

A simulation study of capillary transport, preferential retention and distribution of salts in historic sandstone buildings

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Abstract Salt crystallisation is a major problem of deterioration in historic stone buildings, monuments and sculptures. The capillary rise of soil water is one of the primary sources of salts in stone structures, which evaporates leaving the salts behind. It has been noted that the spatial distribution profile of different species of salts crystallised in historic stone buildings is not homogeneous, i.e. different salts crystallise at different locations. The capillary transport and inhomogeneous spatial distribution of different salts in the porous building materials has been considered to be a result of solubility-dependent crystallisation; however, the factors responsible for this phenomenon are not clearly known. This paper aims to investigate the factors influencing the differential distribution of salts during capillary rise of soil water. In this study, the capillary transport of salts was simulated on two different sandstones—Locharbriggs, a Permo–Triassic, red sandstone and Stoke Hall, a Carboniferous, buff sandstone. The experiments were carried out under controlled environmental conditions to eliminate the possibility of evaporation-driven crystallisation of salts depending on their solubilities. The results indicate that fractionation or differential distribution of salts takes place even in the absence of evaporation and crystallisation. The sandstones exhibit properties like an ion exchange column, and ionic

species present in the salt solution show differential distribution within the porous network of sandstone.

Keywords Built heritage · Stone weathering · Capillary rise · Salt transport · Salt crystallisation

Introduction

It is generally understood that the crystallisation of salts is not uniform within the porous matrix of the stone and that some salts crystallise at the stone surface (efflorescence) whereas others crystallise within the pores (subflorescence) (Arnold 1982; Arnold and Zehnder 1990, 1991). This inhomogeneous distribution and crystallisation of salt may be an important factor in stone decay as the severity of damage is attributed to the location of crystallisation within the stone (Rodriguez-Navarro and Doehne 1999). Although visually alarming, efflorescence is generally less harmful than subflorescence, as crystallisation in confined spaces can generate stresses to cause severe damage. The transport and distribution within the porous network is generally considered to be dependent on the rate of evaporation, salt concentration and their solubilities. It is suggested that when water evaporates, the solutions become more and more concentrated and a concentration gradient develops with respect to the various salts present in it. Various species of salts start to crystallise out from the solution depending on the extent of saturation, whereas the most soluble salts remain in the solution and are transported further during capillary rise (Arnold 1982; Arnold and Zehnder 1990, 1991; Matteini 1991). Thus, different salts precipitate at different locations in the pores as the solution moves further on. This sequential precipitation of different salts from the solution results in fractionation of different

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salts. It is also postulated that the transport of salt solution depends on viscosity, surface tension and vapour pressure (Rodríguez-Navarro and Doehne 1999; Ruiz-Agudo et al. 2007). The authors argue that higher surface tension and lower viscosity lead to faster transport, while low vapour pressure reduces the rate of evaporation and, therefore, result in differential rates of transport of salts, which, in turn controls the location of crystallisation upon saturation. However, sandstones and soil water are heterogeneous and geochemically active systems, and therefore, it is highly unlikely that the transport and distribution of salts is controlled merely by their physical characteristics.

This research aims to investigate the processes that control the relative retention and distribution of salts during capillary transport of soil water. It was devised to determine the distribution behaviours of salt ions during capillary rise in sandstones structures/sculptures with an aim of determining the potential importance of hydrogeochemical interactions on the spatial distribution of crystallisation. A study based on flow-through method (induced flow of salt solution through sandstone pores) and quantitative estimation of cation exchange capacities (CEC) combined with geochemical modelling, as part of this research, has already indicated that ion exchange significantly influences the relative transport of ionic species in the capillaries, which results in differential distribution of salts within the pores and that the sandstones with higher capacity to exchange ions would have greater influence on relative retention and distribution of salts during capillary transport of ground water (Pandey et al. 2014). In this paper, the transport and relative distribution of cations and anions within the pores under natural capillary rise is presented.

Stone types and properties

This study was carried out on Locharbriggs sandstone (Locharbriggs Quarry, Dumfries) and Stoke Hall sandstone (Stoke Hall Quarry, Derbyshire). The Locharbriggs is lower Permian, red sandstone whereas the Stoke Hall is Carboniferous sandstone. The mineralogical examination of the samples of both types of sandstones was carried out qualitatively using polarised light microscopy (PLM) and scanning electron microscopy with energy-dispersive analysis (SEM-EDAX).

The results indicate that Locharbriggs sandstone is predominantly sub-arkosic and is mainly composed of polycrystalline quartz, potassium and plagioclase feldspars and rock fragments with concentrated zones of iron oxides, which produces characteristic dark lamination. It contains kaolinitic clays, and the mineral grains are coated with the iron oxide that gives the rock its red colour. The edge-to-edge cementation is probably due to pressure solution

compaction, but a small amount of silica cement is also present (Baraka-Lokmane et al. 2009; Pandey et al. 2014). Stoke Hall sandstone is poorly sorted with medium- to coarse-grained minerals and is primarily composed of polycrystalline quartz along with plagioclase feldspars, biotite mica, kaolinite and rock fragments. There is some silica cement present; however, no carbonate cement was noticed (Pandey et al. 2014).

Effective porosity was measured according to European Standard [EN 1936] and using mercury intrusion porosimetry. The results indicated the average porosities of Locharbriggs and Stoke Hall sandstones were approximately 22 and 11%, respectively. The hydraulic conductivity for Locharbriggs sandstone was measured to be 6×10^{-7} m/s and for that of Stoke Hall was 5×10^{-8} m/s (Pandey et al. 2014).

Experimental methodology

The column samples with dimensions $150 \times 25 \times 25$ mm (± 2.5 mm accuracy) were cut from single stone blocks to minimise variations in stone properties between samples. The samples were washed repeatedly (about 5–6 times) by vacuum saturation in deionised water followed by drying to remove any salts present in the pore spaces. Washed samples were then dried to constant weight and stored in a desiccator. Two salt solutions were used to study cation and anion transport, respectively—the first contained four cations (Ca^{2+} , Mg^{2+} , K^{+} , Na^{+}) and the second four anions (F^{-} , Cl^{-} , NO_3^{-} , SO_4^{2-}). The salts used to prepare the stock solutions for experiments were NaCl, KCl, MgCl_2 and CaCl_2 for cations and NaF, NaCl, NaNO_3 and Na_2SO_4 for anions. Each of the two solutions contained one common counter ion (Na^{+} in the anion solutions and Cl^{-} in the cation solutions) to maintain uniformity in the effect of the counter ions on the transport and retention behaviour of cations/anions. Four different salt concentrations ranging from 0.01 to 1.0 mol/L were trialed, and it was noticed that 0.1 mol/L was the most suitable since concentrations of 0.5 and 1.0 mol/L were too concentrated to give noticeable ionic exchange and retention, while 0.01 was too dilute to yield salt concentrations above background level in the drilled powder. 100 ml of salt solution was taken in a glass container, and six samples of each type of sandstone were placed in the solution vertically to allow capillary rise. The entire assembly was kept in an environmental chamber under controlled conditions of 20 °C and 99–100% RH. A high RH level was maintained to prevent evaporation and crystallisation of salts from the samples. The capillary rise was monitored periodically, and salt solution replenished every hour to keep the level constant in the container. Samples were removed from the salt solution as soon as the

wetting front reached the top of the column, sealed in airtight plastic bags and frozen in dry ice for about 10–12 h. The freezing was done to prevent any further movement and redistribution of ions and to cool the drill bits when the powder was being drilled out. Drilling was carried out with a pillar drill using 6.0-mm-diameter diamond tip masonry bits at a speed of 350 RPM without any liquid coolant. The drill bit was cleaned with deionised water after each drilling to prevent contamination of ions. Powder samples were collected from 6–7 points (about 18–20 mm apart) along the height of the columns starting from the base of the samples and stored for ion chromatography. In Locharbriggs samples, seven-point drilling measurements were carried out for cation analysis and six-point measurements for anion analysis. Stoke Hall samples were drilled at six points for both anion and cation analysis.

Although the samples used in this experiment were thoroughly washed with deionised water several times before starting the experiments, it was not possible to remove all the preexisting salts from the pores. Therefore, it was necessary to carry out a background check on the concentration of salts already present in the samples. Three control samples each from Locharbriggs and Stoke Hall sandstone were analysed for the background concentrations of salts already present in the pores. Each control sample column was drilled at five points along the height (every 25 mm), and powder sub-samples were collected.

The powder sub-samples were then dried overnight at 80 °C in a ventilated oven, homogenised with gentle grinding, and 200 mg of powder was weighed and dispersed in 25 ml of deionised water. The suspension was stirred manually and left to settle down, and supernatant solution was filtered with a syringe mounted 0.2-µm filters and analysed with Dionex DX500 ion chromatograph to measure the concentration of ions in each powder sub-sample. The mean values of ionic concentration (mg/kg) obtained from three samples (5 drills/sample) of Locharbriggs and Stoke Hall sandstone are given in Table 1.

Results and discussion

Cation transport and distribution

The control samples of Locharbriggs indicate the presence of all the four cations; however, the amount of K⁺ is greater than that of Na⁺, Mg²⁺ and Ca²⁺. The amounts of Mg²⁺ and Ca²⁺ are very low. The most probable source of high amounts of K⁺ is decaying potassium feldspar and illite clays noted to be present in the Locharbriggs sandstone. Stoke Hall sandstone on the other hand has a high amount of Na⁺ as compared to other three cations in the control samples, most probably coming from the decaying

Table 1 Mean values (mg/kg) for different cations and anions measured from three control samples each of Locharbriggs and Stoke Hall sandstones

	Locharbriggs (mg/kg)	Stoke Hall (mg/kg)
Cations		
Na ⁺	0.22	0.61
K ⁺	0.97	0.16
Mg ²⁺	0.05	0.07
Ca ²⁺	0.14	0.11
Anions		
F ⁻	–	0.02
Cl ⁻	0.61	0.17
NO ₃ ⁻	–	–
SO ₄ ²⁻	0.26	–

sodium feldspar present in the sandstone. The concentrations of K⁺, Mg²⁺ and Ca²⁺ are very low in Stoke Hall sandstone. The mean values of different cations in all the control samples were subtracted from the values obtained from powder sub-samples of experimental sample columns, to achieve more realistic data.

Since sandstones contain very heterogeneous porosity distribution, the total pore volume at each drill point may be different which would result in different amounts of salts. However, variation in their relative molar ratio on various locations along the z-axis would indicate any fractionation during transport, because the initial salt solution was used in equimolar concentration. The cation transport and distribution from six replicate samples of Locharbriggs and Stoke Hall sandstones, subjected to cation solution transport by capillary rise, is presented in terms of mean millimolar (mM) fraction (Figs. 1, 2). However, an average of the actual cationic concentration in mg/kg obtained from all the samples (after subtracting the

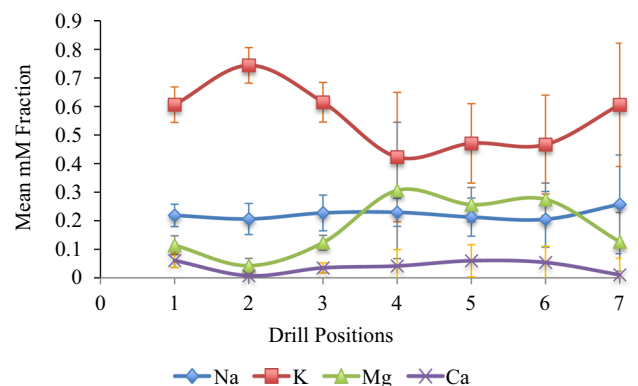


Fig. 1 Cation distribution in Locharbriggs sandstone (1–7 are drill positions from bottom to top of the samples, error bars represent standard deviation)

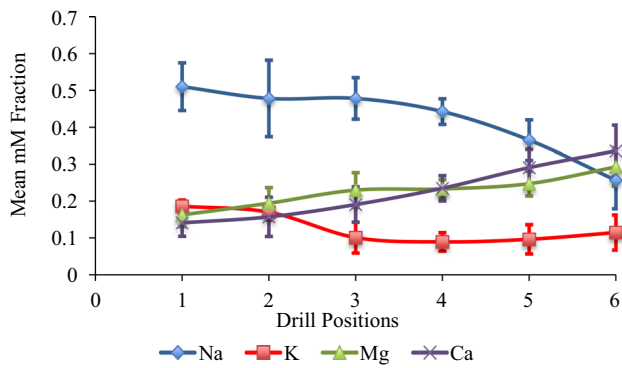


Fig. 2 Cation distribution in Stoke Hall sandstone (1–6 are drill positions from *bottom* to *top* of the samples, *error bars* represent standard deviation)

mean cationic values of control samples) is given in Tables 2 and 3.

The cation distribution analysis of Locharbriggs sandstone indicates that the cations are not equally distributed during transport even though the source reservoir contained equal concentration of cations. K^+ appears to be always dominant as the salt solution moves towards the top of the column. Na^+ concentration is much lower than that of K^+ and shows a flat trend from bottom to top of the column. Mg^{2+} and Ca^{2+} are present in very small amounts with a more or less flat profile in the sample. The higher concentration of K^+ than Na^+ indicates that it is being

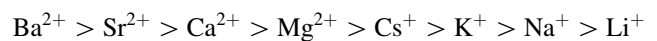
Table 2 Locharbriggs sandstone: mean values of actual concentration (mg/kg) of cations and anions from six samples each

Locharbriggs sandstone				
Cation distribution—mean values (mg/kg)				
Drill points	Na^+	K^+	Mg^{2+}	Ca^{2+}
1	0.69	3.20	0.40	0.36
2	0.43	2.71	0.10	0.03
3	0.69	3.15	0.39	0.17
4	0.25	0.81	0.23	0.11
5	0.23	1.00	0.31	0.13
6	0.27	1.14	0.38	0.13
7	0.23	0.85	0.13	0.04
Anion distribution—mean values (mg/kg)				
Drill points	F^-	Cl^-	NO_3^-	SO_4^{2-}
1	0.40	0.88	1.53	2.41
2	0.18	0.84	1.64	2.57
3	0.05	1.07	1.98	3.44
4	0.03	0.85	1.63	2.73
5	0.04	0.83	1.59	2.46
6	0.03	0.46	0.93	0.90

Table 3 Stoke Hall sandstone: Mean values of actual concentration (mg/kg) of cations and anions from six samples each

Stoke Hall sandstone				
Cation distribution—mean values (mg/kg)				
Drill points	Na^+	K^+	Mg^{2+}	Ca^{2+}
1	0.84	0.52	0.28	0.40
2	0.84	0.49	0.33	0.44
3	1.01	0.38	0.51	0.69
4	0.84	0.30	0.47	0.77
5	0.62	0.29	0.42	0.81
6	0.40	0.31	0.45	0.86
Anion distribution—mean values (mg/kg)				
Drill points	F^-	Cl^-	NO_3^-	SO_4^{2-}
1	0.28	0.48	0.79	1.42
2	0.07	0.54	0.93	1.75
3	0.01	0.54	0.96	2.64
4	0.02	0.52	0.87	1.78
5	0.02	0.62	1.07	0.99
6	0.02	0.57	1.02	0.41

replaced more from the exchange sites as compared to Na^+ . Generally, K^+ is favoured (preferentially retained) over Na^+ , and therefore, Na^+ is expected to be dominant in the salt distribution profile (because it is less attracted to exchange sites). Mg^{2+} and Ca^{2+} have higher exchange selectivities as compared to K^+ and Na^+ and are more strongly attracted to exchange sites. A general order of affinity to exchange sites of common cations is given as under (Blatchley and Thompson 2007):



However, the salt distribution profiles will be greatly influenced by the initial amount of ions present on the exchange sites. Because the ionic composition in the control samples indicates a high presence of K^+ in Locharbriggs, it can be anticipated that the exchange sites are occupied mostly by K^+ . Since the cation exchange selectivities of Mg^{2+} and Ca^{2+} are higher as compared to Na^+ and K^+ , the exchange sites are readily taking up these cations. Therefore, a high concentration of K^+ in the sample is most probably because Mg^{2+} and Ca^{2+} displaced K^+ from the exchange sites in solution and also because there is additional K^+ in the salt solution, most of which is not being taken at the exchange sites in the presence of Mg^{2+} and Ca^{2+} . The concentration of Ca^{2+} cations remains very low indicating that most of the Ca^{2+} cations are being exchanged preferentially with other cations. The rising concentration of Mg^{2+} towards the top of the sample

as compared to Ca^{2+} also indicates that Ca^{2+} has higher preference over Mg^{2+} .

Similar exchange phenomena took place in the Stoke Hall sandstone column experiment. This sandstone shows a higher amount of Na^+ in the control samples, which indicates that most of the cation exchanger may have attained equilibrium with Na^+ . When the salt solution containing other cations comes to contact with exchange sites, Na^+ is probably being replaced with other cations. Combined with the Na^+ already present in the salt solution, the total amount of Na^+ in the powder collected from all the points is higher than other cations. The decrease in Na^+ profile and steady increase in the Ca^{2+} and Mg^{2+} profiles in the powder sub-samples collected towards the top of the columns may be because of reversed exchanged phenomenon due to higher concentration of Na^+ .

The samples were dried completely before the experiments, and the drying process may have moved cations from exchange sites towards precipitated phases. The dried sandstone columns have a fixed composition of cation exchanger, but with the introduction of salt solution, the cation exchanger readjusts its cation composition to attain equilibrium with the salt solution (Appelo and Postma 2005). All the powder sub-samples drilled from the columns were dispersed in deionised water for ion chromatographic analysis, and all the cations will have again readjusted to equilibrium with water. The filtrate that was used for ion exchange analysis, therefore, only contained cations, which were released into the water. Dispersing the drilled powder in water does not release cations from exchange sites, and thus, the results indicate that Na^+ and K^+ are released more as compared to Ca^{2+} and Mg^{2+} as the latter are preferentially taken at the exchange sites.

Anion transport and distribution

The anion analysis of control Locharbriggs samples indicates the presence of Cl^- and SO_4^{2-} anions in very low quantities whereas F^- and NO_3^- are absent in the control samples. The control column samples of Stoke Hall sandstone, on the other hand, show F^- and Cl^- in low quantities and NO_3^- and SO_4^{2-} are not present. The source of F^- in this sandstone is most probably contaminated water and/or decaying biotite $[(\text{K}(\text{Mg},\text{Fe})_3(\text{AlSi}_3\text{O}_{10})(\text{F},\text{OH})_2)]$, which is present in significant quantities. The mean values of different anions (except F^-) from all the control samples were subtracted from the experimental values obtained from the sample columns. The concentration of F^- was noted to be extremely low, and therefore, it was considered negligible.

Like the cation distribution experiments, six replicate samples of each Locharbriggs and Stoke Hall sandstone

subjected to anion solution transport by capillary rise. The data analysis was again carried out in terms of molar fraction to take into account the variation in the total amount of salts at each drill point. The results are presented in terms of mean millimolar fraction (Figs. 3, 4). An average of the anionic concentration in mg/kg obtained from all the samples (after subtracting the mean anionic values of control samples) is given in Tables 2 and 3.

The anion distribution in Locharbriggs sandstone shows that F^- drops dramatically after the first couple of drill point whereas Cl^- , NO_3^- and SO_4^{2-} exhibit a similar profile in all drill points. The results indicate that fluoride is being preferentially exchanged, whereas Cl^- , NO_3^- and SO_4^{2-} continue to flow through the pores with little or no attraction to the exchange sites. At the very top of the sample, there is some indication of SO_4^{2-} being preferentially adsorbed and the replaced Cl^- and NO_3^- from the exchange sites are indicated by a slight increase. Anion exchange is much weaker than cation exchange, and Cl^- , NO_3^- and SO_4^{2-} are poorly retained anions. The affinities of anions for positively charged surfaces follow the order (Juo and Franzluebbbers 2003):



In the Stoke Hall sandstone, F^- shows a similar trend as in the Locharbriggs and drops rapidly. SO_4^{2-} exhibits a peculiar characteristic of considerable increase in the third drill point and then rapid decrease in the amount. The most likely reason for the behaviour is that F^- ions are being preferentially retained and SO_4^{2-} concentration continues to build up in the pore solution until nearly all the F^- ions are exhausted. From this point, SO_4^{2-} ions are probably preferentially exchanged, which is indicated by a drop in SO_4^{2-} concentration in the pore solution.

There is also a possibility that precipitation of SO_4^{2-} took place in the solution. Since the anion salt solution contained a high concentration of Na^+ (0.5 M), it is possible that Na^+ replaced exchangeable Ca^{2+} from the

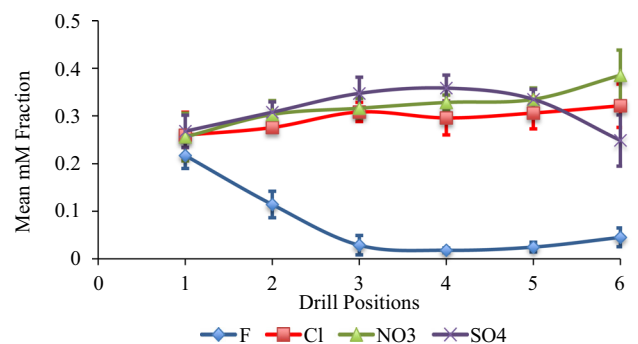


Fig. 3 Anion distribution in Locharbriggs sandstone (1–6 are drill positions from bottom to top of the samples, error bars represent standard deviation)

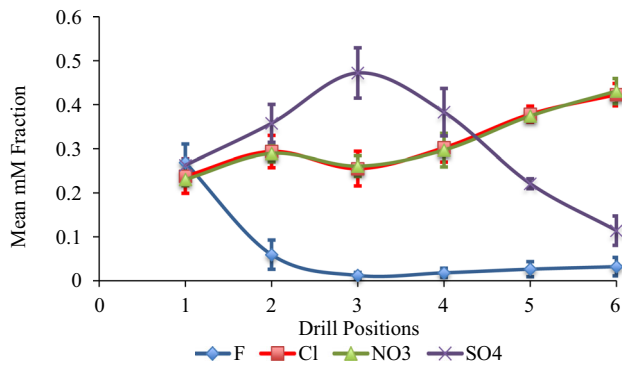


Fig. 4 Anion distribution in Stoke Hall sandstone (1–6 are drill positions from *bottom* to *top* of the samples, error bars represent standard deviation)

exchange sites. The anion solution was in equilibrium with atmospheric CO₂, and it may also be possible that dissolved CO₂ caused dissolution of some of the carbonate minerals (calcite, dolomite etc.), releasing Ca²⁺ in the pore water. By the time the solution front reached the middle of the column, the amount of Ca²⁺ and SO₄²⁻ reached their saturation limit and may have precipitated as gypsum (Tellam, pers. Comm, 2009). The precipitation of gypsum consumed most of the SO₄²⁻, which resulted in a drop in SO₄²⁻ content in subsequent drill points. The precipitated gypsum then re-dissolved and dissociated into Ca²⁺ and SO₄²⁻ when powder sub-samples were dissolved in water resulting in a higher concentration indicating precipitation at the particular drill point. The phenomenon of precipitation of gypsum in groundwater flow has been modelled, which supports this explanation (Wissmeier and Barry 2008). Cl⁻ and NO₃⁻ profiles show a trend of increasing concentration towards the top of the sample, which may be a result of exchange with some other anions such as HCO₃⁻, present in the sample from dissolution of carbonates.

In both sandstones, F⁻ is being readily adsorbed and this is because F⁻ is the second most electronegative ion known to exist. Adsorption of F⁻ is mainly a process in which coordinated OH⁻ exchanges with F⁻ as a specific adsorption called ‘ligand exchange’ and is not reversible, and therefore, no F⁻ was released in water when powder sample was dispersed in water. Iron oxyhydroxides such as goethite (FeOOH), certain micas and kaolinitic clays are major sources of OH⁻ for F⁻ adsorption. F⁻ can also be adsorbed on cations present in 2:1 hydrous silicates such as Mg²⁺, Al³⁺, Fe²⁺, Fe³⁺, Ni²⁺ and Mn²⁺ (Polomski et al. 1982). In Locharbriggs sandstone, presence of iron oxyhydroxides could provide loci for F⁻ adsorption. Stoke Hall sandstone contains decaying biotite mica and iron oxides, which can act as fluoride exchanger.

Conclusions

The results indicate fractionation of various ions takes place in the transport pathway even when environmental conditions are such that no evaporation and crystallisation take place. The differences in anion and cation distribution in both sandstones depend on the nature of the exchange composition of salts present in groundwater. Thus, it can be concluded that the natural stone acts as an ion exchange column and controls the rate of ionic transport through it in a preferential order. This implies that in course of capillary transport of salts, different ionic species have varying rates of transport, and depending on their rates of transport, some ions are exuded on the surface where as others remain in the pores. It can, however, be argued that a specific stone type can only retain ion preferentially until all the exchange sites are in equilibrium with the ions in the solution and in that condition ion exchange may no longer impact on transport and distribution of ions. However, the soil water chemistry changes continuously due to various environmental and geochemical processes such as rock weathering, dissolution, precipitation, leaching of evaporitic deposits, dilution of soil water concentration from rainwater infiltration and contamination from various sources. Since the ion exchange is a readily reversible process, any change in the concentration and/or chemistry of soil water will alter the equilibrium, making ion exchange a continuous phenomenon.

Although, it may be possible that the transport and distribution of salts is affected by environmental conditions, crystallisation of a salt phase takes place upon supersaturation and for soil water concentrations, supersaturation can take place in an ‘evaporation zone’ limited to superficial layers of the stone. Geochemical processes taking place in the pores would, however, significantly control the composition of salt solution that reaches the evaporation zone.

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