

Host-Guest Interaction of Iodine with Zeolite A

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Abstract

Introduction of I₂ molecules into zeolite A (sodium form) was made through a three-step procedure: water removal from zeolite, introduction of iodine via gas phase and removal of I₂ from ZA surface by dissolution in absolute ethanol. Interactions of zeolite A saturated with incorporated iodine vapor was studied by vibration spectroscopy (FT Raman and FTIR), DTG and XRD. Results show that about 50% of iodine exists in the framework voids of zeolite A in the form of elemental iodine (molecules, clusters, crystals) and the rest exists in the ionic forms. The ionic forms of iodine (formed by disproportionation of iodine) interact with the negatively charged aluminosilicate framework of zeolite A and with compensating sodium ions. The possible characters of interactions are also discussed.

Keywords: Zeolite A, Iodine, Host-guest interaction, iodine nanocrystals, Raman spectroscopy, DTG

Introduction

The rigid and well-defined three dimensional structures of zeolites, with channels and voids, provide a possibility to introduce different host materials into zeolite crystals.¹⁻⁵ Synthesis pathways and the influence of various organic and inorganic molecules embedded in zeolites on physico-chemical properties of the modified zeolites were studied using a variety of analytical techniques such as XRD,⁶⁻⁹ SAXS/WAXS,¹⁰ SEM,^{6,9} LLS,⁹ NMR,^{7,8} ESR,² TGA/DTG.¹¹ The nature of intermolecular host-guest bonds was dominantly examined through their vibrational properties using FTIR^{4,8,12} and FT Raman spectroscopy.^{4,5,8,12-18,20,24}

Light-sensitive materials are very interesting subjects for investigations because of their potential use in future technologies as light (information) storage and processing at molecular (nano-size) level.^{1,2} Silver halides are well known as light sensitive materials. The crystals of AgX in solution can be dissolved in access of both silver and halide ions forming colorless cationic or anionic species (complex molecules).

Since introduction of iodine into zeolite framework is an important step in the formation of silver iodide based light sensitive materials, the objective of this work is a study of the host-guest interactions of iodine molecules with the framework of zeolite A (sodium form of zeolite A).

Experimental

Materials

The following chemicals were used for the experiments:

- Zeolite A, SILKEM, Kidričevo, Slovenia.
- Doubly sublimated crystalline iodine, KEMIKA, Zagreb, Croatia
- Absolute ethanol, ALKALOID, Skopje, FYR Macedonia.

Samples preparation

Samples S1 and S2 were prepared in a three-step procedure: The first step, dehydration of zeolite was performed as follows: 0.600 g of zeolite A was put into a glass tube (4 cm³) and heated at 550 °C for 3 hours. The dehydrated sample (0.470 g) was then cooled down to room temperature in a desiccator over dry silicagel. Thereafter, predefined amount of iodine crystals (0.700 g for preparation of a sample S1 and 0.140 g for preparation of sample S2) was added into the tube. Iodine was separated from zeolite in the tube by glass-wool layer. Air from tube was evacuated by an oil vacuum pump. The tube was then sealed and heated at 220 °C for 5 hours in laboratory convection oven (second step). The initial pressure of iodine vapor (2.77 MPa for the sample S1 and 0.55 MPa for the sample S2) was calculated using the general gas

equation, neglecting the fact that a part of the iodine was introduced into the zeolite before the temperature of 220 °C was reached. In spite of faster cooling and sublimation of iodine at empty side of the tube, a part of it was sublimed on the zeolite surface and had to be removed by rinsing with absolute ethanol. The samples were rinsed with small portions (*ca.* 20 ml) of absolute ethanol until the effluent was colorless (third step). Samples S1 and S2 were divided into 4 parts; one for characterization and the rest for preparation of samples 3-8 by additional heating. That heating was performed under normal pressure, in the air, for 3 h (see Table 1) at selected temperature: 70 °C (S3, S4), 210 °C (S5, S6) and 410 °C (S7, S8).

Samples characterization

Powder XRD analysis was performed using Phillips PW 1775 powder diffractometer with Cu K $_{\alpha}$ radiation (40 kV, 30 mA, 0.020 degrees s $^{-1}$). Differential thermogravimetry (DTG) measurements were made at Mettler-Toledo TA 4000 system (5 °C min $^{-1}$ in the air). FT Raman spectra were made by Bruker's Equinox 55 Raman spectrometer with a FRA 106/s FT Raman module (Nd:YAG laser, 100 scans) while FTIR spectra were recorded on a Perkin-Elmer System 2000 FTIR spectrometer (KBr wafer, 100 scans).

Results and discussion

Diffusion of iodine into the α and β voids of the zeolite framework began with the air evacuation, and continued more intensely during heating. Introduction of the iodine into the zeolite framework was followed by the color change of samples from white (zeolite A) to dark-red (sample S1 obtained after introduction of iodine into zeolite A at 2.77 MPa) or brown-red (sample S2 obtained after introduction of iodine into zeolite A at 0.55 MPa – see Table 1).

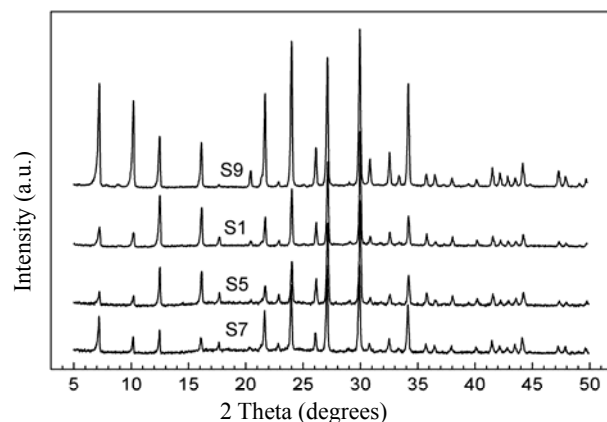


Figure 1. X-ray diffraction patterns of: zeolite A (S9), sample after introduction of iodine into ZA (S1), sample obtained after heating of S1 at 210 °C for 3 h (S5), and after heating of S1 at 410 °C (S7).

Fig. 1 shows that there are no changes in the positions of the diffraction peaks of zeolite A (diffractogram S9)²¹ after introduction of iodine into the framework voids (diffractogram S1). This means that introduction of iodine into the framework voids of zeolite A does not distort the zeolite framework. The change in the relative intensities of peaks is caused by higher electron density of iodine relative to the electron densities of the zeolite framework atoms (Al, Si, O) and compensating sodium ions. The insensitivity of the zeolite framework structure to the presence of iodine is also revealed by analysis of the relevant FTIR spectra; A comparison of the IR spectra S9 (zeolite A)²² and S1 in Fig. 2 shows that introduction of the iodine into zeolite A does not influence the positions of the characteristic IR bands at 381 cm $^{-1}$ and 467 cm $^{-1}$ assigned to 6-R and D4-R secondary building units¹³ of zeolite A framework.

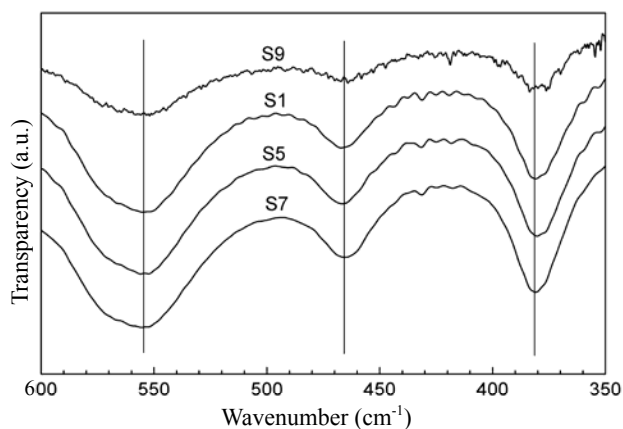


Figure 2. The FTIR spectra of: zeolite A (S9), after introduction of iodine into ZA (S1), sample obtained after heating of S1 at 210 °C for 3 h (S5), and after heating of S1 at 410 °C (S7).

In contrast to insensitivity of XRD and IR spectra on the presence of iodine in the zeolite framework voids (see Figs. 1 and 2), the Raman spectrum of crystalline iodine exhibits two strong stretching bands at 180 cm $^{-1}$ and 189 cm $^{-1}$ (dotted line in Fig. 3). Based on the analysis of the polarized Raman spectra of single iodine crystals, Congeduti et al.,¹⁹ assigned these two bands to the two symmetry modes, A $_g$ and B $_{3g}$.

On the other hand, according to Knops-Gerrits et al.,¹⁷ the most intense bands in the Raman spectra of zeolites are structure sensitive ones which appear between 300 and 600 cm $^{-1}$. These bands are assigned to the motion of an oxygen atom perpendicular to the T-O-T bonds (T = Si, Al) of 4 to 12 member rings; generally - smaller rings correspond to higher frequencies and vice versa. Other framework vibrations appear at higher wave numbers (up to 1210 cm $^{-1}$). Hence, the fact that zeolites are weak Raman scatters^{4,17,18} below

300 cm^{-1} while iodine exhibits very strong bands (non-overlapping Raman bands^{5,19} - see Fig. 3), make this technique very attractive for the study of host-guest interactions in zeolite-iodine systems. This is firmly revealed by the Raman spectra of the samples obtained by introduction of gaseous iodine into zeolite A (solid and dashed spectra in Fig. 3). FT Raman spectra of the sample S1 (solid curve in Fig. 3) and S2 (dashed curve in Fig. 3) have the Raman bands at positions similar to those of crystalline iodine, and thus indicate the presence of molecular iodine in the voids of the zeolite A framework.

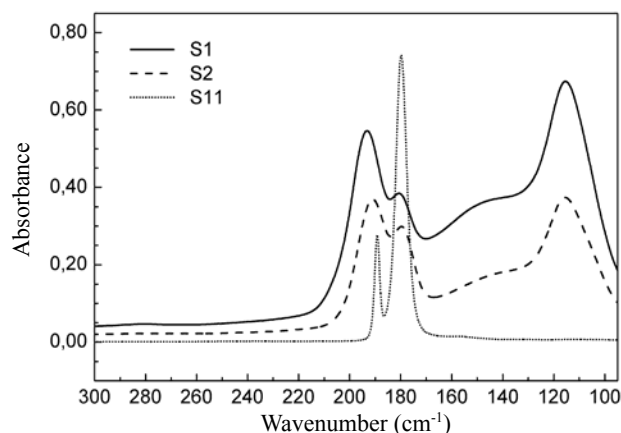


Figure 3. The FT Raman spectra of ZA samples after introduction of iodine with initial pressure of 2.77 MPa (S1), and 0.55 MPa (S2) and spectrum of molecular I_2 crystals (S11).

However, in contrast to crystalline iodine (dotted spectrum in Fig. 3) the band at 189 cm^{-1} in the spectrum of iodine incorporated into the zeolite A framework is stronger than the band at 180 cm^{-1} , and slightly shifted towards higher frequency. Here it should be noticed that: (a) intensity of both peaks decreases with decreasing initial pressure of gaseous iodine (and thus with the amount of iodine incorporated into zeolite), (b) shifting of the peak at 189 cm^{-1} (relevant to crystalline iodine; dotted curve in Fig. 3) towards higher frequency increases with increasing amount of iodine incorporated into zeolite A, (c) both Raman bands of iodine incorporated into zeolite A are broadened relative to the corresponding Raman bands of crystalline iodine (d) the shifted band in both cases (191 and 193 cm^{-1}) is stronger than band at 180 cm^{-1} which is opposite to the bands of pure I_2 crystals, and (e) the Raman spectra of iodine incorporated into zeolite A exhibits a shoulder in the frequency range 125–160 cm^{-1} and a band at 115 cm^{-1} . The specific profile of the FT Raman spectra of the iodine incorporated into zeolite indicates that iodine in the voids of the zeolite A framework exists not only in the crystalline form (bands at 180 cm^{-1} and near 189 cm^{-1}), but also in other different forms (molecules, clusters, *etc.*); some of these forms interacting with

the zeolite framework and/or compensating cations (Na^+). In this context, it is evident that different forms of iodine have different “binding” energies, and thus it can be assumed that the different forms of iodine can be desorbed at different temperatures.

Results of differential thermogravimetric (DTG) analysis show the weight loss of 1.63 wt.% of the mass sample S1 and 1.06 wt.% of the mass of sample S2, during their heating from room temperature to 70 $^{\circ}\text{C}$ (shoulder in the temperature range 25 – 70 $^{\circ}\text{C}$ of the DTG curve in Fig. 4). Here, it is interesting that the FT Raman spectra of both samples (S1 and S2) have the Raman bands characteristic for crystalline iodine (S11 in Fig. 5),¹⁹ after their heating at 70 $^{\circ}\text{C}$ for 3 h, *i.e.*, after removal of the loosely held iodine. This undoubtedly shows that a part of iodine incorporated in zeolite A exists in the form of (nano)crystalline iodine.¹⁹ Also the shoulder in the frequency range 125 – 160 cm^{-1} and the band at 115 cm^{-1} , remained in the Raman spectra of the samples S1 and S2 (solid and dashed curves in Fig. 5) after their heating at 70 $^{\circ}\text{C}$. This leads to the conclusion that the loosely held iodine exists in the form of molecules or even different clusters²⁰ which are responsible for the peaks broadening (see Fig. 3). On the other hand, change in the ratio of the symmetry modes A_g and B_{3g} of the crystalline iodine, followed by the shifting of the band at 189 cm^{-1} towards higher wave numbers (see Fig. 3) are probably caused by weak interactions of iodine molecules/clusters with iodine nano-crystals and/or the aluminosilicate environment of the zeolite framework.

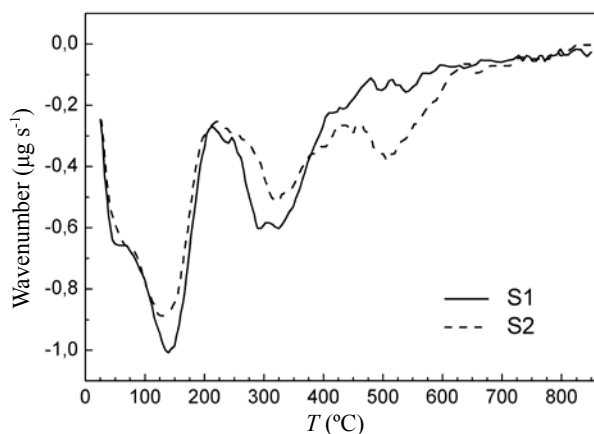


Figure 4. The DTG curves of the ZA samples after introduction of iodine with initial pressure of 2.77 MPa (S1), and 0.55 MPa (S2).

Since the boiling point of crystalline iodine is 184.4 $^{\circ}\text{C}$,²³ it is reasonable to assume that the DTG curve in the temperature range 70 – 210 $^{\circ}\text{C}$ with the temperature peak at 140 $^{\circ}\text{C}$ (see Fig. 4) can be ascribed to the temperature-induced desorption of the crystalline

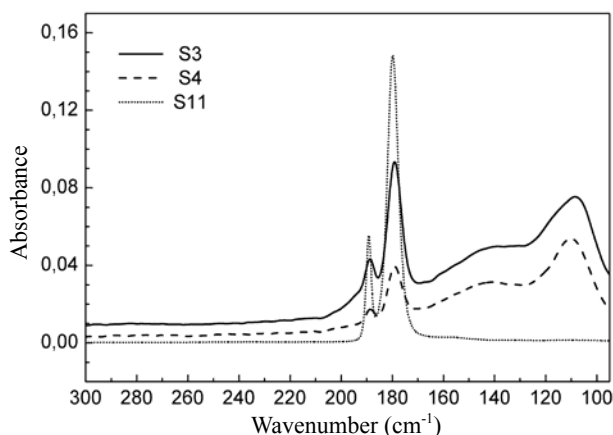


Figure 5. The FT Raman spectra of sample S1 after heating at 70°C for 3 h (S3) and sample S2 after heating at 70°C for 3 h (S4). Spectrum of iodine crystals (S11) is of comparison.

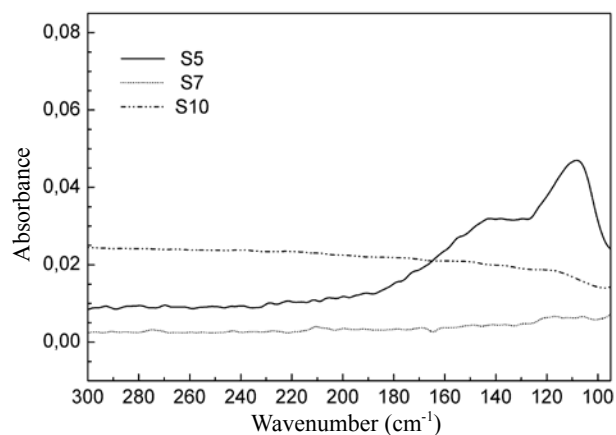


Figure 6. The FT Raman spectra of the samples after heating of S1 at 210 °C (S5) and 410 °C for 3 h (S7). FT Raman spectrum of pure NaI (S10).

iodine. Of course, due to tendency of iodine to sublime even at room temperature, and with respect to the (nano)dimensions of iodine crystals positioned in the α and β cages of zeolite A framework, the removal of crystalline iodine starts at temperatures which are considerably lower than is the boiling temperature.

FT Raman spectra of the samples S1 and S2 heated at 210 °C for 3 h confirm this assumption; the spectra do not have FT Raman bands at 180 and 189 cm^{-1} characteristic for crystalline iodine,¹⁹ but only shoulders in the frequency range 125 – 160 cm^{-1} and the band at 115 cm^{-1} (see Fig. 6). This undoubtedly shows that the weight loss of the samples S1 (15.7 wt.%) and S2 (14 wt.%) in the temperature range 70 °C to 210 °C (see Fig. 4) is caused by the removal of crystalline iodine from the voids in the framework structure of zeolite A. As expected, the desorption of iodine from zeolite A at 210 °C for 3h did not change the peaks position in the XRD pattern of the heated sample S5 (diffractogram S5 in Fig. 1) relative to the peaks positions in the XRD pattern of a fresh sample (diffractogram S1 in Fig. 1) as well as zeolite A (diffractogram S9 in Fig. 1). Also the FTIR spectrum of the sample S1 heated at 210 °C for 3 h (spectrum S5 in Fig. 2) remained unchanged relative to the FTIR spectrum of untreated sample S1 (Fig. 2) and FTIR spectrum of zeolite A (spectrum S9 in Fig. 2).

As described in the Experimental, the total amount of iodine incorporated in the sample during preparation of S1 was 34.04 wt.% and 31.66 wt.% for S2. From the weight loss of the samples during their heating in the temperature range 25 – 70 °C and 70 – 210 °C (see Fig. 4), it can be easily calculated that: (1) 4.8% of the entire amount of iodine incorporated in the sample S1 and 3.4% of the entire amount of iodine incorporated in the sample S2, exists in the form of loosely held iodine molecules/clusters, and (2) 46.2% of the entire amount

of iodine incorporated in the sample S1 and 44.4% of the entire amount of iodine incorporated in the sample S2, exists in the form of (nano)crystalline iodine. This indicates that the pressure of iodine vapor above 0.55 MPa at 220 °C does not have a large influence on the total amount of iodine, on the amount of crystalline iodine and on the amount of iodine, stable above 210 °C, but only on the amount of the loosely held iodine molecules/clusters. The content of the loosely held iodine molecules/clusters is about 54% higher in sample S1 than in sample S2. Here, it is interesting that although both samples initially contain the same amount of iodine, the freshly prepared sample S1 is dark-red while the freshly prepared sample S2 is brown-red. The color of both samples changes to brown (See Table 1) after removal of the loosely held iodine (heating at 70 °C for 3 h), and to yellow after removal of crystalline iodine (heating at 210 °C for 3 h). This indicates that iodine molecules/clusters (< 5% of the total amount of iodine held in zeolite A) and crystalline iodine (44 – 46%) influence the color of the samples much more than the rest of the iodine (about 50% of the total iodine held in zeolite A). Thus the starting color of the samples S1 (dark-red) and S2 (brown-red) is determined by the content of the molecular iodine - loosely held molecules/clusters and iodine nanocrystals.

In contrast to the straightforward explanation of the effects observed during heating of the samples S1 and S2 at 70 °C and 210 °C, the state(s) of iodine remaining in the samples after heating at temperatures > 210 °C is not quite clear. About 34% of the entire amount of iodine introduced in the sample S1 and about 29% of iodine introduced in the sample S2, has been removed from the sample during their heating from 210 °C to 410 °C with the maximum rate of removal at about 300 °C (see Fig. 4). Lower intensity of the DTG “peak” of the sample S2 relative to the intensity

Table 1. Influence of the treatment on the color of the samples.

Sample	Starting material	Starting color	Type of treatment	Color after treatment	Color after DTG (850 °C)
S1	NaZA	white	2.77 MPa I ₂ at 220 °C, 5 h	dark red ^a	white ^b
S2	NaZA	white	0.55 MPa I ₂ at 220 °C, 5 h	brown-red ^a	white ^b
S3	NaZA + I ₂ (S1)	dark red	3 h at 70 °C	brown ^a	white ^b
S4	NaZA + I ₂ (S2)	brown-red	3 h at 70 °C	brown ^a	white ^b
S5	NaZA + I ₂ (S1)	dark red	3 h at 210 °C	orange ^a	white ^b
S6	NaZA + I ₂ (S2)	brown-red	3 h at 210 °C	orange ^a	white ^b
S7	NaZA + I ₂ (S1)	dark red	3 h at 410 °C	yellow ^a	white ^b
S8	NaZA + I ₂ (S2)	brown-red	3 h at 410 °C	pale yellow ^a	white ^b
S9	NaZA	white	-	-	white ^b
S10	NaI	white	-	-	white ^{b,c}
S11	I ₂	dark red	melting	deep purple ^{a,d}	-

^aSamples color before cooling to room temperature.

^bSamples color after cooling in the air and re-hydration (moisture from air).

^cCrystals of NaI remained white all the time (melting point 660 °C, boiling point 1304 °C).²³

^dAfter melting (113.7 °C)²³ I₂ become deep purple, while color of the vapor (184.4 °C)²³ depends on vapor pressure and can be from deep to light purple.²⁰

of the DTG “peak” of the sample S1 corresponds to the lower amount of iodine removed from the sample S2 (29%) relative to the amount of iodine removed from the sample S1 (34% - see Fig. 4). The removal of iodine from the samples S1 and S2 during their heating at 410 °C (upper temperature of the DTG curve in the temperature range 210 – 410 °C; see Fig. 4) was accompanied by the change of color from orange (S3 and S4) to yellow (S5 and S6). This color is caused by the presence of iodine remained in the samples after their heating at 410 °C for 3 h.

Finally, the rest of iodine, that remained in the samples S1 (15%) and S2 (21%) after their heating at 410 °C can be entirely removed from the samples during their heating from 410 °C to about 650 °C, with the maximum removal rate at about 550 °C (see Fig. 4). Again, the lower intensity of the DTG “peak” of the sample S1 relative to the intensity of the DTG “peak” of the sample S2 (see Fig. 4) corresponds to the lower amount of iodine removed from the sample S1 relative to the amount of iodine removed from the sample S2 in the temperature range 410 – 650 °C. After removal of the entire amount of iodine (heating at ≥ 650 °C), both samples are white (see Table 1).

On the other hand, FT Raman spectra of samples S1 and S2 heated at 410 °C for 3 h do not have specific bands and/or shoulders in the frequency range 100 – 300 cm⁻¹ (dotted curve in Fig. 6). This means that the shoulder in the frequency range 125 – 185 cm⁻¹ and the band at 115 cm⁻¹ (solid and dashed curves in Fig. 6) belong to the part of iodine removed from the samples during their heating from 210 – 410 °C. Hence, the fact that about 50% of iodine incorporated into zeolite A can be removed at relatively high temperatures (above

210 °C) leads to the conclusion that about 50% of iodine exists in the elemental form (molecules, clusters, crystals), and that the rest of iodine exists in the form of charged iodine species which are in electrostatic interactions with aluminosilicate zeolite framework and compensating sodium ions. The “mild” colors of the samples heated at ≥ 210 °C (orange after heating at 210 °C and (pale)yellow after heating at 410 °C) corroborates such a conclusion. Namely, it is realistic to assume that relatively high temperature (220 °C) of incorporation of iodine into zeolite A, and the presence of high electrostatic fields (caused by negative charge of aluminosilicate framework and positive charge of Na⁺ ions) can induce disproportionation of a part of iodine molecules to positively and negatively charged iodine species. In this case, negatively charged iodine species (probably I⁻ ions) would interact with Na⁺ ions forming NaI, and positively charged iodine species would replace the “occupied” Na⁺ ions (in NaI) as the compensating “cations” (or complex cation). Since crystalline NaI has a melting point²¹ of 660 °C and taking into consideration that NaI in α and β cages exists rather in molecular than in crystalline form, we assume that the fraction of iodine removed from the samples in the temperature range 410 – 650 °C arises from decomposition of NaI (complex cation). The absence of specific band/shoulder (110 – 185 cm⁻¹) in the FT Raman spectrum of the sample S1 heated at 410 °C for 3 h (S7 - dotted curve in Fig. 6) as well as in the spectrum of pure NaI (dot-dashed curve in Fig. 6) confirms such an assumption. In this context, the shoulder in the frequency range 125 – 160 cm⁻¹ and the band at 115 cm⁻¹ (solid curve in Fig. 6) can be assigned to the positively charged “compensating” iodine species which can be removed from zeolite A during the heating

of the samples in the temperature range 210 – 410 °C. However, the proposed character of the iodine that can be removed from an samples above 210 °C is based on the unproved thesis, at present, and thus will be taken into consideration in the future investigations.

Conclusion

The total amount of the iodine incorporated into the structural voids of zeolite A framework does not dramatically depend on the initial I₂ pressure; samples S1 (0.55 MPa) and S2 (2.77 MPa) contain 31.66 and 34.04% wt., respectively).

Analyses of XRD, DTG, Raman and IR data have shown that about 4% of the entire amount of iodine incorporated in zeolite A, form loosely held iodine molecules/clusters that can be removed from zeolite A in the temperature range 25 – 70 °C, and about 46% of the entire amount of iodine incorporated in zeolite A exists in the form of (nano)crystalline iodine that can be removed from zeolite A in the temperature range 70–210 °C. The fraction of iodine which can be removed from zeolite A at temperatures above 210 °C (about 50% of entire amount of iodine incorporated in zeolite A) probably exists as positively and negatively charged iodine species; negatively charged iodine species (most probably I⁻ ions) are associated with the part of compensating Na⁺ ions (NaI), and positively charged iodine species replace the “occupied” Na⁺ ions as the compensating “cations”.

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Povzetek

Molekule joda (I₂) smo vključili v zeolit A (natrijevo obliko) s tristopenjskim postopkom: iz zeolita smo odstranili vodo, uvedli plinast jod in odstranili površinsko vezan jod z raztapljanjem v etanolu. Interakcijo zeolita A in joda smo proučevali z vibracijsko spektroskopijo (FT Raman in FTIR), DTG in XRD. Ugotovili smo, da je približno 50% joda v zeolitnih prazninah kot elementarni jod (molekule, klastri, kristali), preostanek pa je v ionski obliki. Ionska oblika joda nastane z disproporcionacijo joda. V članku predlagamo možne načine interakcij nastalih ionov z zeolitom A.