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NATURAL ANALOGUES OF CEMENT: OVERVIEW OF THE UNIQUE SYSTEMS IN JORDAN

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ABSTRACT

In many radioactive waste repository designs, cement-based materials are expected to dominate the repository and models of cement evolution predict that leaching of the cementitious material in the repository by groundwater will produce an initial stage of hyperalkaline (pH~13.3) leachates, dominated by alkali hydroxides, followed by a longer period of portlandite and C-S-H (CaO-SiO_2-H_2O) buffered (pH~12.5) leachates. It has also been predicted that, as the hyperalkaline porewater leaches out of the near-field, significant interaction with the repository host rock and bentonite buffer and backfill may occur. This could possibly lead to deterioration of those features for which the host rock formation and bentonite were originally chosen (*e.g.* low groundwater flux, high radionuclide retardation capacity etc).

The precise implications of cement leachate/repository host rock interaction has been studied in the laboratory and in underground research laboratories (URLs) and this work has been supported by study of natural cements in Jordan. These natural cements have been produced by the combustion of organic-rich clay biomicrites and are very close analogues of industrial cement. Following interaction with groundwaters, natural hyperalkaline leachates are produced and these move out of the cement into the surrounding host rock, subsequently interacting with and altering it.

Keywords: cementitious repository, natural analogues, overview

INTRODUCTION

The natural cements and associated hyperalkaline groundwater plumes of the Maqarin and Central Jordan areas are excellent natural analogues of cement-dominated repositories and provide the best sites currently known to examine the processes associated with the long-term behaviour of such systems (see Figure 1). The work carried out in Phases I to IV of the project (see [1], [2], [3], [4], and [5], for details) now provides a consistent picture explaining the origin of the hyperalkaline waters (with *in situ* pH values of up to 12.9, the highest ever measured for natural waters), the persistence of some of the plumes and the sequence of alteration occurring when such leachates interact with various host rock types.

The Jordan Natural Analogue Project was initiated in 1989 with Phase I, continuing with Phase II in 1991, Phase III in 1993 and Phase IV in 2001. The Maqarin site appears to be unique in that the hyperalkaline groundwaters in the area are the product of leaching of an assemblage of natural cement minerals produced as a result of high temperature-low pressure pyrometamorphism of marls (*i.e.* clay biomicrites) and limestones, producing a mineral assemblage which belongs to the sanidinite and pyroxene hornfels facies [6]. These natural cements are a reasonable analogy of the industrial OPC (Ordinary Portland Cement) which is likely to be used in waste isolation and repository construction [7],[8] ,[9].

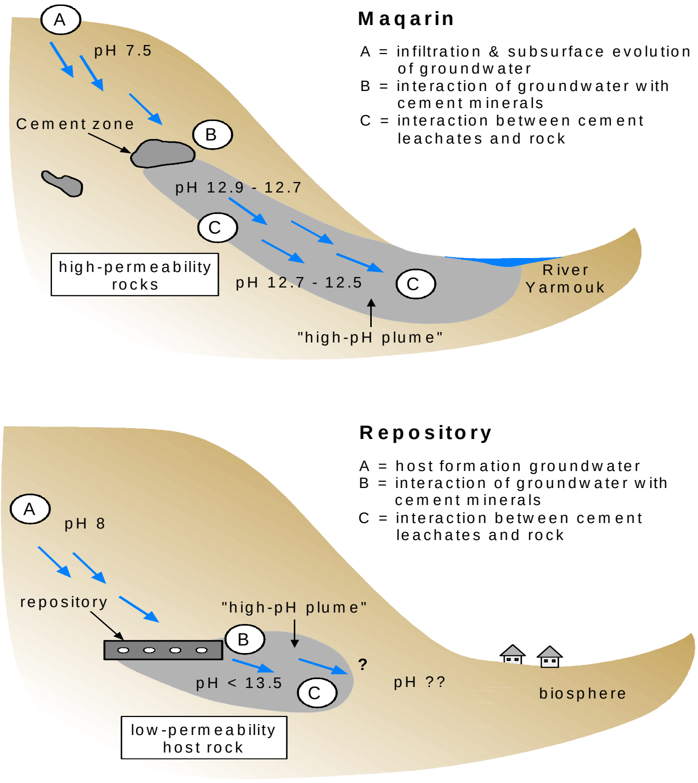

FIGURE 1: THE BASIS OF THE ANALOGY (FROM [10]). THE NATURAL CEMENT LEACHATES AT MAQARIN ARE ANALOGOUS TO THE HYPERALKALINE LEACHATES EXPECTED TO BE PRODUCED FROM A CEMENTITIOUS REPOSITORY. FIGURES ARE APPROXIMATELY TO SCALE.

In Jordan as a whole, at least three different types of hyperalkaline groundwater alteration have been identified and they appear to represent, by analogy, three different stages in the theoretical evolution, of a cementitious repository for the disposal of radioactive wastes (cf. [11]). They are:

- Stage 1: early, currently active, high pH Na/KOH leachates (Western Springs, Maqarin)
- Stage 2: intermediate, currently active, lower pH $Ca(OH)_2$ buffered leachates (Eastern Springs, Maqarin)
- Stage 3: late, currently inactive, near-neutral pH, silica-dominated leachates (Daba and Khushaym Matruk regions in central Jordan).

Whilst Phase I and Phase II were very much site-specific and process oriented (*e.g.* studies of the source term and its interaction with the host rock; testing the applicability of available thermodynamic data to hyperalkaline conditions; predicting the extent of hyperalkaline water/rock interaction using coupled models *etc*), Phase III provided a more regional perspective to the geological and hydrogeochemical evolution of the entire cementitious system. Phase IV went on to better define the local hydrogeology (and the impact of the cement leachates on it), examine the impact of the leachates on matrix diffusion in the host rock, study the microbiology of the Maqarin site in further detail, look at the behaviour of Re (as an analogue of Tc IV and VII) and examine clay alteration (as an analogue of the alteration of bentonite buffer in a cementitious repository) at the newly studied Khushaym Matruk site. Finally, comparison of the long-term alteration of the advective-flow dominated Maqarin site with the diffusive-flow dominated Khushaym Matruk site allows better prediction of the likely affects the hyperalkaline leachates will have on a wide range of repository host-rock types.

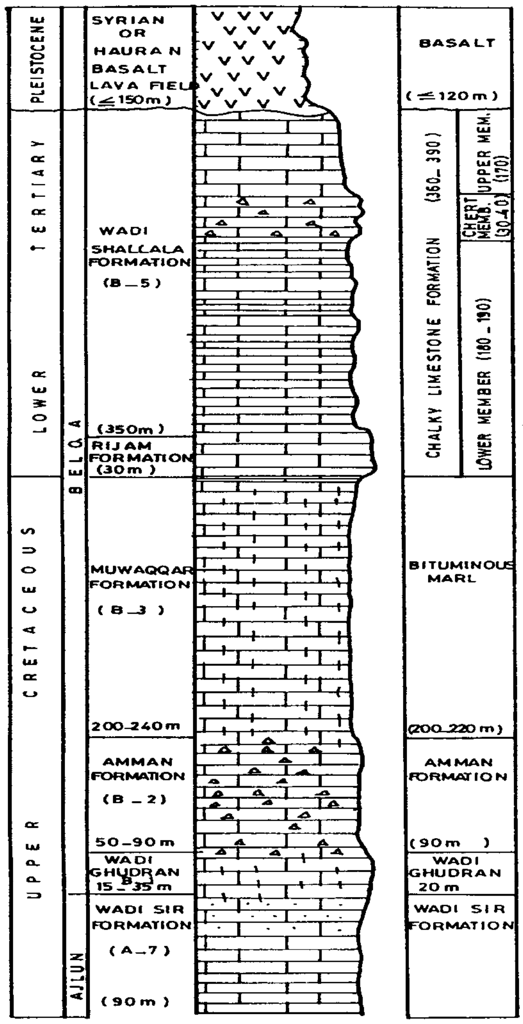

FIGURE 2: REGIONAL LITHOSTRATIGRAPHIC CORRELATION CHART (FROM [12])

SITE DESCRIPTION

The Maqarin study area is located in the Yarmouk River valley at the Syrian-Jordanian border, 16 km north of the provincial town of Irbid, and the Khushaym Matruk site is in central Jordan, about 75 km south-southwest of Amman. A generalised stratigraphic column is presented in Figure 2 and of most interest is unit B-3, known locally as the Bituminous Marl

Formation, and units B-4 and B-5, the Chalky Limestone Formation. The natural cement is formed by the local combustion of the B-3 unit which is regionally an aquaclude. It appears that fractures or joints in the B-3 to -5 units (or, in some cases, karstic dissolution of the B-4 and -5 units) allow penetration of air or oxygenated groundwater into the kerogen-rich (up to 15 wt%; [7],[8]) B-3 unit. Spontaneous combustion is thought to occur when the abundant pyrite (up to 1-2 wt%; [6]) in the B-3 unit oxidises exothermically and heat accumulates in the bituminous marl due to the low thermal conductivity, so igniting the kerogen (see also discussion in [2]).

Generally, the natural cement is found as localised 'pods' in the B-3 unit, but it can be found occasionally in the B-4 unit. Interaction of 'normal' (i.e. pH 7-8) groundwaters with the natural cements produces the hyperalkaline groundwaters (which are directly analogous to leachates from cementitious repositories) and these go on to interact with the B-3 unit, forming the so-called hyperalkaline plume downstream of the cement body (cf. Figures 1 and 3). As shown in Figure 3, at the cement/host rock interface, the hyperalkaline leachates have not yet reacted with the host rock and so have a high pH and high concentrations of Na, K and Ca, reflecting the cement porewater chemistry. As the plume reacts with the host (aluminosilicate-bearing) rock, the pH falls, as do the Na, K and Ca concentrations in the groundwater, while the concentrations of Al and Si rise to a small degree. Beyond the distal edge of the plume, in the, as yet, undisturbed host rock, the groundwater pH is near neutral, the Na, K and Ca concentrations are lower and the concentrations of both Al and Si are higher than in the plume waters.

This pattern has consequences for the secondary mineralogy (Figure 3, bottom): C-S-H phases will be found in the fractures (through which the plume has migrated) in the proximal part of the plume, reflecting the fact that the leachate has not yet reacted with the host rock and is equilibrated with the C-S-H phases which make up the cement. As the leachate moves downstream and interacts with the aluminosilicates in the host rock (and the host rock groundwater and porewater), the Al concentration increases, precipitating C-A-S-H (CaO-SiO_2-Al_2O_3-H_2O) phases. At the distal edge of the plume, the leachate has reacted with an even larger volume of host rock (and the host rock groundwater and porewater) and eventually precipitates zeolites as the Al concentration in the groundwater becomes high enough and the pH low enough. As these secondary phases have much larger volumes than the primary phases they replace, the matrix porosity and flow porosity slowly decrease until being effectively sealed.

DISCUSSION AND CONCLUSIONS

It is all but impossible to cover all of the areas of interest touched upon over the 15 years of the Jordan Natural Analogue Study (detailed in over 5,000 pages of reports and papers) so, here, focus is placed on those results which are of direct relevance to a cementitious radioactive waste repository SA. These are split into two groups, quantitative and qualitative.

Quantitative conclusions

The conceptual model for the evolution of a cementitious repository-derived hyperalkaline plume in a generic host rock (see Figure 3) is largely consistent with observations at the sites, although the diffusive system at Khushaym Matruk still requires further, detailed study [5].

Hyperakaline pore fluid conditions generated by minerals directly analogous to those in modern industrial cements are long-lived (up to one hundred thousand years) under the advective flow conditions found at the Maqarin sites [7],[8].

Reactions between hyperalkaline waters and the host rock mostly have positive reaction volumes and thus open porosity (fractures or porous media) will be sealed by the precipitation of secondary phases.

Interaction between hyperalkaline waters and the host rock occur extensively and (small aperture) fracture sealing occurs within timescales (years to hundreds of years) which are short in repository terms. Further work is required at Khushaym Matruk to establish timescales of sealing under diffusive conditions [5].

The altered rock matrix appears to be accessible to diffusion of aqueous species to a depth of several centimetres, but further study (e.g. on existing core material from Maqarin and in the laboratory with core infiltration experiments) is required to produce data of use to SA (cf. [5]).

Sequences of minerals predicted by coupled (geochemical and transport) codes are very close to those observed in the hyperalkaline alteration zones at Maqarin, even if the specific phases cannot be represented due to a paucity of relevant thermodynamic and kinetic data (cf. [13]) and some phases may be indicative of other reaction periods (*cf.*[5]).

The effects of the site hydrology (and tectonic/erosional processes) upon fracture sealing needs to be considered on a repository site-specific basis and fully integrated with the results of *in situ* experiments such as the HPF project when they become fully available (cf. [14]). Only then can an estimate be made of the long-term impact of the hyperalkaline plume on radionuclide retardation in the geosphere.

For example, as the hyperalkaline groundwaters react with the host rock, the fractures generally seal (due to the greater volume of the secondary phases), diverting flow elsewhere. At Maqarin, however, the active site tectonics means that the sealed flow paths are generally re-activated numerous times, so building up a confusing sequence of mixed secondary phases. However, in a tectonically quiescent host rock, the fractures may remain sealed, causing significant changes to the original site hydrogeology (see discussion in [15] for details).

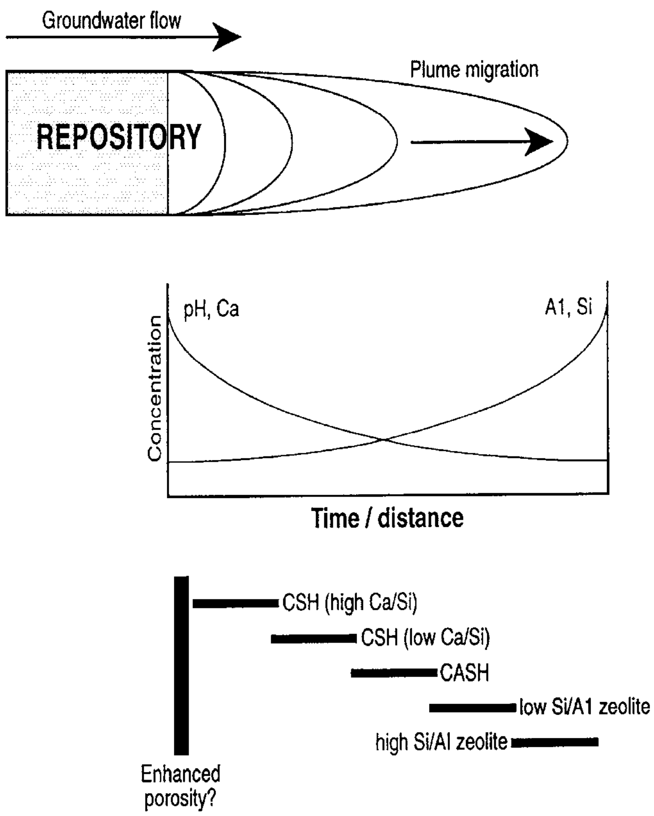

FIGURE 3: CONCEPTUAL MODEL OF THE HYPERALKALINE PLUME EVOLUTION (FROM [16]). AS THE HYPERALKALINE LEACHATES LEAVE THE CEMENT (TOP) AND ENTER THE HOST ROCK, THE GROUNDWATER PH (MIDDLE) INCREASES TO AROUND 13.3 (INITIALLY) AND CA LEVELS CLIMB DRAMATICALLY. AS THE LEACHATES INTERACT WITH THE HOST ROCK, PH AND CA LEVELS DROP AS CSH AND CASH SECONDARY PHASES FORM IN THE HOST ROCK AND AL AND SI LEVELS INCREASE (MIDDLE) AS ALUMINOSILICATE MINERALS IN THE HOST ROCK DISSOLVE. THIS GRADUAL BUFFERING ACTION OF THE HOST ROCK MEANS THAT THE SECONDARY PHASES WHICH PRECIPITATE IN THE HOST ROCK VARY WITH DISTANCE AWAY FROM THE CEMENT SOURCE (BOTTOM).

Thermodynamic databases of elements of interest to radioactive waste disposal provide conservative (*i.e.* solubilities are overestimated) estimates of solubility, despite the fact that the representation of the solubility controlling solid phases is too simplistic [17].

The amounts of colloidal material generated at the cement zone/host rock interface appears to be low, although any future analogue and laboratory work would benefit from a common approach to minimise method-inherent differences, so allowing better comparison of data [18].

Microbes are present in the hyperalkaline groundwaters, although their precise activity is currently difficult to define [19].

Qualitative conclusions

The natural cement at Maqarin and Khushaym Matruk may be considered a good analogy to an industrial cement and the leaching behaviour of both cement types is very similar[5].

All fractures examined within the plume at Maqarin (other than those currently water conducting) are sealed [5]. However, as the apertures of the fractures examined to date are generally small (mm to cm), it is currently possible to state only that thin fractures in a repository host will be probably self-sealing.

As a consequence of the fast rate of interaction between hyperalkaline waters and the wallrocks, it seems likely that radionuclides released from a cementitious repository will migrate through rock that has already been altered by the high-pH plume (although it must be emphasised that the radionuclide release scenarios for most repositories are still somewhat unclear here). This alteration affects both the geochemical (mineralogy) as well as the physical (porosity, connectivity) properties of the rocks.

Once a fracture is sealed, no further alteration can take place unless new pore-space is created by fracture reactivation. In this case, the fracture may then ”see” a pulse of hyperalkaline leachate with a composition no longer in equilibrium with the existing fracture infill (*ie* secondary minerals) and this will initiate further interaction with both the host rock and the fracture infill [7],[8], possibly releasing any radionuclides associated with the original secondary mineralogy [15]. The numerous phases of fracture precipitation (and dissolution) identified at Maqarin bear witness to recurrent events of alteration/precipitation, sealing and reactivation as does the range of ages so far reported for infill mineralogy.

In a highly porous rock (or flow system), it is possible that reaction will not rapidly seal the flow porosity. In this case, the distal part of the plume may be over-run by the middle part which may, in turn, be over-run by the proximal part of the plume (see Figure 3). Partial to complete replacement of previous secondary phases is to be expected, with the potential implications this has for radionuclide retardation. Such flow systems are probably of little relevance to deep repository host rocks.

Where very wide fractures are present, the same processes described immediately above may also occur and this could be of more significance to a repository [5].

Due to the high permeability in this surficial environment, the length of the hyperalkaline plumes downstream of the cement zones appear to be on the order of hundreds of metres.

In the lower advection rate systems of relevance to deep repository host rocks, plume lengths will probably be much smaller.

Re, studied as an analogue of Tc, is leached from the natural cement and, in some cases, is re-deposited in secondary fracture filling minerals (especially calcite and gypsum), possibly in the Re-VII form [20].

I retardation in the altered host rock is currently under investigation. Preliminary results indicate that I is present in the altered/non-altered host rock, as well as in the secondary fracture-fill minerals (T.Ishidera, *pers comm*, 2010). These data need to be integrated with the information on matrix diffusion in the altered host rock before a detailed assessment of I retardation can be made.

In those parts of the flow system which may be defined as 'open' (in the geochemical sense), or groundwater dominated, C-S-H phases are seen to dominate the secondary mineral assemblage whereas 'closed', or rock dominated, parts are zeolite dominated. By comparison with Maqarin and Khushaym Matruk, deep repository host rocks with low groundwater fluxes might be expected to be zeolite dominated.

Cement carbonation studies: this could effectively protect a cementitious repository from significant groundwater leaching and can be clearly seen in certain areas of the Maqarin site but has not, to date, been studied in detail

Clay alteration by the hyperalkaline leachates has been observed at Khushaym Matruk and would appear to show a decrease in expandibility of mixed layer illite-smectite following interaction with the leachates [5]. However, the number of samples analysed to date is small, so care must be taken with the extrapolation of these results to SA.

Despite major differences between the rock types at the Eastern and Western Springs sites at Maqarin and between Maqarin and Khushaym Matruk, the mineralogical composition of the secondary minerals at all sites is very similar, implying that similar reactions could be expected to occur at a repository host rock, *ie* the mineralogical information from Jordan appears to be directly transferable to repository conditions (see also discussion in [21]).

RECOMMENDATIONS FOR FURTHER WORK

The sites studied as part of the Jordan Natural Analogue Project remain the best available analogy of the long-term behaviour of OPC and the host rock surrounding a cementitious repository. Although much has been done over Phases I-IV, further effort could usefully be expended on [22]:

- the effects of the site hydrology upon the degree and form of fracture sealing: this remains open due to the difficulties of studying the sites, but perhaps too much emphasis was placed on the Eastern Springs at Maqarin at the expense of the Western Springs (and even Wadi Sijin). In addition, full integration of the data from the HPF experiment at Grimsel has not yet been possible and this would certainly aid the extrapolation of both data sets to potential repository sites
- do larger aperture fractures seal? The data from Maqarin do not help here but this process has not been addressed at the sites at Daba and Sweilah. In addition, provisional data from the new low alkali cement natural analogues in the Philippines and Cyprus suggest that wider fractures and joints can seal. If possible, this should be addressed in detail at these sites and should include an estimation of likely rates of sealing.
- matrix diffusion appears to continue in the matrix behind fractures as they seal, but the results are not statistically valid and this needs to be repeated before such an important mechanism can be included in repository SAs
- testing of thermodynamic databases and their associated codes: changes in both the databases and the codes mean that it would be worth updating these tests
- colloids: additional work at the Western Springs would be useful as the current data represent no more than a single snapshot. Future assessments should strive, where possible, to develop a common approach to minimise method inherent differences between NA and laboratory studies and try to access the distal part of the hyperalkaline plume where it is likely that most colloid formation will occur
- nitrate reduction: for those national programmes with a large waste nitrate inventory (e.g. Japan, UK), further examination of the nitrogen chain in the Western Springs would be of interest. This could include examination of denitrifying and/or ammonium reducing microbes – for example, are the populations significantly higher than other microbes? An assessment of the ammonium data would be of use to examine if the exchange of high levels of ammonium onto minerals in both the hyperalkaline plume and the clay biomicrite reduces radionuclide retardation. Apophyllite crystals from Maqarin show a significant deficiency in K^+ site occupancy (up to 25%) compared to ideal apophyllite stoichiometry, indicating probable major lattice substitution of K^+ by NH_4^+.
- assessment of Thaumasite Sulphate Attack (TSA) may affect concrete in waste disposal systems. Until recently, it was thought that TSA could only occur at low temperatures (less than 10°C). Recent observations and published thermodynamic data suggest that TSA also occurs at temperatures around 25°C or even higher. Maqarin is a site in which thaumasite and thaumasite-ettringite solid solution precipitation occur at ~25°C and therefore constitutes

a unique place to test predictions of thermodynamic models of TSA.

- deep repository host rocks with low groundwater fluxes might be expected to be zeolite dominated and the implications of this on radionuclide retardation could be examined at Khushaym Matruk, even if the zeolites are not necessarily a product of the hyperalklaine plume interaction
- Khushaym Matruk appears to be an excellent analogy for a diffusively controlled host rock and should be the focus of additional studies to improve the statistical relevance of the results presented here
- a full integration of the results of Phases I-IV with the now extensive body of laboratory data and (much fewer) URL experiments and modelling studies remains to be carried out. This would appear to be the most cost-effective manner to advance understanding of the likely long-term performance of the numerous existing and planned cementitious repositories

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