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SOILS

Influence of Adding Jordanian Scoria on the Properties of Clay-based Geopolymer

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ABSTRACT

This work aims to investigate the influence of adding Jordanian scoria on the characteristics of clay-based geopolymer. The clay deposit and scoria were gotten from north-east Jordan. The chemical, mineralogical, and microstructural properties of the used materials were examined. Scoria was added to clay-based geopolymer mixtures in different ratios: 0%, 10%, 20%, 30%, 40%, and 50%. Comprehensive experimental tests were conducted to assess the effect of adding scoria on the properties of the produced geopolymer. The results revealed decreased compressive strength and dry density, whereas porosity, water absorption, cation exchange capacity, and specific surface area increased as the ratio of scoria increased. The mineralogical and microstructural analysis of the geopolymers after adding scoria indicates the formation of mineral phases, namely hydroxy-sodalite and hydroxy-cancrinite in addition to the gel phase.

Keywords: Clay-based geopolymers; Scoria; Clay deposits; Geopolymerization; Volcanic materials.

Efectos en las propiedades de geopolímeros a base de arcilla tras añadir escoria volcánica de Jordania

RESUMEN

Este trabajo se enfoca en investigar los efectos en las características de geopolímeros a base de arcilla tras añadirles escoria volcánica de Jordania. La arcilla y la escoria volcánica fueron tomadas de depósitos en el noreste de Jordania. Luego se examinaron las propiedades químicas, mineralógicas y microestructurales de los materiales usados. La escoria volcánica fue añadida en diferentes proporciones en mezclas de geopolímeros con base en arcilla: 0 %, 10 %, 20 %, 30 %, 40 %, y 50 %. Posteriormente se realizaron pruebas experimentales para evaluar los efectos en las propiedades de los geopolímeros producidos tras haber añadido escoria volcánica. Los resultados revelan un decrecimiento en la fuerza compresiva y en la densidad seca, mientras que la porosidad, la absorción de agua, la capacidad de intercambio catiónico, y el área de superficie específica se incrementaron cuando la proporción de escoria volcánica también se incrementó. El análisis mineralógico y microestructural de los geopolímeros después de la adición de escoria volcánica indica la formación de fases minerales como la hidroxisodalita e hidroxicancrinita además de la fase de gel.

Palabras clave: Geopolímeros basados en arcillas; escoria volcánica; depósitos de arcillas; geopolimerización; materiales volcánicos.

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1. Introduction

Geopolymerization is a chemical reaction between alumino-silicate oxides and alkali solutions yielding polymeric Si-O-Al bonds with amorphous to semi-crystalline three-dimensional silico-aluminate structures (Davidovits, 1991). The primary materials used in the geopolymerization process to provide a high proportion of alumina and silica are aluminosilicates. In most cases, a total composition of Al_2O_3 and SiO_2 of more than 70% is recommended (Davidovits, 1991; Cioffi et al., 2003). Al_2O_3 and SiO_2 make up 70–90% of the clay composition (Abdullah et al., 2018). The alkaline solution acts as a reactor for the dissolution of aluminosilicates and the contribution of SiO_2 to the geo-polymerization reaction (Singh et al., 2005). The most common alkaline activators used are NaOH or KOH and silicate solutions (Na_2SiO_3 or K_2SiO_3) (Xu and van Deventer, 2002; Barbosa et al., 2000). Various types of additives could be used in the activation process to improve the properties of geopolymers, including sand grains (Subaer and van Riessen, 2010; Bumanis et al., 2017; Lemougna et al., 2011), crushed brick (Barbosa & MacKenzie, 2003), corundum (Kamseu et al., 2010), vermiculite (Medri et al., 2015), and zeolitic tuff (Almjadleh et al., 2014; Khoury, 2003). The use of additives depends upon the content of aluminosilicates, mineral components, and the shape and size of the grains (Lukkonen et al., 2018). Previous studies have shown that the use of additives in alkali activation may improve the workability of mixtures (Koehler and Fowler, 2009) and the physical characteristics of the products, such as mechanical strength, durability, and stability (Buchwald et al., 2009.

Scoria aggregates have a black color, are light in weight, and have a vesicular texture, with diameters ranging from millimeters to centimeters (Sen, 2014). The vesicular texture caused by volcanic gases escape during the eruption. Minerals that form in hot water-rich fluids, such as zeolite, calcite, and quartz, are sometimes reintroduced into these vesicles (Stiegeler, 1976). The scoria clasts oxidize and turn a dark reddish-brown color (Sen, 2014). Scoria contains significant amounts of alumina (Al_2O_3) and silica (SiO_2), making it reactive as a pozzolanic material (Swamy, 1987). As a result, it has a wide range of industrial applications, including cement replacement, lightweight concrete manufacturing, insulating materials, filter materials, absorbents, and other architectural applications (Hossain & Lachemi, 2010).

Numerous studies examined the use of volcanic materials (pozzolanic materials) such as volcanic tuff, volcanic ash, and zeolitic tuff as source materials or additives in geopolymerization. In Jordan, several studies on the development of geopolymer composites have been performed with volcanic materials as additives for cement replacement and water treatment (Yousef et al., 2009; Hamaideh et al., 2014; Al-Zboon et al., 2019; Alshaaer et al., 2015; Al-Zboon et al., 2016). The researchers noted that the composites enhanced the compressive strength and the adsorption capacity.

The present work aims to compensate for the effects of adding different ratios of Jordanian scoria (10%, 20%, 30%, 40%, and 50%) on the mechanical and structural properties of clay-based geopolymer in order to utilize the scoria in geopolymer production for waste water treatment.

2. Materials and Methods

A sample of clay deposits was collected from Al-Azraq area in northeast Jordan, approximately 110 km northeast of Amman (Figure 1). The collected sample was crushed, ground, and sieved to a grain size less than 63 µm, then mixed to become homogenized. To enhance the workability and reactivity, the prepared sample of clay was burned in a furnace at a temperature in the range of 660–700 °C for 2 hours. The aggregates of scoria were obtained from Tall Hassan area, about 125 kilometers northeast of Amman. The aggregates were ground to be suitable for mixing. X-ray diffraction (XRD) analysis of clay and scoria was performed by the use of a diffractometer (Shimadzu XRD-7000) at a scanning rate of 2.0°/min. The chemical compositions of both materials were obtained using an X-ray fluorescence (XRF) spectrometer (Shimadzu XRF-1800).

For geopolymer synthesis, sodium silicate solution (Na_2SiO_3) with silica content ranging from 25.6 to 27.6% and sodium oxide content ranging from 7.5 to 8.5% was mixed with 10 M of sodium hydroxide solution (NaCl, purity 95%) to prepare an alkaline solution at a mixed ratio of 1.5. A mixture of clay and alkaline solution was prepared at a solid/liquid ratio of 1.4 by using a mixer at different speeds to give a homogenous admixture. The mix was molded in three cubic steel molds (50×50×50 mm) and cured for 24 hours in a dry oven at 80°C. A group of specimens were kept dry in lab conditions for 7 days for measuring dry density and unconfined compressive strength (UCS). A group of specimens were soaked in tap water for 24 hours to measure the water absorption percentage (Wabs). For porosity measurement, the specimens were evacuated by a vacuum after drying in an oven at 105°C and then immersed in water for 24 hours. The specimens were then wiped to give them a dry surface and weighed.

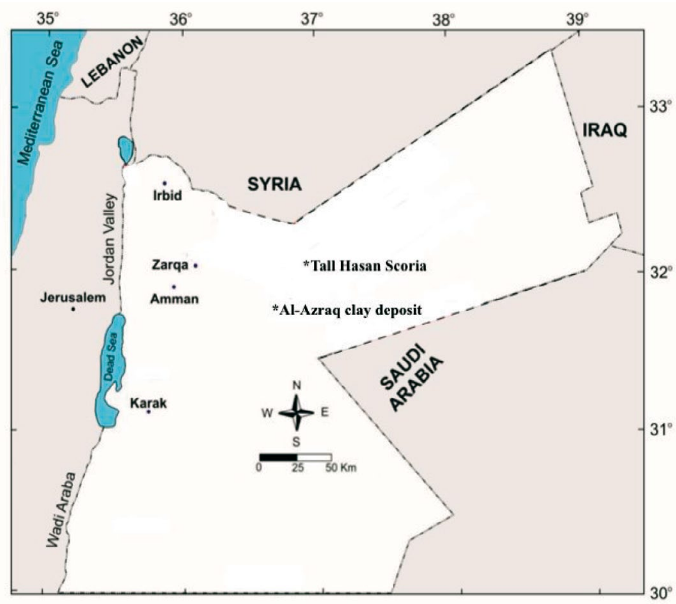


Figure 1. Location map of the materials sample area (modified after Khoury et al., 2015)

To examine the cation exchange capacity (CEC in meq/100g) for the prepared geopolymers, methylene blue dye was used as a cationic dye popular in industrial waste water. The dry specimens of geopolymer were ground, sieved to a particle size of 425 µm, washed with deionized water to remove the excess NaOH, and then dried at 105 °C. An initial concentration of methylene blue dye solution was added to the specific weight of the geopolymer. A UV-visible spectrophotometer was used for measuring the equilibrium concentrations of methylene blue dye. The specific surface area of geopolymers (SSA/m^2 g^{-1}) was determined according to the adsorption of molecules method as presented by Santamarina et al. (2002). A methylene blue dye solution was used to measure the total surface area.

To examine the effects of adding scoria on the characteristics of clay-based geopolymers, different mixtures were prepared with different ratios of scoria (0, 10, 20, 30, 40, and 50).

The experimental work was summarized as a flowchart in Figure 2.

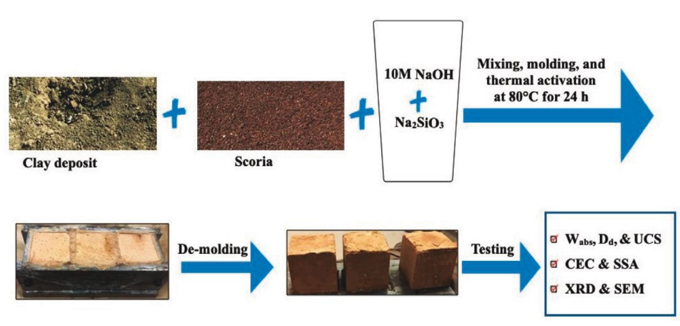


Figure 2. Flowchart of experimental works

3. Results and Discussion

Material Characterization

The results of the chemical analysis of the clay deposit show that silica (SiO_2) and alumina (Al_2O_3) are the major constituents of the used clay, representing 76.47% of the total contents (Table 1). The SiO_2/Al_2O_3 molar ratio is 4.66. The respective loss on ignition is 14.4%, which may indicate the presence of high organic content or water and amorphous phases. The chemical composition of scoria shows that the sample is mostly rich in silica (46.62%) and alumina (14.83%). In addition to iron (13.31%), calcium (9.81%), and magnesium (10.20%) oxides. The alkali content is less than 1%. Al-Swaidani (2018) investigated the chemical composition of scoria samples collected from several countries. The results show that the silica and alumina ranges are 40 to 60% and 10 to 20%, respectively. The iron oxide ranges between 5 and 16%, followed by magnesium (2–13%) and calcium (5–13%). The alkali content varies between 1 and 7%.

Table 1. Major elements of the base materials

Oxides wt.%	Al_2O_3	CaO	Fe_2O_3	K_2O	L.O.I	MgO	MnO	Na_2O	SiO_2	SO_3	TiO_2
Clay deposit	12.28	1.17	6.86	2.89	14.40	4.15	0.04	0.21	56.26	0.05	1.34
Scoria	14.83	9.81	13.31	0.83	2.00	10.20	0.22	0.31	46.62	0.06	2.03

Figures 3 and 4 show the XRD diffractograms of clay deposits and scoria. The results show that the dominant minerals in clay are illite, quartz, and kaolinite, with some amounts of montmorillonite. The clay sample shows broad peaks between 3–20° and 25–30° that indicate the presence of an amorphous phase in the raw clay (Figure 3). The used clay is considered common clay deposits or less pure clay resources containing multiple clay minerals (Khalifa et al., 2020).

The mineralogical composition of scoria indicates that the detected minerals are anorthite and augite as major minerals, with the addition of calcite and forsterite minerals (Figure 4). Previous work on scoria has confirmed this result (Djobo et al., 2014; Tchakoute et al., 2015; Al-Swaidani, 2018; Song et al., 2021). They revealed that the major minerals are plagioclase, olivine, and pyroxene.

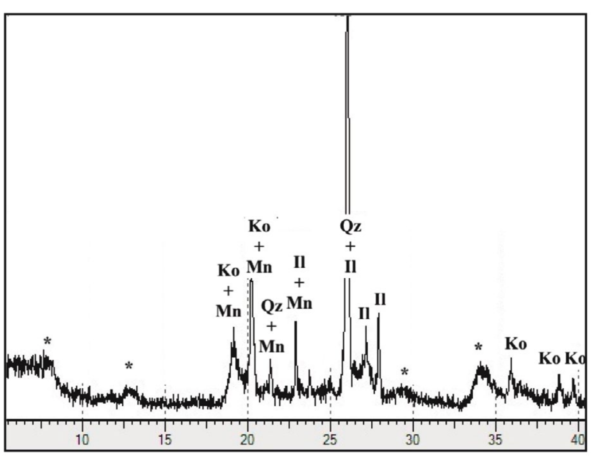


Figure 3. XRD spectrum of Al-Azraq clay deposit. Qz: quartz, Il: illite, Ko: kaolinite, Mn: montmorillonite, and *: amorphus phase

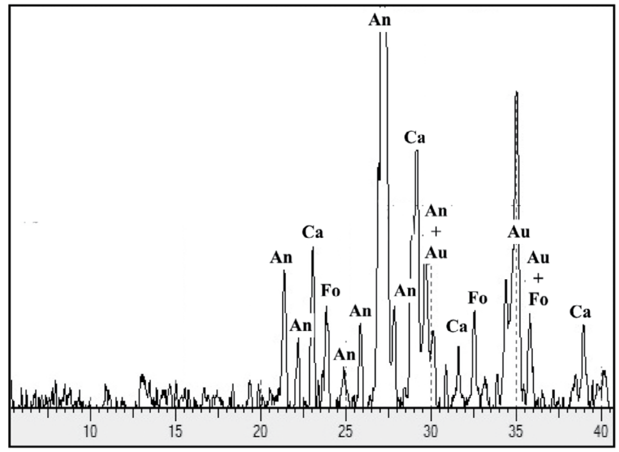


Figure 4. XRD spectrum of scoria. An: anorthite, Au: augite, Ca: calcite, and Fo: forsterite

Characterization of clay-based geopolymers

The effects of adding different ratios of scoria to clay during alkali activation were reflected in the percentage of water absorption, porosity, dry density, and compressive strength values. Figure 5 demonstrates that as the scoria ratio increases, the percentages of water absorption and porosity increase practically linearly. An increase in water absorption might be an indication that a highly porous matrix is developing. On the other side, as the scoria ratio rises, dry density and compressive strength almost linearly reduce (Figure 6). The dry density of the specimen decreased due to the low density of scoria. Furthermore, when the scoria ratio increases, the percentage of porosity also increases, affecting the strength of the mixtures. Many researchers found that the addition of volcanic materials to the cement mortar mixture increased water absorption and reduced density due to the high void ratio and specific surface area (Yourself et al., 2012; Al-Zboon and Al-Zou'by, 2014; Al-Zboon et al., 2019). The unreacted particles of scoria would behave as coarse aggregates in the matrix, decreasing the mechanical strength.

The results of CEC and SSA are shown in Figure 7 and demonstrate the influence of adding scoria during the alkali activation. According to the findings, the specimen with 50% scoria had the highest CEC (25.73 meq/100g) for methylene blue dye as a water contaminant, which was higher than the CEC for the free-scoria specimens (21.45 meq/100g). The measured SSA indicates that, in comparison to the free-scoria specimens (22.22 m^2/g), the specimen with 50% scoria had the highest SSA (29.67 m^2/g).

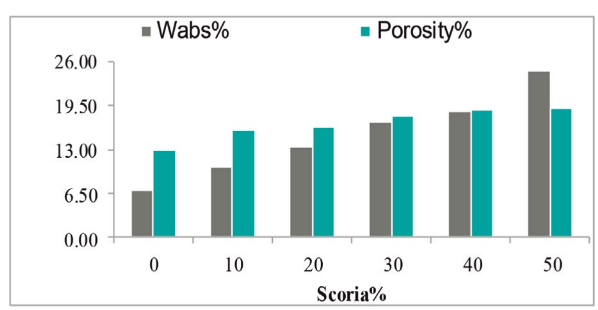


Figure 5. Water absorption and Porosity% Vs. scoria ratio

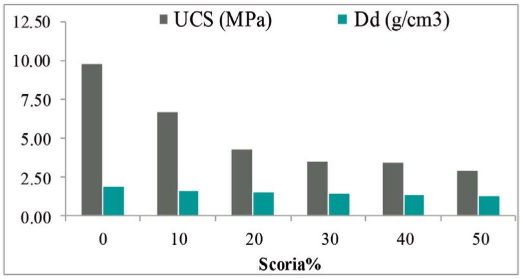


Figure 6. USC and dry density vs. scoria ratio

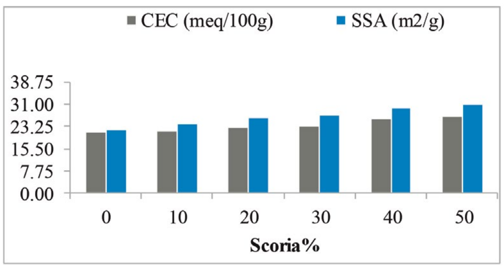


Figure 7. CEC and SSA vs. Scoria ratio

Figure 8 shows the XRD diffractograms of the clay-based geopolyer in contrast to the geopolymer with 50% scoria. The pattern revealed the formation of mineral phases of hydroxyl sodalite and Hydroxy-cancrinite, in addition to the remains of quartz and the remains of unreacted clay minerals. The broad peaks developed at 10◦-20◦ and 28◦-36◦ values in the XRD chart after adding 50% Scoria. This is an indication of the partial dissolution of scoria and the formation of a gel phase in the matrix. Referring to previous studies, solid and stable phases of hydroxy-sodalite, zeolite, and feldspathoids were formed after alkali activation of aluminosilicate materials due to the dissolution of Si and Al beside the gel phase (Yousef et al. (2009), Panias et al. (2007), Rahier et al. (2011), Slaty et al. (2013, 2015), and Alshaaer et al. (2016)).

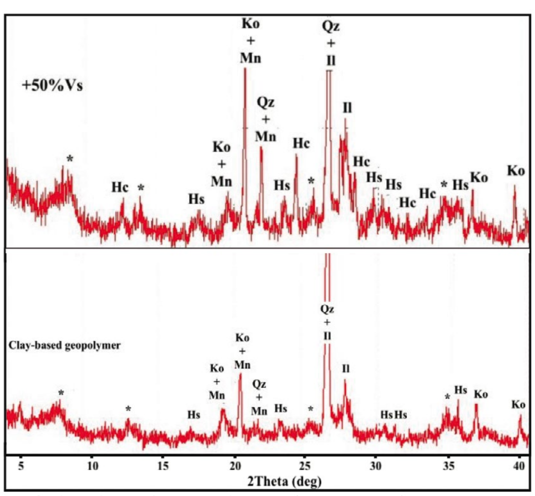
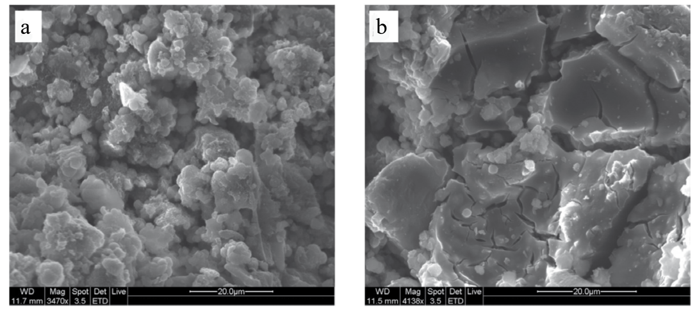


Figure 8. XRD patterns of clay-based geopolymer in comparison to geopolymer with scoria. Qz: quartz, Il: illite, Ko: kaolinite, Mn: montmorillonite, Hs:hydroxysodalite, Hc:hydroxycancrinite, and *: amorphus phase

The SEM micrographs of the clay-based geopolymer and the geopolymer with 50% scoria are presented in Figures 9 a & b. The gel phase appeared as spheres on the surface of the activated material and filled the pore spaces. The presence of the micropores indicates the development of new cavities in the matrix. The micrographs show that the presence of scoria results in a highly fractured structure. This result is consistent with the compressive strength result. Although the activation process created more cavities, scoria is considered a porous material, which provided appropriate diffusion pathways for metal ions and subsequently improved the adsorption of metals inside these pores (El-Eswed et al., 2013).



Figures 9. SEM micrographs **a.** clay-based geopolymer **b.** geopolymer with 50% scoria

4. Conclusions

This research utilized scoria as an additive material, resulting in an improvement in the removal efficacy of clay-based geopolymers for methylene blue dye. The used clay is predominantly composed of multiple clay minerals. The results revealed that adding more scoria for reaction components causes an increase in surface area and cation exchange capacity of the microstructure, thus causing a lower compressive strength and density. It can be concluded that scoria could be used as an additive material to improve geopolymer properties for environmental applications like waste water treatment.

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