Condensed summary of current R&D on cementitious sealants for Deep Boreholes with HLW

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Abstract

Cement-based materials for use as sealants in underground waste storages must be erosion-resistant and chemically stable. Placement of highly radioactive waste (HLW) in boreholes may require that the rock is cement-grouted and stabilized by constructing concrete plugs. Where smectitic clay seals are in contact with concrete there is mutual degradation, and low-pH cement with inorganic superplasticizers, like talc, are recommended for preparing the concrete. This paper reviews our current state-of-knowledge concerning the grout and concrete sealing very deep boreholes (DBD) for purpose of high-level radioactive waste disposal. In this concept, the lower 2 km section of 4 km deep holes bored in crystalline rock could host waste-containers while the upper parts are sealed by dense clay and concrete[1]. The parts of such a hole that intersect fracture-poor rock are sealed with dense expandable clay while concrete is cast where fracture zones are intersected. The paper summarizes the available experimental results concerning the performance of grouts and concrete with talc as superplasticizer in contact with smectitic clay.

Keywords: Boreholes, Clay, Concrete, DBD, Radioactive waste, Talc, VDH,

1 Scope

The basic idea of disposal of high-level radioactive waste (HLW) according to concepts termed DBD is to locate the waste deep in rock with very heavy, saline, stagnant formational waters that are

[1] At present, continued R&D will show if the holes can be only 2.5 km deep with HLW in the lower 2 km parts

unlikely to rise to contaminate shallow ground waters (Brady *et al*, 2009; Sapiie and Driscol, 2009). Internationally, several models for deep disposal of highly radioactive waste (HLW) have been launched (cf. Nirex, 2004), in the UK by the University of Sheffield, and in the USA by Sandia National Laboratories. The present authors propose a concept (cf. Pusch *et al*, 2012b) for the disposal of highly radioactive waste, involving placement of the heat-producing waste in the lower 2 km part of 4 km deep holes bored in granitic rock, i.e. the deployment zone, that relies on the sealing capacity of engineered barriers in the form of concrete and clay in the upper parts of the so-called VDH holes (Fig.1). The described concept requires that those parts of such holes that are located in fracture-poor rock are sealed with dense expandable clay, while concrete is cast where pre-grouted fracture zones are intersected. Matters of particular importance are 1) function of the grout, concrete, and clay seals, 2) chemical stability and physico/chemical evolution of contacting concrete and clay seals. The clay seals consist of highly compacted smectite clay confined in perforated tubes ("supercontainers"), that are used also for hosting HLW canisters separated and surrounded by well fitting very dense clay blocks.

Fig.1.

The criteria for the cement-based grout is to be sufficiently fluid for sealing off finer fractures and to provide erosion resistance during the installation of concrete and clay seals. For the concrete it is required that it is coherent at casting and has a sufficiently high bearing capacity and low compressibility for carrying the load of subsequently installed series of clay and concrete seals. The hydraulic conductivity of the hardened concrete should be lower than that of the surrounding fracture zone. Since the concrete must perform acceptably for up to 100,000 years according to most national environmental protection agencies, and the cement component will ultimately be dissolved and lost, the rest, i.e. the aggregate components of the concrete, must still provide sufficient support for overlying clay and concrete seals in deep boreholes. The aggregate grains must therefore be very densely packed and have a granular composition that resists erosion.

The present study reviews new types of chemically stable organic-free cement-poor grout for sealing fractured rock, and talc-based concrete for casting under water-saturated conditions within deep boreholes. The properties of special importance considered are fluidity, mechanical strength, rate of strengthening, and minimized weakening when in contact with smectite-rich clay.

2 Composition and function of cementitious sealing materials for VDH storage of radioactive waste

2.1 Grout in fracture zones

The role of the grout in sealing VDHs is to minimize the risk of erosion and loss of cement particles and ultra-fine aggregate particles from the concrete to be cast, and from adjacent clay seals. The recommended recipe is a mixture of 10 % low-pH cement, 10 % talc and 80 % finely milled quartzite and silica flour (Pusch *et al*, 2012a; Mohammed *et al*, 2013, Mohammed, 2014, Mohammed *et al*, 2014a, 2015a). The grouting is made through 70-100 mm cored holes that are bored in conjunction with the detailed site characterization of host rock during exploration of the VDH location. Injection is made using the highest pressures possible with vibrations superimposed (Pusch, 1994; Mohammed, 2014). Very fine fissures will not be sealed but fractures with a hydraulic aperture of a few tens of micrometers can be successfully tightened.

The proposed grout type contains a low concentration of cement for minimizing the increase in porosity that will follow during dissolution and erosion of the cement component over the long-term. The granular composition is optimized according to packing theory (Pusch *et al*, 2012a; Mohammed *et al*, 2013). Using a fly ash-based low-pH cement, which is more stable than Portland cement and chemically reacts with talc, provides an ultimately high strength but a slow strengthening rate, which can be accelerated by adding the strongly thixotropic clay mineral, palygorskite. Once forced into rock fractures the grout stiffens and serves as a filter that hinders fine particles from adjacent clay seals to migrate through it and be lost in the fracture zone (Pusch et al, 2012a; Mohammed, 2014). Talc

($3MgO.4SiO_2.H_2O$)[2] is hydrophobic and low-viscous and does not form gels. It has no negative impact on the environment and is chemically stable in ordinary groundwater.

Penetration of new cement grouts, containing low-pH cement, powdered quartz and talc, has been investigated in laboratory experiments (Mohammed, 2014). The aggregate/cement ratio was very high 11.2-17.6 and hence also the density, but the water/cement ratio was also high (1.18-1.60) making the grouts behave as Bingham fluids. The penetrability into slots simulating rock fractures with an aperture of 100-500 µm was determined using static, constant pressure, and superpositioning oscillatory pressure waves on the injection pressure. The aim was to test the hypothesis that "dynamic" injection can increase the penetrability of cement-based grouts (Pusch, 1994), and to work out theoretical models for predicting the penetration into fractures.

The experiments highlight the role of the rheological properties and the filtering behaviour of certain grouts (Mohammed *et al*, 2014b). The following observations were drawn from the study of the low-pH cement-talc grout types:

- Effective penetration of grouts into fractures with smaller aperture than 100 µm requires that the viscosity is lower than 0.05 Pas. Here, injection under static, constant pressure is preferable,

- Effective penetration of grouts in fractures with an aperture of 100-500 µm can be achieved for grouts with a viscosity of 0.05-1.0 Pa s. Injection under dynamic pressure conditions is optimal,

- Grouts with a viscosity of 1-50 Pa s can enter fractures with apertures larger than a few millimeters,

[2] VWR International Company, UK.

- Measuring of the viscosity of freshly prepared grouts can be made by viscosimeters and capillaries. The latter is practical for rapid checking of the fluidity on the construction site,

- Theoretically predicted penetration depths are in fair agreement with laboratory test data,

- Dynamic injection has successfully been made on full scale in 760 mm diameter boreholes (Pusch, 1994).

2.2 Concrete cast in VDH

2.2.1 Preparation of holes

Deep VDHs have varying diameters which causes rock fall and other types of excavation damage that must be smoothened and stabilized by first boring to a somewhat larger diameter than intended, followed by casting of concrete between packers, in turn followed by re-boring to the intended diameter (Pusch *et al*, 2013a). The holes need to be stabilized and rinsed before installation of the supercontainers (cf. Fig.1). Stabilization involving reaming, concrete casting and re-boring can be made by applying techniques used commercially in deep drilling projects (Brady *et al*, 2009). After cleaning, the topography of the borehole walls is scanned for determining the actual hole geometry as part of exploring placeability of the supercontainers. Techniques, tools and experience developed at deep-drilling in the petroleum industry are utilized.

2.2.2 Concrete seals

Preparation

The concrete cast where the holes intersect fracture zones has the purpose of supporting clay seals placed upon it and of preventing clay particles from the adjacent clay seals migrating along fractures.

A promising candidate concrete is akin to the grout mentioned, except for the granulometry of the aggregate, and consists of 6-10 % low-pH cement (by weight), 10 % talc and 80 % well graded quartzite with silica flour (Pusch *et al,* 2012; Mohammed, 2014). Merit 5000 cement manufactured by SSAB Merox AB, Oxelösund, Sweden, was used for preparation of the concrete in a series of experiments for investigating the curing mechanisms and evolution of physical properties. The aggregate was finely crushed quartzite and talc was added as fluidizer. The concrete recipe, developed on the basis of modern packing theory (Mohammed *et al*, 2012), is shown in Table 1.

Table 1

The concrete is emplaced by squeezing it out from a container, starting at the bottom of the depth interval to be filled and pulled up in parallel (Fig. 2). The pump pressure required to bring the concrete on site while displacing the mud, which weighs only 1100 to 1200 kg/m^3, is very moderate. The length of each concrete plug is determined by the axial extension of the intersected part of the fracture zone. Concrete can be conventionally pumped down and out through a tube but better precision is expected by charging the container with a predetermined amount of coherent, rather stiff concrete prepared by mixing the solid cement components with “Dry Water”. It consists of microscopic droplets confined in microscopically thin silica “shells” that break and release water when compacted (Forsberg *et al*, 2017). The equipment can be used also for installation of dense clay plugs in holes of limited length.

Fig.2

Construction of VDH concrete seals where the holes are intersected by fracture zones means that concrete is cast upon placed clay seals up to the upper end of the respective fracture zone, where the next clay seal will be installed. The chemical interaction between concrete and clay causes mutual degradation that must be taken into consideration (Warr and Grathoff, 2010; Pusch *et al*, 2013b). Ordinary concrete with Portland cement as binder is not suitable because of its poor chemical stability over longer periods of time and because it generates a plume of high pH, more than 12, which attacks contacting clay seals. Commonly used organic superplasticizers for achieving fluidity of cast concrete are expected to give off organic colloids that can transport radionuclides and are suitably replaced by inorganic fluidizers of which talc is recommended (Pusch *et al*, 2012a, Mohammed *et al*, 2013). As in the described grout, talc replaces organic superplasticizers in the concrete proposed here (Mohammed *et al*, 2014a).

Evolution of stable concrete seals

The concrete proposed to be used in a VDH of the described type has a density of 2070 kg/m^3 and will be exposed to temperatures up to about 60^{o}C in the lowest part of the sealed zone and up to 150^{o}C in the deployment zone (cf. Polsky and Capuano, 2008; and Nirex, 2004). Samples of talc-concrete samples were cured in hydrothermal cells where they were exposed to a temperature gradient of 20-75^{o}C and 35-150^{o}C, respectively for 73 days followed by determination of the hydraulic conductivity and compressive strength (Mohammed, 2014; Mohammed *et al*, 2015b).

Table 2 gives the unconfined compressive strength of samples cured under different conditions. As for all brittle materials failure took place in the form of fracturing parallel to the loading direction.

Table 2

The strength of the 75^{o}C and 150^{o}C samples was almost the same, indicating that the strengthening processes were complete already at 75^{o}C. The strength of the sample cured at 150^{o}C, being the maximum temperature in the deepest part of the VDH concept considered, indicates that talc-concrete has a potential of being sufficiently stable early after placement but extended testing is required for assessing its long-term performance. The fact that the density of the concrete is very high suggests that it will serve acceptably even if the cement component dissolves. In a real VDH, sufficient bearing capacity is reached in a few days with the initial low-electrolyte water in the pores of the clay, and subsequent intrusion of more saline groundwater, especially at depth, will cause saturation with calcium leading to even higher strength. These findings agree with the results obtained by i.a. Behnood and Ziari, 2008, and Emerson *et al*, (2010).

Table 3 gives data of the hydraulic conductivity of the concrete made with the samples in oedometer cells before loading them, using distilled water as percolate. For avoiding leakage along the cell/concrete contacts the samples were first extruded from the cells and coated by smearing dense smectite-rich clay paste on them. The paste had a hydraulic conductivity of less than E-11 m/s Mohammed, 2014; Mohammed *et al*, 2015).

Table 3

The high value for the sample cured at room temperature indicates the presence of large interconnected voids. The reduction to 1/10 of this value for the sample exposed to 75^oC in the hydrothermal treatment suggests that these voids were partly filled with gels or other precipitates formed by chemical interaction of cement and talc. For the highest temperature in the hydrothermal treatment, 150^oC, further reduction of the conductivity by 5 times occurred, indicating that precipitation had continued and that further blocking of voids and channels had taken place (Pusch *et al*, 2013b). It is reasonable to believe that significantly elevated temperature accelerates hydration which in turn produces more hydration phases able to chemically bind more ions.

The described changes in physical performance are closely related to changes in chemical composition as indicated by Table 4, showing that dissolution and precipitation processes took place with almost constant relative amounts of the respective elements up to 75^oC, which will prevail at about 2 km depth in the “sealed” part of the VDH. For the deepest part of the deployment zone, with temperatures up to 150^oC, one would expect more significant changes but the table merely indicates that the concrete lost iron and became enriched in aluminium[3].

Atomic absorption spectroscopy analysis was made of the water circulated at the cold ends of the hydrothermal cells. The concentration of leached Na decreased over the sampling period at all three temperatures. The most rapid rate of Na decrease was seen in the sample heated to 75°C and the

[3] Comment by Warr and Grathoff in Mohammed et al, 2015b

slowest in the 20°C sample. The lowest amount of leached Na, K, Ca and Mg was recorded at the highest temperature of 150°C, indicating that this concrete was thermally the most stable (Pusch et al, 2013a; Mohammed, 2014).

Table 4

Electron microscopy showed abundant cement phases in the form of thin interconnecting grain coating in the 25°C sample (Fig.3a). In contrast, the 75°C sample has noticeably coarse cement coating that partly fill void spaces (Fig.3b). The coarsest type of cement with thick clusters and pore fillings was observed in the concrete subjected to 150°C, whereby the precipitation of crystals as large as 2 µm was observed. This sample showed the strongest resistance to degradation.

The observations confirm that the enhanced thermal conditions led to more extensive precipitation of cement phases that provided both strength and lower hydraulic conductivity.

Fig.3

2.3 Interaction of clay and concrete in the VDH seal

2.3.1 Mechanisms and consequences

The clay and concrete seals in a VDH will undergo chemical and mineralogical changes that can significantly affect their sealing functions in the deployment part but should have little impact on the less heated, upper part. The two components are expected to have about the same porewater pH (<10), but are still expected to undergo mutual degradation as found in earlier investigations that show the types of cement/clay interaction that can take place at moderate temperatures (Warr and Grathoff, 2010). A recent series of hydrothermal tests of talc-concrete in contact with montmorillonite-rich clay was conducted at the Luleå University of Technology in Sweden for finding out the reactions between low-pH concrete and clay at temperatures up to 150^oC and their impact on the physical properties (Mohammed, 2014b; Mohammed *et al.*, 2015a). The results are summarized here.

Experimental

The hydrothermal cells of 50 mm diameter and 70 mm height contained two 20 mm high samples of Na-montmorillonite-rich clay[4] blocks with an equally thick disc of talc-concrete of the earlier described type cast between them (Fig.4). The clay samples had a dry density of 1500 kg/m^3 and were saturated with distilled water and 3.5 % $CaCl_2$ solution, respectively. The concrete cast over the lower clay sample had a (total) density of 2070 kg/m^3. The upper ends of three cells were connected to vessels with distilled water and the lower ones to vessels with 3.5 % $CaCl_2$ salt solution, all three being heated for 2 months at 21^oC, 100^oC and 150^oC, respectively. The fluid pressure was held at 500 kPa for avoiding boiling.

Unconfined compression was made from 40 kPa to 1500 kPa. Fig.4 shows the successive breakdown of the sample set stored at 21^oC temperature for two months before loading. Failure began in the clay at a pressure around 500 kPa (upper sample with distilled water). The saltwater-saturated clay (lower sample) was more brittle and slightly stronger, failing at about 600 kPa when the total compression of the whole set was about 20 %. The concrete remained intact until the pressure had reached about 4.3 MPa and the total compressive strain was about 50 %. Fig.5 shows the stress/strain behaviour of the set of clay and concrete.

Fig.4

Fig.5

[4] Smectite-rich Tertiary Holmehus clay of S/I type with a Na-montmorillonite content of 90 % (Pusch, Kasbohm, Hoang-Minh, Knutsson, Nguen.Thanh, 2015).

Loading of the samples cured at 100^{o}C showed that the ductility of the clay saturated with distilled water was still evident. It failed at a pressure of about 600 kPa, i.e. slightly more than for the 21^{o}C sample. The saltwater clay below the concrete had higher strength and failed at about 1 MPa pressure when the total compression was about 8 %, indicating 50 % higher shear strength than of the 21^{o}C clay sample saturated with salt water. The concrete remained intact until the pressure had reached about 4.3 MPa as in the 21^{o}C set. The total compressive strain was then about 45 %.

Fig.6 shows the successive breakdown of the sample set cured at 150^{o}C temperature, indicating that the ductility of the clay samples had disappeared. Both clay samples broke into a granular form, the one with distilled water at a pressure of about 1100 kPa, corresponding to a shear strength of 550 kPa, and the one with salt water at about 1300 kPa when the total strain was about 15 %. The corresponding shear strength was about 550 kPa for the clay with distilled water, i.e. about the same as for the 100^{o}C sample, while the salt-water clay had a shear strength of about 650 kPa, reaching a total strain of about 15 %. The concrete remained intact until the pressure was 3.3 MPa and the total compressive strain reached about 42 %.

Fig.6

On comparing the compressive strength of talc-concrete samples that were cured separately, and those cured in contact with the smectitic clay, both had the same strength after curing at room temperature. However, the talc-concrete sample was twice as that in contact with clay when cured at 150^{o}C. This indicates that the mutual interaction of the two materials involved stiffening and strengthening of the clay, and loss of strength of the concrete, possibly by becoming argillitic.

3 Summary

The VDH concept implies that the rock and sealants stay stable physically and chemically for very long periods, which requires that chemically induced changes, particularly dissolution, are keep at a minimum. The upper half of the 4km steeply oriented holes serves to isolate the lower part in which HLW canisters surrounded by dense clay in large containers and installed in clay mud. Where the holes are located in rock lacking significantly water-bearing fractures they will be sealed with dense clay, while intersection of water-bearing fracture zones requires casting of concrete that will occasionally be in contact with clay seals.

Preceding functional analyses (Pusch, 2008) had shown that the clay should be of smectite type for having a potential to expand and self-heal and adapt to the rock, and that the concrete should have low porosity and only little cement for having sufficient bearing capacity and sufficiently chemically stable. It is also a requirement that the concrete should not contain organic superplasticizers, and talc is presented as an alternative. In this review, which focuses on the concrete seals, we suggest that pre-grouting of the rock may be needed for preventing cement gels and clay particles from migrating into intersected fracture zones and reduces degradation of the clay and concrete seals. Summarized are suitable grout types of low-pH cement with talc as a superplasticizer and palygorskite for gel stabilization and filtering (Table 5). These mixtures may be further modified depending on the fracture geometry and nature of the mineral coating required.

Table 5

Based on the compilation the results of previous studies of suitable sealant materials for the VDH concept, and re-interpretation of relevant laboratory- and field investigations that led to this review, we provide the following recommendations for grouts:

- Select grout mixtures with high density by basing the granular composition on a suitable packing theory, and by selecting very quartz-rich aggregate material. Selection of finely crushed quartzite mixed with silica flour is preferable, and using a minimum amount of low-pH cement is the second most important requirement. Adding palygorskite can provide thixotropic strength, which is regained after injection, and filtering ability.

For concrete in VDH the following comments are valid:

- Concrete consisting of 6-10 % low-pH cement, 10 % talc and 80 % well graded quartzite with silica flour and a water/cement ratio of 3-4 gives suitable concrete for being cast in VDH. pH is 10, which is well below the value that is critical for contacting smectitic clay seals,

- Heating of talc/low-pH concrete to the moderate temperatures in the upper 2 km part of VDH causes only small changes in any period of time. In the deepest part of VDH the temperature will be up to 150^{o}C during several hundred years and stay over 100^{o}C for hundreds of thousands of years causing stiffening and brittleness of the concrete that undergoes comprehensive chemical changes,

- Concrete in contact with smectite clay enhances stiffening and strengthening of the smectitic clay and brittleness and weakening of talc-concrete at 150^{o}C. This reduces the ductility and self-sealing potential of both components in the lower part of VDH where the radioactive waste will be enclosed in very dense “argillaceous” matter.

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Figure captions

Fig.1. VDH. Left: Casing-supported (C) holes sealed with clay in "supercontainers", and concrete, cast on site, to 2 km depth. Right: In the lower, "deployment" zone (2-4 km), sets of supercontainers with HLW canisters (W) surrounded by clay, and separated by blocks (B). The supercontainers are of copper, steel or titanium and submerged in soft clay mud (DM), (Pusch, 1994, 2012; Pusch *et al*, 2012a).

Fig. 2. Equipment for placing prepared specific amount of coherent concrete. (By courtesy of Drawrite AB).

Fig.3. Typical SEM micrographs of the hydrothermally treated concrete samples imaged with a secondary electron detector. a) 20^{o}C, c) 150^{o}C (Warr, Greifswald University).

Fig.4. Upper: Experimental set-up. Lower: Compression stages of sample cured at room temperature. Initial failure took place in the upper clay sample at about 500 kPa pressure (Mohammed, 2014).

Fig.5. Compression stages for the 21^{o}C samples. Initial failure took place in the upper clay sample at about 500 kPa (Stage A). Failure of the lower, salt clay sample occurred at about 600 kPa pressure

(Stages B and C). The concrete began to fail at about 48 % total compression (Stage E), (Mohammed, 2014).

Fig.6. Compression stages of the samples kept at 150^{o}C temperature. Initial failure took place in the upper clay sample at 1100 kPa pressure (Mohammed, 2014).

FIGURES

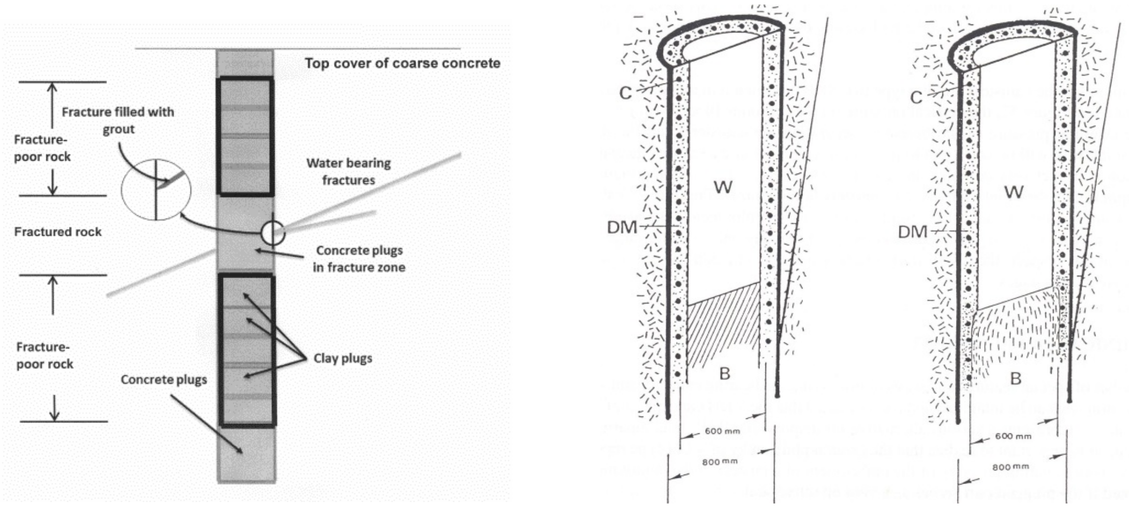


Fig.1.

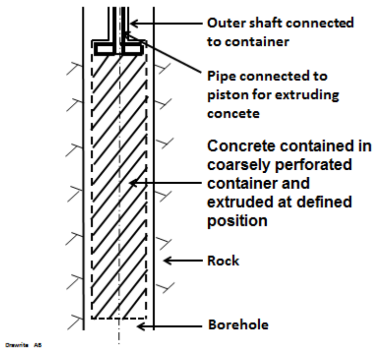
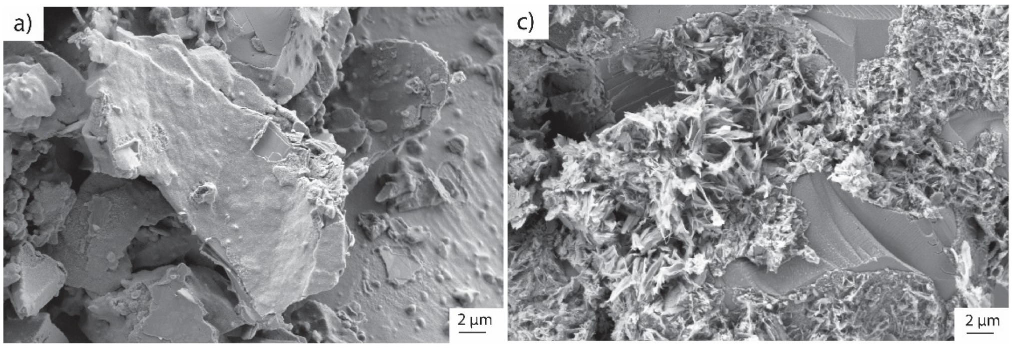


Fig. 2.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2 µm
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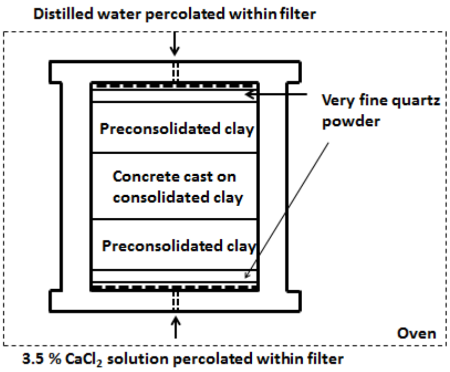
Fig.3.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Very fine quartz powder
Preconsolidated clay
Concrete cast on consolidated clay
Preconsolidated clay
Oven
3.5 % $CaCl_2$ solution percolated within filter

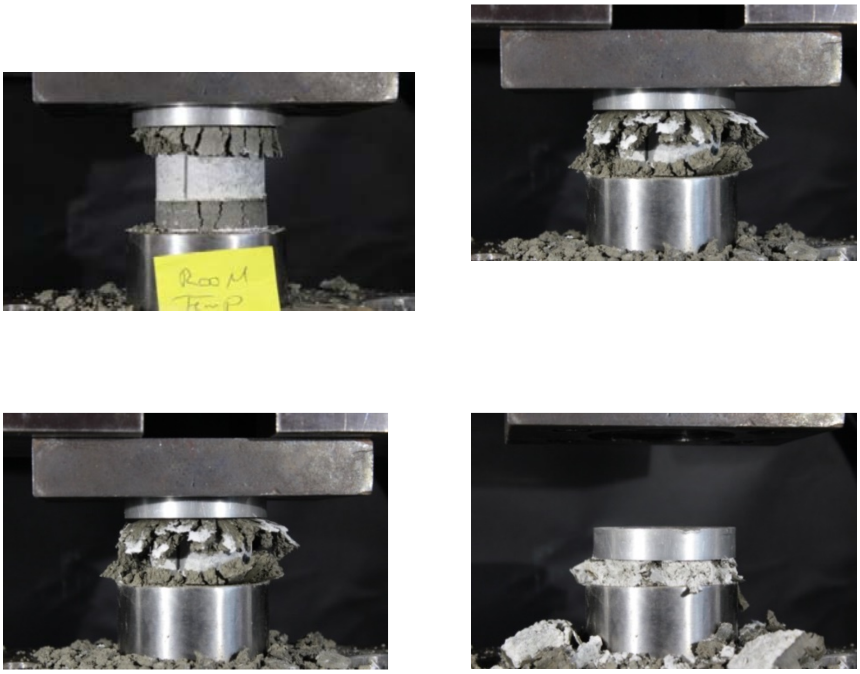

Fig.4.

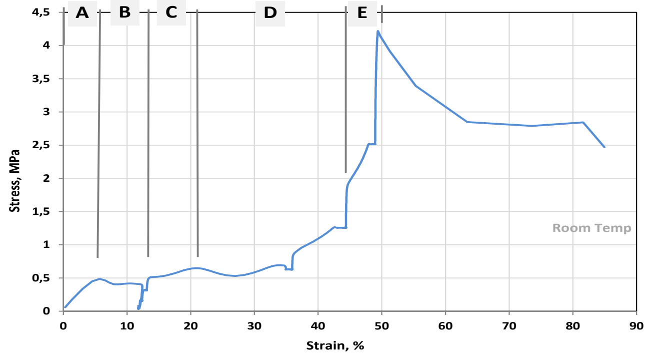

Fig.5.

Fig.6.

Tables

Table 1. Talc-concrete recipe (Mohammed et al, 2013).

Merit Cement (%)	Talc (%)	Aggregate (%)	Water/cement ratio	Aggregate/cement ratio	Density, kg/m^3	pH
6.5	9.5	84.0	3.6	12.8	2070	10

Table 2. Uniaxial compressive strength of samples exposed to hydrothermal treatment for 73 days (Mohammed, 2014; Mohammed *et al*, 2015b).

Sample of talc-concrete cured at respective temperatures	Compressive strength (MPa)
Room temperature 20^{o}C	4.52
Heating at 75^{o}C	9.16
Heating at 150^{o}C	9.00

Table 3. Hydraulic conductivity of talc-concrete samples cured at different temperatures and percolated for one month (Mohammed, 2014; Mohammed *et al*, 2015a).

Talc-concrete sample cured at respective temperatures	Hydraulic conductivity, m/sec
Room temp. (20^{o}C)	1.8E-08
Heating (75^{o}C)	2.5E-09
Heating (150^{o}C)	5.6E-10

Table 4. Change in chemical composition of talc-concrete cured at different temperatures (Pusch *et al*, 2013b).

Temperature	CO_2	Na_2O	MgO	Al_2O_3	SiO_2	CaO	TiO_2	FeO
20°C	28,6	0,1	9,8	3,8	60,4	0,9	0,5	3,4
75°C	21,7	0,1	19,0	2,3	54,0	1,2	0,2	5,3
150°C	23,5	0,3	10,5	7,5	56,7	1,5	n.d	n.d

Table 5. Components and mix proportions in grams of investigated grouts (Mohammed, 2014; Mohammed *et al*, 2014a).

Grout components (g)	Merit 5000 cement (low-pH)	6.25	6.25	6.25
	Palygorskite	9.38	12.50	18.75
	Ground crushed quartzite	43.75	43.75	43.75
	Distilled water	70	80	110
	Packing degree	0.393	0.386	0.374
Mix proportions	Water content %	118	128	160
	Water/cement ratio	11.2	12.8	17.6
	Cement/aggregate ratio	0.14	0.14	0.14
	Palygorskite/Agg. ratio	0.21	0.29	0.43
	Fluidity	High fluidity	Very high fluidity	Very high fluidity