Development of Alkali-Activated Luminescent Composite

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Abstract

Alkali-activated materials are the alternative binders in which latent hydraulic materials or pozzolans are used as the main binder component. Luminescence is a process in which atoms or molecules are excited and emit light. This phenomenon is used in the construction industry, mainly in safety, lighting, and design. These materials are often used as paint or spray as a safety element to mark escape routes, staircases, etc. They are also used for decorative purposes, for example, in luminescent concrete, where the luminescent component is often contained as aggregate. The experiment was focused on testing the possibility of preparing a luminescent material based on alkali-activated granulated blast furnace slag, where the luminescent pigment is applied as part of the binder component. The experiment compared the luminescent properties of the created mixture with cement-based materials and subsequently verified the rheological and physical-mechanical properties of the prepared composites. It was found that a luminescent alkali-activated composite can be prepared, but the influence of the luminescent pigment on the final properties, especially the flexural and compressive strength, where a significant decrease in the monitored parameters was recorded, must be taken into account.

Keywords: *Luminescence, Alkali-activated, Blast Furnace Slag, Composite*

1. Introduction

Luminescence is a physical phenomenon emitted by substances called luminophores. It is created by the excitation of an atom by the action of other radiation, electrons, etc., and the subsequent return of the atom to its basic state, resulting in the emission of a photon. A substance's luminescence can, therefore, be observed after it is irradiated by another radiation source. According to the afterglow duration, it is divided into fluorescence, which disappears almost immediately, and phosphorescence, which can last up to several hours. [1, 2]

Currently, it is used in medicine and the construction industry, mainly to produce safety elements and decorative items.

Using color pigments in construction is common, but luminophores are rarely used. This is due to the relatively high price of these materials. However, the potential for their use is high. Therefore, the aim of this experiment was to verify the possibility of preparing substances with a luminescent component, verify the basic properties of the prepared composite, and find out how much the use of pigment affects the final properties.

The pigment was used as an admixture in mortars based on cement, white cement, and alkaline-activated blast furnace slag in amounts of 5, 10, and 15% by weight of the binder. The pigment replaces a proportional part of the finest fraction of sand so that the amount of binder component remains the same in all mixtures for a possible comparison of strengths. The goal was to verify the influence of the luminescent pigment in predetermined quantities on the length of the luminescence and the chosen mechanical properties. The recipes were subjected to the extinction time and luminescence intensity test. The recipes with the best results were used to produce standard specimens; specimens with dimensions of 40 x 40 x 160 mm were prepared to test the mechanical properties.

2. Luminiscence and alkali-activated materials

Luminescence is simply the process where atoms or molecules are in an excited state and emit light. An excited state is a physical phenomenon where electrons in the electron shell of an atom are transferred to higher energy levels than the usual state. Substances in which luminescence occurs are called luminophores. Luminophores can be gases, liquids, or solids, except metals. Luminescence most often results in the emission of ultraviolet but also infrared or visible photons. [1, 2]

The term luminescence comes from Latin (lumen = light) and was introduced by physicist and science historian Eilhard Wiedemann in 1888. It is a substance that absorbs radiation of a suitable wavelength, which gradually reaches an excited state, where it subsequently emits the received energy in the form of electromagnetic radiation after a specific time. However, it must be true that the lifetime of the excited state must be longer than the period of light oscillations $\tau = 10 - 15$ s. [3, 4]

Luminescent radiation is characterized mainly by the intensity of the luminous effect, various color shades, extinction duration, and uniform radiation scattering. [2]

Fluorescence and phosphorescence distinguish according to the luminescent effect's duration. The difference lies in the mechanism by which the atom returns to its ground state. At the beginning of the process, electrons appear in the ground state, i.e., they have the lowest energy level S1. In fluorescence, a permitted transition occurs in the substance from a higher energy level, S1, to a lower energy state with the same multiplicity, S0. The duration of the effect is in the order of 10^(-9) s to 10^(-6) s. The luminescent effect disappears almost immediately after removing the substance's radiation source. Fluorescence occurs when a UV light source illuminates a white fabric

or when checking for counterfeit banknotes, where the so-called fluorescent dyes are used. They are also used to investigate the flow of groundwater. The name of this phenomenon is derived from the mineral fluorite, in which this phenomenon was observed. In phosphorescence, light radiation is caused by a transition between levels with different multiplicity, moving from a higher to a lower energy level and emitting a photon (T1 → S0). The phosphorescence time is much longer than in fluorescence at 10^(-2) s. The luminescence phenomenon continues even after the radiation source is removed. It can be observed for several hours. [1 - 3]

Luminescence is divided according to how the excitation or excitation energy is supplied. It can be divided into several subgroups:

Photoluminescence - excitation energy is supplied using electromagnetic radiation of a suitable wavelength, such as UV or visible radiation. From the point of view of use, it is the most important, as it is used in analytical chemistry and biochemistry. [5, 6]

Chemiluminescence - this type of luminescence occurs only in certain kinds of exothermic reactions, where after a chemical reaction, the release of reaction heat occurs, part of which is radiated in the form of light. [1, 5]

Electroluminescence - arises from an effect where energy is supplied by applying an electric field and passing an electric current through a substance. Adequate substances are semiconductors with a wide band gap, which is located between the conduction and valence bands. [2, 3]

Bioluminescence - bioluminescence involves specific biochemical reactions resulting in living organisms' luminescence. It is made possible by luciferin and the enzyme luciferase. In the presence of oxygen, luciferin oxidizes to oxyluciferin, which causes the molecule to transition to an excited state and emit radiation. The enzyme luciferase catalyzes the reaction. [1, 7]

Thermoluminescence - this process causes thermal energy delivery by electromagnetic radiation, which is preceded by the cooling of the substance. This results in a luminescent effect. [2, 6]

Luminophores are substances that can absorb energy and subsequently emit it in the form of light. The composition of the luminophor determines the color of the radiation. They occur in solid, liquid, and gaseous states. They usually glow at the same or greater wavelength than the radiation produced. Gaseous luminophores include monoatomic and polyatomic gases such as Na, He, Ne, Bi, and CO_2. Luminescent compounds are divided into organic and inorganic. The carrier of radiation is a molecule in organic compounds such as aromatic hydrocarbons (naphthalene, anthracene, pyrene, etc.). In inorganic compounds such as uranyl ions or lanthanide ions, luminescent radiation occurs due to the grouping of atoms. In both cases, this process occurs in liquids as well as solids. [1 - 4]

Alkali-activated systems are perspective materials that, thanks to processed raw materials and valuable properties, occupy leading positions in possible substitutes for materials based on Portland cement. These are materials prepared by alkali activation of latently hydraulic substances, or pozzolans. The indisputable advantage of these materials is the applicability of secondary raw materials, for which there is often no optimal use, particularly the possibility of processing fly ash, various types of slag, etc. Another significant advantage, compared to cement-based binders, is a low carbon footprint. However, it is necessary to mention the other side of the matter. As already published in [8], these are binders whose diversity of used raw materials, preparation, and storage conditions leads to an even wider range of final properties, resulting from the number of resulting hydration products and their mutual combinations, which may occur depending on the parameters mentioned above. This variability is problematic due to the need for strict adherence to technological discipline in realizing materials, as its non-observance leads to fluctuations in monitored parameters. At the same time, repeatability and reproducibility are necessary conditions for use in the construction industry.

3. Materials and methods

In the experiment, Portland cement CEM I 52.5 R (specific surface according to Blain 350m^2/kg), white cement CEM I 52.5 R white (specific surface according to Blain 400m^2/kg, rapid increase in strength, low Fe_2O_3 content of about 0.15% and MnO about 0.015%), as well as blast furnace finely ground granulated slag (specific surface according to Blain 400m^2/kg, SiO_2 34%, Al_2O_3 12%, FeO 1%, MnO 1.4%, CaO 42%, MgO 9%, P_2O_5 0.05%, CaS 1.3%), sodium water glass (silicate module 2.0, dry matter content 45.5%, bulk density 1520kg/m^3), quartz sand (fraction 0.2 mm), powder luminophore PLE-P-Z-4B green color (granularity 65-85µm and luminosity 640 mcd/m^2 in the first 10 minutes and then luminosity 105 mcd/m^2 for at least 60 minutes), and mixing water.

Luminosity of the pigment in the mixture, depending on its quantity and the binder used, and also how long the luminescence effect would last. For this stage, material samples were prepared in small plastic molds with a diameter of d=55 mm and a height of h=10 mm. For each type of binder, three mixtures were created, while each mixture contained a different amount of pigment, namely 5, 10, and 15% of the weight of the binder. Two mixtures with cement (Portland and white) were prepared, containing cement, filler, pigment, and water, and one alkali-activated mixture containing blast furnace slag, water glass, filler, pigment, and water. The amount of individual components used during mixing depends on the reference mixture consisting of 450g of binder, 1350g of filler, 126g of water glass, and 225g or 110g (for alkali-activated mixture) of water; however, they were converted to the required volume of the mold + a reserve of 5%.

Based on the results of this phase, two mixtures (alkali-activated slag mixture and white cement mixture, both with 15% of pigment replacement) were selected for further experimentation, and two reference mixtures (alkali-activated slag mixture and white cement mixture) that did not contain any pigment were tested for comparison.

An automatic Beton System MI-CM5A laboratory mixer was used to prepare the mixtures according to the EN 196-1 standard. [9] The individual components were weighed using a digital scale with an accuracy of ± 1g. The properties of the fresh mixture and the mechanical properties after the prescribed curing period (7 and 28 days) were tested. During the curing, the test specimens were stored in an environment with a relative humidity of 98% and a temperature of 31°C.

4. Results and discussion

The first test samples were demolded after hardening and exposed to daylight with a value of 5410 lux for 60 minutes. The intensity of the light was measured with a luxmeter LX-103. After 1 hour, the duration of the luminescence effect of individual bodies in a dark room was visually estimated using a stopwatch. The luminescence of the samples cannot be determined due to the low intensity of the radiation that the device could not capture.

The results show that bodies based on alkali-activated blast furnace slag and white cement with the highest amount of pigment (15%) were suitable for further testing. The intensity and duration of the luminescence effect in these mixtures are significant compared to Portland cement, as seen in Figure 1 and 2. It can, therefore, be stated that the intensity of the luminescence radiation and the duration of luminescence are influenced by the color of the binder.

The difference is quite evident in the pictures, depending on the amount of pigment and the binder system used. The best results were achieved by mixtures with 15% pigment and, at the same time, mixtures where alkali-activated slag and white cement were used as binders.

The photos were not edited; they were taken under the same lighting conditions and with the same device. It is clear that the mixture with white cement emits radiation in a different shade than the original shade of the pigment, from which it follows that the pigment can probably react with the alkaline environment of the mixture, similar to what was found in [10].

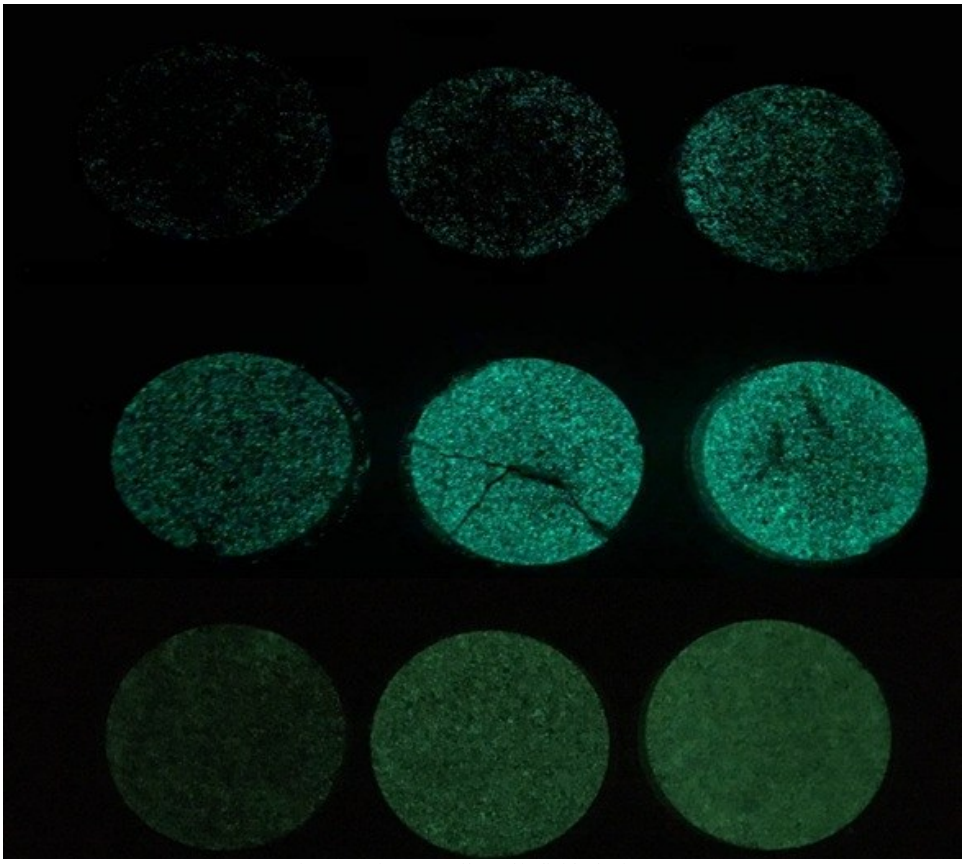

Fig. 1 Luminescent effect of individual mixtures.
Above (from left to right): Mixtures with Portland cement pigment in amounts of 5, 10, and 15%
In the middle (from left to right): Mixtures with blast furnace slag, pigment in amounts of 5, 10, and 15%
bottom: (from left to right): Mixtures with white cement, pigment in amounts of 5, 10, and 15%

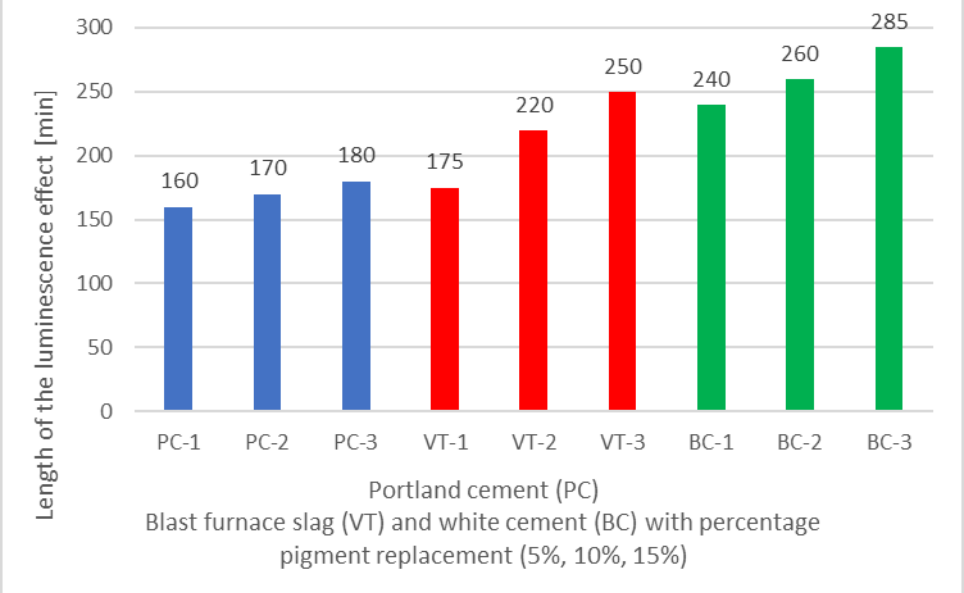

Fig. 2 Results of measuring the length of the luminescence effect

The consistency of fresh mortar was determined in accordance with the EN 1015-3 standard immediately after mixing the mixture using the Beton System RS-15 shaking table. [11]

The test for determining the air content in fresh mortar was carried out in accordance with EN 1015-7 standard. The air content was determined immediately after mixing the fresh mixture using the pressure method. [12]

The procedure for calculating the bulk density of the fresh mixture is according to the EN 1015-6 standard. It is determined as weight and volume, and the mixture must be stored and thoroughly compacted in a measuring container of known volume. [13]

The procedure for calculating the bulk density of hardened mortar is according to the EN 1015-10 standard. The measurements always take place before determining the mechanical properties of the bodies. [14]

The flexural strength was determined in accordance with the EN 1015-11 standard. It is determined by applying a uniform three-point load until the body breaks. The Formtest Mega-100 press was used as the breaking device. The test bodies consist of hardened mortar in the shape of a prism with dimensions of 40 x 40 x 160 mm. The beams were tested after 7 and 28 days from manufacture. [15]

The compressive strength is determined according to the EN 1015-11 standard. The test is carried out on two parts of the prism, which was used in the flexural strength test. The Formtest Mega-100 press was used to determine the strengths. The test was carried out after the flexural strength of the bodies was determined after 7 and 28 days of curing. [15]

Tab. 1. Properties of tested mixtures.

Parameter	White cement ref	White cem 15%	Alkali-activated Slag ref	Alkali-activated Slag 15%
Consistency [mm]	195	220	210	250
Air content [%]	3.1	4.3	4.1	4.6
Bulk density fresh mixture [kg/m^3]	2190	2200	2200	2220
Bulk density 7 days [kg/m^3]	2280	2270	2280	2310
Bulk density 28 days [kg/m^3]	2240	2240	2310	2320
Flexural strength 7 days [MPa]	6.5	8.1	4.8	0.6
Flexural strength 28 days [MPa]	8.2	8.5	7.2	0.8
Compressive strength 7 days [MPa]	29.9	48.4	43.2	3.2
Compressive strength 28 days [MPa]	61.3	62.7	81.9	4.3

It is clear from the results that the flow of the mortar increases with the content of the luminescent pigment, regardless of the binder component. This phenomenon can cause the removal of part of the finest filler fraction. Furthermore, it can be seen that the increase in flow is more pronounced in the blast furnace slag-based mixture than in the cement-based one, although the specific binder surface of both mixtures is the same.

In mixtures where luminescent powder was used, the mixture showed an increase in air content compared to the reference mixture, which is interesting, as the consistency was more liquid.

The luminophore does not significantly affect the change in the bulk density of the fresh mixture.

The results show that the bulk density of the hardened mixture increased for each mixture compared to the fresh mixture. The standard slag-based test specimens exceed the bulk density of the white cement specimen for each mixture.

The strength results proved to be problematic. Both compressive and flexural strengths were significantly reduced in mixtures with alkali-activated slag and pigment to only one-tenth of the strength of the reference mixture. The authors also encountered this problem in [10], who, while researching the possible application of luminescence in pavement materials, also noted a significant reduction in strength. This may be due to the interaction of the luminophore with an alkaline environment, although according to the manufacturer, the pigment should be stable even in a highly alkaline environment.

No such tendencies were recorded for bodies based on white cement; on the contrary, it seems that the pigment positively affected strength, and these mixtures with an admixture of pigment achieved slightly higher values.

5. Conclusion

The experiment tested whether a material with luminescent properties can be produced using a luminescent pigment. It was found that the preparation is possible, but the resulting luminescence effect depends on the binder (color) used and the amount of pigment. For economic reasons, the optimal amount of pigment was determined as 15% of the binder weight.

The results show that the luminescent pigment affects the properties of the prepared mixtures. When applied with white cement, it slightly changes its color, and when mixed with alkali-activated slag, it reduces the flexural and compressive strengths by up to 90% of the reference values. Therefore, it is necessary to study this issue in more detail in the intended use case. In further experiments, detailed analyses of the tested mixtures will be carried out to assess the suitability of the pigment for the given application or to focus on the use of other types of luminophores.

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