

**A short review of the formation,
stability, and cementing properties
of natural zeolites**

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Högskolan i Luleå 1977-10-03

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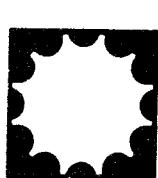
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A SHORT REVIEW OF THE FORMATION, STABILITY AND CEMENTING PROPERTIES OF NATURAL ZEOLITES

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A SHORT REVIEW OF THE FORMATION, STABILITY, AND
CEMENTING PROPERTIES OF NATURAL ZEOLITES

Introduction

The buffer mass for embedding nuclear waste canisters way down in tunnels or bore holes must fulfil a number of requirements. A 10-15% (by weight) sodium bentonite and 85-90% quartz mass has been suggested by Pusch & Jacobsson in a number of reports and this composition is at present the major alternative of the KBS commission. In most respects this buffer mass has excellent properties but its ion exchange capacity is moderate or low. For this reason zeolites have been suggested as an active component in the mass. The question has been, however, whether zeolites are chemically stable together with other minerals such as montmorillonite which still has to be used to give the buffer mass suitable plastic and permeable properties. It is the main object of this report to give an answer to this question and to illustrate any behaviour of zeolites relevant to the problem of finding a suitable buffer mass.

The author's intention has been to find out from geological formations containing zeolites, information concerning their genesis, environment at and after their formation, and their stability as single minerals or in a mixture with others.

Artificially zeolites will not be discussed as they will not give us the information we look for as regards to geological circumstances.

Table 1. Simple zeolite classification (Meier, 1968).

| Species | Idealised unit cell Contents | Isostructural species |
|--|--|---|
| <i>Analcime group</i> <u>Analcime</u> | $\text{Na}_{16}[\text{Al}_{16}\text{Si}_{32}\text{O}_{96}], 16\text{H}_2\text{O}$ | Wairakite, leucite, pollucite, viscite, kehoite |
| <i>Natrolite group</i> Natrolite Thomsonite Edingtonite | $\text{Na}_{16}[\text{Al}_{16}\text{Si}_{24}\text{O}_{96}], 16\text{H}_2\text{O}$ $\text{Na}_4\text{Ca}_8[\text{Al}_{20}\text{Si}_{20}\text{O}_{80}], 24\text{H}_2\text{O}$ $\text{Ba}_2[\text{Al}_4\text{Si}_8\text{O}_{20}], 8\text{H}_2\text{O}$ | Mesolite, scolecite Gonnardite |
| <i>Chabazite Group</i> Gmelinite <u>Chabazite</u> | $\text{Na}_4[\text{Al}_4\text{Si}_{16}\text{O}_{64}], 24\text{H}_2\text{O}$ $\text{Ca}_2[\text{Al}_4\text{Si}_8\text{O}_{24}], 13\text{H}_2\text{O}$ | |
| Erionite Levynite | $(\text{Ca etc.})_{1.5}[\text{Al}_6\text{Si}_2\text{O}_{72}], 27\text{H}_2\text{O}$ $\text{Ca}_3[\text{Al}_3\text{Si}_{12}\text{O}_{36}], 18\text{H}_2\text{O}$ | Linde T |
| Cancrinite hydrate Sodalite hydrate | $\text{Na}_6[\text{Al}_6\text{Si}_6\text{O}_{24}], 5\text{H}_2\text{O}$ $\text{Na}_6[\text{Al}_6\text{Si}_6\text{O}_{24}], 4\text{H}_2\text{O}$ | Nosean, Zhdanov's G, danalite, tugtupit?, (synthetic Ca-alumino-sulphate) |
| <i>Phillipsite group</i> <u>Phillipsite</u> Gismondite Barrer's Pt | $(\text{K, Na})_{10}[\text{Al}_{10}\text{Si}_{20}\text{O}_{80}], 20\text{H}_2\text{O}$ $\text{Ca}_4[\text{Al}_4\text{Si}_8\text{O}_{32}], 16\text{H}_2\text{O}$ $\text{Na}_6[\text{Al}_6\text{Si}_{10}\text{O}_{36}], 15\text{H}_2\text{O}$ | Harmotome Linde B, garronite (?) |
| <i>Heulandite group</i> Brewsterite <u>Heulandite</u> Stilbite | $(\text{Sr, Ba, Ca})_2[\text{Al}_4\text{Si}_{12}\text{O}_{36}], 10\text{H}_2\text{O}$ $\text{Ca}_4[\text{Al}_4\text{Si}_8\text{O}_{24}], 24\text{H}_2\text{O}$ $\text{Na}_2\text{Ca}_4[\text{Al}_{10}\text{Si}_{26}\text{O}_{72}], 28\text{H}_2\text{O}$ | <u>Clinoptilolite</u> |
| <i>Mordenite group</i> Mordenite Dachiardite Epistilbite Ferrerite Bikitaitte | $\text{Na}_8[\text{Al}_8\text{Si}_{16}\text{O}_{64}], 24\text{H}_2\text{O}$ $\text{Na}_5[\text{Al}_5\text{Si}_{19}\text{O}_{65}], 12\text{H}_2\text{O}$ $\text{Ca}_3[\text{Al}_6\text{Si}_{14}\text{O}_{42}], 16\text{H}_2\text{O}$ $\text{Na}_2\text{Mg}_2[\text{Al}_6\text{Si}_{10}\text{O}_{72}], 18\text{H}_2\text{O}$ $\text{Li}_2[\text{Al}_2\text{Si}_4\text{O}_{12}], 2\text{H}_2\text{O}$ | |
| <i>Faujasite group</i> <u>Faujasite</u> Linde A ZK-5 Paulingite | $(\text{Na}_2, \text{Ca})_{32}[\text{Al}_{64}\text{Si}_{128}\text{O}_{384}], 256\text{H}_2\text{O}$ $\text{Na}_{198}[\text{Al}_{98}\text{Si}_{98}\text{O}_{384}], 216\text{H}_2\text{O}$ $\text{Na}_{24}[\text{Al}_{24}\text{Si}_{72}\text{O}_{192}], 90\text{H}_2\text{O}$ $(\text{K}_2, \text{Ca}, \text{Na}_2)_{152}[\text{Al}_{152}\text{Si}_{320}\text{O}_{772}], \sim 700\text{H}_2\text{O}$ | Linde X, Linde Y ZK-4 |

Some of the more important zeolites we have to deal with and which are of special interest to us are classified in Table 1 according to Meier (1968). Table 2 gives us a more practical classification.

Analcime or analcite, phillipsite, chabazite and clinoptilolite have been suggested as possible components in our buffer mass.

Since we will deal a little with geological periods of time in this report Table 3 is given here to show the terminology.

Table 2.

FORMULAE AND AVERAGE CHEMICAL COMPOSITIONS OF ZEOLITES MOST ABUNDANT IN SEDIMENTARY ROCKS (data from many sources). The compositions of all zeolites are extremely variable and, therefore, the percentages in the table can only be taken as approximations.

| Mineral | SiO ₂ | Al ₂ O ₃ | CaO | Na ₂ O | K ₂ O | H ₂ O | Others | |
|--|------------------|--------------------------------|------|-------------------|------------------|------------------|--------|---------------------------------|
| * <i>Laumontite</i> Ca Al ₂ Si ₄ O ₁₂ ·4H ₂ O | 50.7 | 21.9 | 12.1 | 0.5 | 0.2 | 13.7 | 1.0 | } Calcium-rich |
| <i>Wairakite</i> Ca Al ₂ Si ₄ O ₁₂ ·H ₂ O | 55.9 | 23.0 | 11.7 | 1.1 | 0.2 | 8.5 | 0.1 | |
| * <i>Stilbite</i> (Ca, Na ₂ , K ₂) Al ₂ Si ₇ O ₁₈ ·7H ₂ O | 56.0 | 16.7 | 7.7 | 0.9 | 0.7 | 17.9 | 0.3 | |
| <i>Chabazite</i> Ca Al ₂ Si ₄ O ₁₂ ·6H ₂ O | 60.3 | 12.7 | 3.2 | 2.9 | 0.9 | 18.1 | 0.9 | } Sodium and Calcium subequal |
| <i>Mordenite</i> (Ca, Na ₂ , K ₂) Al ₂ Si ₁₀ O ₂₄ ·7H ₂ O | 66.3 | 11.5 | 2.6 | 3.1 | 1.0 | 14.0 | 1.5 | |
| * <i>Natrolite</i> (From igneous rocks) Na ₂ Al ₂ Si ₃ O ₁₀ ·2H ₂ O | 47.2 | 27.1 | 0.2 | 15.4 | 0.3 | 9.6 | 0.4 | } Sodium-rich |
| <i>Analcite</i> NaAlSi ₂ O ₆ ·H ₂ O | 64.0 | 16.9 | 0.1 | 9.5 | 0.2 | 8.7 | 0.5 | |
| <i>Heulandite and Clinoptilolite</i> (Ca, Na ₂) Al ₂ Si ₇ O ₁₈ ·6H ₂ O | 64.7 | 12.4 | 1.5 | 3.8 | 3.1 | 13.2 | 1.4 | } Potassium and sodium subequal |
| <i>Erionite</i> (Ca, K ₂ , Na ₂ , Mg) _{4.5} Al ₉ Si ₂₇ O ₇₂ ·27H ₂ O | 56.8 | 13.8 | 0.2 | 6.2 | 4.1 | 15.1 | 3.8 | |
| <i>Phillipsite</i> deep sea ($\frac{1}{2}$ Na, Ca, K) ₃ Al ₃ Si ₃ O ₁₆ ·6H ₂ O | 54.1 | 17.7 | 1.3 | 3.8 | 6.8 | 15.6 | 0.2 | |
| subaerially altered tuff | 56.1 | 15.1 | 0.2 | 6.3 | 4.3 | 15.6 | 2.2 | |

Table 3. Time concept of geological nature (Harbaugh, 1974).

Geologic time scale in general use. Names of epochs in Cenozoic Era are included; other epoch names are omitted. Estimates of time in millions of years are subject to change as age-dating techniques are improved and applied more widely.

| Era | Period | Epoch | Estimated Duration in Millions of Years | Estimated Millions of Years Ago | |
|-----------|----------------|---------------|---|---------------------------------|-----|
| Cenozoic | Neogene* | Quaternary | | (Recent) | |
| | | | Pleistocene | 2.5 | |
| | Paleogene* | Tertiary | | Pliocene | 4.5 |
| | | | | Miocene | 19 |
| | | | | Oligocene | 12 |
| | | | | Eocene | 16 |
| | | | | Paleocene | 11 |
| | | | | | |
| Mesozoic | Cretaceous | | 71 | 65 | |
| | Jurassic | | 54 | 136 | |
| | Triassic | | 35 | 190 | |
| Paleozoic | Permian | | 55 | 225 | |
| | Carboniferous† | Pennsylvanian | 30 | 280 | |
| | | Mississippian | 35 | 310 | |
| | Devonian | | 50 | 345 | |
| | Silurian | | 35 | 395 | |
| | Ordovician | | 70 | 430 | |
| | Cambrian | | 70 | 500 | |
| | Precambrian | | | 570 | |
| | | | 4000+— | | |

No worldwide divisions of the Precambrian have been devised, although various local classifications exist. By definition, the Precambrian embraces the time between the origin of the earth and the beginning of the Cambrian Period.

Natural zeolites

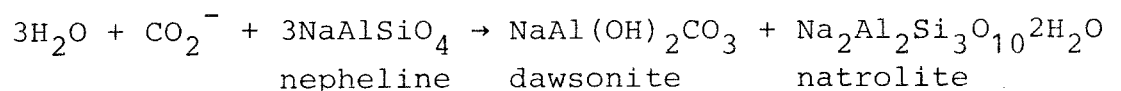
Zeolites are formed in alkaline environment. Gases or solutions of volcanic origin, enriched saline solutions by evaporation are environments where zeolite formation takes place. In the case of saline or saline-alkaline lakes with a high concentration of Na and in marine environments where the Na-content naturally is high, zeolitization occurs in soft sediments. In seawater pH is naturally about 8.0-8.4 but this situation is changed when the seawater penetrates the sediment. Atmospheric CO₂ is removed and pH will then increase in the pore water to about 9 and this is favourable to the formation of a zeolite. Zeolitization in lakes and in the oceans can take place at low temperatures 2-3°C according to Boles (1971). Ross (1941) noticed in his investigation of a transformed volcanic ash at Wikieup in Arizona that both analcite and bentonitic clays were formed from volcanic ash if the concentration of Mg and Ca is sufficiently high in the percolating solutions. If, on the other hand the concentration of Na-ions is high analcite will be formed. In Wikieup the mass ratio of analcite and bentonite was 4 to 1. This can, according to Ross, be taken as a proof of a coexistence between bentonite and analcite in solid rocks with the zeolites as cementing agent. Also Coombs (1965) noted that zeolite/montmorillonite can coexist but in the form of cemented solid rock (siltstone).

In deep drilling cores from the Pacific Ocean Couture (1977) found authigenic clinoptilolite in Cretaceous layers but montmorillonite and phillipsite in Tertiary layers. Biogene SiO₂ was said to contribute to the formation of clinoptilolite from the volcanic ash or glass while the phillipsite/montmorillonite is a direct product from the volcanic ash or glass. The origin of clinoptilolite can have been phillipsite/montmorillonite.

According to many scientists an important factor for the formation of clinoptilolite may be that the Cretaceous sea was warmer than today. Couture (1977) believed that clinoptilolite must be either an authigenic formation or a redeposition of a material originating from the sea. No terrestrial sources are known to this author. Clinoptilolite occurs in abundance in Oligocenic and older sediments and rock-forming sediments. According to Sheppard (1973) clinoptilolites are mainly of Cenozoic age and their reaction is alcalic. The same author noticed that erionite and chabazite are not known from pre-Eocene formations.

Hay (1963) showed that zeolites occurring in Tanganyika are formed by Na-carbonate- and bicarbonate solutions which have affected volcanic glass and nepheline ash. The carbonate solutions are concentrated by evaporation in the soil and surface rock.

Combined hydratization and carbonization seem to be the reason for zeolitization.



Further, Hay observed that phillipsite and calcite form the cement in recent and several thousand year old ash deposits.

In eolian tuffs which contain mineral grains, rock fragments and clay pellets of medium sand grain size the cementing mass consists of one or more zeolites. It can be added that cavities in all existing tuffs are often covered by phillipsite, sometimes also by chabazite, analcime and natrolite. Hayashi and Sudo (1957) described a rock-forming bentonite where the pore spaces among others contain zeolite (mordenite). The above mentioned (Hay, 1963) zeolitization occurred

in Pleistocene under two dry seasons. The conversion to zeolite has taken place fairly rapidly, that is within 8000-20000 years. The pH at the time of formation of the zeolites must have been 9.5-10.6. The enrichment of ions was caused by a transportation of capillary water. The dry clima caused an increased salinity which was not favourable to the formation of clays.

The key mechanism of zeolite formation is probably a function of the Si/Al ratio in the gel developed by the saline-alkaline ratio and pH (cf. Mariner and Surdam, 1970). The SiO_2 -gel does not necessarily originate from volcanic glass but can also be a weathering product from feldspars according to the same authors. Then the formation of phillipsite should increase if the Si/Al ratio is low. If the Si/Al ratio is high with increasing pH the formation of clinoptilolite should be favoured. In conglomerates, sandstones, silt- and mudstones zeolites generally co-exist with calcite and different iron compounds constitute the cementing mass between the soil elements. Authigene Ca-montmorillonite, Ca-heulandite and polycrystalline quartz occur together in equilibrium in a locality from Upper Cretaceous in Crazy Mountains according to Sims (1969).

In marine deposits it is shown that phillipsite and clinoptilolite are the dominating zeolites in co-existence with montmorillonite but in none-marine deposits analcime together with montmorillonite is more abundant.

Coombs (1952) investigated a Triassic layer altered to a bentonite and with residues of clinoptilolite, volcanic glass, aggregates of heulandite, microcrystalline quartz and clay minerals, analcime and quartz partly transformed to albite (a feldspar).

Later, the same author (1959) made a literature review on a large number of cases where zeolites act as a cementing substance in sandstones, siltstones, arcoses and mudstones. The occurrence of natural zeolites with reference to temperature and depth in some selected localities is illustrated by Table 4, (Coombs, 1959).

Table 4. Temperature of occurrence of some natural zeolites and related minerals.

| Mineral | Occurrence | Temperature (T ^o C) | Depth (m) |
|---|------------------------------|-----------------------------------|--------------|
| Phillipsite | Deep sea sediments | 0 ^o | 4000-5000 |
| Chabazite, phillipsite, natrolite | Masonry, Roman baths | 40-70 ^o | Surface |
| Clinoptilolite, analcime | Diagenesis | Low | ? |
| Stilbite | Hunters, Boulder Hot Springs | ?64-?73 ^o | Surface |
| Clinoptilolite | Yellowstone | 125 ^o | 19-26 |
| Analcime | Yellowstone | 125-155 ^o | 26-60 |
| Mordenite | Wairakei | 150-230 ^o | 73-300 |
| Heulandite | Wairakei | within range of mordenite | |
| Lammonite | Wairakei | 195-220 ^o | 150-275 |
| Warakite | Wairakei | 200-250 ^o | 180-600 |
| Prebnite | Wairakei | ~200 ^o | ~100 |
| Albite | Wairakei | 160-240 ^o | 100-600 |
| Adularia | Wairakei | 230-250 ^o | 385-650 |
| Zeolite and adularia | Steam Boat Springs* | 170 ^o | 52 |

Note (1) The maximum temperatures recorded for the Wairakei bore holes are 250-260^oC.

(2) The appearance of albite and other minerals at abnormally low temperatures and shallow depths in some Wairakei holes suggest that in these cases they may have been deposited under an earlier regime of higher temperatures.

* S White (1955)

Coombs stated that zeolites are stable minerals at low temperatures or, under special circumstances, at a small depth. This gives open pores while at greater depth with increasing pressure minor pores are developed under expelling of water. The zeolites will be less stable and an albitization can occur under such circumstances.

Further Coombs (1959) showed that analcime and heulandite can coexist with quartz and closed grain phyllosilicates. He also mentioned that Rengarten (1950) showed that authigenic zeolites occur as cementing substance in many Russian sedimentary rocks from Perm to Tertiary.

Investigations by Sheppard (1973) indicate that neither analcime nor K-feldspar originate directly from the volcanic glass. The formation passes through other zeolite stages both for analcime and K-feldspar. The last mentioned mineral can originate from analcime if the conditions are suitable.

"The paragenesis of silicate minerals in the tuffaceous rocks is attributed to chemical variables of the pore water, such as the $\text{Na}^+ + \text{K}^+ : \text{H}^+$ ratio, Si : Al ratio, the proportion of cations and the activity of H_2O ." Sheppard and Gude (1972) stated that conglomerates, sandstones and siltstones in the "Big Sandy Formation" are often cemented by calcite, clay minerals, zeolites and opal. It appears that clinoptilolite and analcime are the most frequent cementing zeolites. Fig 1 shows traces of zeolite cementation. In mudstone from the "Big Sandy Formation" authigenic zeolites and K-feldspars are shown. According to the author analcime is the dominating mineral.

In order to give the reader of this report a conception of the detailed appearance of some zeolites a sequence of scanning microscope pictures is shown here. Figs 2 and 3, however, show rock samples of Wyoming-bentonites. They give a general idea of the tight appearance of the montmorillonite particle arrangement in bentonite. In comparison the zeolite configuration is more open and the crystallites well formed.



Fig 1. Sandstone cemented by finely crystalline clinoptilolite. Unpolarized light. (From Sheppard, 1973).

Analcime, often termed analcite, is generally present in sedimentary rocks and together with K-feldspar. The investigation of the "Big Sandy Formation" show evidence of co-existence of montmorillonite-phillipsite-clinoptilolite and montmorillonite-clinoptilolite-quartz. The stability of the system as concerns geochemistry is not clear. However, it is shown that certain analcime crystals disclose a thin film of montmorillonite (see fig 5). It is also shown that authigenic montmorillonite is replaced by analcime. This indicates instability. In "Big Sandy Formation" there is a clear proof of analcime transformation to K-feldspar (fig 4). The mineral transformation in

"Big Sandy Formation" is considered to depend on differences in the chemistry of the pore water.

According to Sheppard (1973), Huntley (1959, 1962), Garrels and Christ (1965), Hess (1966) we have the following statement concerning the process of formation:

"Zeolites and feldspars are favoured over clay minerals by relatively high alkali ion to hydrogen ion activity ratios and by relatively high silica activities. The high alkali ion to hydrogen ion activity ratio necessary for the formation of zeolites in a tuff can be attained in the depositional environment of a saline, alkaline lake or in the postdepositional environment by hydrolysis and solution of silicic vitric material by subsurface water."

Iijima and Hay (1968) and Surdam and Parker (1972):

"Tuffs in the Eocene Green River Formation of Wyoming are altered to montmorillonite where they were deposited in fresh water, to clinoptilolite and mordenite in slightly saline water, to analcime in moderately saline water and to K-feldspar in highly saline water."

The formation of K-feldspar governed by the above statement is obvious in many places according to Sheppard. Probable contributing factors are 1) a low activity of H_2O , 2) a relatively high K^+/H^+ ratio and 3) a high activity of SiO_2 . A high pH produces an increased solubility of SiO_2 which, however, contributes to a stabilization of K-feldspar (Surdam & Parker, 1972).

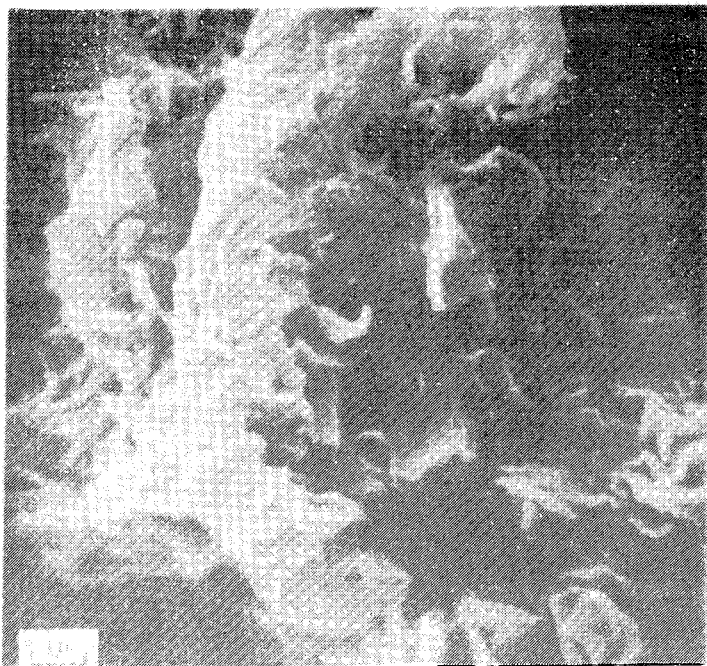


Fig 2. Rock sample of bentonite from Clay Spur, Wyoming (A.P.I. No 26) (Cretaceous). Bohur & Hughes, 1971.

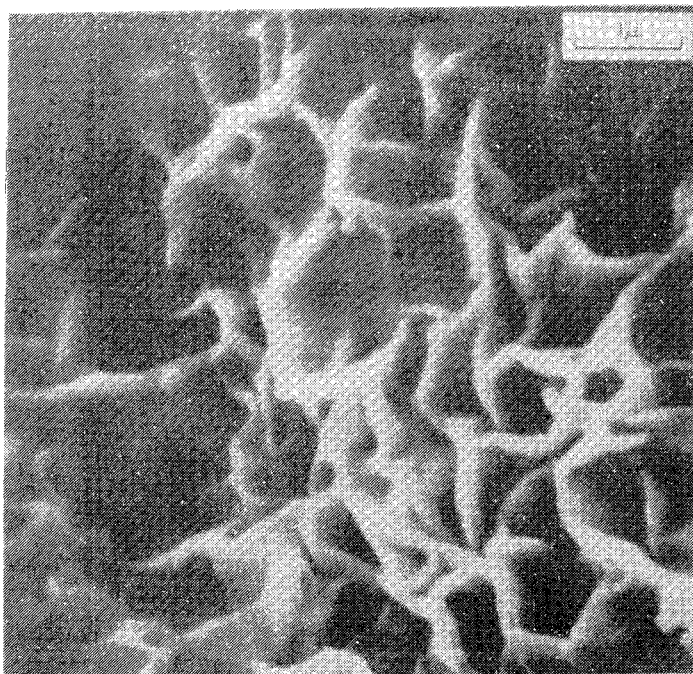


Fig 3. Rock sample of Wyoming bentonite (A.P.I No 26). Bohur & Hughes, 1973.



Fig 4. Scanning electron micrograph of large analcime crystals in a matrix of micron-size crystals of potassium feldspar from a saline-lake tuff near Barstow, California. (Sample 25-43-136).

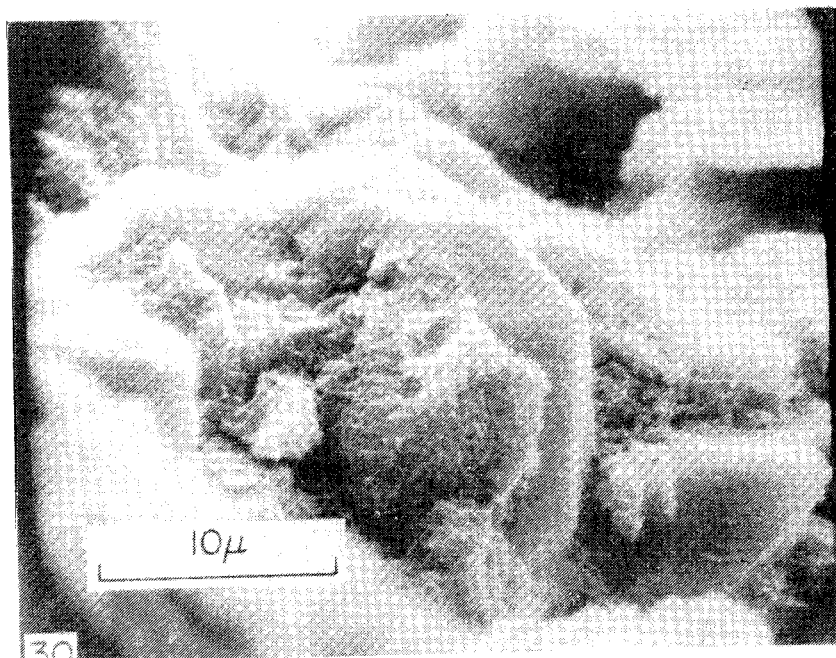


Fig 5. Scanning electron micrograph of a large analcime crystal coated with montmorillonite from a lacustrine tuff near Tejuapan, Oaxaca, Mexico.

Various zeolites are shown in the series of pictures given by Figs 6-10. Fig 6 shows etched crystals of clinoptilolite, the etching being caused by pore-water dissolution. Figs 7-10 show open-structured masses of different zeolites. It is quite obvious that such materials are much more permeable than montmorillonitic soils. The brittleness of zeolite crystals can be demonstrated in Fig 9, where a fracture in a chabazite crystal is shown.

In this review I have mentioned a few authors and presented their basic ideas about zeolites, which are of interest to us. These ideas are representative also of a large number of other investigators who are not mentioned specifically in this report.

The author's study has yielded definite objections against the use of zeolites in the buffer mass. They are:

1. Cementing properties are observed by many investigators.
2. The permeability of pure zeolites is much too high to be accepted for buffer mass components.
3. Zeolites are unstable at higher temperatures with increasing depth and pressure. They are easily converted to feldspars.
4. Zeolites are brittle. In fact there is not a single geological evidence of a non-cemented quartz-bentonite-zeolite mixture above sea level.
5. The heat conductivity is poor ($\lambda \leq 0.2 \text{ W/m, } ^\circ\text{C}$)
6. We have too little knowledge about their

mechanical properties in a mixture with other minerals.

The positive properties of the zeolites are their good ion exchange properties. On the other hand we do not know enough about their long time fixation ability

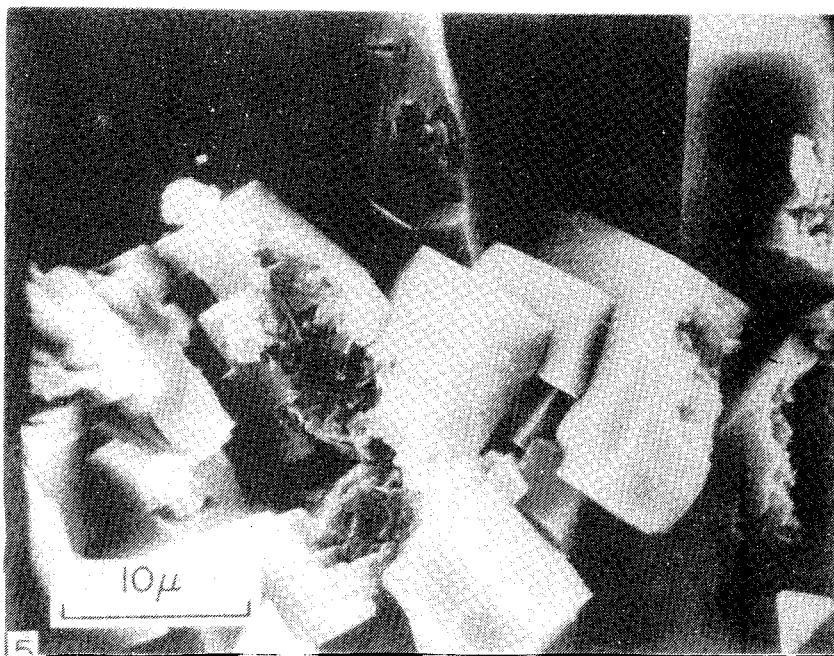


Fig 6. Scanning electron micrograph of clinoptilolite from Hungry Valley, Nevada. The severely etched crystals are probably due to pore-water dissolution, after formation in a saline-lake environment.

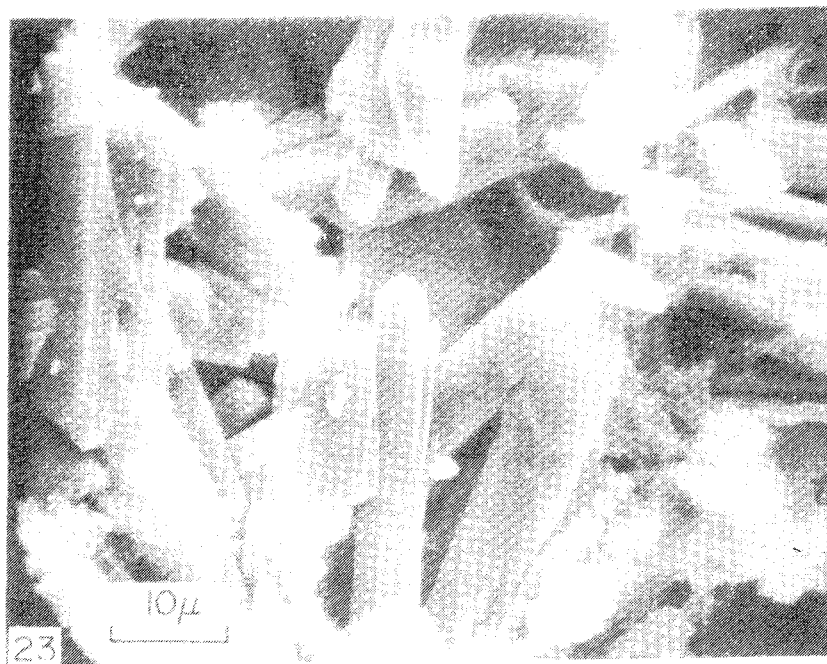


Fig 7. Scanning electron micrograph of phillipsite laths and prisms from a saline-lake tuff near Kirkland, Arizona. Montmorillonite is also abundant in this sample.

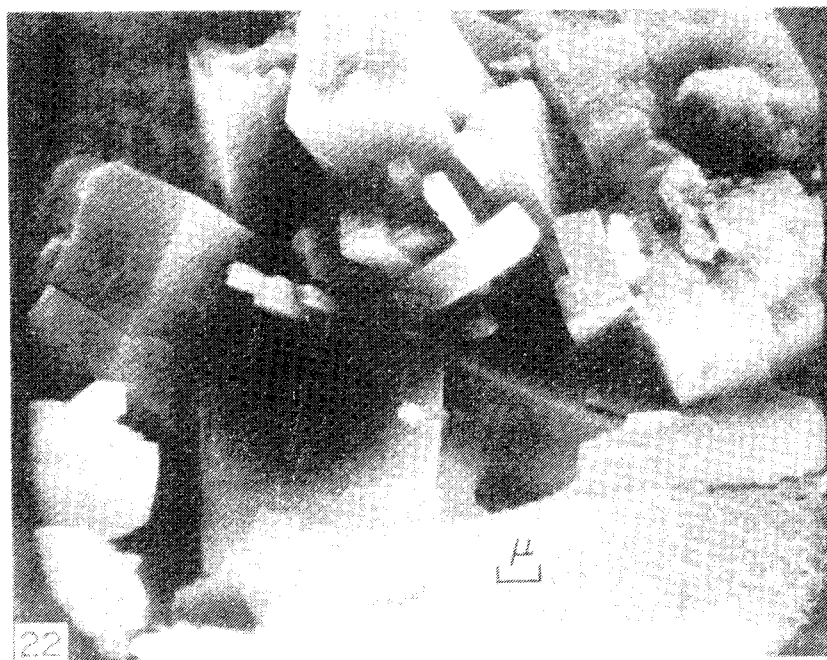


Fig 8. Scanning electron micrograph of phillipsite prisms from a marine tuff near Naples, Italy. Note the "cracking" of many crystals along cleavage surfaces parallel to the axis of elongation. Montmorillonite is also present in this sample.

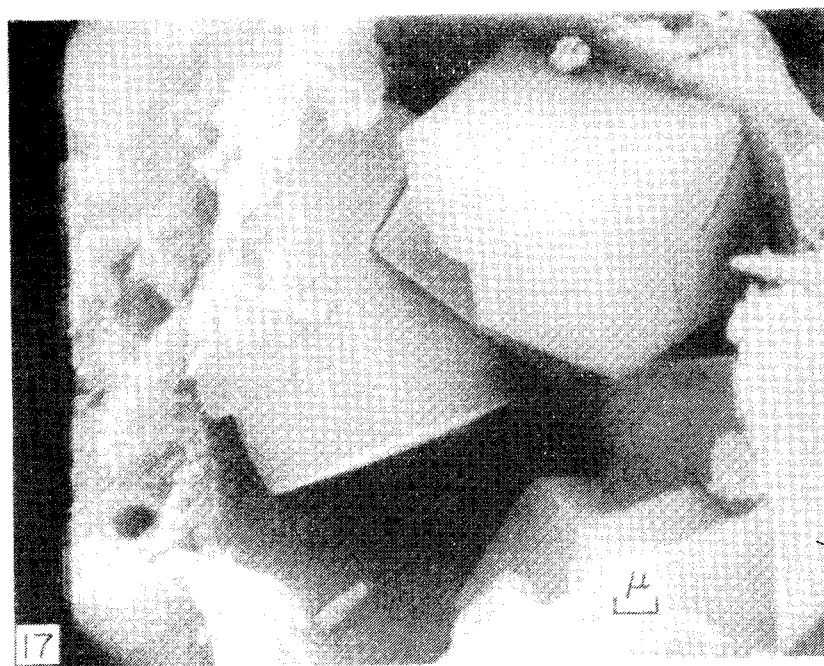


Fig 9. Scanning electron micrograph of well-formed chabazite crystals from a marine tuff near Kruft, Germany, currently mined as a pozzolan.

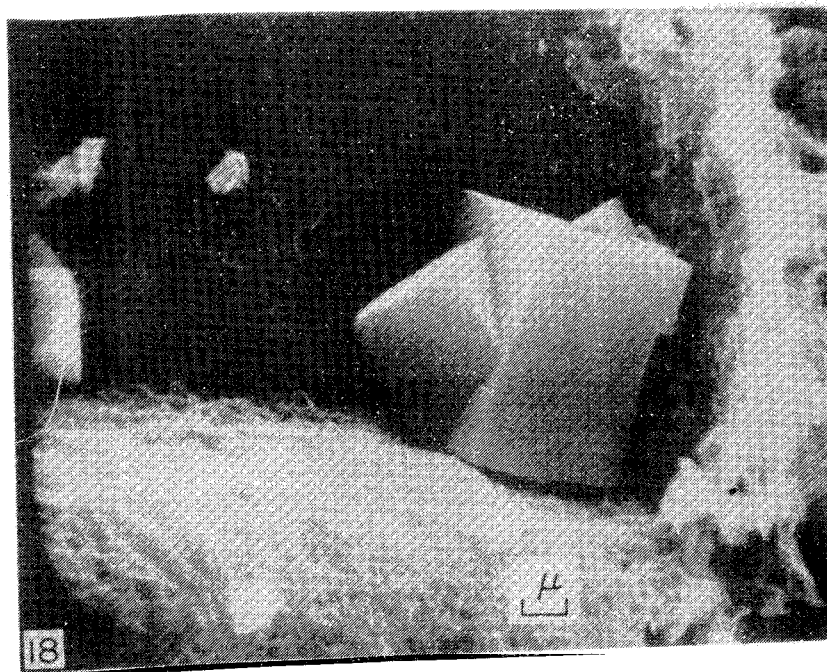


Fig 10. Scanning electron micrograph of an isolated chabazite crystal in a saline-lake tuff near Durkee, Oregon. The surrounding matrix is probably montmorillonite.

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