

Pseudo-cubic crystal structure and phase transition in doped ye'elimitite

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Supporting Information Placeholder

ABSTRACT: Sodalites are tridimensional alumino-silicate materials containing cages where loosely bonded anions are located. Ye'elimitite, $\text{Ca}_4[\text{Al}_6\text{O}_{12}]\text{SO}_4$, is outstanding as an aluminate sodalite with a flexible framework accepting several type of dopants with important structural consequences. Moreover, ye'elimitite is also important from an applied perspective as it is the most relevant phase in calcium sulfoaluminate cements. The crystal structure of stoichiometric ye'elimitite has recently been unraveled but the structure of dopant-containing ye'elimitite, which is presents in cements, is not well studied. Here, we report the pseudo-cubic crystal structure of doped ye'elimitite, $\text{Ca}_{3.8}\text{Na}_{0.2}\text{Al}_{5.6}\text{Fe}_{0.2}\text{Si}_{0.2}\text{O}_{12}\text{SO}_4$, from high-resolution synchrotron powder diffraction data. The powder pattern is indexed with a cubic cell and a structural model is reported based on the $I\bar{4}3m$ space group. However, this compound displays diffraction peak narrowing on heating. Furthermore, some high-angle split peaks become single peak on heating and a phase transition is measured at 525°C. Therefore, it is concluded that the crystal structure at room temperature has lower symmetry although it can be described as cubic. The structural study at 800°C suggests a truly cubic structure and we speculate that this phase transition, on heating, is likely related with the dynamical disordering of the sulfate anions. Finally it is concluded that the high temperature cubic state was not quenchable to ambient, even when the tested chemical substituents are introduced into the structure.

INTRODUCTION

Sodalites with general composition $\text{M}_4[\text{T}_6\text{O}_{12}]\text{X}$ have been known for many years as both naturally-occurring minerals and synthetic compounds¹. The general formula refers to a structure that is (ideally) a body-centered cubic unit cell with a lattice parameter of 9 Å and where M is a relatively low-charged caged cation such as Ca, Na or Sr²⁺; T occupies tetrahedral site(s) and is typically Si or Al; and X is the loosely-bonded caged anion which is either spherical (in the case of Cl) or tetrahedral (in the case of SO₄, WO₄, and CrO₄). Sodalites are interesting materials for several reasons such as their ferroic phase transition³ from the high-temperature cubic phase to complex superstructures at lower temperatures.

Stoichiometric calcium sulfoaluminate or ye'elimitite, $\text{Ca}_4[\text{Al}_6\text{O}_{12}]\text{SO}_4$, can be described as a sodalite where M = Ca, T = Al, and X = SO₄, and crystallizes as a tectoaluminosilicate sodalite structure⁴. This structure was first analyzed by Hanstead and Moore⁵ using X-ray powder diffraction,⁶ and Saalfeld and Depmeier⁷ reported atomic parameters for a cubic crystal structure with space group $I\bar{4}3m$ and $a=9.195$ Å. In 1995, Calos et al.⁸ published an orthorhombic crystal structure, Pcc2 space group, which was revised in 2013 by joint neutron and X-ray powder diffraction Rietveld refinement and atomistic calculations⁴. Recently, the disordered crystal structure of cubic stoichiometric ye'elimitite at 800°C has been satisfactorily studied in the $I\bar{4}3m$ space group using a split-atom model⁹.

Stoichiometric ye'elimitite at room temperature is orthorhombic Pcc2. However, the substitutions of Ca²⁺ by Na⁺ and Al³⁺ by B³⁺, Si⁴⁺ or Fe³⁺ seems to restore the cubic symmetry¹⁰. In the

case of iron, its maximum substitution degree may depend on the crystal structure of ye'elimitite⁵, due to the competition between aluminum and iron. Idrissi et al.¹¹ studied the Al/Fe substitution, $\text{Ca}_4\text{Al}_{6-2x}\text{Fe}_{2x}\text{SO}_{16}$, where x was comprised between 0 and 3, and other authors found that the maximum iron solid solution was reached at 22.61 wt%, expressed as Fe₂O₃¹². The polymorphism of ye'elimitite and its solid solutions in the presence of different amounts of Na⁺ and Fe³⁺ were also investigated¹³.

In addition, the interest in the ye'elimitite structure has recently increased because it is the major component in calcium sulfoaluminate cements (CSA)¹⁴⁻¹⁶, and the second most relevant phases in sulfoelite cements, BCSA, (~25 wt%)¹⁷⁻¹⁹. CSA and BCSA cements are environmentally-friendly materials as they allow decreasing the CO₂ footprint in cement fabrication. On average, for every ton of ordinary Portland cement (OPC) produced, 0.97 tons of CO₂ are released into the atmosphere^{20,21}, with the cement industry contributing around 6% of all anthropogenic CO₂ emissions. By comparison, CSA cements are produced with significantly lower CO₂ emissions relative to OPC, achieved through the use of lower amount of carbonated raw-materials (part of calcite is replaced by gypsum) and a reduced clinkering temperature. The overall CO₂ emission reduction can amount up to 40%.²¹

The effect of iron in the formation of ye'elimitite in belite calcium sulfoaluminate clinkers has been also studied^{22,23}. The iron content of ye'elimitite in these industrially produced clinkers was analyzed and it resulted relatively low^{24,25}. In a recent work²², orthorhombic and cubic ye'elimitite polymorphs were observed to occur in BCSA clinkers doped with B³⁺ and Na⁺.

However, the ye'elimite phase was cubic when Na^+ , Fe^{3+} and Si^{4+} were simultaneously present. Bullerjahn et al.²³ also found a mixture of cubic and orthorhombic ye'elimite in their cements and the cubic polymorph increased at the expense of the orthorhombic one with increasing the iron content. A reverse correlation between the amount of ye'elimite and tetracalcium aluminoferrite (C_4AF) was found, which agrees with the incorporation of iron into ye'elimite framework²³.

The structure of stoichiometric ye'elimite has been recently established as orthorhombic⁴ at room temperature and cubic⁹ at high temperatures. The objective of this study is to clarify the crystal structure of doped (disordered) ye'elimite. To this end, highly crystalline single phase iron-silicon-sodium doped ye'elimite, $\text{Ca}_{3.8}\text{Na}_{0.2}\text{Al}_{5.6}\text{Fe}_{0.2}\text{Si}_{0.2}\text{O}_{12}\text{SO}_4$, has been synthesised. The high temperature structure and phase transition have been studied by thermodiffraction. At 800°C, doped ye'elimite seems to be truly cubic. At room temperature, synchrotron powder diffraction also indicates a cubic diffraction pattern. However, the thermodiffraction study shows peak narrowing with some high-angle partially split peaks (at room temperature, RT) becoming single peaks (after a phase transition measured at 525 °C). Therefore, we are forced to conclude that the RT structure is pseudo-cubic. The reported RT structure is important from both basic (f.i. phase transitions) and applied research (cement analysis) points of views.

EXPERIMENTAL SECTION

Synthesis conditions.

Doped ye'elimite with a nominal composition of $\text{Ca}_{3.8}\text{Na}_{0.2}\text{Al}_{5.6}\text{Fe}_{0.2}\text{Si}_{0.2}\text{O}_{12}\text{SO}_4$ was synthesized following the methodology reported elsewhere⁴ for stoichiometric ye'elimite. Suitable amounts of CaCO_3 (99.95%, Alfa Aesar), Al_2O_3 (99.997%, Alfa Aesar), Fe_2O_3 (99.945%, Alfa Aesar), SiO_2 (99.56%, ABCR), Na_2CO_3 (99.999%, Sigma Aldrich) and $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ (ground natural single-crystal from Málaga, Spain) were used to obtain approximately 8 g of powder. The mixture was ground for 1 hour in an agate mortar with ethanol. The resulting powder was dye-pressed (20 mm diameter and 500 MPa) and heated at 1250°C for 4 hours (heating rate of 5 °C/min) followed by a rapid cooling. Finally, the sample was ground in a tungsten carbide mortar.

Synchrotron X-ray powder diffraction (SXRPD).

SXRPD data was collected in Debye-Scherrer (transmission) mode using the X-ray powder diffraction station of ALBA, the Spanish Synchrotron Radiation Facility (Barcelona, Spain)²⁶. The wavelength, 0.6202(2) Å, was selected with a double-crystal Si (111) monochromator and determined from a Si640d NIST standard ($a=5.43123$ Å) measurement. The diffractometer is equipped with a detector system based on crystal analyzers in the diffracted beam especially suited for high-resolution experiments giving also a flat background. The capillary (0.5 mm of diameter) was rotated at 400 rpm during data collection to improve diffracting particle statistics. The data acquisition time was very large, ~4 hours, to attain a very good signal-to-noise ratio over the angular range 1–45° (2 θ). The temperature inside the experimental hutch was 26(1)°C.

Analysis of SXRPD data.

Rietveld analysis was performed using the GSAS suite of programs and the EXPGUI graphic interface²⁷. Final global optimized parameters were: background coefficients, zero-shift error, cell parameters, and peak shape parameters using a pseudo-Voigt function corrected for axial divergence²⁸. We used as starting model the recently reported high temperature cubic structure⁹. The atomic positional coordinates and anisotropic atomic displacement parameters (ADPs) of ye'elimite were optimized.

Variable Temperature Laboratory X-ray Powder Diffraction (VT-LXRPD).

VT-LXRPD data were recorded on an X'Pert PRO MPD diffractometer (PANalytical) equipped with a Ge (111) primary monochromator, using strictly monochromatic $\text{CuK}\alpha_1$ radiation ($\lambda=1.54059$ Å) and an X'Celerator detector for the sample mixed with NIST silicon in order to decorrelate the thermal expansion from the sample height changes as a function of temperature. The sample stage was an Anton Paar HTK1200 camera working under static air. Data were collected with temperature interval ranging of 100°C, from RT to 800°C after a delay time of 15 min to ensure thermal equilibration. Data were acquired over the angular range 10–78° (2 θ) with a step size of 0.017°, resulting in 2 hours acquisition time per pattern. The sample was then cooled to RT where a final RT pattern was also recorded. A Rietveld study was carried out as described above to extract the unit cell variation, the sample height changes and the peak shape evolution with temperature.

A higher quality pattern was collected at 800°C in the range 5–100 (2 θ) with a step size of 0.017°, resulting in a 6 h data collection time but for the pure sample (i.e. without added silicon). A Rietveld structural study was also performed on this pattern following the methodology detailed in the previous section.

Permittivity measurements.

Electrical characterization was carried out on cylindrical pellets (~10 mm in diameter and ~1 mm thickness) obtained by dye-pressing ~0.1 g of powders mixture at 500 MPa. The pellets were sintered at 1250°C for 2 h after a heating rate of 5 °C/min. Electrodes were made by coating opposite pellet faces with METALOR[®] 6082 platinum paste and heating to 800°C at a rate of 5 °C/min for 15 min in air to decompose the paste and to harden the Pt residue. Impedance data were collected using a Hewlett-Packard 4284 A impedance analyzer over the frequency range 20 Hz – 1 MHz from 300 to 700°C at a heating rate of 10 °C/min.

Results and Discussion

Synthesis.

Several stoichiometries were tested and the selected one is that with the highest dopant contents but yielding a single phase sample. The synthesis procedure followed to prepare doped ye'elimite, $\text{Ca}_{3.8}\text{Na}_{0.2}\text{Al}_{5.6}\text{Fe}_{0.2}\text{Si}_{0.2}\text{O}_{12}\text{SO}_4$, was very similar to that reported for stoichiometric ye'elimite⁴, but the temperature

was reduced 50°C relative to the synthesis temperature for stoichiometric ye'elimite, due to the presence of iron oxide in the reaction medium. The addition of SiO₂, Na₂O and Fe₂O₃ stabilized the high temperature cubic form of ye'elimite at room temperature.

Structural study of doped ye'elimite at room temperature.

SXRPD was used to study the crystal structure of doped ye'elimite, Ca_{3.8}Na_{0.2}Al_{5.6}Fe_{0.2}Si_{0.2}O₁₂SO₄. The high-resolution SXRPD pattern was auto-indexed using DICVOL06²⁹ in a cubic unit cell by using 20 lines. This cell indexes all observed diffraction reflections. Table 1 shows the auto-indexing results, including the figures of merits³⁰.

Table 1. Values for the unit cell for doped and stoichiometric ye'elimite cubic samples auto-indexed with DICVOL06, including figures of merit³⁰.

	a(Å)	V (Å ³)	T(°C)	M(20)	F(20)
Doped-ye'elimite SXRPD	9.1974(7)	778.0	26	58.4	116
Doped-ye'elimite LXPDP	9.2533(1)	792.3	800	281	200
Stoich.-ye'elimite LXPDP	9.2475(1)	790.8	800	449	343

We have used the cubic crystal structure recently reported for stoichiometric ye'elimite at 800°C⁹, space group I4̄3m, as starting model for the Rietveld refinement. Once the overall parameters were optimized, the structural parameters were varied. No attempts to locate the dopants have been carried out as they should be disordered in the cubic structure. The final Rietveld disagreement factors are shown in Table 2 where they are compared to those obtained for previous RT cubic⁷ and HT cubic structures⁹. The recorded pattern clearly contains the diffraction reflections of the cubic sodalite structure. Furthermore, when tetragonal and orthorhombic symmetries were tested, the disagreement values were very high and the refinements did not converge. Figure 1 shows the SXRPD Rietveld plot using the refined cubic structure.

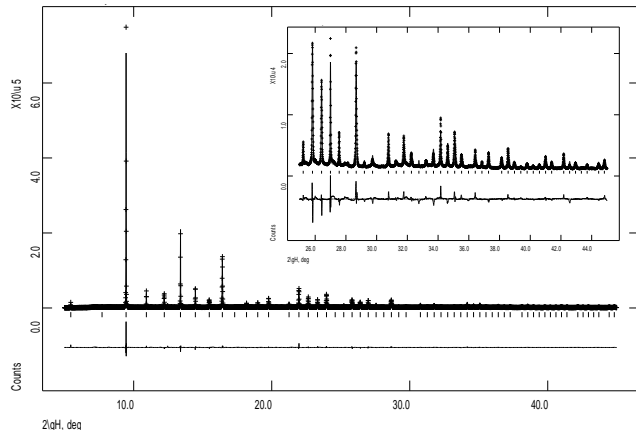


Figure 1. SXRPD Rietveld plot for the cubic structure of doped ye'elimite at RT. Inset details the high-angle range.

The refined unit cell parameter is a=9.1970(1) Å and the cell volume is 777.93(2) Å³. The anisotropic displacement parameters (ADPs) were also optimized as in reference 9. The refined atomic parameters (coordinates and anisotropic displacement parameters) for the RT cubic structure of doped ye'elimite are given in Table S1, provided as supplementary information. Selected interatomic distances and angles are given in Table

S2 (supplementary information). The AlO₄ tetrahedrons have four 1.734(1) Å bond distances and somewhat distorted angles with four values of 105.6(1)° and two large one, 117.4(2)°. The SO₄ tetrahedron is described by the oxygen split model⁹ which lead to reasonably S-O bond distances of 1.474(6) Å. Unsplit oxygen-sulfate models invariably lead to much shorter, unrealistic, S-O bond distances. The corresponding CIF file is also deposited.

Table 2. Agreement factors for the Rietveld refinements of SXRPD and HT-LXPDP patterns for doped and stoichiometric ye'elimite cubic samples by using previously-reported cubic structures, references 7 and 9, and after atomic parameter refinement.

	T °C	Structure-1 ⁷		Structure-2 ⁹		This study	
		R _{WP} /%	R _F /%	R _{WP} /%	R _F /%	R _{WP} /%	R _F /%
Doped- SXRPDP	26	19.9	17.4	15.4	16.9	11.0	7.9
Doped-LXPDP	800	14.4	16.7	6.6	4.4	5.8	3.0
Stoichiometric-LXPDP	800	22.6	19.5	11.4	6.5	9.9	3.2

Variable-temperature study and phase transition.

A VT-LXPDP study, up to 800°C, was performed for doped ye'elimite. The full thermodiffractometric study is reported in supplementary information, Figure S1, and Figure 2 shows two selected high-angle regions. Fig. 2.a displays the sharpening of the (622) reflection which was slightly split at room temperature and becomes a single sharp peak at high temperatures. Fig. 2.b shows the sharpening of the peaks at 75.8 ° (2θ) which corresponds to the (721), (552) and (633) reflections which are intrinsically overlapped for cubic symmetry. Fig. 2.b also shows the thermal evolution of the (331) reflection of silicon for the sake of comparison which does not show any appreciable width change with temperature. All attempts to index the low temperature patterns with symmetry lower than cubic were fruitless as the observed splitting at high angles was very small and only observable for some diffraction reflections.

On the other hand, changes in the widths of the diffraction peaks with temperature were clearly measurable. Figure 3 plots the FWHMs (Full Width at the Half Maximum) values for the thermodiffractometric study including the data for ye'elimite and silicon. Figure 3 shows that the FWHM variation with temperature for Si is negligible. However, a very clear sharpening for doped ye'elimite up to 500 °C is measured and then, the FWHM changes are negligible.

Figure 4 shows the refined volume as a function of temperature of the doped phase obtained from VT-LXPDP patterns combined with Rietveld analysis. Selected Rietveld plots on heating are deposited in supplementary information: Figures S2 to S6. The volumetric thermal expansion parameter (α_v) was calculated by fitting the data with a linear equation $V=n+mT$, where $\alpha_v=m/n$. The linear coefficient of thermal expansion for a cubic system is defined as $\alpha_L=\alpha_v/3$. The final α_L values of this sample, assuming cubic structure, were $6.6 \cdot 10^{-6} \text{ }^\circ\text{C}^{-1}$ (20-400°C interval) and $9.0 \cdot 10^{-6} \text{ }^\circ\text{C}^{-1}$ (500-800°C interval), which are similar to values found for aluminosilicate sodalites³¹. In the case of the stoichiometric sample, a linear thermal expansion coefficient was calculated for each unit cell

parameter³² (in the temperature range of 20 to 450°C), Table 3. The coefficient was also calculated for stoichiometric ye'elimite (between 500 and 800°C), Table 3, for the cubic structure. From this study it is concluded that doped ye'elimite has similar thermal expansion values to stoichiometric ye'elimite. From the VT-LXRPD study, the thermal expansion coefficient calculated for silicon was $4.2 \cdot 10^{-6} \text{ }^\circ\text{C}^{-1}$ which is fairly close to reported values, $3.9 \cdot 10^{-6} \text{ }^\circ\text{C}^{-1}$ and $4.15 \cdot 10^{-6} \text{ }^\circ\text{C}^{-1}$.^{33,34}

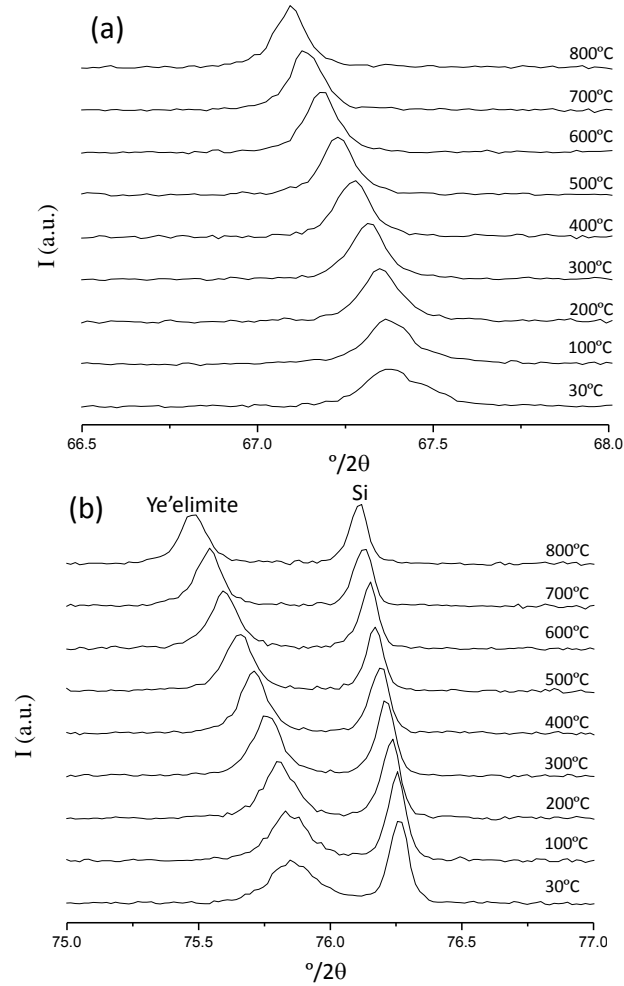


Figure 2. Selected high-angle regions of the VT-LXRPD data for doped ye'elimite. (a) Pseudo-cubic (622) reflection of doped ye'elimite; (b) a second region showing reflections from ye'elimite and Si (internal standard, for details see the text).

Table 3. Linear thermal expansion coefficients for doped and stoichiometric (St.) ye'elimite at different temperatures.

T / °C	20-450	20-450	20-450	20-400	500-800
$\alpha / ^\circ\text{C}^{-1}$	α_a	α_b	α_c	α_L	α_L
St.	$1.7 \cdot 10^{-6}$	$2.0 \cdot 10^{-6}$	$11.5 \cdot 10^{-6}$	$5.1 \cdot 10^{-6}$	$8.3 \cdot 10^{-6}$
Doped	-	-	-	$6.6 \cdot 10^{-6}$	$9.0 \cdot 10^{-6}$

The 800°C powder pattern for doped ye'elimite was also auto-indexed using DICVOL06²⁹ in a cubic unit cell. The results are given in Table 1. Figures of merit³⁰ for this indexing are much higher than those obtained at RT which points toward a truly cubic structure at high temperature. A Rietveld refine-

ment was performed using the previously reported split-atom model for the sulfate anion⁹. The refinement in space group $\bar{1}\bar{4}3m$ is very good as indicated by the low R-factors (see Table 2) and the flatness of the difference curve of the Rietveld plot (see Figure 5). However, this structure is not reported as it is essentially identical to that already reported for stoichiometric ye'elimite⁹. The final refined unit cell parameter at 800°C was $a=9.2544(1) \text{ \AA}$.

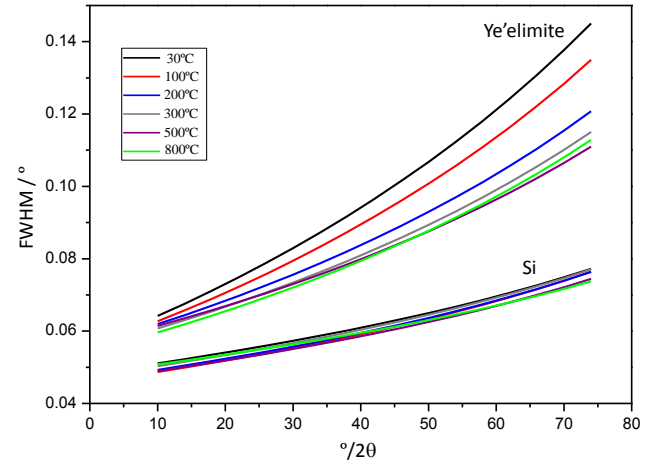


Figure 3. Full-width at the half-maximum variation with temperature for doped ye'elimite and silicon.

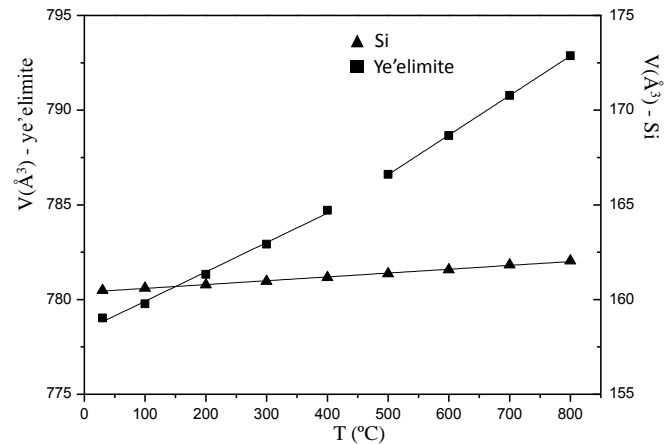


Figure 4. Refined volume for the doped ye'elimite and silicon (internal standard) from RT to 800°C as a function of temperature. Errors are smaller than the points.

The high-temperature form of stoichiometric ye'elimite has also been thoroughly studied for the sake of comparison. Firstly, the pattern was auto-indexed using DICVOL06²⁹ in a cubic unit cell, Table 1. We have not included the peak at 25.5° peak because we have recently noticed that it arises from the sample holder. The absence of this peak in the ye'elimite pattern has been already pointed out⁹. It is worth noticing that the indexing figure of merit³⁰ values for stoichiometric ye'elimite are higher than those of doped ye'elimite (both patterns were collected in identical conditions and at the same temperature). The cubic structure was verified by fitting the powder pattern with the structure recently reported⁹. Table 2 shows the agreement factors obtained using published^{7,9} and refined data. The final unit cell parameter is $a=9.2497(1) \text{ \AA}$.

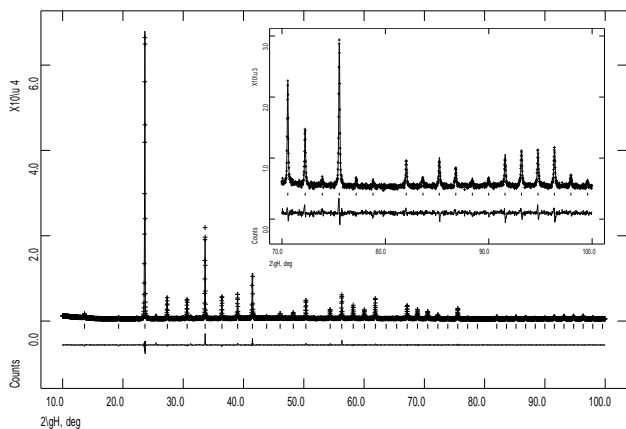


Figure 5. HT-LXRPD plot for doped ye'elimite at 800°C fitted with a refined cubic crystal structure. Inset detail the high-angle range.

The high temperature crystal structure of doped ye'elimite is cubic. Therefore, if the room temperature structure of this sample is also cubic, no phase transition would be expected of heating. The variable-temperature permittivity study for doped ye'elimite, see Figure 6, shows the presence of a peak at 525°C, which is an experimental evidence of a phase transition. For the sake of comparison, the similar measurement for stoichiometric ye'elimite is also shown in Figure 6 where the transition is observed at 470°C.⁴ This transition, also observed by differential scanning calorimetry and thermodiffraction was due to the orthorhombic-to-cubic transition and justified by the dynamical disorder of the sulfate anions within the cages.

We justify the change observed at 525°C for doped ye'elimite as the pseudo-cubic-to-cubic phase transition also related to dynamical disorder of the sulfate anions. The difference in temperature is likely due to the effect of the dopants stabilizing the static disorder of the sulfate anions within the cages.

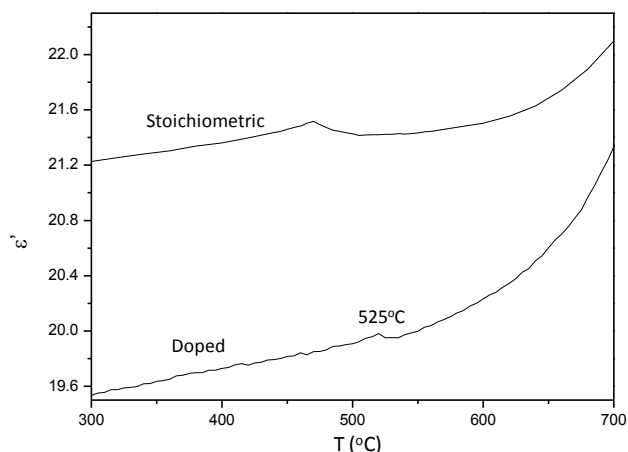


Figure 6. Permittivity signal for stoichiometric ye'elimite and doped ye'elimite.

The complete picture that emerges from this study is that the average high-temperature structure of ye'elimite (both doped and stoichiometric) is cubic. On cooling, stoichiometric ye'elimite undergoes a cubic-to-orthorhombic phase transition at 470°C likely due to the freezing and spatial ordering of the sulfate anions. The associate splitting of the diffraction peaks is clearly seen in Figure 7. On cooling, doped ye'elimite undergoes a related cubic-to-pseudo cubic phase transition at

525°C also likely due to the freezing of the sulfate anions. However, the long-range ordering of the sulfate anions does take place, due to the presence of the dopants, and the orthorhombic structure does not develop. Short-range correlations still develop at room temperature and this is evident in the background of the synchrotron pattern (see Figure 7 intermediate panel) as small humps.

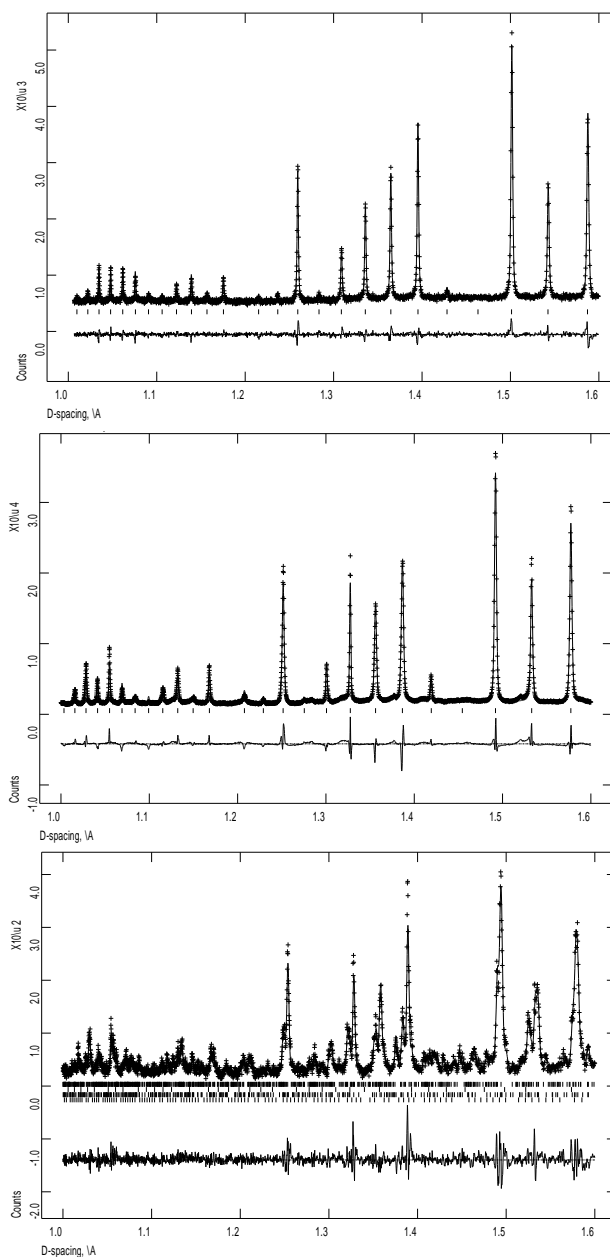


Figure 7. Selected region (1.0 to 1.6 Å) for the LXRPD pattern of doped ye'elimite at 800°C (top), SXRPD pattern of doped ye'elimite at RT (intermediate), and LXRPD pattern of stoichiometric ye'elimite at RT (bottom).

CONCLUSIONS

The crystal structure of doped ye'elimite, $\text{Ca}_{3.8}\text{Na}_{0.2}\text{Al}_{5.6}\text{Fe}_{0.2}\text{Si}_{0.2}\text{O}_{12}\text{SO}_4$, has been studied by high-

resolution synchrotron powder diffraction data. A cubic structural description is reported based on $I\bar{4}3m$ space group with a split-atom model for the sulfate anions. A thermodiffraction study shows a narrowing of the diffraction peaks on heating with some split peaks at room temperature evolving to single reflections at high temperatures. A variable temperature permittivity study shows a phase transition on heating at 525°C. The structural study at 800°C suggests a truly cubic structure and we speculate that the observed phase transition is likely related with the freezing of the sulfate anions on cooling. For stoichiometric ye'elimite, this freezing is accompanied by an ordering of the sulfate anions which provokes the cubic-to-orthorhombic phase transition. However, for doped ye'elimite the freezing is not accompanied by long-range ordering of the sulfate anions but they are locally disordered due to the presence of dopants.

ASSOCIATED CONTENT

Supporting Information. CIF file for the pseudo-cubic crystal structure at room for doped ye'elimite, $\text{Ca}_{3.8}\text{Na}_{0.2}\text{Al}_{5.6}\text{Fe}_{0.2}\text{Si}_{0.2}\text{O}_{12}\text{SO}_4$. Table S1 gives the final (refined) atomic parameters (coordinates and anisotropic displacement parameters) and Table S2 the interatomic distances and angles for doped ye'elimite at room temperature. Figure S1 displays the full thermodiffraction study for doped ye'elimite with Si added as internal standard. Figures S2-S6 display the Rietveld plots for doped ye'elimite at selected temperatures: 30, 100, 300, 500, 700 °C, respectively.

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Author Contributions

This is part of the Ph.D. of Ms. Ana Cuesta. The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

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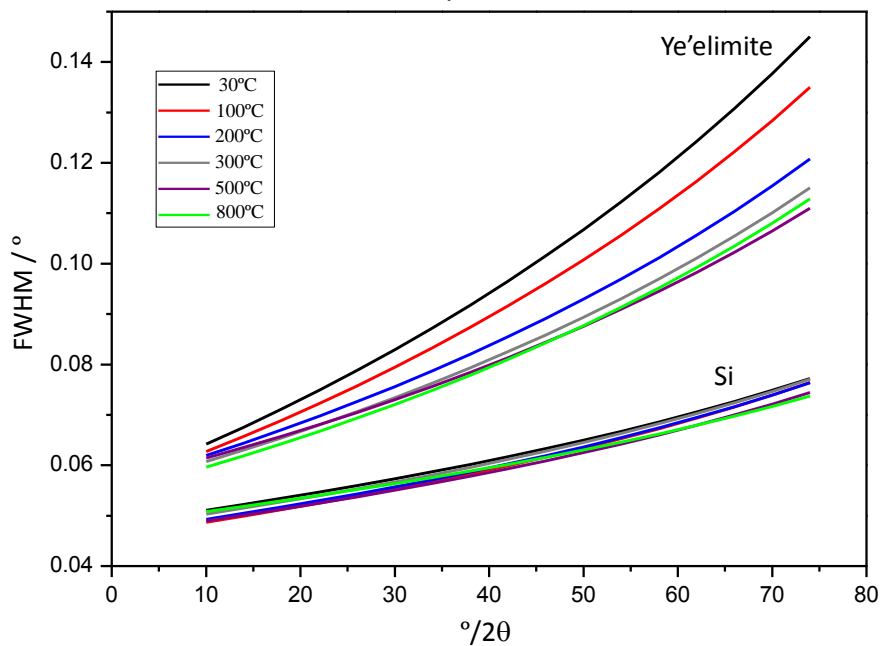
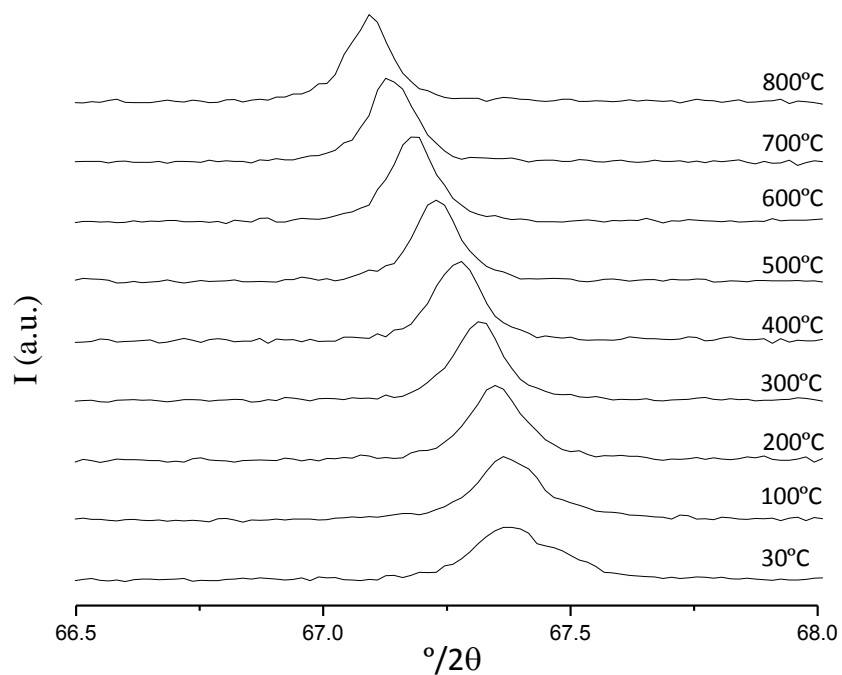
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TOC.



SYNOPSIS TOC TEXT (60 words):

A room-temperature pseudo-cubic structure for doped ye'elimite is reported. The powder pattern of this compound shows narrowing of the diffraction peaks on heating, see figure, which is fully consistent with a measured phase transition at 525°C. This transition is present in all sodalite compounds.