SOIL STRUCTURAL CHANGES FOLLOWING IRRIGATION WITH A POTASSIUM RICH WINERY WASTEWATER

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Abstract

Research on the soil structural effects of potassium (K^+) in irrigation wastewaters has received limited attention due to the typically low abundance of K^+ in most waters. Winery processing wastewater however tends to have elevated concentrations of both K^+ and sodium (Na⁺). This study investigated the contrasting cation dynamics under municipal and winery wastewater following irrigation to a heavy clay soil. Under laboratory conditions, accelerated annual irrigation, drying and rainfall cycles enabled temporal changes to be investigated.

Despite the higher Na^+ concentration in winery wastewater, irrigation caused a significant increase in the exchangeable potassium percentage of the soil indicating preferential retention of K⁺ over Na⁺. Although clay dispersion was evident in soils irrigated with both wastewaters, importantly, this was greater under municipal wastewater due to the higher dispersive potential of Na⁺ over K⁺. Soils receiving municipal wastewater also required a greater electrolyte concentration to maintain flocculation. It is thought precipitation and solubilisation of inorganic carbon during irrigation, drying and rainfall cycles, plays an important role in buffering a dramatic decrease in drainage electrolyte concentration otherwise expected during rainfall cycles. Furthermore changes in soil pH_(1:5) and effective cation exchange capacity (CEC_e) associated with high bicarbonate loading appear to favour retention of monovalent cations, thereby influencing clay dispersion.

Introduction

In Australia, wastewater from many sources, including winery wastewater, are reclaimed for irrigation. In South Australia for instance, both winery and municipal wastewaters are distributed and sold to growers for irrigating grape vines. Both waters are rich in Na⁺ and, in the case of winery wastewater, K⁺ concentration is also high due to waste from grape lees, spent juice and potassium hydroxide (KOH) cleaners (Kumar & Christen, 2009). A high concentration of monovalent Na⁺ in soils has often shown to disrupt soil structure, leading to changes in many key soil physical properties such as hydraulic conductivity, infiltration rate, bulk density and soil aeration (e.g. Rengasamy and Olsson 1991; Halliwell *et al.* 2001; Menneer *et al.* 2001; Stevens *et al.* 2003). Based on the large hydrated ion size and its affinity to clay minerals, high levels of exchangeable K⁺ in soil also has the potential to cause clay swelling and dispersion (Levy and Feigenbaum 1996). Research on the soil structural effects of K⁺ in irrigation wastewaters has however received less attention due to the typically low abundance of K⁺ in most wastewaters (Arienzo et al. 2008).

In arid regions, higher bulk density and lower infiltration rates typical of many B horizon soils often restricts the migration of salts down the soil profile. These B horizon soils are therefore more susceptible to soil dispersion relative to those of the A horizon due to an accumulation of salt at the interface between A and B horizons (Soil and Land Program 2007). This is particularly apparent during winter rainfall when soil electrolyte concentration decreases dramatically (Suarez et al. 2008). The composition of the drainage water, including that resulting from both irrigation and rainfall, entering the B horizon will differ greatly from that applied to the soil surface due to a number of biological, chemical and physical processes occurring during passage through the A horizon. This experiment was therefore carried out in two stages. Initially, changes in the composition of drainage within the A horizon was investigated from soil irrigated with municipal and winery wastewater irrigation. This was followed by a subsequent experiment to determine the influence of these two drainage waters on the structure of B horizon soils, particularly in relation to the temporal changes between winter and summer drainage i.e. irrigation and rainfall.

Methods

Irrigation of the A Horizon

The objective of this experiment is to determine the effect of municipal and winery wastewater irrigation on the chemical composition of drainage from soils of the A horizon. Sixteen intact columns (150 mm length x 100 mm width) of a duplex Luvisol soil (FAO, 2006) were collected from a depth of 0-150 mm in the soil profile at the Viticultural Research Station in Nuriootpa, Barossa Valley, South Australia. The A horizon soils were well structured and approximately 150 to 200 mm deep before a sharp transition to a B horizon. This profile is a good representation of soils widely used for viticulture in the Barossa region where winery and municipal wastewater are used for irrigation. A fibre-glass wick (600 mm) was mounted at the base of each column to maintain a fixed tension (approximately -60 kPa) thereby create a 'hanging soil-water column' similar to field conditions.

Winery and municipal wastewater was sourced from Wastewater Treatment Plants in Barossa Valley and was applied to soil columns at a depth of 60 mm per day for ten days and then allowed to drain for 4 days before applying a low ionic water solution, akin to rainfall, at a rate of 60 mm per day for 2 days. This cycle was repeated 9 times in continuous fashion over the course of six months. Care was taken to balance the time over which irrigation was applied each day, typically 8 hours, with the period of free draining, 16 hours, to ensure adequate diffusion of soluble ions throughout the soil matrix. For each treatment and replicate, a bulk sample of soil drainage was collected every 5 days of consecutive wastewater irrigation, approximately 8 pore volumes (p.v.), and every 2 days following rainfall irrigation (approximately 3.2 pore volumes). Analysis of basic cations was carried out on filtered (0.45 μ m) samples using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). Dissolved inorganic carbon (DIC) was measured on filtered samples (0.45 μ m) using a Wet Oxidation Total Carbon Analyser (O.I. Analytical).

Physico-chemical processes in B horizon soils

The objective of this experiment was to determine structural changes in B horizon soils irrigated with municipal or winery wastewater followed by the application of rainwater.. From the same soil profile, a further series of soils were collected from the B horizon at a depth of 600 mm, approximately 100 mm below the point of transition between the A-B horizon interfaces. In the laboratory, these soils were air dried by force draft oven at 40° C for 48 hours then sieved through 2 mm mesh. These B horizon soils were then equilibrated with drainage waters collected from the A horizon where either winery or municipal wastewater

had been irrigated. For each irrigation treatment (i.e. municipal and winery wastewater), drainage water previously collected from the A horizon at the end of each irrigation cycle was bulked together to give a single sample. In similar fashion, drainage during rainfall cycles were also bulked together to give a single rainfall sample for each of the irrigation treatments.

Soils were initially equilibrated with wastewater drainage then dried prior to being equilibrated with rainfall drainage. This was achieved by placing 200 gram of soil in a 2 L plastic container with the corresponding drainage water, inverted gently three times to ensure adequate mixing of soil and solution then left for 48 hours. Soils were then placed on filter paper (Whatman 42, 2.5 μ m) for 24 hours during which time the solution was drained. Evaporation losses that may otherwise alter the chemical concentrations of the equilibration solutions were reduced by keeping soils covered with plastic wrap. This pre-treatment process was repeated a total of three times over the course of nine days. Soils were then left to drain for approximately 48 hours. Once drained, all soils were air dried at 40°C for 48 hours then again sieved gently through 2 mm mesh.

To investigate the effect of rainfall drainage on soil dispersion, B horizon soils previously equilibrated with winery wastewater or municipal wastewater drainage were subsequently equilibrated with rainfall drainage, diluted with deionised water by 25, 50 or 75 %. This essentially gave four solutions of similar sodium adsorption ration (SAR) and potassium adsorption ratio (PAR) concentration yet decreasing electrolyte concentration for each of the two irrigation types. This emulates changes in soil water chemistry likely to occur in the B horizon during rainfall cycles of varying intensities and subsequent dilutions of the soil-water solution.

Dispersion measurements were determined based on the technique of Rengasamy (2002). All measurements were carried out in duplicate and under a constant temperature of 24°C. Here, a 20 gram soil sample was placed into a 120 mL transparent container (100 mm in height) and 100 mL of the corresponding rainfall drainage was added. Sealed containers were gently inverted four times to ensure adequate mixing of soil and solution and left overnight to equilibrate with the rainfall drainage water. Following this equilibration period, containers were again gently inverted four times before being left for an appropriate sedimentation time based on Stokes Law, where a clay particle with equivalent spherical diameter of 2 μ m and particle density of 2.61 g cm⁻³ falls 50 mm in water of 24°C (Rengasamy 2002).

Following sedimentation, 10 mL (\pm 0.1 mL) of the clay suspension was removed from a depth of 50 mm using a syringe specially modified with an upward pointing inlet tube. This enabled the suspension to be drawn downward through the column of settling clay suspension so that settled soil fractions at the base of the container were not disturbed. Dispersed clay in suspension was then quantified by measuring optical density at 641 nm (Thellier and Sposito 1989) and converting to mass concentration (grams per litre) based on a standard curve of clay concentration against optical density. Immediately prior to photospectroscopic determination, each 10 mL suspension was inverted 10 times.

Exchangeable cations were determined on B horizon soils by adding 20 mL of 0.1 M $BaCl_2/0.1$ M NH_4Cl to 2.0 g of soil (Gillman & Sumpter, 1986), shaking end-over-end for two hours, then centrifuging at 3450 g for 10 minutes. The supernatant was filtered (0.45 µm, Millipore) and analysed for total cations using ICP-OES. The CEC_e was determined as the sum of Ca²⁺, Mg²⁺, K⁺ and Na⁺. The sodium adsorption ratio (SAR) of the wastewaters is

defined as Na/(Ca+Mg)^{0.5} where the concentration of Na, Ca and Mg is in mmol_c per litre, while the potassium adsorption ratio (PAR) is defined as $K/(Ca+Mg)^{0.5}$.

Results

The composition of the two wastewaters and rain water applied to the soil A horizon are shown in Table 1. This shows that both wastewaters have a similar composition of monovalent cation but vary in their Na : K ratio's, being 13 : 1 and 2 : 1 for municipal and winery wastewater respectively. The electrical conductivity (EC) and pH of the two wastewaters prior to their application to soils is similar. Relative to the wastewaters, rainwater has a substantially lower in EC and pH and the cation and carbon composition is also lower.

Chemical parameter	Municipal wastewater	Winery wastewater	Rainwater	
pН	8.41 ±0.28	8.54 ±0.33	6.52 ±0.19	
$EC (dS m^{-1})$	1.70 ±0.14	1.38 ±0.11	0.13 ± 0.01	
	$(mmol_c L^{-1})$			
Ca^{2+} Mg^{2+} Na^+	1.05 ± 0.24	1.61 ±0.26	0.24 ± 0.09	
Mg^{2+}	1.50 ±0.11	0.89 ± 0.08	0.20 ± 0.07	
Na ⁺	$13.64 \hspace{0.1cm} \pm 1.09$	7.50 ± 0.61	0.72 ± 0.17	
\mathbf{K}^+	1.19 ± 0.07	3.94 ±0.24	0.04 ± 0.02	
DIC^\dagger	2.78 ±0.36	2.19 ± 0.24	0.20 ± 0.09	
	$(mmol_c L^{-1})^{0.5}$			
SAR^1	12.2	7.0	1.7	
PAR^{2}	1.1	3.8	0.1	

Table 1. Chemical composition of municipal and winery wastewater and rainwater

[†] Dissolved inorganic carbon. ¹SAR (sodium adsorption ratio) = $Na^{+}/(Ca^{2+}+Mg^{2+})^{0.5}$.

Concentration of Na⁺, Ca²⁺ and Mg²⁺ is in mmol_c L^{-1} . ²PAR (potassium adsorption ratio) = K⁺/(Ca²⁺+Mg²⁺)^{0.5}. Concentration of K⁺, Ca²⁺ and Mg²⁺ is in mmol_c L^{-1} .

Irrigation of the A Horizon

The volume of drainage collected from the base of the A horizon in the column experiment was similar to that which was applied and generally drainage pH was also similar, suggesting pH buffering capacity of this soil is generally low. Notably drainage pH under both municipal and winery wastewater did not decrease greatly during rainfall cycles, despite the considerably lower pH of rainwater. This indicates a net loss in alkalinity during rainfall that buffers changes in soil pH. Given the apparently low buffering capacity of the soil, changes in net alkalinity would presumably coincide with a decrease in soil pH_(1:5), suggesting continual change in soil $pH_{(1:5)}$ may be occurring in response to irrigation and rainfall cycles.

A net reduction in the electrolyte concentration was apparent following the nine irrigation and rainfall cycles. The EC of soil drainage during irrigation cycles was similar to that of the input solution throughout the nine irrigation cycles. Importantly however, as with drainage pH, drainage EC was substantially greater during rainfall cycles that input rainwater and therefore indicates a net reduction in the electrolyte concentration during these cycles. This illustrates the importance of a 'leaching fraction' achieved under regular rainfall cycles or irrigation with good quality water in mitigating high soil EC, in particular where highly saline irrigation waters such as municipal and winery wastewater are used.

Parameter	Winery wastewater drainage		Municipal wastewater drainage		
	irrigation	rainfall	irrigation	rainfall	
рН	8.62	8.49	8.09	8.23	
$EC (dS m^{-1})$	1.39	0.88	1.82	1.00	
Ca^{2+} (mmol _c L ⁻¹)	0.97	0.30	0.93	0.31	
Ca^{2+} (mmol _c L ⁻¹) Mg ²⁺ K ⁺	0.42	0.03	0.59	0.09	
K ⁺	2.02	1.22	0.64	0.37	
Na ⁺	4.25	2.85	7.68	4.29	
DIC^\dagger	5.73	4.04	5.76	3.77	
$SAR^{1} (mmol_{c} L^{-1})^{0.5}$	5.09	7.32	8.79	10.93	
PAR ²	2.42	3.14	0.74	0.93	

Table 2. Chemical composition of drainage waters collected from the base of the A horizon during irrigation with either municipal or winery wastewater each of which was followed by application of rainfall.

[†]Dissolved organic carbon.

¹SAR (sodium adsorption ratio) = Na⁺/(Ca²⁺+Mg²⁺)^{0.5}. Concentration of Na⁺, Ca²⁺ and Mg²⁺ is in mmol_c L⁻¹. ²PAR (potassium adsorption ratio) = K⁺/(Ca²⁺+Mg²⁺)^{0.5}. Concentration of K⁺, Ca²⁺ and Mg²⁺ is in mmol_c L⁻¹.

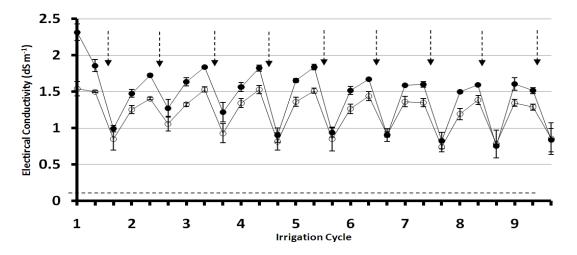


Figure 1. Electrical conductivity (EC) of drainage solutions from A horizon soils irrigated with municipal wastewater (•) and winery wastewater (•). Regular applications of rainfall included in each irrigation cycle are indicated by a dashed-downward (\downarrow) arrow. Error bars represent the standard deviation between replicates. Average EC of the input solutions was: 1.7 and 1.4 dS m⁻¹ for municipal and winery wastewater respectively (Table 1). The EC of input rainfall was 0.1 dS m⁻¹ and is indicated as a dashed line (—).

Dissolved inorganic carbon (DIC) dynamics

Irrigation with municipal and winery wastewater resulted in an accumulation of DIC while rainfall caused a decline (Table 3). Soil drying has been shown to encourage formation of carbonate precipitates in arid soils irrigated with either water or wastewater (Wu *et al.* 2008; Eshel *et al.* 2007; Levy 1980; Lal 2002). As soils dry, DIC in solution is concentrated and at high soil pH (as is the case under municipal and winery wastewater) and adequate exchange of CO_2 with the atmosphere, the formation of CO_3^{2-} precipitates tends to occur (Stumm and

Morgan 1996). This invariably involves in the subsequent precipitation of a conjugate cation, typically Ca^{2+} or Mg^{2+} (Suarez *et al.* 2008).

One major implication resulting from the solubilisation of CO_3^{2-} is a net production of alkalinity in the form of hydroxide ions with the resulting effect of increasing soil pH. This process has been observed in fields amended with liming materials (e.g. CaCO₃) (Davis and Burgoa 1995; West and McBride 2005; Bolan *et al.* 2003b) and in drying environments where alkaline water is used for irrigation (Artiola and Walworth 2009; Entry *et al.* 2004; Gardner 2004; Johnson *et al.* 1979; Khokhlova *et al.* 1997; Wu *et al.* 2008).

Table 3. Cumulative gain and loss $(mmol_c)$ of dissolved inorganic carbon (DIC) during irrigation and rainfall cycles. Values preceded by a minus sign (-) indicate a loss from the soil system, while a positive sign (+) indicates a gain. The standard deviation (S.D.) is shown after each mean value.

	Irrigation	Rainfall	Δ -inorganic carbon
Winery	$+ 82.2 \pm 8.4$	- 24.2 ±2.4	+ 58.0
Municipal	$+45.2 \pm 1.8$	-23.4 ± 1.9	+21.8
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The balance of DIC between input and output solutions was ascribed a delta (Δ) value (i.e. change in carbon), where $\Delta DIC = (\text{input volume } (L) \text{ x concentration } (\text{mmol}_c L^{-1})) - (\text{drainage volume } (L) \text{ x concentration } (\text{mmol}_c L^{-1}))$. A change in the delta value is indicative of either a gain or loss within the soil-plant system.

During rainfall cycles a net loss of DIC was observed. This coincided with a net increase in drainage pH and EC and is likely to be associated with the solubilisation of $CO_3^{2^-}$ that has previously precipitated. Lowering of soil pH and decreasing ionic strength of soil solution encourages reversal of the DIC equilibrium whereby mobile HCO_3^- anions are formed (Bolan and Hedley 2003; Bolan *et al.* 2003a). A number of studies have shown a close link between DIC precipitation and its dissolution with wetting and drying cycles (Eshel *et al.* 2007; Khokhlova *et al.* 1997). Where municipal or winery wastewater is used for irrigation, DIC may continue to accumulate in soils during the irrigation season however will decrease during winter rainfall if both soil pH and ionic strength of the soil solution are lowered.

Changes in soil chemical properties

As described in the materials and methods, B horizon soils were initially equilibrated with drainage waters from the A horizon where municipal wastewater or winery wastewater had been irrigated. The chemical characteristics of the B horizon soils prior to, and after equilibration with wastewaters are shown in Table 4. In all cases, $pH_{(1:5)}$ of the treatment soils were consistently greater than the $pH_{(1:5)}$ of the non-irrigated (control) soil and a significant increase in CEC_e was also evident.

A notable trend with increasing CEC_e was an associated binding of certain cations present in soil solution. In B horizon soils equilibrated with winery wastewater drainage, exchangeable K^+ was nearly twice that of exchangeable Na^+ despite the higher Na^+ in the drainage water. Correspondingly the exchangeable K^+ percentage (EPP) increased from 7 to 18 %. Only moderate increases in exchangeable Na^+ percentage (ESP) were evident. It is hypothesised that preferential binding of K^+ over Na^+ , apparent under winery wastewater, may restrict increases in ESP. This suggests the sodicity risk associated with winery wastewater irrigation is reduced in the presence of K^+ . Potassium can impact on the structural stability of soils when EPP is high, importantly however, the relative effect of K^+ on clay dispersion is likely to be less severe than Na^+ .

In B horizon soils equilibrated with municipal wastewater drainage, the reverse was true whereby exchangeable Na^+ was twice that of exchangeable K^+ and ESP increased from 7 to

18 %. This exceeds trigger values beyond which structural decline in Australian soils is thought to become evident i.e. 6 % (Northcote and Skene 1972). Under both municipal and winery wastewater drainage, exchangeable Ca^{2+} and Mg^{2+} were similar as was the portion of cumulative monovalent cations that comprised the CEC_e.

Table 4. Soil chemical characteristics of the non-irrigated (initial) B horizon soil and soils equilibrated with drainage water from the A horizon where municipal or winery wastewater has been irrigated.

	Initial	Soils irrigated with Winery wastewater	Soils irrigated with Municipal wastewater
pH _(1:5)	6.81	7.37	7.26
EC _(1:5)	0.123	0.193	0.267
CEC _e (cmol _c kg ⁻¹ soil)	17.39	19.95	19.98
Ca ²⁺ Mg ²⁺ K ⁺	10.85	10.58	10.30
Mg^{2+}	4.04	3.45	4.20
\mathbf{K}^+	1.22	3.67	1.86
Na ⁺	1.28	2.25	3.62
ESP	7.37	11.26	18.14
EPP	7.03	18.42	9.30

A high electrolyte concentration, as measured by high EC, in soils helps to mitigate the effects of soil dispersion. Under field conditions, percolation of rainwater will dilute soil solution EC thereby increasing the risk of soil dispersion (Suarez *et al.* 2008). This is likely to be most evident in soils with high monovalent cation concentrations. In this study, the dispersion of the B horizon soils equilibrated with winery or municipal wastewater in response to rainfall drainage waters from the A horizon was investigated. The two rainfall drainage waters are distinguished as winery-rain and municipal-rain. These solutions were diluted by various fractions to give a declining EC concentration (Table 5).

Equilibration solution	Dilution factor	ESP	EPP	pН	EC	Dispersed clay	
		(%)	(%)		$(dS m^{-1})$	(g kg ⁻¹ soil) [#]	(% total clay)
Winery rain	0	11.2	19.3	8.61	0.848	77.4 ±14.0 a B	12.7
	25	11.4	19.9	8.56	0.660	70.7 ±12.8 a A	11.6
	50	10.0	18.5	8.56	0.459	63.9 ±14.5 a A	10.5
	75	10.5	18.9	8.33	0.229	111.4 ±8.5 b B	18.2
Municipal rain	0	19.0	9.9	8.73	0.978	83.1 ±2.8 a B	13.6
	25	18.8	9.7	8.67	0.738	111.2 ±19.6 a B	18.3
	50	18.3	10.6	8.63	0.544	$186.9 \hspace{0.2cm} \pm 8.5 \hspace{0.2cm} b \hspace{0.2cm} B$	30.6
	75	17.4	9.8	8.56	0.312	266.4 ±22.0 c C	43.6

Table 5. Clay dispersion of a Barossa B-horizon soil, previously irrigated with winery or municipal wastewater, in response to rainfall drainage. Soil $pH_{(1:5)}$ and $EC_{(1:5)}$ are determined from the clay suspension following determination of dispersed clay.

[#]The standard deviation (S.D.) between replicates is shown after the average values. Within each water type, values followed by similar lower case letters are not significantly different (P<0.05). Between water types, for each dilution factor, values followed by similar upper case letter are not significantly different (P<0.05).

In the case of soils irrigated with winery wastewater, greater K^+ retention lead to an increase in clay dispersion, especially at low EC (Table 5; Figure 3). This was however lower than soils irrigated with municipal wastewater due to the greater dispersive potential of Na⁺ relative to K⁺. Although dispersion decreased with increasing EC in both winery- and municipal wastewater-irrigated soils, the latter required a higher electrolyte concentration to maintain flocculation (Figure 2). A higher salt content to preserve a flocculated state is undesirable due to its impact on plant growth and microbial activity of the soil.

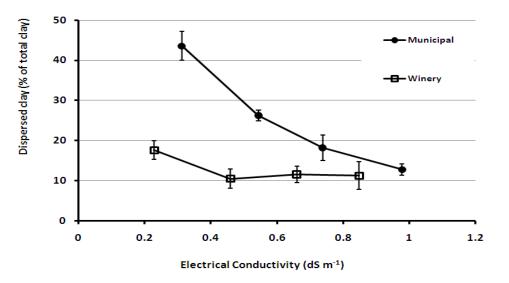


Figure 2. Dispersion of B horizon soils equilibrated with A horizon drainage where municipal or winery wastewater has been irrigated and subsequently equilibrated with rainfall drainage of varying dilutions (0, 25, 50 and 75 % dilutions differing in electrical conductivity (EC) value) to provide contrasting solution salinity concentrations. Error bars indicate the standard deviation between replicates.

Conclusions

Irrigation of municipal wastewater introduces a considerable amount of Na^+ to the soils, while under winery wastewater, both Na^+ and K^+ loading is high. The retention of K^+ however is far greater than for Na^+ , and therefore under winery wastewater irrigation, EPP increases more than ESP.

Based on this series of experiments, it is hypothesised that high evapotranspiration rates in summer concentrate salts in soils irrigated with wastewater, causing the SAR, and in the case of winery wastewater PAR of the soil solution to increase. Where the SAR of the wastewater is high a subsequent increase in ESP will occur. When the PAR of the wastewater is high the retention of Na^+ is limited by the presence of K^+ and therefore only EPP increases. The high EC of winery and municipal wastewater enables soils to remain flocculated during irrigation.

During winter, a rapid decline in soil solution EC occurs. Dissolution of inorganic carbon is able to raise the EC of the percolating drainage water to a concentration that prevents soil dispersion. Dilution of soil water brought about by high rainfall may however lower drainage EC. The likelihood of clays to disperse in response to K^+ is considerably lower relative to Na⁺ therefore, although accumulation of monovalent cations in soils is not desirable, high exchangeable K^+ is less detrimental that an equivalent concentration of exchangeable Na⁺ on soil structure. Furthermore, soils with high EPP (i.e. under winery wastewater irrigation) require lower EC concentrations to maintain a state of flocculation relative to soils with high

ESP (i.e. those irrigated with municipal wastewater). Essentially this study indicates that heavy rainfall may lead to dispersion in soils irrigated with municipal wastewater while those irrigated with winery wastewater will remain flocculated until soil-water has been significantly diluted, in this case by approximately 75 %.

On a practical level, the importance of maintaining a low soil ESP is apparent, particularly in clay soils that have greater propensity to disperse than sandy soils. In areas with dry summers followed by high rainfall in winter, clay dispersion may occur where municipal wastewater is used for irrigation due to a coinciding of high ESP and low EC. This process will be less apparent under winery wastewater irrigation due to the lower ESP and relatively lower effect of EPP on clay dispersion.

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