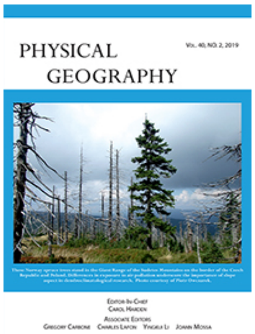

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ARTICLE

Laboratory study of the effect of temperature difference on the disintegration of redbed softrock

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ABSTRACT

To explore the impact of temperature difference (TD) on the disintegration of redbed softrock, three types of redbed rock, collected from Nanxiong Basin, were analyzed under three different treatments: TD, wetting and drying (WD), and TDWD-temperature difference and WD. To better understand the influence of different ranges of TD on disintegration during WD cycles, pH (hydrogen ion concentration) values, electrical conductivity (EC) values, and concentration of cations in leachate released during treatment were measured. The results show that no significant change can be observed under single TD treatment but that TD can increase the disintegration rate by accelerating the water–rock interaction. The effect of TD is more significant for rock with weak resistance to disintegration.

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KEYWORDS
Rock disintegration; temperature difference (TD); moisture–temperature interaction; redbed softrock; wetting and drying (WD)

Introduction

Redbeds, sedimentary rocks typically consisting of conglomerate, sandy conglomerate, sandstone, siltstone, shale, and mudstone, are predominantly red in color due to the presence of ferric oxides. Redbed softrock refers to siltstone, shale, or mudstone with low mechanical strength.

Redbed softrock covers a large area in China and is one of the common strata encountered in surface and underground engineering. Due to its specific lithological characteristics, the disintegration of redbed softrock is closely related to geological disasters and deformations, for example, rockfall (Yan et al., 2016), landslides, and debris flows (Chen, Wang, & Li, 2008; He et al., 2013; Wang, Wang, Wang, & Ji, 2006); therefore, the disintegration problem of redbed softrock has drawn the attention of a number of researchers in both engineering and geomorphology. Also, disintegration of soft intercalated rock layers causes collapse of cliffs, as typified by the Danxia landform (Peng, Qiu, & Pan, 2014).

Engineering studies that have addressed the topic of the disintegration of softrock have been based on the process of water–rock interaction. The main research foci have been on the impact of water–rock interaction on mechanical properties (Deng et al., 2016; Zhang, Zhang, Liu, & Zhang, 2015; Zhang, Li, & Chen, 2008; Zhou, Tan, et al., 2005; Zhu, Xing, Wang, & Xu, 2004); mineral composition, and microstructure (Liu &

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Lu, 2000; Yang, Wang, Li, Li, & Dai, 2014). Other researchers have focused on quantitative research on softrock disintegration characteristics, for example, the softening coefficient (Wang et al., 2009), fractional dimension number (Su, Zhao, & Liu, 2005), coefficient of disintegration-resistance (Gamble, 1971; Wang, Deng, Deng, & Zhu, 2017), and the quantitative relationship between different moisture contents and the amount of disintegration (Zhang et al., 2016), as well as the surface hardness of rocks (Sumner & Nel, 2002), the intensity of disintegration in different solutions (Liang, Tan, Jiang, & Jiao, 2015), and even the effect of moisture application type on the weathering effect (Sumner & Loubser, 2008) and power-dissipation characteristics during disintegration (Ming & Jinwu, 2015). Of these themes, the impact of water–rock interaction on mechanical properties has been most studied. For the mechanisms of softening and disintegration, it has been universally assumed that the processes of clay mineral swelling, ion-exchange absorption, dissolution of soluble minerals and new formation of minerals, and micro-mechanism of water–rock interactions all combine to cause rock to disintegrate (Lv, 2013; Wang, Cao, & Chen, 2016; Wu, Liu, & Wang, 2010; Zhou, Tan, Deng, Zhang, & Wang, 2005).

Beyond the effect of moisture conditions on rock decay, temperature difference (TD) is considered to be an important factor (Weiss, Siegesmund, Kirchner, & Sippel, 2004), and many studies have been conducted on the impact of thermal differences on rock decay (Gómez-Heras, Smith, & Fort, 2006; Sousa, Río, Calleja, Argandoña, & Rey, 2005; Yatsu, 1988). Due to the subtle impact of short-term heating-cooling cycles on the physical disintegration of rock in both natural and built environments (Yamaguchi, Yoshida, Kuroshima, & Fukuda, 1988; Zhou, Deng, et al., 2005), the impact of temperature on rock decay has not been studied as much as the influence of moisture.

In recent years, increasing attention has been given to the impact of temperature on rock decay, specifically, on physical-mechanical properties (Saiang & Miskovsky, 2011; Wang, Xu, Liu, & Wang, 2016), salt weathering (Aly, Gomez-Heras, Hamed, Burgo, & Soliman, 2015), identification of aspect-related differences in thermal and moisture characteristics (McAllister, Warke, & Mccabe, 2016), and on quantitative analysis of the relationship between temperature and rates of stone decay. Among them, few studies have focused on the impact of TD on redbed softrock decay. The macrostructure deformation of a rock body resulting from the incoordination of internal stresses caused by TDs as well as the impact of TDs on the process of salt weathering (Aly et al., 2015; Zhang, Chen, Wang, & Liu, 2015), to some extent reflects the influence of TD on rock decay.

On the whole, the effects of TD on rock decay can be divided into direct and indirect effects. Due to the property of rock to expand when heated and contract when cooled, the direct effect of TD relates to the efficiency of thermal stress. Many studies (e.g. Wang, Huang, & Huang, 1997; Zhu et al., 2006) have shown that temperature oscillations cause a thermal stress of 0.35–6.33 MPa at depths of 6.1–7.6 m and that this stress can lead to mechanical disintegration at the surface. However, without a moisture effect, a TD can hardly cause disintegration alone (Yamaguchi et al., 1988; Zhang, Chen, & Liu, 2012a). Griggs (1936) found no change in a coarse-grained granite during an experiment in which the rock was subjected 89,400 times to TDs of 110°C. The indirect effect of TDs is that the combination of heating and wetting can significantly deteriorate stones (Hale, 2003; Zhang, Chen, & Liu, 2012b). Temperature changes can change the water content in rock. In particular, a change of water phase will form multiphase-damaged media and cause expansion and contraction (Geng-She, Yi-Bin, & Wei, 2002). Additionally, freeze-thaw action (Hall, 2007; Hall & André, 2001) and dissolution of soluble material can also cause disintegration (Goudie, 1989).

Although some researchers (Zhang et al., 2012b, 2015) have quantitatively analyzed the relationship between TDs and mudstone decay, their research did not analyze the impact of TDs on the dissolution of minerals. The aim of this paper is to analyze the impact of TDs on disintegration during wetting and drying (WD) treatment, and to understand how TDs influence physico-chemical characteristics of the clay-rich sediment.

Materials and methods

Experimental materials

Nanxiong Basin (24°33′–25°24′N, 113°52′–114°45′E) is a redbed basin located in the north of Guangdong Province. A subtropical monsoon climate prevails, with long hot summers and short winters. Based on meteorological observations collected from Nanxiong Station (1956–2010), the mean annual temperature of Nanxiong Basin is 19.6°C and mean annual precipitation is 1555.1 mm (Yan et al., 2017). Three types of purple mudstones were sampled, from the Nongshan Formation (En) in Dahangkeng Village, the Shanghu Formation (Esh) in Huangtian Village, and the Zhutian formation (Kzt) in Jiangtian Village, and respectively named D, H, and J (Figure 1). All samples used in this study were extracted from 2 m depth.

Methodology

Measurements of mineral composition

X-ray diffraction (XRD) analysis of mineral composition was commissioned by SGS Unconventional Petroleum Technical Testing (Beijing) Co., Ltd by Rigku Smartlab9 (D/MAX2200 type). The method refers to the SY/T5163-2010 analysis method for clay minerals and ordinary non-clay minerals in sedimentary rocks by XRD. X-ray fluorescence (XRF) analysis of elemental composition was conducted by X-ray fluorescence spectrometer (ZSX Primus). Thin-section identification method was used to determine the lithology of samples.

Procedures and treatments of experiment

The experiment was designed to address three factors that influence rock disintegration: moisture, temperature, and moisture–temperature interaction. The influence of moisture was conceptualized as rock saturation. The influence of temperature was conceptualized as fluctuations in temperature that were characteristic of Nanxiong County. The last factor, moisture–temperature interaction, refers to different combinations of moisture and temperature acting on rock at the same time. Prior to the beginning of the experiment, the mineralogical and chemical compositions of samples from three sites in Nanxiong Basin were determined by XRD and XRF.

Overall, 27 samples were subjected to three treatments (Figure 2). To ensure homogeneity, the samples for each treatment were selected from the same rock and cut into

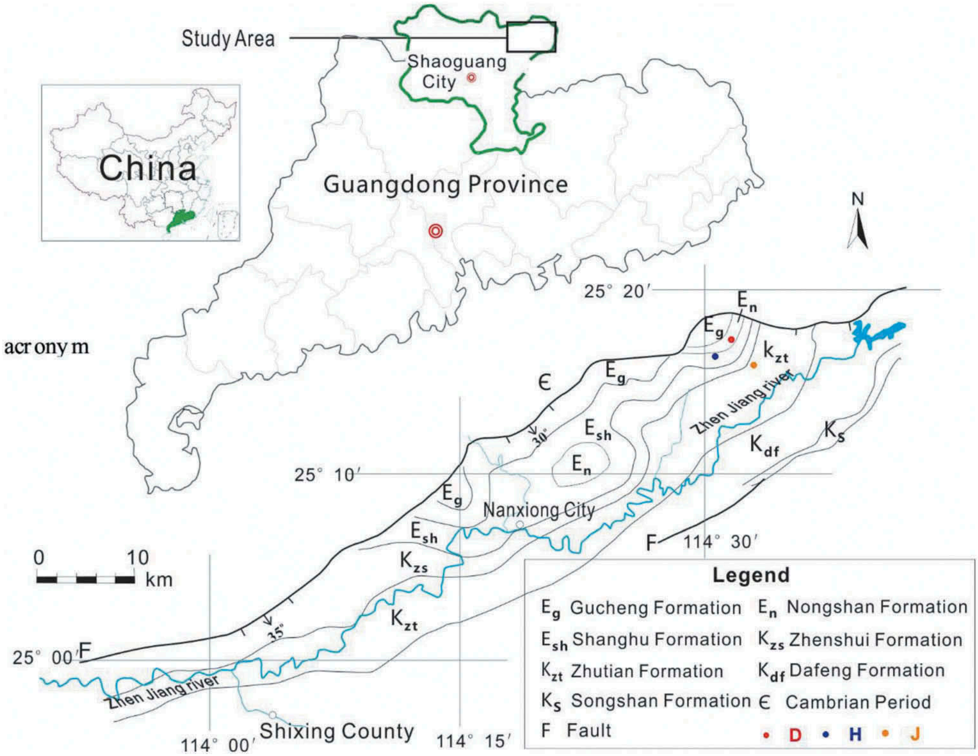

Figure 1. Map of Nanxiong Basin in Guangdong Province and the sampling sites.

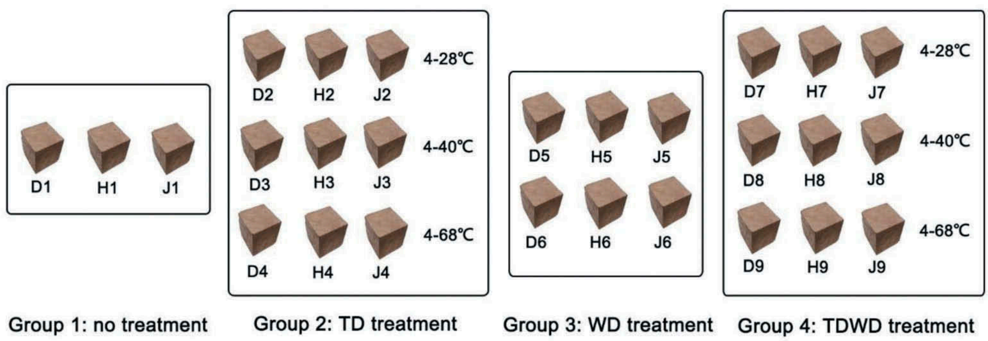

Figure 2. Graphic showing the design of the disintegration experiment.

similarly sized cubes (50 ×50 × 50 mm) with an electric saw. Before treatment, all samples were subjected to the following treatments:

(1) Temperature difference (TD): Air-dried samples were placed in a refrigerator at 4°C for 24 h and then heated in an oven at 28, 40, or 68°C for 24 h, respectively.
(2) Wetting-drying treatment (WD): Air-dried samples were immersed in a 1000-ml beaker for 24 h, and then dried in a desiccator with phosphorus pentaoxide. The amount of water absorbed by the sample was recorded.

(3) TD and wetting-drying treatment (TDWD): The immersed samples were placed in a refrigerator at 4°C for 24 h and then heated in an oven at 28°C (referring to the average temperature in summer), 40°C (extreme highest air temperature), or 68°C (surface extreme temperature) oven for 24 h, respectively. When heating, each wet samples was put in a sealed dry container with phosphorus pentaoxide.

Five cycles of each treatment were completed, and photographs were taken after each cycle.

Analysis of microscopic morphologic characteristics of fragments and determination of leachate collected after treatment

The surface microscopic morphologic characteristics of specimens were examined by scanning electron microscopy (SEM). In addition, in consideration of temperature as a major control on rock breakdown through its effects on chemical weathering processes (Warke & Smith, 1998), electrical conductivity (EC), pH, and concentration of cations (Na^{+}, Ca^{2+}, Mg^{2+}, Mn^{2+}, Fe^{2+}, Fe^{3+}, Cu^{2+}, Al^{3+}, K^{+}, S^{2-}, Si^{4+}) using inductively coupled plasma mass spectroscopy (ICP-MS) were determined in the filtrates collected after each round of treatment.

The Mann–Whitney *U* test (MWU test) is a non-parametric statistical hypothesis test for assessing whether one of two samples of independent observations tends to have larger values than the other. In this research, the MWU test was used to determine the statistical significance of pH, EC, and detected elements under different temperature treatments.

Results

Composition of sample minerals

The mineral composition and physiochemical features of unweathered sediments are shown in Tables 1 to 3. The three analyzed unweathered samples mainly differ in their clay content.

The influence of TDWD on the rock decay

Results from the weathering experiment showed that the disintegration of dry rock free from any other treatment and caused by TDs can be described as imperceptible. The WD rock disintegrated rapidly; the disintegration rate caused by the TDWD treatment was the highest of all treatments, likely because the rock had undergone both WD cycles and TD cycles (Figure 3). Figure 3 shows that the larger the TD, the smaller the fragments in size for rock treated with TDWD. Results from this study showed that

Table 1. Lithology and main mineral composition of the redbed parent rock from the three sites.

Site	Lithology	Quartz (%)	Calcite (%)	Hematite (%)	Clay (%)	Clay		
						Illite(%)	Kaolinite(%)	Chlorite(%)
D	Silt-bearing Mudstone	45.8	14.8	3.5	28.7	33	57	10
H	Silty mudstone	33.9	19.4	3.7	36.8	23	65	2
J	Siltstone	26.4	14.3	2.4	42.4	31	53	16

Rock at site D is silt-bearing mudstone (Nongshan Formation), rock at site H is silty mudstone (Shanghu Formation), and rock at site J is siltstone (Zhutian formation).

Table 2. The main physiochemical features of redbed parent rocks (%) from the three sites.

Site	SiO_2	Al_2O_3	Na_2O	MgO	K_2O	CaO	MnO	Fe_2O_3	TiO_2	P_2O_5	LOI
D	62.58	12.45	0.91	1.98	3.03	5.39	0.01	4.76	0.68	0.12	7.88
H	55.46	14.13	0.87	2.17	2.99	8.10	0.15	5.20	0.63	0.15	10.26
J	55.26	13.40	0.88	2.70	3.70	7.92	0.13	4.93	0.63	0.23	10.08

Rock at site D is silt-bearing mudstone (Nongshan Formation), rock at site H is silty mudstone (Shanghu Formation), and rock at site J is siltstone (Zhutian formation). LOI is loss-on-ignition.

Table 3. A brief summary of thin-section identification.

Site	Lithological features seen under the microscope
D	Sandy texture, support type: base support, cementation type: contact cementation, content of cement: 15% and is mainly mud, weakly consolidated, little calcium in concretion forms.
H	Anisometric sandy texture, mainly mud and ferruginous cementation, secondarily clay calcite, cementation type: basal cementation, content of cementation: 10%.
J	Silty texture, 30% cement content, and mainly mud and ferruginous cement with little calcium cement; cementation type: porous.

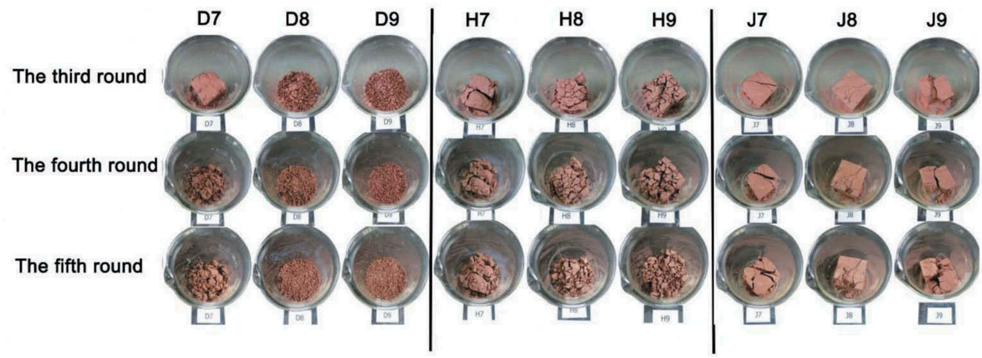

Figure 3. The disintegration of rocks after each cycle of TDWD treatment with different temperature differences. Samples D9, H9, and J9 were subjected to the greatest temperature difference (4°C for 24 hr to 68°C for 24 hr).

TD alone cannot cause disintegration; however, when TD and WD work together, TD can cause the disintegration rate to increase compared with the disintegration rate of rock only through WD. Additionally, during TDWD, the impact of TD on disintegration is more obvious for D and H rock than for J rock.

Also, results from the experiment showed that J rock has higher disintegration resistance than D and H rocks.

The influence of TDWD on the microstructure of redbed softrock

According to the visual interpretation of images from SEM, the following observations can be pointed out: surfaces of fragments free from treatment are much more compact than those after treatment. Without treatment, mineral layers are strongly cemented and porosity is lower. However, after TDWD with a TD of 4–28°C, surfaces of rock fragments are covered with mineral particles, and small holes have appeared. After TDWD with a TD of 4–68°C, the surface is more broken, a layered structure is not visible, the number of holes has increased, and a loose structure has formed (Figure 4).

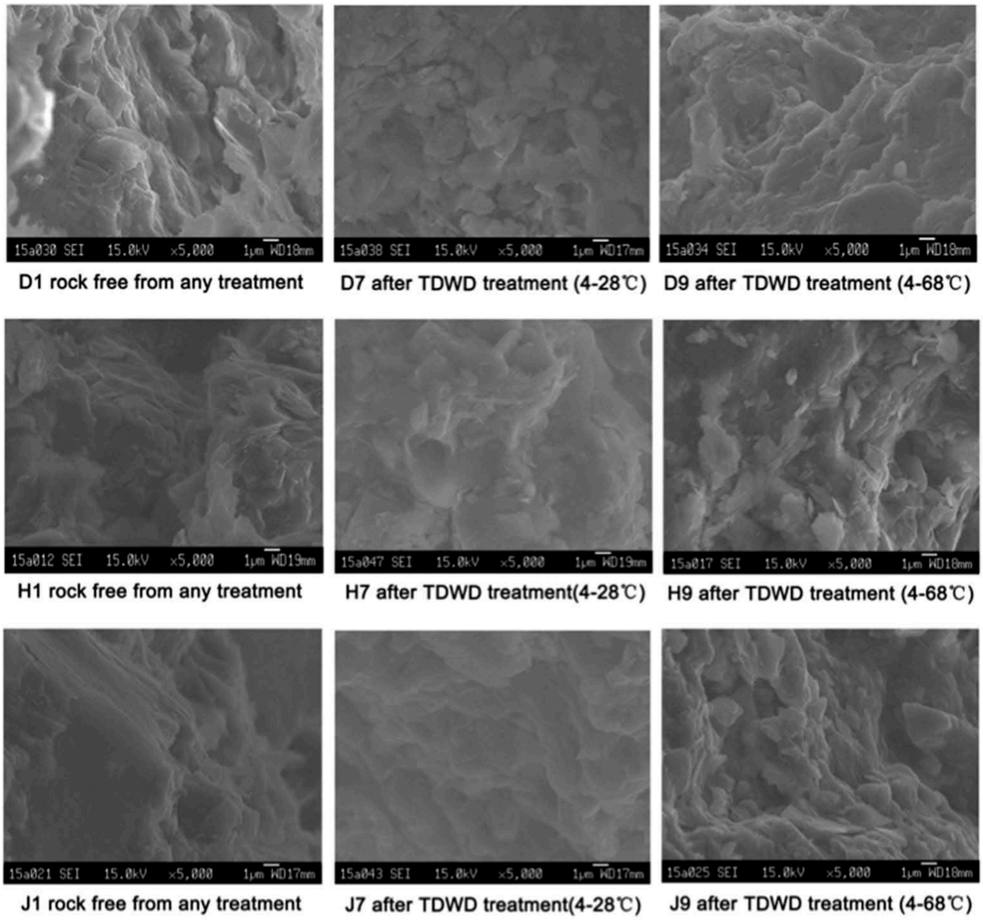

Figure 4. SEM images of softrock specimens free from and after different treatments. The surfaces of D9, H9, and J9 are more broken and contain more holes than those of D1, H1 and J1. The surface of J1 is smoother and flatter than those of D1 and H1.

By comparing images of D1, H1, and J1 (Figure 4), we find that the surface of J1 is more compact and smooth than the surfaces of D1 and H1. Surfaces of D1 and H1 are uneven and not compact or dense in arrangement between layers, which makes it easier for water to permeate into the inside of the rock. The SEM results can partly explain why D rock shows stronger disintegration than J rock. Therefore, we deduced that the surface features might be one of the reasons affecting disintegration.

The influence of TDWD on the pH value in filtrate

The pH value is the composite reflection of a rock's chemical properties. Its changing values illustrate the complexity of water–rock interactions. As shown in Figure 4, TDWD caused significant change in pH values of the filtrate, which showed slightly alkalinity, with pH value ranging between 7 and ~8.1. The general trend is that pH value increases at the beginning and decreases during the TDWD treatment (Figure 5). However, no significant influence caused

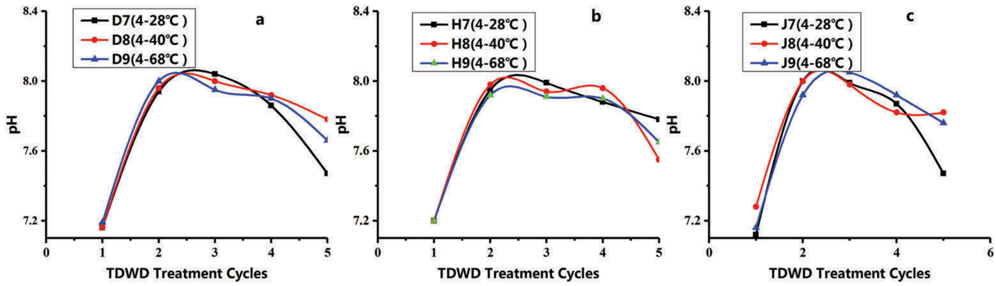

Figure 5. The changing trend of pH values in three types of purple mudstone. Rock D is silt-bearing mudstone (Nongshan Formation), rock H is silty mudstone (Shanghu Formation), and rock J is siltstone (Zhutian formation).

by different TD on pH value was found, according to the MWU test ($\alpha > 0.1$). The same changing regularity was found by Zhou CY (Zhou, Deng, Tan, Lin, & Wen, 2004).

The influence of TDWD on the EC value in filtrate

EC values of leachates collected after each cycle of treatment were measured. All EC values were between 55 and 90 μS/cm. As the number of cycles of TDWD increased, the cumulative EC value increased significantly (Figure 6). Results show that EC values for D and H rocks treated with TDWD with a TD 4–28°C are lower than EC values for samples experiencing TDs of 4–40°C or 4–68°C (MWU test: D rock, $\alpha = 0.094$ (<0.1), H rock, $\alpha = 0.117$ (>0.1), J rock, $\alpha = 0.917$ (>0.1)). This means that TD exercised a more significant influence on D rock than on H and J rocks. A possible reason is that TD would have more influence on rocks with low disintegration resistance than on rocks with higher disintegration resistance.

The influence of TDWD on the concentration of cations in filtrate

Analyses of solutions collected from each cycle of treatment shows that a total of 10 cations (Na^+, Ca^{2+}, Mg^{2+}, Mn^{2+}, $Fe^{2+(3+)}$, Cu^{2+}, Al^{3+}, K^+, S^{2-}, Si^{4+}) were detected in

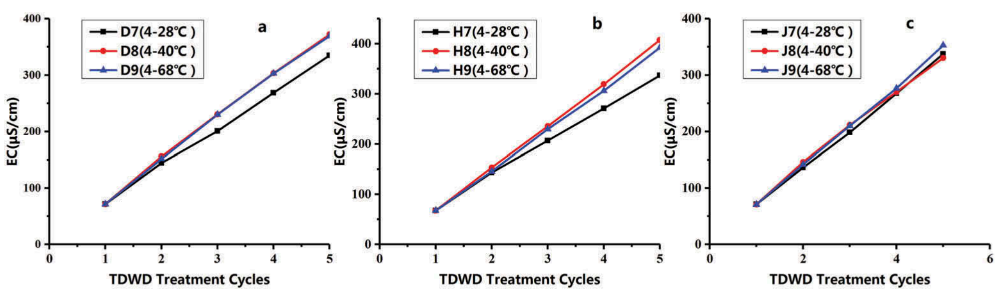

Figure 6. Trends of cumulative EC values for D, H, and J rocks in cycles of TDWD treatment. Rock D is silt-bearing mudstone (Nongshan Formation), rock H is silty mudstone (Shanghu Formation), and rock J is siltstone (Zhutian formation).

leachate. Among them, concentrations of Na^+, Ca^{2+}, Mg^{2+}, Si^{4+}, K^+ were significantly higher than those of the other detected cations.

Figure 7 shows that the cumulative concentration of Ca^{2+} after treatment with TDs of 4–40°C and 4–68°C is higher than with the 4–28°C treatment (MWU test on concentrations of Ca^{2+}: J rock, $\alpha = 0.602$ (>0.1), H rock, $\alpha = 0.094$ (<0.1), D rock, $\alpha = 0.097$ (<0.1)). The MWU test shows that the amount of TD has a statistically significant influence on the concentration of Ca^{2+} in leachate in D and H rocks, but not in J rock.

For Na^+, the cumulative concentration in leachate from treatments subjected to a TD of 4–68°C is much higher than that from treatments with 4–40°C and 4–28°C temperature ranges, although trends for cumulative concentrations of Na^+ with 4–40°C and 4–28°C are similar. As is especially apparent in Figure 7(b,c), treatment with a TD of 4–68°C caused Na^+ concentration to greatly increase by 50 and 36% for H and J, respectively, compared to the 4–28°C treatment (MWU test of concentrations of Na^{2+}: J rock $\alpha = 0.117$ (>0.1), H rock $\alpha = 0.753$ (>0.1), D rock, $\alpha = 0.047$ (<0.1)). The MWU test shows that the range of TD has a statistically significant influence on the concentration of Na^{2+} in leachate in D rocks, but not in H and J rocks.

Altogether, Figures 7 and 8 show that the range of TD significantly influences concentrations of Ca^{2+} and Na^+ during TDWD treatment. On the whole, the larger the TD, the higher the concentrations of Ca^{2+} and Na^+.

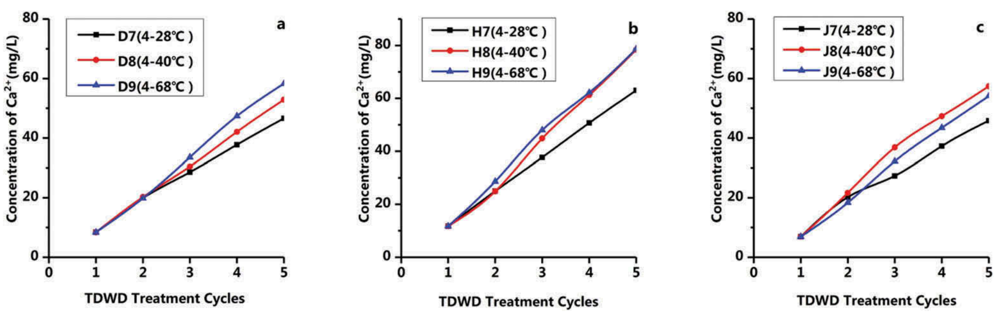

Figure 7. Trends of cumulative concentration of Ca^{2+} undergoing TDWD cycles. Rock D is silt-bearing mudstone (Nongshan Formation), rock H is silty mudstone (Shanghu Formation), and rock J is siltstone (Zhutian formation).

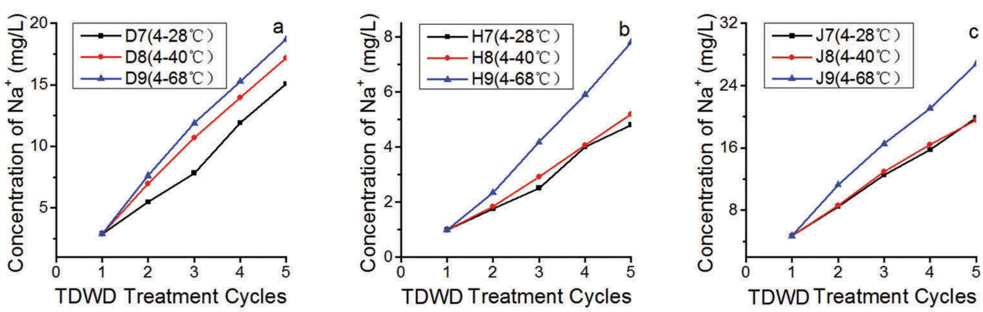

Figure 8. Trends of cumulative concentration of Na^+ as the WDTD cycles. Rock D is silt-bearing mudstone (Nongshan Formation), rock H is silty mudstone (Shanghu Formation), and rock J is siltstone (Zhutian formation).

Figure 9 shows that the magnitude of TD has a more significant influence on exchangeable cations, such as Na^{+}, Ca^{2+}, K^{+}, Si^{4+}, than on cations such as Mg^{2+}, Mn^{2+}, Al^{3+}, Si^{4+} that mainly dissolved from less soluble minerals. Therefore, rock–water interaction was accelerated by TD and, during this process, the solution effect was accelerated. We conclude that the influence of the magnitude of TD on increasing the disintegration rate occurred partly

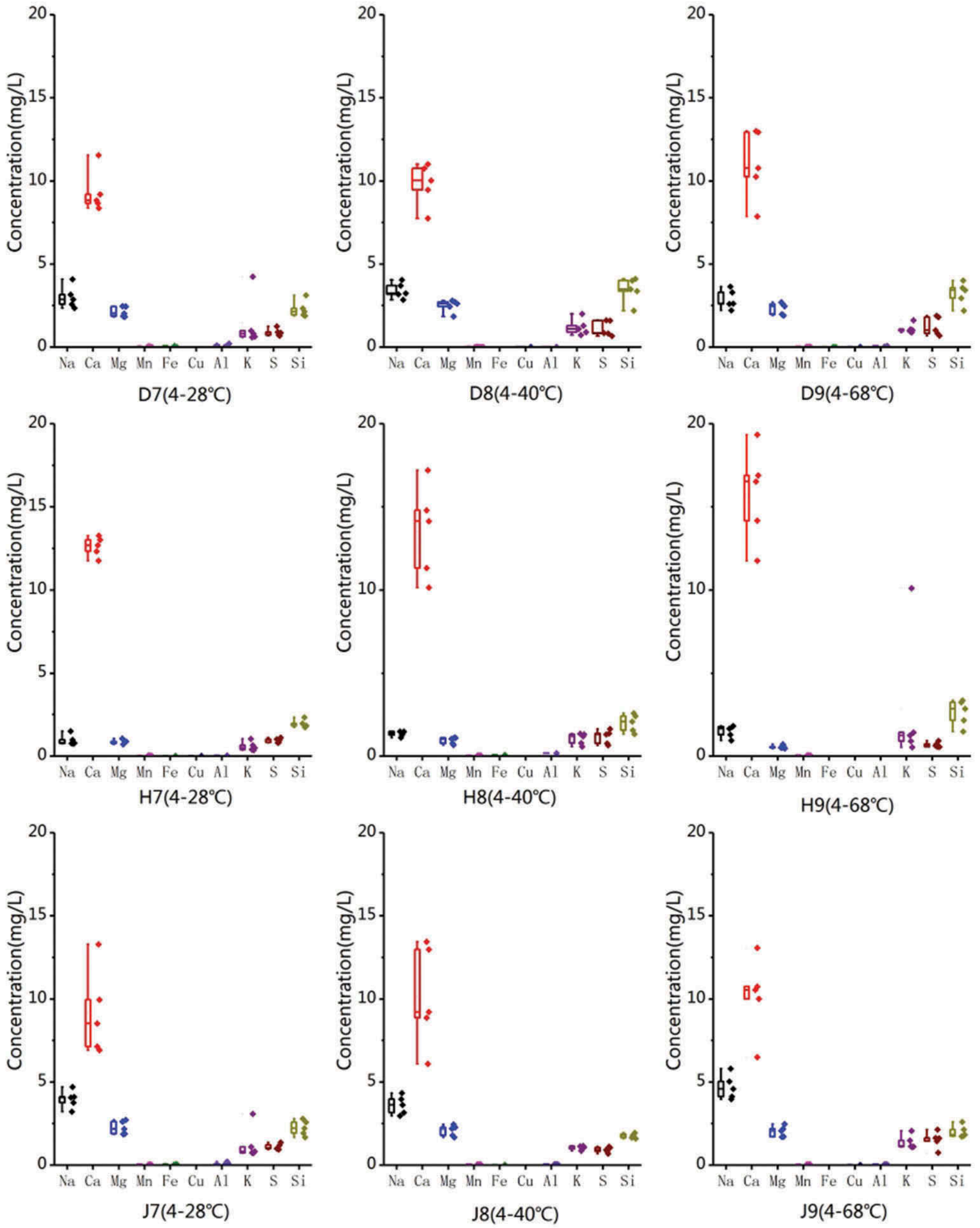

Figure 9. Box-plot of concentrations of detected cations. Rock D is silt-bearing mudstone (Nongshan Formation), rock H is silty mudstone (Shanghu Formation), and rock J is siltstone (Zhutian formation).

by accelerating the water–rock interaction. Given the same temperature when immersed in water, this implies that larger TDs in water–rock interaction create more cracks inside of the rock, which increase water–rock contact.

Discussion

The lithologies of D, H, and J are silt-bearing mudstone, silty mudstone, and siltstone, while, from high to low, rates of disintegration are J > H > D. From high to low, XRD results show that clay content is D > H > J and calcite content is H > D > J. Regarding their mineral constitutes, although calcite is known to be especially sensitive to temperature fluctuations (Eppes & Griffing, 2010; Widhalm, Tschegg, & Eppensteiner, 1996), the J and D rocks with the lowest calcite content showed totally different anti-disintegration abilities. Illite is known for shrink-swell minerals (Zhang, Xu, & Hu, 2016), but notably rock J, having the highest clay content, is not easy to decay. Thus we surmise that grain size might explain the decay of poorly cemented rocks.

Results from SEM observation show that TDWD treatment caused minerals to dissolve and promoted the development of cracks on rock surfaces; overall, TDWD treatment increased rock disintegration. At the same time, the results showed a significant influence of TD on disintegration; with larger TD imposed, disintegration rates during WD cycles were higher. In previous research, the main cause of damage in heating-cooling processes has been thought to result from differential thermal expansion and contraction of individual mineralogical components and of interior and exterior portions of the stone (Ghobadi & Babazadeh, 2015). For example, thermal expansion of quartz is three times that of feldspar, which exerts variable pressures upon heating, and stresses resulting from this process can combine to generate tensile strain sufficient for fracturing to occur (Ivanovich, 1990; Krynine, 1957; Selley, 2000). Secondly, water–gas phase transformation occurs frequently to water inside stone during cycling TD, affecting fracture development through volume change and migration effects (Wang et al., 2016). Steam splitting has an important role in accelerating fracture development and void communication (Hettema, Wolf, & Pater, 1998). Due to the rapid dissolution of cementation caused by the spreading of fractures, the mechanical property of rock decreases rapidly and, as a result, its disintegration rate increases.

The EC results also reveal that the trend of cumulative EC values during cycles of TDWD treatment is consistent with the result found by Higuchi (2013), that EC values fluctuate down from regolith on the surface to raw rock in a badland (Higuchi, Chigira, & Lee, 2013).

Although previous results have suggested that swelling clay minerals are an important determinant of rock decay (Brown, 1981; Doostmohammadi, Moosavi, Mutschler, & Osan, 2009), the expansible clay mineral content of the rock did not show a positive relationship with the extent of decay under the same treatment in our study.

In addition to the common factors that increase the rock decay process, such as (1) stronger thermal stress causing increased rock breakdown (Eppes & Griffing, 2010; Hall & André, 2001; McKay, Molaro, & Marinova, 2009) and (2) faster wet-dry alternation under higher temperatures, causing rapid evaporation of moisture and subsequent strong swelling-shrinking (Geng-She et al., 2002) and the dissolution of easily soluble elements (Goudie, 1989), and thus also to ultimate rock decay.

Rock decay rates significantly relate to temperature conditions during wetting-drying cycles (Zhang et al., 2016). Our data agree with this finding; moreover, it is more significant to D rock than J and H rocks. This implies that temperature variations have a more significant effect on more easily weathered rocks. In the present study, when the TD was 4–68°C, a significant difference in the release of Na^+ and Ca^{2+} cations appeared for H and J rocks. This implies that the threshold of TD efficiency for fatigue differs among the three rock types (Zhang et al., 2015). However, whether the main effect of TD is to cause rapid moisture variations or result in strong expansion and contraction still needs further research.

Conclusion

TDs can accelerate the disintegration rate by promoting the interaction between water and rock; moreover, this effect is more significant for rock that is more apt to disintegrate. For EC values of leachate collected after treatment, the influence of TDs is significant; specifically, the larger the TD, the higher EC values will be, especially for rock that disintegrates more readily. TDs exert a strong influence on the release of exchangeable cations during WD cycles, as a whole. The larger the TD, the higher the concentration of cations will be. If the TD is over the threshold of TD efficiency for fatigue, the influence can be significant.

In addition, in present study, we found that mineral constituents including calcite and illite can not determine the extent of decay; thus the grain size might be the main reason accounting for decay extent.

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Disclosure statement

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