500, 6000 rpm of the activating disks) and muller LB–2 (by activating for 30, 45, 60 min.). The pH control of mechanically activated compositions of PG with additives was carried out by using ion meter pH–330i. X-ray phase analysis method was used to carry out phase analysis of the hardening and hardened powder of the samples. X-ray analysis was done by diffractometer "Dron-2". The obtained data of the radiography analysis were processed by software "Origin 50".

3. Influence of the mechanical activation methods on the properties of phosphogypsum and its compositions

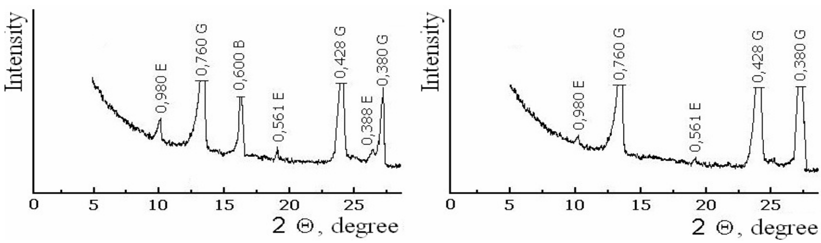
After the analysis of the possibilities to use PG for the formation of the products, it was determined that both types of PG, which have to be utilised until the binding process has started (and the amount of the hydrated water shall not exceed 7.4%), are suitable for the reprocessing. It was estimated that, in order to produce sufficiently strong (compressive strength up to 32 MPa) and water resistant (softening coefficient up to 0.92) compositions of mechanically activated PG, cement and pozzolanic additives, the amount of cement can be decreased up to 10%. This remaining amount of cement is sufficient to neutralise acidic additives as well (pH in all cases reaches 11.2–11.7).

It is expedient to neutralise the impurities of acidic PG with additives during the mechanical activation, i. e. PG and additives shall be supplied to the mechanical activation unit at the same time. In this way the cooling and the related hydration of PG is avoided. It was found when the disintegrator with the intensity of 3000 rpm is used for the activation of uncooled and unhydrated PG with the mix of cement and opoka, the amount of deliquescent acidic impurities (for the case when contemporary parameters for the production of orthophosphorous acid are used) has no influence on the strength of the samples (correlation coefficient $R = 0.1347$).

During the investigations the system of PG–cement–pozzolanic additives was analysed. PG, produced from the apatites of Kovdor deposit, was used in the analysis. Cement was used as neutralising additive, carbonaceous opoka and microsilica were used as pozzolanic additives. The concentrations of CaO in the compositions of PG, dried inactivated, dried activated (disintegrator at the intensity of 3000 rpm) and freshly taken from waste disposal conveyor, cement and pozzolanic additives in liquid phase, were compared with each other.

It was determined that mechanical activation of PG lowers CaO concentration in the liquid phase of compositions, where less initial ettringite is created (Fig. 1). Therefore, during exploitation these compositions are more resistant to the climatic conditions. CaO concentration and the amount of the initial ettringite created depends on the type of the pozzolanic additive. CaO concentration of the compositions with carbonaceous opoka in the liquid phase is larger that the one of the compositions with microsilica.

CaO concentration hardly depends on the type of the PG, therefore, the same proportions of pozzolanic additive and cement are used for the preparation of both compositions: PG produced from the apatites of Kovdor deposit and PG produced from the mixture of Morocco phosphorites and apatites of Kovdor deposit.



a b

Fig. 1. X-ray diffraction pattern of hardened samples:
a – composition with non-activated PG; b – composition with activated PG

During the analysis of the influence of mechanical activation on the properties of the PG with additives, the disintegrator (with various speeds of 3000, 4500, 6000 rpm of activating disks) and muller (by changing activation period for 30, 45, 60 min.) were used.

It was determined that the most suitable activation method by using disintegrator is 3000 rpm, and the activation process is occurs within several seconds. It has a big advantage in the technological process, because overall formation time of the products lasts only 8–10 min. approximately. However, in this case, due to the air intake during the disintegration, the porosity, water impregnation increase. In order to produce higher density, more water resistant and higher strength products with more homogenous structure, the activation with the muller should be applied. It was determined that the best period for the activation with the muller is ~45 min. This activation period ensures the best strength and water resistance properties of the compositions, but the formation process of the samples lasts up to 60 min. approximately. With the purpose to improve the structure and properties of the produced compositions by using disintegrator, the two-stage mechanical activation was tested. Initially, PG with additives was activated by using the disintegrator with the intensity of 3000 rpm (this ensures fast activation process), and then with the muller for 3 min. (in order to compact the material). In order to assess the expediency of the usage of two-stage mechanical activation, the sand concrete was selected. The achieved results are provided in Figures 2, 3.

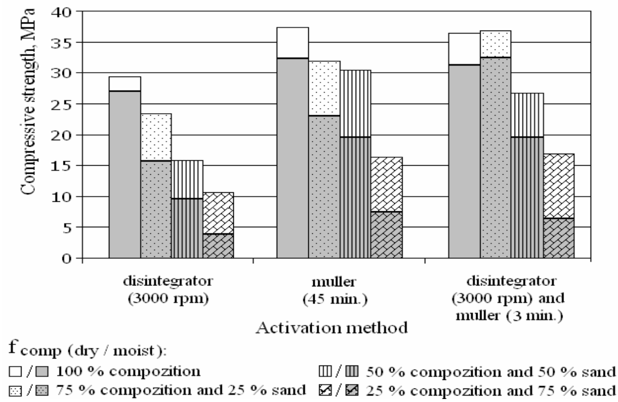


Fig. 2. Influence of activation method on the compressive strength of the samples

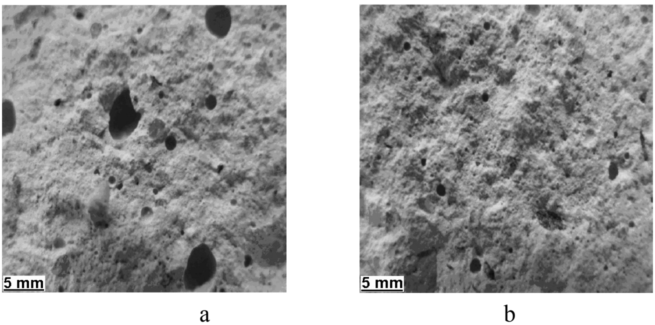


Fig. 3. View of sand concrete structure:
a – formed from the formation mass produced with the disintegrator;
b – formed from the formation mass produced by employing two-stage activation

The strength and water resistance properties of the composition, activated by employing two-stage activation method (with disintegrator and muller for 3 min.), and sand concrete samples, produced from this composition, are very similar (comparing the samples with the equal amount of sand) as the properties of the samples produced from the formation mass activated with the muller for 45 min. However, when the two-stage mechanical activation is used, the activation and samples' formation period decreases up to 10–15 min.

Surface pictures of the sand concrete samples (with the sand amount of 25%), formed from the composition of PG, activated by applying disintegrator (Fig. 3, a) and two-stage activation method (Fig. 3, b), cement and opoka are provided in Figure 3.

In Figure 3 provided pictures show that in the structure of sand concrete, produced from the formation mass activated with disintegrator (Fig. 3, a), quite large pores (up to the size of 5 mm) are noticed, whereas, no pores, larger than 1 mm (Fig. 3, b), are found in the structure of sand concrete produced from the composition formed by employing two-stage activation method.

It was determined that dried PG can be used for the production of dry mixes. The samples of the compositions with dried and mechanically inactivated PG are characterised by low strength (compressive strength up to 5 MPa), softening coefficient (up to 0.4) and high water impregnation (up to 16,4%). Whereas, after the activation of dried PG, compressive strength increases up to 18–22 MPa, softening coefficient – up to 0.6–0.7, and water impregnation decreases up to 10.9–11.1%.

4. Influence of other additives on the properties of phosphogypsum and its compositions

The possibilities to use low energy consumption additives – carbonaceous opoka, GSG, monoethanolamine were analysed. In addition, their influence on the properties of mechanically activated PG were determined.

During the analysis of the influence of opoka and GSG on the properties of mechanically activated PG the prism-shaped samples 4×4×16 cm were formed by vibration. Additives, in a form of dry powder, were added during the activation with the disintegrator (at the intensity of 3000 rpm). During the investigation of the influence of monoethanolamine, its various amounts were diluted by the water of 20°C temperature (ratio of water and hard particles is 0.4). Samples were hardened in laboratory conditions.

It was determined that opoka is sufficiently effective additive neutralising the acidic impurities of PG. Even low amount of opoka (3% or 5%) increases pH values to 5.43–5.99. With the compositions of 10% and 15% amount of opoka pH becomes almost neutral (6.19–6.80). GSG is more effective neutraliser of the acidic impurities than opoka, even with 3 % of compositions pH reaches 6.41–6.43. With larger amounts pH becomes alkaline (7.81–11.35). The increasing amount of opoka slightly decreases the ratio of water and hard particles of the compositions. Due to this the density increases and water impregnation decreases slightly. GSG influences the properties of hardened compositions in other way. It was determined, that when the amount of GSG increases, the density of the samples increases marginally, whereas water impregnation decreases considerably. The larger the amount of GSG additive, the lower the water impregnation. It can be explained by the fact, that GSG is active in respect of the gypsum and acts in the composition not as an inert filler, but as an active component, with gypsum forming new formations that are more water resistant. This is noticed by the increase of the softening coefficient of the hardened composition (Fig. 4).

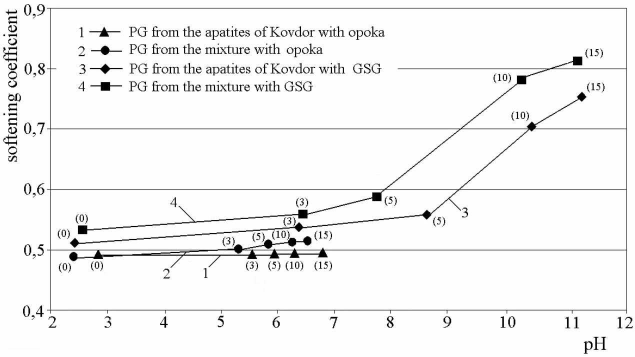


Fig. 4. Relation of softening coefficient of the hardened samples with composition's pH (in brackets the amount of additive is specified, % comparing to the amount of phosphogypsum)

Curves of Fig. 4 show that with the increase of pH the values of softening coefficients increase as well: when pH is up to 9, the softening coefficient does not exceed 0.6, whereas, when pH reaches 10 and more, the softening coefficient increases up to 0.71–0.82.

Considering the above-mentioned results (Section 1), obtained during the analysis of the properties of compositions of samples produced from the PG, cement and pozzolanic additives (pH reached up to 11.7, and the softening coefficient of hardened samples reached 0.92), it can be assumed that the water resistance of the samples depends on pH of the composition's medium.

Monoethanolamine is very effective additive, neutralising acidic impurities of PG, increasing the diffusiveness and decelerating the hydration and binding of the PG. However, when the amount of monoethanoamine is increased, the strength of compositions decreases. It is enough to use 0.1–0.5% of monoethanolamine to neutralise acidic additives of PG. The compressive strength of such samples reaches 12.1–13.5 MPa, but they are completely not resistant to the influence of water. Monoethanolamine can be used for the deceleration of the binding of compositions of PG, cement and pozzolanic additives. The best amount is 1.5–2.0%, for the case when the PG produced from the apatites of Kovdor deposit is used, and 3,0% when the PG produced from the mixture of Morocco phosphorites and apatites of Kovdor deposit is used. Such amounts of monoethanolamine decelerate the binding of compositions up to 2–2.5 hours and do not influence their strength negatively. The compressive strength of the compositions with monoethanolamine additive reaches up to 31.5 MPa, and softening coefficient – up to 0.92.

5. Industrial tests of the usage of phosphogypsum for the production of masonry products

The pilot technological production line for the masonry products from the compositions of PG with additives was installed and tested (Fig. 5).

Fig. 5. Experimental production line of stone wall blocks:
1 – disintegrator; 2 – mixer; 3 – belt-conveyor; 4 – press

The purpose of industrial tests was to form the blocks of large size from the activated PG and its compositions and prove the results, obtained in laboratory conditions, by applying various products' formation methods (moulding, vibration, pressing, vibropressing). Prism-shaped samples 4×4×16 cm were cut from the hardened products to determine the physical-mechanical properties.

The samples, cut from the non-activated PG blocks with no the additives, are characterised by low density (720–1220 kg/m^3), strength (3–6 MPa) and high water impregnation 27.4–33.1 %). No positive results were achieved even after the pressing of the non-activated PG, the strength of such samples hardly reaches approximately 6 MPa. The samples formed by moulding from the PG, activated by disintegrator and muller, with no additives and from the pressed inactivated composition of PG, cement and pozzolanic additives, are not distinguished by high strength and water resistance as well.

The formation methods (moulding, vibration, pressing, vibropressing) applied for the samples from the mechanically activated compositions of PG with cement and pozzolanic additives showed positive results. The physical and strength properties of the samples, for which the following compositions were used, depend on the formation method of the samples:

- properties of the compositions produced by applying moulding method: compressive strength 19.1–20.0 MPa; water impregnation 13.6–13.9%; softening coefficient 0.55–0.7;
- properties of the compositions produced by applying the vibration method: compressive strength 28.3–28.7 MPa; water impregnation 8.8–9.1%; softening coefficient 0.89–0.92;
- properties of the compositions produced by applying the pressing method: compressive strength 28.3–33.7 MPa; water impregnation 6.1–7.9%; softening coefficient 0.86–0.92;
- properties of the sand concrete (when binding material consists of compositions of PG, cement and pozzolanic additives) produced by applying vibration and vibropressing:
    - with 25% sand: compressive strength 25.9–31.7 MPa; water impregnation 9.1–11.4%; softening coefficient 0.77–0.82;
    - with 50% sand: compressive strength 20.3–27.0 MPa; water impregnation 9.4–11.9%; softening coefficient 0.52–0.61.

The obtained results do not differ from the properties of the samples formed in laboratory conditions, and this implies that mechanically activated compositions of PG, cement and pozzolanic additives could be recommended for the manufacturing of mural products. These compositions could allow to produce of wide scale of masonry products. Considering the physical and strength characteristics of the products, they can by used for the masonry works of both, supporting and partition constructions.

General conclusions

1. The determined mathematical relations show that, when the mechanical activation of the hemihydrate phosphogypsum is applied, the main factor determining the strength properties of the samples is the time period till the mechanical activation process, but not the degree of impurity by the deliquescent acidic impurities.

Both types of phosphogypsum are suitable for the reprocessing. In order to produce the binding material of stable properties from this phosphogypsum, the best way is to reprocess the phosphogypsum which is uncooled and in which the binding process has not started. The time period for the retention of the phosphogypsum till the mechanical activation process must last not longer than 60 min.

2. Mechanical activation and its method influences the properties of the compositions and the samples formed from these compositions. The following was determined:
   - mechanical activation lowers the concentration of free CaO in the hardening cement and pozzolanic additives compositions of extractive hemihydrate phosphogypsum in liquid phase, smaller amounts of initial ettringite (comparing to the compositions when the phosphogypsum, that is not mechanically activated, is used) are created in these compositions. Therefore, the compositions with activated phosphogypsum are more resistant to the climatic conditions during exploitation;
   - the most suitable activation period with the muller is about 45 min. The samples, formed from the formation masses activated by using this method, are characterised by high compressive strength (up to 37.3 MPa) and softening coefficient (up to 0.94). However, the formation period of these samples lasts up to 60 min.;
   - when the disintegrator is used for the activation – at the intensity of 3000 rpm –, the compressive strength of the samples reaches 29.6 MPa, and softening coefficient approximately 0.90. However, the activation with disintegrator is convenient technologically because the formation process lasts only 8–10 min.;
   - two-stage mechanical activation – initially with the disintegrator (at the intensity of 3000 rpm), and then with the muller for 3 min. – allows us to produce the samples with the analogous properties, as when muller is used for the activation for 45 min., and to decrease the formation period for the samples up to 10–15 min.
3. It was determined that the dried mechanically activated extractive hemihydrate phosphogypsum is suitable for the production of the dry mixes. The density of the samples, formed from this phosphogypsum with the cement and pozzolanic additives is 1120–1140 kg/m^3, compressive strength is 18–22 MPa, water impregnation is 10.9–11.1%, softening coefficient is 0.6–0.7.
4. After the analysis of the possibilities to use low energy consumption additives, such as carbonaceous opoka, granulated slag glass,

monoethanolamine, for the neutralisation of the acidic impurities of the phosphogypsum, the following recommended amounts of these additives and properties of the samples were determined:

- carbonaceous opoka 10–15%. pH of formation masses 6.19–6.80. Compressive strength of the samples 12.9–18.7 MPa, water impregnation 13.2–14.9%, softening coefficient 0.48–0.53;
- granulated slag glass 10–15%. pH of formation masses 10.2–11.4. Compressive strength of the samples 18.4–22.8 MPa, water impregnation 9.4–10.6%, softening coefficient 0.70–0.82;
- monoethanolamine 0,1%. pH of formation mass pH 8.6. Compressive strength of the samples 13.5 MPa, water impregnation 14.8%.

5. After the formation of mural blocks, by employing manufacturing process, and analysis of their properties, the data obtained during the laboratory tests were confirmed. Practically, for the masonry works of supporting constructions, protected from the direct climatic conditions, it is expedient to use the products, produced from the following mechanically activated compositions of phosphogypsum with additives:
   - phosphogypsum 80%, cement 10%, opoka 10%. pH of formation mass 11.3–11.7. Compressive strength of the samples 26–30 MPa, water impregnation 9.7–11.0%, softening coefficient 0.87–0.92;
   - phosphogypsum 85%, cement 10%, microsilica 5%. pH of formation masses pH 11.2–11.6. Compressive strength of the samples 26–32 MPa, water impregnation 9.0–9.9%, softening coefficient 0.88–0.94.
6. The pilot technological production line for the masonry products from the compositions of extractive hemihydrate phosphogypsum with additives was installed and tested. The industrial tests showed that the mechanical activation of the phosphogypsum with additives imply that this largest amount of technogenic waste material in Lithuania could be reprocessed to the masonry products, which could replace nowadays used silicate, ceramic and other high energy consumption masonry products in future.

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MECHANINĖS AKTYVACIJOS IR PRIEDŲ POVEIKIS EKSTRAKCINIO PUSHIDRAČIO FOSFOGIPSO IR JO GAMINIŲ SAVYBĖMS

Mokslo problemos aktualumas. Fosfogipso perdirbimas tampa vis aktualesnė problema ne tik Lietuvoje, bet ir visame pasaulyje. Tiek Lietuvos, tiek ir pasaulio mokslininkai jau seniai bando spręsti fosfogipso perdirbimo problemą, tačiau iki šiol jis dažniausiai būdavo utilizuojamas pilant į vandens telkinius, užpilant neeksploatuojamas šachtas arba transportuojant į sąvartynus.

Lietuvoje šiuo metu fosfogipsas neperdirbamas, o transportuojamas į sąvartynus. Sprendžiant fosfogipso perdirbimo problemą atlikta nemažai darbų, tačiau ankstesnių mokslinių tyrimų trūkumas yra tai, kad taikant iki šiol sukurtus perdirbimo būdus (plovimas, priemaišų neutralizavimas kalkių pienu) prarandama fosfogipse akumuliuota cheminė energija (apie 160 kWh/t), o gaunamo produkto savybės labai priklauso nuo rūgščiųjų priemaišų atliekose kiekio, t. y. bandinių, pagamintų iš neutralizuoto fosfogipso savybėms poveikį turi ortofosforo rūgšties gamybos parametrai bei atliekų filtracijos efektyvumas. Be to, taikomos kenksmingų rūgščiųjų priemaišų neutralizavimo technologijos, pagrįstos sudėtingais technologiniais procesais bei didesnėmis nei statybinį gipsą gaminant iš gamtinės žaliavos energijos sąnaudomis.

Mokslininkai jau seniai siekė atlikti platesnius tyrimus ir gauti apibendrintus duomenis apie ryšius tarp fosfogipse esančių žalingų priemaišų kiekio, perdirbimo technologinių parametrų, gaminių kietinimo sąlygų bei gaminių savybių, taip pat nustatyti šių ryšių tendencijas. Tačiau konkrečių gaunamų fosfogipso bandinių savybių ir jame esančių rūgščiųjų priemaišų kiekio bei perdirbimo technologijų procesų parametrų sąryšis lieka neaiškus.

Tyrimų objektas

Darbo tyrimų objektas yra ekstrakcinis pushidratis fosfogipsas ir mechaninės aktyvacijos bei priedų poveikis jo savybėms.

Darbo tikslas ir uždaviniai. Darbo tikslas yra pasiūlyti naujus, efektyvius rūgščiąją ekstrakcinio pushidračio fosfogipso terpę neutralizuojančius priedus ir efektyvią fosfogipso perdirbimo mechaninės aktyvacijos technologiją, leisiančią šias gausiausias Lietuvoje mineralines technogenines atliekas perdirbti į kokybišką kompozicinę gipso rišamąją medžiagą ir statybinės paskirties gaminius iš jos. Ištirti mechaninės aktyvacijos būdų, tame tarpe ir dvipakopės mechaninės aktyvacijos poveikį pramoniniu būdu gauto ekstrakcinio pushidračio fosfogipso ir bandinių iš jo fizinėms mechaninėms savybėms.

Darbo tikslui pasiekti darbe reikia spręsti šiuos uždavinius:

1. Ištirti dviejų atmainų – gauto iš Kovdoro vietovės apatitų bei Maroko fosforitų ir Kovdoro vietovės apatitų mišinio – ką tik nuo atliekų šalinimo konvejerio nuimtų, neatvėsusių ir nesusihidratavusių ekstrakcinių pushidračių fosfogipsų savybes ir jų pokyčius dėl mechaninės aktyvacijos ir neutralizuojančių priedų poveikio bei apibrėžti jų panaudojimo galimybes stabilių savybių kompozicinei gipso rišamajai medžiagai ir kokybiškiems gaminiams gauti.
2. Ištirti skirtingų mechaninės aktyvacijos dezintegratoriumi ir statgirnėmis parametrų poveikį mechaniškai aktyvuoto ekstrakcinio pushidračio fosfogipso su neutralizuojančiais priedais savybėms ir nustatyti tinkamiausius aktyvacijos režimus. Ištirti dvipakopės mechaninės aktyvacijos poveikį dezintegravimo būdu gaunamos medžiagos struktūrai ir savybėms.
3. Ištirti džiovinimo poveikį kompozicinės gipso rišamosios medžiagos iš ekstrakcinio pushidračio fosfogipso fizinėms mechaninėms savybėms ir nustatyti galimybes jį naudoti sausiems mišiniams gaminti.
4. Nustatyti galimybes naudoti energijai neimlius priedus (karbonatinę opoką, granuliuotą aukštakrosnių šlaką, monoetanolaminą) rūgščiajai fosfogipso terpei neutralizuoti ir ištirti jų poveikį gipso rišamosios medžiagos hidratacijos ir kietėjimo procesams ir sukietėjusių bandinių savybėms.

5. Gamybiniu būdu suformuoti iš mechaniškai aktyvuotų kompozicinių gipso rišamųjų medžiagų didelių matmenų sieninės paskirties gaminius ir ištirti iš jų išpjautų bandinių savybes.

Tyrimų metodika. Darbe buvo tirtos 2 atmainų fosfogipso: gauto iš Kovdoro vietovės apatitų ir gauto iš Maroko ir Kovdoro vietovės apatitų mišinio savybės. Kenksmingoms fosfogipso priemaišoms neutralizuoti buvo naudoti šie priedai: portlandcementis, granuliuotas aukštakrosnių šlakas, opoka, monoetanolaminas. Pucolaniniais priedais naudota opoka ir silicinės mikrodulkės (SiO_{2MD}).

Mechaninė aktyvacija ir rūgščiųjų priemaišų neutralizacija atlikta dezintegratoriumi DIA–01, kai aktyvuojančių diskų apsisukimų skaičius yra 3000, 4500, 6000 per minutę ir statgirnėmis LB–2 30, 45, 60 min. Neaktyvuoto fosfogipso ir mechaniškai aktyvuotų E-PG su priedais mišinių pH kontrolė buvo atlikta jonomačiu pH–330i. Rentgenofaziniu tyrimų metodu buvo atlikta kietėjančių ir sukietėjusių bandinių miltelių fazinė analizė. Pagrindinis naudotas prietaisas – difraktometras DRON–2. Gauti rentgenografinės analizės duomenys buvo apdoroti kompiuterine programa „Origin 50“.

Mokslinis naujumas. Rengiant disertaciją buvo gauti šie medžiagų inžinerijos mokslui nauji rezultatai:

1. Nustatyta, kad mechaninė pushidračio fosfogipso aktyvacija sumažina laisvojo CaO koncentraciją kietėjančių fosfogipso cemento pucolanų kompozicijų skystojoje fazėje, todėl sukietėjusi medžiaga yra atspari ardančiųjų mineralų (etringito, taumasito) susidarymui.
2. Ištirtas skirtingų neutralizuojančių priedų (portlandcemenčio, opokos, granuliuoto aukštakrosnių šlako, monoetanolamino) poveikis ką tik nuo atliekų šalinimo konvejerio nuimto mechaniškai aktyvuoto fosfogipso savybėms. Nustatyta, kad, esant šiuolaikiniams ortofosforo rūgšties gamybos parametrams, fosfogipso užterštumo tirpiosiomis rūgščiosiomis priemaišomis lygis neturi poveikio gaunamos kompozicinės gipso rišamosios medžiagos ir bandinių iš jos savybėms, jei jos neutralizuojamos mechaninės aktyvacijos metu. Nustatyta, kad rūgščiosioms priemaišoms neutralizuoti naudojant mineralinės kilmės priedus, rišamosios medžiagos pH turi poveikį bandinių stiprumo ir atsparumo vandeniui savybėms.
3. Sukurtas dvipakopės mechaninės aktyvacijos būdas (iš pradžių dezintegratoriumi 3000 aps./min intensyvumu, po to statgirnėmis 3 min), leidžiantis pagerinti sukietėjusios dezintegravimo būdu gautos kompozicinės rišamosios medžiagos struktūrą, sumažinti poringumą ir

vandens įgertį, padidinti tankį, stiprį ir suminkštėjimo koeficientą bei sutrumpinti gaminių formavimo laiką.

Praktinė vertė. Pagrindinė šiame darbe gautos rišamosios medžiagos pritaikymo sritis yra mūro gaminių gamyba. Taikant darbe siūlomą fosfogipso su priedais mechaninės aktyvacijos technologiją, gauta stabilių savybių rišamoji medžiaga, kurios gamybos procesas pasižymi nesudėtinga technologija (ją sudaro vos kelios technologinės operacijos), kurioje nenaudojamas kuras, nereikia daug energijos, beveik nesusidaro atliekų. Šie veiksniai sudaro galimybę mūro gaminių rinkai pasiūlyti pigesnius gaminius lyginant su kitais rinkoje esančiais analogiškais gaminiais. Ateityje gaminiai iš fosfogipso galėtų pakeisti pastatų atitvaroms naudojamus keraminius, silikatinius ir kitus energijai imlius dirbinius.

Ginamieji teiginiai

1. Taikant mechaninę aktyvaciją fosfogipse esančių tirpiųjų rūgščiųjų priemaišų kiekis neturi poveikio formavimo masių ir bandinių iš jų stiprumo savybėms.
2. Mechaniškai aktyvuojant gautos fosfogipso su priedais rišamosios medžiagos ir bandinių iš jos savybes lemia priedo rūšis ir kompozicijos pH.
3. Dvipakopė mechaninė aktyvacija leidžia sumažinti gaminių formavimo trukmę ir gerinti sukietėjusios medžiagos struktūrą ir savybes.

Darbo struktūra. Disertaciją sudaro įvadas, penki skyriai, bendrosios išvados, literatūros sąrašas ir autoriaus publikacijų disertacijos tema sąrašai. Darbo apimtis – 152 puslapiai, tekste panaudota 13 numeruotų formulių, 36 iliustracijos, 23 lentelės. Rašant disertaciją panaudoti 164 literatūros šaltiniai.

Įvadiniame skyriuje aptariama tiriamoji problema, darbo aktualumas, aprašomas tyrimų objektas, formuluojamas darbo tikslas ir uždaviniai, aprašomas darbo mokslinis naujumas, nurodoma darbo rezultatų praktinė reikšmė, įvardijami ginamieji teiginiai. Pirmasis skyrius skirtas literatūros apžvalgai. Skyriaus pabaigoje formuluojami apibendrinimas ir išvados, tikslinami disertacijos uždaviniai. Antrajame skyriuje aprašytos naudotų medžiagų charakteristikos ir savybės, tyrimų metodai ir aparatūra ir bandinių eksperimentams paruošimas. Trečiajame skyriuje aprašomas mechaninės aktyvacijos būdų poveikis fosfogipso ir jo kompozicijų savybėms. Ketvirtajame skyriuje tiriamas energijai neimlių priedų poveikis mechaniškai aktyvuoto ekstrakcinio pushidračio fosfogipso savybėms. Penktasis skyrius skirtas fosfogipso panaudojimo mūro gaminiams gaminti gamybiniams bandymams.

Bendrosios išvados

1. Nustatytos matematinės priklausomybės rodo, kad taikant pushidračio fosfogipso mechaninę aktyvaciją, pagrindinis veiksnys, lemiantis bandinių stiprumo savybes yra jo išlaikymo iki mechaninės aktyvacijos trukmė, o ne užterštumo tirpiosiomis rūgščiosiomis priemaišomis lygis. Perdirbti tinkamas abiejų atmainų fosfogipsas. Siekiant iš jo gauti stabilių savybių rišamąją medžiagą, geriausia perdirbti neatvėsusį ir nepradėjusį rištis fosfogipsą. Jo išlaikymo iki mechaninės aktyvacijos trukmė turi būti ne ilgesnė kaip 60 min.

2. Mechaninė aktyvacija ir jos būdas turi poveikį kompozicijų ir iš jų suformuotų bandinių savybėms. Nustatyta, kad:
   - mechaninė aktyvacija mažina laisvojo CaO koncentraciją kietėjančių ekstrakcinio pushidračio fosfogipso cemento pucolanų kompozicijų skystojoje fazėje, jose susidaro mažesni pirminio etringito kiekiai, palyginti su kompozicijomis, kai naudojamas mechaniškai neaktyvuotas fosfogipsas. Todėl eksploatuojant kompozicijos su aktyvuotu fosfogipsu yra atsparesnės aplinkos poveikiams;
   - tinkamiausia aktyvacijos statgirnėmis trukmė yra ~45 min. Bandiniai, suformuoti iš tokiu būdu aktyvuotų formavimo masių pasižymi dideliu gniuždomuoju stipriu (iki 37,3 MPa) ir suminkštėjimo koeficientu (iki 0,94), tačiau jų formavimo trukmė yra iki 60 min;
   - aktyvuojant dezintegratoriumi (3000 aps./min intensyvumu), bandinių gniuždomasis stipris siekia iki 29,6 MPa, o suminkštėjimo koeficientas apie 0,90. Tačiau aktyvacija dezintegratoriumi patogi technologiškai, nes visas bandinių formavimo procesas užtrunka tik 8–10 min;
   - dvipakopė mechaninė aktyvacija – iš pradžių dezintegratoriumi (3000 aps./min intensyvumu), po to statgirnėmis 3 min – leidžia gauti analogiškų savybių bandinius, kaip ir aktyvuojant statgirnėmis 45 min ir sumažinti bandinių formavimo trukmę iki 10–15 min.

3. Nustatyta, kad džiovintas mechaniškai aktyvuotas ekstrakcinis pushidratis fosfogipsas tinka sausiems mišiniams gaminti. Bandinių, suformuotų iš tokio fosfogipso su cementu ir pucolanais tankis yra 1120–1140 kg/m^3, gniuždomasis stipris 18–22 MPa, vandens įgertis 10,9–11,1%, suminkštėjimo koeficientas 0,6–0,7.

4. Ištyrus galimybes rūgščiosioms fosfogipso priemaišoms neutralizuoti naudoti energijai neimlius priedus – karbonatinę opoką, granuliuotą

aukštakrosnių šlaką, monoetanolaminą, nustatyti tokie rekomenduojami jų kiekiai ir bandinių savybės:

- karbonatinė opoka – 10–15 %. Formavimo masių pH 6,19–6,80. Bandinių gniuždomasis stipris 12,9–18,7 MPa, vandens įgertis 13,2–14,9 %, suminkštėjimo koeficientas 0,48–0,53;
- granuliuotas aukštakrosnių šlakas – 10–15 %. Formavimo masių pH 10,2–11,4. Bandinių gniuždomasis stipris 18,4–22,8 MPa, vandens įgertis 9,4–10,6 %, suminkštėjimo koeficientas 0,70–0,82;
- monoetanolaminas – 0,1 %. Formavimo masės pH 8,6. Bandinių gniuždomasis stipris 13,5 MPa, vandens įgertis 14,8 %.

5. Suformavus gamybiniu būdu sieninius blokelius ir ištyrus jų savybes, buvo patvirtinti laboratorinių tyrimų metu gauti duomenys. Praktiniam naudojimui laikančiosioms konstrukcijoms, apsaugotoms nuo tiesioginių atmosferos poveikių, mūryti tikslinga naudoti gaminius iš šių mechaniškai aktyvuotų fosfogipso su priedais kompozicijų:
   - 80 % fosfogipso, 10 % cemento, 10 % opokos. Formavimo masių pH 11,3–11,7. Bandinių gniuždomasis stipris 26–30 MPa, vandens įgertis 9,7–11,0 %, suminkštėjimo koeficientas 0,87–0,92;
   - 85 % fosfogipso, 10 % cemento, 5 % SiO_{2MD}. Formavimo masių pH 11,2–11,6. Bandinių gniuždomasis stipris 26–32 MPa, vandens įgertis 9,0–9,9 %, suminkštėjimo koeficientas 0,88–0,94.

6. Įdiegta ir išbandyta mūro gaminių iš ekstrakcinio pushidračio fosfogipso su priedais kompozicijų bandomoji technologinė linija. Gamybiniai bandymai parodė, kad mechaninė fosfogipso su priedais aktyvacija sudaro prielaidas perdirbti šias gausiausias Lietuvoje technogenines atliekas į mūro gaminius, kurie ateityje galėtų tapti pakaitalu šiandien naudojamiems silikatiniams, keraminiams ir kitiems energijai imliems mūro gaminiams.

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Sergejus GAIDUČIS

MECHANICAL ACTIVATION AND ADDITIVES INFLUENCE ON EXTRACTIVE HEMIHYDRATE PHOSPHOGYPSUM AND ITS PRODUCTS CHARACTERISTICS

Summary of Doctoral Dissertation
Technological Sciences, Materials Engineering (08T)

Sergejus GAIDUČIS
MECHANINĖS AKTYVACIJOS IR PRIEDŲ POVEIKIS EKSTRAKCINIO PUSHIDRAČIO FOSFOGIPSO IR JO GAMINIŲ SAVYBĖMS

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