

Synthesis and Characterization of Analcime (ANA) Zeolite using a Kaolinitic Rock

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Abstract

Analcime is nowadays an important component in dental porcelain systems, in heterogeneous catalysis, in the nanoelectronic field, in selective adsorption and in stomatology. Analcime synthesis from a kaolinite rock coming from Romana (Sassari, Italy) is here presented. A synthesis protocol is proposed that aims to make an improvement of synthesis conditions compared to the past. The hydrothermal treatment is in fact here achieved without aging times and without the use of sodium silicate, as reported in the literature. Lower calcination temperature, synthesis temperatures and crystallization times are verified in this work. The kaolin is subjected to calcination at the temperature of 650°C and then mixed with NaOH. The experiment is performed at ambient pressure and $170 \pm 0.1^\circ\text{C}$. The degree of purity of Analcime is calculated in 97,57 % at 10h. Analcime is characterized by X-ray diffraction, infrared spectroscopy, scanning electron microscopy, inductively coupled plasma optical emission spectrometry and thermal analysis. Density is also calculated. Cell parameters and the amount of amorphous phase in the synthesis powders is estimated with quantitative phase analysis using the combined Rietveld and reference intensity ratio methods.

The experimental conditions make the synthesis protocol particularly attractive from an economic point of view. Both chemical and physical characterization of Analcime is satisfactory making the experimental protocol very promising for an industrial transfer.

Introduction

The structure of zeolites consists of three-dimensional frameworks of SiO_4 and AlO_4 tetrahedra arranged to form channels containing water and exchangeable cations (sodium, potassium, calcium etc). Analcime (ANA) zeolite is the smallest-pore zeolite and it exhibits a compact structure compared to other zeolites with an idealized unit cell of $\text{Na}_{16}\text{Al}_{16}\text{Si}_{32}\text{O}_{96} \cdot 16\text{H}_2\text{O}$ [1]. Analcime is usually of cubic symmetry. Saha [2] described the cubic unit cell composed of four-, six-, and eight-membered oxygen rings forming three non-intersecting channels. Every sodium ion is surrounded by four oxygen ions and two water molecules, which make up a distorted octahedron [3]. Analcime occurs in various geological environments. Type H analcime is formed under hydrothermal conditions by dissolution of source materials such as nepheline, plagioclase, albite, quartz, volcanic glass and recrystallization from hydrothermal solutions. A particular case of Analcime which is formed under hydrothermal conditions is that which is formed by cationic exchange K-Na from Leucite [4]; this analcime is called X type Analcime. Morphologically it retains the leucite habit, but it is dusty and often the crystal core is still leucite [5]. This cation exchange reaction requires 3 days at 300°C and 1 Kbar and the use of Leucite crystals of at least 100 microns size, as well as an activation energy of 8Kcal / mol [6]. The thermal [5] and morphological [7] analyses allow to distinguish the type of analcime (H or X). Then there is the I type analcime which segregates itself directly from the magma and occurs inside igneous rocks [8].

Analcime exhibits a variety of applications in technology especially in selective adsorption and heterogeneous catalysis [9], in the nanoelectronic field [10], in stomatology [11] and in ceramics for

denture [12].

Even if Analcime is found in nature, abundant supplies of this mineral occur in limited regions of the world. For this reason, recent research is moving towards the synthesis of this mineral by using different sources of silica and alumina.

Usually, analcime is synthesized under hydrothermal conditions using aluminosilicate clear solutions or gels, in the presence of an alkaline medium and at temperature variable from 100 to 310°C. Some studies demonstrates that the more Na₂O is in the initial raw materials, the lower the temperature at which analcime crystallizes [13]. The alternative route is to synthesize analcime from cheap local materials, i.e. by the conversion of natural glasses [14], conformed ashes [15], rhyolitic tuff [16], rice husk [17-18], clinker [19], quartz syenite powders [20] and clays [21-22] which represent highly reactive raw materials. However not only analcime prevails among the synthesis [23].

As regards the use of clays, kaolin is the material already tested in the synthesis of analcime (Hegazy et al., 2010; Atta et al., 2012). Among clay minerals, in fact, kaolinite is the most used phyllosilicate in the zeolitic synthesis because of its particularly ample supply and availability and the well-known reactivity of thermally treated kaolin clays (metakaolin) with alkali [24-25-26-27-28-29-30]. Hegazy et al. [21] synthesized analcime by hydrothermal reaction of kaolin and commercial sodium silicate solution at 200°C for 24h under autogenous pressure. Atta et al. [22] synthesized analcime from kaolin after 72 h aging and 24 h reaction time at temperature of 180°C.

The scope of the present work is to synthesize monomineralic powders of Analcime by testing a natural rock, *i. e.* a kaolinitic rock coming from Romana (Italy). This kaolin has already been successfully used in the past in the synthesis of useful minerals [28-30]. Roman kaolin has the advantage of being characterized by an excess of silica compared to common kaolins, being its mineralogical composition made of kaolinite plus minor amounts of quartz and opal-crystobalite [28]. This peculiar characteristic gives the material a Si/Al ratio greater than 1 and it makes it suitable for the synthesis of minerals characterized by a Si/Al ratio higher than 1:1, like analcime. So, the use of Romana kaolin avoids the addition of sodium silicate and it makes the synthesis protocol simpler and above all cheaper than in the past.

This work aims to improve the previous attempts made in the past for the synthesis of the analcime from kaolin by avoiding aging times and by lowering: i) synthesis temperatures ii) crystallization times and iii) kaolin calcination temperatures. It must also be said that no one in the past has actually conducted a study aimed at characterizing the purity of the synthetic product, i.e. at investigating the possible presence of unreacted and/or amorphous material. Systematic samplings during the experimental run enable to follow the progress in the crystallization of the mineral and allows to determine the time at which the climax in the crystallization is reached. The degree of purity of the synthesized powders expressed in terms of absence of amorphous phase and impurities coming from the natural kaolinite

sample is here defined through a quantitative phase analysis approach using the combined Rietveld and reference intensity ratio methods [31].

Materials And Methods

The kaolin used in this study comes from a mine located in Romana (Sassari, Italy). For the chemical composition of kaolin and its mineralogical, morphological and spectroscopic characterization, see Novembre et al. [28]. The kaolin was triturated, and the sandy fraction was separated by retention in a sieve; the fraction below 90 μm was then collected, suspended in distilled water, sonicated, and centrifuged for separation of the silt fraction and collection of the clay fraction [30]. Preliminary calcination of kaolin was carried out in open porcelain crucibles heated in a Gefran Model 1200 furnace (Gefran Spa, Brescia, Italy) to the calcination temperature (650°C) at a pressure of 1 atm. The heating rate of the sample was 1.5°C s⁻¹. Once the calcination temperature was reached, the crucibles were left in the furnace for 2 h and then removed and cooled at room temperature. The NaOH used in the synthesis protocol were purchased from Riedel-de Haën (Honeywell Riedel-de Haën, Bucharest, Romania). The purity of the reagent was of 99%. Kaolin of Romana is characterized by an excess of silica compared to the common stoichiometry of commercial kaolins [28], therefore the experimental protocol did not provide for the addition of sodium-silicate. 2 g of metakaolinite have been directly dissolved in 20ml of a NaOH (8%) solution. The initial mixture had the composition: 6.25 SiO₂ – 1.00 Al₂O₃ – 3.6 Na₂O. The mixture was homogenized for two hours with a magnetic stirrer. Then was put inside a stainless-steel hydrothermal reactor and heated at 10 °C/min until the desired temperature (170°C). Synthesis products were sampled periodically from the reactor, filtered with distilled water and dried in an oven at 40°C for a day.

Kaolin and products of synthesis were analysed by powder X-ray diffraction (XRPD); the instrument was a Siemens D5000 operating with a Bragg-Brentano geometry (CuK α =1.518 Å, 40 kV, 40mA, 4-120° 2theta scanning interval, step size 0.020° 2theta). Identification of analcime and relative peak assignment was performed with reference to the following JCPDS code: 00-019-1180. Both the crystalline and amorphous phases in the synthesis powders were estimated using quantitative phase analysis (QPA) applying the combined Rietveld and reference intensity ratio (RIR) methods; corundum NIST 676a was added to each sample, amounting to 10% (according to the strategy proposed by Novembre et al. [30- 31] and the powder mixtures were homogenized by hand-grinding in an agate mortar. Data for the QPA refinement were collected in the angular range 5-110° 2theta with steps of 0.02° and 10s step⁻¹, a divergence slit of 0.5° and a receiving slit of 0.1mm.

Data were processed with the GSAS software [32] and the graphical interface [33] starting with the structural model proposed by Gatta et al. [34] for Analcime. The following parameters were refined: background parameters, zero shift, cell parameters and peak profiles.

Morphological analyses were obtained by means of scanning electron microscopy (JEOL JSM-840 served by a LINK Microanalysis EDS system, with operating conditions of 15kV and window conditions

ranging from 18 to 22 mm) [35].

Induced coupled plasma optical emission spectroscopy technique (ICP-OES, Perkin Elmer Optima 3200 RL) was performed on synthesized powders through previous fusion (Pt meltpot) in lithium meta-tetra borate pearls and subsequent acid solubilisation and analytical determination [36].

Density of Analcime was calculated by He-pycnometry using an AccuPyc 1330 pycnometer. [37].

The infrared analysis was performed with a spectrometer FTLA2000, served by a separator of KBr and a DTGS detector; the source of IR radiation was a SiC (Globar) filament. Samples were treated according the method of Novembre et al. [38] using powder pressed pellets (KBr/sample ratio of 1/100, pressure undergone prior determination 15t/cm²); spectra were processed with the program GRAMS-AI (GRAMS/AI™ Spectroscopy Software, Thermo Scientific Company).

Differential thermal analysis (DTA) and thermogravimetry (TG) were performed using a Mettler TGA/SDTA851e instrument (10°/min, 30-1100°C, sample mass of ~10 mg, Al₂O₃ crucible) (Mettler Toledo, Greifensee, Switzerland).

Results

Results of XRPD analyses performed on the synthesis run conducted at 170° are illustrated in Figure 1.

Appearance of Analcime is evident at 5h. The existence field of the ANA zeolite is very large, in fact the phase remains isolated for a long time; peaks grow in height until reaching the maximum intensity at 10 hours. The intensity of the peaks remains unchanged even at 30 hours, proving that the climax of crystallization is reached only 10h after the start of the thermal treatment. Results of the QPA analyses conducted on samples at 5, 10 and 30h are illustrated in Table 1. Analcime percentage increases over time at the expense of the amorphous component and reaches its climax at 10 hours (97.57%). There is no substantial change in the percentages of the crystalline fraction versus the amorphous one passing from 10 to 30 hours.

For the sample at 10h the observed and calculated profiles and difference plots for ANA and corundum NIST 676a are reported in Figure 2. Cell parameters of ANA, refined with cubic symmetry space group *Ia-3d*, remain constant within error as a function of the experimental run time. The results of the Rietveld refinements provide cell values that are in good agreement with the structural model proposed by Gatta et al. [34].

Figure 3a-b reports SEM images of ANA crystals at 5 h and 10h, respectively. It results an average maximum length of crystals observed to be around 25 µm. Chemical analysis performed on samples at 10h (180 °C) resulted in the stoichiometry of Na_{6.00}Al_{5.98}Si_{12.02}O₃₆. The density of Analcime from sample at 10h (180 °C) was determined to be 2.261(5) g/cm³.

Further characterizations were carried out on the sample at 10h (170 °C). Figure 4 illustrates the infrared spectrum of the sample. The significant broad peaks are located at 3467 and 1638 cm^{-1} for O-H stretching and bending, respectively. The bands at 1029 and 1091 cm^{-1} are attributed to the stretching mode of O-Na-O. The bands at 768, 742, 698 and 625 cm^{-1} are attributed to Si-O-Si symmetric stretching vibration. Bands at 446 and 416 cm^{-1} are characteristic of O-Si-O bonding mode. Data are coherent with those available in the literature [10-22-39-40].

Thermogravimetric analysis conducted on samples at 10h (170 °C) revealed a gradual and continuous water loss up to 1000°C (Figure 5). In particular three well defined dehydration steps are observed at 140, 415 and 980 °C. The first two peaks correspond to removal of physical absorbed and occluded water. The loss at 980 °C is due to gradual removal of water in micropores. A total weight loss of 13,5 % is obtained. The three step-dehydration process here observed is in agreement with findings by Sandoval et al. [19].

Conclusion

A goal of fundamental importance that research today seeks to pursue is the green synthesis of minerals achieved by using naturally derived reagents. At the same time, research seeks to reduce synthesis times and temperatures in order to create inexpensive protocols from an economic point of view. Analcime has already been synthesized in the past from natural materials, one of these is kaolin [21-22]. This work describes the synthesis of Analcime using a kaolinitic rock coming from Romana (Sassari, Italy). When our results are compared with the past literature, a reduction of calcination temperature of kaolinite, of the synthesis temperature, and of crystallization times is evident. Moreover, no aging time is required in the synthesis protocol.

Hegazy et al. [21] operate a calcination temperature of kaolinite of 900°C, while we reduced it to 650°C. In addition, our protocol does not provide for the use of sodium silicate, as indicated by these authors. Romana kaolin, in fact, is characterized by itself by an excess of silica compared to pure kaolins [28]; this makes the experimental procedure cheaper and faster.

Hegazy et al. [21] get analcime at 24 h at 200°C, and Atta et al. [22] get Analcime after 72 h aging and 24 h reaction time at 180°C; in our work ANA zeolite crystallizes at only 5 h at 170°C. The existence field of the zeolite is very large, in fact no phase replaces it in the time interval 5-30 hours.

In mineralogical synthesis processes in general, but when synthesizing starting from natural reagents in particular, great attention must be paid to characterizing the degree of purity of the synthetic products. In fact, amorphous residues or even impurities of the starting material can often be present or even the desired phase is not isolated, but in coexistence with others. Industry requires at least 90% purity. It must be said that none of the authors in the past has estimated the degree of purity of the synthetic powders, from this it is not possible to establish whether these are representative of the climax of crystallization, or to exclude the presence of minor other phases and/or amorphous residues.

The degree of success of an experiment is here established from calculation of the percentage of crystallization vs. amorphous material and other impurities. In our case, already at 5h of the synthesis run, 93.47% is reached, which is already a sufficient achievement for the parameters required by industry. At 10h it then exceeds 97%. This percentage remains unchanged for a long time, therefore the stability range of the mineral is wider than that obtained by the previous authors in the literature. All other characterization, i. e. chemical-physical, morphological, and spectroscopic characterization of experimental products testifies the efficacy of the experimental protocol proposed here. All these results suggest that transfer to an industrial production scale would be possible.

Last but not least, this work does not use a commercial kaolin but a kaolinite rock from a disused quarry. This further reduces the costs of the experimental protocol. It also gives the protocol an added value, as the synthesis of a useful mineral is obtained through the enhancement of an otherwise unused georesource. In addition, as Romana's kaolin is characterized by an excess of silica with respect to commercial kaolins, this study opens the way to the use of so-called "impure" kaolins, providing for their enhancement in the synthesis processes of useful minerals characterized by a Si/Al ratio higher than 1:1.

Declarations

Acknowledgements

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References

1. Meier, W.M. & Olson, D.H. *Atlas of Zeolite Structure Types, 4th ed.* (ed. Elsevier Science) 405pp (London, 1996).
2. Saha, P. Geochemical and X-ray investigation of natural and synthetic analcites. *Mineral.* **44**, 300 (1959).
3. Palubinskaite, D., Sinkeviciene, I., Siauciunas, R., Sadunas, A. Determination of the parameters of Analcime synthesis and the obtaining of Leucite by ion exchange. *Sci.* **6 (1)**, 36-43 (2000).
4. Karlsson, H.R. & Clayton, R.N. Oxygen and hydrogen isotope geochemistry of zeolites. *Cosmo. Acta* **54**, 1369-1386 (1990).
5. Giampaolo C. & Lombardi, G. Thermal behaviour of analcimes from two different genetic environment, *J. Mineral.* **6**, 285-289 (1994).
6. Gupta, A.K. & Fyfe, W.S. Leucite survival: the alteration to analcime. *Mineral.* **13**, 361-363 (1975).
7. Putnis, A., Putnis, C., Giampaolo, C. The microtexture of analcime phenocrysts in igneous rocks, *J. Mineral.* **6**, 627-632 (1994).
8. Karlsson, H.R. & Clayton, R.N., Analcime phenocrysts in igneous rocks: primary or secondary? *Mineral* **76**, 189-199 (1991).

9. Tatlier, M., Baris Cigizoglu, K., Tokay, B., Erdem-Senatalar, A. Microwave vs. conventional synthesis of analcime from clear solutions. *Cryst. Growth* **306**, 146-151 (2007).
10. Liu, B.S., Tang, D.C., Au, C.T. Fabrication of analcime zeolite fibers by hydrothermal synthesis *Mesop. Mat.* **86**, 106-111 (2005).
11. Balandis, A., Traidaraite, A. The influence of Al containing component on synthesis of analcime of various crystallographic systems. *Sci-Poland* **25**, 637-647 (2007).
12. Rasmussen, S.T., Groh, C.L., O'Brien, W.J. Stress induced phase transformation of a cesium stabilized leucite porcelain and associated properties. *Mater.* **14**, 202-211 (1998).
13. Breck, D.W. Zeolite Molecular Sieves. Structure, Chemistry and Use. Robert E. Krieger Publishing Company, Malabar, Florida, 771 pp. (1984).
14. Derkowski, A. Experimental transformation of volcanic glass from Streda nad Bodrogom (SE Slovakia). *Geol Carpath* 2002, vol. 53. In: Proceedings of the XVII Congress of Carpathian-Balkan Geological Association Bratislava, September 1-4 2002.
15. Garcia, J., Cazorla, D., Linares, A. Selective synthesis of zeolite briquettes from conformed ashes. *Chem. Tech. Biot.* **77**, 287-291 (2002).
16. Abe, H., Aoki, M., Konno, H. Synthesis of analcime from volcanic sediments in sodium silicate solution. *Miner. Petr.* **42**, 81-92 (1973).
17. Petkowicz, D.I., Rigo, R.T., Radtke, C., Pergher, S.B., dos Santos, J.H.Z. Zeolite NaA from Brazilian chrysotile and rice husk. *Mesop. Mat.* **116**, 548-554 (2008).
18. Azizi, S.N. & Ypusefpour, M. Synthesis of zeolites NaA and analcime using rice husk ash as silica source without using organic template. *J. Mat. Sci.* **45**, 5692-5697 (2010).
19. Sandoval M.V., Henao, J.A., Rios, C.A., Williams, C.D., Apperley, D.C. Synthesis and characterization of zeolite ANA framework by hydrothermal reaction of natural clinker. *Fuel* **88**, 272-281 (2009).
20. Ma, X., Yang, J., Ma, H., Liu, C., Zhang, P. Synthesis and characterization of analcime using quartz syenite powder by alkali-hydrothermal treatment. *Mesop. Mat.* **201**, 134-140 (2015).
21. Hegazy, E.Z., Abd El Maksod, I.H., Abo El Enin, R.M.M. Preparation and characterization of Ti and V modified analcime from local kaolin. *Clay Sci.* **49**, 149-155 (2010).
22. Atta, A.Y., Jibril, B.Y., Aderemi, B.O., Adefila, S.S. Preparation of analcime from local Kaolin and rice husk ash. *Clay Sci.* **61**, 8-13 (2012).
23. Navickas, A.A., Spokauskas, A.A., Stuopys, A. Factors that influence analcime formation. *Geologija* **42**, 19-25 (2003).
24. Novembre, D., Di Sabatino, B., Gimeno, D. & Pace, C. Synthesis and characterization of Na-X, Na-A and Na-P zeolites and hydroxysodalite from metakaolinite. *Clay Miner.* **46**, 336-354 (2011).
25. Novembre, D., Di Sabatino, B. & Gimeno, D. Synthesis of Na-A zeolite from 10 Å halloysite and a new crystallization kinetic model for the transformation of Na-A into HS zeolite. *Clay Miner.* **53**(1), 28-36 (2005).

26. Novembre, D., Gimeno, D., Pasculli, A. & Di Sabatino, B. Synthesis and characterization of sodalite using natural kaolinite: an analytical and mathematical approach to simulate the loss in weight of chlorine during the synthesis process. *Environ. Bull.* **19**(6), 1109–1117 (2010).
27. Novembre, D., Pasculli, A., Pace, C., Gimeno, D. & Di Sabatino, B. Synthesis of sodalite from natural kaolinite. A way to simulate the loss in weight of chlorine during synthesis process by an analytical and mathematical modelling. *Online Soc. Geol. It.* **11**(2), 548–549 (2010).
28. Novembre, D., Gimeno, D., d'Alessandro, N. & Tonucci, L. Hydrothermal synthesis and characterization of kalsilite by using a kaolinitic rock from Sardinia, Italy, and its application in the production of biodiesel. *Mag.* **82**(4), 961–973 (2018).
29. Novembre, D., Gimeno, D. & Del Vecchio, A. Improvement in the synthesis conditions and studying the physicochemical properties of the zeolite Li-A(BW) obtained from a kaolinitic rock. *Rep.* **10** (1), 5715-5723 (2020).
30. Novembre, D., Gimeno, D. & Del Vecchio, A. Synthesis and characterization of Na-P₁ (GIS) zeolite using a kaolinitic rock. *Rep.* **11**, 4872-4883 (2021).
31. Novembre, D., Gimeno, D., d'Alessandro, N. & Tonucci, L. Hydrothermal synthesis and characterization of kalsilite by using a kaolinitic rock from Sardinia, Italy, and its application in the production of biodiesel. *Mag.* **82**(4), 961–973 (2018).
32. Larson A.C. & Von Dreele R.B. GSAS: General Structure Analysis System. Document Laur 86-748, Los Alamos National Laboratory (1997).
33. Toby, B.H. EXPGUI, a Graphical User Interface for GSAS. *Journal of Applied Crystallography*, **34**, 210-213 (2001).
34. Gatta, G. D., Nestola, F., Ballaran, T. B. Elastic behaviour, phase transition, and pressure induced structural evolution of analcime. *Am. Mineral.* **91**, 568-578 (2006).
35. Novembre, D., Di Sabatino, B., Gimeno, D., Garcia Valles, M. & Martinez-Manent, S. Synthesis of Na-X zeolites from tripolaceous deposits (Crotone, Italy) and volcanic zeolitized rocks (Vico Volcano, Italy). *Mesopor. Mat.*, **75**, 1-11 (2004).
36. Novembre, D. & Gimeno, D. The solid-state conversion of kaolin to KAlSiO₄ minerals: the effects of time and temperature. *Clays and Clay Min.* **65**(5), 355–366 (2017).
37. Novembre, D., Gimeno, D. & Poe, B. Synthesis and Characterization of Leucite Using a Diatomite Precursor. *Rep.* **9**, 10051–10061 (2019).
38. Novembre, D., Pace, C. & Gimeno, D. Syntheses and characterization of zeolites K-F and W type using a diatomite precursor. *Mag.* **78**, 1209–1225 (2014).
39. Mozgawa, W. The relation between structure and vibrational spectra of natural zeolites. *Mol. Struct.* **596**, 129-137 (2001).

Tables

sample + 10% corundum Nist 676a	5h	10h	30h
R_{wp}	0,19	0,18	0,18
R_p	0,14	0,15	0,14
χ^2	2,08	2,18	2,17
space group ANA	<i>la-3d</i>	<i>la-3d</i>	<i>la-3d</i>
<i>a</i> (Å)	14.2071(0.0051)	14.2041(0.0037)	14.2052(0.0035)
<i>b</i> (Å)	10.0515(0.0005)	10.0536(0.0018)	10.0539(0.0021)
<i>c</i> (Å)	10.0418(0.0007)	10.0427(0.0004)	10.0416(0.0006)
% amorphous	6.53(13)	2.43(11)	2.47(11)
ANA	93.47(17)	97.57(14)	97.53(14)

Table 1. Results of the QPA analyses conducted on samples synthesized at 170 °C.

Figures

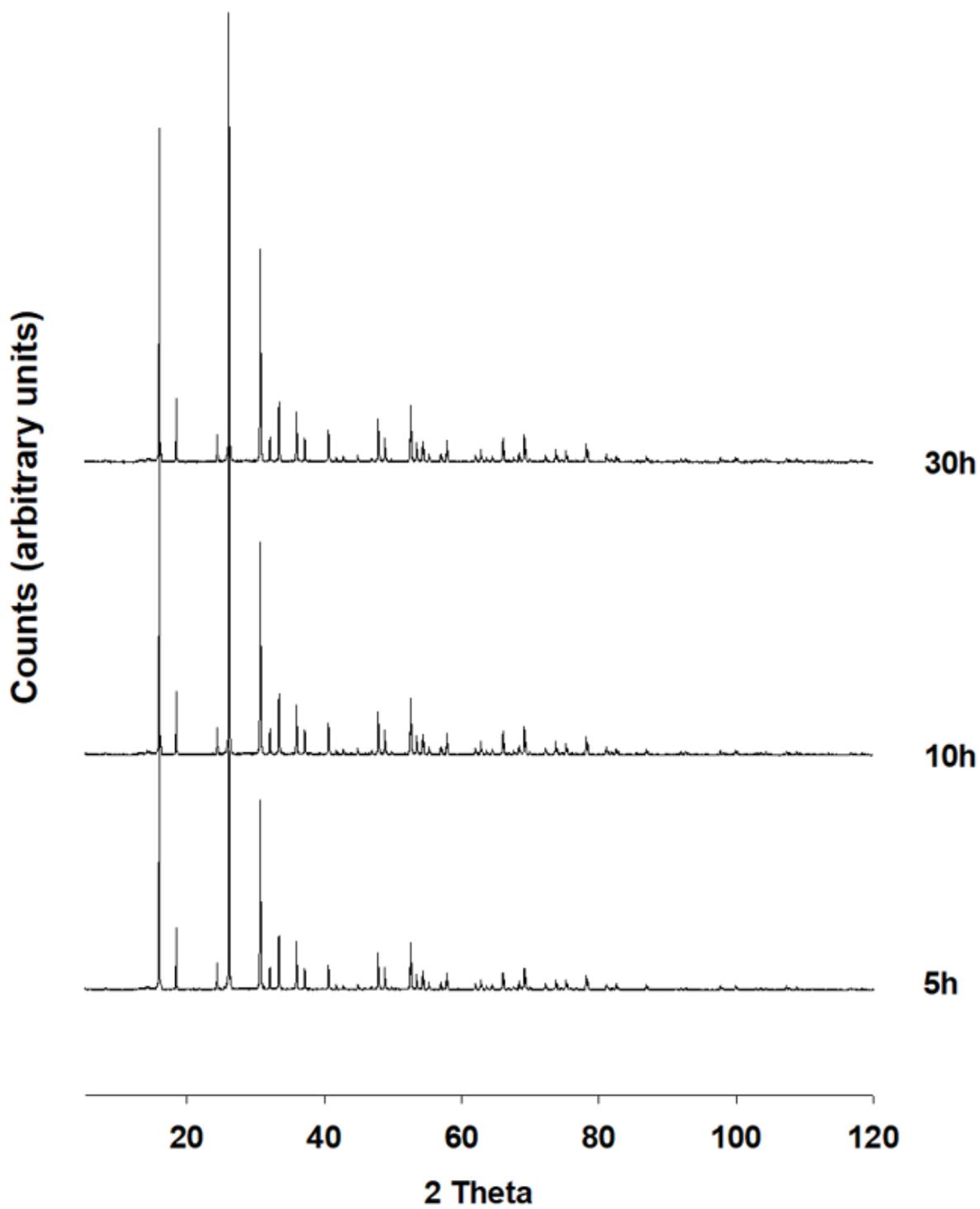


Figure 1

X-ray diffractometric sequence of the synthesis run at 170 °C.

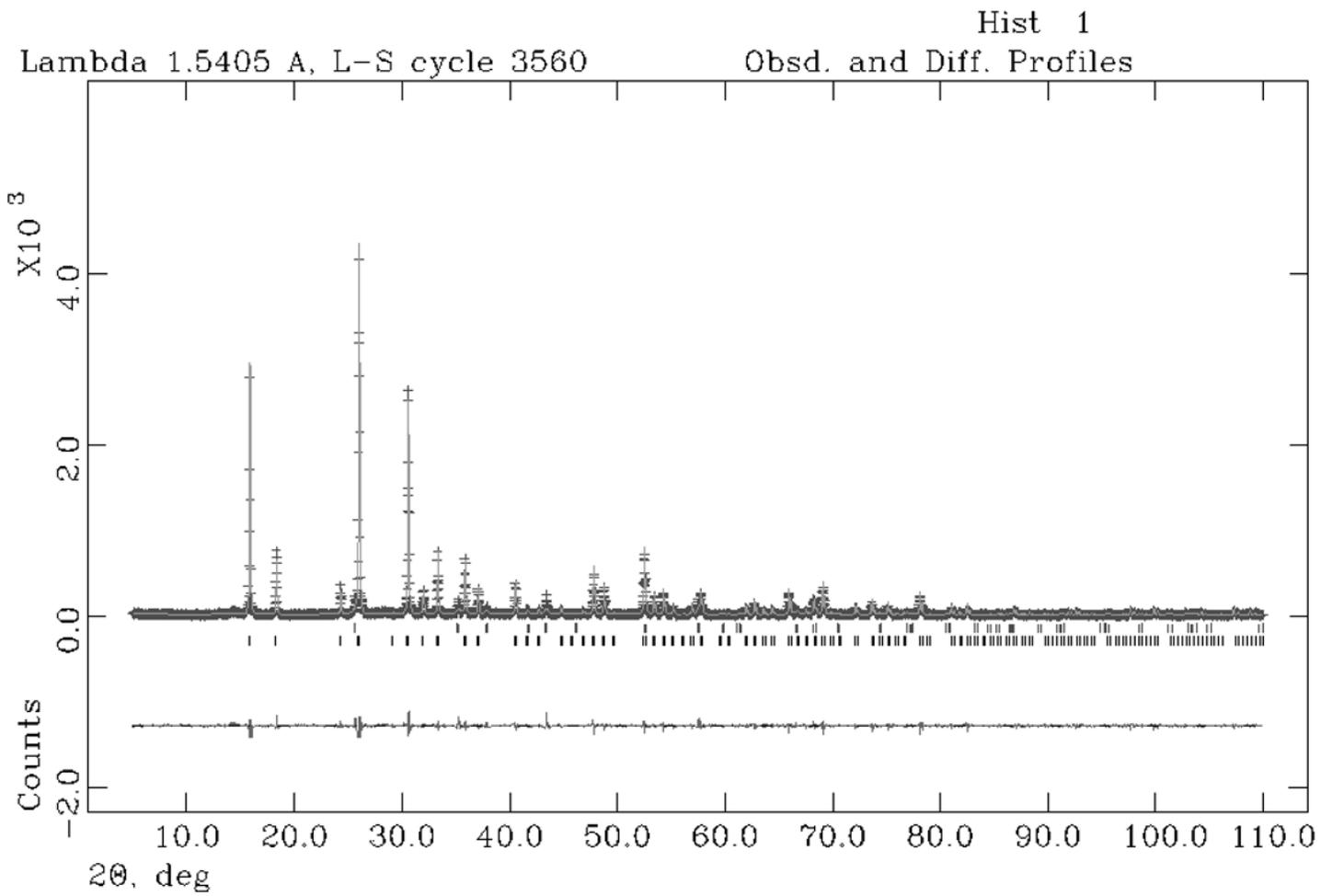


Figure 2

Rietveld refinement plot: Observed (+) and calculated profiles and difference plot for Analcime zeolite (10h at 170 °C) and corundum NIST 676a with tick marks at the position of the Bragg peaks. From the bottom: Analcime zeolite, corundum NIST 676a.

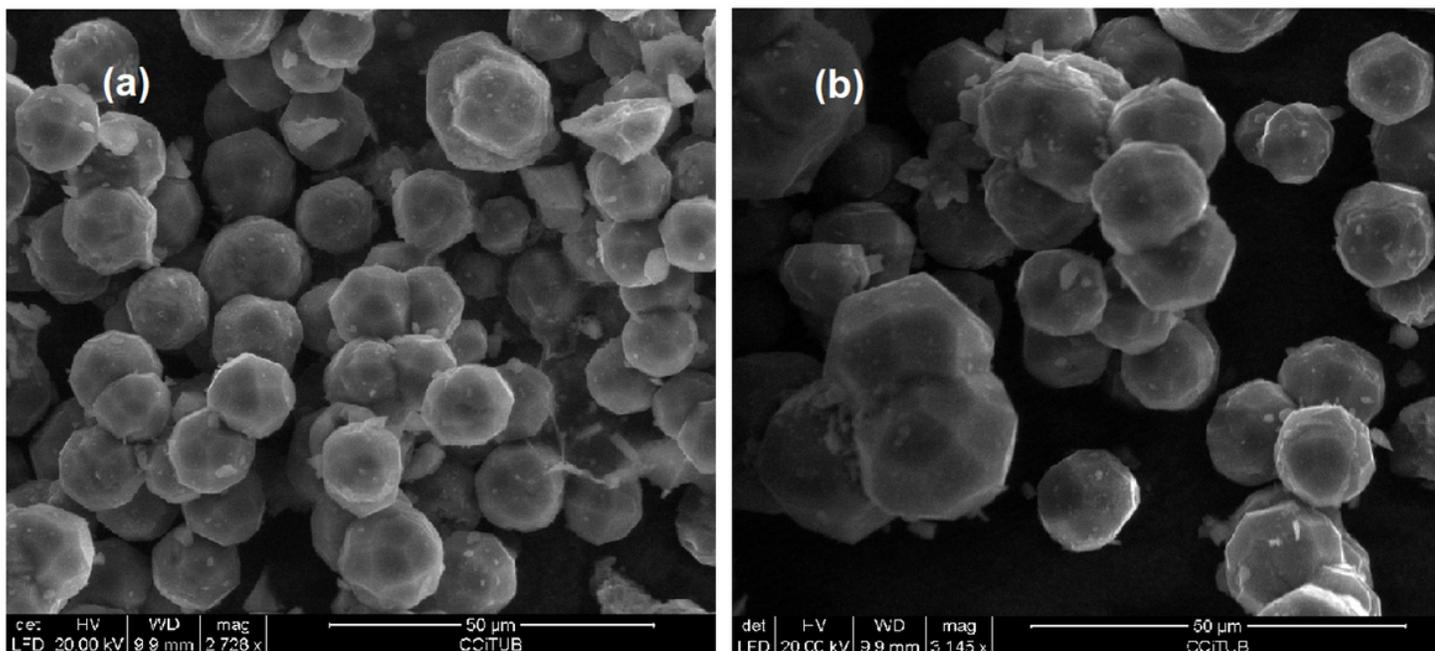


Figure 3

SEM images of ANA zeolite crystals obtained at 5 h (a) and at 10 h (b) of the synthesis run.

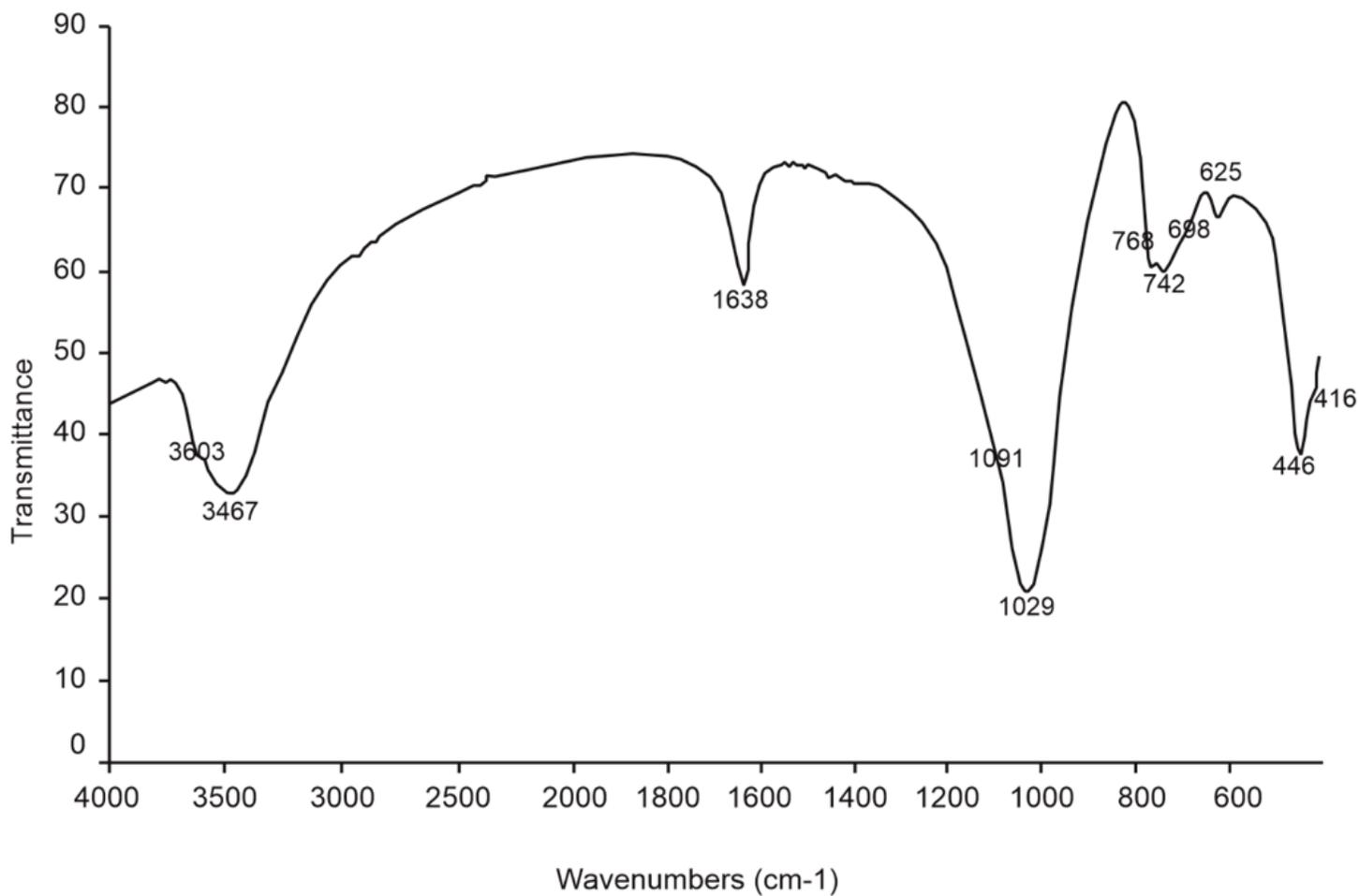


Figure 4

IR spectrum of the Analcime at 10h (170 °C).

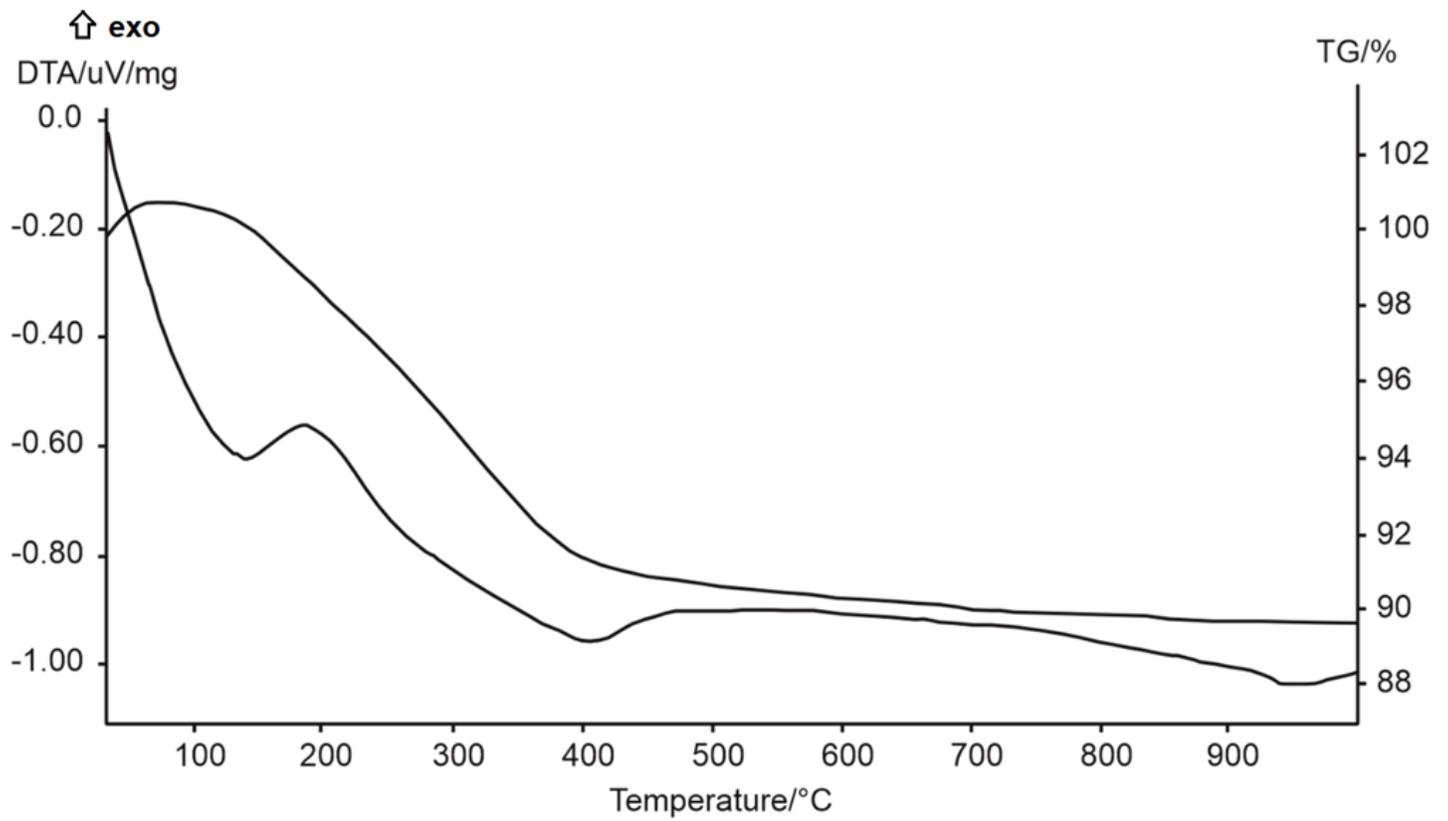


Figure 5

DTA-TG analysis of the sample at 10h (170 °C).