Synthesis, Thermal properties and electrical conductivity of Na-Sialate geopolymer

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Abstract

In this work, the central focus is upon the synthesis of new Na-Sialate geopolymer concerning as ionic conductive material for electric and electrochemical devices. The studied material was prepared at ambient temperature by a mixture of metakaolin and sodium hydroxide.

In order to study the change in gel structure and phase composition at each heating step. The synthetic compound was characterized by several techniques.

The phase identification of Na-Sialate geopolymer, was carried out by powders X-ray diffraction, showed an amorphous character at ambient temperature. As well as minor amounts of high temperature crystalline phases were appeared which were: zeolite (300-600°C), Nepheline (NaAlSiO_4) (900-1150° C). Above 1200°C, the structural reorganization occurs due to the phase crystallization of corundum ($\alpha\text{-Al}_2\text{O}_3$). The dissolution of the grains by the liquid phase formed induces the conversion of the structure of the material from sialate $[-\text{Si-O-Al-O}]$ to sialate siloxo $[-\text{Si-O-Al-O-Si-O-}]$. These results were confirmed by Fourier transform infrared spectroscopy (FT-IR).

The thermal analysis offered a thermal stability of Na-geopolymer up to 1400°C.

The electrical measurements were applied to investigate Na^+ ions conductivity of Na-geopolymer. The results showed that Na-Sialate geopolymer materials exhibit high ionic conductivity of approximately $5.10^{-5} \text{ S.cm}^{-1}$ in air, at room temperature.

Introduction

Geopolymer is an inorganic polymer. The materials are generally produced from clays activated by an alkali or an alkali-silicate solution [1–3]. When treated below 100°C, they are generally X-ray amorphous and consist in cross-linked AlO_4 and SiO_4 tetrahedral. The arrangement of the silicon and aluminum tetrahedral into a three-dimensional network structure generates cavities in which water molecules and monovalent cations (type Na^+ and/or K^+) will be trapped.

The empirical formula proposed is: $\text{M}_n((-\text{SiO}_2)_z\text{-AlO}_2)_n \cdot w\text{H}_2\text{O}$ where M is a positive cation, such as sodium or potassium, z is 1, 2 or 3, n is the degree of polycondensation, and w is the mole number of hydrated water (usually up to 7) [1].

The polysialates are based on aluminosilicate units such as sialate $[-\text{Si-O-Al-O}]$ (PS), sialatesiloxo $[-\text{Si-O-Al-O-Si-O-}]$ (PSS) or sialate disiloxo $[-\text{Si-O-Al-O-Si-O-Si-O-}]$ (PSDS).

The physical evolution of materials during heating is a critical factor in determining their suitability and performance for applications ranging from construction to refractories and adhesives.

Previous works on the effect of high temperatures on geopolymers have already examined phase changes using XRD and NMR [4–9].

Several researchers have focused on the use of geopolymer in high temperature environments for targeted industrial applications [1–4]. Nowadays, a novel potential application for geopolymers is to use those materials as precursors for the production of ceramics [5–6]. Different charge balancing cations (Na, K and Cs) were investigated. It was shown that the Si/Al ratio and the heating rate have an influence on the physical and structural evolution, the onset temperature of crystallization, and the phase stability of alkali-based geopolymers [8–9, 13]. Previous research comparing different Al/Si ratios has shown that a ratio of 2 results in relatively high strength geopolymers [10–13].

Likewise, alkali-activated geopolymers are known as high ionic conductors with a conductivity value at about $1.5 \times 10^{-6} \text{ S.cm}^{-1}$ in air at room temperature [6].

In the present work, the structural characterizations, thermal evolution and ionic conductivity of new Na-Sialate geopolymer have been investigated by different experimental studies.

Materials And Experimental Techniques

2.1. Geopolymer preparation

Na-geopolymer was synthesized from commercial kaolin calcined at 700°C for 6 h to obtain the activation phase (metakaolin), and sodium hydroxide solution, which the molar ratios

Si/Al = 1 and Na/Al = 1. The chosen ratio of H₂O/NaOH of the sodium hydroxide solution was 10. The present work was carried out using a previously composition found to prepare the formulation of Na-PS [7–8].

From the first minutes, a paste was obtained and evolved chemically and rheologically very quickly. This paste was composed from gel solution and interstitial liquid. After several hours, it would harden to form an amorphous solid.

The specimen was kept at room temperature for 7 days, and then calcined at different temperatures.

2.2. Experimental techniques

2.2.1. X-ray powder diffraction

The phase identification and the crystal structure of Na-geopolymer were performed by X-ray diffractometry (BRUKER-AXS-D8-Advance) with CoK α radiation ($\lambda = 0.178 \text{ nm}$) and a scanning rate of 2°/min from 10° to 60° (2 θ).

2.2.2. IR spectroscopy

The IR measurements, obtained using a Perkin Elmer 1600 FT spectrometer, were carried out at ambient temperature between 400 and 4000 cm^{-1} .

2.2.3. Thermal analysis

The thermal behaviors of geopolymer were characterized by differential thermal analysis (DTA). This analysis was performed by Instrument: SDT Q600 V8.1 Build 99, the temperature was raised from room temperature to 1400°C at a heating rate of 10°C/min under dry air flow (100 mL.min⁻¹) with α-Al₂O₃ as reference.

2.2.4. Electrical conductivity

Electrical conductivity measurements were performed on geopolymer pellet sintered at 300°C. Specimen pellet was produced with a diameter ≈ 5 mm and thickness ≈ 2.4 mm. Thin platinum films (~10 nm) were deposited on each face of the pellet by radiofrequency sputtering using a Sputter Coater Polaron SC7620 apparatus. The impedance spectra were recorded over the frequency range from 1MHz to 1Hz every 50°C from 75°C to 725°C, under dry air flow, using a Schlumberger Solartron 1260 frequency response analyzer connected to a Schlumberger Solartron 1296 dielectric interface. An ac voltage of 350 mV was used, and a waiting time of 20 min was necessary to reach thermal equilibrium after each 50°C heating or cooling step.

Equivalent circuits modeling was used to analyze the impedance spectra and the corresponding parameters were obtained by complex nonlinear least squares (CNLLSQ) fitting [14] performed on commercial software (Zview) [14].

Results And Discussion

3.1. Characterization of Raw Material

3.1.1. X-ray powder diffraction

The chemical composition of kaolin was determined with Thermo Nitron FXL (Table 1).

Table 1 Chemical composition of kaolin (% of mass).						
SiO ₂	Al ₂ O ₃	FeO ₃	MgO	Na ₂ O	CaO	K ₂ O
49.17	44.05	0.69	0.27	1.43	0.81	2.13

The XRD pattern of kaolin (Fig. 1) was analyzed by Le Bail method, without atoms, using the pattern matching routine of the FULPROF program [15]. The profile refinement results indicate that the diffraction pattern corresponds to a mixture of two phases: Kaolinite (C 1; a = 5.055(1) Å, b = 8.984(5) Å, c = 7.376(4) Å, α = 91.730(6)°, β = 104.92(3)°, γ = 89.67(4)°, and Muscovite (C 2/c; a = 5.180(2) Å, b = 8.932(3) Å, c = 20.034(3) Å, α = 90.0°, β = 95.14(8)° and γ = 90.0°). Semi-quantitative analysis using the RIR (reference intensity ratio) method shows that the commercial kaolin is a mixture of 94% of kaolinite and 6% of Muscovite.

3.1.2. IR Spectroscopy

- Crystal structures

Kaolinite with general formula: $\text{Al}_2(\text{OH})_4\text{Si}_2\text{O}_5$ is 1:1 type layer silicates and consists of a two-layer arrangement of tetrahedra and octahedra sheets. The tetrahedral layer is composed of SiO_4 linked in a hexagonal array. The bases of the tetrahedra are approximately coplanar and the apical oxygen atoms are linked to a second layer containing aluminum ions and OH groups. The aluminium ions located in the center of octahedra are surrounded by 4(O) and 2(OH) [16].

A major component of the separation force is the hydrogen-bonding force between hydroxyl groups on octahedral sites and oxygen atoms of SiO_4 tetrahedra, as van der Waals force among the atoms.

- Unit cell OH stretching vibrations

The identification of proton positions in the structure of layer silicate are important to interpret infrared spectroscopy data of kaolin.

The kaolinite contains four (OH) groups in the primitive unit cell. The observed band at 3620 cm^{-1} can be assigned to the (OH) stretching vibration of one hydroxyl group which lies within the layers and is nearly parallel to the layer surface, a (001) plane [17–18]. The other three (OH) groups, lie on the upper surface of the layers, and are oriented at angles of $60\text{--}73^\circ$ to the (001) plane [17–18]. The three hydroxyl groups couple to give a strong in-phase symmetric stretch, observed, in our case, at 3691 cm^{-1} and two weak out-of-phase vibrations at 3669 cm^{-1} and 3652 cm^{-1} [19]

The IR spectrum of kaolin (Fig. 2) exhibits additional bands at $3677, 3727, 3730, 3740\text{ cm}^{-1}$ in the (OH) stretching region that are not reported at the previous research works. These extra bands were observed in the Raman spectrum of kaolinite at 3686 and 3710 cm^{-1} [18], and assigned to transverse optical modes involving in-phase coupled vibrations of the layer-surface hydroxyl groups. Farmer suggested that the corresponding IR bands have transition moments nearly perpendicular to the layer surface, and appear in the higher frequencies range of the longitudinal optical modes of macroscopic crystals.

- Vibrations below 1200 cm^{-1}

The Infrared vibration bands of kaolin below 1200 cm^{-1} displayed in Fig. 3, represent cooperative motions of structural ionic groups. The IR vibration bands may divided into vibrational, rotational and translational motions of anion-cation associations. Although, the proportion of each factor in the production of vibrations is difficult to evaluate at the present time. That is why the detailed vibrational assignments of bands in this region are controversial. To reveal the general characteristics of these network vibrations we have adapted assignments that present a general agreement.

The moderately strong broad band at 1033 cm^{-1} and the strong sharp band at 1004 cm^{-1} correlated to the antisymmetric and symmetric stretching modes of Si-O-Si, respectively [20]. A medium band appeared at 1117 cm^{-1} correspond to the apical Si-O vibrations [20–22]. The weak bands at 940 and 912 cm^{-1} assigned to the deformation modes of inner and inner-surface of $\text{Al}_2\text{O-H}$ [21–22].

The domain observed from 800 to 400 cm^{-1} is the skeleton vibrational modes region. There is a general agreement that the medium band observed at 428 cm^{-1} , the weak band at 650 cm^{-1} and the strong bands at 460 and 528 cm^{-1} belong to Si-O-Si deformation banding modes [21–22].

Three medium bands appeared at 688 , 752 and 795 cm^{-1} were assigned to the deformation modes of $\text{Al}^{\text{VI}}\text{-O-Si}$ bands [21–22].

A weak band near of 413 cm^{-1} ascribed to out-of-plane OH librations [23].

3.2. Characterization of Na-geopolymer:

3.2.1. X-ray diffraction:

The XRD diagrams of metakaolin, geopolymer at ambient temperature and geopolymer heated for 1 h at 300 , 600 and 900 are shown in Fig. 4.

The metakaolin comes from the calcination at 700°C of the kaolin. Metakaolin presents a relatively amorphous XRD diagram with noticeable crystalline peaks relative to the muscovite phase. This is characteristic of the potentially reactive phase in geopolymerization.

The XRD pattern of geopolymer showed the typical amorphous structure of metakaolin. In addition, the characteristic diffraction peaks of muscovite remained after geopolymerization, which suggests that muscovite did not participate in the geopolymerization reaction. While, the amorphous structure of metakaolin transformed from one structure to another structure.

A broad peak characteristic of amorphous structure with two peaks corresponding to muscovite and zeolite phases were observed at 300°C and persisted until 600°C .

After treatment at 900°C , muscovite crystalline phase remained stable, while the zeolite disappeared (Fig. 5).

After heating at 950°C , Na-geopolymer transformed to crystalline material consisting predominantly in nepheline (NaAlSiO_4) with a few low intensities, minor peaks are ascribable to the muscovite crystalline phase. On heating at 1100°C , the amount of the amorphous phase increases with the decrease of crystalline peaks intensities of the nepheline phase. The relative decrease should be ascribed to the appearance of the liquid phase in the matrix. The liquid will affect crystalline phases by partial dissolution (Fig. 6).

At 1200°C, diffraction lines corresponding to corundum ($\alpha\text{-Al}_2\text{O}_3$) are superimposed on the broad amorphous background feature (Fig. 6). These indicate that the dissolution of grains by liquid phase induces the conversion of the structure of the material from sialate $[\text{-Si-O-Al-O}]$ to sialate siloxo $[\text{-Si-O-Al-O-Si-O-}]$ and the formation of new crystalline phase ($\alpha\text{-Al}_2\text{O}_3$). This development of sialate to sialate-siloxo was confirmed by IR Spectroscopy.

3.2.2. IR Spectroscopy:

Figure 7 illustrates the infrared spectra of kaolin, metakaolin and geopolymer treated at different temperatures for 1 h.

The IR spectrum of kaolin showed a dihydroxylation phenomenon during its transformation to metakaolin correlated by the appearance of peaks near of 3650 cm^{-1} .

On the IR spectrum of metakaolin, a peak appeared at 1037 cm^{-1} corresponds to a large concentration of Si-O bonds. Two infrared bands are located at 569 cm^{-1} and 648 cm^{-1} assigned to Si-O-Al stretching vibrations; a peak at 439 cm^{-1} arises from Si-O-Si bending vibration. Indeed, an IR band observed at 806 cm^{-1} is assigned to the Al-O bending mode of $[\text{AlO}_6]$ octahedra [20–21].

Due to the presence of water molecules in geopolymer, the strong characteristic peaks at approximately 3331 cm^{-1} and 1631 cm^{-1} were attributed to stretching and bending vibrations of hydroxyl groups, respectively [22, 23]. After geopolymerization, the chemical environment around regular arranged chain structures of the Si-O bond altered, along with the formation of Al-O-Si bonds. Subsequently, the strong asymmetrical stretching vibration peak of the Si-O bond on the IR spectrum of metakaolin (1037 cm^{-1}) shifted to a lower wavenumber (960 cm^{-1}). This indicated that the solidification process of the geopolymer is a chemical reaction, according to a generation of new substance.

Table 2
The wavenumber of kaolin, metakaolin and geopolymer treated at different temperature

Kaolin	Métakaolin	Geopolymer	100°C	300°C	900°C	1100°C	1200°C
3740	-	3331	3344	3347	-	-	-
3730	-	1631	1647	1654	-	-	-
3727	-	-	1564	1549	1367	-	-
3677	-	-	1379	1371	-	-	-
-	-	-	-	1221	-	970	-
-	-	-	-	-	975	-	991
1117	1037	-	-	-	-	-	846
1033	-	960	962	968	-	-	-
1004	-	843	853	848	708	682	-
940	806	-	-	-	-	654	699
912	-	-	716	729	-	556	636
-	-	697	-	-	555	465	574
795	648	545	-	-	456	-	442
752	569	470	558	572	-	-	-
688	-	421	462	467	-	-	-
650	439	-	-	-	-	-	-
528	-	-	-	-	-	-	-
460	-	-	-	-	-	-	-
413	-	-	-	-	-	-	-

The principal new band appeared at 960 cm^{-1} is assigned to the asymmetric stretching vibration of Si–O–T band links in the geopolymer frameworks (T:Si or Al in tetrahedral coordination). This band is known to be sensitive to connectivity and Si/Al ratio [24–25], and in this case, it is observed at a wavenumber consistent with the presence of predominantly Si–O–Al bonds, which agrees well with the stoichiometry showed in previously studied systems [24–25].

It was also found that the broad and strong peak observed at approximately 802 cm^{-1} ; which belongs to the stretching vibration of hexa-coordinate $\text{Al}^{(\text{VI})}\text{-O}$ in metakaolin; almost disappeared after geopolymerization.

A new peak located at 697 cm^{-1} corresponds to the bending vibration of tetra-coordinated $\text{Al}^{(\text{IV})}\text{-O-Si}$ in a cyclic structure emerged on the FT-IR spectra of the geopolymers. This phenomenon signified the

formation of aluminosilicate networks with the transition from hexa-coordinated $\text{Al}^{(\text{VI})}$ to tetra-coordinated $\text{Al}^{(\text{IV})}$ during the geopolymerization process, as observed by Sitarz et al. [26].

After heating at 100°C, the geopolymer produces a marked decrease in the water bands (Fig. 7).

Apart from this obvious change in the sample hydration on heating to 1100°C, small changes also occur in the Al-Si-O region, especially the development of the broad zeolites band at about 725 cm^{-1} (see Figure.7, T = 300 and 600°C, consistent with the XRD results).

In summary, all the results indicate that the removal of hydration water from a well-treated Na-PSS1 polymer brings about no significant change to the structure. Furthermore, the structure displays a high degree of thermal stability at higher temperatures, retaining its X-ray amorphous character and the atomic environment of its constituents, Al, Si and Na components.

Finally, on the spectrum of 1200°C a double band observed at 635 and 592 cm^{-1} (Fig. 7) relative to the presence of crystalline corundum ($\alpha\text{-Al}_2\text{O}_3$) phase [27] (already detected through XRD). It supports a hypothesis of the conversion of the structure of the material from sialate $[\text{Si-O-Al-O}]$ to sialate-siloxo $[\text{Si-O-Al-O-Si-O-}]$ according to the appearance of $\alpha\text{-Al}_2\text{O}_3$ crystalline phase. The principal band at 960 cm^{-1} characteristic of sialate shifted to a lower wavenumber (846 cm^{-1}). The principal new band at 846 cm^{-1} is consistent with the presence of sialate-siloxo [24–25].

3.2.3. Thermal analysis by TG-DTA

The thermal analysis curves TG and DTA of Na-geopolymer are shown in Figure 8.

Below 250°C, the TG curve indicates that the sample present about 14% of mass loss related to the evaporation of free water (~70%). This phenomenon is well evidenced on the DTA thermogram by the presence endothermic peaks appearing from ambient temperature until approximately 250°C with a minimum values occurring at 50°C and 150°C.

The remainder is either more tightly bound or less able to diffuse to the surface, and continues to evolve gradually up to about 600°C.

Between 250 and 550°C, an endothermic reaction occurring on the material, caused by the dihydroxylation of the octahedral sheet (constitutive water) of muscovite. On the TG curve, the hydroxyl groups of the tetrahedral sheet of muscovite are gradually removed up to 850°C. An exothermic peak resulting by the crystal reformation (zeolite phase), then appears between 550 and 750°C. It can also be inferred that the exothermic peaks at 850-1150°C are due to the formation of nepheline (NaAlSiO_4).

A large and intense exothermic peak started from 1150°C attributed to the structural reorganization.

3.2.4. Electrical conductivity of Na-Sialate geopolymer

As mentioned above, from 300 to 900°C, Na-sialate geopolymer presents the same disordered structure of the nepheline. The crystal structure of Nepheline is characterized by layers of six-membered tetrahedral rings of exclusively oval conformation. The rings are built up by regularly alternating AlO_4 and SiO_4 tetrahedral. The stacking of the layers parallel to the c axis results in a three-dimensional network containing channels that are occupied by the Na cations [28]. This topology is in favor of an easy movement of Na^+ ions throughout the structure. For this reason ionic migration in nepheline is extensively reported [29–31].

The electrical measurements were performed on geopolymer pellets sintered at 300°C to remove free water.

Figure 9 shows the impedance plot at different temperatures of the Na-sialate geopolymer. The data was refined with an electrical equivalent model, the polarization part of the sample and the electrode polarization were modeled by a constant phase element (CPE), while the migration part was represented by a resistance R. The complex impedance spectra were characterized by the appearance of a semi-circle centered below the x-axis. At room temperature, the diagram shows two contributions, with two tangled semi-circles. These two relaxations, modeled by two parallel R//CPE circuits, correspond to ion migration in the grains and in the grain boundaries. The refinement of Na-Sialate geopolymer at room temperature gives bulk high ionic conductivity of about $5.10^{-5} \text{ S. cm}^{-1}$. At higher temperatures, the characteristic frequencies become so close that it is impossible to distinguish the contributions. A total resistance comprising both grain and grain boundaries contribution is then determined.

According to these results, the temperature dependence of electrical conductivity can be analyzed following an Arrhenius plot (Fig. 10).

Showing Fig. 10, two different behaviors of the geopolymer can be evidenced. At low temperature, the total conductivity slowly decreases under 200°C. This phenomenon can be attributed to the loss of water which was absorbed on the surface of the pellet leading to protonic conduction. Then, from 200°C to 725°C, the conduction properties seem to approximately follow an Arrhenius law both on heating and cooling with deduced activation energies which are 0.26 eV and 0.28 eV respectively.

Thus, Na-Sialate geopolymer presents lower σ than the specific ionic conductivity of single crystals of Na-Nepheline along the crystallographic c-axis [28] but higher than the value measured in the polycrystalline sample [6, 30–31]

Conclusions

The Na-Sialate geopolymer was synthesized by a mixture of commercial kaolinite dehydroxylated at 700°C, and sodium hydroxide pellets.

The thermal analysis demonstrated a thermal stability of geopolymer up to 1400°C. On the heating rate, an irreversibly loss of free water (approximately 14 wt %) appeared by an endothermic reaction at the

temperature range of 100-200°C.

The X-Ray powder diffraction of geopolymer unveiled an amorphous character at ambient temperature with characteristic diffraction peaks of muscovite phase. After heating at different temperatures, a zeolite crystalline phase appeared between 300°C and 600°C. At 950°C, Na-geopolymer was transformed into a crystalline material consisting predominantly in nepheline (NaAlSiO_4).

From 1200°C, diffraction peaks corresponding to corundum ($\alpha\text{-Al}_2\text{O}_3$) were appeared, this indicate that the Na-sialate geopolymer was converted into Na-sialate siloxo-geopolymer. Results confirmed by IR spectroscopy.

The electrical measurements of geopolymer pellet showed an ionic conductivity value of $\sigma = 5 \times 10^{-5} \text{ S.cm}^{-1}$ at room temperature with a probable joint contribution of H^+ and Na^+ ions. Above 200°C, Na^+ seems to remain the only charge carrier with a low activation energy of approximately $E_a = 0.26 \text{ eV}$.

Declarations

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Figures

Figure 1

Le Bail refinement of the lattice parameters of Kaolin. The point symbols represent the observed diffraction pattern, the solid lines represent the calculated pattern, and the curves at the bottom of the figure represent the difference between the observed and calculated patterns. The short vertical lines mark the positions of possible Bragg reflections.

Figure 2

Infrared bands of kaolin between 3000cm^{-1} and 4000cm^{-1}

Figure 3

Infrared bands of kaolin below 1200cm⁻¹

Figure 4

Mineralogical composition of metakaolin, geopolymer and geopolymer heated for 1 h at different temperatures (with radiation CuK α)

Figure 5

X-ray powder diffractograms of geopolymer heated for 1 h at 950°C and 1100°C (with radiation CuK α)

Figure 6

Typical X-ray powder diffractogram of geopolymer heated for 1 h at 1200°C

(* α -Al₂O₃)

Figure 7

FT-IR spectra of kaolin, metakaolin and geopolymer cured at different temperatures

Figure 8

The TG-DTA curves of Na-Sialate geopolymer

Figure 9

Nyquist plot of impedance diagrams measured at different temperatures

Figure 10

Temperature dependence of $\ln(\sigma T)$ versus $1000/T$ for Na-sialate geopolymer.