Chemical routes to materials



Valorization of aplite in alkali-activated materials

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ABSTRACT

Geopolymers belong to the broad family of alkali-activated materials that are considered to have much smaller carbon dioxide (CO_2) footprint than traditional Portland cements. A wide range of metallurgical wastes are utilized as silicious sources to produce geopolymer components with a prospect of numerous applications in the construction field. At the same time, efforts have been also made to the valorization of aplite rock, which is found to be abundant in Finnvolldalen of Norway and consists mainly of quartz and alkali feldspar, resembling the composition of Na-rich pozzolans. In this framework, the current study focuses on the synthesis of inorganic polymers made of aplite and metakaolin as precursors. In addition, the production of synthetic Na-waterglass is also tested as candidate soluble silica donor in geopolymer systems, through the hydrothermal treatment of aplite. The obtained results confirm that inorganic polymers produced by hydrothermally treated aplite (HTA) and metakaolin (MK) lead to materials with enhanced compressive strength values compared to specimens produced by untreated aplite and MK. At the same time, sodium silicate solution from aplite and commercially available waterglass present similar properties, indicating that aplite can be used as alternative raw material in the production of sodium silicates.

Introduction

Within decades of industrial activities, a huge amount of stored industrial wastes in landfills and untapped side-streams have been accumulated in Europe, constituting an enormous wealth that waits to be unlocked and valorized. Extensive research has been done to investigate the possibility of utilizing a variety of industrial solid residues or wastes [1–5]. However, little research has targeted utilization of natural aluminosilicate type rocks as a source material for geopolymerization, a green technology based on an exothermic heterogeneous chemical reaction between a solid aluminosilicate raw material and an alkali metal

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silicate solution under mild conditions [2, 6–12]. In this point of view, the aplite rock, that is found worldwide, mainly in Japan, Russia, U.S.A, Norway and Finland [13], could be an alternative natural aluminosilicate source to produce high added value materials using the geopolymerization technology.

Aplite is an intrusive rock in which quartz and alkali feldspar (microcline and albite) are the dominant components. Aplite is usually Na-rich and contains SiO_2 and Al_2O_3 which seem to be promising for being utilized in the development of aplite-based geopolymer materials. However, the high SiO_2 aplite content is attributed to crystalline silicious phases with no presence of amorphous silica, indicating limited reactivity of the sample, that prohibits the possibility of further utilization as silicious raw material for geopolymer production. This necessitates the need for further treatment on aplite to liberate reactive Si in the system, promoting the geopolymerization process.

The broad chemistry of raw materials that are used currently in geopolymers, render aplite a promising precursor to be used either as main silicious source (after appropriate treatment to convent the crystalline silica content into amorphous silica) or as secondary raw material in geopolymer production. Recent studies have used aplite as a source material or addition for the synthesis of cementitious [14], ceramics [15] and geopolymers, along with the addition of blast furnace slag and micro silica, activated by potassium and silicate solution-containing systems [16, 17]. However, the addition of an industrial by product, namely a slag, complexes the geopolymer matrices (including potentially heavy metals) preventing the development of a final product with reproducible properties (e.g.,, compressive strength) [18]. Thus, the current study focuses on the selection of aplite as secondary raw material for the development of geopolymer materials with good mechanical properties and decreased environmental impact. At the same time the production of a synthetic sodium silicate solution is also tested after hydrothermal treatment of raw aplite, as an alternative suggestion to the commercial Na-waterglass.

Materials and methods

Laboratory/analytical equipment and reagents

The synthesis of geopolymer pastes was performed using a Hobart mixer under intensive agitation. The fresh pastes were molded in 50 mm cubic molds (3 specimens per mold), sealed with an organic membrane and cured at laboratory furnaces.

The dissolution of aplite in sodium hydroxide solution was studied in a 400–mL Inconel autoclave reactor, connected to a mechanical stirrer with controlled speed. Ceramic Buchner funnels and filter-paper types of MN 640 *d* and MN 615 ¼ were used for solid/liquid separation. Typical borosilicate glassware was used for volumetric measurements and plastic bottles were used for the storage of the solutions.

Reagent grade sodium hydroxide (Merck Chemicals, Darmstadt, Germany, 99% purity) was used as the alkaline activator prepared by dissolving sodium hydroxide pellets in deionized water. Sodium silicate solution (Merck, extra pure, SiO_2 : 26.65 wt%, Na_2O : 8.35 wt%, *d*: 1.35 g/cm³) was also added in the liquid phase of the mixture in selected mix designs.

The particle size distribution (PSD) of aplite was determined by laser diffraction using a Malvern Mastersizer Analyzer.

The XRF analysis of the aplite was performed with a SPECTRO XEPOS ED-XRF Analyzer. Quantitative elemental chemical analysis of wet samples (produced by dissolution of aplite) was performed by Atomic Adsorption Spectroscopy (AAS) and Inductive Couple Plasma Optical Emission Spectroscopy (ICP–OES). In more detail, AAS analysis was performed with a PerkinElmer[™] PinAAcle 900 T Atomic Adsorption Spectrometer. ICP–OES analysis was performed with a PerkinElmer[™] Optima 800 Optical Emission Spectrome-ter.

Crystallographic analysis of aplite was performed in a Malvern-PANalyticalTM X'Pert Pro diffractometer, with CuKa radiation (V = 40 kV k αt I = 30 mA).

Phase identification of aplite was performed with BrukerTM DIFFRAC.EVA software and use of ICDDTM Diffraction databases PDF-4+ και PDF-4 Minerals. Quantitative analysis of the identified phases was performed with the BrukerTM DIFFRAC.TOPAS software.

The compressive strength of produced geopolymers performed by the servo-hydraulic loading machine Advantest 9, after 7 and 28 days of hardening according to ASTM C109 [19]. For compressive strength measurements, three cubic specimens of 50 mm edge were used for each material and the mean value of the three measurements was reported as the final compressive strength of the material. In order to achieve smooth and uniform dimensions appropriate for mechanical performance

Table 1 Particle sizedistribution of aplite	$d_{10}(\mu m)$	d ₅₀ (μm)	$d_{90}(\mu m)$
distribution of upile	0.83	10.34	57.60

evaluation, the measurements took place after cutting the final materials using a band saw.

Materials used

Prior to any analytical technique and synthesis of the geopolymers, aplite was ground to a small particle size in a ball mill with porcelain milling media, to avoid any potential contamination and then dried at 105 °C for at least 24 h. The *d*10, *d*50, and *d*90 values, which are intercepts for 10%, 50%, and 90% of the cumulative mass of aplite after milling, are presented in Table 1.

The raw materials used for the synthesis of geopolymer slurries consist either of solely aplite originated from the mining processing of Namsskogan, Norway, or of aplite and metakaolin (Al₂O₃: 42 wt% and SiO₂: 53.9 wt%) produced in the laboratory after thermal treatment of pure kaolinite from VWR (Bole White) at 750 °C. The chemical and mineralogical analysis of aplite is shown in Table 2 and Fig. 1, respectively.

Subsequently, a quantitative (Rietveld) analysis was also performed and the results are shown in Table 3.

According to the chemical analysis (Table 2), aplite is rich in SiO₂ being equal to 87 wt%, while Al₂O₃ is substantially lower reaching a level up to 6 wt%. CaO, K_2O and Na₂O are the main impurities in aplite. Mineralogical analysis (Fig. 1) of ground aplite confirms that crystalline quartz is the main host mineral of SiO₂ while the remaining amount of silica exists inside the feldspars, more specifically in microcline and albite which are the host minerals of Al₂O₃ in aplite. This is also confirmed by the quantitative analysis of raw aplite (Table 3) revealing that a major part of the material is quartz, followed by secondary phases attributed to albite, and microcline.

Therefore, based on the characterization results, it is concluded that raw aplite rock is not a promising precursor material for alkali activation for two reasons: (a) Silica exists in crystalline quartz which exhibits very

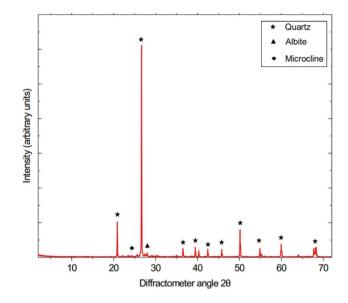


Figure 1 XRD pattern of raw aplite.

Table 3 Quantitative analysis (wt%) of raw aplite

SiO ₂	NaAlSi ₃ O ₈	KAlSi ₃ O ₈
(Quartz)	(Albite)	(Microcline)
77.2	14.8	8.02

limited dissolution in NaOH solutions under atmospheric conditions [20] and: (b) The remaining silica and alumina found in feldspars (microcline and albite) are non–soluble minerals in NaOH solutions, not only at atmospheric conditions but also under elevated temperatures and pressures.

Given the above conditions, grinding of aplite was considered to be a requisite to improve its dissolution ability by increasing substantially its specific surface area and probably deliberating micronized quartz crystals, exhibiting thus superior dissolution capability. The reason why Na–waterglass and metakaolin are used as additives in the mix designs of geopolymer synthesis is the lack of easily dissolved silica and alumina during geopolymerization, as indicated by the physicochemical characterization of aplite (Table 2 and Fig. 1). Metakaolin is an amorphous, easily dissolved material in NaOH

Table 2	Chemical analysis
of raw ap	olite

%wt Equivalent oxides								
$\overline{Al_2O_3}$	CaO	SiO ₂	K ₂ O	Fe ₂ O ₃	Na ₂ O	Others	LOI	Total
6.12	1.41	87.24	1.74	0.94	1.65	0.17	0.15	99.42



solutions, being, therefore, a good precursor for easily dissolved aluminum and silicon in alkali activation [21].

Experimental methodology

Two different routes are followed for the implementation of the experimental work based on the valorization of aplite either as: (a) Raw material/filler to produce geopolymers, or as: (b) A precursor to produce synthetic sodium silicate solution, after hydrothermal treatment of aplite, with direct use in geopolymer production: (a) Aplite as a raw material/filler for geopolymer production: Aplite and/or aplite-metakaolin blend is activated with NaOH and/or sodium silicate solution. Preliminary experiments on using ground aplite powder as the sole precursor for the synthesis of geopolymers are performed according to the following procedure: Ground aplite is mixed with an alkali activator (either 8 M NaOH solution or a doped NaOH solution with Na-waterglass) at a solid to liquid ratio (S/L) in the range 2.5-4.5 g/mL [22]. The selected activator's concentration is equal to 8 M according to published studies, reporting that at lower alkalinity level, the OH⁻ ion amount is inadequate to facilitate the dissolution of silicate and aluminate species that promote polymerization [23]. On the other hand, under higher alkaline conditions the oligomeric silicate species lose their stability in favor of mononuclear silicate species [6, 24]. The resulting paste is casted in cubic molds (triple samples) and cured at 70 °C under atmospheric pressure for several days (7-10 days). A second experimental series is also performed, where part of aplite is substituted with metakaolin to boost the paste in dissolved silica (e.g.,, 70A-30MK denotes that the solid precursor is composed by 70 wt% aplite and 30 wt% metakaolin) and the solid to liquid ratio is 1.1 g/mL. The curing temperature is increased to 80 °C for 3 days and then the materials are demolded and further cured at ambient temperature (4 or 25 days) for compressive strength measurements. The experimental conditions are summarized in Table 4, where five different mix designs are presented. (b) Hydrothermal treatment of aplite for synthetic waterglass production with direct use in geopolymer synthesis: A new series of experiments is performed using aplite at autoclaved conditions to examine the option of producing synthetic sodium silicate solution that could be directly used (instead of commercial waterglass) for the production of geopolymers. The selected system to study is the one where the wt% substitution of

Table 4	Mix designs	(MD), at at	mospheric	conditions
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Mix design (wt%)	Temperature (°C)	Activating solution
MD1: 90A-10MK	70 ^a	8 M NaOH
MD2: 70A-30MK	70 ^a	8 M NaOH
MD3: 50A-50MK	80 ^b	8 M NaOH
MD4: 50A-50MK	80 ^b	$6.7 \text{ M} (\text{NaOH} + \text{WG})^{c}$
MD5: 30A-70MK	80 ^b	$6.7 \text{ M} (\text{NaOH} + \text{WG})^{\text{c}}$

At ambient temperature: a) 4 days (early) and b) 25 days (long) compressive strength measurements; ^a70 °C: 7–10 days; ^b80 °C: 3 days. ^cNaOH: 30% *w/w* and WG: 70% *w/w*

Table 5 Mix design at autoclaved conditions

Mix design (wt%)	Temperature (°C)	Activating solution		
MD6: 50HTA-50MK	200	8 M NaOH		
Treated Aplite and MK: Metakaolin				

HTA, Hydrothermally

aplite with metakaolin is 50% (50A-50MK). The whole amount of aplite needed for the paste preparation is added in an Inconel autoclave (detailed description at chapter 2.1) at a solid to liquid ratio of 0.5 g/mL with 314 mL of 8 M NaOH solution, heated in the temperature of 200 °C for 2 h at a pressure of 15 bars. After the end of the hydrothermal treatment, a small amount of liquor is withdrawn, filtered and sent for dissolved Si chemical analysis. Then the pulp is mixed thoroughly with the proper amount of metakaolin to have the 50 wt% substitution of aplite, forming a paste with a solid to liquid ratio of 1.1 g/mL (this ratio has been calculated by adding the initial aplite mass that was fed to the autoclave with the remaining mass of aplite and the mass of added metakaolin to form the 50A-50MK solid precursor). The pastes are casted and cured for 3 days at 80 °C and then remain additionally 4–25 days at ambient temperature before the measurement of compressive strength. The experimental conditions are summarized in Table 5.

Results and discussion

Valorization of aplite as raw material/filler for geopolymers production

According to the preliminary experiments performed using aplite as the main raw material to produce

geopolymer specimens, the pastes failed to set under any of the studied conditions even if high curing temperature (70 °C) and prolonged curing time (up to a week) were applied. The results indicate and support the lack of minerals dissolution (quartz, microcline and albite) from aplite under the mild activation conditions applied in laboratory. Considering that in a typical geopolymer paste the molar ratio SiO₂/Na₂O should be in the range of 3.6–5 (w/w) and the molar ratio SiO₂/ Al_2O_3 in the range of 3.5–4.5 (w/w) [22], the absence of any significant dissolution, principally of quartz and secondarily of (Na, K, Ca) feldspars in NaOH activator inhibit the setting procedure of the paste. Even in the case of doping the activating solution with Nawaterglass that has a SiO₂/Na₂O molar ratio 3.3 (w/w), the absence of any significant quartz dissolution keeps the SiO₂/Na₂O molar ratio lower than the value of 3.5 (w/w) and thus hinders the setting of the paste.

To overcome the setting difficulty observed above, the strategy of doping aplite with metakaolin was adopted in the second series of tests (Table 4), where part of aplite was substituted by metakaolin. In this experimental series, metakaolin works as an active precursor and thus donor of dissolved aluminum and silicon ions to the geopolymer network, while aplite behaves as a filler. Five different mix designs are examined at a substitution ratio of MK–A in the range of 10–50 wt%. The selected alkaline activator is either NaOH or NaOH with sodium silicate solution. The compressive strength values of the produced specimens are presented in Fig. 2.

It is seen from the results that the substitution of aplite with metakaolin to an amount as high as 30 wt% (MD1 & MD2) leads to pastes that cannot set. Higher substitution to 50 wt% (MD3), results in pastes that set within the timeframe of 3 days with compressive strength after 7 and 28 days 2.2-2.6 MPa, respectively. This can be explained by the molar ratios of produced pastes compared to the typical molar ratios encountered in geopolymer pastes, according to the literature [22]. According to calculations (Table 6), the molar ratios of the pastes in MD1 and MD2 are far away from the corresponding typical MR values. The pastes are deficient in silica and alumina as the Na₂O/SiO₂ (3.71 (w/w) for MD1 and 1.24 (w/w) for MD2) and Na₂O/ Al_2O_3 (8.10 (*w/w*) for MD1 and 2.70 (*w/w*) for MD2) molar ratios are extremely higher than the corresponding typical values in geopolymer pastes (Na₂O/SiO₂: 0.2-0.28 (w/w) and Na₂O/Al₂O₃: 0.8-1.2 (w/w)). The molar ratios in the paste from MD3 that set are closer

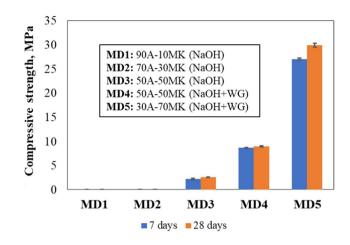


Figure 2 Compressive strength values of mix designs after 7 and 28 days of hardening

 Table 6
 Molar ratios in pastes

MR values $SiO_2/Al_2O_3(w/w)$		Na_2O/Al_2O_3 (w/w)	Na ₂ O/SiO ₂ (w/w)	
Geo-pastes	3.5–4.5	0.8–1.2	0.2–0.28	
MD1	2.18	8.10	3.71	
MD2	2.18	2.70	1.24	
MD3	2.18	1.62	0.74	
MD4	4.34	1.16	0.27	
MD5	3.59	0.85	0.24	

to the typical values although are still far from them $(Na_2O/SiO_2: 0.74 \ (w/w)$ and $Na_2O/Al_2O_3: 1.62 \ (w/w))$. Metakaolin is not enough to offer to the paste soluble silica and alumina. It helps the system to start polymerization and give solidified compact materials with low compressive strength but the SiO_2/Al_2O_3 molar ratio of 2.18 (w/w) will be always lower than the typical value of 3.5–4.5 (w/w) (almost half the typical value) indicating that the paste must be doped with higher amount of soluble silica.

If the system is dopped with soluble silica by using Na-waterglass (MD4), the molar ratios will be within the typical values and the compressive strength of material will almost quadruple (Fig. 2) reaching a value of 8.7 MPa at 7 days and 9 MPa after 28 days. The molar ratios SiO_2/Al_2O_3 : 4.34 (*w/w*) and Na_2O/Al_2O_3 :1.16 (*w/w*) for MD4 are very close to the highest limit of the typical molar ratio values (SiO_2/Al_2O_3 : 0.8–1.2 (*w/w*)) indicating that doping with soluble alumina is necessary to



improve further the properties of geopolymer materials. To test this observation, a new experiment with 30 wt% aplite and 70 wt% metakaolin in the presence of Na-waterglass in the activator (MD5) was performed. Alumina doping took place through the increased amount of metakaolin in the mixture of solid precursors. The molar ratios SiO_2/Al_2O_3 : 3.59 (*w*/*w*) and Na_2O/Al_2O_3 : 0.85 (*w*/*w*) in this test are located closer to the lower limits of the typical molar ratio values indicating a substantial doping with soluble alumina. Under those conditions the mechanical strength of produced material improved substantially reaching a value of 27 MPa at 7 days followed by 30 MPa after 28 days, revealing the substantial role that soluble aluminum plays in the geopolymer network of material.

The produced materials coming out by MD3, MD4 and MD5 experiments (with successful setting) are depicted in Fig. 3.

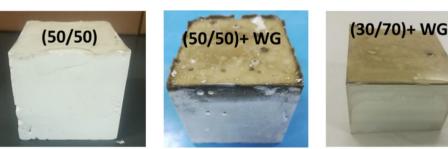
It is evident that doping of aplite with metakaolin as well as Na-waterglass, results in well-shaped and structured materials with appreciable mechanical properties (Fig. 2). This type of doping is not the most cost-effective option (metakaolin price: 300–400 \$/t, Na–waterglass: 700–900 \$/t in solid state) and thus, alternative solutions must be found to render the aplite-based geopolymers less costly and thus more attractive.

Hydrothermal treatment of aplite for synthetic waterglass production with direct use in geopolymers

To decrease or even eliminate the doping with Nawaterglass, silicon must be transferred to the paste by the aplite dissolution in NaOH solutions. To achieve this, the hydrothermal treatment of aplite in autoclave is necessary as it has been confirmed by literature [25]. At 200 °C and under the given conditions (Table 5) the hydrothermal treatment of aplite is performed, resulting in a viscous aqueous phase with dissolved silicon content of 146 g/L. By assuming that the density of the liquor after the hydrothermal treatment has a value of 1.35 g/mL (which is a common density for a Na-waterglass solution), then the silica content of the aplite waterglass reaches the value of 23.2 wt%. In any case, this measurement indicates that the hydrothermal treatment of ground aplite can offer to the system appreciable amount of dissolved silica, forming a type of synthetic waterglass that has the potential to completely substitute the commercial one. The compressive strength of 13 MPa achieved after 7 days followed by 15 MPa after 28 days, is directly comparable and higher with the one of 8.7 MPa achieved with the addition of Na-waterglass (MD4 in Table 4) and again dictates that hydrothermally treated aplite has the potential to act as a substitute of Na-waterglass. The experiment MD6 (50HTA-50MK) in Table 5 and MD4 (50A–50MK + WG) in Table 4 have similar molar ratios and for this reason give enhanced values of compressive strengths. The physical appearance of the material MD6: 50HTA–50MK is shown in Fig. 4. The material is compact and very well shaped and structured.

The comparison of 28 days compressive strength of MD6: 50HTA–50MK (15 MPa) with the respective mix formulation of 50A-50MK (2.6 MPa) shows that the hydrothermal treatment of ground aplite improves the strength of material at almost 6 times at the expense of the cost of the hydrothermal treatment in autoclave. In the formulation 50HTA–50MK the value 1.62 (w/w) of the molar ratio Na₂O/Al₂O₃ indicates that the system is deficient in Al₂O₃ (as this molar ratio Na₂O/Al₂O₃ value in typical geopolymers in in the range of 0.8–1.2 (w/w)) and thus must be doped with easily dissolved Al_2O_3 to improve the geopolymerization performance. To make such an adjustment in the composition of the paste is a difficult issue, as a good donor of Al₂O₃ must be sought that its addition to this system wouldn't affect substantially the Na₂O and SiO₂ content. This

Figure 3 Aplite-Metakaolin materials of different ratios: **a** 50A–50MK, **b** 50A–50MK with WG & **c** 30A–70MK with WG.





(b)

(c)



Figure 4 Aplite-Metakaolin materials of mix design 50A–50MK after autoclave treatment.

 Table 7 Specifications of commercial sodium silicate solution

 compared to synthetic aplite waterglass (WG)

WG type	Na ₂ O (wt%)	SiO ₂ (wt%)	Si (g/L)
Commercial WG	8.35	26.65	167.5
Synthetic aplite WG	8.88	23.20	146

type of modification was not investigated within the frame of this work, but it is something that could be done as a future step in the aplite hydrothermal treatment optimization. On the other hand, the hydrothermal treatment of ground aplite leads to a synthetic aplite Na-waterglass as indicated by the measured Si content, reaching the value of 146 g/L (~70% Si extraction based on raw aplite chemical analysis). The pH of sodium water glass that was used for the synthesis of the fresh mortars is equal to 11.8. The sodium silicate solution that was produced after the hydrothermal treatment of aplite reaches a pH value equal to 12.3. Thus, the treated aplite leads to a synthetic waterglass, possessing almost similar properties with the commercial one, as confirmed by Table 7.

Conclusions

In the current work, aplite was tested as raw material for the production of geopolymers and as a candidate soluble silica donor in geopolymer systems after hydrothermal treatment. Using solely ground aplite as a solid precursor and NaOH solution as alkaline activator, results in unsuccessful setting of pastes and this approach has no potential for geopolymer production. Using a 50/50 mixture of ground aplite with metakaolin as a solid precursor and NaOH solution as alkaline activator (MD3), results in successful setting of pastes and in production of geopolymers with compressive strength of 2.2–2.6 MPa at 7 days and 28 days, respectively. Using a 50/50 mixture of ground aplite with metakaolin as a solid precursor and NaOH solution doped with Na-waterglass (as soluble silica and alumina donor) as alkaline activator (MD4), results in successful setting of pastes and in production of geopolymers with improved compressive strength of 8.7–9 MPa at 7 days and 28 days, respectively. By applying higher aplite substitution with metakaolin and doping with waterglass (MD5), an excellent material was produced that had 27 MPa compressive strength at 7 days and 30 MPa at 28 days. This test elucidates the need for doping aplite with enough soluble aluminum and silica. The doping with silica can also be achieved directly by performing hydrothermal treatment of aplite at elevated temperatures to produce a synthetic Na-waterglass. The doping with aluminum needs a proper donor, probably fine alumina trihydrate.

Using a 50/50 mixture of hydrothermally treated ground aplite with metakaolin as a solid precursor and NaOH solution as alkaline activator (MD6), results in successful setting of pastes and in production of geopolymers with improved compressive strength of 13 MPa at 7 days and 15 MPa at 28 days (compared to the same recipe MD4 where Na-waterglass was added as soluble silica donor with compressive strength values of 8.7–9 MPa). The hydrothermal treatment of ground aplite (200 °C, 2 h, 8 M NaOH) in autoclave, leads to a synthetic Na-waterglass with characteristics very similar to the commercial Na-waterglass. As a conclusion, the most promising formulation is the 50HTA-50MK in NaOH activator that gave 13-15 MPa compressive strength at 7–28 days. This formulation needs optimization toward: (a) the improvement of the paste composition (e.g., by doping with dissolved alumina) that would potentially increase the mechanical strength and (b) the decrease in the cost of materials (e.g., by optimization of the hydrothermal treatment of aplite, substitution of metakaolin partially or totally with silica fumes, and/or kaolin).



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Author contributions

Georgia Maria Tsaousi and Georgia Flesoura designed and performed the experiments under the supervision of Professor Dimitrios Panias; Georgia Maria Tsaousi analyzed the data and wrote the paper. All authors read and approved the final manuscript.

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Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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