Effect of Irrigation with Diluted Winery Wastewater on Phosphorus in Four Differently Textured Soils

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The wine industry needs solutions for wastewater treatment, as environmental legislation for its disposal is increasingly being enforced due to non-compliance. The feasibility of re-using diluted winery wastewater was assessed in a pot experiment under a rain shelter over four simulated irrigation seasons. Four soils varying in parent material and clay content, *viz.* aeolic sand from Lutzville containing 0.4% clay, alluvial sand from Rawsonville containing 3.3% clay, granite-derived soil from Stellenbosch containing 13% clay, and shale-derived soil from Stellenbosch containing 20% clay, were irrigated with wastewater diluted to 3 000 mg/L COD (chemical oxygen demand), whereas the control received municipal water. Irrigation with diluted winery wastewater increased the $pH_{(KCl)}$ in the shale- and granite-derived soils into the optimum range for P availability. Although $pH_{(KCl)}$ in the aeolic sand was initially above the optimum range, relatively high Na^+ levels also caused available P to increase as the $pH_{(KCl)}$ increased. The $pH_{(KCl)}$ in the alluvial sand increased beyond the optimum range, thereby causing a reduction in the available P. This indicates that irrigation with diluted winery wastewater may only enhance P absorption if the $pH_{(KCl)}$ shift is towards the optimum. It must be noted that the results represent a worst-case scenario, *i.e.* in the absence of rainfall or crops.

INTRODUCTION

Wineries generate large volumes of wastewater, and increased wine production in South Africa is exerting more pressure on natural resources. In many cases, winery wastewater is not suitable for the irrigation of agricultural crops (Mulidzi *et al.*, 2009). Due to the intensification of environmental legislation (Department of Water Affairs, 2013), the wine industry is expected to find solutions for the treatment or re-use of winery wastewater (Van Schoor, 2001). This initiated the development of guidelines for the management of wastewater and solid waste at wineries (Van Schoor, 2005). A shortage of good-quality water leads to an increasing demand to irrigate with poor-quality water such as saline groundwater, drainage water and treated wastewater (Jalali *et al.*, 2008). The negative effects of re-using untreated municipal and other industrial wastewater for agricultural irrigation are well documented (Bond, 1998; Papini, 2000; Mulidzi, 2001; Arienzo *et al.*, 2009; Christen *et al.*, 2010; Laurenson *et al.*, 2012; Laurenson & Houlbrooke, 2011; Mosse *et al.*, 2011; Arienzo *et al.*, 2012). The impact of irrigation with treated wastewater on the environment has not been investigated widely, although the demand for the use of sewage effluent due to water shortages is increasing (Walker & Lin, 2008). The disposal of wastewater through land application has been practised for many years by the majority of wineries (Mulidzi, 2001; Laurenson & Houlbrooke, 2011). Effective disposal of wastewater through re-use depends on the irrigation technology, as well as on soil properties (Oron *et al.*, 1999). Furthermore, a study carried out in some of the South African grape-growing regions confirmed that the impact of using undiluted winery wastewater for irrigation differed substantially between soils (Mulidzi, 2001).

Most of the previous studies and reports focused on the negative effects of irrigating with winery wastewater on soil physical and chemical status (Bond, 1998; Arienzo *et al.*, 2009; Christen *et al.*, 2010; Mosse *et al.*, 2011; Arienzo *et al.*, 2012; Laurenson *et al.*, 2012; Howell & Myburgh, 2014). However, it has also been shown that re-using wastewater for irrigation and as fertiliser has positive effects, such as pH increases, water- and mineral salt-retention characteristics, as well as restoration and maintenance of soil micro-flora (Papini, 2000). It was also proposed that re-using potassium-rich wastewater could enhance soil fertility (Mosse *et al.*, 2011). In this regard it is possible that phosphorus (P) applied via irrigation with winery wastewater could contribute to the nutrient requirements of agricultural crops. The solubility of phosphate (PO_4^{3-}) compounds, or P

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availability to plants (P is adsorbed by plants in the ionic form $H_2PO_4^-$), strongly depends on the soil pH (Sharpley *et al*., 1988; Conradie, 1994; Busman *et al*., 2002; Devau *et al*., 2009). In acidic soils, particularly where pH (water) is less than 5.5, aluminium (Al^{3+}) and iron (Fe^{3+}) will react with PO_4^{3-} to form amorphous phosphates (Busman *et al*., 2002). The amorphous Al^{3+} and Fe^{3+} phosphates gradually change into insoluble PO_4^{3-} compounds that are not available to plants. Phosphate also becomes increasingly insoluble if the soil $pH_{(KCl)}$ exceeds 7 (Conradie, 1994; Busman *et al*., 2002). In alkaline soils, *i.e.* pH > 7, calcium (Ca^{2+}) is the dominant cation that will react with PO_4^{3-}, to form a general sequence of calcium phosphates, *viz.* dibasic calcium phosphate dihydrate, octocalcium phosphate and hydroxyl apatite (Busman *et al*., 2002). The formation of each of these compounds decreases the solubility of phosphate. On the other hand, PO_4^{3-} solubility can also increase in high pH soils when exchangeable sodium (Na^+) releases inorganic PO_4^{3-} (Sharpley *et al*., 1988). When Na^+ replaces exchangeable Ca^{2+}, Mg^{2+} and Al^{3+}, the negative potential of the surface increases, which results in the desorption of PO_4^{3-} (Naidu & Rengasamy, 1993). It has also been reported that water-soluble PO_4^{3-} increases as Na^+ saturation increases in alkaline soils (Abrol *et al*., 1988 and references therein). Similar to the effect of exchangeable Na^+, soluble P increased to above pH 7 when a silty clay soil was alkalinised with potassium hydroxide (KOH) (Devau *et al*., 2009). A number of field and laboratory studies have shown that irrigation with winery wastewater increases soil pH, particularly if the water contains high levels of potassium (K^+) and Na^+ (Lieffering & McLay, 1996; Papini, 2000; Mulidzi, 2001; Laurenson *et al*., 2010; Laurenson & Houlbrooke, 2011; Mulidzi *et al*., 2015). However, it was also reported that the opposite effect on soil pH is possible (Bueno *et al*., 2009). Therefore, the extent to which the P applied *via* winery wastewater can be absorbed by plants will depend indirectly on the effect of the wastewater irrigation on the soil pH. However, this does not rule out the possibility that applied P may be lost by leaching during winter rainfall.

The objective of the study was to determine the effect of irrigation with diluted winery wastewater on P in four differently textured soils.

MATERIALS AND METHODS

Experiment layout

Four pedogenetically different soils from three grape-growing regions in the Western Cape were included in the study, *viz.* (i) aeolic sand from Lutzville containing 0.4% clay, (ii) alluvial sand from Rawsonville containing 3.3% clay, (iii) granite-derived soil from Stellenbosch containing 13% clay, and (iv) shale-derived soil from Stellenbosch containing 20% clay. The properties and characteristics of the soils were presented previously (Mulidzi *et al*., 2016). Bulked soil samples were collected from the topsoil layers, *i.e.* approximately 0 to 30 cm deep. Soils were packed into 3.54 dm^3 PVC pots to a bulk density of 1 400 kg/m^3, as described by Mulidzi *et al.* (2016). The pot experiment was carried out under a 20 m x 40 m translucent fibreglass rain shelter at ARC Infruitec-Nietvoorbij near Stellenbosch. Control treatments of all soils were irrigated with water supplied by the Stellenbosch municipality. The winery wastewater treatments were irrigated with wastewater diluted to a chemical oxygen demand (COD) of 3 000 mg/L, as described by Mulidzi *et al.* (2016). The undiluted wastewater was obtained from the wastewater collection pit at a winery near Rawsonville and transported in a tank to Stellenbosch. Treatments were applied over four simulated irrigation seasons consisting of six irrigations each. Consequently, a total of 24 irrigations were applied over the four simulated seasons. Details of the pot experiment layout and wastewater dilution procedure, as well as the irrigation system and irrigation scheduling, were presented earlier (Mulidzi *et al*., 2016).

Analyses

Water samples were collected and analysed prior to each irrigation. Phosphorus was not determined in the irrigation water. Triplicate soil samples were collected from the bulked soil to determine the initial soil chemical status. Due to destructive soil sampling, each experimental "plot" consisted of four pots. Following each simulated irrigation season, the soil in one of the pots was sampled, *i.e.* after six, 12, 18 and 24 irrigations. Soil samples were taken from the 0 to 10 cm and 10 to 20 cm layers in the pots of all replications. Soil samples were air dried and passed through a 2 mm mesh sieve. The P and $pH_{(KCl)}$ were determined by a commercial laboratory (BEMLAB, Strand). Phosphorus was determined according to the Bray No. 2 method, *viz.* extraction with 0.03 M NH_4F (ammonium-fluoride) in 0.01 M HCl (hydrochloric acid). The P concentration in the extract was determined by inductively coupled plasma optical emission spectrometry (ICP-OES) using a spectrometer (PerkinElmer Optima 7300 DV, Waltham, Massachusetts). The $pH_{(KCl)}$ was determined in a 1 M potassium chloride (KCl) suspension.

Statistical procedures

Each soil/water treatment was replicated four times in a completely randomised design. The four soils were randomly allocated within each block. The treatment design was a split-plot, with soil type as the main plot factor and soil depth as the sub-plot factor. Analyses of variance were performed separately for each season using SAS version 9.2 (SAS, 2008). The Shapiro-Wilk test was performed to test for non-normality (Shapiro & Wilk, 1965). Student's "t" least significant difference (LSD) was calculated at the 5% significance level to facilitate comparison between treatment means (Ott, 1998).

RESULTS AND DISCUSSION

Some soil physical characteristics, as well as cation and $pH_{(KCl)}$ responses of the four soils to irrigation with diluted winery wastewater, were presented previously (Mulidzi *et al*., 2015; 2016). The irrigation amounts applied to the Rawsonville sand, Lutzville sand and Stellenbosch shale soil were comparable (Table 1), but the Stellenbosch granitic soil received substantially less water due to its coarse texture and high gravel content (Mulidzi *et al*., 2016). During the period that the pot experiment was carried out, the P content in the wastewater obtained from the same winery near Rawsonville, diluted to a COD of 3 000 mg/L, was 4.8 ± 1.6 mg/L (Howell & Myburgh, 2014). The amount of

TABLE 1
Irrigation volume during four simulated seasons in which four different soils were irrigated with diluted winery wastewater.

	Irrigation applied (mm/season)				
Soil	**Season 1**	**Season 2**	**Season 3**	**Season 4**	**Total**
Rawsonville sand	291	289	287	289	1156
Lutzville sand	281	282	282	281	1126
Stellenbosch shale	246	250	246	245	987
Stellenbosch granite	181	180	184	183	728

P applied during irrigation of a field experiment that was irrigated with the diluted wastewater was 1.3 ± 0.4 kg P per hectare per irrigation. Based on these results, the annual application would amount to 9.4 ± 2.6 kg P per hectare per year if six diluted wastewater irrigations were applied. This indicates that the annual amount of P applied *via* the diluted wastewater was relatively small.

The initial P contents were 217 mg/kg, 6 mg/kg, 8 mg/kg and 15 mg/kg in the Rawsonville sand, Lutzville sand, Stellenbosch shale and Stellenbosch granite soils respectively. With the exception of the Rawsonville sand, P contents in the soils were in line with values normally expected for vineyard soils (Conradie, 1994). The initial P levels in the Rawsonville sand were more than tenfold the maximum of 20 mg/kg recommended for grapevines in soils containing less than 6% clay (Conradie, 1994). This was also more than double the P level at which wheat yields were reduced in a red, sandy soil near Vaalharts (Eloff & Laker, 1978). It was previously reported that P levels could range between 10 and 400 mg/kg for the duplex and gradational soils in Australia (Naidu & Rengasamy, 1993 and references therein). The foregoing confirms that high levels of P are not uncommon in agricultural soils. Irrigation with municipal water had a minimal effect on the P contents in all of the soils (data not shown). The change in extractable P of the four soils after wastewater irrigation is shown in Fig. 1. The P content in the 10 to 20 cm layer of the Rawsonville sand only tended to be higher compared to the top layer following the third irrigation with diluted winery wastewater, thereby indicating that attenuation of P did not occur in the top layer (Fig. 1A). The drastic decline in available P in the Rawsonville sand during the third season of winery wastewater irrigation (Fig. 1A) was possibly due the formation of stable complexes with constituents in the wastewater from which P could not be extracted by the Bray II reagent (Eloff & Laker, 1978). Since no leaching occurred when irrigations were applied (Mulidzi *et al*., 2016), it could not have contributed to the decline in available P.

In contrast, irrigation with diluted winery wastewater increased soil P substantially more in the 0 to 10 cm layer compared to the 10 to 20 cm layer of the Lutzville sand and the Stellenbosch granite soil over the four simulated seasons (Fig. 1B & 1D). This trend indicates that P attenuation occurred in the top layer of these soils. The very large increase in plant-available P in the top layer of the very sandy red soil from Lutzville is striking. It confirms the ability of non-acid red sandy soils to retain applied P in plant-available forms, as reported by others (e.g. Eloff & Laker, 1978). On the one hand there is little movement of P in the soil, but there is also little fixation of P into unavailable forms. Available P in the Lutzville sand increased as the pH (KCl) increased well above 7 where the diluted wastewater was applied (Fig. 1B). This trend suggests that the increasing amounts of sodium applied *via* the wastewater increased the soluble PO4^{3-}, instead of insoluble calcium phosphates being formed. Although the P content in the 10 to 20 cm layer of the Stellenbosch shale tended to be lower after the first simulated season, it increased at the same rate over time as in the 0 to 10 cm layer (Fig. 1C). This indicates that no P attenuation occurred from the second season onwards.

In the case of the initially acidic Stellenbosch shale and granite soils (Fig. 1C & 1D), the amorphous Fe^{3+} and Al^{3+} phosphates became more soluble as the pH$_{(KCl)}$ increased towards the optimum, as proposed by Busman *et al*. (2002). Since P was not determined in the irrigation water, models to estimate the effect of irrigation with diluted winery wastewater on soil P based on the amounts applied could not be created. However, the general variation in available P for the four soils could be illustrated with a plot of relative P, as calculated for each soil and layer, against pH$_{(KCl)}$ (Fig. 2).

After the fourth season, available P in the Rawsonville sand was still above the norm of 20 mg/kg proposed by Conradie (1994) for grapevines in sandy soils (Fig. 1A). However, this must be regarded as an atypical situation due to the initially high levels. After four simulated seasons of irrigation with the winery wastewater, Bray II P in the Lutzville sand reached over 40 mg/kg, thus far exceeding the norm of 20 mg/kg (Fig. 1B). This indicates that the winery wastewater is a good source of P on such soils. On the other hand, the fact that the Bray II P content of this soil increased by nearly 40 mg/kg after four seasons could serve as a warning that long-term continuous application of winery wastewater could cause the accumulation of excessive P levels in such soil over time. After the fourth season, P in the Stellenbosch shale soil (Fig. 1C) was well below the norm of 30 mg/kg for grapevines in soils containing more than 15% clay (Conradie, 1994). The much smaller increases in available P in the two Stellenbosch soils indicate much larger P fixation into unavailable forms in these acidic soils than in the Lutzville soil. Likewise, P in the Stellenbosch granite soil (Fig. 1D) was less than the lower threshold of 25 mg/kg for soils containing 6% to 15% clay (Conradie, 1994). The much smaller increases in available P in the two Stellenbosch soils indicate much larger P fixation into unavailable forms in these acidic soils than in the Lutzville soil. Although the lower thresholds were not reached, this situation does not rule out the possibility that it could be achieved if diluted winery wastewater was applied over a longer period. However, if

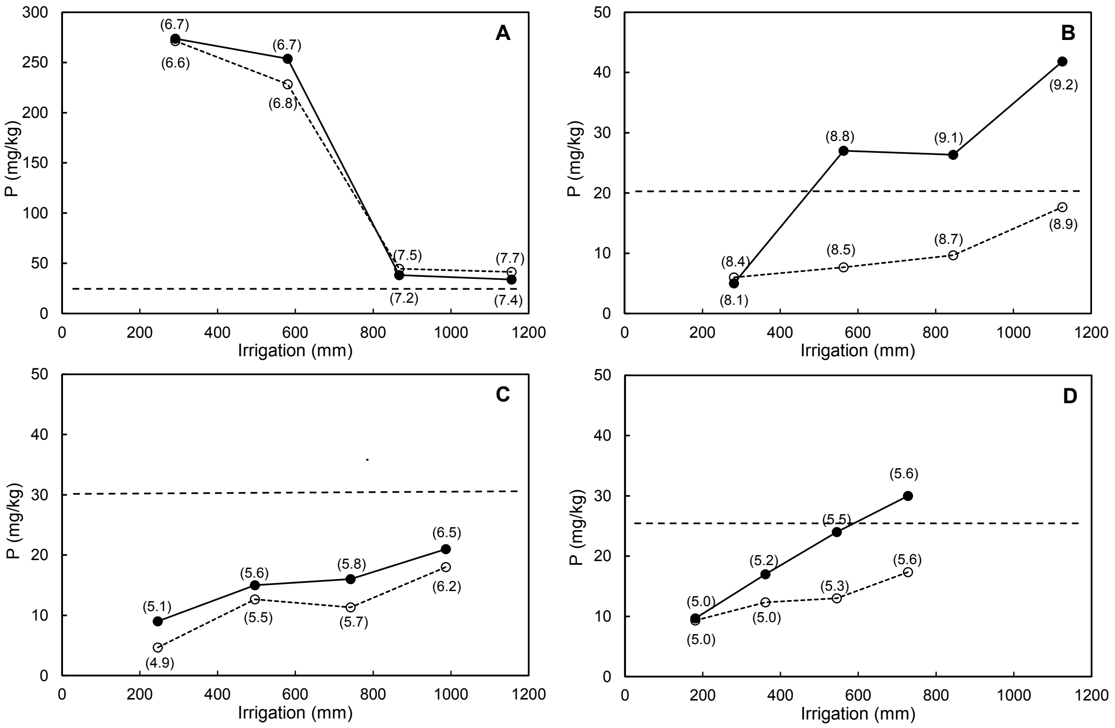

FIGURE 1

Effect of irrigation with diluted winery wastewater on P (Bray 2) in the 0 to 10 cm layer (solid circles) and 10 to 20 cm layer (open circles) in (A) Rawsonville sand, (B) Lutzville sand, (C) Stellenbosch shale and (D) Stellenbosch granite soils over four simulated seasons. Values in brackets indicate the soil $pH_{(KCl)}$. Dashed lines indicate the P (Bray 2) thresholds for grapevines based on clay content (Conradie, 1994).

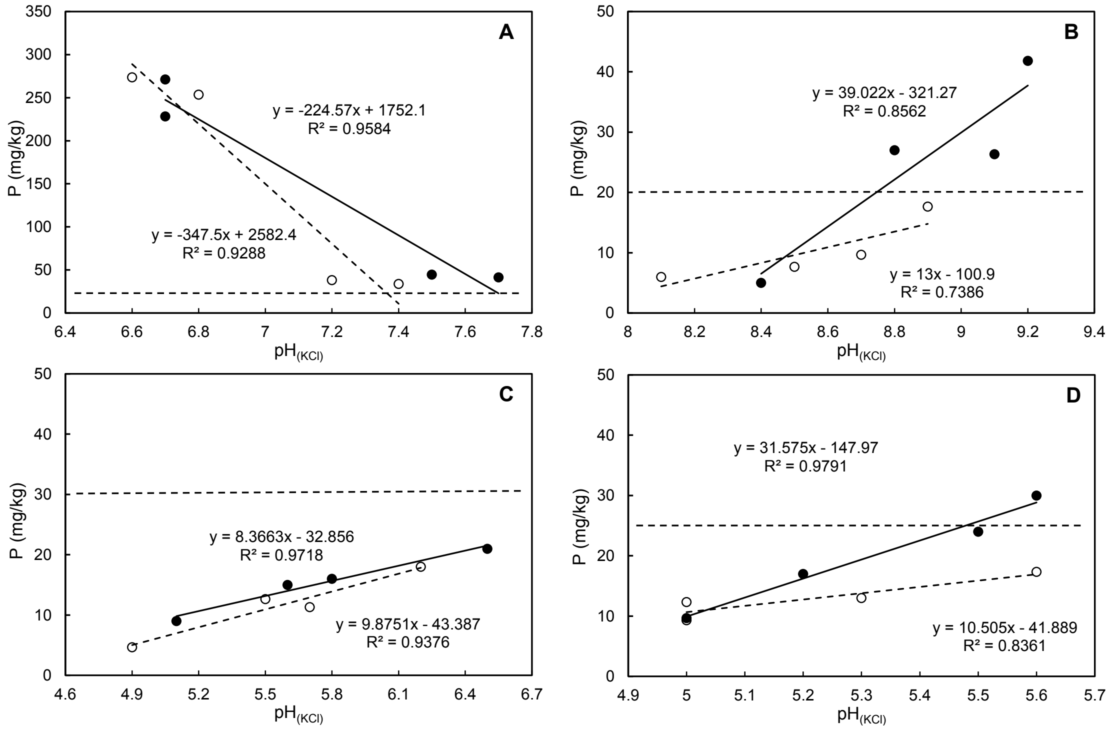

FIGURE 2

Relationship between P (Bray 2) and $pH_{(KCl)}$ in the 0 to 10 cm layer (solid circles) and 10 to 20 cm layer (open circles) in (A) Rawsonville sand, (B) Lutzville sand, (C) Stellenbosch shale and (D) Stellenbosch granite soils following irrigation with diluted winery wastewater over four simulated seasons. Dashed lines indicate the P (Bray 2) thresholds for grapevine based on clay content (Conradie, 1994).

the P applied *via* winery wastewater were to be absorbed by grapevines and cover crops, the minimum thresholds might not be exceeded to the extent that no fertilisers will be required.

CONCLUSIONS

Where diluted winery wastewater was applied, the level of soluble P in the shale and granite soils increased. Although the initial $pH_{(KCl)}$ in the aeolic sand was higher than the optimum range, the presence of relatively high levels of Na^+ caused available P to increase as the $pH_{(KCl)}$ increased. In the case of the alluvial sand containing unusually high initial levels of P, the $pH_{(KCl)}$ increased out of the optimum range, thereby causing a substantial reduction in the level of available P. These results indicate that irrigation with diluted winery wastewater could promote P absorption by grapevines if the $pH_{(KCl)}$ shift is towards the optimum. Since the level of P applied *via* diluted winery wastewater appears to be generally low, the application of P fertilisers will probably still be necessary to ensure adequate uptake by grapevines. It should be noted that the results represent a worst-case scenario, *i.e.* in the absence of rainfall or crops. Determining the effect of seasonal leaching by winter rainfall on the chemical status in soils irrigated with diluted winery wastewater is part of an ongoing study.

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