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ORIGINAL ARTICLE

Development of functional geopolymers for water purification, and construction purposes

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Abstract This paper deals with the development of functional geopolymers based on local resources such as kaolinitic soil and zeolitic tuff for the construction of water storage containers and water transfer channels. The effect of water content on the mechanical performance and physical properties of synthesized geopolymers was evaluated. The results confirmed that the optimum ratio of water is 28% of clay fraction, which revealed observable improvements of physical, mechanical, and adsorption properties of the geopolymeric products. Such geopolymers showed the highest compressive strength, density, and maximum adsorption capacity toward cadmium among the products and precursors tested. The residual soluble salts in produced geopolymers were markedly reduced by using this optimum water content.

1. Introduction

It has been observed (Ingles, 1970) that through chemical polymerization reactions, the aluminosilicates such as clay (kaolinite) could be hardened and transformed into aluminosilicate polymers, also known as geopolymers, which are useful construction materials (Patfoort and Wastiels, 1989). Geopolymer can be defined as an amorphous, three-dimensional structure resulting from the polymerization of aluminosilicate monomers in an alkaline solution (Rowles and O'Connor, 2003). Geopolymers belong to the class of alkali activated cements (Shi et al., 2006).

The exact mechanism of the alkali-activation of kaolinite is not precisely known until now. There is a good possibility for

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Abbreviations: $W\%$, percentage of water absorption; W_w, weight of immersed specimen (g); W_d, weight of specimen after drying (g); JHK, natural kaolinite from Jordan; JZT, zeolitic tuff; M-JZ, geopolymer prepared from Jordanian kaolin and zeolitic tuff filler

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Na^+ or K^+ from the alkaline solution to exchange for the hydrogen ions on the broken edges of the clay. As a result of this ion exchange, repulsion between the Na^+ or K^+ ions will break down some clay particles into smaller grains of the soil and bind them together (Gemerts et al., 1989). Much more work has been done recently on understanding the reaction between thermally activated clays such as metakaolinite and alkaline solutions such as silicate solutions (Hajimohammadi et al., 2011; O'Connor and MacKenzie, 2010; Sagoe-Crentsil and Weng, 2007). Reaction mechanisms have been proposed for this geopolymerization involving the breakdown of the solid aluminosilicate into smaller 'monomers' where Al is already tetrahedrally coordinated. The second step is the polycondensation of these monomers into the geopolymer (Rahier et al., 2007; Duxon et al., 2007). As a result of these reactions, solid, hard, and stable materials with hydroxysodalite, feldspathoid, or zeolite like structures are formed (Davidovits, 1998).

When aluminum is in fourfold coordination, a negative charge is created and therefore the presence of cations such as Na^+ is essential to maintain the electric neutrality in the geopolymeric matrix (hydroxysodalite). Hydroxysodalite, which ranges from amorphous to microcrystalline material, consists of SiO_4 and AlO_4 tetrahedra linked alternately by sharing all the oxygen atoms. Positive ions ($Na^+, K^+, Li^+, Ca^{2+}, Ba^{2+}, NH_4^+$, and H_3O^+) must be present in the framework cavities to balance the negative charge of Al in the fourfold coordination.

There is a large-scale pollution in many areas around the world by micropollutants, an example being the Zarka basin in Jordan (Abderahman and Abu-Rukah, 2006). Low cost construction materials with high adsorption capacity are needed for constructing water storage systems, transportation channels, and low cost filters. To lower the cost, fillers could be used in different amounts in geopolymers depending on the workability of the overall mixture and the physical stability of the end products. These materials are usually the least expensive of the major ingredients of geopolymers. However, fillers may participate in geopolymerization process (functional reactive fillers) and thus are very important in establishing the performance of geopolymers. Zeolitic tuff-based geopolymers were already introduced (Alshaaer et al., 2009; Yousef et al., 2009) as construction materials for water treatment, storage, and transportation. The zeolite may also improve the adsorption capacity of the geopolymer.

Little work was found in the literature on the adsorption behavior of geopolymers. Wang et al., 2007) reported an amorphous aluminosilicate geopolymer resulting from solid-state conversion of fly ash. The synthesized geopolymer was found to have a higher adsorption capacity toward Cu^{2+} ion (92 mg Cu/g adsorbent) than the fly ash itself (0.1 mg Cu/g adsorbent).

This research focuses on optimizing the properties of geopolymers using natural zeolitic tuff as reactive filler. The zeolitic tuff which has a large availability in Jordan and many other places in the world is characterized by a high adsorption capacity and stability. The first concern is determining the optimum water content in the geopolymeric mixes. This will provide a material with suitable mechanical properties for the purpose of water storage and high adsorption capacity for water purification. The second concern is the adsorption characteristics of the produced geopolymers which will be compared to their starting materials, i.e. zeolitic tuff and kaolinite, toward Cd(II). This heavy metal is toxic and responsible for causing kidney damage, renal disorder, high blood pressure, bone fraction, and destruction of red blood cells. The main sources of Cd(II) in the environment are the industries of metal finishing, electroplating, pigments and plastic (Drush, 1993; Zhou et al., 1993).

2. Materials and methods

2.1. Materials

2.1.1. Kaolinite (JHK)

Kaolinite with a purity of 60% (Yousef et al., 2009) was collected from El-Hiswa deposit, which is located in the south of Jordan about 45 km to the east of Al-Quweira town. The elevation of the upper surface deposit ranges between 850 and 950 m above sea level. Thirty-two individual samples were collected from different layers of an outcrop, weighing in total 32 kg. These samples were mixed to represent 8 m of the upper layers of this outcrop. Preparation of the kaolinite samples involved crushing (using Jaw crusher, RETCH-BB1A) of oven dried clay (at 105 °C) to a grain size less than 425 μm. Then the samples were mixed in 50 L plastic drums for several times to make them more homogenous.

For a better understanding of the geotechnical properties of the samples and for optimizing the filler/clay/water/NaOH ratios, which should be used in the fabrication of specimens, kaolinite samples were tested for their plasticity limit. The plasticity limits are important to optimize the amount of water needed for the specimens' preparation. The plasticity limits, according to the ASTM D424 Plastic Limit and Plasticity Index of Soils, for the kaolinite (JHK) was found to be 22%.

The chemical composition of the kaolinitic soil (JHK) was determined using XRF analysis (Bruker System S4 Pioneer) as shown in Table 1.

2.1.2. Zeolitic tuff (JZT)

The zeolitic tuff was from North–East Jordan deposits (Aritayn area) located around 50 km to the east from Amman. The mineral composition of this tuff belongs mainly to the phillipsite type of zeolites (Nawasreh et al., 2006; Khoury et al., 2003; Yousef et al., 1999). The chemical composition of the zeolitic tuff (JZT) was determined using XRF analysis (Bruker System S4 Pioneer) as shown in Table 1.

2.2. Fabrication of geopolymer specimens

Seven different series of geopolymers with different water ratios were prepared from kaolinitic soil (JHK), functional reactive filler (zeolitic tuff, JZT), and an alkaline solution as shown in Table 2. The zeolitic tuff was added as a reactive filler to increase the adsorption capacity of the end products toward micropollutants. Sodium hydroxide was used in small amounts (15 to 20 wt.% compared to kaolinite) to avoid any residual alkali in the material and for economical reasons as well. It has been found (Alshaaer et al., 2007) that the optimum water content for high quality construction is close to the plasticity limit of the solid ingredients mixture (i.e. clay and filler).

The weighed filler (zeolitic tuff) and kaolinitic soil were mixed first and then sodium hydroxide solution was added. Good mixing is important to obtain homogeneous and identical specimens because when the ingredients of zeolite-kaolinite based geopolymer are mixed, a semi-dry mixture is formed.

Table 1 Chemical composition of kaolinite (JHK), and zeolitic tuff (JZT).

	SiO_2%	TiO_2%	Al_2O_3%	Fe_2O_3%	MnO%	MgO%	CaO%	Na_2O%	K_2O%	P_2O_5%	$SO_{3\%}$	Cl%	L.O.I%
JHK	48.80	0.86	25.10	7.50	0.01	0.21	0.68	0.21	1.40	0.16	2.93	0.10	12.00
JZT	38.30	3.12	11.2	13.10	0.180	10.30	7.39	1.96	1.09	0.41	0.10	0.17	12.60

Table 2 Composition of synthesized geopolymers.

Series	Clay (JHK)		Zeolitic tuff (JZT)		NaOH		Water	
ID	Ratio	%	Ratio	%	Ratio	%	Ratio	%
M-JZ.1	100	42.0	100	42.0	14	5.9	24	10.1
M-JZ.2	100	41.7	100	41.7	14	5.8	26	10.8
M-JZ.3	100	41.3	100	41.3	14	5.8	28	11.6
M-JZ. 4	100	41.0	100	41.0	14	5.7	30	12.3
M-JZ.5	100	40.7	100	40.7	14	5.7	32	13.0
M-JZ.6	100	40.3	100	40.3	14	5.6	34	13.7
M-JZ.7	100	40.0	100	40.0	14	5.6	36	14.4

Thus, good mixing is needed to avoid the agglomeration of the mixture and to keep the mixture non-compacted.

Specimens were fabricated and tested as illustrated in Fig. 1. Each mixture (series) was divided into eight specimens (50 g each). The specimens were molded immediately after weighing to avoid carbonation, drying and decrease of the workability of the mixture. The specimens were molded in a stainless steel cylinder (diameter of 25 mm and height of 45 mm) at a pressure of about 15 MPa (Carver hydraulic laboratory press). After unmolding the eight specimens of each series were cured by placing them in a ventilated oven (Binder-ED115) at 80 °C for 24 h. The specimens were not covered. After this curing time, the specimens were removed and allowed to cool down at room temperature. The eight cured specimens of each series were divided into three groups. Three specimens (Group-1) were further dried in an oven at 40 °C for seven days. Three other specimens (Group-2) were immersed in plastic containers filled with tap water at room temperature for seven days. The remaining two specimens (Group-3) were used for XRD and TGA measurements, leaching, and adsorption experiments.

2.3. Mechanical and physical characterization of geopolymers

Water absorption of the immersed specimens (Group-2) was calculated for each series according to Eq. (1) where W% is

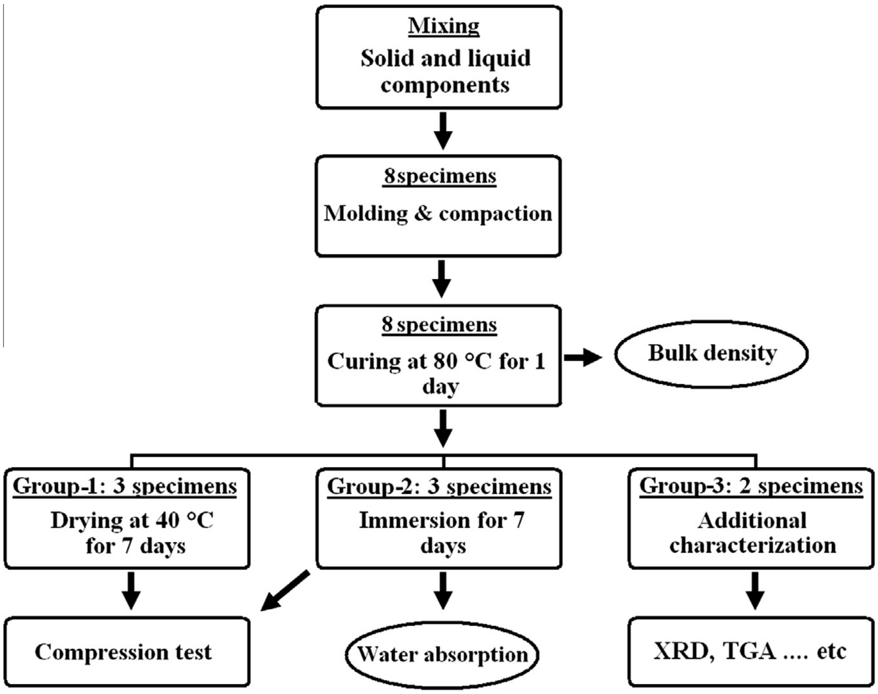

Figure 1 Experimental design of fabricating and testing the prepared geopolymers.

the absorption of water expressed as a percentage; W_w and W_d are the weights of the immersed specimen and of the specimen after drying, respectively.

$$W\% = ((W_w - W_d)/W_d) \times 100\% \quad (1)$$

For density measurements, the eight specimens were weighed after the curing using an electronic balance (SPE2001, Scout Pro) and their dimensions were measured using a digital micrometer (electronic digital caliper, 0.155 mm).

The compression test was performed using a CONTROLS testing machine (Model T106 modified to suit with standard testing), where the load was applied and increased by a displacement rate of 2 mm/min.

2.4. X-ray diffraction (XRD)

The X-ray diffraction (XRD) measurements were carried out (X-ray diffractometer-6000, Shimadzu) on powdered samples of synthesized geopolymers. The XRD patterns were measured from 5° to 80° 2-θ at a scan rate of 2°/min. The crystalline phases were identified by using the software package supplied with the instrument.

2.5. Thermo gravimetrical analysis (TGA)

Samples of about 40 mg were tested by TGA (using STA 409 thermal analyzer, NETZSCH) to evaluate the structural and water loss changes as a function of temperature with a constant heating rate of 5 $K.min^{-1}$.

2.6. Scanning electron microscope (SEM)

The morphology of the specimens was studied using an Inspect F50 scanning electron microscope (Netherlands). The samples were pre-coated with platinum under an argon atmosphere.

2.7. Determination of skeletal density using helium-pycnometry

The measurements of skeletal density (using Micrometrics AccuPyc 1330 He-pycnometer) were performed as follows: helium was first loaded in a calibrated reference volume, and then expanded in a chamber filled with the sample (Lowell et al., 2004). The difference in the pressure of the helium in the known cell volume without and with the specimen gives the 'skeletal' volume of the material. Dividing the mass of the specimen by this volume, true density is yielded. Four samples were prepared to measure the variations of skeletal density (by using helium-pycnometry) as a function of temperature. The volume of each of them is around 6 cm^3. The samples were crushed into small aggregates. Afterward, they were heated for 24 h at 105 °C before testing. The specimens were put immediately in the testing chamber to avoid any moisture uptake.

2.8. Adsorption of Cd(II) ion

Samples of synthesized geopolymers and their raw materials were ground and sieved into aggregate sizes of 250 μm and 500 μm. 0.5000 g of sample was placed in a 500 mL closed reagent bottle, to which 250 mL of 100 ppm Cd(II) standard solution was added. The solution was adjusted to pH 4 and shaken in a shaker water bath at 25 °C and 320 rpm for 24 h. A 1.0 mL sample of adsorbate solution was withdrawn at different contact times (0–72 h) and diluted to 5 mL with distilled water. The cadmium concentration of the diluted solutions was measured using the atomic absorption spectrometer.

2.9. Water-soluble salts

To estimate the water-soluble salts in the end geopolymer, a representative sample, 5 g, of synthesized geopolymers (M-JZ.1−M-JZ.7) was prepared from specimens of group 2 after performing the compression test. This sample was powdered, thoroughly homogenized, and dried at 105 °C for 24 h. Five grams of the powdered sample was placed in a bottle to which 200 mL of distilled water was added. The soil–water mixture was excessively stirred in a mechanical shaker until a constant Electrical Conductivity of the solution was reached. The Electrical conductivity (EC) and pH values of the solution were measured (using WTW multi-line P4, pH and EC meter) as a function of time; until they became constant (complete leaching of water-soluble salts was achieved). The concentration (ppm) of dissolved Na^+ was determined in the solution using an atomic absorption spectrophotometer (PERKIN-ELMER model A Analyst 200) for low concentrations and a flame photometer (Sherwood Model 410) for high concentrations.

3. Results and discussion

3.1. Microstructural characteristics

The XRD patterns of kaolinite (JHK), zeolitic tuff (JZT), and synthesized geopolymers (M-JZ.1−M-JZ.7) are shown in Fig. 2. The kaolinite spectrum showed quartz accompanied with kaolinite. The XRD spectrum of zeolitic tuff showed the characteristic peaks of phillipsite-K. Upon geopolymerization, the intensity of the phillipsite related peaks reduced due to dissolution as shown in the XRD pattern of M-JZ.1. All geopolymer spectra showed at least two peaks corresponding to new feldspar mineral that is formed as a result of geopolymerization.

SEM analysis, Fig. 3 indicates that the microstructure of geopolymers (M-JZ.4), is characterized by the coexistence of geopolymer gel, partially unreacted kaolinite sheets, and residual zeolitic tuff, Fig. 3A. SEM image reveals also that due to geopolymeric reactions the gaps between partially reacted kaolinite sheets have been filled with formed sodium aluminosilicate particles, Fig. 9B. This finding is in agreement with the results reported by Rahier et al. (2007), who produced partially amorphous sodium aluminosilicates after activation of metakaolinite with sodium silicate.

Thermo gravimetric analysis, TGA, was carried out on synthesized geopolymers and their precursors. Cumulative and differential thermo gravimetric analysis curves of the precursors, kaolinitic and zeolitic tuff, and geopolymers (M-JZ.1–M-JZ.7) are shown in Fig. 4. The TGA curve for kaolinite (JHK) proves that heating to 200 °C decreases the mass of the sample by 2.3% Fig. 4 (A). This decrease reflects removing water adsorbed on the surface. Further 8.4% mass loss related to dehydroxylation of kaolinite between 400 and 650 °C is shown in Fig. 4 (B). Therefore, the amount of kaolinite minerals was calculated to be around 60% (Yousef et al., 2009). Zeolitic tuff (JZT) exhibits a total mass loss of 13% in two steps with heating up to 1000 °C. The TGA curve of zeolitic tuff (JZT) reveals that thermal events occur in two temperature

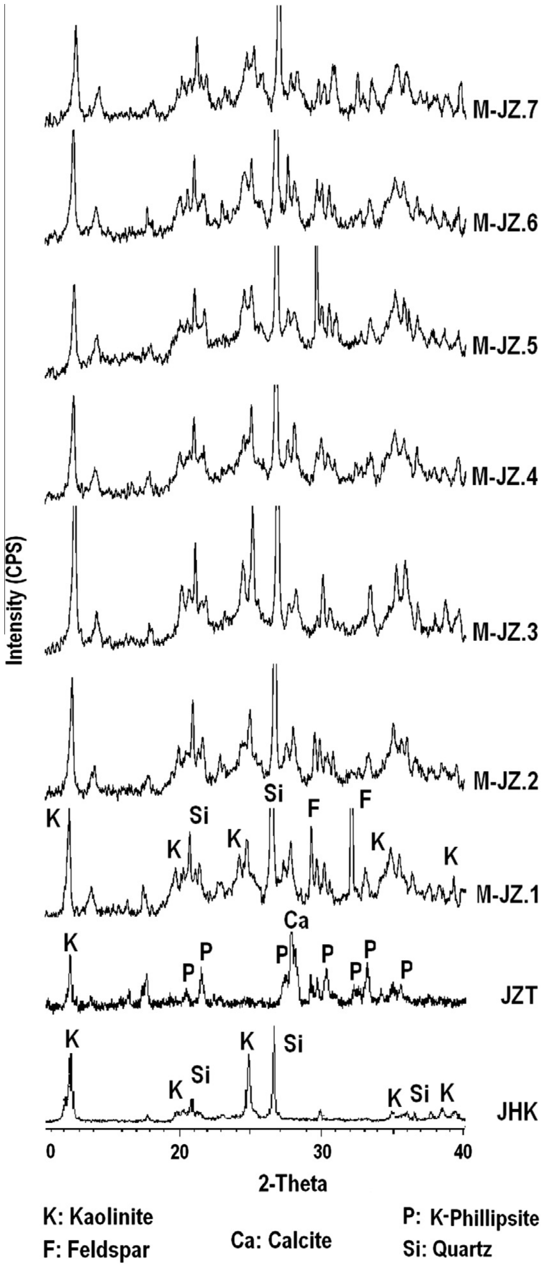

Figure 2 Qualitative X-ray diffraction spectra of the precursors and the geopolymers.

ranges: 25–400 °C and 670–870 °C as shown in Fig. 4 (B). In the first zone, the decrease of weight is around 8% which can be attributed to the release of zeolitic water while the second peak of weight loss between 670 and 870 °C could be a result of the loss of CO_2 from calcite (Frost et al., 2009).

The TGA curves of Fig. 4 show the mass loss percentage of synthesized geopolymers with different water contents (M-JZ.1–M-JZ.7). The total mass losses vary from 9.5% to 10.7% as shown in Fig. 4 (A). The derivatives of the TGA curve, Fig. 4B, show that major mass loss percentages occur in three temperature ranges, from 100 to 300 °C due to the evaporation of chemically bonded water (also called zeolitic water), between 400 and 600 °C mainly from the dehydroxylation of non-reacted kaolinite, and the last temperature range is 670–870 °C, which could be a result of the loss of CO_2 from calcite as mentioned above.

3.2. Mechanical and physical properties of geopolymers

3.2.1. Bulk density and water absorption

As shown in Fig. 5, bulk density of synthesized geopolymers increases slightly from 1.79 g/cm^3, (M-JZ.1), up to 1.83 g/cm^3, (M-JZ.3), with increasing water contents from 24% up to 28%. Parallel to this trend, the water absorption decreases from 16.5% (M-JZ.1) to 13.5% (M-JZ.3). This reduction of water absorption and the increase in bulk density indicate the closure of the open porosity and formation of compacted structure and that the water contents in geopolymers increase up to the optimum value, of 28%.

On the other hand, the bulk density of geopolymers shows a decreasing trend from 1.83 g/cm^3 (M-JZ.3) to 1.72 g/cm^3 (M-JZ.7) with increasing water from 28% up to 36%. Within this range of water contents, the water absorption increased from 13.5% to 18%. These two trends of decreasing bulk density and increasing water absorption indicate the formation of high porosity since the excess water leaves pores after evaporation. Noteworthy, the above results of bulk density and water absorption are in good agreement and show that the optimum water content is 28% of the clay. Using this optimum water content in geopolymers fabrication yields a dense geopolymeric structure and reduces the open pores.

3.2.2. Compressive strength

The compression strengths of the seven series of synthesized geopolymers are shown in Fig. 6. Although they did not show disintegration, the strength of the immersed specimens is mostly below 50% of the strength of the same dry specimens. The M-JZ.3 wet specimens series (water content = 28%) showed the highest strength. Higher or lower water contents lead to a decrease in the strength. This compression index of the geopolymers confirms that the optimum water content value, in a range of 24–36%, to obtain strong geopolymeric structure with high mechanical performance is 28% of the clay fraction.

3.2.3. Skeletal density

Fig. 7 exhibits the variation of the skeletal density of synthesized geopolymers with water content. The results show that the maximum skeletal density, 2.617 g/cm^3, is obtained by using a water content of 28% (M-JZ.3). This is in good agreement with the above mentioned results: bulk density, water absorption, and compression strength.

3.3. Adsorption kinetics

The adsorption capacity of synthesized geopolymers (M-JZ.1–M-JZ.7) and the precursors JHK and JZT toward cadmium ions were investigated. Different kinetic models were used to fit the obtained data of adsorption kinetics of Cd(II) onto geopolymers and raw materials, i.e., pseudo-first order, pseudo-second order, and intraparticle diffusion models. Among the three kinetic models employed, the pseudo-second order model was selected depending on the correlation coefficient values of the models. Thus, the pseudo-second order kinetic model (Eq. (2)) was used to fit the kinetics data:

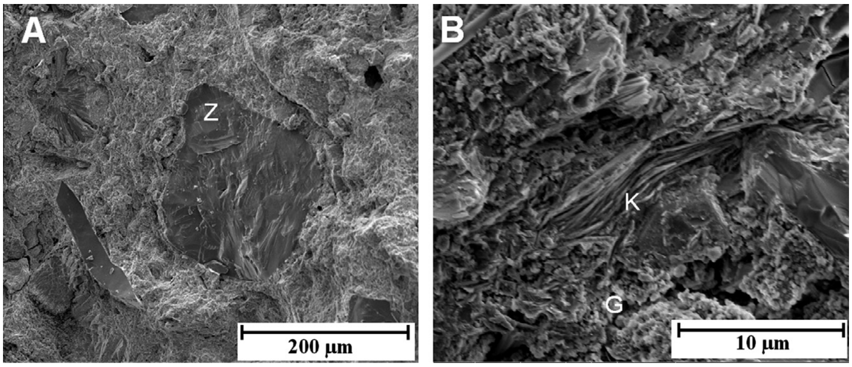

Figure 3 SEM images geopolymers (M-JZ3); (Z: zeolitic tuff, K: kaolinite, G: geopolymer gel).

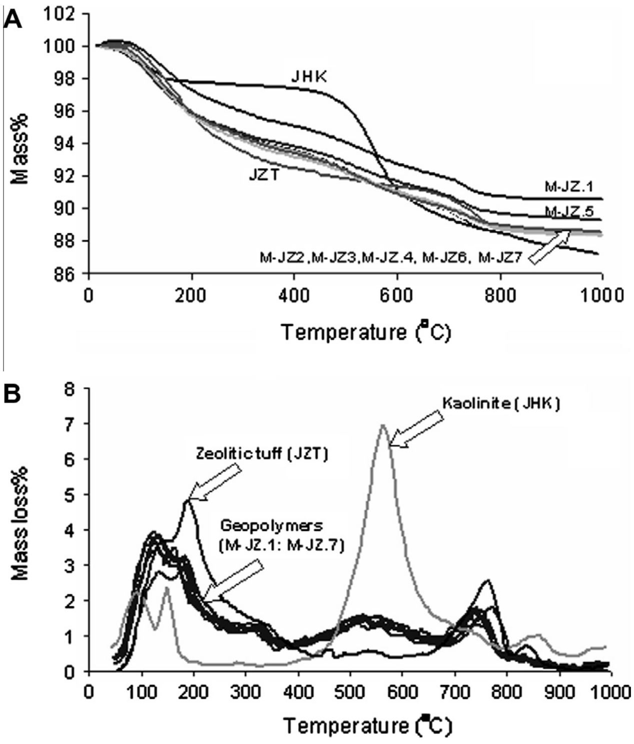

Figure 4 Cumulative (A) and Derivative (B) of the thermal gravimetric analysis of the precursors, kaolinite (JHK) and zeolitic tuff (JZT), and geopolymers (M-JZ.1:M-JZ.7).

$$t/Qt = 1/(kQe2) + t/Qe \quad (2)$$

Where Qe is the amount of Cd(II) adsorbed at equilibrium (mmol Cd(II)/g adsorbent); t is the time (min); Qt is the amount of Cd(II) adsorbed (mmol Cd(II)/g adsorbent) at time t; k is the equilibrium rate constant of pseudo-second order adsorption (g adsorbent/mmol Cd(II). min). By plotting t/Qt versus t, the value of the slope $1/Qe$ and the intercept $1/(kQe2)$ can be used for the determination of k and Qe (Table 3).

Fig. 8 shows that, the amounts of adsorbed Cd(II) onto geopolymers (M-JZ.1–M-JZ.7) are larger than those of kaolinite raw material (JHK). Sample M-JZ.3, which shows the highest mechanical strength and bulk density, revealed the highest adsorption capacity among all investigated samples even higher than raw zeolite (JZT).

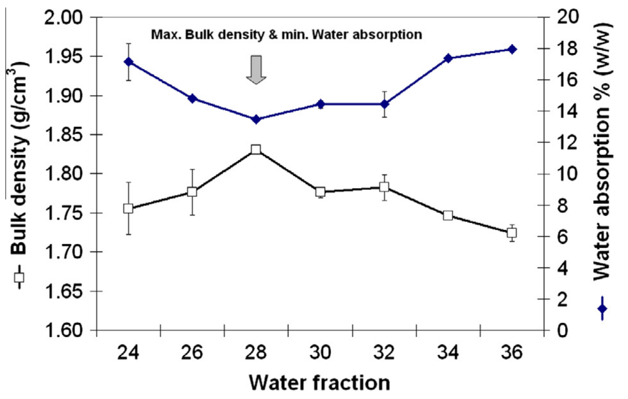

Figure 5 Bulk density and water absorption of the geopolymers as a function of water content.

Table 3 shows that the geopolymer sample M-JZ.3 has the lowest k value among the samples investigated. This is in agreement with the minimum water absorption and maximum bulk density, which means that a more perfect geopolymerization leads to a more closed and denser structure with more diffusion limitations toward the adsorption of Cd(II) ions.

3.4. Water-soluble salts

Efflorescence is caused when soluble salts and other water dispersible materials come to the surface of the partially immersed material. The most usual reaction occurs when residual alkali hydroxides (e.g. sodium hydroxide, potassium hydroxide...) are transported by water to the surface through capillaries in the material. There it combines with carbon dioxide from the air to produce sodium carbonate (depends on the alkali solution) and water. The presence of water-soluble salts in the end products is the major factor of formation of the efflorescence. Therefore, we will explore the presence of these water-soluble salts (i.e. sodium salts) in the material in the following paragraphs.

The Synthesized geopolymers (M-JZ.1–M-JZ.7) exhibit similar trends toward the electrical conductivity of their solutions and the concentration of leached Na^+ as shown in Fig. 9. As the water percentage of clay increases from 24% (M-JZ.1) to 28% (M-JZ.3), both electrical conductivity and leached Na^+ concentration decrease from 1967 to 1459 μS/cm

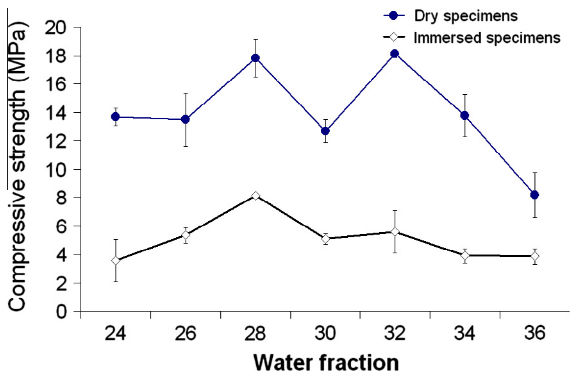

Figure 6 Variation of compressive strength of the geopolymers with water content.

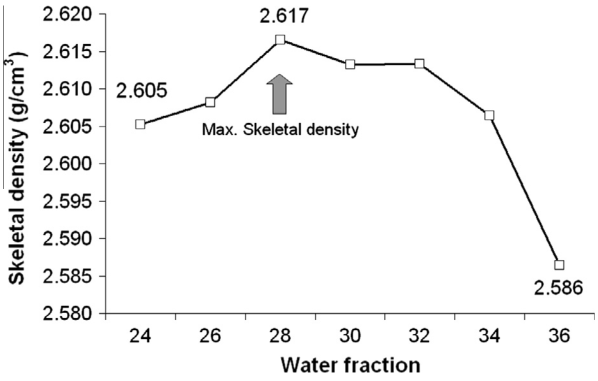

Figure 7 Variation of skeletal density of the geopolymers with water content.

Table 3 The results of *Qe* and *k*2.

Adsorbent	*Qe* (mmol/g adsorbent)	*k* (g/mmol min)
M-JZ.1	0.154	0.022
M-JZ.2	0.207	0.026
M-JZ.3	0.304	0.005
M-JZ.4	0.234	0.047
M-JZ.5	0.175	0.013

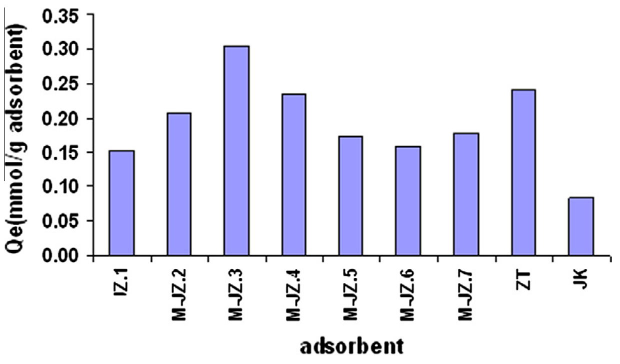

Figure 8 Amount of Cd(II) ion adsorbed at equilibrium(Qe) onto geopolymers (M-JZ.1–MJZ.7) and raw materials (JZT and JHK).

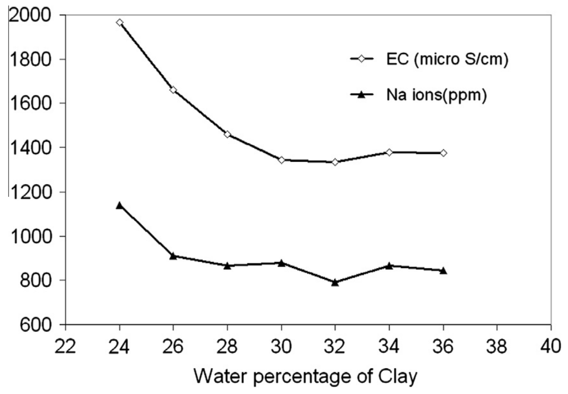

Figure 9 EC and Na^+ concentration of the seven series of geopolymer solutions.

and from 1141 to 876 ppm, respectively. Neither electrical conductivity nor Na^+ concentration shows a remarkable change as the water content ranges from 28% to 36%. Accordingly, it may be concluded that an increase in the water percentage up to 28% of the clay reduces significantly the residual chemicals in the structure of the end geopolymer products.

4. Conclusions

Low cost geopolymeric materials could be produced by using natural kaolinitic and zeolitic tuff as precursors. It was observed that water content plays the major role in the end product properties. Using a water content of 28% increases the strength and skeletal density and decreases water absorption and residual salts of the geopolymer. This water content value increases the adsorption capacity of the end geopolymeric products toward micropollutants (i.e. Cd(II)). The rate of adsorption, however, goes down as a result of forming more compact structures. Accordingly, these geopolymeric products could be used for the construction of water storage containers, water channels/pipes, and low cost macro-scale water filtration systems.

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