



Effect of high-pressures on the electrical resistivity of natural zeolites from Deccan Trap, Maharashtra, India

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Abstract

We report here the electrical resistivity measurements on two natural zeolites—natrolite and scolecite (from the Killari borehole, Maharashtra, India) as a function of pressure up to 8 GPa at room temperature. High-pressure electrical resistivity studies on hydrous aluminosilicate minerals are very helpful in understanding the role of water in deep crustal conductivities obtained from geophysical models. The results obtained by magneto-telluric (MT) soundings and direct current resistivity surveys, along with the laboratory data on the electrical resistivity of minerals and rocks at high-pressure–temperature are used to determine the electrical conductivity distribution in continental lithosphere. The electrical resistivity of natural natrolite decreases continuously from $2.9 \times 10^9 \Omega \text{ cm}$ at ambient condition to $7.64 \times 10^2 \Omega \text{ cm}$ at 8 GPa, at room temperature. There is no pressure-induced first order structural phase transitions in natrolite, when it is compressed in non-penetrating pressure transmitting medium up to 8 GPa. On the other hand scolecite exhibits a pressure-induced transition, with a discontinuous decrease of the electrical resistivity from 2.6×10^6 to $4.79 \times 10^5 \Omega \text{ cm}$ at 4.2 to 4.3 GPa. The observed phase transition in scolecite is found to be irreversible. Vibrational spectroscopic and X-ray diffraction studies confirm the amorphous nature of the high-pressure phase. The results of the present high-pressure studies on scolecite are in good agreement with the high-pressure Raman spectroscopic data on scolecite. The thermo gravimetric studies on the pressure-quenched samples show that the samples underwent a pressure-induced partial dehydration. Such a pressure-induced partial dehydration, which has been observed in natural scolecite could explain the presence of high conductive layers in the earth's deep-crust.

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1. Introduction

Geophysical surveys, especially the electrical and electromagnetic methods are increasingly being used at sites contaminated by organic and inorganic pollu-

tants. (Aristodemu and Betts, 2000; Cozzolino et al., 2000; Atekwana et al., 2000). To interpret, the data obtained by geophysical surveys, laboratory measurements on minerals at controlled thermodynamic conditions, are quite useful. Laboratory investigations of the electrical properties of hydrous silicates have attracted much attention in recent years, because

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they are useful to interpret the electrical properties of lower crust and lower mantle (see e.g., Duba et al., 1997; ELEKTB Group, 1997; Xu and Shankland, 1999; Xu et al., 1998, 2000; Freund, 2002, 2003; Mahadevan et al., 2003). In a recent study, it has been suggested that the electrical resistivity studies on hydrous minerals at laboratory can be successfully applied to interpret the electrical imaging to map clay deposits (Giao et al., 2003). Magneto-telluric investigations in the Killari region of Maharashtra, carried out by Sarma et al. (1994) indicate the presence of anomalous fluid-filled zones at shallow levels in the crust, which could have promoted brittle failure. In the Deccan Trap, zeolites like heulandite, mordenite, scolecite, natrolite and laumontite, are also formed due to late phase hydrothermal activity during the consolidation of lava flows in the west coast geothermal system (Pitale, 1996). These alteration minerals are recorded dominantly as fractures fillings and alteration of vein minerals, which could influence the electrical properties of the rock samples. In order to understand the nature of such deep-seated fluids, we undertook a systematic study of pressure dependence of natural hydrous minerals from the Deccan Trap area.

Natural zeolites are usually produced by processes related to hydrothermal activity or diagenesis, and occur widely in sedimentary rocks and in cavities of igneous rocks. Zeolites and phyllosilicates possess an important property of desorbing water at elevated temperature and regaining it back on cooling to room temperature, and hence they are widely used in industrial applications such as ion exchange materials, molecular sieves and catalysis. The minerals of the natrolite group (natrolite, tetranatrolite, paranatrolite, mesolite, scolecite, thomsonite, gonnardite and edingtonite) are commonly found as later stage crystallization products within cavities in basaltic rocks, in various hydrothermal deposits, and as alteration products of nepheline in nepheline syenites, phonolites and related rocks. These zeolites have the general formula $(\text{Na,Ca,Ba})_{8-16} (\text{Al, Si})_{40} \text{O}_{80} \cdot n \text{H}_2\text{O}$. Their structural properties have been extensively discussed in several monographs (Sand and Mumpton, 1978). The phase stability, the phase changes and the dehydration of zeolites during heating at ambient pressure has been the subject of numerous experimental studies (Gottadi and Galli, 1985). However, there are only very few studies on the effect of pressure at room

temperature on the stability of zeolites (Belitsky et al., 1992; Gillet et al., 1996; Parthasarathy and Gowd, 2000; Ballone et al., 2002; Arletti et al., 2003). Earlier high-pressure studies on natural zeolites based on visual observation, Raman spectroscopic and XRD suggested that natrolite group zeolites should undergo at least three pressure-induced phase transitions, while they are compressed in penetrating liquids up to 5 GPa. They also suggested that pressure-induced irreversible amorphization of scolecite might occur at the pressure of 6 to 7 GPa. The purpose of our present study is to investigate the pressure-induced transitions of natural natrolite and scolecite by the electrical resistivity method up to 9 GPa and to investigate a nature of high-pressure phase of the natural zeolites from Deccan Trap.

2. Geological settings

The samples of the fibrous zeolites, natrolite and scolecite were obtained from the secondary fillings of the Killari borehole of Deccan Flood Basalts (DFB). Deccan flood basalts occupy ~1.5 million km² of the Indian Peninsula. The basaltic pile has been classified into various litho stratigraphic divisions on the basis of geochemical criteria (see Mahoney, 1988; Subbarao, 1999; for summary). In order to understand the subsurface structure of the basement and the possible reason for the occurrence of the large magnitude earthquake at Killari, a borehole has been drilled by National Geophysical Research Institute (NGRI) and Atomic Mineral Division (AMD) Hyderabad. The borehole is located near Killari village, in Deccan Trap (18°03'07" N, 76°33'20" E). The drilling in the DFB province revealed an aggregate thickness of 338 m of basalt. Geochemical characterization shows that the lower four flows (below 178 m depth from the surface) belong to the Poladpur Formation and the rest of the upper five flows to the Ambenali Formation (Gupta and Gupta, 2003), both belonging to the upper most Wai Subgroup of the Deccan basalt Group. All these flows are of aa-type (blocky lavas). Each flow has an amygdaloidal and/or vuggy top, a massive interior and chilled glassy basal part. In some flows, pipe amygdales are observed between the quenched basal part and the massive central part. In addition, there are thin cooling cracks. The secondary

minerals were precipitated from fluids that migrated through the basalts, and there is evidence that most zeolites were formed at temperatures below 150 °C (Jeffery et al., 1988). It is also assumed that fluid was meteoric, ground water rather than hydrothermal in origin. The dissolved material in the fluids must have come from fluid–rock interaction (Parthasarathy et al., 2001). Earlier work on Deccan zeolites have been focused on the possible presence of the zeolite zonation in the Deccan basalts (Sukheswala et al., 1974). In the Western Coast geothermal area, zoning of zeolites stellerite–mordenite–stilbite–scolecite were observed. The water content varies from 7 H₂O (for stellerite) at shallow depth to 3 H₂O (for scolecite) at greater depth of 150 m, indicating a decrease of water content with the increase of depth in the west coast geothermal system (Pitale, 1996).

3. Experimental

High-pressure electrical resistivity studies were carried out in an opposed Bridgman anvil cell system. The Bridgman opposed anvil cell system consists of tungsten carbide anvils with the tip diameter of 6 mm. The sample is embedded in MgO as pressure transmitting medium, surrounding with pyrophyllite gaskets. We used internal pressure standard of high-purity bismuth, which exhibit three pressure-induced solid–solid phase transitions at 2.5, 2.7 and 7.4 GPa respectively. The high-pressure cell was also calibrated by studying the electrical resistivity of manganin (in the pressure range up to 2.5 GPa), pressure-induced phase transitions in tellurium (Parthasarathy et al., 1984; Parthasarathy and Holzappel, 1988) and thallium (Parthasarathy and Gowd, 2000). The typical uncertainty involved in the pressure measurements is 5%. For electrical resistivity measurements, high-purity copper wires were used as electrical probes. The standard four-probe method has been used to measure the electrical resistivity. Keithley instruments calibrator/source meter model No. 263, electrometer (model No. 614), digital multimeter (model No. 199) were used as measuring instruments. Five experimental runs were made to check the reproducibility of the transition pressure. The values of the transition pressure were found to be reproducible within the experimental error. The change in the dimensions of the

sample has been ignored while calculating the absolute value of the electrical resistivity, as the change of resistivity is several orders of magnitude. For the spectroscopic investigations on the pressure-quenched samples we have used sodium chloride as pressure transmitting medium.

The starting materials were characterized by powder XRD, DTA and FT-IR spectroscopic techniques. The chemical compositions of the natural zeolites were characterized by scanning electron microscope with energy dispersive mode and also by electron microprobe method. The composition of the samples (from the cavities in Deccan basalts) were determined to be Na_{15.2} Ca_{0.06} (Al_{15.8} Si_{24.17} O₈₀) · 16.7 H₂O (natrolite) and Ca_{7.8} (Al_{15.8} Si_{24.3} O₈₀) · 23.9 H₂O (scolecite). Sample powders were prepared in acetone medium in porcelain mortar. For powder diffraction studies, the sample powders were smeared onto low background quartz plate. The diffraction patterns were obtained by Philips diffractometer and also Siemens D-5000 powder diffractometer with HOPG graphite monochromator. Cu K α radiation with a wavelength of 0.15406 nm was used in all the diffraction experiments. Differential thermal analysis and thermogravimetric studies were performed on about 15 mg powder sample using a Mettler Toledo Star System apparatus. The analyses were performed in the temperature range between 300 and 900 K at a heating rate of 10 K/min. Triplicate runs were made in static air atmosphere. Fourier transform-infrared spectroscopic (FT-IR) studies on the silica samples were carried out in ambient conditions, using BIORAD 175C FT-IR spectrometer adopting potassium bromide pellet method. Samples were scanned in the frequency range of 4000 to 300 cm⁻¹.

4. Results

Fig. 1 shows the powder X-ray diffraction pattern of natrolite sample, exhibiting 26 well resolved diffraction peaks, all of which can be indexed to an orthorhombic cell with unit cell parameters $a = 1.830$ (2) nm, $b = 1.863$ (2) nm and $c = 0.660$ (1) nm; (Fdd2). The values of the cell parameters are found to be in agreement with those for synthetic pure natrolite $a = 1.829$ nm; $b = 1.864$ nm; $c = 0.659$ nm. The chemical composition studies show the samples are homo-

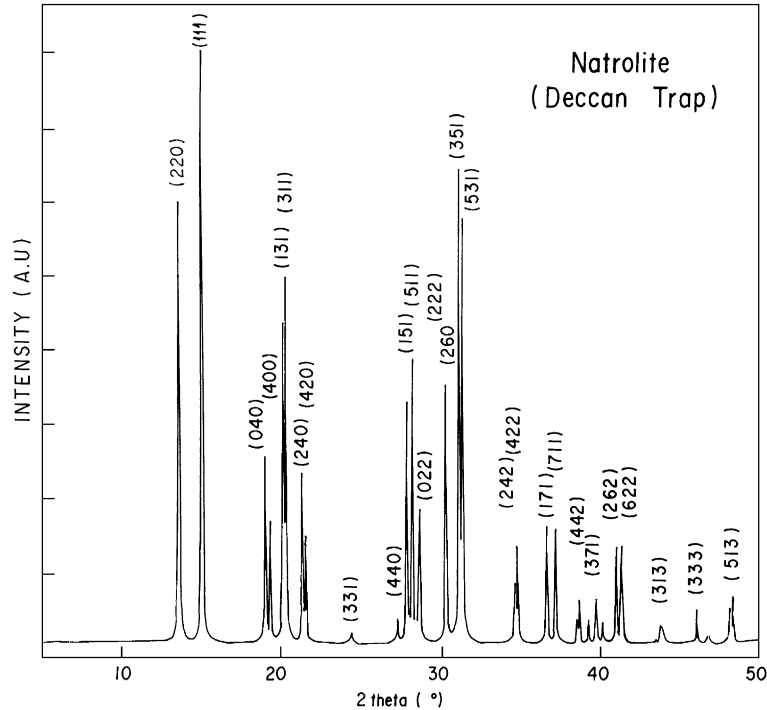


Fig. 1. Powder X-ray diffraction pattern of natural natrolite from Deccan Trap.

geneous and almost pure. The compositional purity of the natural zeolites from Deccan basalt has also been observed by the other workers (Subbarao, 1999). Powder XRD studies on the scolecite from Killari borehole showed 46 diffraction peaks, all of which were indexed to a monoclinic cell with the cell dimensions $a=0.6522$ nm, $b=1.8968$ nm, $c=0.984$ nm and $\beta=110^\circ$ with the space group C1c1 (Fig. 2).

Fig. 3 shows the pressure dependence of the electrical resistivity of natrolite and scolecite at room temperature. Usually the conductivity decreases with the increase of pressure, due to the decrease of porosity and the reduction of possible interstitial migration of ions. However, the electrical resistivity of the studied natural zeolites is found to be decreasing with the increase of pressure, which could be due to the pressure-induced partial dehydration and permanent densification of the zeolites. The natrolite sample is found to be more conducting than scolecite throughout the pressure range of the investigation. This is due to the fact that the ionic mobility of Na-ions is in natrolite. The electrical resistivity of natrolite decreases from 10^9 Ω cm at 0.5 GPa to 10^3 Ω cm at 8 GPa. The initial

increase in the electrical resistivity with pressure up to 0.5 GPa, is caused by ionic conduction in natrolite at low-pressures. Earlier high-pressure studies using water, glycerol, and perfluoroether showed that natrolite compressed in water undergoes two pressure-induced transitions I–II at 0.75 GPa with $\delta V=+6\%$ and natrolite II–natrolite III at 1.25 GPa with $\delta V=-3\%$ and no further transition up to 5 GPa. However, when natrolite is compressed in a non-penetrating pressure medium, there is no phase transition up to 5 GPa. The absence of any phase transition in our sample up to 8 GPa indicates that the pressure-transmitting medium (MgO) did not diffuse within the sample during the investigation.

High-pressure behaviour of scolecite is found to be distinctly different from that of natrolite (Fig. 3). The electrical resistivity of scolecite decreases exponentially with the increase of pressure up to 4.2 GPa and drops discontinuously from 2.6×10^6 to 4.79×10^5 Ω cm at 4.2 to 4.3 GPa, indicating a pressure-induced transition in scolecite. Gillet et al. (1996) have observed that, upon compression at room temperature, scolecite undergoes a series of

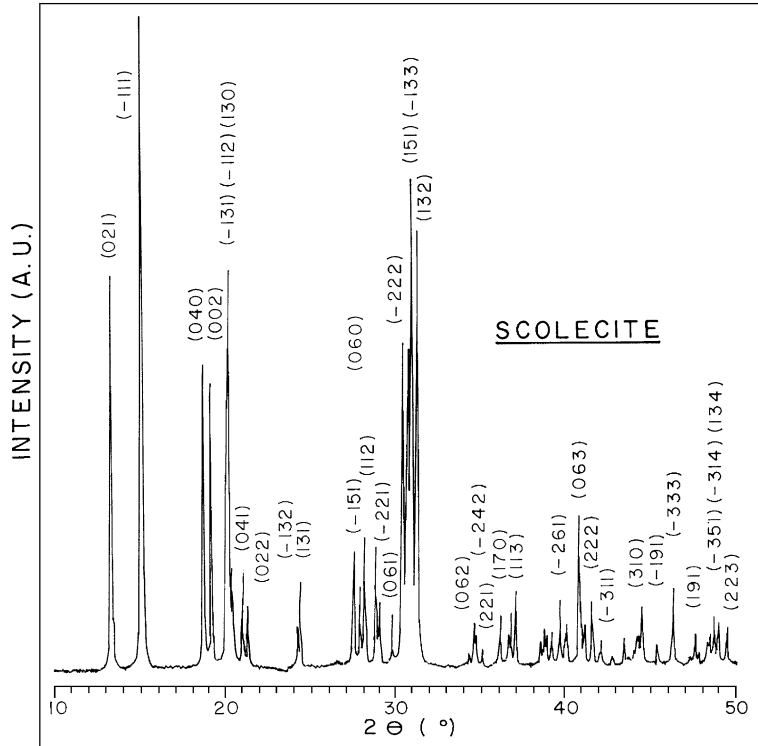


Fig. 2. Powder X-ray diffraction pattern of natural scolecite from the Deccan Trap.

modifications. Raman spectra of scolecite do not vary much up to 1.8 GPa. Between 2 and 4 GPa slight changes like band broadening and peak disappearing were detected in the 100–350 cm^{-1} region. Beyond 6 GPa, new broadband appears indicating amorphization of scolecite. Gillet *et al.* (1996) have observed that the volume changes of scolecite during compression prior to pressure-induced amorphization are essentially achieved by closure of the T–O–T angles to 130°. Further closure of these angles cannot occur probably because of repulsion between O and Si atoms. Zeolites accommodate further compression by a disordering process leading ultimately to an amorphous solid. The observation of the low value of the transition pressure for the phase transition in scolecite indicates that the shear stresses have important role in controlling the pressure-induced transitions in natural zeolites.

High-temperature differential thermal analysis (DTA) and thermo gravimetry (TG) traces of scolecite are shown in Fig. 4, exhibiting two main water loss at room-pressure: the first one at 247 °C, the second one

at about 435 °C with the enthalpy change of 81.63 and 49.87 kJ/mol, respectively. The first dehydration initiated at about 157 °C, shows a single endothermic peak with a maximum at 247 °C. During this first dehydration process the water loss has been estimated to be 44% and the remaining 56% water is removed during the second dehydration process with a transition temperature of 435 °C. The third endothermic peak at about 540 °C is due to the irreversible thermal amorphization of scolecite (Fig. 4). These endothermic peaks can be attributed to the loss of two types of combined water within the voids of the minerals. One type corresponds to the water coordinated around Ca^{2+} ion (8 molecules), the second one to water coordinated around Na^{+} ion. These water molecules have according to the coordination distance, different forces of interaction with their neighbourhood and hereby also different dissociation energies. The samples subjected to 7 GPa show only one endothermic peak at about 480 °C, which could be due to the dehydroxylation and phase transition of amorphous scolecite. The high-pressure amorphous scolecite

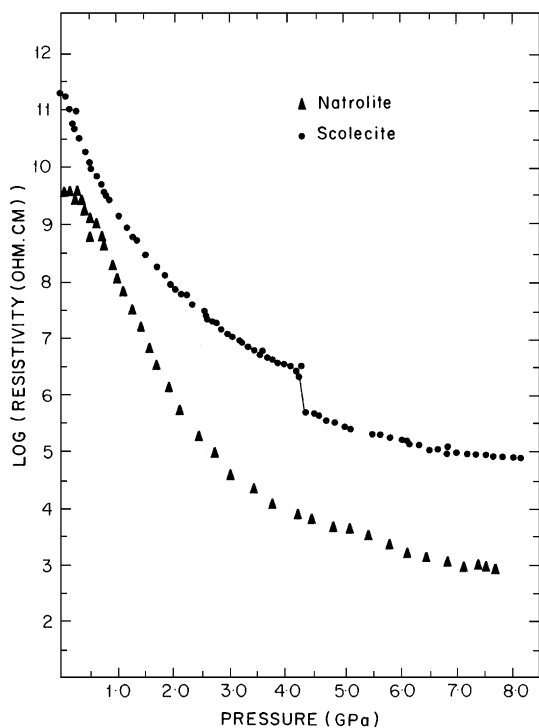


Fig. 3. Pressure dependence of the electrical resistivity of natural natrolite and scolecite up to 8 GPa at room temperature.

exhibits only weight loss of 7.6 wt.% up to 800 °C, indicating the partial dehydration of the scolecite at high-pressure and temperature.

The thermal behaviour of natrolite showed only one water loss at about 360–380 °C and the water loss begins at 350 °C. Alberti and Vezzalini (1983) have studied single-crystal XRD of natrolite at high-temperatures and showed that natrolite transforms to α -metanatrolite near 160 °C. Further heating to 510 °C results in inverse transformation to β -metanatrolite (Belitsky et al., 1992). In metanatrolite, Na atoms occupy positions near those occupied by H₂O in natrolite. It is interesting to note that the amorphous phase has not been found in natrolite up to 800 °C. Dehydrated natrolite has a contracted structure with highly deformed channels. Although, scolecite and natrolite belong to the same class of fibrous aluminosilicate zeolites, their high-temperature behaviour is found to be distinctly different.

High-temperature electrical behaviour of natrolite has revealed that the dehydration starts at 280 °C and ended at 450 °C. The process is indicated by change

of slope in the Arrhenius plot of log (resistivity) against reciprocal temperature plot. The conductivity activation energy was found to be 1.7 eV. During the cooling cycle, the electrical resistivity of natrolite lies far below the heating curve and is characterized by a small activation energy (~0.5 eV), indicating the partial dehydration of natrolite and an irreversible conversion of natrolite to metanatrolite.

We have also carried out powder-X-ray diffraction and FT-IR spectroscopic studies on the pressurized samples of scolecite. Our studies confirm the pressure-induced amorphization of scolecite at 4.5 GPa. The infrared spectra of starting material of scolecite and its high-pressure phase are shown in Fig. 5(a) and (b). At room temperature and pressure scolecite exhibits five distinct infrared peaks in the frequency range 3000 to 4000 cm⁻¹ (Fig. 5a). These bands are attributed to the symmetric stretch of OH vibrations of the water molecules at crystalline site. At high-pressure the lattice gets deformed due to pressure-induced amorphization, which has been demonstrated by the observation of very broad absorption bands centred at 3580 and 3400 cm⁻¹ (Fig. 5a). The spectra obtained for high-pressure scolecite is found to be similar to that of high-temperature amorphous phase of scolecite.

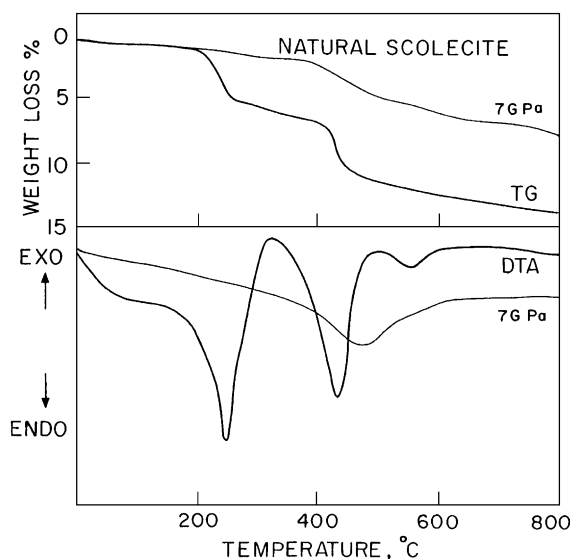


Fig. 4. Differential thermal analyses (DTA) and thermogravimetric (TG) traces of scolecite (both starting material as well as the samples subjected to 7 GPa pressure) at high-temperatures.

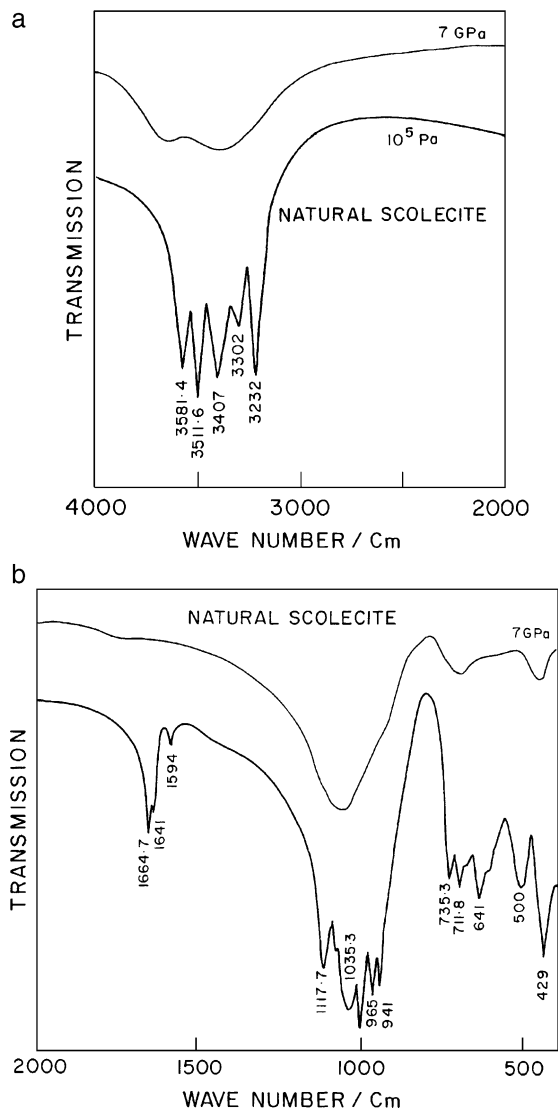


Fig. 5. (a) Fourier-transform infrared (FT-IR) spectra of natural scolecite (lower spectrum is for starting material and the upper spectrum is for the sample subjected to 7 GPa). Spectrum a shows the absorption bands in the frequency range 2000 to 4000 cm^{-1} . (b) The FT-IR spectra of natural scolecite in the frequency range 400–2000 cm^{-1} .

The vibrational bands at 1664.7 and 1641 cm^{-1} of scolecite are attributed to bending modes of H_2O ; the bands at 1117 to 941 are attributed to the antisymmetric stretching modes of aluminosilicate tetrahedra; the bands in the frequency range 735 to 710 cm^{-1} are assigned to the symmetric stretching vibration of Al–Si–O bonds. The libration mode of H_2O is

observed at 500–425 cm^{-1} (Fig. 5b). It is clear from the FT-IR spectrum of the pressure-quenched scolecite (Fig. 5b) that the high-pressure phase of scolecite is less hydrous and amorphous in nature. The broad bands at 1000, 740, 500 cm^{-1} (Fig. 4b) and 3580 and 3400 cm^{-1} (Fig. 5a) were identical to the spectra of typical hydrous aluminosilicate glasses. The chemical and infrared spectroscopic studies on the pressurized scolecite indicate that only a small amount of water about 0.8% were removed in the pressure range of 1 to 5 GPa.

5. Discussion

High-pressure studies on zeolites can provide important insights into the general properties of aluminosilicate frameworks for several reasons. Pressure is energetically a far stronger driving force than temperature and thus one can explore a far greater range in structural behaviour by compressing the structure rather than heating it. Pressure allows the repulsive regime of the inter-atomic potential to be explored directly. Pressure can also induce structural change and a dense amorphous phase synthesised at room temperature by merely applying high-pressure. For example a dense amorphous phase of hydrocalcite, which can be used as radioactive sink material is synthesised by applying pressure of 6 GPa (Parthasarathy et al., 2002). The decrease of resistivity of the natural zeolites (Fig. 3) with increase of pressure can be explained in terms of increase in mobility of charge carriers, i.e., sodium cations. It is well known that the significance of the zeolite water is its location not in the sites of the crystalline lattice, but in the channels of the aluminosilicate framework of zeolites. Hence a gradual extraction of water is effected through the channels, accompanied by certain reduction in density but with retention of structural volume. As a result of dehydration, therefore, channels are deformed, possibly creating more favourable conditions for greater mobility of current carriers. Since the most probable carriers are sodium cations, whose ionic radius is rather large ~ 0.103 nm, and which therefore needs space for migration. The formation of channels in the natural zeolites, is the factor stimulating the diffusion of sodium-ions and increasing their mobility. In a

recent study on the conductivity behaviour of hydrous and anhydrous silicic melts, Gaillard (2004) has suggested that both the mobility and content of sodium alone could contribute to the high electrical conductivity of most terrestrial silicate melts at mantle conditions.

The number of current carriers increases simultaneously, since water molecules are associated with sodium ions and their elimination reduces the number of bonds, and consequently, the binding energy of sodium ions. This fact is confirmed by the observation of anisotropic “swelling” of zeolite crystals under high-pressure. It is also associated with reversible phase transformations of the polyhedral type. Recent experiments on high-pressure behaviour of zeolites, with different pressure transmitting media like perfluorodibutyl ether and water suggest that enhancement of molecular mobility in the water (as media) is caused by an additional H₂O sorption due to pressure (Moroz et al., 2001). This means the water mobility enhances with the pressure, which causes the resistivity decrease with loading. In present experiments we used only solid pressure-transmitting medium, which is not only non-penetrating but also reduces the transition pressure due to non-hydrostatic stress. The high-pressure experiments, using silicon oil as a non-penetrating pressure-transmitting medium showed pressure-induced disorder at 8.5 GPa at room temperature. However, the compressibility increases discontinuously at about 6 GPa, which was attributed to reorganization of the hydrogen-bonding network, with the formation of water dimers. (Ballone et al., 2002). This non-hydrostatic stress is responsible for the observation of lower transition pressure for scolecite at 4.2 GPa compared to 5.2 GPa in hydrostatic medium. It has been suggested that the pressure-induced increase in coordination number of Al atoms from 4 to 5 probably triggers the irreversible pressure-induced amorphization. The present studies on high-pressure behaviour of natural zeolites are useful in understanding the dehydration behaviour of natural zeolites at a greater depth. Also in recent times rock metamorphism and earthquakes are linked in the context of rock failure due to the high-internal fluid pressures, and decrease of rock strength as the consequence of the rapid release of water from the hydrous minerals at high-pressures (Kornprobst, 2002).

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