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Thermally Induced Phase Transformation of Mn – LTA and Mn –FAU Zeolite to Anorthite Phases

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Abstract:

Data on thermally induced transformations of Mn exchanged zeolites LTA and FAU topology are presented in this paper. Thermally induced phase transformation of Mn-exchange zeolites are followed in the range from 700 to 1300 °C. Both frameworks collapse into amorphous intermediate products after heating between 600 and 650 °C. Prolonged heating of the intermediate product above 1100 °C results directly in formation of a disorder Mn-anorthite$_{LTA}$ and Mn-anorthite$_{FAU}$. The parameter of unit cell of Mn-anorthite$_{LTA}$ and Mn-anorthite$_{FAU}$, in temperature range between 700 and 1300 °C, was observed in space group C-1. The phase conversions in the temperature range investigated were followed by thermal, X-ray powder diffraction and FT-IR analyses.

Keywords*: Ion exchange of zeolite; Thermal treatment; X-ray powder diffraction analysis.*

1. Introduction

Aluminosilicate ceramic can be used for various applications, such as thermal, chemical, biological and dielectric ones [1]. During the last 80 years of the twentieth century, a new method of synthesizing aluminosilicate ceramic materials by using thermally induced phase transformation of ion transformed zeolites of various topologies was promoted [2-9]. Different methods, like "sol-gel" method [10-12] or hydrothermal synthesis [13], of reaction in the solid state ("*solid - solid*" reaction) [14-16], was the conventional routes for the synthesis of phases in the system $M - SiO_2 - Al_2O_3$ (where: $M = Li^+$, Na^+, K^+, Rb^+, Cs^+ or Ba^{2+}, Ca^{2+}, Sr^{2+}). The problem of these methods was the appearance of phases which are not expected. However, zeolites have a low density aluminosilicate network, which is transformed into structures with a higher density network during thermal treatment. The thermal collapse of zeolite network can be at least directly to produce another crystalline or amorphous phase. All of these characteristics point to the possibility of using zeolites as precursors for the production of high-quality ceramic products. Synthetic zeolites with LTA, FAU, or GIS topologies are the most commonly used zeolites as ceramic material precursors.

The syntheses of alkaline-earth aluminosilicate phases with thermally induced

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transformations of ion exchange LTA or FAU framework zeolites in the system of $MAl_2Si_2O_8$ (M = Ca^{2+}, Sr^{2+}, Ba^{2+} or Pb^{2+}) are presented in the literature [17-20].

Literature data for thermally induced transformations of Mn exchanged zeolites have so far not been published. In the nature, are not known the monomineral species of Mn-zeolite or Mn-feldspar. Content of Mn in natural feldspar is in ranges between 2 and 200 ppm [21]. Eberhard [22] presented the first crystallographic data for synthetic feldspars $MnAl_2Si_2O_8$, [values of the parameters of unite cells were a = 8.1 Å, b = 12.7Å, c = 7.2Å, α = 93.5°, β = 116°, and γ = 90°]. Matsui and Kimata [21], observed the three component system $MnAl_2Si_2O_8 - CaAl_2Si_2O_8 - SrAl_2Si_2O_8$. They applied X-ray analysis to the monocrystal to determine structure of Mn-anorthite with chemical formula $Ca_{0.71}Mn_{0.19}Na_{0.05}Al_{1.86}Si_{2.13}O_8$, The structure of Mn-anorthite is represented by disorder distribution of Si/Al, as well as the appearance of distorsition of average distances of Mn cation. All this leads to a decrease of values the crystallographic axis c and reduces it to 7 Å. The unit cell parameters were a=8.131(2), b= 12.847(3), c= 7.07(1) Å and V=662 $Å^3$.

The work presented here continues previous studies on thermally induced structural conversions of LTA− and FAU− framework zeolites exchanged with alkali metal cation and syntheses of $MAl_2Si_2O_8$ (M= Ca^{2+}, Sr^{2+}, Ba^{2+}, Pb^{2+}) aluminosilicate ceramics [3-6, 17-20]. The interest of this work is focused on the MnO – CaO − Al_2O_3 − SiO_2 phase system. Motive for this work was: *a*.) observe the process of transformation of Mn- LTA and Mn-FAU zeolite during the thermally treatment with X-ray powder diffraction and SEM method *b*.) to investigate the structural (*a*, *b*, *c*, *V*) parameters of new synthesized phasees of Mn-anorthite.

2. Materials and Experimental Procedures

The calcium form of the LTA (Si/Al=1.00) and sodium form of the FAU (Si/Al=1.34) zeolite structure types [23], manufactured by Union Carbide Co., were used as starting materials. The partially-exchanged Mn^{2+} forms of these zeolites were prepared after several successive exchanges from the 0.17 M $Mn(NO_3)_2$ solutions of the solid/liquid ratio (S/L) 1/30. Chemical compositions of the samples were analyzed by an atomic absorption spectrophotometer (AAS), incorporating a Perkin-Elmer 390 instrumental device. The presence of different cations was determined by EDAX analysis. Thermal behavior of the partially Mn-exchanged zeolites was investigated by using a Netzsch simultaneous analyzer; model STA-409 EP and DTA cells. Both starting zeolite precursors were examined at a heating rate of 10 ^{o}/min. A Netzsch-421 type furnace was used for heating the samples at temperatures over 800-1300 ^{o}C for 1h.

The corresponding X-ray powder diffraction patterns were obtained by a Philips PW-1710 automated diffractometer, using a Cu tube operated at 40 kV and 30 mA. The instrument was equipped by a diffracted beam curved graphite monochromatic and Xe-filled proportional counter. The diffraction data of thermally treated samples were collected in the range of 4 to 70° 2θ Bragg angles counting for 1.0 sec in 0.02° steps, for routine phase analysis.

The investigations of crystal morphology of thermally treated partially Mn-synthesized phases were carried out by scanning electron microscopy, using a JEOL 840A instrument. The investigated samples were gold sputtered in by a JFC 1100 ion sputterer.

3. Results and Discussion

3.1. Chemical and DTA/TG analysis

The results of the chemical composition of starting samples and analysis after ion exchange of zeolites with Mn-cation are presented in Table I.

Tab. I The chemical composition of Mn-LTA zeolite determined with AAS and EDAX methods.

Oxide [%] AAS analysis of LTA zeolite					
SiO_2	Al_2O_3	MgO	CaO	Na_2O	H_2O
36.18	28.82	2.42	10.71	5.21	15.98
Oxide [%] AAS analysis of FAU zeolite					
SiO_2	Al_2O_3	MgO	CaO	Na_2O	H_2O
40.20	27.17	1.83	2.36	8.75	18.60
Oxide [%] AAS analysis of Mn-LTA zeolite					
SiO_2	Al_2O_3	MnO	CaO	Na_2O	H_2O
34.62	27.82	18.70	8.63	0.5	10.52
EDAX analysis of Mn-LTA zeolite					
Si	Al	Mn	Ca	Na	O
16.18	14.72	14.48	6.16	0.15	54.36
Oxide [%] AAS analysis of Mn-FAU zeolite					
SiO_2	Al_2O_3	MnO	CaO	Na_2O	H_2O
39.25	29.07	27.01	-	4.34	13.0
EDAX analysis of Mn-FAU zeolite					
Si	Al	Mn	Ca	Na	O
18.34	13.96	13.55	-	3.22	50.93

The chemical analysis of Mn-LTA and Mn-FAU zeolite (see Table I) revealed that the starting zeolitic precursore, were not completely modified with Mn^{2+}. The Ca content of the zeolite of LTA topology remained at 6.16 %, while the Na content of the zeolite of FAU topology remained at 3.22 %. The Si: Al ratio does not change during the ion exchange process; for LTA zeolite, it is 1:1, and for FAU, it is 1:1.25. Thermal stability of ion exchanged zeolite LTA and FAU topology with Mn^{2+} cation, was observed with DTA/TG analysis and the results are shown in Fig. 1.

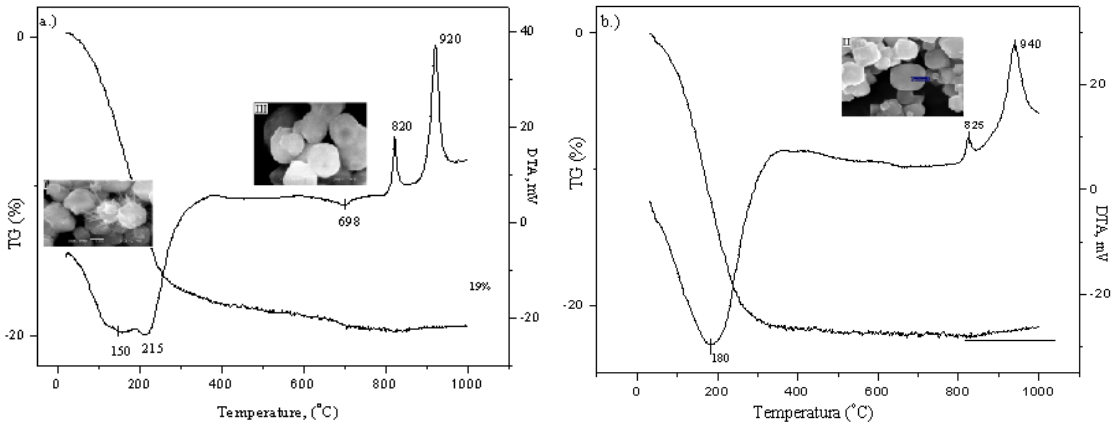


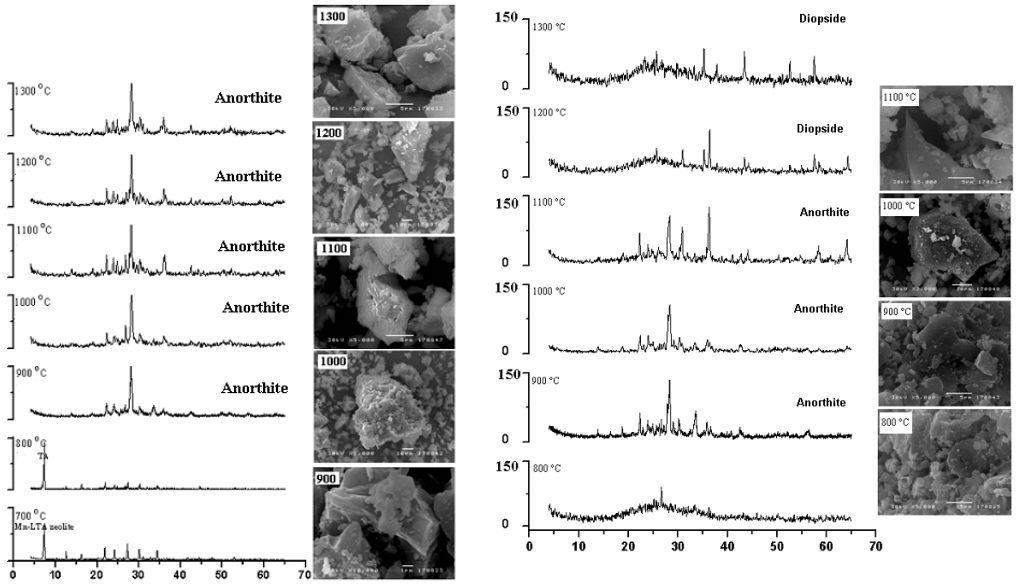
Fig. 1. The DTA/TG diagrams of Mn^{2+} ionic exchange zeolite; a.) Mn-LTA zeolite; b) Mn- FAU zeolite.

The endothermic effects (dehydration processes) in both zeolites are present at temperatures below 200 ^oC (Fig. 1). Endothermic effects in this temperature range (below 200 ^oC), are related to the release of the weakest bound water in the structure of zeolites. As a consequence of amorphization structure for both zeolites at temperature about 820 ^oC the first exothermic peak appears. The second exothermic peak for Mn-LTA is at 920 ^oC and for Mn-FAU zeolite is at 940 ^oC. This temperature around 900 ^oC is the results of crystallization the

zeolite precursore into feldspars topology. Different temperature dehydrations of manganese-zeolite precursors are due to corresponding topological arrangements of Si and Al.

3.2. X-ray powder diffraction and SEM/EDS analysis

During the thermal treatment of Mn-modified zeolites (LTA and FAU topology) at temperature of 900 ^{o}C, new Mn-anorthite phase$_{LTA}$ and Mn-anorthite phase$_{FAU}$ were formed. The structural changes of Mn-anorthite phases were observe during the thermal treatment (from 800 to 1300 ^{o}C) and are presented in (Fig. 2a-b).



a) b)

Fig. 2. The comparative X-ray powder diffraction diagrams of thermal treated zeoiltes a) Mn-LTA framework zeolite in temperature range 700-1300 ^{o}C; b) Mn-FAU framework zeolite in temperature rang 800-1300 ^{o}C.

Tab. II The parameters of unit cell of new anorthite phase for both zeolite precursore in temperature rang 900-1300 ^{o}C.

Mn-anorthite$_{LTA}$							
T(^{o}C)	parameter of unit cell (Å)						
	a	*B*	*c*	*α*	*β*	*γ*	*V(Å^3)*
900	8.131(4)	12.851(4)	7.024(4)	94.03(3)	115.85(3)	90.79(3)	658.04(2)
1000	8.130(4)	12.856(4)	7.014(4)	93.91(3)	116.03(3)	91.07(3)	656.42(3)
1100	8.105(4)	12.824(4)	7.007(4)	93.73(3)	115.96(3)	91.09(3)	652.56(3)
1200	8.110(4)	12.835(4)	7.024(4)	93.47(3)	116.02(3)	91.06(3)	654.778(3)
1300	8.099(4)	12.832(4)	7.007(4)	93.76(3)	116.00(2)	91.07(2)	652.243(3)
Mn-anorthite$_{FAU}$							
	a	*b*	*c*	*α*	*β*	*γ*	*V(Å^3)*
900	8.053(4)	12.753(4)	7.043(4)	95.80(3)	116.10(3)	89.76(3)	645.67(2)
1000	8.034(4)	12.737(4)	7.036(3)	95.74(3)	116.17(2)	89.80(2)	642.3(2)
1100	8.031(4)	12.729(4)	7.032(4)	95.47(3)	116.20(3)	89.97(3)	641.3(3)

The parameters of unit cell for anorthite phases (from LTA and FAU zeolite topology) in a temperature range from 900 to 1300 ^{o}C are presented in Table II. The parameters were refined in space group C-1, using the starting model from literature data [21].

The first phase of transformation during thermal treatment corresponds to the process of dehydration. With increasing the temperature of annealing, mobility of extraframework cations in the structure of zeolite is increasing. The decrease in peak intensity, as well as shifts in d-values (Fig. 2a, diagram 700-800 ^{o}C) are a consequence of cations mobility. With an increase in temperature over 800 ^{o}C, the T–O–T bond breaks and the zeolite structure collapses. These changes lead to distortions of the topological symmetry and involves formation of an amorphous phase. Collapse of the Mn-aluminosilicate framework occurs at different temperatures for Mn−LTA (Fig. 2a between 800 and 900 ^{o}C) and Mn−FAU (Fig. 2b 800 ^{o}C). Based on literature data the processes of Ca–LTA and -FAU zeolite amorphization are completed at about 400 ^{o}C [5], Sr-LTA, Sr-FAU zeolite at about 900 oC [4, 19], Pb–LTA and Pb−FAU zeolite at between 600 and 650 ^{o}C [17].

At temperatures higher than 800 ^{o}C, there is a structural reorganization of the (Si, Al) O_4 tetrahedra, ie SIJ. Based on the literature data [24], it was observed that extrafarmework cations play a significant role in the coordination of tetrahedral rings during structural thermal transformation. The Si–O–Al bridges, D4R SIJ, were found to be brokend in such a way as to leave a cation with six members preserved around the extraframework cation [20, 24]. During formation the stable phase of Ca/Mn/Na anorthite, this symmetrical configuration around the extrafarmework cation Ca is retained, Fig. 3.

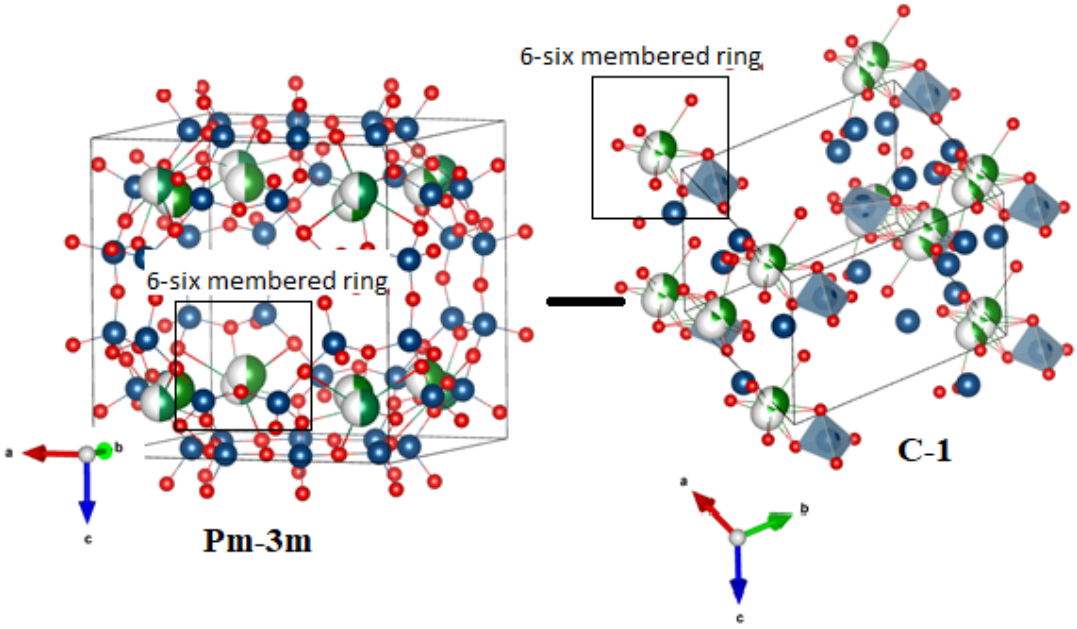
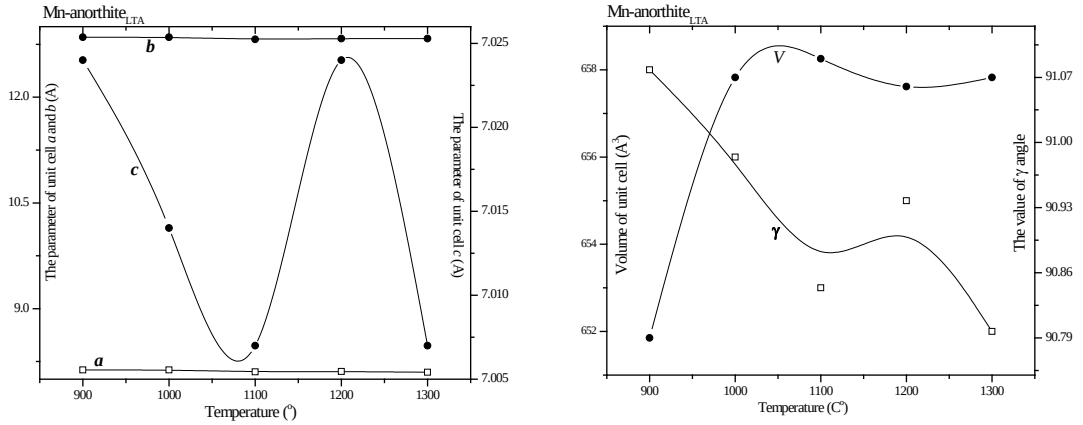


Fig. 3. The symmetrical configuration around the extrafarmework cation Ca in structure of thermal treated Mn-LTA zeolite (in temperature range from roome temperature to 800 ^{o}C).

The final stage of thermal conversion includes the polymorphous transformations to the more stable crystalline phase. After an annealing temperature of 800 ^{o}C, zeolite precursor of LTA topology directly recrystallized to anorthite, which is stable up to temperatures of 1300 ^{o}C. The zeolite of FAU topology was transformed to anorthite gradually, first by forming intermediate−amorphous phase, at a temperature of 800 ^{o}C (Fig. 2a-Fig. 2b). The process of crystallization into anorthite phase occurs by temperature increasing to 900 ^{o}C. Its stability in the process of prolonged heating is temperature/time dependent. The anorthite phase$_{FAU}$ is stable over a small temperature range of 900-1100 ^{o}C. Increasing temperature to 1300 ^{o}C leads to the formation of a phase of mineral diopside. Annealing temperature of 900 ^{o}C leads to gradual crystallization and the crystals have rounded edges and are agglomerated

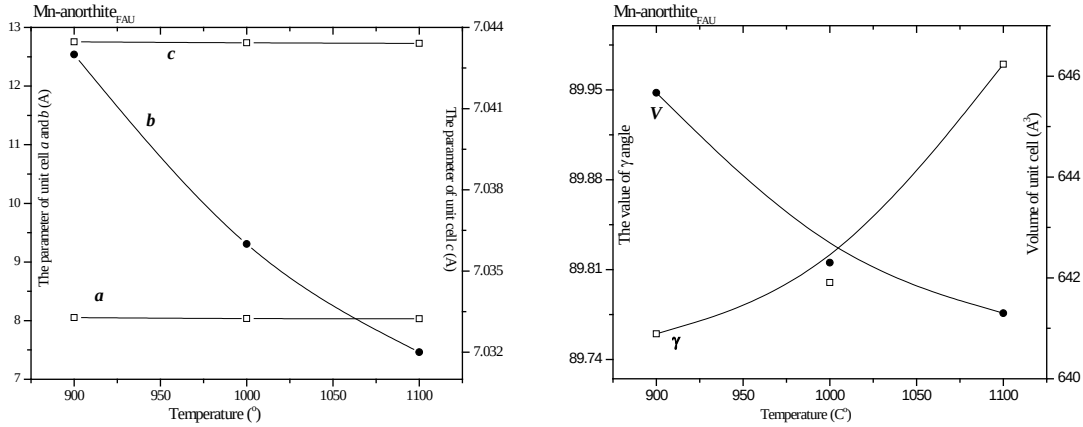
(Fig. 2a-b). The results of this investigation of the partially modified Mn- zeolite whit different topology show that they are completely transformed into the stable structure of anorthite, Mn-anorthite$_{LTA}$ and Mn-anorthite$_{FAU}$ during thermal treatment (in temperature range of 900-1100 ^{o}C).

The parameters of unit cell (Mn-anorthite$_{LTA}$ and Mn-anorthite$_{FAU}$) in correlation with temperature, during the thermal treatment are presented in Fig. 4 and Fig. 5.



a) b)

Fig. 4. The value of unit cell (***a***, ***b***, ***c***, ***V*** and angle **γ**) of Mn-anorthite$_{LTA}$ during the thermal treatment in temperature rang 900-1300 ^{o}C.



a) b)

Fig. 5. The value of unit cell (***a***, ***b***, ***c***, ***V*** and angle **γ**) of Mn-anorthite$_{FAU}$ during the thermal treatment in temperature rang 900-1100 ^{o}C.

During the thermal treatment changes occur along the axis *c* for Mn-anorthite$_{LTA}$ and along axis *b* for Mn-anorthite$_{FAU}$. Based on the literature data, after thermal treatment, the parameters of unit cell in the system of $MnAl_2Si_2O_8$–$CaAl_2Si_2O_8$–$SrAl_2Si_2O_8$ was decreasing and are correlated with ion exchange of Mn/Ca. The results for Mn-anorthite$_{LTA}$ are in accordance with the literature data [21].

Deviations in the anorthite phase, synthesized from the zeolite of the FAU topology, are probably a consequence on of higher concentrations of Na/Mn in relation to cation Ca in the structure. The composition of M cations influenced the structural changes of feldspar,

which had a significant effect on changes in the bond length M–O [25]. It has also been observed, an increase of coefficients of thermal expansion depends on bond length of the Na–O, Ca–O or K–O. To maintain the balance of structure, it is important to establish the relationship between interatomic and tetrahedral positions in the cage [25]. In the structure of Mn feldspar$_{FAU}$ is present 3.22 % Na and 0.2 % Ca (Table I), in the structure of Mn feldspar$_{LTA}$ is present 6.16 % Ca and 0.15 % Na (Table I). Probably the newly formed Na–O and Mn–O bonds, as well as their relationship with the T–O–T bonds lead to changes along the ***b*** axis for Mnanorthite$_{FAU}$ and ***c*** axis for Mnanorthite$_{LTA}$.

Also, was observed values of the gamma angle, which is sensitive to temperature change. Based on the literature data [26, 27], a correlation was established between the value of the gamma angle and content of Al in tetrahedral position. The highest ΔAl value correspond the highest value of γ angle. Lower value of γ angle (91.01) indicates higher degrees of disorder. The thermal treatment of both Mn-anorthite, after 1100 ^{o}C, induced the highest value of γ angle and indicate the disorder distribution in tetrahedral framework. At a temperature of 1100 ^{o}C at which both anorthite phases are present, the angle values are 90.85 for anorthite$_{LTA}$ and 90.2 for anorthite$_{FAU}$. The differences in values of γ angle, are a consequence of different ratio Si/Al in the two structures, the Si/Al ratio for zeolite of LTA topology is 1:1, while for the FAU topology Si:Al ratio is 1:1.25.

3.3. FT-IR analysis

In infrared spectra, the wavenumbers and band intensities depend on the Si/Al ratio, the coordination of Al atoms, as well as the content of extraframework cations in the structure. The FT-IR spectra (in temperature range of 900 to 1300 ^{o}C) of thermal treated Mn-anorthite$_{LTA}$ and Mn-anorthite$_{FAU}$ are presented in Fig. 6.

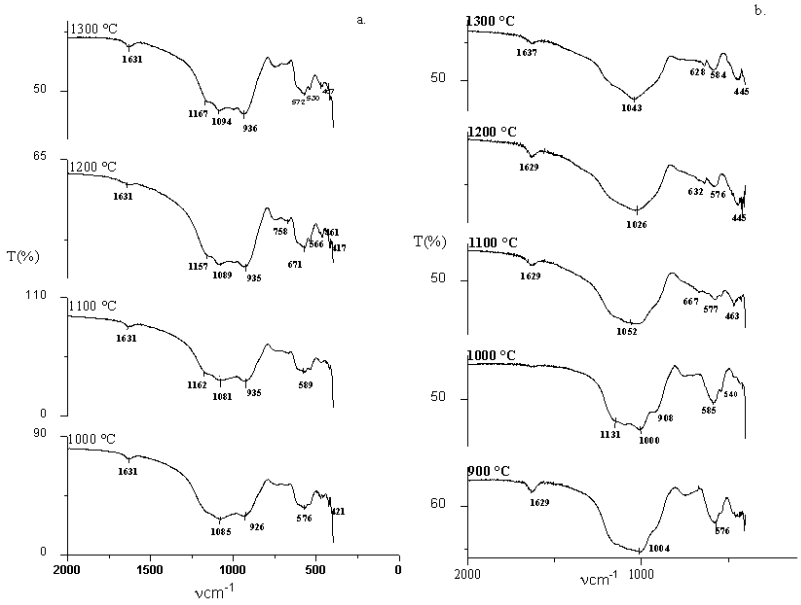


Fig. 6. Comparative FT-IR spectra of thermal treated Mn-anorthite; a) FT-IR spectra of Ca/Mn/Na-anorthite$_{LTA}$ b) FT-IR spectra of CA/Mn/Na-anorthite$_{FAU}$.

The intensive vibration mode in the infrared spectra of the analyzed Mn-anorthite indicates a strong vibrational mode at a frequency of about 1000 cm^{-1}.These vibrations are assigned to the symmetric (v_1) and asymmetric stretching (v_3) vibration of SiO_4 and AlO_4 tetrahedra [28]. The intensity and position of this mode depend on the distribution of Si^{+4}/Al^{+3}

and their coordination in the structure of aluminosilicate. The Si (Al) − O stretching bands, (Fig. 6) show a significant difference between the two anorthite structures, Table III.

Tab. III Characteristic positions of vibration modes in the spectra of Mn anortite$_{LTA}$ and Mn anortite$_{FAU}$, in the spectral range 350-1200 cm^{-1}.

Mn-anorthite$_{LTA}$						
T^a(^{o}C)	v^b (cm^{-1})	vib.mod	v^b (cm^{-1})	vib.mod	v^b (cm^{-1})	vib.mod
1000	1085, 926		665		572, 478, 463	
1100	1081, 935		662		575, 468, 462	v_2
1200	1089, 935	v_1, v_3	660	v_4	570, 468	
1300	1094, 936		660		570,464	
Mn-anorthite$_{FAU}$						
T^a(^{o}C)	v^b (cm^{-1})	vib.mod	v^b (cm^{-1})	vib.mod	v^b (cm^{-1})	vib.mod
900	1004, 908		720		559,532, 438	
1000	1026	v_1, v_3	728	v_4	596, 534	v_2
1100	1060		680		599, 459	

[a] *Temperature*
[b] *vibration band*

The differences in values are a consequence of different ratio Si/Al, Si/Al ratio for zeolite of LTA topology is 1:1, while for the FAU topology Si:Al ratio is 1:1.25. The higher values of vibration mod (v_3) correspond to the vibration along Si−O−Si bond, while the vibrations along Si−O−Al bond are lower [29]. Values of the vibration modes in the region of 660 cm^{-1} correspond to the bending vibration along whit the O–Si (Al)–O bridges [30]. These values increase for Mn-anorthite$_{FAU}$ and correspond to vibrations along the Si–Si (Al) bonds. They probably show the process of ordering distribution of Si/Al atoms.

The intensive vibration mod in range 500-570 cm^{-1} correspond to the mixing symmetric/asymmetric stretching vibration along the Si−O−Si or Si−O−Si−Al bond [31]. This band corresponds to the vibrations of Si(Al)O_4 tetrahedral with double-four membered-tetrahedral rings in zeolite topology [24]. Changes in the intensity of this band in the observed spectra indicate the breaking of Si–O–Al bridges within D4R secondary building units.

The vibration modes in the range of 468-478 cm^{-1} correspond to O–Si–O bending and M–O is stretching vibrations. The most significant spectral variations caused by exchanging Ca, Na or K, occur in this spectral region [32]. Based on the literature data, the presence of vibration between 440 and 450 cm^{-1} may be related to the presence of six-membered rings (S6R) in the tetrahedron. The presence of these vibrations corresponds to a higher Ca and Mn content in the structure, that is, the presence of Ca–O and Mn–O bonds.

4. Conclusion

The thermally induced phase transformation of Mn - modified zeolite is observed. The samples of the LTA-Mn and Mn-FAU zeolites were annealed for 1 hour at temperatures ranging from 800 to 1300 ^{o}C. Increasing the annealing temperature causes dehydration of the Mn - zeolite aluminosilicate structure and network collapse. The amorphization has occured in different temperature, 800 ^{o}C for Mn-LTA and 700 ^{o}C for Mn - FAU zeolite. After the collapse of the structure at a temperature of 900 ^{o}C, the precursor of zeolite of LTA topology

directly recrystallized into anorthite, which is stable up to a temperature of 1300 ^{o}C. The anorthite phase for both samples is stable over a small temperature range of 900 to 1100 ^{o}C. Increasing annealing temperature to 1100 to 1300 ^{o}C leads to the formation of amorphous phase and gradual recrystallization of the minerals from the group of spinels. Based on the results of the X-ray powder diffraction analysis, it can be concluded that values of the unit cell parameters do not change significantly, while parameter γ increases with temperature. Thermal treatment induced the highest value of γ and indicate the disorder distribution in the tetrahedral framework.

Acknowledgments

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Сажетак: *У овом раду су представљени резултати о термички индукованим трансформацијама измењених зеолитских прекурсора Mn–LTA и FAU топологије. Фазне трансформације посматране су температурном опсегу од 700 до 1300 °C. На температурама између 600 и 650 °C долази до аморфизације кристалне структуре зеолитских прекурсора. Након жарења на температурама изнад 1100 °C долази до кристализације нових фаза алумосиликата, Mn-анортита$_{LTA}$ и Mn-анортита$_{FAU}$. Параметри јединичне ћелије одређивани су у просторној групи C-1 (за Mn-анортит$_{LTA}$ у температурном опсегу од 900 до 1300 °C, а за Mn-анортит$_{FAU}$ од 900 до 1100 °C). Метода рендгенске дифракције праха на поликристалном узорку, SEM/EDX, као и метода Инфрацрвене спектроскопије коришћене су за детаљне анлизе ново добијених фаза.*

Кључне речи*: јонска измена зеолита, термичка обрада, анализа рендгенске дифракције праха.*