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REUSE OF RESIDUAL DIATOMACEOUS EARTH FOR THE PRODUCTION OF GEOPOLYMERS – A REVIEW

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ABSTRACT

The high pollution levels produced by the manufacturing of conventional Portland cement have motivated several studies in an attempt to modernize this process with alternative binders such as cements based on use of alkaline activation of aluminosilicates. The reaction between aluminosilicates (such as metakaolin, fly ash and blast furnace slag) and an alkaline solution (sodium hydroxide or potassium hydroxide) triggers a geopolymerization process, developing a material with good mechanical and thermal properties, with a vast spectrum of applications in construction, helping in the reduction of the environmental impact and due to it, lower carbon footprint. Most geopolymer concrete was focused on fly ashes, metakaolin and blast furnace slag, this paper focuses on use of residual diatomaceous earth in the manufacture of geopolymer concrete, which are very rich in aluminosilicates.

This review paper presents an extensive bibliographic review of studies related to the manufacture of geopolymer cements based on diatomaceous earth. The chemical composition of diatomaceous earth, density, surface area, absorption capacity, different drying methods and different calcination processes are investigated factors. Mortar blends based on diatomaceous earth, temperatures and curing times, physical and mechanical properties of the obtained samples are also subject to analysis.

KEYWORDS: “Geopolymerization”; “Residue valorisation”; “Residual diatomaceous earth”.

1. INTRODUCTION

This research arises within the scope of the project “BacchusTech – Integrated Approach for the Valorisation of Winemaking Residues (POCI-01-0247-FEDER-069583)”, financed by “Fundo Europeu de Desenvolvimento Regional (FEDER)”. The consortium led by Caves Campelo S.A. brings together the Polytechnic Institute of Bragança, Polytechnic Institute of Viseu, Hanze University of Applied Sciences and University College Copenhagen. In addition, companies in the cosmetics (Cosmetek Lda.), construction materials (Pavimir Lda.) and food (BEPPO Gelados Lda.) sectors also joined the consortium, companies that showed interest in the new materials to be developed.

Caves Campelo S.A. uses an average of 4,000 kg/year of commercial diatomaceous earth, currently using Silite Mini Speed diatomaceous earth, a white-coloured material, very fine and with a low flow velocity. After using these earths in wine filtration, it is discarded and sent to waste management facilities. This project aims to develop a new innovative process that allows the extraction, purification and concentration of bioactive compounds present in winemaking residues, in this case, the residual diatomaceous earth resulting from the filtration of white and red wines. Bioactive substances recovered from residual diatomaceous earth will be exploited as functional ingredients for food and cosmetic industries. After this process, the extracted diatomaceous earth will be later transformed into geopolymers, to be used as an alternative to the conventional Portland cement and as an innovative membrane filtration, to be used again in treatments for winery liquid effluents.

Therefore, meeting society's growing environmental requirements, the consortium seeks with this project to convert current environmental issues into business opportunities, valuing diatomaceous earth in environmental applications and building materials, ultimately evaluating the company's environmental sustainability, resulting from implementation of these technologies through a life cycle analysis approach [1].

2. GEOPOLYMERIZATION

2.1. Traditional Aluminosilicates

The high pollution levels produced by the manufacture of Portland cement, associated with a carbon dioxide emission of around 7% of the world total emissions, and the entry of the Kyoto protocols in 1997, resulted in a series of attempts to modernize the manufacture of cement with alternative binders, where cements based on the alkaline activation of aluminosilicates have become a target of research all around the world [2].

The ancient mortars contained aluminosilicates of volcanic origin. Many of these structures are still standing today and are proof of the high durability of these lime-based cements and mortars with materials with pozzolanic properties. The investigation of the chemical and physical properties of these ancient structures is still ongoing, in particular for restoration purposes [3].

The main examples of aluminosilicates used nowadays in construction are metakaolin, slag obtained in blast furnaces, and fly ashes obtained from burning coal in thermoelectric power stations [4].

Metakaolin is a material of pozzolanic origin obtained from kaolinitic clays. The calcination temperature for treatment of metakaolin is around 700-800 °C and it is usually composed by 50-55% of SiO_2 (silicon oxide) and 40-45% of Al_2O_3 (aluminium oxide). Other oxides such as Fe_2O_3, TiO_2, CaO and MgO can also be present, but in small quantities. Regarding the size of its particles, some authors refer to an average of approximately 3 μm in diameter being 99% of its particles smaller than 16 μm. Metakaolin properties are highly dependent on the raw material from which it comes and on the calcination temperature to which it was submitted. The density of the material presents values between 2.40-2.70 g/m^3 [5].

Blast furnace slag is a waste product from steel mills and is formed inside the furnaces from the melting of iron ore and dripping of liquid pig iron. The composition of blast furnace slag depends on the ores and the type of fuel used (mineral coal or wood coal), usually composed by SiO_2, Al_2O_3 and Fe_2O_3, the density of the material presents values between 2.90-3.16 g/cm^3. Several studies show how this waste can be used in the constitution of compound cements for the manufacture of concretes and mortars. This substitution brings numerous environmental, socio-economic and compressive strength benefits, and is pointed out by many researchers as a material with the necessary characteristics for partial replacement of cement even in large quantities [6].

Fly ashes is normally obtained from burning coal, with great availability in the market in general because it is cheaper than Portland cement and metakaolin, however, it doesn't present mechanical characteristics as high and as fast in curing time as these mentioned constituents. Furthermore, fly ashes provide good levels of durability, low mixing water requirements and, eventually, higher mechanical resistances in the long term. It is usually composed by SiO_2, Al_2O_3, Fe_2O_3, and the density of material presents values around 2.30 g/m^3 [7].

2.2. Use of Diatomaceous Earth

Natural diatomaceous earth is a natural material formed by diatomites deposited in the ocean and lakes over thousands of years. Diatomite is a mineral of biogenic and sedimentary origin, formed by the accumulation of diatomaceous algae that fossilized since the Pre-Cambrian period, presenting a white colour and being highly rich in aluminosilicates. Several physical properties of diatomite add commercial value to this raw material, such as low apparent density, high porosity and surface area, which are extremely necessary and important properties for different kinds of industries [8].

Diatomaceous earth is marketed in two ways: calcined and non-calcined. The difference between the two is very important depending on what purpose is being used for. Calcined diatomaceous earth has been treated at a temperature above 1000 ºC. The purpose of this is to further harden the exoskeletons of the diatoms in order to create a better filtering agent. This process causes the amorphous silica that makes up the exoskeleton of the diatom to turn in to crystalline silica. They are used mainly as water filters in swimming pools and as absorbents for heavy liquids. The crystalline silica can be toxic to humans and animals when inhaled. Calcined diatomaceous earth is not used for animal feed and is not food grade. After use and when calcined, diatomaceous earth loses its filtering capacity, it becomes a residue with toxic properties to the environment. The residual diatomaceous earth is finally sent to treatment management facilities for this type of residue. Non-calcined diatomaceous earth meaning that it has not been treated at a high temperature. The amorphous silica remains in its natural state and is not considered harmful to animal or human health. It is marketed in its dry state, a fine white powder, used in the agricultural sector as an insecticide and/or ecological fertilizer [9].

Microstructural studies and analysis carried out on several samples collected in the pyramids of Giza revealed evidence that supports the hypothesis of the use of artificial stones in their construction, a material constituted by an aggregate of fine limestone previously ground and calcium silicates, produced from a reaction between diatomaceous earth and fine limestone. These earths are believed to have been collected from diatomaceous earth deposits at Fayoum, located about 70 km south of Cairo, Egypt [10].

Nowadays, as with traditional aluminosilicates, it is believed to be possible to develop the process based on the alkaline activation of aluminosilicates with diatomaceous earth, natural or residual. The main characteristics of this material are the high porosity and low thermal conductivity, however, it also has pozzolanic properties very similar to fly ash and metakaolin [11].

2.3. Conventional Alkaline Activation for Geopolymer Concrete

Alkaline activation, or geopolymerization, is a reaction that consists in the hydration of aluminosilicates with alkaline or alkaline earth type substances. The alkaline type substances used can be NaOH (sodium hydroxide) or KOH (potassium hydroxide) [12].

The reaction mechanism of the geopolymer or geopolymerization occurs by the exothermic phenomenon, due to polycondensation. This is, the binders originated through alkaline activation are divided into two phases, the first is the dissolution of the aluminosilicates from the raw material used when mixed with an alkaline solution (activator), thus obtaining the gelation phase, and the second phase, polycondensation, where the polymerization and hardening process of the reaction products in the polymeric structure is developed [13].

During this chemical reaction, the water expelled over the curing process is beneficial to the performance of the geopolymers, as it eliminates discontinuous nano-pores from its formation matrix. Water only provides workability to the mix, unlike what happens in Portland cement mixes, where the chemical reaction of water helps in the hydration process. However, although the water expelled in the curing process is beneficial, geopolymers are very sensitive to the curing process, especially with regard to the risk of retraction that arises associated with water loss [14].

The use of alkaline solutions in aluminosilicate rich materials opens up new opportunities for obtaining special cements. In recent years, several geopolymeric cements based on blast furnace slag, volcanic rocks, fly ash and iron silicates have emerged, as an alternative to Portland cement. In addition to help reducing environmental impact, geopolymer cements offer high initial strength, low retraction, freeze-thaw resistance, abrasion resistance, sulphate resistance and corrosion resistance, these properties make this material ideal for long-term containment of pathologies in cementitious surfaces [15-16].

2.4. Alkaline Activation using Diatomaceous Earth

In order to develop geopolymer cements, a completely amorphous and inorganic binding material is required. Different studies reveal several ways of drying and calcining original and/or residual diatomaceous earth, with different temperatures and calcination times, reaching important data on its chemical composition and diverse physical properties, mainly the amount of SiO_2 (silicon dioxide) present in the material, the main compound that reacts with the alkaline solution. Distinct geopolymers are also achieved with this material, changing the amount of alkaline reagent used in mixing, adopting different compositions and aggregates in the mixture, different curing processes, and various physical, mechanical and thermal characterization tests.

In [17], natural diatomaceous earth collected in Mina Ponte – Bahia (Brazil) show moisture values ranging from 30 to 60%, being subsequently naturally dried until it as moisture values ranging between 10 and 20%. The chemical characterization of this earth reveals a SiO_2 value equal to 88.2%. When this material is calcined at temperatures ranging between 800 and 1000 °C the percentage of SiO_2 increases to 90%. It is also known that the density of diatomaceous earth increases from 2.0 to 2.3 g/cm^3 and the surface area decreases in values in the range of 10-30 m^2/g to 0.5-5.0 m^2/g after the calcination process.

In [18], natural diatomaceous earth collected in the Nile River – Cairo (Egypt) was naturally dried, after this procedure their chemical characterization was carried out, which revealed a SiO_2 value equal to 66.88%. Other characterization tests revealed that the dry density of the material is equal to 0.63 g/cm^3 and the wet density is equal to 1.66 g/cm^3, the surface area is equal to 55.66 m^2/g. After undergoing a calcination process, it is known that water is released at temperature values in the range of 100-200 °C and organic matter was removed at temperature values in the range of 250-550 °C. The dry density of the material decreases from 0.63 g/cm^3 at normal temperature to 0.43 g/cm^3 at 1000 °C, and the surface area increases from 55.66 to 64.80 m^2/g on heating up to 400 °C, however, decreases to 30.46 m^2/g at 1000 °C.

In [19], natural diatomaceous earth collected in Lampang (Thailand) has a SiO_2 value equal to 77.46% and a surface area equal to 46.8 m^2/g. After undergoing a calcination process at 800 °C the material was sieved and divided into small, medium and large particles, the SiO_2 value for small particles rose to 79.76%. After this procedure, they were mixed for 5 minutes with different solutions of NaOH and KOH and water was added for another 5 minutes. The final mix obtained was placed in cubic specimens of 50x50x50 mm and wrapped in plastic film to prevent loss of moisture during the curing process. After 1 hour of curing the samples were placed in an electric oven at 60, 75 and 90 °C for 5 days, at the end of the established days the samples were removed from the oven and allowed to cool at room temperature until they were demoulded, and finally submitted to compression tests. The results revealed that the geopolymer activated with NaOH has greater compressive strength. The ideal temperature and curing time are 75 °C for 5 days and different ratios of Na_2O/Al_2O_3 (Sodium Oxide/Aluminium Oxide) used in the mixture significantly alter the geopolymer properties, with ratios from 1,0 to 3,0 increasing compressive strength from 1.08 to 5.88 MPa as well as density from 0.93 to 1.5 g/cm^3.

In [20], residual diatomaceous earth used in the Colombian beer industry was employed as a source of aluminosilicates to obtain geopolymers based on fly ash and metakaolin. This material underwent a calcination process at a temperature of 400 °C for 3 hours, then shown a density equal to 2.06 g/cm^3, a surface area equal to 12.99 m^2/g, and after chemical characterization a SiO_2 value equal to 91.86%. The mixtures were prepared for 10 minutes with 70% fly ash, 30% metakaolin, and an alkaline solution of diatomaceous earth, NaOH and water. The mixtures were then placed in 20x20x20 mm cubic specimens for 24 hours at 60 °C with 90% relative humidity and then kept at room temperature until reaching sufficient age to be subjected to compression tests. The results revealed that the compressive strength value at the end of 28 days is equal to 36 MPa and at the end of 360 days is equal to 38 MPa.

In [21], residual diatomaceous earth from the Spanish wine industry was used as a source of aluminosilicates to obtain geopolymers. This material was calcined at 650 °C for one hour. The chemical composition reveals a SiO_2 value equal to 80.88%. Mixtures of calcined and non-calcined diatomaceous earth, with water and NaOH, were then placed in cubic specimens of 100x100x100 mm and cured at 20 °C with 95% relative humidity for 28 days. The specimens obtained were submitted to compression tests and the results show that the calcination process significantly improved the mechanical properties of mortars developed with calcined diatomaceous earth, due to the elimination of organic matter. At the end of 28 days, the specimens with non-calcined diatomaceous earth presented a compressive strength value equal to 16.66 MPa, and the calcined diatomaceous earth specimens a value of 39.32 MPa.

In [22], residual diatomaceous earth collected in South Bohemian (Czech Republic) in a earth treatment and processing company was used in mortars modified with Portland cement. This material showed a high moisture content, a value of 62%, having been subsequently dried at 100 °C for 24 hours and then ground for 2 minutes in a vibrating mill. A SiO_2 value equal to 78.20% was verified after chemical characterization, a density value between 1.98-2,06 g/cm^3 and a surface area value between 1,67-2,06 m^2/g is also known. In this case, there was no alkaline activation of the aluminosilicates, but the residual diatomaceous earth was used as an aggregate in mixtures with Portland cement, silicon sand, ground limestone and additives (EVA copolymer and cellulose ether). The reference mixture is composed of 37% Portland cement CEM I 42.5R, 16% silicon sand, 40.3% ground limestone and 6.7% additives, while in one of the mixtures with the residual earths was used 37% Portland cement CEM I 42.5R, 16% silicon sand, 34.77% ground limestone, 5.53% residual diatomaceous earth and 6.7% additives. The samples were placed in specimens of 100x100x100 mm and submitted, at the end of 28 days of curing, to tests of resistance to compression and bending, before and after being subjected to a temperature of 200 °C at a speed of 10 °C per minute. In relation to the reference mixture, the results revealed an increase in compressive strength from 18.1 to 20.6 MPa and an increase in flexural strength from 5.0 to 5.2 MPa. For specimens subjected to 200 °C, the compressive strength increased from 14.3 to 20.4 MPa and the flexural strength from 4.5 to 5.1 MPa.

3. CONCLUSIONS

The use of aluminosilicate rich materials in the production of geopolymer cements is a consolidated fact, the materials achieved with this manufacture process have already shown to offer good characteristics of mechanical resistance, abrasiveness, corrosion, and with less CO_2 emissions during their manufacture when compared to Portland cement. It is also a fact that diatomaceous earth has very similar properties to the materials used in the production of geopolymer cements and it is firmly believed to be possible to use this material in such mixtures.

The different studies using residual diatomaceous earth reveal high values of moisture present in the residue, due to its porosity and its high absorption capacity. To obtain a material that is completely inorganic and capable of being used in different mixtures, it is necessary for it to undergo a calcination process. It is known that at temperatures between 100-200 °C the humidity present in the residual diatomaceous earth is completely eliminated, the organic matter is eliminated at temperatures between 250-850 °C, and its porous structure is improved at temperatures above 900 °C. The percentage of SiO_2 present in the material is improved after the calcination process, its density also increases and the surface area increases during the calcination process above 400 °C, but decreases at temperatures of 1000 °C and above.

To obtain geopolymer cements, the alkaline reagent to be used is NaOH, the alkaline activation with this reagent yields better results of compressive strength and density of the geopolymers achieved. The amount of reagent used in the manufacture of geopolymers directly influences the density and strength of the geopolymer. The temperature, air humidity, and curing time of the materials are also very important and influence all the characteristics of the geopolymer obtained. Geopolymers made from calcined residual diatomaceous earth show higher compressive strength than geopolymers made from non-calcined residual diatomaceous earth.

The calcined residual diatomaceous earth can be used in alkaline activation mixtures, added to alkaline activation mixtures with fly ash and/or metakaolin, and also used as aggregate in mixtures with Portland cement. The materials obtained by introducing calcined residual diatomaceous earth in mixtures with Portland cement show improvements in compressive strength and flexural strength characteristics and also strength improvements after being subjected to elevated temperatures.

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