

Desalination of Saline Sludges Using Ion-Exchange Column with Zeolite

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Abstract: A flushing process followed by a zeolite based ion-exchange process were developed for the treatment of saline sludges from oil and gas exploration sites. The particle size distribution of sludge sample indicated the presence of very fine sand and clay. The electrical conductivity of the sludge was 42.2 dS m^{-1} indicating very saline sludge and the Cation Exchange Capacity (CEC) was 40 cmol kg^{-1} which was very suitable for ion-exchange process. A 500 g sample of saline sludge (containing $\text{CaCl}_2:\text{MgCl}_2:\text{NaCl}$ ratio as 1:1.16:36.61) was washed using demineralized water in a mixed reactor and the sludge was allowed to settle for 36 h. The optimal number of washes was determined to be two washes with an overall salt removal efficiency of 94.47%. The treated sludge contained $515 \text{ mg salt kg}^{-1}$ sludge (or 0.05% w w^{-1}) and was suitable for agricultural application. The washwater was passed through a Mountain Stronach zeolite (chabazite) based ion-exchange column for salt reduction. The sodium salt removal efficiency was 75.34%. This was increased to 99.79% when using two ion-exchange columns. The Ca and Mg ions were under regulatory limits and required no further treatment. The final salt concentration in the wash water was 314.0 mg L^{-1} which was below the limits established by the Canadian Guidelines. For complete removal of total salts, a series of ion-exchange columns with different zeolites (for removal of NaCl, MgCl_2 and CaCl_2) will be required.

Key words: Saline sludge, salt, zeolite, washing, demineralization, desalination, EC, CEC, ion-exchange, NaCl, MgCl_2 , CaCl_2

INTRODUCTION

Soils may be salinized as a result of resource extraction and other human activities. However, most of soil salinity in western Canada is resulting from oil and gas exploration^[1]. Saline water and sludge are usually produced during this process and stored on site. The current disposal of contaminated materials is inefficient and costly^[2]. The remoteness of many sites complicates the disposal process as these sites are only accessible in winter^[3]. Furthermore, the disposal process results in the storage of contaminated materials with no further treatment.

The result of not treating the contaminated material is the destruction of the vegetation and creation of bare areas^[2,4,5] as well as contamination of ground water^[3,6]. The regulating bodies responsible for oil and gas operations in Canada are beginning to enforce clean ups to eliminate any potential environmental hazards^[7]. Therefore, an economically sustainable efficient technology is required to remove salts from contaminated soils and sludges associated with oil and gas production.

The objective of this study was to develop a treatment process for a salt-contaminated sludge associated with natural gas and oil production. The specific objectives were: to develop an efficient salt removal process from saline sludge, to develop a water remediation system for the saline wash water and to evaluate the performance of the two processes.

BACKGROUND

Sludge remediation: Sludge is normally composed of largely water, biological compounds (microbial cells and various cellular products), organic solids, metals and several other pollutants which are usually above standard guidelines^[8]. The complex physiology of sludge offers great challenges in separation of hazardous compounds^[9]. Presently, there are different techniques used for the treatment of contaminated sludges including: conditioning, thickening/dewatering, composting and washing processes.

Conditioning of the sludge can be performed through chemical and thermal treatments. Chemical additives such as lime, ferric chloride and cyclodextrins

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are used to act as chemical forces to drive together colloidal particles in the sludge^[10-12] whereas thermal conditioning involves heat and pressure as a method to drive particles together^[8,13,14].

Sludge thickening/dewatering is a process of removing water to reduce the volume of a sludge stream and decrease the cost of its handling^[8,9,15]. Thickening/dewatering methods include centrifugation^[16], sedimentation^[17], vacuum filters^[18], belt filter press^[19], drying beds^[20] and sludge lagoons^[21].

Sludge composting results in moisture removal, volume reduction, stabilization of waste and destruction of pathogens. Composting techniques include: windrows^[22], biopiles^[23] and bioreactors^[8]. Windrows are elongated, dome shaped piles, under 3 m in height, normally constructed on a lined surface (with geotextile material) with a liquid collection system^[8]. The biopiling process begins with mixing the contaminated sludge/soil with straw and fertilizer amendments and the bio-pile is then turned over once a month to accelerate the bio-remediation process^[23]. Bioreactors are closed composters which are used when controlled conditions such as temperature and humidity are essential^[8].

Washing is currently used to treat soil/sludges containing organic and inorganic contaminants. This technology is considered to be a relatively low-cost alternate to land disposal^[24]. Soil washing is a physical/chemical treatment process which normally involves:

- Excavation and staging of contaminated soil/sludge
- Pretreatment of soil/sludge to remove large objects and oversized clods
- Washing the soil/sludge with water (chemical extracts may be used in the wash water) to separate the contaminants
- Recovering the clean soil/sludge fraction that can be redeposited on site^[25]

The removal efficiencies are highly dependent on the physical and chemical characteristics of the soil/sludge and contaminants. The process depends on the Cation Exchange Capacity (CEC) of the soil/sludge which is a measure of the soil ability to exchange cations. Cations are held by electrostatic forces on the soil/sludge particle surfaces to balance the negatively charged clays^[26]. A high CEC in soil indicates a stronger bond between the cations located on the surface of the soil (higher capacity to hold onto cations) and it is less likely that they will exchange with other cations. In comparison to other sludge remediation

techniques, sludge washing has many advantages including: less time required, higher treatment efficiency, low requirements of chemicals/biological agents to treat pollutants in sludge and ease of operation^[24].

Water demineralization: Methods of the demineralization of water include: distillation, membrane separation and electro dialysis processes. Distillation is based on the fact that the water evaporates, while the salt components stay behind^[27].

Membrane separation processes are pressure intensive operations in which solutes of different molecular weights are separated from solution by applying moderate to high differential pressure across membranes^[28,29]. There are different membrane separation processes based on particle sizes: microfiltration, ultrafiltration, nanofiltration and reverse osmosis. In principle, large molecules are separated from solution through membranes made from synthetic polymers as well as ceramic materials^[28]. Unlike conventional filtration, in these processes the solution flows parallel to the membrane, cleaning away any particles remaining on the membrane surface. Microfiltration membrane pore-size range between 0.1-10 μm and requires up to 2-3 atm differential pressure. Ultrafiltration operates around 3-10 atm differential pressures and utilizes 0.001-0.1 μm pore-size membranes.

In case of nanofiltration, differential pressure above 15-20 atm is applied to 0.0001-0.001 μm pore-size membrane to separate low-molecular-weight organic solutions like alcohol from organic solutes, suspended solids and polyvalent ions. Osmosis is a driving force for the flow of solvent molecules due to a difference in chemical potential on the two sides of a membrane^[30]. Reverse osmosis is the movement of water in a direction opposite to that it would move to in a normal osmosis^[28]. Unlike microfiltration and ultrafiltration, reverse osmosis operates similar to nanofiltration, albeit at very high pressure (up to over 20-30 atm) and separates salts as well as macromolecules and particles, allowing only the water molecules. In fact, in many European countries almost all newly set up water desalination plants for potable water are based on reverse osmosis process despite having higher installation and operating costs^[29].

Electrodialysis is another option in removing the salts from the saline solution utilizing electrophoresis of ionic substances in solutions and the selective permeability of ionized inorganic and ionized organic exchange membranes.

Electrodialysis has several advantages over all other techniques of wastewater demineralization including: no evaporation are required, low power requirement for pumping, relatively cheaper, use of naturally occurring catalysts that are easier to regenerate and ease of operation^[31].

Zeolites are composed of SiO_4 tetrahedra (Fig. 1) in which each oxygen is shared between two tetrahedra, occasionally Al^{3+} ions in place of Si^{4+} ^[32]. The chemical composition for various types of zeolites is shown in Table 1^[34]. The ion-exchangeability is due to the presence of extra-frame cations, located in the regular array of channels and cages. Cations are bound to the lattice and to water molecules, which normally fill the zeolite micropores. Substitution is stoichiometric and unless partial or total exclusion occurs, the entire cation exchange capacity can be covered^[35].

Zeolites are commonly used in water softening processes to exchange Ca^{2+} for Na^{+} ^[36]. Electrodialysis can be performed on saline water (containing CaCl_2 , MgCl_2 and NaCl) using an ion-exchange column consisting of zeolite as a medium, in which the Na^+ , Ca^+ and Mg^+ ions can be exchanged for H^+ ion and the Cl^- ion can be exchanged for OH^- ion.

EXPERIMENTAL APPARATUS

Soil washing unit: A simple soil washing unit was constructed as seen in Fig. 2. It consisted of three cylinders, one for the clean water (10 cm diameter and 100 cm height), the second for the contaminated soil (10 cm diameter and 25 cm height) and the third for the collection of wash water (10 cm diameter and 25 cm height). The cylinders were constructed from Plexiglas pipes. The bottom of each cylinder was made of a Plexiglas circular plate which was glued into the bottom end of the pipe and fastened with four screws.

To prevent sediments from traveling through the tubing, a GeoTech[®] screen (GeoTech Environmental Inc., Tamarac, Florida) was placed at the bottom of the soil cylinder. The cylinders were connected by 1.5 cm diameter Tygon[®] tubing. Two 1.5 cm ball valves (Northeast Equipments Ltd., Halifax, Nova Scotia) were used to facilitate the movement of clean water into the soil cylinder and the movement of saline water into the wash water collection cylinder. A motor with speed controller (Dayton Electric MFG. Co., Chicago, Illinois) was used to mix the water/soil mixture to allow for the transfer of salts from the soil to the water.

Water desalination unit: A simple water desalination unit was constructed as shown in Fig. 3. It consisted of

Table 1: Chemical composition of some natural zeolites^[34]

Zeolite	Idealized unit-cell formula	Si / Al ratio
Chabazite	$\text{Ca}_6[\text{Al}_{12}\text{Si}_{24}\text{O}_{72}].40\text{H}_2\text{O}$	1.43-4.18
Clinoptilolite	$\text{Na}_3\text{K}_3[\text{Al}_6\text{Si}_{30}\text{O}_{72}].20\text{H}_2\text{O}$	2.92-5.04
Erionite	$\text{Na}_2\text{K}_2\text{MgCa}_{1.5}[\text{Al}_9\text{Si}_{27}\text{O}_{72}].27\text{H}_2\text{O}$	3.05-3.99
Ferrierite	$\text{Na}_2\text{Mg}_2[\text{Al}_6\text{Si}_{30}\text{O}_{72}].18\text{H}_2\text{O}$	3.79-6.14
Heulandite	$\text{Ca}_4[\text{Al}_8\text{Si}_{28}\text{O}_{72}].24\text{H}_2\text{O}$	2.85-4.31
Laumontite	$\text{Ca}_4[\text{Al}_8\text{Si}_{16}\text{O}_{48}].16\text{H}_2\text{O}$	1.95-2.25
Mordenite	$\text{Na}_8[\text{Al}_8\text{Si}_{40}\text{O}_{96}].24\text{H}_2\text{O}$	4.19-5.79
Phillipsite	$\text{Na}_2\text{K}_2\text{Ca}[\text{Al}_6\text{Si}_{16}\text{O}_{52}].12\text{H}_2\text{O}$	1.45-2.87

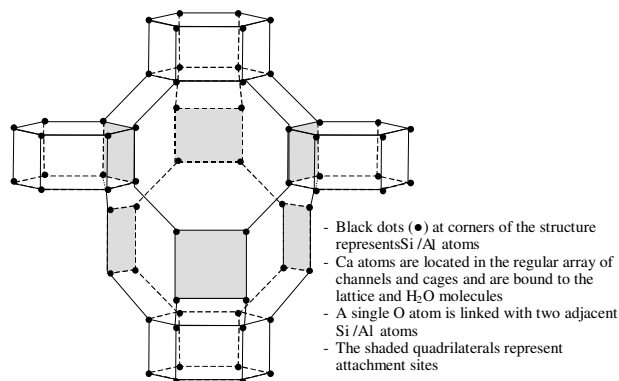


Fig. 1: Three dimensional structure of chabazite, the dominant from in Nova scotian mountain stronach zeolites^[33]

three cylinders, one for the waste water (10 cm diameter and 50 cm height), the second was used as ion-exchange column (5 cm diameter and 25 cm height) and the third was used for collection of clean water (10 cm diameter and 25 cm height).

The cylinders were constructed from Plexiglas pipes. The bottom of each cylinder was made of a Plexiglas circular plate which was glued into the bottom end of the pipe and fastened with four screws. A GeoTech[®] screen (GeoTech Environmental Inc., Tamarac, Florida) was placed at the bottom of the ion-exchange column before filling it with zeolite. The three cylinders were connected with 1.5 cm diameter Tygon[®] tubing. A motor with speed controller (Dayton Electric MFG. Co., Chicago, Illinois) was used to mix the wash water.

MATERIALS AND METHODS

Sludge: Sludge samples were collected in July from a gas production field in British Columbia, Canada. The sludge characteristics are shown in Table 2. The analyses were performed at Philip Analytical Service, Dartmouth, Nova Scotia. The sodium absorption ratio (SAR) was calculated using the concentration of Na, Ca

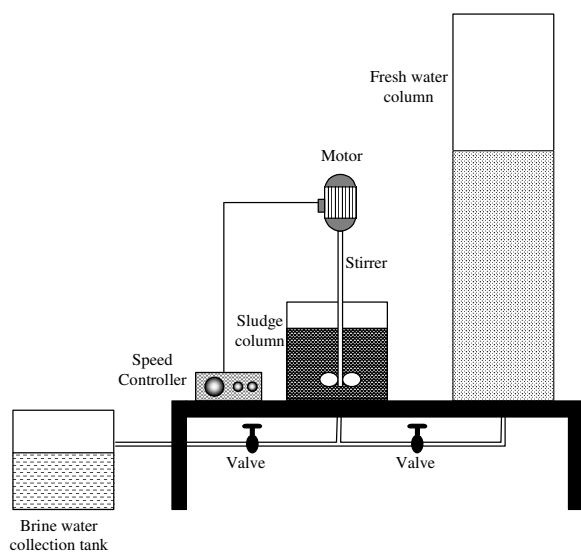


Fig. 2: Sludge demineralization unit

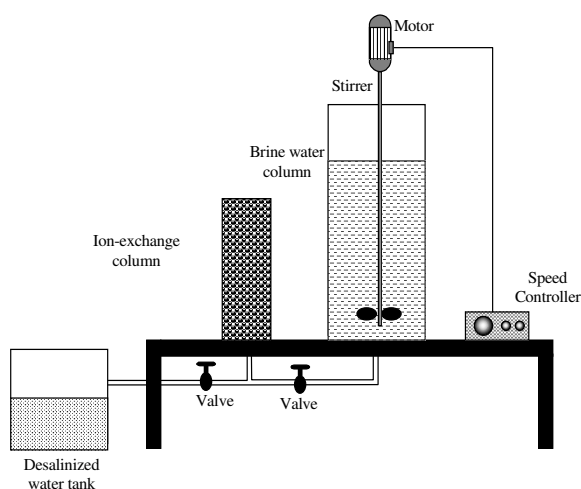


Fig. 3: Water desalination unit

and Mg ions (m mol L^{-1}) according to the following equation^[1].

$$\text{SAR} = \frac{[\text{Na}^+]}{[\text{Ca}^{++}] + [\text{Mg}^{++}]} \quad (1)$$

A 500 g sample was placed in a convection oven (Fisher Scientific) to dry at 105°C for 24 h according to the procedure described by Black *et al.*^[37]. The dried sample was in the form of large clumps (50 mm in length). These clumps were reduced in size using a hammer. Then, 100 g of this dried granule sample were placed in the sieve shaker (W.S. Tyler Incorporated,

Table 2: Sludge characteristics

Item	Value	Industrial limit ^[71]
Soluble elements (mg kg^{-1})		
Calcium	95	na
Magnesium	78	na
Sodium	3400	na
Chloride	5750	na
Total salt (mg kg^{-1})	9323	--
Electrical Conductivity (dS m^{-1})	24.2	4
Sodium Absorption Ratio	35.1	12
CEC (cmol kg^{-1})	40.3	--
pH	6.7	6.6-8.5

na = not applicable

Table 3: Size limits of soil separated^[37]

Name of separate	Diameter range (mm)
Very coarse sand	2.0-1.0
Coarse sand	1.0-0.5
Medium sand	0.5-0.25
Fine sand	0.25-0.10
Very fine sand	0.10-0.05
Silt sand	0.05-0.002
Clay	<0.002

Table 4: Sieve analysis results

Mesh	Screen opening (mm)	Mass fraction retained	Average particle diameter in increment (mm)	Cumulative fraction smaller than screen opening
14	1.410	0.487	-	0.513
18	1.000	0.091	1.21	0.422
20	0.850	0.048	0.92	0.374
30	0.595	0.066	0.72	0.308
40	0.425	0.057	0.51	0.251
80	0.180	0.150	0.30	0.100
100	0.150	0.015	0.17	0.085
Pan	-	0.085	0.08	0.000

Gastonia, North Carolina) for 30 min.

The diameter ranges for various soil particles as reported by Black *et al.*^[37] are presented in Table 3. Based on particle size distribution (Table 4), the soil/sludge composition can be determined in terms of percentage of sands, silt and clay. The results indicate that most of the sludge is made up of very fine sand and clay.

The value of 48.7% of very coarse sand particles was not considered representative of the sample because the large clumps were formed during drying and could be broken to smaller particles with a relatively small finger pressure. When the sludge was mixed with water, the clumps did dissolve and it was, therefore, assumed that the sludge had a very high clay content.

Zeolite: The zeolites (Fig. 4) used as the ion-exchange medium is a Nova Scotian variety called Mountain



Fig. 4: Mountain stonach zeolite

Stonach. It was collected from Stonach Pit in Nova Scotia and had a Carbon Exchange Capacity (CEC) of 144 cmol kg⁻¹.

EXPERIMENTAL PROCEDURE

Salt solubility: The solubility of salts was determined at different temperature as shown in Table 5. The amount of water required (mL kg⁻¹ soil) for each salt type is shown in Table 6.

Sludge demineralization: A 500 g sample of sludge was placed in the sludge column and mixed for approximately 30 seconds. One L distilled deionized water (EC = 0 at room temperature) was then added by percolating up through the bottom of the sludge column. The sludge/water mixture was mixed at a constant speed (50 rpm) for 10 min. The water was then drained. Several washes were performed. After each wash, the wash water was centrifuged (International Equipments Company, Needham Heights, Massachusetts) for 40 min and then filtered under vacuum using Whatman Filter Paper (No. 40, Fisher Scientific, Montreal, Quebec). The solids were returned to the sludge column for the next wash. This process was continued until no salts were transferred from the sludge to the wash water (as indicated by constant electrical conductivity values). The samples obtained for the sludge and wash water were analyzed for salt concentration and the salt removal efficiency was calculated.

Table 5: Salt solubility

Salt	Solubility (g L ⁻¹)			
	0°C	10°C	20°C	100°C
CaCl ₂	595	650	745	1590
MgCl ₂	528	535	545	727
NaCl	357	358	360	391

Table 6: Water requirement for the sludge (at room temperature)

Parameter	NaCl	CaCl ₂	MgCl ₂
Amount of salt available (mg kg ⁻¹ soil)	8642.71	263.05	305.49
Solubility (mg mL ⁻¹)	360	545	745
Water requirement (mL kg ⁻¹ soil)	24	0.48	0.41



Where:

$$\text{Na} = 22.99 \text{ g mol}^{-1}$$

$$\text{Cl} = 35.45 \text{ g mol}^{-1}$$

$$\text{Ca} = 40.08 \text{ g mol}^{-1}$$

$$\text{Mg} = 24.31 \text{ g mol}^{-1}$$

Water desalination: To test the salt removal from the wash water, the zeolite was placed in the column and the wash water was filtered up through zeolite column until the water filled the entire column. The water was kept in the column for 20 min and was then drained. The salt concentration in the treated water was determined and the desalination efficiency was calculated.

RESULTS AND DISCUSSION

Sludge characteristics: The total amounts of measured and calculated salts are summarized in Table 7. The CaCl₂:MgCl₂:NaCl ratio was 1:1.16:36.61. The calculated total salt concentration was less than that obtained from the laboratory analysis by 1.2% which is acceptable analytical error. The electrical conductivity (EC) is a measure of the solution ability to conduct electricity which is related to the concentration of ions and their electrical charges.

The EC value was 42.2 dS m⁻¹ indicating a very saline sludge. The SAR value was 35.1 which is higher than industrial limit of 12 indicating high level of

sodicity. The Cation Exchange Capacity (CEC) is an important factor in determining the suitability of washing process for the removal of salts. The CEC for the saline sludge was 40 cmol kg⁻¹ which is considered to be high indicating that the soil texture is clay to clay loam^[26].

Sludge washing: Initially, three washes were performed on each sludge sample. Two liters of water were used in each wash for each kg of sludge. The results showed that the majority of the salt was removed in the first wash and no salt was removed in the third wash. The sludge water mixture took 36 h to completely settle.

The salt removed in each wash and the total removal efficiency results are presented in Table 8. The average salt removal efficiency was 94.47%. The treated sludge contained 515 mg salt kg⁻¹ sludge (or 0.05% w w⁻¹) and can be applied on agricultural land^[38].

Soil/sludge washing was implemented recently for removal of heavy metals (Cd, Co, Cr, Cu, Mn, Ni, Pb and Zn). Chemicals have also been added to the water to facilitate the washing process. These include: ethylenediaminetetraacetic acid (EDTA)^[39], ferric chloride^[40] and high-concentration chloride solutions (CaCl₂ and NaCl)^[41].

Andrade *et al.*^[39] investigated the effect of water to soil ratio (between 1:5-2:5) on the chelating efficiency of heavy metals (Cr, Cu, Ni, Pb and Zn) during washing process which required 24 h of settling time after 16 h of constant agitation at 125 rpm. Their results showed removal efficiency of 95.0-99.9%. Makino *et al.*^[40]

have reported two washes with a soil to water ratio of 2:3 and a settling time of around 24 h after each wash to attain optimal removal of Cd (up to 66%) from soil.

No study has been reported on sludge washing for desalination purpose. In this study, the quantity of fresh water required as well as overall process time (sludge-fresh water mixing and settling time) are within the range reported in the literature for soil washing. Thus, the results obtained from the present study would provide the basis for the development of a large scale sludge desalination process.

Desalination of wash water: The zeolite (Chabazite) in the ion-exchange column was tested using upflow percolation. The Chabazite (Ca₆[Al₁₂Si₂₄O₇₂].40H₂O) can only remove the Na ions. The results are shown in Table 9. A sodium removal efficiency of 75.25% was achieved.

Table 7: Mass balance on soluble salts

Salt	Concentration (mg kg ⁻¹)
NaCl	8642.71
Na	3400.00
Cl	5242.71
CaCl ₂	263.05
Ca	95.00
Cl	168.05
MgCl ₂	305.49
Mg	78.00
Cl	227.49
Total calculated	9211.25
Total lab analysis	9323.00
Error (%)	+1.20

Table 8: Salt removal from the sludge

Sample	Original salt (mg)	Salt removal (mg)			Removal efficiency (%)
		Wash 1	Wash 2	Total	
1	4661.50	2723.19	1680.40	4403.59	94.47
2	4661.50	2739.80	1660.92	4400.72	94.41
3	4661.50	2746.64	1660.50	4407.14	94.53
4	4661.50	2733.40	1670.60	4404.09	94.48
Average	4661.50	2735.78	1668.11	4403.89	94.47

Original soil sample = 500 g, Total salt concentration = 9323 g/kg soil

Table 9: Sodium removal from the wash water

Sample	Original salt (mg)	Final salt (mg)	Salt reduction (mg)	Removal efficiency (%)
1	400	98	302	75.50
2	400	100	300	75.00
3	400	97	301	75.25
4	400	99	301	75.25
Average	400	99	301	75.25

Average salt concentration = 2200 mg/L, Average sodium concentration = 800 mg/L, A removal efficiency of 99.79 % was obtained with a two column ion-exchange system.

These results suggest that a series of ion-exchange column (with Chabazite) will most likely be required to attain a higher removal efficiency of Na ions. When a two column ion-exchange system (with Chabazite) was used the Na removal efficiency increased from 75.25-99.79%.

Use of zeolites for water desalination/purification have been reported by many researchers^[42-44]. Caputo *et al.*^[42] reported reductions in contaminants of up to 99.99% using a series of zeolite columns depending upon type of zeolite, valance of salt cation and salt concentration. Zhao *et al.*^[44] reported 89.8% reduction of ammonium in municipal wastewater after repeated regeneration cycle in zeolites.

Utilization of zeolite columns in series for desalination of wash water obtained from the sludge washing process could be a plausible alternative to the cost intensive reverse osmosis and other membrane based processes. The sludge investigated in the present study was enriched in Na ions. Therefore, use of Na-free zeolite (Chabazite) was the logical approach for the removal of Na ions which resulted in a removal efficiency of sodium salt of 99.79% using two column ion-exchange system. However, the wastewater had a total salt concentration of 2200 mg L⁻¹ of which 2039 mg L⁻¹ is the sodium chloride (the remaining 161 mg L⁻¹ were in the forms of calcium and magnesium chlorides). Therefore, the overall salt removal efficiency will only be 92.9%. The treated water will contain 144.27 MgCl₂, 142.22 CaCl₂ and 126.52 mg L⁻¹ NaCl giving a total salt concentration of 314.0 mg L⁻¹. The salt concentration in the treated wash water is below the established Canadian limits^[7].

CONCLUSIONS

The particle size distribution of sludge sample indicated the presence of very fine sand and clay. The electrical conductivity of the sludge was 42.2 dS m⁻¹ indicating very saline sludge and the Cation Exchange Capacity (CEC) was 40 cmol kg⁻¹ which was very suitable for ion-exchange process. The desalination of the saline sludge by washing and removal of salt using zeolite based ion-exchange column was very effective.

Two washings of sludge using demineralized water achieved 94.47% salt removal efficiency and resulted in final salt concentration of 515 mg salt kg⁻¹ sludge (or 0.05% w w⁻¹). The treated sludge can be applied on agricultural land as per the Canadian Guidelines. The ion-exchange treatment of the wash water was also very effective. A single column with zeolite (Chabazite) achieved 72.25% Na removal efficiency while the two

column ion-exchange system achieved Na removal efficiency of 92.96%.

The Ca and Mg ions were under regulatory limits and required no further treatment. The final salt concentration in the wash water was 314.0 mg L⁻¹ which is below the limits established by the Canadian Guidelines. For complete removal of total salts, a series of ion-exchange columns with multiple zeolites (for removal of NaCl, MgCl₂ and CaCl₂) will be required.

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