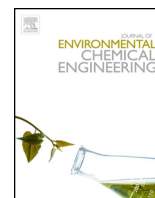




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Co-valorization of marine sediments and construction & demolition wastes through alkali activation

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ABSTRACT

In the present experimental study, the co-valorization potential of marine sediments and construction & demolition wastes (C&D wastes) through alkali activation is investigated. Specimens were produced by mixing raw materials, namely marine sediments and C&D waste components (tiles, bricks and concrete) with the activating solution consisting of KOH and sodium silicate solution. The produced specimens were then subjected to compressive strength testing. The effect of the molarity of the alkaline activating solution as well as the percentage of each waste in the initial mixture on the compressive strength of the final products was also assessed. Also, the effect of high temperature heating (400–800 °C), immersion in water or seawater and subjection to freeze–thaw cycles on the structural integrity of the produced specimens was investigated. Analytical techniques, namely X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) were used for the identification of the morphology and structure of the final products.

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1. Introduction

Alkali activation of aluminosilicates can be accomplished at relatively low temperature and results in the production of materials, called geopolymers or inorganic polymers [1]. These cementitious materials, consisting of Si–O–Al bonds, are characterized by partially or fully amorphous polymeric structure, high early strength and high chemical and temperature resistance. They are suitable for a number of applications in the construction industry and are characterized by lower environmental footprint when compared with traditional construction materials, such as Ordinary Portland Cement (OPC) and concrete [2–5]. Although geopolymers can be produced with cost and properties comparable to OPC, there are some drawbacks such as loss of workability, quick setting time as well as health and safety implications when working with strong alkali solutions. This is the main reason why geopolymers have been proposed to be used mainly as pre-cast concrete (in railway traverses, pipelines, hydraulic structures, pre-tensioned concrete structures etc), where the above drawbacks can be easily adapted.

Today, most research efforts focus on valorization of several solid waste streams and the production of materials with suitable

physico-chemical, mechanical and thermal properties, that are suitable as alternative binders for the construction industry, fire/corrosion resistant materials or matrices for the encapsulation of hazardous elements [6–10]. Waste valorization results in minimization of the consumption of natural resources, reduction of the volume of wastes that are ultimately landfilled and reduction of the emissions of greenhouse gases (GHGs) [11–15].

Dredging operations in ports and rivers, in order to improve navigation, result in the excavation of big volumes of sediments that may often require special treatment and remediation [16]. In Europe, 100–200 Mm³ of sediments are dredged annually to maintain efficiency in navigational waterways [17,18]. The management of sediments is an important issue and has several environmental and social implications [19]. The most important *ex situ* remediation technologies investigated so far include phytoremediation [20], electrokinetic remediation [21], bioleaching and stabilization [22–24] and development of technosols through composting [25].

Very interesting recent studies investigated the potential of calcination of marine sediments, in temperatures varying between 400 °C and 750 °C, for the subsequent production of geopolymer source materials and showed that increase of calcination temperature increases reactivity of the raw materials [26–29]. Other recent studies examined the use of different types of river and lake sediments for the production of bricks with high insulating capacity or aggregates which are suitable for cement

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mortars [30,31]. Liao et al. [32] investigated the potential of water reservoir sediments, mixed with sodium hydroxide, for the production of lightweight aggregates after calcination at higher temperature, ranging between 1045 °C and 1085 °C. Snellings et al. [33] investigated the effect of flash calcination of dredging sediments, carried out between 820 °C and 905 °C, on their properties and reactivity and mentioned that alternative supplementary cementitious materials (CSM) suitable for blended cements can be produced. In all these studies the pretreatment step of calcination, which results in increased energy consumption, is included. Thus, it will be interesting to explore in the near future cost and more energy efficient alternative routes for the valorization of marine sediments.

C&D wastes represent an important waste stream and huge quantities are produced every year in most countries [34–37]. The European Commission considers C&D wastes as priority waste stream for reuse and underlines their important environmental benefits as a result of their valorization [38]. C&D wastes are mainly used as recycled aggregates in concrete, as additives in cement production and as road base materials. The main factors that hinder the wider use of recycled C&D wastes in the construction industry include the lack of standards and specifications for recycled concrete production, the distance between demolition sites and recycling plants as well as lack of confidence from end users for the quality of the final products [39]. So far, studies have been carried out to investigate the potential of alkali activation of C&D wastes for the production of geopolymer binders and defined their properties and microstructure [40–43]. Recently, it has been shown that two of the components of C&D wastes namely bricks and tiles, which contain amorphous/semi-crystalline silica or aluminosilicate compounds, can be successfully alkali activated [44,45], while recycled demolition aggregates and crushed brick can be used for the production of geopolymers comprising calcium carbide residue, fly ash and slag as precursors [46] and ceramic sanitaryware as an alternative for the development of new sustainable binders [47].

The present experimental study aims to investigate the co-valorization of marine sediments and C&D wastes through alkali activation, define the morphology and determine the main factors that affect the properties of the final products. The study has a noticeable degree of novelty since so far very few studies are available in the international literature investigating valorization of marine sediments through alkali activation.

2. Materials and methodology

Marine sediments were collected from the ports of Souda in Chania, Crete, and Patras in NW Peloponnese, Greece. C&D waste components, including tiles, bricks and concrete, were collected from various demolished buildings of the city of Chania. Both sediments and C&D waste components were pulverized using a FRITSCH-BICO pulverizer (Germany) and homogenized. A Master-sizer S (Malvern Instruments) particle size analyzer was used for particle size analysis (Table 1). Table 2 shows the chemical composition, in the form of oxides, of sediments and each component of C&D wastes, as derived from an X-ray fluorescence energy dispersive spectrometer (Bruker-AXS S2 Ranger). Loss on

ignition (LOI) was determined by heating wastes at 1050 °C for 4 h. The weight percent of each raw material subjected to alkali activation as well as the codes of all specimens produced are presented in Table 3. The activating solution was prepared by dissolving the required amount of KOH pellets (Sigma Aldrich) in distilled water to obtain solution molarity 2–12 M and then adding sodium silicate solution (Na_2SiO_3 , Merck, $\text{Na}_2\text{O}=7.5\text{--}8.5\%$, $\text{SiO}_2=25.5\text{--}28.5\%$). The final solution was allowed to cool at room temperature for 24 h prior to use. The liquid/solid (L/S) ratio varied between 0.25 and 0.35 for each combination in order to improve the flowability characteristics of the resulting paste. An indicative composition of the starting mixture is (% weight): raw materials 82%, H_2O 6%, KOH 3% and Na_2SiO_3 9%.

The produced paste was cast in cubic metal moulds of 5 cm edge, which were vibrated for a few minutes to eliminate the presence of air voids in the reactive mass and improve the properties of the final solidified specimens. The moulds remained at room temperature for 2 h to allow early initiation of reactions, development of structural bonds and solidification of the paste. Then, the specimens were demoulded and sealed in plastic bags to prevent fast evaporation of water during curing and heated at 80 °C in a laboratory oven (ON-02G) for 24 h. After cooling and aging at room temperature for 7 days, compressive strength measurements were done using a MATEST C123N load frame. All tests and measurements were carried out in triplicate.

The morphology and structure of the final products was studied using analytical techniques. Fourier Transform Infrared Spectroscopy (FTIR) analysis was carried out, using pellets produced after mixing a pulverized sample of each specimen with KBr at a ratio of 1:100 w/w, with a Perkin-Elmer Spectrum 1000 spectrometer (USA). Scanning Electron Microscopy (SEM) analysis was performed with a JEOL 6380LV scanning electron microscope equipped with an EDS INCA microanalysis system with low vacuum, pressure 30 Pa, voltage 20 kV and 10–12 mm working distance from the detector. X-ray Diffraction (XRD) analysis of the raw materials and the produced specimens was performed using a Bruker AXS (D8 Advance type) diffractometer with Cu tube, scanning range from 4° to 70° 2 θ , step 0.02° and measuring time 0.2 s/step. The qualitative analysis was assessed with the use of the DIFFRAC^{plus} EVA v. 2006 software and the Powder Diffraction File (PDF-2) database.

The thermal behaviour of the specimens was evaluated after heating them at 400, 600 or 800 °C for 1 h in a laboratory furnace N-8L Selecta. The structural integrity of selected specimens was assessed after immersing them in tap water and seawater for a period of up to 3 months or subjecting them to 20 freeze–thaw cycles, according to ASTM standard C1262-10 [48].

3. Results and discussion

3.1. Compressive strength

Fig. 1 shows the evolution of the compressive strength of the specimens produced from single wastes, namely C&D waste components (tiles, bricks and concrete), Patras sediments and Souda sediments vs KOH concentration. Specimen codes are given in Table 3, while error bars denote standard deviation of three specimens.

Regarding sediments, it is seen from this data that only Patras sediments can be alkali activated. The compressive strength of the produced specimens increases from 8.5 to 19 MPa when KOH molarity increases from 2 to 4 M, while further increase of molarity (6 to 12 M) has no additional beneficial effect. Souda sediments are only slightly alkali activated (maximum compressive strength is 5 MPa). This is mainly due to their lower content of SiO_2 which reduces the molar ratio of $\text{SiO}_2/\text{Al}_2\text{O}_3$ (4.5 compared to the

Table 1
Particle size of the raw materials.

| | Souda sediments | Patras sediments | Tiles | Bricks | Concrete |
|----------------------------|-----------------|------------------|-------|--------|----------|
| size (μm) | <120 | <120 | <140 | <140 | <190 |
| d_{50} (μm) | 9 | 8 | 14 | 7 | 10 |

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