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Article

Compositional Features and Swelling Potential of Two Weak Rock Types Affecting Their Slake Durability

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Abstract: Weak and weathered rocks are well known for their sensitivity to changes in moisture content. Degrading behavior is common in weak rocks with moisture-sensitive mineral components and present numerous stability problems. The slake durability is a measure of the resistance to weakening and disintegration of rock materials which quantitatively distinguishes durable from non-durable rock materials. Several rock material parameters interact on the process of disintegration when exposed to cyclic moisture changes, whereby the content of clay is believed to play a major role. This manuscript evaluates the overall material composition of flysch and serpentinite rocks cored from the wall of the shotcrete-lined headrace tunnel of a hydropower project, including minerals, structure, porosity, the presence of micro-discontinuities, and swelling potential, and links these properties to the slake durability. Further, the different methods used to assess compositional features affecting the durability of weak rocks are evaluated and discussed. The manuscript argues that the mineralogical composition and microstructures present in the intact rock and the content of moisture-sensitive constituents, as swelling clays, control the long-term durability of weak rock material. It is demonstrated that XRD assessments are not sufficient to detect the content of brucite and swelling components, and that methods as thin section and SEM analyses should be carried out in the assessment of weak and weathered rock mass.

Keywords: weak rocks; slake durability; rock composition; optical methods; construction stability

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1. Introduction

Instabilities in underground openings caused by different rock degradation processes which lead to substantial maintenance and repair costs are frequently reported [1]. The degradation potential of the intact rock is highly dependent on different material properties which interact on the rock durability, as rock composition, porosity, fracturing, structural/textural characteristics, and swelling potential [2–7]. The initial rock properties may change during weathering and/or alteration processes and can result in a weak and moisture-sensitive rock structure [8], especially if constituting minerals are decomposed or altered into clay minerals. However, the alteration process may also act on the rock in a positive way, as when the re-welding of existing rock discontinuities occurs through the deposition of ferrous oxides, calcite, or siliceous filling material [9]. As the intact rock is one of the most important parameters influencing the mechanical response of rock masses in underground excavations, the degree and nature of weathering and/or alteration should be characterized.

The characteristic behavior of clay-bearing rocks when unloaded during tunnel construction and exposed to water during operation are their unique tendencies to swell and slake [5]. Cyclic wetting and drying are considered as one of the main processes that can induce micro-fissures in the rock material and may result in disintegration [10,11]. The behavior is strongly dependent on the interaction between the rock constituents and moisture, which often results in volume changes, dissolution, the development of fractures, and flaking of the surface layers [12–14]. Voids, including pores and micro-fractures, provide access routes for water to gain access to water-sensitive minerals, leading to swelling and further material degradation [4,5]. Swelling of minerals, such as smectites, mixed-layer clays, and zeolites results in a volume increase of the crystal lattice and expansion forces in the rock material [15,16]. If the volume increase is prohibited, the swelling results in the development of a swelling pressure which, in turn, may lead to fracturing of the surrounding rock material. These changes of material properties may, in turn, accelerate the total degradation of the rock mass when exposed to moisture changes, as in the case of hydropower tunnels. Further, the presence of minerals which may dissolve into components that deteriorate the shotcrete lining of tunnels should be assessed.

Researchers have attempted to quantify the degradation susceptibility of weak clay-bearing rocks aiming to classify the rock types and assess required support in underground openings. Different procedures are available to determine the durability of materials from which the selection of appropriate engineering parameters can be obtained [5], as for example the slake durability test [2]. The slake durability test aims to accelerate weathering to a maximum by combining the processes of slaking and sieving whereby the latter requires some motion in the test process. The technique is discussed in the ISRM-suggested method for determination of the slake-durability index [17]. However, the suggested method is unable to distinguish the material parameters involved as well as specific material characteristics examined.

Recent studies have highlighted the importance of clay mineralogy, fabric, and strength in controlling the physical breakdown of rock material and it is possible to use these features to determine the durability and identify potentially unstable rock materials. The influence of geological structures, textural features, and mineralogy on the breakdown of coal bearing mudstones and shales were discussed by [18]. The authors emphasized that bedding and stratification on small scales may give rise to planes of weaknesses in the rock material, and that microfracture orientations relative to these structures play an important role on the disintegration behavior. The authors further stressed that the capillarity and the build-up of capillary pressure are important factors in terms of water-uptake and consequential disintegration, where the presence of swelling clay minerals affects the rate of degradation.

The influence of the number of drying and wetting cycles and control of mineralogical composition and strength on durability of weak and clay-bearing rocks were presented in [19]. The study indicated that the type and amount of clay minerals are the main factors influencing the variations of the slake durability and also found a strong correlation to the amount of swelling clay. A positive correlation was also found between the durability index and the total carbonate mineral content, meaning that high carbonate content increases the slake durability of the rocks.

The mineralogical composition and textural features of some weathered rocks from Japan were studied by [20], who linked these properties to the slake durability of rocks. They found that the difference in slake durability can be attributed to occurrences of smectite and zeolite as alteration minerals, and also pointed out a general decrease in slake durability with an increase in the weathering degree of the rocks. According to [21], there is an influence of amorphous clay-sized materials on the swell-shrink properties of weathered rocks and soils. The amorphous materials include alumino-silicates, iron and aluminium oxides, silica, and other complexes too small or too poorly crystalline to produce recognizable crystalline phase peaks on X-ray diffraction (XRD) patterns. To detect these materials, the use of an electron microscope or similar methods were required. The results of this study showed that up to 74% of the clay-sized fraction of the materials tested was amorphous, mainly due to a high degree of weathering of the original rock material. The amorphous material also showed abundant swelling behavior despite the lack of recognized smectites.

Research was carried out on the effect of initial wetting on the durability of rocks, where a modified version of the slake durability test was proposed [16]. The modified version introduced a wetting phase of the samples prior to the cycles of wetting and drying in the slake durability test, and the temperature and consequential dehydration exerted on the samples between the cycles was constrained [16]. Both the ISRM [17]-suggested slake durability test and the modified version of the test [16] were performed on duplicate samples to compare the obtained slake durability indices and to evaluate the effect of initial saturation on the durability of different rock types. The results of the research [16] implied that some of the tested rock samples have a lower durability when initially wetted compared to their initial dry condition. This finding was explained by the content of clay-sized and weathered minerals, whereby the bulk mineralogical composition was assessed by XRD-analysis. In addition, the composition of the disintegrated material (<2 mm) after wetting of the samples was determined, whereby a slightly higher content of clay-sized and amorphous constituents was detected. However, a further assessment of material properties affecting the slake durability of weak rocks was suggested in order to better understand the differential behavior of the tested samples.

Hence, the main aim of this manuscript is to present results on rock composition consisting of X-ray diffraction analyses (XRD), optical methods (thin section analysis), and scanning electron microscopy (SEM) in order to identify critical textural and compositional features of the weathered rock material. The manuscript further presents swelling tests performed to assess the swelling potential of the rock material as swelling is considered as an important indicator on potential degradation behavior. The mineralogical composition, structural features, and swelling potential of the tested rock samples are then discussed and linked to rock durability. Finally, the different methods used to assess the compositional features and durability of the rocks are evaluated. The manuscript is to be considered as a continuation of the research work published by [16].

2. Materials and Methods

The research methodology of this paper is based on the foundation of a study presented by [16] on the effect of initial moisture content on the slake durability indices of flysch and serpentinite rocks. In the study, the authors found out that some rocks showed a lower durability when initially wetted and there was a differential disintegration behavior in a modified test compared with the ISRM-suggested [17] test method. The authors also concluded that the XRD analysis is not sufficient to understand mineralogical composition. The research question here is how do compositional features, such as texture, mineral distribution, and micro-fracturing affect the slaking behavior of samples of similar origin? What is the nature of the amorphous constituents and how do they influence rock durability? Is there a link between swelling potential and degree of slaking/disintegration? Are the traditional methods to assess rock composition diagnostic when applied to weak and weathered rocks?

To answer the research question abovementioned, similar rock samples as in the study of [16] were used in this study, including duplicate samples. The main aim was to uncover material properties affecting the slake durability of the rocks and to gain further understanding on the differential moisture sensitivity of the tested samples. To enable an understanding of the structural features and swelling potential of the material, further mineralogical analyses and swelling tests were carried out. However, the serpentinite samples were not prioritized in the XRD analyses, and the swelling tests of this rock type were limited to bulk sample tests due to relatively small amount of material loss of particles <2 mm during the slake durability test, similar composition among the samples, and high content of chrysotile (asbestos). The aim of the study was to obtain extended mineralogical, structural, and swelling analyses so that an increased understanding of factors influencing the durability and disintegration behavior of the tested rocks is established. Hence, the study mainly focused on detecting compositional changes of the material after the slake durability tests to evaluate structural/textural characteristics of the rock material and to detect swelling minerals and indicate the swelling potential of the material. The rock samples used for the study were the slaked material of Flysch (Flysch 6 to Flysch 11) and Serpentinite (Serp. 5–8) samples and the duplicate samples of Flysch (Flysch 6 to Flysch 11) and Serpentinite (Serp. 6 and Serp. 8). The test methods used were XRD, thin section, and SEM analysis as well as the swelling test.

2.1. XRD-Analyses

In order to detect eventual differences in the mineralogical composition of the samples after the slake durability tests compared with the composition prior to the tests, two separate XRD analyses were performed on each flysch sample, i.e., the material retained in the drum (>2 mm) and the disintegrated material passed through the drum and left in the container (<2 mm). The analyses were conducted using Bruker D8 ADVANCE where identification of crystalline phases was performed with DIFFRAC.SUITE.EVA software combined with the PDF-4+ database. A semi-quantitative estimation on the content of amorphous phases was performed and the quantification of the minerals was performed by Rietveld refinement in Topas, normally with an accuracy of 1–2 percent. The accuracy was, however, reduced with higher content of amorphous phases. Further, glycolation was used on fraction sizes of <6 μm to identify existence of swelling clays.

2.2. Thin Section Analyses

Since weathered and amorphous constituents lack long-range crystallographic order and produce broad humps with low intensities in XRD patterns due to the high degree of weathering, XRD may not produce adequate information on the mineralogical composition. Further, voids and microfractures control the rate of water ingress into the material and this is a factor which separates durable from non-durable rocks which are otherwise similar. Optical methods were therefore chosen to obtain complementary information on mineralogy, and to enable a determination of the structural features which could be helpful in interplaying on the slaking behavior.

Duplicate samples of Flysch 6 to Flysch 11 and Serp. 6 and Serp. 8 were used for the thin section analysis with an aim to gain information on porosity, micro-fracturing, grain-size distribution, clay content, and signs of weathering. The samples were impregnated with blue epoxy, aiming to detect open discontinuities and pores. An initial inspection of the samples was obtained by transmitted light microscopy of polished thin sections using a Nikon Eclipse E600 microscope. Optical micrographs were studied by using parallel and cross-polarized light. Further, the textural features were described by studying the thin sections in an Olympus bx51 microscope. Scans were obtained by stitching 5× microscope images together using the Olympus stream motion and the Märzhauser Wetzlar automated stage. In order to calculate the porosity, the scans of thin sections of each sample were uploaded in the software FIJI bundle of Image J. Color threshold was applied to identify the porosity using blue dye color.

2.3. SEM Analyses

The samples Flysch 9 and Serp 6 were analyzed by SEM to further assess clay mineralogy, amorphous constituents, crack infillings, and matrix composition. The first step was to concenter the textural features of interest by thorough assessments of the thin sections of the chosen samples, and then carry out focused SEM-analyses on these features. SEM data were acquired on the Hittachi SU6600 FEG SEM from the electron microscopy laboratory. Samples were coated with a 15 nm layer of carbon to avoid charging. SEM was operated at 15 kV acceleration voltage and 35 nA beam current for optimization of EDS analysis. EDS spectra and maps were acquired using the Bruker esprit 1.4″ software. SEM backscatter images were used to document detailed mineral associations in the samples, mainly using the compositional mode but also using the 3D mode to image clay mineral structures.

2.4. Swelling Test

Swelling behavior can be assessed using both volume expansion and development of pressure induced by the rock material on its surroundings. Free swelling tests on rock powder are index tests normally used to examine the swelling potential of swelling gouge in the weakness-zones and are also applied nowadays to identify the presence of swelling minerals in the pulverized material of intact rocks. The test measures the nearest percentage volume change of 10 mL of oven-dried powder when immersed in water.

Swelling tests under oedometric conditions are frequently carried out to determine the potential swelling pressure initiated by the rock when volume expansion is prohibited [22]. The oedometer test is the most common procedure of swelling pressure where the samples are radially constrained, and the axial pressure is measured. The test is usually performed on oven-dried and compacted powder samples. The prepared samples first undergo a compaction phase (24 h) prior to unloading (2 h) and swelling (24 h). The powder tests are well established, quicker, and straightforward, and they provide the possibility to use wide ranges of rock materials as the sample preparation does not rely on the quality of the rock material. However, ideally, the oedometer test should be performed on intact rock structures as this will comprehend the controlling factors of the material structure on the swelling capacity of the tested rock.

Through testing, all samples first underwent the free swelling test and the oedometer swelling pressure test. The oedometer swelling pressure test was performed according to the ISRM standard [17]. For the free swelling tests, all samples were prepared and tested according to the procedure given in Statens Vegvesen [23] for testing of the swelling gouge. The slaked material of the flysch samples was additionally tested to detect eventual changes in swelling potential after 4 cycles of slake durability tests. It is emphasized that both tests are meant to check if swelling can be a contributing factor on rock degradation and disintegration.

3. Results

3.1. XRD Test Results

The XRD analyses carried out on flysch rocks prior to the slake durability tests indicated quartz, chlorite, calcite, plagioclase, mica, and k-feldspar and main mineral constituents. However, the composition varied between the different samples, where high amounts of quartz and plagioclase were related to lower amounts of mica, chlorite, and amorphous phases, and vice versa. The mineral constituents of the serpentinite samples were found as difficult to assess by the XRD analysis. This is mainly caused by large amounts of weathered and amorphous phases.

The results of the XRD analysis carried out on the bulk samples prior to the slake durability test are summarized in Table 1. In the analysis of the results, only minerals consisting of more than 2 percent were accounted for and the values were rounded up to the nearest percent. Swelling clays were not quantified, but noted as present if these were detected after glycol treatment of the sample material. As the deviation between crystalline and amorphous phases was semi-quantitative, the accuracy of the results decreased with the increasing number of amorphous phases. In the table, the quantification of each mineral was based on the XRD results of the bulk composition. The retained material in the drum and the disintegrated material after slake durability testing was studied, including the dissolved material after the wetting phase of the modified test. Figure 1 compares the results, which should be considered as indicative on changes in mineral distribution due to the exposure of wetting and drying in both slake durability test procedures (according to ISRM standard [17] and the modified version presented by [16]).

Table 1. XRD analysis results of flysch and serpentinite samples before slake durability test, given in percentage (updated in [16]).

Minerals	Flysch						Serpentinite			
	6	7	8	9	10	11	5	6	7	8
Amorph	23	28	37	40	43	48	57	58	56	58
Brucite	-	-	-	-	-	-	1	1	-	1
Calcite	15	14	25	13	10	8	-	-	-	-
Chlorite	6	9	14	16	18	16	9	5	11	11
Chr./liz.	-	-	-	-	-	-	13	10	18	19
Enst./fost.	-	-	-	-	-	-	-	13	-	-
K-feldspar	2	2	1	1	1	1	-	-	-	-
Kaolinite	-	-	-	-	-	-	12	8	10	8
Magnetite	-	-	-	-	-	-	-	-	2	1
Mica	4	4	4	8	8	10	-	-	-	-
Népouite	-	-	-	-	-	-	4	2	2	3
Plagioclase	13	12	4	7	6	6	-	-	-	-
Pyrope	-	-	-	-	-	-	2	2	-	-
Quartz	37	31	14	15	14	11	-	-	-	-
Swelling clay detection [1]	yes	yes	yes	no	yes	no	yes	no	no	no

[1] Corrensite is the only detected mineral with known swelling potential, but is not quantified.

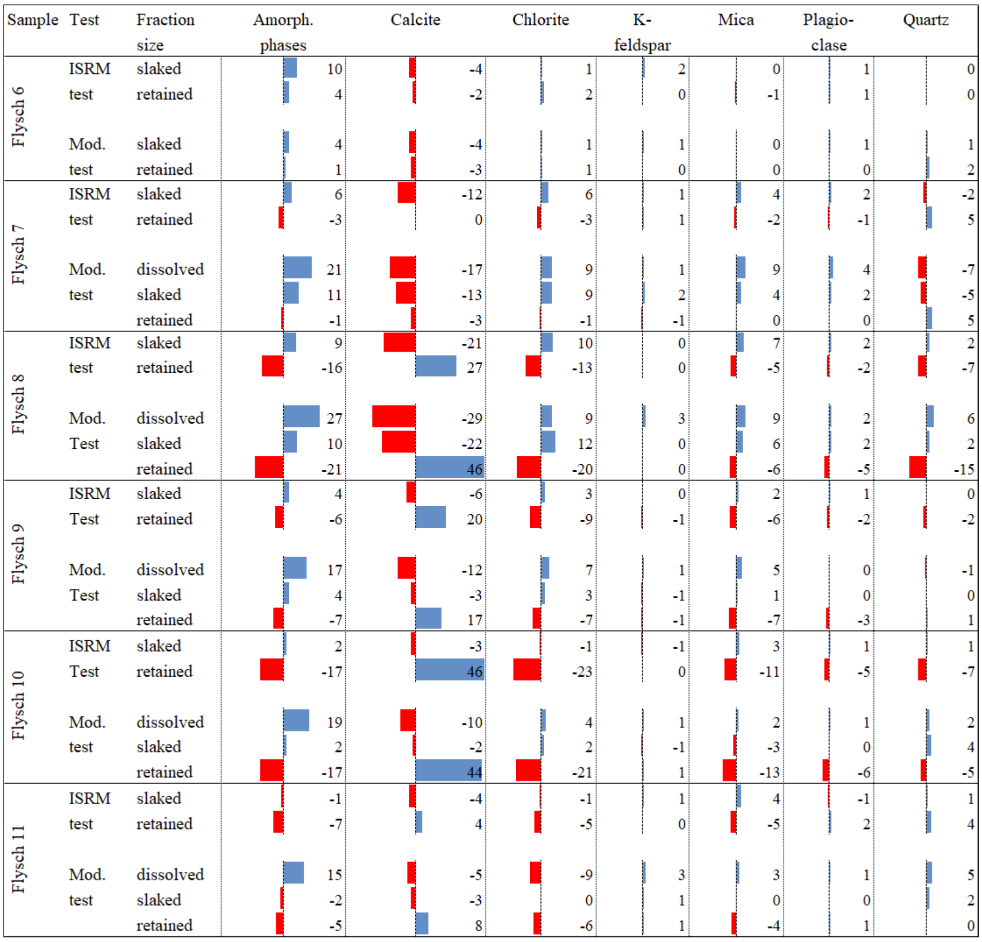


Figure 1. Illustration showing changes in mineral distribution of the slaked, retained, and dissolved material after the slake durability tests based on the XRD test.

In Figure 1, a red color indicates a decreasing order of the respective mineral compared with the bulk composition of the samples and a blue color indicates an increasing concentration. No color indicates no changes in mineral concentration.

It should be noted that the slaked and dissolved materials in Figure 1 are based on XRD analyses of the particles passing through the drum with a mesh size of 2 × 2 mm^2. The comparison of the XRD results prior to slaking (Table 1) and the corresponding results after slaking (Figure 1) shows that there was an increasing amount of mica, chlorite, and amorphous constituents on the disintegrated rock material passing through the drum (<2 mm). The calcite content increased in the retained material, whereas only minor changes were identified for the content of k-feldspar, plagioclase, and quartz.

3.2. Thin Section Test Results

The textural features, including mineral characteristics, grain size, porosity, microfracturing, and clay distribution of the samples were determined by the thin section analyses using plane-polarized light and cross-polarized light. The rocks were thereafter categorized into three main groups, i.e., sandstone (Flysch 6 and Flysch 7), mudstone/siltstone (Flysch 8, Flysch 9, Flysch 10, and Flysch 11), and weathered serpentinite (Serp. 6 and Serp. 8). The samples within each category have slightly different features regarding heterogeneity (grain size and mineral distribution), porosity, permeability (fracturing and cementing), weathering, and textural distribution of clay minerals (Table 2).

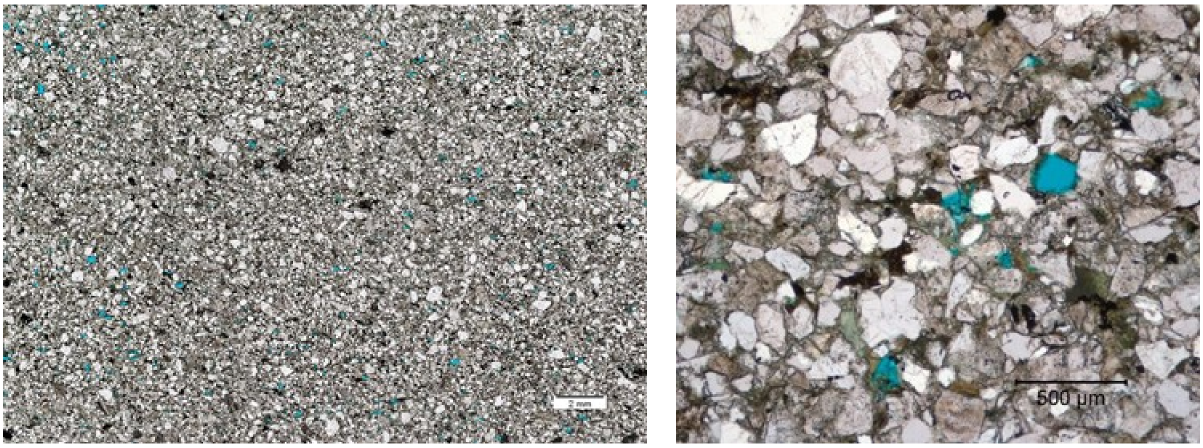
Table 2. Main textural features of the samples.

Specimen Name	Homogeneity Condition	Clay Matrix	Fracture Characteristics	Porosity (%)
Flysch 6	Homogeneous sandstone	Low, occasionally substitutions of feldspar/mica grains	-	0.06
Flysch 7	Homogeneous sandstone	Low, occasionally substitutions of feldspar/mica grains	-	1.03
Flysch 8	Heterogeneous mudstone, moderate microfracturing	Medium-high, distributed in matrix	Fractures mostly cemented with calcite. Some clay-filled microfractures penetrating matrix.	<0.01
Flysch 9	Heterogeneous mudstone, extensive microfracturing	High, distributed in matrix, pores and at grain borders	Both calcite-cemented and open or clay-filled microfractures.	1.02
Flysch 10	Heterogeneous mudstone, moderate microfracturing	Medium-high, distributed in matrix, pores and at grain borders	Both calcite-cemented and open/clay-filled microfractures.	0.25
Flysch 11	Laminated mudstone, extensive micro-fracturing	High, distributed in matrix, pores and at grain borders	Mainly open or clay-filled microfractures.	1.96
Serp 6	Heterogeneous serpentinite	Not detected	Veins as part of the serpentinite structure. Thin fractures along some veins.	0.30
Serp 8	Heterogeneous serpentinite	Not detected	Veins as part of the serpentinite structure. Thin fractures along some veins.	[1] 11.42

[1] It is likely that part of the porosity is due to disintegration of the samples during sample preparation.

Flysch 6 is very fine-to-medium-grained sandstone with angular/subangular grains. The rock is carbonate cemented, and the sand grains are dominantly of quartz, plagioclase feldspar, micrite clasts, and occasionally mica, chlorite, and microcline feldspar. Some rock fragments of carbonate, micaschist, volcanic rocks, and chert are observed. Diagenesis was verified by compaction and mica folding, chloritization of mica, chlorite as pore-linings and replacement within igneous rock fragments, and by precipitation of pore-filling calcite. The porosity and degree of micro-fracturing were very low.

Flysch 7 is also very fine-to-medium-grained sandstone with subangular grains. Similar minerals and clast types, as in sample Flysch 6, were identified, except lesser chlorite rims. In addition, heavy minerals of garnet, zircon, and Cr-spinel were observed. The rock is carbonate cemented with some minor disconnected visible pores. The porosity and degree of micro-fracturing were seen as very low. However, the porosity was slightly higher than that of Flysch 6. A thin section scan of the sample Flysch 7 in plane-polarized light is shown in Figure 2.



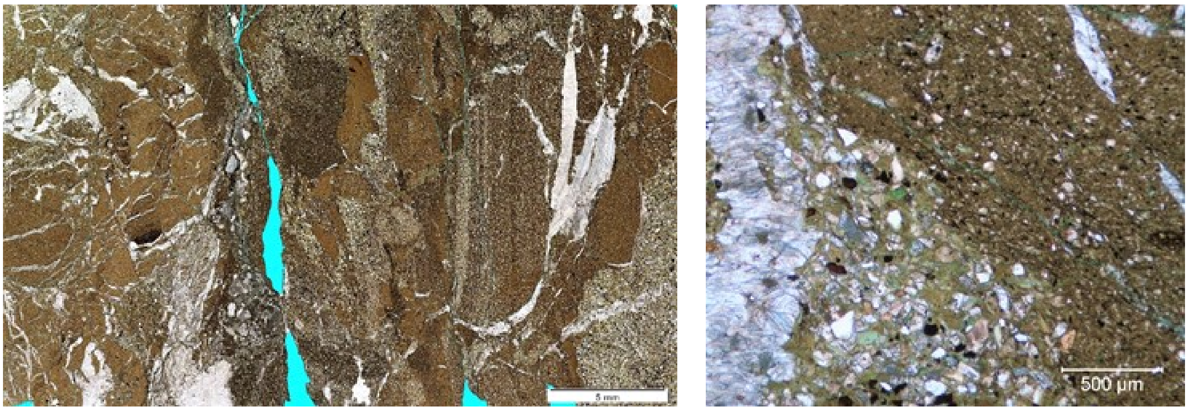
(**a**) (**b**)

Figure 2. Thin section scan of Flysch 7. (**a**) Overall texture. (**b**) Closer view on mineral grains and pores.

Flysch 8 is a very fine-grained-to-silty heterogeneous mudstone with an abundance of very fine-grained calcite fragments and trace amounts of quartz. The finest-grained brown zones are richer in clay. The rock is transected by coarse crystalline calcite cemented patches and veins. Calcite twins were observed, indicating a geological history of deformation processes. Some microfractures were observed along calcite veins and traversing the matrix. The porosity seems very low, and the microfractures were mostly carbonate cemented, resulting in a low permeability. A thin section scan of the sample in plane-polarized light is shown in Figure 3.

Figure 3. Thin section scan of Flysch 8.

Flysch 9 is a heterogeneous mudstone with rock matrix consisting of zones of clay-rich, brown matrix with silt-sized silica and opaque minerals and sandy mudstone with very fine-grains of detrital quartz, chert, feldspar, chlorite, mica, and opaque minerals. Calcite is dispersed in matrix and fracture-filling cement (veins). The rock structure showed a high content of microfractures. The calcite cemented veins penetrated different matrix zones, indicating that the cementing of the fractures occurred in a late stage of the rock's history. Figure 4 shows a thin section scan of the Flysch 9 in plane-polarized light.



(**a**) (**b**)

Figure 4. Thin section scan of Flysch 9. (**a**) Overall texture. (**b**) Detailed (closer) view of the matrix.

Flysch 10 is also a very fine-grained and heterogeneous mudstone with a brown clay matrix, possibly including clay minerals as illite, smectite, and/or chlorite. Very fine sand grains of quartz, mica, feldspar, and chert were observed in addition to coarse fragments up to granule size of different lithologies including rock fragments of calcite biomicrite, calcite fossils, porphyric felsic igneous rock, weathered microcline, and chert with chlorite. The rock was found to be transected with bended carbonate cemented veins, and some of them initiated along deformed brachiopod shells. The porosity looked low, but some microfractures seemed open, which make the rock permeable. A thin section scan of the sample in plane-polarized light is shown in Figure 5.

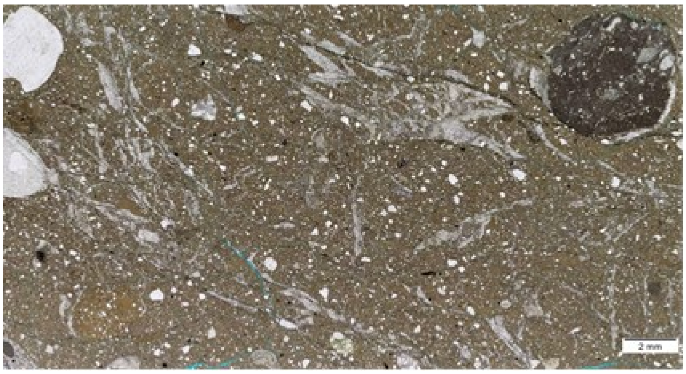
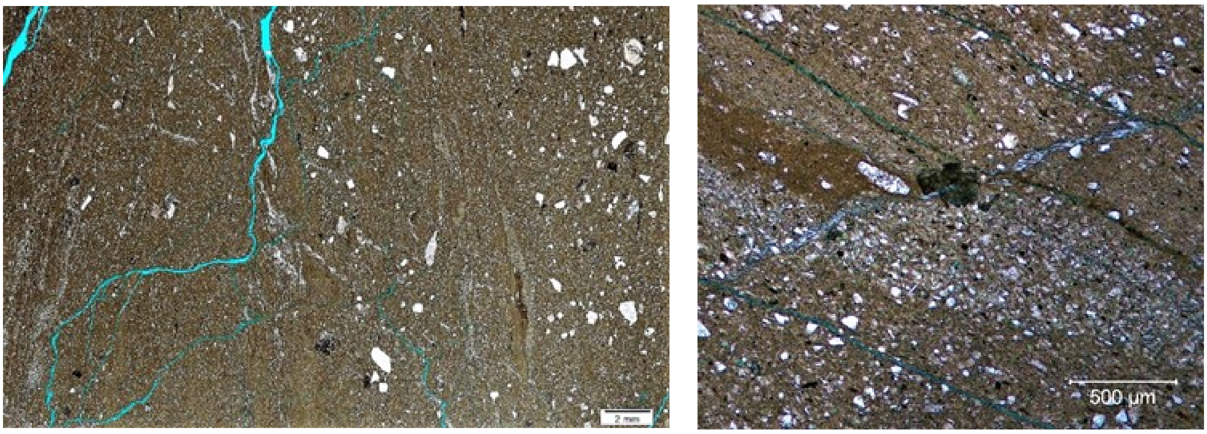


Figure 5. Thin section scan of Flysch 10.

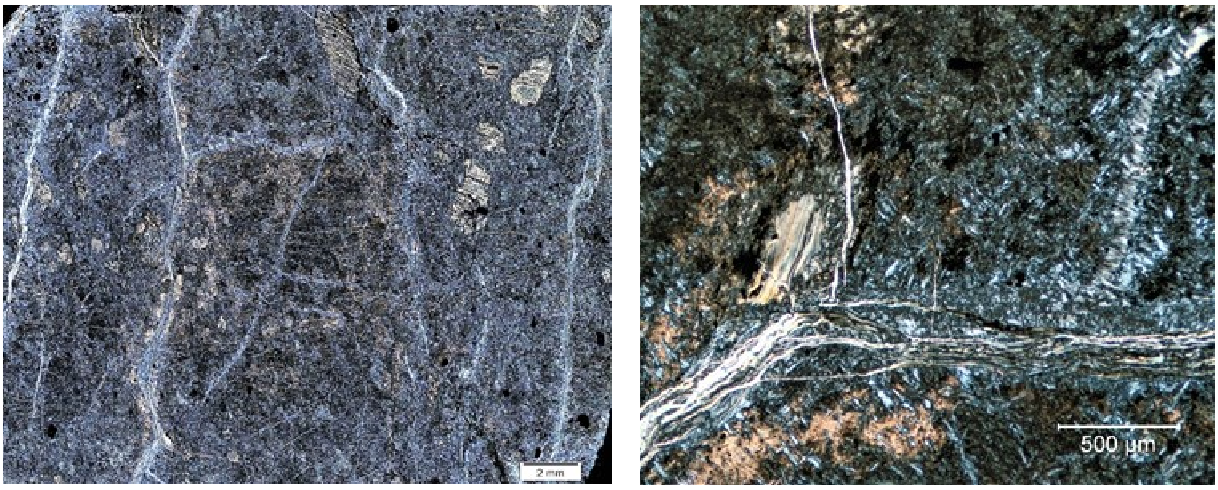
Flysch 11 is a silty mudstone with scattered fine sand grains of quartz, feldspar, and chert. Coarser calcite fossil fragments were observed and illite and silica (opal, amorphous) were possibly intergrown in the matrix. The matrix contained zones of different grain size, resulting in a laminated structure of the rock. Extensive micro-fracturing resulted in high permeability, whereas some of the fractures were filled with clay. A thin section scan of the sample in plane-polarized light is shown in Figure 6.



(**a**) (**b**)

Figure 6. Thin section scan of Flysch 11. (**a**) Overall texture. (**b**) Detailed view with laminated structures with clay-filled micro fracturs.

As serpentinites form by hydrothermal alteration of ultramafic rocks, which is associated with volume expansion, fracturing, and veining, the rocks often show a chaotic texture. This is confirmed by the analyses performed on the serpentine samples. Serp. 6 and Serp. 8 are heterogeneous serpentinite rocks with matrices consisting of a high amount of material with low crystallinity and stacking faults in the crystal structure that were challenging to account for in the XRD modeling, including various types of serpentine minerals such as lizardite and antigorite with additional brucite. The samples are characterized by mesh textures defined by fibrous serpentine minerals within and between pseudomorphs after igneous minerals, and with a network of crosscutting irregular veins and fractures. The porosity is very low; however, the thin section of Serp. 8 contains areas of empty space assumingly due to sample disintegration during preparation. The structural features and identification of mineral constituents were best viewed in cross-polarized light, where brucite displays flesh color and the serpentine displays different shades of gray (Figure 7).



(**a**) (**b**)

Figure 7. Cross polarized images of sample Serp 6. (**a**) Overview image of flesh-colored brucite occurring between fibers of serpentine. (**b**) Detailed image showing brucite matrix (flesh color) surrounding serpentine fibers.

As seen in Figure 7, the brucite tends to concentrate near fractures but is also found in the matrix. The figure shows a cross-polarized thin section scan of Serp. 6 showing overall texture (a) and detail of brucite infiltrated by serpentinite minerals near a fracture (b).

3.3. SEM Analysis Results

The samples Flysch 9 and Serp. 6 were further analyzed by SEM to enable detailed assessment of the matrix mineralogy, crack infillings, and matrix composition. A SEM-scan of Flysch 9 is shown in Figure 8.

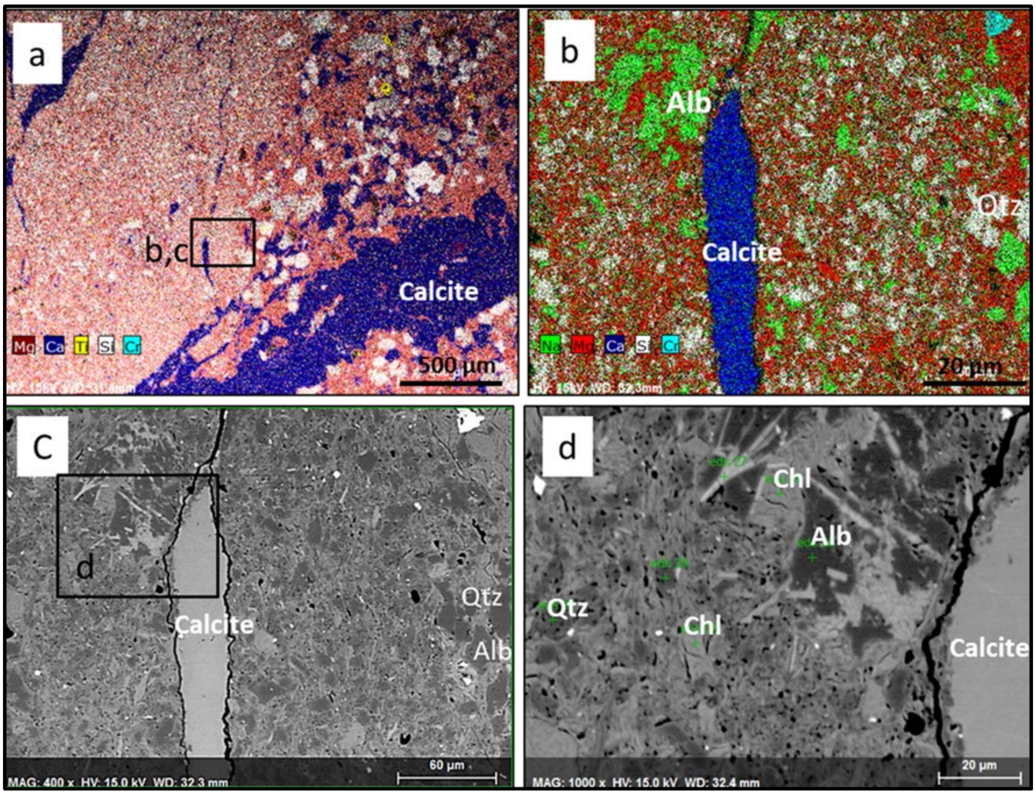


Figure 8. SEM images of the sample Flysch 9 (Alb = albite, Chl = chlorite, Qtz = quartz): (**a**) overview EDS map, showing the boundary between a more coarse-grained area and area dominated by a fine-grained matrix, (**b**) zoom EDS map on the fine-grained matrix, showing the distribution of calcite, albite, quartz, and fine-grained chlorite/corrensite, (**c**) backscatter image of same area as in (**b**), showing more details of the mineral textures, (**d**) detailed backscatter image showing the intricate intergrowth between quartz, chlorite and albite.

Figure 8a displays differences in clastic grain size and diagenetic calcite cementation as patches in matrix and fractures. Figure 8b–d represents the mudstone patches and verifies similar mineralogy as described in Section 3.2 above. The chlorite seen in matrix had a similar composition to the chlorite within the rock fragment (Figure 8d), which was also verified by EDS analysis. The images suggest that the chlorite appears to be the main clay mineral.

Figure 9 shows details on the texture and mineralogy of sample Serp. 6. The minerals and their distribution were identified by EDS chemical-point analysis in combination with BE-images (Figure 9a), and by EDS mapping of selected elements over a larger area (Figure 9b). Serpentine is the main rock mineral, and is also present as crosscutting veins (Figure 9b) which also contain magnetite. Fibrous intergrowths of serpentine and brucite can be seen adjacent to the vein.

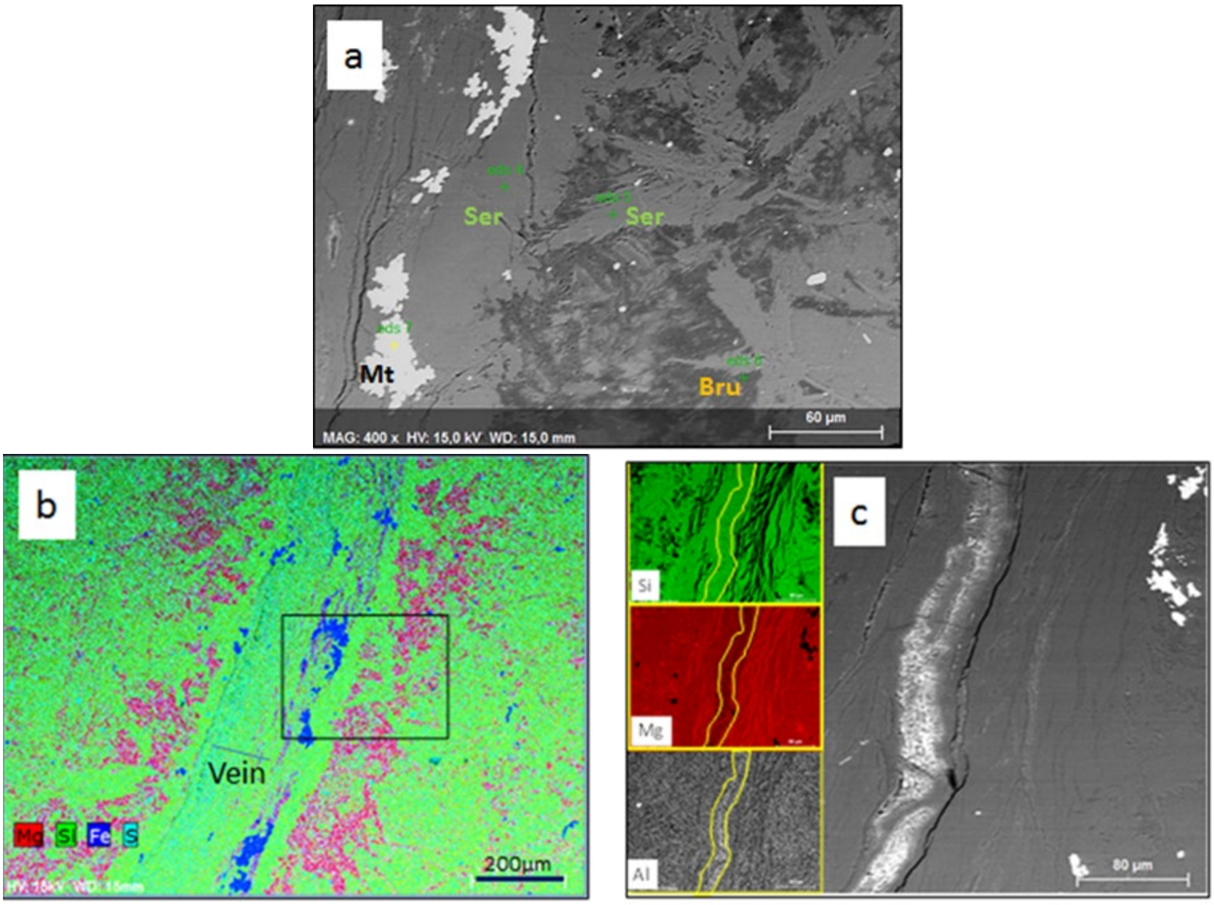


Figure 9. SEM BE-image of Serp 6: (**a**) Shows magnetite (Mt) bright, brucite (Bru) dark and serpentine (Ser) grey, (**b**) Shows EDS Mg, Si, Fe, S distribution map, the central part shows the serpentine vein and distribution of brucite (purple), (**c**) SEM BE-image of serpentine, thin zones of brucite (dark), and magnetite (bright spots), and the figure also shows microfracture along the vein; Al + Si maps (left) indicate kaolinite in the vein (Mg absent).

3.4. Swelling Test Results

Swelling pressure tests and free swelling tests were performed on the bulk material of all samples prior to the slake durability tests. The results are shown in Table 3 and were classified according to [24].

Table 3. Swelling pressure (SP) and free swelling (FS) results prior to the slake durability tests.

Sample ID	Swelling Pressure [MPa]	Classification (SP)	Free Swelling [%]	Classification (FS)
Flysch 6	0.17	Moderate	110	Moderate
Flysch 7	0.07	Low	133	Moderate
Flysch 8	0.25	Moderate	158	High
Flysch 9	0.16	Moderate	135	Moderate
Flysch 10	0.16	Moderate	147	High
Flysch 11	0.24	Moderate	120	Moderate
Serp. 5	0.12	Moderate	160	High
Serp. 6	0.31	High	142	High
Serp. 7	0.14	Moderate	149	High
Serp. 8	0.43	High	141	High

As seen in Table 3, all the flysch samples, except sample Flysch 7, showed a moderate swelling potential based on the oedometer swelling pressure tests, whereby the samples

Flysch 8 and Flysch 11 provided the highest swelling pressures. The pressures partly correspond to the obtained free swelling results, where all samples showed moderate to high volume expansion. Serpentinite samples showed high volume expansion in the free swelling tests and samples Serp. 6 and Serp. 7 also indicated high swelling pressures in the oedometer tests. The results indicated the presence of the swelling minerals in all tested samples.

After the slake durability tests, the flysch samples were again tested for swelling pressure (SP) potential and free swelling (FS) by preparing material from the retained material in the drum (>2 mm) and the slaked material passing the drum mesh (<2 mm). The aim of the experiment was to see if it is possible to detect changes in swelling potential after the rock material underwent several wetting cycles and link this to the changes in mineralogical composition.

The obtained swelling test results were then compared with the results obtained from the bulk samples. The calculated deviations from the bulk material results did not reveal a clear pattern, neither for the oedometer tests nor for the free swelling tests. The explanation may be that the swelling minerals were retained in both fractions after a slake-durability test. It was therefore not possible to detect eventual changes in swelling potential with this approach.

4. Discussion

The content of moisture-sensitive rock components may affect the durability of rocks when exposed to cyclic moisture changes during construction and operation of engineering structures, as in hydropower tunnels. It is assumed that the most moisture-sensitive rock components are clay minerals such as smectite and kaolinite [16] (Selen et al., 2019). In addition, clay-like minerals such as chlorite and mica are susceptible to weathering agents and may transform into swelling clay minerals or intermediate states of these mineral groups. Optical methods and swelling index tests can be informative procedures in assessments of moisture-sensitive constituents and their effect on slaking properties of the rock material.

4.1. The Effect of Compositional Features on the Slake Durability

As some minerals are more vulnerable to weathering agents and to mechanical abrasion as of the slake durability test, XRD analyses were performed on the flysch material after the completion of both slake durability test procedures in order to detect changes in mineralogical composition of the slaked material. The results can to some extent be used as indications on the rock components most vulnerable to slaking. Based on the XRD results of the flysch material presented in Table 1 and the observed compositional changes after both the slake durability test procedures illustrated in Figure 1, it can be implied that the clay-sized and easily weathered minerals are the first to leave the sample when exposed to moisture changes and minor mechanical abrasion in both test procedures. A similar trend was also found for the material dissolved and/or disintegrated during static wetting in the modified test (material components which leaves the sample without mechanical abrasion). This indicated that the exposure to water is the main cause of weakening and disintegration, rather than mechanical abrasion. This erosion trend was most prominent for the samples that had the highest bulk content of amorphous and clay-like minerals and the lowest durability in both slake durability tests (Flysch 8 to Flysch 11), which indicated a link between the content of these minerals and the durability of the rocks. It is noteworthy that the difference between the composition of the retained material in the drum (>2 mm) and the composition of the material passing through the drum after wetting and slaking (<2 mm) was most prominent when comparing the content of amorphous phases. As amorphous minerals are intermediate products of weathered primary minerals, correlation between the state of weathering and the resistance to slaking can be argued. On the other hand, calcite seems concentrated in the retained material in both the test procedures, indicating that this constituent contributes to cementing of the rock and an increased resistance to dissolution and slaking.

The thin section analyses enabled assessments of the textural characteristics of the samples, including the distribution of clay and carbonate in the rock. The content of clay and amorphous constituents from the XRD-analysis were re-calculated to present the percentage of the sample as a total and compared to the findings of the thin section analysis. The sample Serp. 8 was excluded from this comparison as the thin section of the sample was damaged during preparation. An overview of the main features of each sample is shown in Table 4. Access routes for water are defined here as open fractures or connected pores, including carbonate cemented fractures whereby some water flow is possible along the borders between matrix and cement. The clay-sized constituents grasp the detected clay components from the XRD-analyses including kaolinite, chlorite, weathered micas, and amorphous phases. The position of the clay-sized particles was mainly categorized as occasional (in pores or between some grains), as a main constituent of the matrix, or as infillings of microfractures. Microscale weakness zones are defined here as features which represent a heterogeneity or discontinuity in the material structure, as laminations due to differences in material composition or grain-size, and fractures (open or cemented). The estimated moisture sensitivity was based on the combination of structural features which favor the access of water to sensitive material constituents, as clay minerals and brucite.

Table 4. Overview of the main features of the tested samples.

Sample ID	Access Routes (Permeability)	Clay Sized and Amorphous Content (%)	Position of Clay Sized and Amorphous Content	Microscale Weakness Zones	Estimated Moisture Sensitivity
Flysch 6	Low	33	Few and occasional	Low	Low
Flysch 7	Low	41	Few and occasional	Low	Low
Flysch 8	Medium	56	Mainly in matrix	Moderate high	Moderate
Flysch 9	High	64	Mainly in matrix	Very high	Very high
Flysch 10	Medium high	69	Matrix and microfractures	High	High
Flysch 11	Very high	74	Matrix and microfractures	Very high	Extreme high
Serp. 6	Low	77	Matrix and microfractures	Moderate	Moderate

For the flysch samples, mainly Flysch 8 to Flysch11, the amorphous content was identified as clay/silt-sized constituents of the matrix, which most likely is the product of extensive weathering. Based on the thin section microscopy, it was possible to identify amorphous silica in the matrix. A chlorite-rich clay matrix was observed in the thin section and SEM analyses, but it was not possible to identify subsequent components of smectite based on the methods used in this study. The combination of microfractures and weathering products as clay-like minerals may contribute to lowering the resistance of the rock to moisture changes. The presence of extent of clay minerals, microfractures, and compositional heterogeneity will define resistivity to moisture change for weak rocks. The presence of calcite in veins, pores, and matrices, on the other hand, positively contribute to cement the rock material.

The serpentinite samples show a chaotic structure with a composition dominant on altered and amorphous constituents. The amorphous constituents are assumed to result from weathering of serpentine minerals as lizardite, chrysotile, and particularly of brucite, which is more susceptible to weathering. The brucite in sample Serp. 6 is most concentrated along the serpentine veins, assumingly formed in association with hydrothermal fluids in veins parallel to fractures. Based on optical microscopy and SEM-analysis (Figures 8 and 9), sample Serp. 6 contained a notably high amount of brucite compared with the XRD results. Herewith, XRD seems to underestimate the amount of brucite and further has a problem in fitting the observed diffraction pattern due to structural complexities of the serpentine and brucite minerals. This causes an underestimation of the brucite content which appears to be at least by 18–20 percent in comparison with the optical microscopy and SEM observations. Hydro-magnesite as a common brown-colored weathering product of serpentinite in association with amorphous iron oxide is described in [25]. However, hydro-magnesite was not identified in the XRD-analysis of the samples herein, but brown coloring was noted in the thin section analysis.

4.2. Link between Amorphous Constituents, Swelling Potential and Durability

All samples showed a swelling potential when pulverized and exposed to water in the oedometer tests and free swelling tests (Table 3), which indicated the presence of swelling clay minerals in both the flysch and serpentinite samples. On the other hand, the bulk composition analyses on the samples Flysch 9 and Flysch 11 did not reveal any swelling clay minerals according to the XRD results, but both the samples swelled in swelling tests. The serpentinite samples show high swelling potential based on both swelling tests despite that swelling clay minerals were not detected by XRD, with exception of sample Serp. 5. This may be explained by the amorphous clay-sized material which may hold swelling potential even when no smectites are detected by XRD. Hence, it is reasonable to assume that the amorphous clay-sized mineral content acts as an important indicator on the weathering degree and swelling potential. The weathering of feldspar is found to result in various combinations of non-crystalline intermediates, including different subgroups of swelling clays, which may fall into the category of amorphous phases in an XRD-analysis. As concluded by [21,26–28], amorphous clay-sized constituents may hold swelling potential even for immature transformation processes from the mother mineral into smectites. This may be an explanation of the high content of clay matrix seen in some of the thin section analysis of the flysch samples. Lizardite can also be transformed into smectites during hydrothermal alteration processes, which may result in immature and amorphic swelling components in serpentinites. This means that high swelling potential may exist in the case of samples containing amorphous constituents, even if the XRD analysis is unable to detect any clay structures. Moreover, the hydration of chrysotile can result in expansion of rock material artificially dried at high temperatures in the lab, which may explain the high swelling results of the pulverized serpentinite samples.

The swelling test results on bulk material prior to the slake tests were compared to the content of amorphous constituents in order to detect a link between weathering degree and swelling of the samples. By including the slake durability results from the previous study [16], it was possible to analyze correlations between the content of amorphous minerals, detected swelling clays, measured swelling potential, and slake durability of the different samples. The detection of clays was either obtained by XRD, thin section (TS), or SEM analysis. Additionally, the estimated moisture sensitivity from the structural analyses (Table 5) can explain the differences between the slake durability indices obtained by the two slake durability test methods performed, i.e., initially dry samples (SDI4) and initially wet samples (MSDI4).

Table 5. Summary of amorphous content, swelling potential, and slake durability of the samples.

Sample ID	Amorphous Content (%)	Swelling Clay Detected	Swelling Pressure (MPa)	Free Swelling (%)	SDI4	MSDI4	Estimated Moisture Sensitivity
Flysch 6	23	Yes (XRD)	0.17	110	97.6	97.8	Low
Flysch 7	28	Yes (XRD)	0.07	133	93.4	91.9	Low
Flysch 8	37	Yes (XRD)	0.25	158	70.8	73.4	Moderate
Flysch 9	40	Yes (TS/SEM)	0.16	135	48.9	43.9	Very high
Flysch 10	43	Yes (XRD)	0.16	147	64.9	51.2	High
Flysch 11	48	Yes (TS)	0.24	120	43.3	31.8	Extreme high
Serp. 5	57	Yes (XRD)	0.12	160	93.4	96.9	Moderate
Serp. 6	58	No	0.31	142	91.8	92.2	Low-moderate
Serp. 7	56	No	0.14	149	92.9	90.1	Low-moderate

Serp. 8	58	No	0.43	141	89.2	92.9	Low-moderate

No clear correlation was found between the measured swelling potential (oedometer tests and free swelling tests) and the slake durability indices of the material. The flysch samples showed weaker durability with increased swelling while the serpentinite samples swelled highly but also had relatively high durability. This may be due to the fact that the overall compositional features of the rock samples were destroyed during preparation (pulverizing) of the samples prior to the swelling tests, changing samples to a different state compared with the intact state prior to slake durability tests. Durable constituents of the rock and eventual cementing of microfractures and pores for which the intact rock will increase the strength and durability will therefore not be properly reflected in the swelling test results. The correlation found for the flysch samples may be occasional or explained by a primary weakening of the rock material due to weathering where swelling clay minerals are present as weathering products, i.e., the influence of swelling minerals on rock durability is dependent on the overall compositional features of the rock material. The swelling potential of the serpentinite did not seem to affect the durability of this rock type, as test results showed.

While comparing the mineral composition of the samples with the measured durability, a correlation was identified for the content of amorphous constituents and the slake durability indices for the flysch samples. An increase in weathering degree with clay-like products corresponded well to a decreasing durability. This resembles well the estimated moisture-sensitivity of the flysch samples (Tables 4 and 5), where increased permeability and the composition of the matrix resulted in decreased resistance to repeated cycles of wetting and drying. In contrast, this correlation did not yield for the serpentinite samples, which may be explained by their textural composition favoring an increased structural strength of the rock material. In addition, it should be kept in mind that the rock was already disintegrated into lumps at arrival, indicating that major disintegration took place prior to the slake durability tests performed. It is therefore reasonable that the lumps tested are more durable than the original material. The comparison of the content of amorphous minerals and the slake durability indices is illustrated in Figure 10.

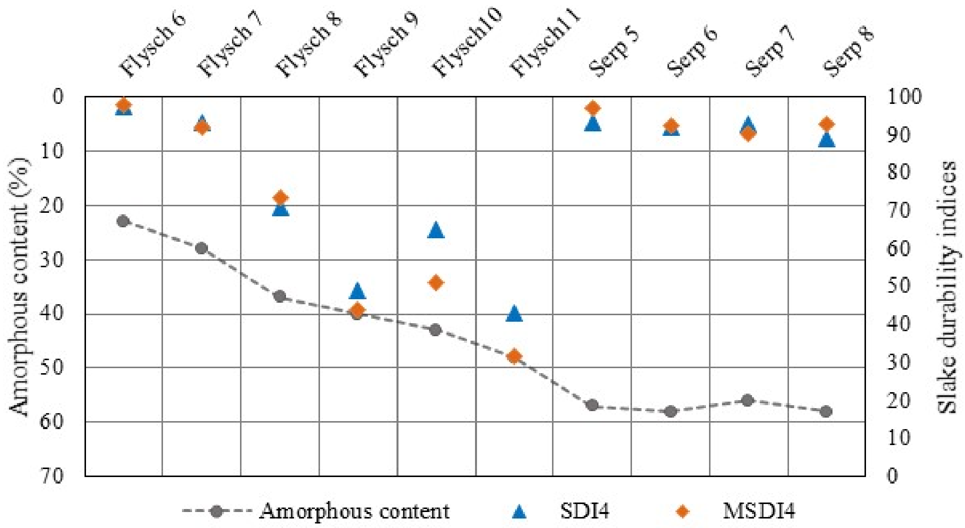


Figure 10. Correlation between the content of amorphous minerals and slake-durability indices.

4.3. Appropriateness of the Applied Methods

The free swelling test and oedometer swelling pressure test were performed in order to assess the swelling potential of the tested rock types. The tests are standardized index tests that aim to classify the swelling magnitude based on volume expansion characteristics (free swelling tests) or maximum pressure induced (oedometer test under the condition of zero deformation) of pulverized rock samples. The relatively poor correlation between oedometer and free swelling test results indicated that the evaluation of swelling potential should not rely entirely on these methods, but instead be supplemented with detailed mineralogical assessment, as was demonstrated in this manuscript. It is noted here that the structural features of the samples were destroyed during the pulverizing process and that the index tests on pulverized rocks may not fully correspond to the intact rock behavior assessments. Further, the swelling results of the slaked material of the flysch samples showed no clear pattern on the changes in swelling potential prior to and after the slake durability tests. Still, the tests are useful as first-hand estimations on the swelling potential and as indicators on the presence of swelling minerals in the bulk rock material.

The differential slaking behavior of the tested rocks when first wetted and then exposed to repeated moisture changed compared with the conventional slake durability test, certainly dependent on the availability of access routes for the water to penetrate the material and the nature of the fractures including crack infillings. The water weakening effect due to saturation has major control over the durability of the rocks, and the modified slake durability test revealed this inherent vulnerability of saturation prior to the mechanical abrasion exerted to the samples during the conventional slake durability test. As the structural features of rock materials may act as controlling factors on the resistance to weathering and disintegration behavior, it is important for future investigations that thin section analysis is carried out in order to gain information on porosity, micro fracturing, grain-size distribution, clay content, and signs of weathering. In addition, samples may be further analyzed with SEM to assess the clay mineralogy, amorphous constituents, crack infillings, and matrix composition and to evaluate the moisture sensitivity.

The importance of optical analyses was considerable in the assessments of the serpentinite samples. The texture of the samples was heterogeneous and dominated by altered minerals not recognizable by XRD analysis. The inherent properties of these samples were therefore unclear prior to this study. As was demonstrated by this research, SEM and optical microscopy successfully documented considerable amounts of undetected brucite in the serpentinite samples, which is a weak and vulnerable mineral to dissolution and critical for tunnels supported with shorcrete lining. The research also demonstrated that it is difficult to use standard mineral structure models to fit the XRD patterns for serpentinite samples. This may have been caused by the complexity and variability of the serpentine minerals and perhaps also as a result of amorphization of the sample during crushing (preparation). Even if the slake durability of the tested serpentinite samples were relatively high, the rock type holds the potential to cause problems for tunnels conveying water.

5. Conclusions

The textural features of samples with similar rock origin have shown to be an important material parameter causing differential slake durability and moisture sensitivity. By studying the thin section scans, a microscale heterogeneity in the tested rock samples was revealed and which was not detectable by visual inspection of the samples. The differential presence of microstructures among similar rocks was found to control the slake durability of the tested rock samples as their degradation potential certainly depends on the mineral distribution, porosity, carbonate cementing, and weathering degree of the material components. The crack infillings may either function as accelerators on rock degradation, as fractures filled with clays, or increase the rock durability by carbonate cementing. However, carbonate cementing was found to be less important if the rock composition is dominated by clay minerals, which is in accordance with the findings of [19]. Further, the compositional heterogeneity and presence of microfractures were shown to be important material parameters when comparing the moisture sensitivity and durability of similar rock types, which is in accordance with the study of [18]. As demonstrated, these features are best determined by optical methods where the material structure, fractures, and position of weaker rock components have been evaluated. By use of different image analysis tools, structural and textural properties which differentiate between samples that are otherwise similar can be detected. Optical methods, in particular the thin-section analysis, are therefore recommended in future assessments of rock durability.

Due to the high content of amorphous phases, the error estimate of the values was uncertain and varying between the tested samples, and fitting of the XRD patterns was difficult. In general, when the material consisted of large amounts of weathered constituents, amorphous mineral phases and/or swelling minerals, the results obtained by XRD seemed very approximate and were uncertain. Further, the method does not serve to provide reliable estimations on the content of brucite, which is a mineral that reacts with water and may corrode shotcrete lining applied in underground caverns and tunnels. The method as it stands today is therefore not appropriate for quantifying weak, swelling, and weathered rock materials in engineering projects where these particular material properties are central for durability evaluations. However, the method is useful as a complement to other mineralogical analyses and in confirming findings of optical investigations.

The performed swelling tests provide a fair indication on the presence of swelling minerals when the content of amorphous phases are high, as the amorphous content may include swelling constituents masked in the XRD-diffractogram. Some samples with no detected smectites but high contents of amorphous minerals showed abundant swelling, and the swelling results therefore confirmed the findings of [21]. However, it is realized that the attempt to detect changes in swelling potential based on the performed swelling index tests was not successive as performed in this study. Further, the amount and distribution of potential swelling mineral components in the intact rock structures are assumingly more important than the specific concentration of these minerals. Hence, in order to evaluate the effect swelling has on intact rock durability, swelling tests on intact rock samples are strongly recommended. It is also underlined here that for swelling behavior determinations to be incorporated in rock mass deformation analyses, comprehensive testing programs designed for intact rock swelling should be conducted.

As was demonstrated, the use of SEM backscattered electron image analysis with EDS helps to identify minerals by their main chemical signatures. It is, however, noted here that identifying clay minerals is commonly complicated due to their small size, variable compositions, and different types of mixed-layer minerals. SEM secondary electron images (SEI) is a recommended method as it provides details on the complex and delicate morphology of clay minerals. In combination with EDS and XRD fine fraction analysis, the SEM–SEI method will be helpful in identifying fine particles, and possibly also amorphous weathering textures. For future research, spectroscopic methods should be used to quantify the content of brucite. In hyper-spectral techniques, one can differentiate different types of hydroxyl bearing minerals based on the position and the OH-absorption peak in the infrared spectrum. As the content of brucite may have a severe impact on the durability of rocks and the long-term stability of underground constructions supported with sprayed concrete, revealing the presence of this mineral is worth the effort and thus is strongly recommended.

The authors strongly suggest further research on the compositional control on the slake durability of weak rocks, with emphasis on mineral distribution, compositional heterogeneity, and the nature of microfractures of different rock types. In order to evaluate the correlation between the presence of clay-like minerals, swelling potential, and the durability of clay-bearing rocks, thorough assessments on weathered and altered constituents should be applied on intact rock structures whereby the total of compositional features of the rock is evaluated. In engineering structures that are exposed to water and moisture changes, the modified slake durability test in combination with assessments on compositional features is recommended to be carried out.

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