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Column leaching test on oxidized and non-oxidized tailings in northern Norway

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Abstract. Column leaching test was applied for oxide and non-oxide tailings in the Arctic area. Physico-chemical parameters (pH, pe: potential ecolotric, conductivity, TDS: Total dissolved solids, SO_4^{2-}, salinity, Ni, Co, Zn and Mn) were measured to identified the effect of oxidized degree on heavy metals leaching from tailings. Results showed that oxidized tailings contained higher levels of Co and Ni, and their leachate was highly mineralized and less loaded with heavy metals. Non-oxidized tailings are easy to oxidation and vulcanization to generate acid drainage, weathering play more important role in oxidized tailings. Higher acid generation led to leachate having a low pH level, while strong weathering led to high levels of salinity in leachate. Acid generation and weathering controlled the release of Ni, Co, Zn, and Mn from tailings. Acid generation promotes Co and Ni release and weathering promote Mn release. Strong acid and highly salinity accelerated Co and Ni release from oxidized tailings. The source of Zn changed its origin between oxidized tailings and non-oxidized tailings leachate, with both acid mine drainage generation and weathering playing an important role in its release. Non-oxidized tailings take more sulfide that is easy to generate acid, oxidized tailing have more oxide material that lead to high minerals. Keep tailings from vulcanization and oxidation is a good way to prevent heavy metals leaching from tailings.

1. Introduction

Mining activity produces large volumes of tailings [1]. Tailings are the waste produced after the extraction of the targeted metal from the ore; through crushing and milling, particles are obtained that consist of one mineral and minerals are separated by their physical or surface characteristics [2]. Mine tailings contain mine residue and various kinds of heavy metals such as Mn, Cd, Cr, Ni, Zn and Pb. They are a significant source of the heavy metals discharged to deep soils and groundwater [3,4] and are a potential threat to the environment and human beings. Tailings currently constitute the largest and most extensive industrial solid waste and are likely to do so in the future. Many hazardous and noxious substances are leached from the tailings by draining and soaking in rain and surface water [2,5]. Heavy metals are possibly released from the tailings into the soil and water by weathering and acid drainage [6]. The treatment of tailings leachate containing heavy metals is a major environmental problem affecting all mining countries [7]. Therefore, tailings are an important potential contamination source for the environment and can never be neglected. To assess the risk of these chemicals pose to

the environment, it is important to consider not only the direct impact on the soil but also transport to other environmental compartments [8].

Large amounts of tailings directly discharged in the wild, and some are stored with covers. It is easier for weathering, oxidizing and sulfuring to occur in the uncovered tailings [9]. The oxidized tailings became red and brown with higher Cao, Fe_2O_3 and Al_2O_3 content, and the un-oxidized tailing still keep gray. Many heavy metals are released from oxidized and non-oxidized tailings and enter water and soils through climate change and biochemical action, having different effects on the ecosystem [10]. Although the concentrations of heavy metals were mostly higher in oxidized tailings than non-oxidized tailings, this does not mean that oxidized tailings bring a greater risk to the environment than non-oxide tailings. The potential of heavy metals in tailings, with respect to their mobility and ecotoxicological significance, is determined by their release rather than by the total heavy metal content [11]. The release of heavy metal cations to the water phase (leaching), and therefore their susceptibility to transport processes, depends on their solution speciation and their affinity. Changes in environmental conditions, such as redox and pH, will release heavy metals bound to different fractions, so monitoring of environmental conditions would appear to be a priority, particularly since such conditions in mine tailings are in a state of constant flux [12-14]. It is well known that, under highly acidic conditions, Cd, Co, Cr, Cu, Mn, Ni, Pb and Zn become more soluble and mobile when released from tailings [15]. The chemical and physical properties of the tailings and the prevailing environmental conditions affect the rate of release of heavy metals from tailings. Leaching concentration is a good index for evaluating tailings' potential risk. However, current research focuses on analyzing risk by the total content of heavy metals in the tailings materials themselves [12], which is inaccurate in the evaluation. Investigating the potential for heavy metal leaching from different tailings is important to access the risk of tailings. Evaluation of the degree of effect of oxidization on leaching is necessary for tailings management.

Column leaching test is a kinetic way to determine the weathering characteristics and leachate chemistry of tailings that are deposited by mining. In this paper, we examined the leaching behavior of Ni, Zn, Co and Mn in oxidized tailings and non-oxidized tailings under steady temperature (10°C) and precipitant (120 mm/month) to investigate Ni, Zn, Co and Mn leaching behavior during experimental column leaching in oxidized and non-oxidized mine tailings. In this context, the main objectives of this were applying bench-scale column leaching tests to evaluate the environmental risk of heavy metals leaching from tailings at different redox conditions, and to investigate their influence on heavy-metal leaching from different tailings.

2. Materials and methods

2.1. Study site
Ballangen is a municipality in Nordland County, Norway [16]. Amounts of tailings were piled directly in wild areas or in tailings dams. The mean monthly temperature increases from March to July and decreases from August to November [17]. The maximum value of the mean monthly temperature is 14°C in July, and the minimum is -5°C in January (Ballangen meteorological station located at $68^{\circ}25'20'$ N, $17^{\circ}27'28'$ E). Because of the Gulf Stream, the climate is not as cold as would be expected at this latitude. However, the fall and winter weather include snow, ice and strong winds. The mean annual temperature and precipitation of Ballangen are 4.1°C and 1420 mm, respectively (table 1). From April to October, the soils and tailings are not frozen, some environment risks will occur through heavy metals leaching from tailings by rainfall and weathering.

2.2. Column test conditions
Leaching experiments were conducted using transparent high-density polythene columns (7 cm $\times$50 cm high). To avoid the "side wall effect", the inner wall was polished with sandpaper in advance to increase the roughness. Columns had been soaked in 50% v/v HNO_3 at pH$\leq$1 for 48 h and then thoroughly rinsed with deionized water to remove any residual metals before the experiment. The top

of the leaching column was opened, the bottom was reserved for the outlet of the duct to collect the leachate and was covered with a layer of filter paper; a layer of filter paper was then installed after the 5-mm-thick quartz sand particles were loaded [18]. In order to maintain the evenness of the sample leaching, the specification for filling each leaching column was the same. For the experiments, one column filled with oxidized tailings, the other with non-oxidized tailings. According to the annual precipitation conditions and hydrological characteristics in the study area (table 1), 100 mm/month was set in the experiment. Then 600 ml/2weeks deionized water was added to each column to replicate meteorological conditions. The leaching experiments were carried out under a stable air temperature (10°C), with the thermostat based on the mean temperature of the leaching season (from April to October); water was added every 14 days and leachate was collected (two weeks were a leaching cycle). Leachate's pe, conductivity, SO_4^{2-}, TDS and pH were measured immediately.

Table 1. Distribution of the average annual rainfall in the study area and corresponding simulation leaching amount (HT: high temperature, LT: low temperature, MT: mean temperature).

|	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Annual
HT (℃)	-4	0	3	7	12	13	17	15	14	7	2	3	7.42
LT (℃)	-10	-8	-4	-1	4	6	10	8	7	0	-2	-2	0.67
MT (℃)	-7	-4	0	3	8	9	14	11	10	4	0	1	4.08
Precipitation (mm)	97	83	139	45	118	96	72	104	157	56	149	304	1420

2.3. Analysis
The pH and PE of the leachate were obtained by use of a Hanna pH meter, while a Hanna conductivity meter was used to measure the leachate's conductivity. The concentration of SO_4^{2-} was measured by an automatic potentiometric titrator (Metrohm 877 Titrino plus, Swiss). Tailings and leachate samples were sent to ALS (https://www.alsglobal.se/en; follow EPA 200.7, 200.8) for analysis. Following Swedish standards (SS 028113), tailings were dried at 105 ℃. The dried sample was melted with $LiBO_2$ and dissolved in HNO_3 (1:1 nitric acid and water), according to ASTM 3682. Reference material (CRMs) (GSS-16) as a control sample added in the digestion experiment was in the certified. Leachate was treated according to EPA 200.8 and measured by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES). International certified reference materials for the water (SRM 1643e) were analyzed in the measurement to assess accuracy and precision. Before measuring each sample, a standard solution of different concentrations (0.02, 0.05, 0.1, 0.2, 0.5, and 1.0 mg L^{-1}) was used to calibrate the ICP-AES and obtain a reliable work curve ($R^2 > 0.999$).

Descriptive statistics, Pearson correlation analysis and principal component analysis (PCA) were carried out using SPSS v.24 (SPSS Inc., Chicago, USA).

2.4. Oxide and non-oxide tailings situation
Historically, simple approaches were taken to dispose of tailings, such as discharging in downstream running water or down drains. Because the contaminants in tailings and mine drainage water cause considerable environmental issues, many methods are applied to deal with the tailings, including valley impoundments, ring dikes, in-pit impoundments, specially dug pits, tailings ponds and dams [19]. Once the tailings pond filled up, its surface would be covered by new soil and revegetated plants, which would protect tailings from weathering, oxidation and transporting. In the case of some other tailings ponds without cover, the uncovered tailings would oxidize more quickly. It led difference of heavy metals contents in tailings and from leachate. There was a significant variation between the concentrations found in oxidized and non-oxidized tailings. In the oxidized tailings, there were higher Fe_2O_3, Al_2O_3 and CaO, 17.3%, 4.47% and 3.18%, respectively. And in the un-oxidized tailings, they had lower content with 14.2%, 3.95% and 2.22%, respectively. The Co (83 mg/kg) and Ni (476 mg/kg) content was higher in oxide tailings, while Mn and Zn showed higher concentrations in non-oxidized

tailings (figure 1). According to the total content of heavy metals in tailings, oxidized tailings had a greater risk of having a higher Ni and Co content, and non-oxidized tailings carried more risk of higher concentrations of Mn and Zn. The uncovered tailings (oxidized tailings) were easy to weathering, oxidizing and sulfidation, coupled with rainfall leaching, Mn and Zn are easier to release from tailings, so the oxidized tailings had lower content of Mn and Zn. Tailings with covers (non-oxidized tailings) decreases weathering and biochemical reaction [19], that prevented heavy metals from change forms and releasing, so led to heavy metals steady in the non-oxidized tailings. Oxidation and sulfidation may promote the release of Mn and Zn from tailings and cause oxidized tailings to have a lower heavy metal content. For the non-oxidized tailings, oxidation and sulfidation will lead to Co and Ni stabilization. It is not accurate to assess the risk from the total heavy metal content of tailings for some heavy metals were steady in the tailings, and some were released to the environment to pollute water and soils. Evaluating the risk of tailings by their leaching ability is a more exact way of monitoring the environment.
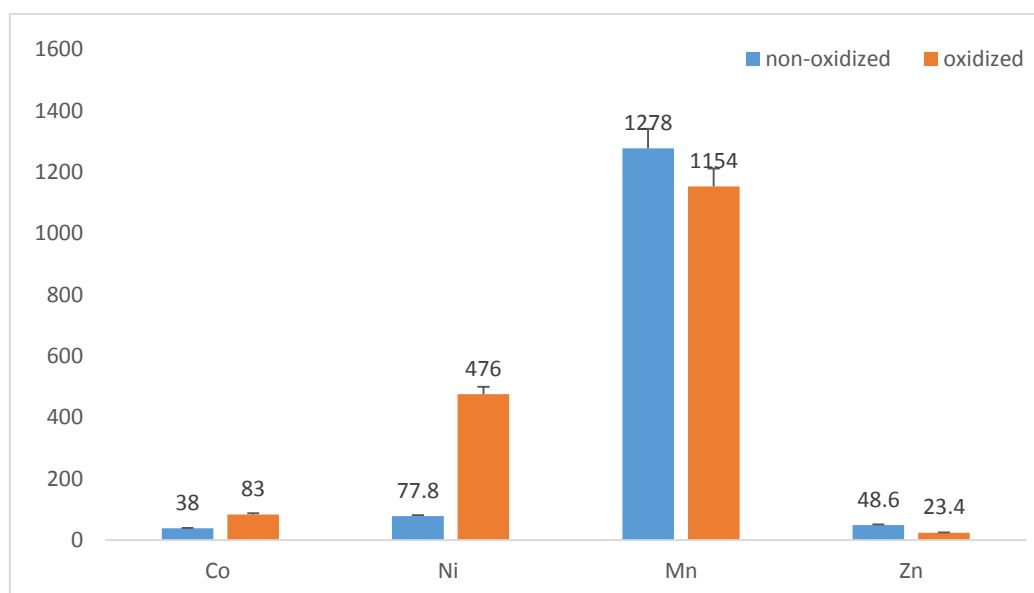
Figure 1. Statistical data for various heavy metals in tailings (in milligrams per kilogram).

2.5. Physicochemical property of the leachate
Leachate pH, pe, conductive and SO_4^{2-} values for the oxidized and non-oxidized tailings, respectively, are shown in figure 2. At the beginning of the leaching experiments, there was little difference between the pH values of oxidized and non-oxidized tailings leachate: 4.01 and 4.42, respectively. The pH increased from the first to the third cycle in both oxidized and non-oxidized tailings leachate. At the first 3 cycles, the pH of oxidized tailings leachate increased to 6.76, and kept a balance, and then to the 12th cycles, it declined to 3.8, respectively. In the non-oxide tailings, the pH increased to 6.17 from the first cycle to the third cycle, and declined to 4 in cycle 10, increasing slightly to 5.5 from cycles 10 to 11, finally decreasing to 3.54. In the cycles, the pH value of oxidized tailings' leachate was higher than that of non-oxide tailings' leachate. The variation in the pH value of oxidized tailings' leachate is smoother than that of non-oxidized.
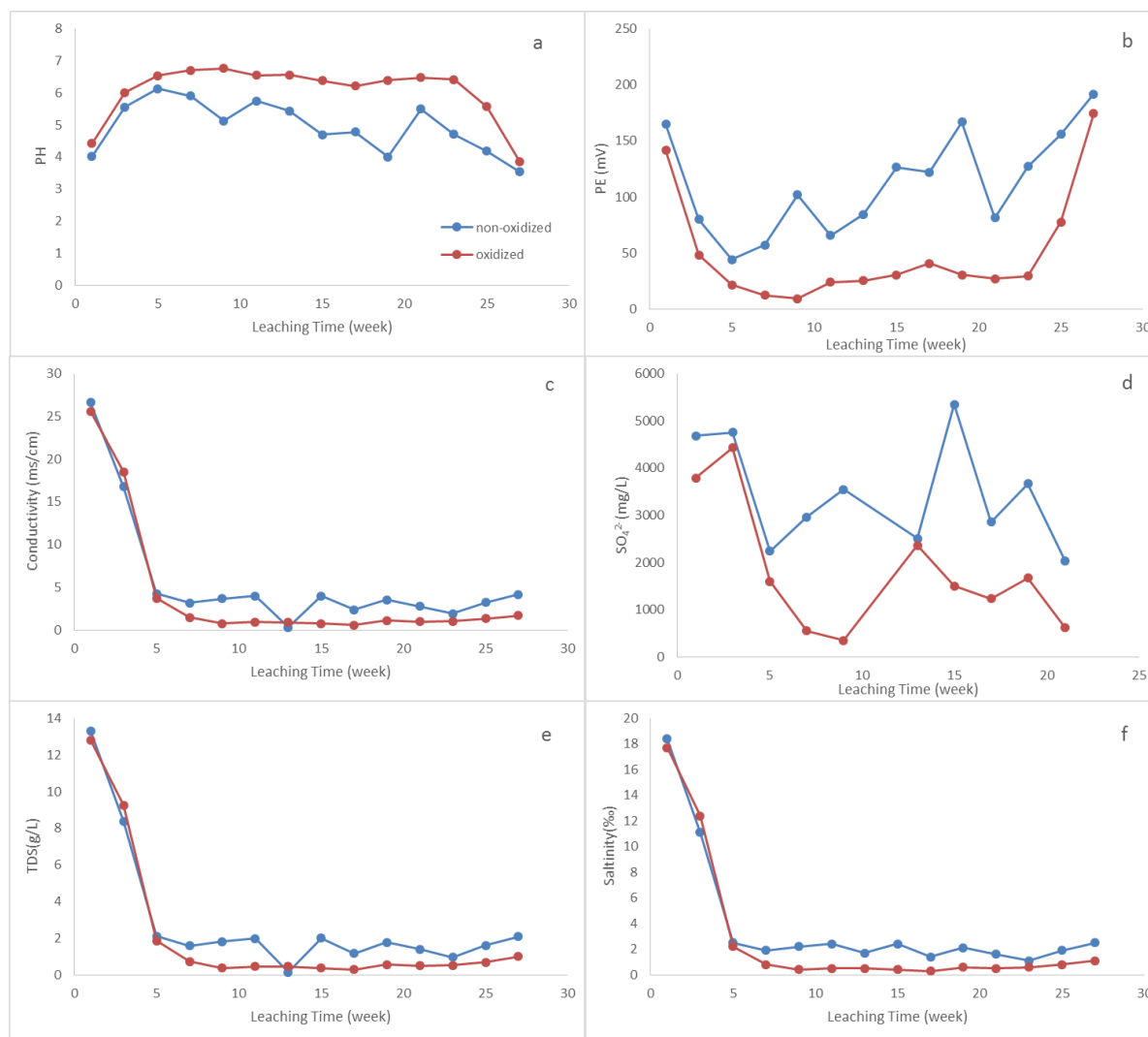
**Figure 2.** Physicochemical variables of the leachate.

The variations in the values of electrical conductivity were between a minimum of 0.291 mS/cm and a maximum of 26.6 mS/cm in non-oxide tailings' leachate; in oxidized tailings' leachate, this changed from 0.579 mS/cm to 25.6 mS/cm (figure 2). The level of conductivity of both types of leachate decreased sharply during the first five cycles and maintained steady low values in the latter cycles. Both of the leachates had a high risk to the surrounding environment of exceeding the standard for the conductivity of irrigation water in the first two cycles, which is 15 mS/cm [20]. So, the leachate in the earlier cycles were highly mineralized and posed a potential risk to groundwater.

PE, pH and conductivity are the main properties of leachate; they affected metals' leaching activity in tailings. Both pH and PE were determined by H^+ concentration; low pH and high PE easily oxidize the tailings and dissolve heavy metals At the start of the leaching cycle, the PE values of oxide tailings and non-oxide leachate were high: 141.6 mV and 164.8 mV, respectively. High PE is beneficial for generating H^+ and accelerating the oxidization of tailings [21], which leads to the creation of more acid mine drainage. Therefore, the non-oxidized tailings generated acid mine drainage more easily for high PE.

The concentration of SO_4^{2-} is an important index to indicate tailings' oxidization and sulfidation; high levels of SO_4^{2-} content mean more intense sulphur oxidation. The non-oxidized tailings showed higher concentrations of SO_4^{2-} during the leaching cycles. Oxidized tailings contain more metal oxides

and sulfides, so it is easier to generate acid mine drainage [22] (Kleiv and Thornhill 2004). After rainfall leaching, the oxidized tailings are more likely to lose SO_4^{2-} by hydrolysis of metal sulphides, so there was a higher level of SO_4^{2-} content in non-oxidized tailings' leachate (figure 2). In the first cycle, there was little change in SO_4^{2-} content, but the SO_4^{2-} decreased more sharply in the oxidized tailings' leachate and maintained low values. The pH trend showed the opposite result to that of SO_4^{2-}. Lower levels of pH will accelerate the actions of oxidizing and sulfuring in tailings [23]. So, the non-oxidized tailings' leachate showed higher concentrations of SO_4^{2-} in the latter leaching cycles.

Leachates' pH was valued from 3.54 to 6.76 in the leaching process (figure 2). However, the level of acid generation differed in the two types of tailings. From the 3rd cycle to the 10th cycle, the pH level of the oxidized and non-oxidized tailings' leachate decreased, indicating that acid was continuously and increasingly generated [24]. The pH value of oxidized tailings' leachate exceeded that of the non-oxidized, which implied that the acid-generating ability of non-oxide tailings was much stronger in the leaching process. This could be attributed to the higher content of organic matter and sulfuret [25]. The conductivity of the leachate is associated with the concentration of ions. Conductivity decreased with the leaching time delay; the electrical conductivity of non-oxidized tailings was higher than that of oxidized tailings, which implied that the ion transport or chemical reaction process was stronger in the non-oxidized tailings (figure 2). During the first four cycles, the leachate brought out a large number of ions and salts, which led to big changes in the leachate's conductivity [26]. Higher pe value showed in non-oxidized tailings' leachate, implying that it had more drastic oxidation and a greater ability to generate acid [27].
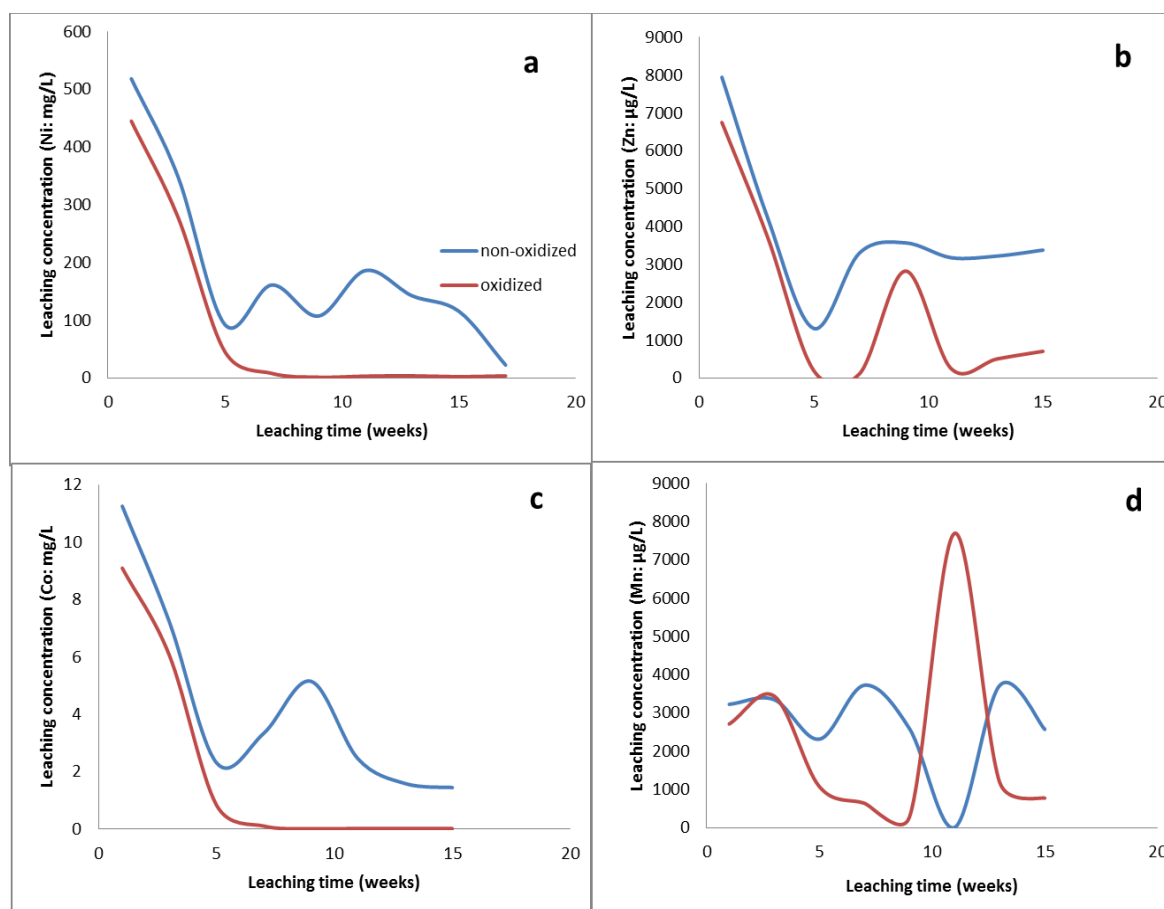
Figure 3. Column sub-annual precipitation cycle leachate Ni, Zn, Co and Mn in oxidized and non-oxidized tailings.

2.6. Heavy metals contained in the leachate

We obtain the element concentrations on a volume basis to account for variations in leachate between cycles. As shown in figure 3, sub full cycle leaching Ni, Zn, Co and Mn from the columns. In most cases, the levels of leached concentrations of Ni, Zn and Co are higher from the non-oxidized tailings than from the oxidized tailings. The concentration of Mn in the leachate is different from that of Ni, Zn and Co, and showing a higher level in oxidized tailings' leachate from the 4th to the 5th cycle and a lower level in the other cycles. The changing trend of Mn in the leachate is in opposition to the variations in SO_4^{2-} (figures 2 and 3) in both leachates: Mn decreased, while SO_4^{2-} increased. Leaching concentrations vary with leaching time. Leaching concentrations of Ni, Zn and Co reached a bottom level at the 3rd cycle in both oxidized tailings' leachate and then kept a steadily low value. Meanwhile, in the non-oxidized tailings leachate, the Ni, Zn and Co content decreased similarly to that of oxidized tailings leachate in the first three cycles; as the leaching operation time elapsed, the leaching concentration continuously increased over two cycles, then peaked, then declined over a cycle and finally stabilized. Although the total concentrations of Ni and Co were higher in oxide tailings, the concentrations in leachate were contradictory (figures 1 and 3). Ni, Zn and Co exhibited a rapid dissolution from oxidized and non-oxidized tailings in cycles 1 and 2. The leaching concentrations of Ni, Zn and Co declined sharply in cycles 2 and 3 in both leaching. Changes in the release of heavy metals across the sub-cycles show a mixed pattern in oxide tailings' leachate and non-oxide tailings' leachate. Compared with the leaching ability of Ni, Zn and Co from oxidized tailings and non-oxidized tailings, its variation is the same as with pH changes (figures 2 and 3). Therefore, non-oxidized tailings easily generate acid and promote Ni, Zn and Co release.

2.7. Effect of tailings oxidized degree on heavy metals' leaching

Both the oxidized and non-oxidized tailings are highly loaded with heavy metals. Their quantification allows us to evaluate the potential for heavy metals and to take steps to anticipate the impact on the environment. Compared with the control columns experiment, changes in physical-chemical variables, total content and leachate concentration from oxidized tailings were significantly different. The degree of tailings oxidation affected their leachate concentration and leachate variables. Correlation analysis is a good way to identify a single influence on leaching metals, and principal component analysis will help us to obtain the mechanisms that govern the behavior of these metals [20]. To interpret the mechanisms that govern the behavior of these metals leaching from tailings, correlation and principal component analysis between chemical parameters was applied to the study. As shown in table 2, Co and Ni were significantly positively correlated with TDS, Conductivity and Salinity in both the leachates (table 2). Co and Ni were negatively correlated with pH in the both leachate. In the non-oxidized tailings' leachate, there was significant positive correlation between Ni and Co (0.904), between Ni and Zn (0.912), and between Co and Zn (0.874) at the 0.01 level, which may suggest a common origin in the leachate [24]. In general, Mn was positively correlated with Zn, and Ni was positive correlated with Co in both leachate.

Table 2. Correlation matrix between leachate physicochemical variables and concentration.

		PH	PE	TDS	Conductivity	Salinity	SO_4^{2-}	Zn	Co	Ni	Mn
	PH	1									
	PE	-1.00**	1									
	TDS	-0.623	0.630	1								
	Conductivity	-0.623	0.630	1.000**	1							
Non-Oxidized	Salinity	-0.644	0.652	0.994**	0.994**	1						
	SO_4^{2-}	-0.711	0.721	0.567	0.567	0.537	1					

Group		PH	PE	TDS	Conductivity	Salinity	SO_4^{2-}	Zn	Co	Ni	Mn
	Zn	-0.836**	0.838**	0.860**	0.860**	0.881**	0.580	1			
	Co	-0.607	0.613	0.943**	0.943**	0.939**	0.453	0.874**	1		
	Ni	-0.598	0.605	0.964**	0.964**	0.976**	0.509	0.904**	0.912**	1	
	Mn	0.111	-0.109	0.069	0.069	0.119	-0.108	0.273	0.088	0.320	1
	PH	1									
	PE	-.998**	1								
	TDS	-.909**	0.892**	1							
	Conductivity	-0.909**	0.892**	1.000**	1						
	Salinity	-.916**	0.900**	1.000**	1.000**	1					
Oxidized	SO_4^{2-}	-0.705	0.697	0.853*	0.853*	0.849*	1				
	Zn	0.088	-0.098	-0.24	-0.24	-0.234	-0.288	1			
	Co	-0.925**	0.909**	0.999**	0.999**	1.000**	0.842*	-0.223	1		
	Ni	-0.936**	0.921**	0.997**	0.997**	0.998**	0.831*	-0.225	0.999**	1	
	Mn	0.071	-0.077	-0.237	-0.237	-0.233	-0.163	0.923**	-0.226	-0.227	1

**. Correlation is significant at the 0.01 level (2-tailed).
*. Correlation is significant at the 0.05 level (2-tailed).

These strong correlations are used to form groups of elements that are governed by the same mechanism during their leaching; these are shown as components 1 and 2 (table 3). The factors 1 and 2 explain 94.19% (76.08% for factor 1 and 18.11% for factor 2) and 86.49% (71.94% for factor 1 and 14.55% for factor 2) of the variance in the two leachates (table 3). Component 1 describes the general loading of the tailings' leachate, which originates from acid generation and weathering. High levels of PE, pH, Ni, Co, SO_4^{2-}, TDS, salinity and conductivity existed in component 1 in both tailings. There were strong correlation coefficients, ranging from -0.598 for Ni-pH to 0.998, for PE (table 2). These results showed that component 1 is the most common group in respect of leaching ability and origin. All the group parameters were generated from the acid mine drainage; during the leaching cycle, tailings were oxidized and sulfured; finally, a lot of acid mine drainage was generated when water flowed across the tailings (Johnson et al. 2001). The high level of Mn in Component 2 correlated weakly with the other parameters (table 2), meaning that the origin of Mn was differed from those variables. In oxidized leachate, Zn obtained high levels in component 2 and significantly positively correlated with Mn (tables 2 and 3), which implied they have a similar origin in the leachate. Climate change serious increased tailings weathering, and lead elements to change forms. So, Zn in oxidized tailings' leachate originated from weathering but not acid releasing for the high pH. This conformed to the results from the tailings' investigation (figure 1): lower levels of Mn and Zn in oxidized tailings for weathering releasing and precipitant transporting. There was much more organic matter and sulfide in the non-oxidized tailings, and stronger sulfidation-oxidation action in non-oxidized tailings led to lower pH, higher conductivity and pe, which is beneficial for the release of Co and Ni. Therefore, lower levels of Co and Ni showed in the non-oxidized tailings for their acid leaching abilities (figure 1). pH was a key influence in the leaching. Many biotic or abiotic parameters influence the levels of pH, such as salinity, temperature, dissolved oxygen, but the most important is biochemical activity. However, changes in the pH values may be due to the different reasons listed above. Decreasing pH will lead to more acid mine drainage, which accelerates the release of heavy metals from tailings. Weathering promoted the release of Mn and Zn from tailings. Non-oxide tailings are easy to weather and generate acid mine drainage when expose in nature.

Table 3. Community component of leachate physicochemical variables (component matrix).

	Oxidized		Non-Oxidized	
	Component 1	Component 2	Component 1	Component 2
pH	**-0.949**	0.001	**-0.8**	0.495
PE	**0.938**	0.003	**0.807**	-0.49
TDS	**0.992**	-0.296	**0.954**	0.163
Con	**0.992**	-0.296	**0.954**	0.163
Sal	**0.993**	-0.289	**0.961**	0.191
SO4	**0.855**	-0.242	**0.676**	-0.498
Zn	-0.258	**0.979**	**0.96**	0.005
Co	**0.995**	-0.278	**0.919**	0.216
Ni	**0.952**	0.293	**0.952**	0.293
Mn	-0.219	**0.979**	0.173	**0.706**

3. Conclusions

Oxidized and non-oxidized tailings were used in column leaching experiments to investigate the impacts of physicochemical variables on heavy metals' leaching from different tailings in the arctic area. The results of this study indicated that oxidized tailings contained higher levels of Zn and Mn, and lower levels of Co and Ni. Non-oxidized tailings carried a higher risk in respect of their higher level of leaching concentrations of Co, Ni and Zn. In the leachate of both tailings, the origin of Co and Ni was dissolution from acid drainage generation, while Mn originated from natural weathering. The origin of Zn varied in oxidized tailings and non-oxidized tailings, with both acid mine drainage generation and weathering playing an important role in its release. Low pH, high conductivity, high salinity and high TDS accelerated the release of Co, Ni and Zn from non-oxidized tailings. In oxidized tailings, those physicochemistry indexes accelerated the release of Co and Ni. Acid and salinity had a weak impact on the leaching of Mn from tailings. Non-oxide tailings hold more sulfide that is easy to generate acid, oxide tailing have more oxidized material that lead to high salinity. Keep tailings from vulcanization and oxidation is a good way to prevent heavy metals leaching from tailings. Discovering the relationship between leaching characteristic and degree of oxidation, is beneficial for understanding the transportation of the heavy metals from tailings and controlling pollution from tailings leaching.

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