# A LABORATORY AND GLASSHOUSE EVALUATION OF AN ANAEROBIC BAFFLED REACTOR EFFLUENT AS A NUTRIENT AND IRRIGATION SOURCE FOR MAIZE IN SOILS OF KWAZULU-NATAL, SOUTH AFRICA

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#### ABSTRACT

Scarcity of water for irrigation is a serious hindrance for small-scale farmers in sub-Saharan Africa. The use of good quality water for irrigation has resulted in increasing pressure on such water which has prompted sourcing of wastewater as an alternative. One possibility, being investigated by eThekwini Water and Sanitation (Durban, South Africa), is to install anaerobic baffled reactors within local communities to treat wastewater to allow its use for agriculture. The success of wastewater irrigation depends on the ability of the soil to assimilate the water, nutrients and any other contaminants that are applied to it. The aim of this project was to investigate the potential of an anaerobic baffled reactor (ABR) effluent as both an irrigation and nutrient source for use in peri-urban agriculture.

The effluent was slightly alkaline (pH 7.40-7.60) and in class C2S1 (mediumsalinity/low sodicity water) according to the United States Soil Salinity Laboratory classification for irrigation waters. It was very low in heavy metals, values being below permissible levels according to the Food and Agricultural Organisation (FAO) of the United Nations and the South African Department of Water Affairs and Forestry (DWAF) guidelines for water use in agriculture. The total solids were low thus particulate matter was minimal with a greater concentration of the major elements found in solution. An investigation was carried out to ascertain the behaviour of the effluent when applied to soil and how the soil was able to adsorb plant nutrients from it. A soil column study was undertaken in the laboratory with three contrasting soil types namely a sandy soil (Longlands, E horizon), an organic soil (Inanda, A horizon) and a clayey soil (Sepane, A horizon). The effluent was leached through the soil while distilled water was concurrently used as a control. Results indicated that after application of 16 pore volumes, leachates from the columns contained concentrations of Na, equal to that in the incoming effluent for all soils. The concentrations of Ca and Mg were lower in the leachates than in the original effluent indicating adsorption in the soils. Phosphorus was the element that was most strongly adsorbed in all soils. While its adsorption in the Ia could be attributed to organic matter and the presence of iron oxides and oxyhydroxides, the clay type and amount in the Sepane was likely to have been responsible for P adsorption. The NO<sub>3</sub>-N, which was initially low in the effluent, increased as leaching

progressed, while the NH<sub>4</sub>-N decreased. In the water-leached columns, elements were leached out of soil because none were added with the water.

At the end of leaching, columns were allowed to drain and then sectioned into 2 cm segments. The 0-2 cm, 8-10 cm and 14-16 cm segments representing the top, middle and bottom parts of the column were analysed for inorganic-N, phosphorus and potassium. The elemental content of the 0-2 cm segment was significantly higher (p<0.05) than the lower segments in all columns for soluble P in all soils. This reflects the immobile nature of P in soils and confirmed the high amounts of P retained by the soils. There were significant differences between the effluent and the water-leached soils in terms of P retention. The amount of inorganic-N and K in the top layer was not significantly different from the other layers. In the Ia 0–2 cm segment, a pH increase of about 1.3 pH units was recorded in the effluent-leached columns when compared to the equivalent segment in the water-leached columns.

A glasshouse study was undertaken to assess the availability to maize of nutrients from the effluent. Maize was grown for 6 weeks in pots filled with the same soil types used in the columns study except that a similar sandy soil, Cartref (Cf, E horizon), replaced the Lo due to inadequate availability of the latter. Fertilizer (N, P and K) was applied at the full recommended rate, half the recommended rate and zero fertilizer for each of the three soils used. This corresponded to 0, 100, 200 kg N ha<sup>-1</sup> for all soils; 0, 40, 80 kg P ha<sup>-1</sup> and 0, 50, 100 kg K ha<sup>-1</sup> for the Cf; 0, 10, 20 kg P ha<sup>-1</sup> and 0, 102.5, 205 kg K ha<sup>-1</sup> for Ia; and 0, 30, 60 kg P ha<sup>-1</sup> and 0, 5, 10 kg K ha<sup>-1</sup> for Se. Lime was applied to the Ia soil at the rate of 10 t ha<sup>-1</sup>. Plants were watered with either effluent or tap water. Dry matter yield and nutrient concentrations for effluent-irrigated maize were significantly higher (p<0.05) than for all equivalent fertilizer applications in the water-irrigated plants. The unfertilized effluent-irrigated plants were not significantly different from the fertilized water-irrigated plants, but performed as well as the water-irrigated plants at half fertilization irrespective of soil type. Phosphorus deficiency was observed in the Ia and Se soils but not in the Cf soil, irrespective of fertilizer treatment. Plants grown on the Cf soil irrigated with effluent and fully fertilized had the highest above-ground dry matter yield (4.9 g pot<sup>-1</sup>) and accumulated the most nutrients namely N, P, K, Ca and Mg than all other treatments. After harvest the most marked changes had occurred in the Cf soil for P as the effluent-irrigated soils were significantly higher (p<0.05) than the water-irrigated soils reflecting the P input from the effluent.

The effect of effluent on soil and plants was further investigated by planting maize on the Ia soil without lime application. Plants that received effluent irrigation and no lime had significantly higher (p<0.05) dry matter yields and accumulated more N, P and K than the water-irrigated with no lime as well as the equivalent limed treatments. This suggests an interaction effect between the lime and the effluent with its effects obvious on above-ground dry matter yield and plant N, P and Mg.

A soil column experiment using the Cf, Ia and Se soils and planted with maize was conducted to assess the ability of plants to take up nutrients with concurrent leaching. Plants from the Cf soil recorded the lowest above-ground dry matter yield which was observed from the less vigorous growth as compared to plants in the Ia and Se soils. This growth pattern could also be explained by the low N accumulation in the plants from the Cf soil. Unlike N, P in plants grown on the Cf soil was significantly higher (p<0.05) than in the plants on the Ia and Se soils, despite having the least P gain from the effluent. The readily available P triggered both more uptake and also greater losses through leaching. The rate at which P was being supplied from the effluent was greater than its uptake by the plants and with limited capacity to hold onto P in this sandy soil there was inevitably loss though leaching.

A pot experiment was conducted to investigate the interaction effects between lime and effluent. Lime type (calcium hydroxide or dolomite) was applied to two acidic top soils namely Inanda and Avalon at 0%, 25%, 50%, 75% and 100% of the recommended rates for these soils. Maize was planted and after 6 weeks it was harvested and evaluated for above-ground dry matter yields and plant nutrient concentrations. Non-significant effects were recorded for above-ground dry matter, N, P and K as a result of altering the liming rate and type within each soil. The effects of lime application were apparent in the soils after harvest as increasing the lime rate caused an increase in pH and an inverse relationship with the exchangeable acidity and acid saturation in soils, as expected. Although the unlimed treatments did not impact on the acidity after harvest when compared to the soils at the beginning of the experiment. Phosphorus accumulation in plants was also unaffected by either lime rate or type which showed that effluent irrigation could influence P availability and further liming would not accrue benefits to the soil so as to influence plant P uptake.

Based on these data, ABR effluent could be perceived as a resource rather than a waste product. It could conveniently be used for irrigation provided there is soil and plant monitoring to assess build-up of elements especially in the long term. Further investigations have to be carried out on other crop types both in the field and glasshouse to ascertain nutrient uptake and effect on different soil types.

### DECLARATION

I, Irene Bongsiysi Bame, declare that

- 1. The research reported in this thesis, except where otherwise indicated, is my original research.
- 2. This thesis has not been submitted for any degree or examination at any other university.
- 3. This thesis does not contain other persons' data, pictures, graphs or other information, unless specifically acknowledged as being sourced from other persons.
- 4. This thesis does not contain other persons' writing, unless specifically acknowledged as being sourced from other researchers. Where other written sources have been quoted, then:
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Signed..... Date.....

As the candidate's supervisor I have /have not approved this thesis for submission.

Signed..... Professor J.C. Hughes Date.....

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# **DEDICATION**

I dedicate this work to my parents,

### **Thomas Tayu Bame**

and

### Mary Shomika Bame

as a small gesture compared to what they have done for me.

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### Chapter 1

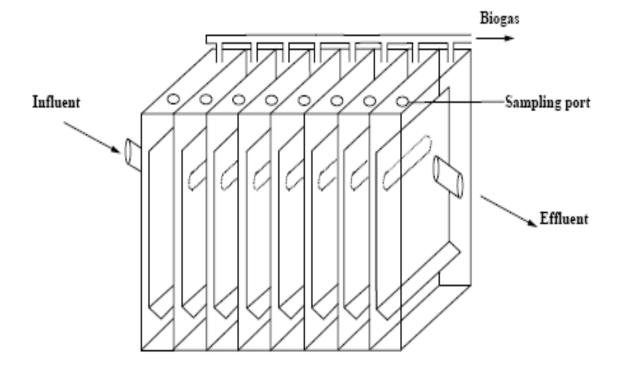
### INTRODUCTION

#### **1.1** Concept of human waste

Worldwide, people both in rural and urban areas have been using human excreta for centuries to fertilize fields and fishponds and to maintain soil organic matter. The recirculation of nutrients from towns to agricultural land is one of the big challenges of our time since most agricultural activities take place in the rural areas far from where most of the nutrients are being lost. Urban and peri-urban agricultural soils are often greatly depleted of organic matter and nutrients which flow from rural areas to urban waste sinks failing to complete the nutrient loop (Esrey and Andersson, 2001). Alternatively these nutrients end up in sewage works and are not recovered which is not in accordance with the principles of sustainable development. Human waste has to be disposed of in one form or the other which could be either on land or water. Whatever disposal measures are embarked upon, there are environmental regulations that govern such disposal. The soil has the potential to assimilate human waste but if this option is taken, it must be ensured that the quality and fertility of soils, crops and water are not negatively affected. There is therefore a need for research on the efficiency and environmental impacts of this organic resource if disposed of onto land.

#### 1.2 Project background and justification

This project stems from an investigation by the Water Research Commission (WRC), South Africa titled "Evaluation of the Anaerobic Baffled Reactor for Sanitation in Dense Peri-urban Settlements" WRC report No. 1248/1/06 (Foxon *et al.*, 2005). The anaerobic baffled reactor (ABR) is a high rate, anaerobic digester consisting of alternate hanging and standing baffles designed to treat wastewater and has undergone improvement in design over the years to make it suitable for treating a wide variety of wastewaters (Barber and Stuckey, 1999). The performance of an anaerobic baffled reactor (ABR) was investigated in treating sewage in peri-urban areas where it would take several years to get water-borne sewage sanitation facilities installed.



**Figure 1.1** Schematic diagram of the anaerobic baffled reactor (adapted from Bell, 2002).

The technology has been identified to have potential in South Africa especially given the rapid expansion of peri-urban areas due to the establishment of new communities that are not connected to the main sewer sytems. It was found to be advantageous from an engineering perspective in that it could be easily installed and maintained as most of the components are readily available. However, this form of treatment resulted in the production of an effluent with no nutrient removal and this was considered to be a limitation in terms of disposal (Snyman and Herselman, 2006) as natural waterways have to be protected from nutrient rich effluents, particularly those that contain nitrogen and phosphorus. This research focuses on a recycling system that promotes a circular rather than a linear movement of nutrients thus closing the nutrient loop (Section 1.1).

In a diagnostic report by the National Planning Commission of South Africa (National Planning Commission, 2011) over 400 million tons of water a day is being taken from the Earth's underground reservoirs which is more than the input from rainfall (Martin, 2006). Agriculture uses about one third of this volume (Seckler *et al.*, 1998) but cannot rely on this supply in the long term as increasingly more is being diverted for urban and industrial use (Molle and Berkoff, 2006). There is bound to be a shortfall in South Africa which has been

ranked the thirtieth driest country in the world as water used is not being replaced by rainfall (Schreiner *et al.*, 2010. An imminent water resource management strategy is required to safeguard water supply in the future through water reuse The use of treated wastewater as a substitute for freshwater will maintain agricultural productivity as well as conserving water. Most water treatment plants focus on treatment strategies that reintroduce wastewaters into rivers which might pose a risk to water bodies considering the cost and inefficiency associated with such treatments. An ABR could be a solution for water recycling which presents a 'holistic' approach to irrigated agriculture that comes with the advantage of nutrients as inorganic fertilizer substitute.

The focus on wastewater reuse and nutrient capture can contribute towards climate change adaptation and mitigation. Energy savings from freshwater pumping and fertilizer savings would possibly prevent mineral fertilizer mining thus reducing their carbon footprint and earning carbon credits (Hanjra *et al.*, 2012).

As a result a new project was developed to evaluate the use of ABR effluent as an irrigation source for peri-urban agriculture. This could then supplement the low rainfall that characterises South Africa generally and supply irrigation that is necessary for agriculture. On-site sanitation in poor peri-urban communities can therefore be linked to such initiatives to improve food security in accordance with the millennium development goals (United Nations Millennium Declaration, 2000). One such initiative is community gardening where the use of ABR effluent could improve livelihoods and build the resilience of communities most vulnerable to food security. However, there is a need to study the implications of using such an effluent for agriculture and its effects on soils, crops and health of the community. This thesis lays the foundation for this research into the effects of ABR effluent on soil and crops.

#### 1.3 Objectives

The main objective of this study was to evaluate the potential of an ABR effluent for use as a nutrient and irrigation source in peri-urban agriculture with the following specific aims to:

review the literature on wastewater use to investigate potential and limitations for use in agriculture (Chapter 2);

- characterise the ABR effluent for the presence of any constituents unacceptable for use in irrigation for agricultural purposes (Chapters 3 and 6);
- evaluate the resulting leachates coming from the use of effluent as compared to leachates from water leaching (Chapter 3);
- vevaluate the ability of soil to assimilate nutrients of significant importance to plant growth from the effluent as well as the effects of the effluent on the properties of different soil types (Chapter 4);
- assess the growth of maize and uptake of nutrients when using effluent as a source of fertilizer (Chapter 5);
- monitor nutrient release in soil and potential for leaching to groundwater when irrigating with effluent (Chapter 6); and
- determine if the effluent possesses any liming effects when applied to soil and the extent to which the liming properties can compare with other types of liming materials (Chapter 7).

The thesis concludes with a summary of the findings and recommendations for future work (Chapter 8).

#### Chapter 2

# WASTEWATER AS A SOIL CONDITIONER AND POTENTIAL NUTRIENT SOURCE FOR PLANTS: A REVIEW OF LITERATURE

#### 2.1 Introduction

Presently, some of the key issues around the world centre on exploitation and limitation of natural resources as well as environmental pollution. Waste reuse is being adopted as a way to evade environmental problems and a recycling method will be determined by the type of waste in question. There is an associated risk involved in the treatment and handling of wastes which must be treated differently. Human wastes have been treated as a pollutant with disposal systems employed to deal with such wastes. Moreover, faecal material is considered to be repulsive and not to be touched. Most of the conventional sanitation systems lack nutrient recycling as most treatment technologies result in nutrients being dumped onto dedicated sites or ending in sewage lines. Nutrient recycling through utilisation of wastewater from sewage treatment technologies therefore presents a solution to disposal. However, there must be an understanding of the impacts of applying such waste on the soil and the ability of the soil to sustain plant growth. This is of utmost importance as the soil has to be able to accommodate both nutrients and water for plant uptake.

Many countries have adopted guidelines for the reuse of wastewater e.g World Health Organisation (WHO), 2006, United States Environmental Protection Agency (USEPA), 1992; South African Department of Water Affairs and Forestry (DWAF), 1996 which are based on the health and environmental risk from microbial pathogens, nutrients and heavy metals. A detailed comparison of these guidelines will not be a part of this review but it is worth mentioning that they have similarities in terms of permissible limits for various constituents. There is concern about the potential health and environmental impacts of these factors and if they survive or concentrate in the treatment processes they accumulate in the environment and may enter the food chain. Maximum permissible concentrations of heavy metals in surface soils irrigated with wastewater are normally given as total concentrations (Pescod, 1992). According to the South African guidelines for utilization and disposal of wastewater sludge (Snyman and Herselman, 2006) if the total metal content (aqua regia) of the soil is found to be between the total investigative level and the total maximum threshold, the mobility of the metals in the soil needs to be assessed. Current guidelines do not specify the types of soils, plants and other factors that have a bearing on how much heavy metal a plant can take up. This review explores the impacts of wastewater irrigation on soil and the possible effects that would be carried onto the crop and the environment as a whole. It further looks at the critical role wastewater has played in agricultural production with some comments concerning the risks involved in wastewater reuse.

#### 2.2 History of using human waste

Human wastes have been used frequently as "night soil" in some areas of the world such as China, Vietnam and Japan (Malkki, 1999) without any major problems for agricultural productivity, although improper use causes hygiene and health problems. Some countries and cultures have been recycling human wastes for agricultural purposes for thousands of years, especially in China and Southeast Asia (Reed and Shaw, 2003) but often human wastes have not been properly sanitized thereby propagating disease.

The practice of reuse has also been common in Europe. Sweden is probably the country with the most advanced system of collection and reuse of human urine, where it is practised by farmers on a large, mechanised scale. In a number of settlements (called 'eco-villages') or apartment blocks in the country the residents have ecological sanitation systems with urine diversion toilets. The urine from the houses or apartments is collected in large underground tanks, and what the residents do not use themselves is collected by farmers in road tankers and used to fertilize their crops (Esrey and Andersson, 2001). In the Netherlands dry toilets were emptied and contents used regularly as fertilizer for arable farming until the second half of the 1900s. Around this period, water toilets were initially not accepted in some Nordic towns the main argument being that agriculture would lose its resource for fertilization (Lindegaard, 2001). Composting and separating toilets have enabled the reclamation of human excreta and the use of the nutrients contained therein as a fertilizer and soil conditioner. In Sweden, organic farmers have expressed interest in using human urine as

liquid manure because of the content of macronutrients and the low heavy metal content (Linden, 1997).

In Africa, urine and faeces have been used in many countries such as Burkina Faso where human faeces were used when planting mango trees (Jönsson *et al.*, 2004). Morgan (2003) has shown that in Kenya the 'neem' and citrus trees have been grown on 'arborloo' pits, while in Zimbabwe the 'fossa alterna' has been used to recycle nutrients as humus in shallow pits. Morgan (2001) has also shown that some farmers practising urban agriculture have collected sewage from disposal points to fertilize their plants and gardens in Malawi. In South Africa, human excreta have been perceived as dirty and unpleasant to handle and, even more so, culturally unacceptable. Studies by Dunker *et al.* (2007) showed that a number of projects on urine diversion (UD) toilets were put in place in the Eastern Cape, Northern Cape, and KwaZulu-Natal but there was still the unwillingness to handle faecal material. Over the years the UD toilets have been converted to sheds for maintenance tools or as animal pens, especially in KwaZulu-Natal.

#### 2.3 Composition of urine and faeces

Human waste constitutes a large nutrient resource and could beneficially be handled in a more sustainable way than in present systems. According to Wolgast (1993), the annual amount of human excreta of one person corresponds to the amount of fertilizer needed to produce 250 kg of cereal, which is also the amount of cereal that one person needs to consume per year. Each year one person produces 500 kg of urine as compared to 50 kg of faeces. These faeces contain some 10 kg of dry matter. Thus, annually one person produces approximately 5.7 kg of nitrogen, 0.6 kg of phosphorus and 1.2 kg of potassium and some micronutrients in a form useful for plants (Wolgast, 1993). Of the human excreta, urine contains some 90% of the nitrogen, 50-65% of the phosphorus and 50-80% of the potassium (Wolgast, 1993). These figures, however, depend on the body weight of the person involved, the climate, water intake and the diet characteristics, especially its protein content. Drangert (1998) has also shown that 4.0 kg N, 0.4 kg P and 0.9 kg K is found in 500 L of urine while 0.5 kg N, 0.2 kg P and 0.3 kg K is found in 50 L of faeces. As shown in Table 2.1, the amount of N, P and K differs for different countries with most excreted in urine.

Country		Nitrogen kg cap <sup>-1</sup> yr <sup>-1</sup>	Phosphorus kg cap <sup>-1</sup> yr <sup>-1</sup>	Potassium kg cap <sup>-1</sup> yr <sup>-1</sup>
China, total		4.0	0.6	1.8
	urine	3.5	0.4	1.3
	faeces	0.5	0.2	0.5
Haiti, total		2.1	0.3	1.2
	urine	1.9	0.2	0.9
	faeces	0.3	0.1	0.3
India, total		2.7	0.4	1.5
	urine	2.3	0.3	1.1
	faeces	0.3	0.1	0.4
South Africa, total		3.4	0.5	1.6
	urine	3.0	0.3	1.2
	faeces	0.4	0.2	0.4
Uganda, total		2.5	0.4	1.4
	urine	2.2	0.3	1.0
	faeces	0.3	0.1	0.4

**Table 2.1**Estimated excretion of nutrients per capita in different countries (Jönsson and<br/>Vinnerås, 2004)

The two most critical nutrient elements for agricultural production worldwide are N and P and the third is K (Heinonen-Tanski and van Wijk-Sijbesma, 2005). In most countries plant nutrients are collected in wastewater treatment plants and a large part pollutes the environment, depending on the system used (Steineck *et al.*, 1999). The plant availability of urine nitrogen is the same as that of chemical urea or ammonium fertilizers. This is to be expected as 90-100% of the urine nitrogen is found as urea and ammonium and this has been verified in fertilization experiments (Kirchmann and Pettersson, 1995; Richert Stintzing *et al.*, 2001). The phosphorus in urine is almost entirely (95-100%) inorganic and is excreted in the form of phosphate ions (Lentner *et al.*, 1981). These ions are directly available to plants and thus it is no surprise that plant availability has been found to be at least as good as that of chemical phosphate (Kirchmann and Pettersson, 1995).

Potassium is excreted in the urine as  $K^+$  ions, which are directly available to plants. This is the same form as supplied by chemical fertilizers and thus their fertilizing effect should be the same (Jönsson *et al.*, 2004). Sulphur is mainly excreted in the form of free sulphate ions (Lentner *et al.*, 1981; Kirchmann and Pettersson, 1995) which are directly available to plants. This is the form of S in most chemical fertilizers. Compared with the urine, which has water soluble nutrients, faeces contain exchangeable water soluble nutrients and nutrients that are combined in larger particles not soluble in water. About 50% of the nitrogen and the majority of the potassium in faeces are water soluble (Berger, 1960; Frausto da Silva and Williams, 2001). Phosphorus is mainly found as calcium phosphate particles that are slowly soluble in water (Frausto da Silva and William, 2001).

With the widespread knowledge of human waste being a resource, a lot of attention has to be given to its use in order to avoid its negative effects. With a better closed nutrient loop, many more people, including low income farmers, would be able to produce more food and other plant products. It would also reduce the pollution effects from unsafe excreta disposal and excessive use of chemical fertilizers and protect surface and groundwater and the air. The recycling of nutrients from urine and faeces is one of the key benefits of ecological sanitation. The nitrogen, phosphorus and potassium found in urine are a valuable fertilizer and the high organic content of faeces makes the composted product (humus) an excellent soil conditioner (Strauss, 2000). In addition, it is important to recover and reuse these nutrients to produce sustainable ecosystems that will reduce the drain on natural reserves and lessen the dependence on artificial chemical fertilizers.

The problems that might arise from use of human excreta are mainly in terms of the health hazards as they contain a number of pathogens (Schönning and Stenström, 2004). However, these can be rendered harmless by various treatment methods (Schönning and Stenström, 2004). A more effective utilization of excreta would also reduce the water-borne enteric microbiological diseases, since there would be less contaminated wastewater and the die-off of enteric microorganisms could be better controlled.

#### 2.4 Ecological sanitation systems

Hannan and Andersson (2002) defined ecological sanitation as "an ecosystem approach to waste disposal based on three key principles – that sanitation should be safe from a health perspective; 'green' or non-polluting; and be based on principles of reuse and recycling of the valuable nutrients in human excreta". Ecological sanitation systems can make an invaluable contribution to sustainable livelihoods and poverty reduction, in both rural and urban areas, by increasing food security through the return of nutrients from excreta to the

soil to increase soil fertility and by reducing pollution and health risks. Such systems also impact positively on food security through better management of scarce water resources and contribute to health through reducing transmission of disease and increasing nutritional intake (Esrey *et al.*, 1998; Esrey and Anderson, 2001; Werner *et al.*, 2004b; Langergraber and Muellegger, 2005).

Ecological sanitation approaches thus greatly aid in saving useful resources and are both financially and environmentally more viable than conventional sanitation systems and offer more from sustainable livelihood and poverty reduction perspectives. In addition, these systems can foster decentralised management systems, with potential for empowering people, providing for local livelihoods and enhancing community cohesion (Esrey *et al.*, 1998). Ecological sanitation is therefore a system that turns human excreta into something useful and valuable with minimal risk of environmental pollution and with no threat to human health. It portrays human excreta as a resource and not a waste product (Austin and Duncker, 2002).

#### 2.5 Wastewater use in agriculture

Wastewater use in agriculture has been established as the most viable reuse option as compared to other uses (Jimenez *et al.*, 2010). Scott *et al.* (2010) reported that unplanned use of wastewater either directly or indirectly is an order of magnitude greater than planned use. In many low-income and middle-income countries, wastewater irrigation either involves the direct use of untreated wastewater or its indirect use from rivers and streams that receive untreated wastewater discharges. Case studies of city and country assessments of varying detail conducted in middle and low-income countries of Africa, Asia and Latin America have recognized that the use of untreated wastewater for the irrigation of high-value cash crops close to urban centres is a widespread practice. An estimated 20 million hectares is under agriculture using treated, partially treated, diluted and untreated wastewater (Scott *et al.*, 2004; Marsalek *et al.*, 2005; Hamilton *et al.*, 2007; Keraita *et al.*, 2008). For millions of poor households wastewater is a highly important productive resource used in profitable but often informal production systems that contribute significantly to the supply of perishable produce, notably fresh vegetables, to urban areas (Scott *et al.*, 2004; Drechsel *et al.*, 2006). In developing countries there are difficulties in sourcing such perishable food, from more distant

locations due to lack of necessary infrastructure and cooled storage trucks for transport, thus supporting agriculture in market proximity. Furthermore, interest in wastewater irrigation is viewed as a substantial and sometimes even primary source of income in addition to contributing towards urban food supply (Drechsel et al., 2006; van Veenhuizen and Danso, 2008). With the economic development of many countries towards large scale urbanization, industrial or domestic wastewaters are either used or disposed of on land for irrigation purposes and this creates both opportunities and problems. Opportunities exist as wastewaters from municipal origin are rich in organic matter and also contain appreciable amounts of major and micronutrients (Feigin et al., 1991; Pescod, 1992; Gupta et al., 1998). For many wastewaters, it is their high content of nutrients (nitrogen and phosphorus) and total dissolved salts that make them able to be treated as waste products, although when recycled can be used as a fertilizer source in irrigated agriculture (Toze, 2006; Scheierling et al., 2011)). Other constituents may be critical in specific cases such as high organic matter content and biological oxygen demand, or high concentrations of particular chemicals. The use of treated wastewater in agricultural soils has been proposed as a sustainable management strategy and as an aspect of integrated water management for water-poor countries (Neubert, 2009). In such countries, the reuse of wastewater has, in recent years, been viewed as a strategy for the future and is being propagated as a concept by industrialized countries. In peri-urban areas of many developing countries, agriculture would be virtually impossible without the use of wastewater for irrigation. Farmers are dependent on it for their existence since it is their only reliable source of water (Friedler, 2001; Rutkowski et al., 2007).

#### 2.5.1 Soil, plant and wastewater interrelationships

The objective of land treatment of wastes is to utilise the chemical, physical, and biological properties of the soil/plant system to assimilate the waste components without adversely affecting soil quality or causing contaminants to be released into water or the atmosphere (Loehr, 1984). The use of wastewaters centres on the need to maintain a productive soil environment for crop production, while minimizing or avoiding degradation of soil and water resources. Municipal wastewaters used for irrigation could influence the physical, chemical and biological properties of the soil (Feigin *et al.*, 1991; Mathan, 1994; Schipper *et al.*, 1996) which in turn play an important role in the transformation of nutrients present in the applied wastewater.

Chakrabarti (1995) observed that rice crops gave a higher yield when irrigated with raw or partially diluted sewage compared to unamended groundwater. Evidently while the additional elements can be a bonus as additional fertilizer, excess carbon and nitrogen can have an adverse effect through excessive microbial activity and growth. Thus care needs to be taken in the concentrations of nutrients in the recycled water to avoid detrimental impacts on soil porosity.

#### 2.5.1.1 Effect of wastewater irrigation on soil physical properties

The main properties that control soil hydraulic conductivity are soil texture, dry bulk density, soil structure, soil solution chemistry, soil cation exchange capacity (CEC) and the microbial activity (Halliwell *et al.*, 2001; Goncalves *et al.*, 2007). These properties tend to be modified during the application of wastewaters especially in tropical soils because of the effects of sodium (Na<sup>+</sup>) which occurs in high concentration in many wastewaters (Goncalves *et al.*, 2007). Studies by Magesan *et al.* (1999) and Halliwell *et al.* (2001) have shown that changes in the porous system of the soil seem to be the dominant factor for infiltration and hydraulic conductivity reduction. Decreases in soil hydraulic conductivity can result in surface runoff and flooding, which leads to superficial contamination by the effluents and soil erosion, especially in a tropical environment (Vinten *et al.*, 1983). Intensive irrigation with treated wastewater in loam and clay soils has been shown to have resulted in a significant increase in *"clay dispersion and eluviation from the upper soil layers"* (Warrington *et al.*, 2007).

The potential risk associated with irrigation using treated wastewater is degradation of soil structure. This is manifested by deterioration of aggregate stability resulting in decreased soil hydraulic conductivity. As a result there is increased susceptibility to surface sealing, runoff and soil erosion problems such as soil compaction and decreased soil aeration (Mandal *et al.*, 2008). Irrigation with water of a moderate SAR of about 6 leads to an ESP of comparable value in the soil and can adversely affect soil physical properties such as soil hydraulic conductivity due to sodium-induced clay dispersion (Halliwell *et al.*, 2001). Studies by Tarchitzky *et al.* (1999) have shown that the presence of dissolved organic matter in treated wastewater, coupled with its higher sodicity, increases clay dispersion and results in higher flocculation values for both specimen and soil clays.

Comparative studies on the effects of irrigation with either treated wastewater or freshwater have shown that irrigation with treated wastewater containing a high load of organic matter and nutrients decreased soil hydraulic conductivity due to pore blockage by the suspended solids present in the treated wastewater (Vinten *et al.*, 1983; Magesan *et al.*, 2000) and by the excessive growth of microorganisms (Magesan *et al.*, 1999). Studies that used wastewater with a greater degree of treatment and thus of better quality, have shown no negative effect on soil hydraulic conductivity (Levy et al., 1999) whereas Tarchitzky *et al.* (1999) reported a reduction in hydraulic conductivity after leaching with treated wastewater. The level of treatment of the wastewater then becomes a factor for consideration.

In other experiments, changes in soil hydraulic conductivity during leaching with deionized water were compared to soils subjected to long term irrigation with either treated wastewater or freshwater (Bhardwaj *et al.*, 2007). Results from these studies showed that irrigation water quality and method of irrigation did not have conclusive effects on aggregate stability of the soil which was used as an indicator of steady state hydraulic conductivity. Levy *et al.* (2005) found that the combined effects of salinity, wetting rate and sodicity on hydraulic conductivity were complex and should be considered simultaneously in estimating hydraulic conductivity.

Similar studies compared the changes in infiltration rate, runoff and erosion during natural or simulated rainfall on such soils (Mamedov *et al.*, 2001; Agassi *et al.*, 2003). These properties were found to vary due to differences in treated wastewater quality, soil texture, calcium carbonate content, intensity of cultivation, irrigation method, and antecedent moisture content in the soil. An exception to these studies was that of Bhardwaj *et al.* (2008) who tested the hypothesis that replacing saline-sodic irrigation water that had been in use for many years, with the considerably less saline-sodic treated wastewater, although with higher loads of organic matter and suspended solids, may help the soil regain its structure and hydraulic conductivity. Bhardwaj *et al.* (2008) examined the hydraulic conductivity of undisturbed soil cores and the aggregate stability of samples taken from soils irrigated with these different water qualities. They found significantly higher hydraulic conductivity and aggregate stability in the treated wastewater-irrigated samples than in those that were subjected to long term irrigation with saline-sodic water. This effect of irrigation with wastewater can be used as a check mechanism especially in monitoring leaching columns.

In an earlier study, effects of sodicity on soil hydraulic conductivity, permeability and seal formation were determined for dry soils that were subjected to rapid wetting either from below or from above, prior to their exposure to leaching or simulated rain. In this study fast wetting led to aggregate slaking (Panabokke and Quirk, 1957). A similar study showed substantial reduction in aggregate slaking by using slow wetting rates (commonly ~2 mm h<sup>-1</sup>) which lessened the susceptibility of soil to seal formation and maintained higher hydraulic conductivity values in comparison to cases where severe aggregate slaking occurred when using much faster wetting (~50 mm h<sup>-1</sup>) (Moutier *et al.*, 2000). Shainberg *et al.* (2001) and Mamedov *et al.* (2001) have also demonstrated the importance of aggregate slaking in determining susceptibility to permeability deterioration which depends on both soil sodicity and clay content.

#### 2.5.1.2 Effect of wastewater irrigation on soil chemical properties

The soluble inorganic constituents of irrigation waters react with soils as ions rather than as molecules. The principal cations are  $Ca^{2+}$ ,  $Mg^{2+}$  and  $Na^+$  with small quantities of K<sup>+</sup> ordinarily present, while the dominating anions are  $CO_3^{2-}$ ,  $HCO_3^-$ ,  $SO_4^{2-}$  and  $Cl^-$  (US Salinity Laboratory Staff, 1954). Interesting to note is the availability status of nitrogen, phosphorus, and potassium to crops which could have been prompted by the higher amounts accumulated in surface soils after receiving loads of sewage than in soils irrigated with water (Yadav *et al.*, 2002). Similarly, the N, P and K amounts in a clayey soil increased significantly after irrigation with municipal wastewater that was screened through filtration media in India (Singh *et al.*, 2012). After one season of wheat cultivation the amount of N, P and K in soil increased from 200, 13.0 and 280.6 kg ha<sup>-1</sup> to 283, 23.9 and 343 kg ha<sup>-1</sup>, respectively.

The concentration of total dissolved solids (TDS) in sewage effluent ranges from 200-3000 mg L<sup>-1</sup> (Feigin *et al.*, 1991) but could be higher in effluents from intensive rural industries and industrial processing. Effluent irrigation can result in the addition to soil of large amounts of salts. An annual application of 1000mm of water with 500mg L<sup>-1</sup> of TDS would add five tons of salt per hectare per year to the soil (Bond, 1998). Problems may arise through removal of water by evapotranspiration and accumulating salts to a concentration considered harmful. Effluent irrigation can be managed such that salt does not accumulate in the root zone, which invariably means it will impact on groundwater. It may be possible to store some salt between the root zone and the water table, if the underlying material is sufficiently porous. However, this is likely to be no more than six tons per hectare for each metre of the profile. Storage of salt from 10 years of irrigation contributing salt at the rate of five tons per hectare per year therefore requires about eight metres of profile between the root zone and the water table (Bond, 1998).

Heavy metals in wastewater are also a limitation on its utilisation. Common treatment processes efficiently remove heavy metals and the larger fraction in raw sewage ends up in the biosolid fraction of the treatment process with very low metal concentrations present in the treated effluents (Sheikh *et al.*, 1987). Although the concentration of heavy metals in sewage effluents are low, long term use of these waters on agricultural lands often results in the build-up of these metals to elevated levels in soils (Datta *et al.*, 2000). Therefore heavy metals tend not to be a cause for concern when irrigating with treated effluent that is not from an industrial source but when present they could be of utmost importance because of their potential bioavailability to crops.

Local conditions such as climate, soil and plant characteristics affect their uptake and it should therefore be determined whether they are within acceptable limits (Kiziloglu *et al.*, 2008). In Bulgaria a study by Angelova *et al.* (2004) confirmed that fibre crops such as flax and cotton did take up heavy metals from heavily contaminated soils as levels were above maximum permissible concentrations according to Bulgarian standards. However, the concentrations detected in leaves and seeds were only a small percentage of the concentration present in soil. Contrary to this, untreated wastewater irrigation in Turkey did not significantly affect the heavy metal content in cauliflower and red cabbage on a short term basis (Kiziloglu *et al.*, 2008). In Kenya, Ofosu-Asiedu *et al.* (1999) examined the uptake of heavy metals by crops irrigated with domestic and industrial wastewater. They found that the levels in the crops were similar to background environmental levels and thus posed no health risks.

Nutrient levels of soils are expected to improve considerably with continuous irrigation with wastewater and, depending on the source, may contain variable amounts of heavy metals that could limit the long term use of effluent for agricultural purposes as the likelihood of phytotoxicity and negative environmental effects increase. The most common organic

nutrient in wastewater is dissolved organic carbon (DOC), which can take various forms depending on the source of the wastewater. The source can also influence the bioavailability of the nutrient. The organic carbon present in recycled water can stimulate the activity of soil microorganisms (Ramirez-Fuentes *et al.*, 2002). Magesan *et al.* (2000) noted that the organic and inorganic nutrients in treated effluent that had a high carbon to nitrogen ratio stimulated the soil microorganisms, which in turn decreased the hydraulic conductivity of the irrigated soil. The reduction in hydraulic conductivity was by excess cell growth and the production of biofilm structures, which clogged the pore spaces between the soil particles.

# 2.6 Microorganisms in wastewater

Although the present study does not investigate in any detail the pathogenic component of wastewater the following brief discussion of some aspects of the microorganisms present in human waste is included to highlight a very important aspect of the use of human waste for agriculture. The most common human microbial pathogens found in recycled water are enteric in origin and include viruses, bacteria, protozoa and helminths. They enter the environment in the faeces of infected hosts through defecation in water, contamination with sewage effluent or from runoff from soil and other land surfaces (Feachem *et al.*, 1983). Increased metabolic activity of soil microorganisms has been observed when sewage effluent is used for irrigation (Meli *et al.*, 2002; Ramirez-Fuentes *et al.*, 2002).

## 2.6.1 Viruses

Viruses are among the most important and potentially most hazardous of the microbial pathogens found in wastewater. Untreated water can contain a range of viruses which are pathogenic to humans. In wastewater, viral numbers have been detected in concentrations in excess of  $10^3$ - $10^4$  viral particles L<sup>-1</sup> (Feachem *et al.*, 1983). Viruses are generally more resistant to treatment processes, more infectious, require smaller doses to cause infection and are more difficult to detect in environmental samples than other microorganisms (Toze, 1999). Detection of viruses in water sources usually involves concentration of viral particles from large volumes of water samples, followed by detection methods such as culturing in suitable host cells, electron microscopy and immunoassays. These detection methods have the disadvantage of being highly inaccurate, time consuming, expensive and requiring

sophisticated laboratories with highly trained personnel (Toze, 1999). Therefore routine assessment of water and wastewater samples for viruses becomes extremely laborious and expensive.

Alternatively, PCR (polymerase chain reaction) can be used to detect viruses. This has the advantage of improving detection limits and the range of viruses detected as well as reducing processing time. Traore *et al.* (1998) were able to detect astrovirus, hepatitis A virus and poliovirus in all samples of experimentally infected mussels. Thus the efficiency, sensitivity and speed with which these PCR methods can detect viruses in infected shellfish could allow these to be used as an important monitor of the quality of treated wastewater. Gajardo *et al.* (1995) found a detection limit of 20 plaque-forming unit mL<sup>-1</sup> for rotaviruses present in samples. Although PCR technology offers the advantages of specificity and sensitivity to detect small amounts of target nucleic acid in a water sample, quantification using PCR is still a difficult and exacting method. It requires skilled operators, expensive equipment and large amounts of material. The PCR detection of pathogens in water and wastewater is therefore more of a qualitative presence/absence test (Toze, 1999) as compared to the 'most probable number technique' (MPN) which provides a more quantitative analysis of particular pathogens (American Public Health Association, 1992).

## 2.6.2 Bacteria

Bacteria are the most common microbial pathogens found in wastewaters (Toze, 1999). Most pathogenic bacteria can be isolated and maintained on solid media, but there are problems associated with the detection and quantification of bacteria in wastewater samples. These include the time and expense involved in identifying and typing of bacteria pathogen isolates and the effects of selective media and/or selective isolation methods. Another dilemma frequently encountered is that viable bacterial strains in the environment can enter a dormancy state, in which they are viable but non-culturable (Porter *et al.*, 1995) and can cause an underestimation of pathogens from wastewaters. Fode-Vaughan *et al.* (2003) have used PCR for the direct detection of *Escherichia coli* in water samples. Tsen *et al.* (1998) used selected regions of the *E. coli* 16SrRNA gene to detect *E. coli* cells in water using PCR and, with an enrichment step, they were able to detect as little as one *E. coli* per 100mL. The presence of *E. coli* is often used as an indicator of faecal contamination (Edberg *et al.*, 2000).

There are protozoan pathogens which have been isolated from water and wastewater. The pathogenic protozoa of most interest are *Entamoeba histolytica, Giardia intestinalis* and *Cryptosporidium parvum* (Toze, 1997). These are common enteric pathogens and have been frequently detected in water contaminated with faecal material. They exist as cysts or oocysts in wastewater and tend to be present in low numbers when compared to bacteria. Detection is difficult because they cannot be easily cultured and the most common detection method for *G. intestinalis* cysts and *C. parvum* oocysts involves using fluorescent labelled immunological stains combined with fluorescence microscopy or flow cytometry (Wallis *et al.*, 1996). Infection from all three of these protozoan pathogens can occur after consumption of food or water contaminated with the cysts or through person to person contact (Carey *et al.*, 2004). *Entamoeba histolytica* has been detected in all parts of the world, although it is more common in tropical regions (Feachem *et al.*, 1983).

## 2.6.4 Helminths

Nematodes and tapeworms are common intestinal parasites that are transmitted by the faecaloral route (Toze, 1997). Commonly detected helminths in wastewater which are of significant health risk include the roundworm (*Ascaris lumbricoides*), hookworm (*Ancylostoma duodenale*), whipworm (*Trichuris trichiura*) and *Strongloides stercoralis*. One of the major sources of helminth infections globally is the use of raw or partially treated sewage effluent and sludge for the irrigation of food crops (World Health Organisation (WHO), 1989).

# 2.7 Conclusions

The concept of human waste reuse presents an opportunity as there is potential for nutrient recycling in agriculture. This is demonstrated in the chemical composition where values for N, P and K are comparable with some of the inorganic fertilizers being used for crop production. The use of human waste has been carried out for centuries and if this was not a beneficial scheme the practice would have been phased out long ago. With the advent of technology and improvement in sanitation, human waste has faced a shift from being handled at household level to more centralised units but the challenges of disposal are still present.

Wastewater use has been extensively documented in the literature and is highly diversified in terms of their characteristics depending on the source. In the past wastewaters have mostly been viewed from a disposal perspective with very stringent policies guiding their disposal. More often wastewaters do not meet the criteria for disposal but if used as an irrigation source, the soil is able to accommodate it for plant uptake. Most studies on the impact of wastewater irrigation on soils are carried out over the long term and usually would have resulted in significant changes in the soil properties especially in terms of heavy metal accumulation. On the other hand, the impact of wastewater irrigation on soil physical properties could have an immediate effect which depends on the quality of the wastewater. However, freshwater has also been found to have adverse effects on soil properties when compared to wastewater. Such results eliminate the dispute over water quality and an understanding of the characteristics of the soil could explain such occurrences. The pathogenic component is still a major cause for concern as some are more easily detected than others. The level of contamination is a function of the degree of treatment of the wastewaters and their use depends on the type of crops to be irrigated. Some of these wastewaters contain constituents that tend to build-up in the soil or more often end up in plant parts. Plant nutrient uptake has been found to be higher when irrigated with wastewater than with freshwater which shows its potential as a nutrient source. Despite the risks involved in wastewater use there is a continued increase in wastewater irrigated agriculture especially in developing countries. In view of this it is certain that wastewater has got a place in irrigated agriculture both as a nutrient and a water source and rather than discourage its use sustainable solutions should be sought through research in order to optimise its use.

# Chapter 3

# LEACHATE CHARACTERISTICS AS INFLUENCED BY ANAEROBIC BAFFLED REACTOR EFFLUENT APPLICATION ON THREE SOILS: A SOIL COLUMN STUDY

# 3.1 Introduction

According to Bond (1998) the key limitations to sustainable soil application of most effluents are threefold namely excessive nitrate leaching to groundwater, salinity and the effects of increasing soil sodicity. Salinity and sodicity are the principal water quality concerns in irrigated areas receiving such waters (Ayers and Westcot, 1985). Phosphorus may also be a limiting factor where there are deep sandy soils promoting its downward movement and eventual enrichment of groundwater. Many countries have experienced groundwater contamination due to nitrates as a result of the application of nitrogen fertilizers and land application of nutrient rich waste (Canter, 1997). Nitrate is the most common chemical contaminant in groundwater aquifers (Spalding and Exner, 1993). It has an impact on the environment if not taken up by plants or denitrified and may end up in streams and groundwater. Studies in India have revealed the danger of nitrate leaching to groundwater and its negative effect on human health (Prakasa Rao and Pattanna, 2000). The actual impact of nitrate leaching depends on a number of factors namely depth to water table, quality of groundwater, soil drainage, soil hydraulic conductivity, scale of wastewater irrigation and agronomic practices (Khan and Hanjra, 2008).

Wastewater salinity and sodicity also play a very important role in soil physical and chemical properties. The sodium adsorption ratio (SAR) is used to define the sodicity of the soil and that of the soil solution or applied water (United States Salinity Laboratory, 1954). Sodium persists in recycled water and is very difficult to remove, usually requiring the use of cation exchange resins or reverse osmosis membranes. Such practices are for high quality water and are not practical or economic for crop and pasture irrigation and thus other management mechanisms need to be employed. The salinity of recycled water can impact on the soil as

well as the crops being irrigated (Katerji *et al.*, 2003). Sodicity can directly affect soil properties through the phenomena of swelling and dispersion (Halliwell *et al.*, 2001).

However, monitoring effluent infiltration and the replacement of the existing soil solution with fresh effluent can provide relevant short term information about such changes (Gloaguen *et al.*, 2007). Soils are generally better suited as a reservoir for wastewater than water bodies because of their ability to buffer and assimilate the water, nutrients and any other contaminants (Bond, 1998) through physical, chemical and microbial processes. An evaluation of the retention capacity of elements from effluent and the composition of leachates as a result of such additions will give a better understanding on the ability of soils to impact on effluent characteristics.

Studies by Foxon et al. (2005) have shown that an anaerobic baffled reactor (ABR) treating domestic wastewater will convert a large amount of wastewater chemical oxygen demand (COD) to methane gas, and will reduce pathogen loads in the wastewater. Despite considerable reduction of pathogen load secondary treatment is required before any conventional irrigation methods are embarked upon. However, there is no nutrient removal, and the amount of pathogens removed is insufficient to render the effluent safe for human contact. The presence of significant amounts of ammonium and phosphorus in the effluent means that it cannot be discharged to surface or groundwater but, theoretically, can be used in irrigation of agricultural land, or disposed of in a soak-away (Foxon *et al.*, 2004). Except in the case where sufficient area and infrastructure is available to build a sub-surface soak-away system, some post-treatment of the effluent is required before it can be reused. It has been recommended that the use of membrane bio-filters in conjunction with the ABR be considered since a bio-filter would remove virtually all COD and pathogens, while allowing nutrients, which have a real economic value as a fertilizer, to be retained for use in agriculture (Foxon et al., 2004). Another post-treatment option is a constructed wetland although this will likely remove much of the nitrogen and phosphorus, as well as the pathogens (Vymazal, 2007). Results from other sections of the ABR project (Foxon et al., 2005) have indicated that the effluent, at its present microbiological quality, is not suitable for irrigation of some food crops. However, the high nutrient levels of the effluent suggest that it holds potential as a fertilizing solution, if the microbial quality can be improved.

Anaerobic digestion is a biological process in which organic matter is catabolised to methane and carbon dioxide. This can be simplified into four steps as follows:

- hydrolysis-a chemical reaction where particulates are solubilised and large polymers converted into smaller monomers;
- acidogenesis- a biological reaction where simple monomers are converted into volatile fatty acids;
- acetogenesis- a biological reaction where volatile fatty acids are converted into acetic acid, carbon dioxide and hydrogen; and
- methanogenesis- a biological reaction where acetates are converted into methane and carbon dioxide while hydrogen is consumed.

Anaerobic digestion greatly minimises excess sludge production as microorganisms within the reactor gently rise and settle due to the flow characteristics, power requirements are reduced and methane gas is produced as an energy source. The ABR works under similar conditions to a septic tank but it increases contact between biomass and wastewater by forcing liquid to flow through biomass beds under the hanging baffles. In this way there is a biological filtering effect in which solid components are physically retained by settling, and liquid components are removed by adsorption and consumption. As a result, an ABR will produce a far superior effluent to a septic tank operating with a similar hydraulic retention time.

Soil column studies have frequently been used to provide information about element release and transport in soil, chemistry of soil and leachates and to carry out kinetic and mass balance studies (Grolimund *et al.*, 1996; Magesan *et al.*, 1999; Kolahchi and Jalali, 2006; Jalali *et al.*, 2008). They therefore constitute a valuable tool for the examination of the changes that take place in the chemistry of soil and in the leachates after addition of the effluent and the downward movement and distribution of nutrients through the soil profile (Egiarte *et al.*, 2006; Kolahchi and Jalali, 2007). Soil column leaching experiments are performed to evaluate the impact of wastewater use on concentrations of different cations and anions in agricultural soils and their leaching towards groundwater (Jalali *et al.*, 2008). The objectives of this study were to evaluate:

- the changes in leachate characteristics after anaerobic baffled reactor (ABR) effluent application; and
- the capacity of different soils to retain plant nutrients from the applied effluent.

# **3.2.** Materials and methods

3.2.1 Soils

# 3.2.1.1 Physical and chemical characteristics

The experiment was carried out using three contrasting soil types namely the A horizon of an Inanda (Ia; humic A, red apedal B, weathered dolerite), the E horizon of a Longlands (Lo; orthic A, E, soft plinthic B), and the A horizon of a Sepane (Se; orthic A, pedocutanic B, unconsolidated material with signs of wetness) (Soil Classification Working Group, 1991). These correspond to a Rhodic Hapludox, Typic Plinthaquult, and Aquic Haplustalf, respectively, according to the USDA Soil Taxonomy (Soil Survey Staff, 2010). The Ia was collected from World's View, Pietermaritzburg under commercial forestry; the Lo from the South African Sugar Research Institute, Mt Edgecombe previously under sugarcane then grassland for about 12 years; the Se from the permaculture site at Newlands-Mashu, near Durban. Soil was collected, air-dried and milled to pass through a 2-mm mesh prior to preparing the soil columns. Soil pH was measured in distilled water and in 1M KCl solution (1:2.5 soil:solution) (Rowell, 1994) using a Radiometer PHM 210 meter. Electrical conductivity (EC) was measured in distilled water (1:2.5 soil:solution) using a CDM 210 electrical conductivity meter. Organic carbon was determined by the dichromate oxidation method (Walkley, 1947) and particle size by the pipette method (Gee and Bauder, 1986). Other analyses were carried out by the Soil Fertility and Analytical Services Division (Department of Agriculture, Cedara) following methods given by The Non-Affiliated Soil Analysis Work Committee (1990).

The clay mineralogy of the three soils was estimated qualitatively using X-ray diffraction. Magnesium and K saturated clays separated from each soil were prepared as smear specimens as follows:

- Mg air-dry
- Mg placed in an atmosphere of ethylene glycol at 60°C for 24 h
- Mg placed in an atmosphere of glycerol at 85°C for 24 h
- K air dry
- K heated in a furnace at 550°C for 4 h.

The smear specimens were run on a Philips PW1050 diffractometer using monochromated CoK $\alpha$  radiation from 3 to 40° 2 $\Theta$  at 1° per minute scan speed with a 0.02° counting interval. Data were collected automatically by a Sietronics 122 micro-processor coupled to the diffractometer.

## 3.2.2 Column study

The columns consisted of polyvinyl chloride tubes, 20 cm long (inner diameter = 5.3 cm). The bottom of each column had a perforated perspex disc (holes of 0.8 cm diameter) of the same diameter as that of the column that was covered with nylon mesh. Glass-fibre mesh was placed on the disc before filling the column with soil to minimise sediment loss from the column during leaching. The columns were filled with soil to a height of about 17 cm by uniform tapping on the bench top to achieve a bulk density of 1.48 g cm<sup>-3</sup> for the Lo, 0.75 g cm<sup>-3</sup> for the Ia and 1.12 g cm<sup>-3</sup> for the Se soil; values equivalent to field bulk densities. Glass-fibre mesh was placed on the soil surface to minimise soil disturbance during the leaching procedure.

The pilot project site for the installation of the ABR is in the Newlands-Mashu Permaculture Centre, eThekwini Municipality, Durban, South Africa where an ABR was being constructed and connected to 80 households in a new housing development that was estimated to produce 60kL of effluent per day with the intention of using the effluent for agricultural purposes. Due to delays in the construction process the effluent used in this study was collected from an ABR located at the School of Chemical Engineering, University of KwaZulu-Natal (UKZN), Durban, South Africa, which was fed manually and produced about 100 L of effluent in 24 hours (Plate 3.1). The effluent was collected and taken to the Soil Science laboratory at UKZN, Pietermaritzburg Campus where the research was conducted.



Plate 3.1A laboratory-scale anaerobic baffled reactor at the School of Chemical<br/>Engineering, University of KwaZulu-Natal, Durban.

Soil columns (Plate 3.2) were leached with either effluent or distilled water in triplicate (total of 18 columns). Prior to leaching the columns were saturated with distilled water by capillary wetting. With an assumed particle density of 2.65 g cm<sup>-3</sup>, a pore volume for the Lo, Ia, and Se soils was calculated to be 168 mL, 270 mL and 217 mL, respectively (Rowell, 1994). Each leaching event comprised of drip flow from the top onto the columns according to the hydraulic properties of each soil which gave a flow rate of 6.4-6.5 cm hr<sup>-1</sup> for the Lo, 5.1-5.8 cm hr<sup>-1</sup> for the Ia and 1.0-1.1 cm hr<sup>-1</sup> for the Se.



Plate 3.2 Soil leaching columns laboratory set-up.

The ABR effluent was analysed for pH and EC. Major elements (N, P, K, Ca, Mg and S) and heavy metals (Fe, Mn, Cu, Zn, Cd, Cr, Co, V and Se) were analysed by inductively coupled plasma emission spectrometry (ICP, Varian 720-ES). The *Escherichia coli* count was done by plating dilutions from the column on eosin methylene blue agar plates and counting colonies formed after incubation at 35°C for 48 hours (American Public Health Association, 1992). The columns were leached with 16 pore volumes (PV) over a period of 21 weeks. Initially leaching was carried out weekly (PV 1–11); then at 2 weekly intervals (PV 12–15) with a 3 week interval to the final PV. This was equivalent to a total of 1218 mm, 1957 mm and 1573 mm water for the Lo, Ia and Se, respectively. Leachate samples from each leaching event were collected and analysed immediately for  $NH_4^+$ -N and  $NO_3^-$ -N with a TRAACS 2000 continuous flow auto-analyser, while pH and EC were measured directly and plating used for *E. coli* count. An aliquot of about 100 mL was taken and acidified with nitric acid for determination of Ca, Mg, Na, P and K by ICP-ES. A chemical balance of inorganic-N, P, K, Ca, Mg, and total C was done at the end of leaching based on their input and output concentrations.

## **3.3** Results and discussion

# 3.3.1 Soil and wastewater properties

The three soils differed in a number of important aspects which was the basis for their selection. These included particle size distribution, organic carbon and base status (Table 3.1).

The Ia is a highly weathered soil dominated in the clay fraction by kaolin, gibbsite and goethite. The Lo is composed of mostly kaolin and quartz. The abundance of quartz makes it a non-reactive soil. The Se has interstratified material predominantly vermiculite, mica, chlorite and illite.

From an agricultural perspective, the ABR effluent contains considerable amounts of plant nutrients with low concentrations of heavy metals, with most being below South African permissible limits (Table 3.2). This effluent meets the criteria for use as an irrigation source (Ayers and Westcot, 1985; Department of Water Affairs and Forestry (DWAF), 1996). The effluent belongs to salinity class C2S1 (medium-salinity/low sodicity water), enabling its use without any major salinity control measures and with little danger of developing harmful levels of exchangeable sodium as the sodium adsorption ratio (SAR) was low at 1.13 (United States Soil Salinity Laboratory Staff, 1954). The NH<sub>4</sub><sup>+</sup>-N content of the effluent was greater than the NO<sub>3</sub><sup>-</sup>-N owing to the fact that the treatment process was anaerobic. The microbiological content of the effluent as shown by the indicator organism for faecal contamination, *E. coli*, is very high.

Downwortow		So	il form <sup>a</sup> and l	horizon
Parameter		Ia A	Lo E	Se A
	( H <sub>2</sub> O)	4.51	5.72	5.80
рН	(1M KCl)	3.83	4.74	4.81
Electrical conductivity (dS m <sup>-1</sup> )		0.05	0.04	0.15
Organic C (g 100g <sup>-1</sup> )		9.60	0.14	3.65
Total N (mg kg <sup>-1</sup> )		5121	533	3036
	Ca <sup>#</sup>	0.85	2.06	10.8
	$\mathrm{Mg}^{\#}$	0.20	0.62	9.13
Extractable base cations (cmol <sub>c</sub> kg <sup>-1</sup> )	$\begin{tabular}{ c c c c c c } \hline Ia & A & Lo & E \\ \hline & (H_2O) & 4.51 & 5.72 \\ \hline & (1M & KCl) & 3.83 & 4.74 \\ \hline & 0.05 & 0.04 \\ \hline & 0.05 & 0.04 \\ \hline & 9.60 & 0.14 \\ \hline & 5121 & 533 \\ \hline & Ca^{\#} & 0.85 & 2.06 \\ \hline \end{tabular}$	0.10	0.25	
	Na	0.14	0.10	0.32
Exchangeable acidity $(\text{cmol}_{c} \text{kg}^{-1})^{\#}$		4.71	0.03	0.09
Acid saturation (%) <sup>#</sup>		79.6	1.07	0.44
Total cations (cmol <sub>c</sub> kg <sup>-1</sup> )		5.92	2.80	20.32
	Mn	16.0	23.7	28.6
Extractable metal cations $(mg kg^{-1})^{\#}$	Cu	4.40	2.23	2.50
	Zn	2.00	1.76	0.09
Extractable P (mg kg <sup>-1</sup> ) <sup>#</sup>		20.0	4.05	1.79
Particle size (%)				
Coarse sand (0.5-2 mm)		3.9	1.8	2.7
Medium sand (0.25-0.5 mm)		5.7	27.3	3.6
Fine sand (0.053-0.25 mm)		26.3	47.5	17.7
Silt (0.002-0.053 mm)		42.2	12.8	42.0
Clay (<0.002 mm)		21.9	10.6	34.0
Clay mineralogical composition (%)				
Vermiculite		*	*	**
Illite		-	*	*
Mixed-layer minerals		-	-	v-m **/ v-c
Kaolin		**	**	*
Quartz		tr	**	*
Feldspar		tr	tr	tr
Goethite		*	*	tr
Anatase		-	tr	tr
Gibbsite		**	-	-

**Table 3.1**Characterisation of the Inanda, Longlands and Sepane soils used for the<br/>column experiment

<sup>a</sup> Inanda (Ia), Longlands (Lo), Sepane (Se) (Soil Classification Working Group 1991).

<sup>#</sup> Analysis conducted by the Soil Fertility and Analytical Services Division (KwaZulu-Natal

Department of Agriculture, Cedara).

\*\* 20-60%; \* 5-20%,; tr < 5%; - not found, v vermiculite; m – mica; c - chlorite.

Parameter	Effluent	Water
Electrical conductivity(dS m <sup>-1</sup> )	0.641	0.003
pH	7.60	6.24
Elements (mg L <sup>-1</sup> )		
Nitrate-Nitrogen	bd*	bd
Ammonium- Nitrogen	14.3	bd
Phosphorus	25.2	bd
Potassium	8.55	0.14
Sulphur	6.6	bd
Calcium	18.9	0.37
Magnesium	26.3	bd
Sodium	32.5	0.26
Aluminium	0.08	bd
Cadmium	bd	bd
Cobalt	bd	bd
Chromium	0.01	0.01
Copper	bd	bd
Iron	0.28	bd
Manganese	0.003	bd
Molybdenum	0.004	0.006
Nickel	0.009	bd
Lead	0.03	0.08
Selenium	0.06	0.02
Vanadium	0.01	0.005
Zinc	bd	0.003
Boron	bd	0.03
Bicarbonate	247	3.05
Chloride	36	1.41
Total carbon	21.4	1.3
<i>E.coli</i> count- colony forming unit (cfu mL <sup>-1</sup> )	$7.5 x 10^4$	bd
Sodium adsorption ratio (SAR) *bd - below detection	1.13	0.14

**Table 3.2**Chemical and *Escherichia coli* (*E. coli*) composition of the anaerobic baffled<br/>reactor effluent and distilled water

\*bd - below detection

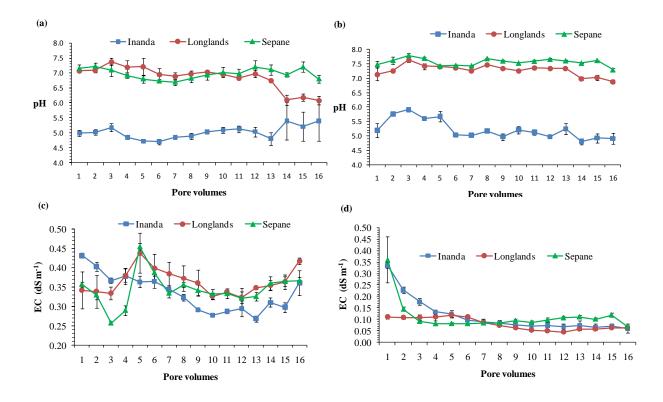
# 3.3.2.1 pH and electrical conductivity (EC)

The pH of the leachates from the Ia soil were initially similar to that of the soil and gradually increased with each leaching event but remained lower than the pH of the original leaching solution (pH 7.60) for the effluent-leached columns after 16 pore volumes (pH 5.40) (Figure 3.1a). However, this was not the case with the Se and Lo soils with final leachate values of pH 6.80 and pH 6.10, respectively, for the effluent-leached columns after 16 pore volumes. It is worthwhile noting that the effluent leachate pH values for these two latter soils were not affected as much as was the case with the Ia.

The observed trend in the Ia is likely a result of the acidic nature of the soil (pH 4.51) as a similar trend was observed for the distilled water leachates (Figure 3.1b). A liming effect of the effluent also becomes apparent in the case of the Ia soil with time as evident in the gradual increase of the leachate pH with pore volume that was not observed when leaching with distilled water.

With regards to the electrical conductivity (EC), the trend was quite different as leachates from soils peaked towards the value of the original effluent at pore volume 5 except the Ia (Figure 3.1c) with the Longlands and Sepane recording EC values close to 0.5 dS m<sup>-1</sup>. The EC for the distilled water leachates consistently decreased with pore volume owing to the fact that the soil solution was becoming more dilute with most of the ions being leached out of soil (Fig. 3.1d).

Anions like sulphate  $(SO_4^{2-})$ , chloride  $(CI^-)$  and nitrate  $(NO_3^-)$  are generally mobile in soil because the anion exchange capacity is much lower than the cation exchange capacity so when anions are leached cations like Ca and Mg are also leached thus diluting the soil solution (Nunez-Delgado *et al.*, 1997). The EC of the leachates from the effluent columns did not show a sharp decline as was the case with the water leachates showing that ions from the effluent were retained in the soil despite the leaching.



**Figure 3.1** The mean pH of (a) effluent, (b) water and mean electrical conductivity (EC) of (c) effluent, (d) water of leachates from soil columns (± SE; n=3).

# 3.3.2.2 Leachate concentrations of major plant elements

Nitrogen was measured as the inorganic fraction ( $NO_3^-N$  and  $NH_4^+-N$ ). Nitrate-N was undetected in the incoming effluent but gradually increased with pore volume in the leachate from all soils leached with effluent (Figure 3.2). The  $NH_4^+-N$ , on the contrary, decreased in the effluent leachates from all soils and was undetectable in the Se soil as was the case also with the water-leached columns. The nitrate concentration in the effluent by the end of the experiment (17.2 mg L<sup>-1</sup>) was comparable with the ammonium concentration in the original effluent at the beginning of the experiment (14.3 mg L<sup>-1</sup>). It is likely that that the  $NH_4^+-N$  in the effluent was being converted into the nitrate form rather than the increase coming from the inherent N in the soil because this would have also been observed from the distilled water-leached columns. The  $NO_3^--N$  being higher in the percolating solution than the  $NH_4^+-N$ is indicative of the nitrification process which produces  $H^+$  ions (Egiarte *et al.*, 2006). An increase in acidity, however, was apparently counteracted by the presence of Ca and Mg ions in the effluent, suggesting a liming effect from the effluent. There was an absence of  $NH_4^+-N$  in the Se leachates from both the effluent-leached and water-leached columns. For the effluent-leached columns this can be explained by the conversion of the  $NH_4^+$ -N to  $NO_3^-$ -N. In the water-leached columns the absence of  $NH_4^+$ -N in the incoming water explains its absence in the leachates (Figure 3.2). The Se soil has got a finer texture resulting in a low hydraulic conductivity relative to that of the Ia and Lo (Section 3.2.2). This promotes interaction between the soil and incoming solution. Sandy soils with low cation exchange capacities permit appreciable movement of  $NH_4^+$ -N through the soil. However, this was not the case with the Lo soil as  $NH_4^+$ -N was absent from the leachates in the latter stages of leaching suggesting that it was converted to  $NO_3^-$ -N (Figure 3.2). The  $NO_3^-$ -N and  $NH_4^+$ -N in leachates from the water-leached columns decreased consistently, except for  $NO_3^-$ -N in the Lo soil, as leaching progressed. Nitrate-N is very mobile in soil and the sandy texture of the Lo soil could have aided its leaching from the soil. The trend in the water-leached columns was such that the initial leachate concentration of these ions was high then a sharp decrease occurred before levelling off except for the  $NH_4^+$ -N in the Se soil as explained earlier.

Phosphorus concentrations in all leachates were low irrespective of soil type (Figure 3.2) owing to the immobile nature of P in soils. Phosphorus was retained even in the Lo despite its low clay content of only 10%. The low P concentrations in the leachates could be attributed to different properties of the respective soils. The predominance of Fe and Al oxides and hydroxides in the Ia was responsible for P retention. The type of clay mineralogy has also been found to be responsible for P retention. Non-significant differences have been found in P retention by kaolinite and vermiculite under alternate wetting and drying conditions (Zia et al., 1992). Retention of P in the Lo soil was not expected but could be a function of the presence of goethite in this soil (Table 3.1). Also, Djodjic et al. (1999) found that more P leached from clay soils than sandy soils when labelled <sup>33</sup>P was added to soil lysimeters which contradicts this study. Preferential flow which is a process where only a small fraction of the available pore space is used to move water and solutes has been proposed as a possible cause for P leaching in clayey structured soils (Beven and Germann, 1982). Most of the water tends to move in cracks and channels from decayed root material. Contrary to this, piston flow was likely the main mode of water movement in the sandy soil which was related to its hydraulic conductivity. With the water-leached columns it could simply be that distilled water contained no P and since leaching was intermittent very little P could be leached out of the soils.

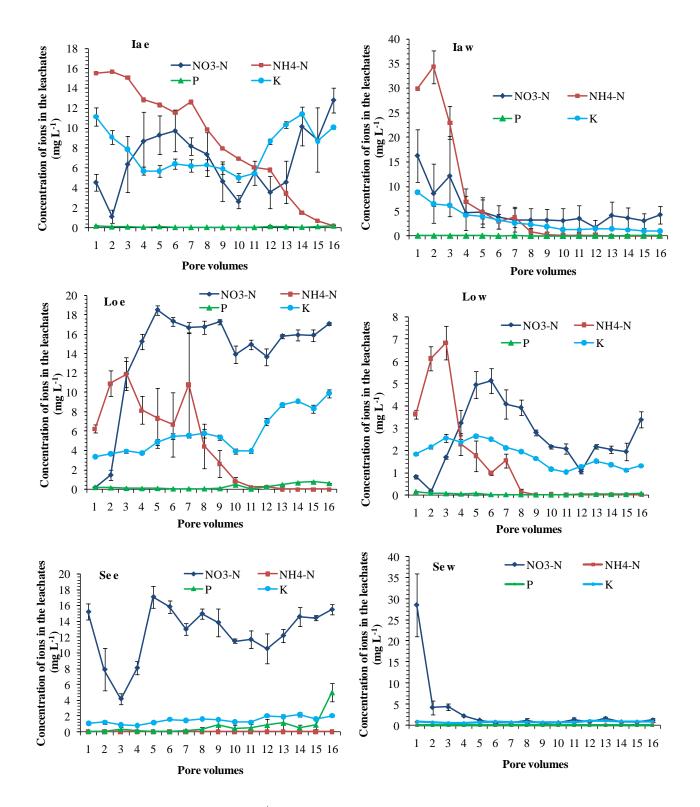
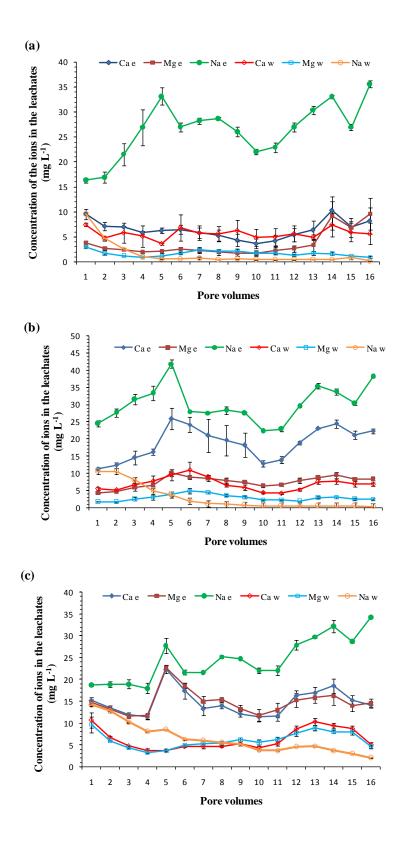


Figure 3.2 Concentration of NH<sub>4</sub><sup>+</sup>-N, NO<sub>3</sub><sup>-</sup>-N, P and K ions from Inanda (Ia), Longlands (Lo) and Sepane (Se) soils leached with (e) effluent or (w) distilled water (± SE; n=3).

Potassium concentration in the leachates from the effluent-leached columns showed a similar trend to the NO<sub>3</sub><sup>-</sup>-N except in the Sepane (Figure 3.2). The K concentration in leachates from the Ia and Lo soils was close to that of the original effluent (8.55 mg L<sup>-1</sup>) by pore volume 16. The K in leachates from the Se soil maintained a constant and low concentration throughout the leaching process. Potassium retention in the Se was apparent which could be linked to the clay type (vermiculite). Vermiculite is associated with K retention particularly in a dry-wet cycle in soil (Chittamart *et al.*, 2010). In the water-leached soils, the concentration of K in leachates decreased as initial leachates had higher concentrations and with no added K there was a progressive decrease with leaching. The Lo recorded a higher K concentration than Se and Ia in the leachate with very little differences between PV 1 and 16.

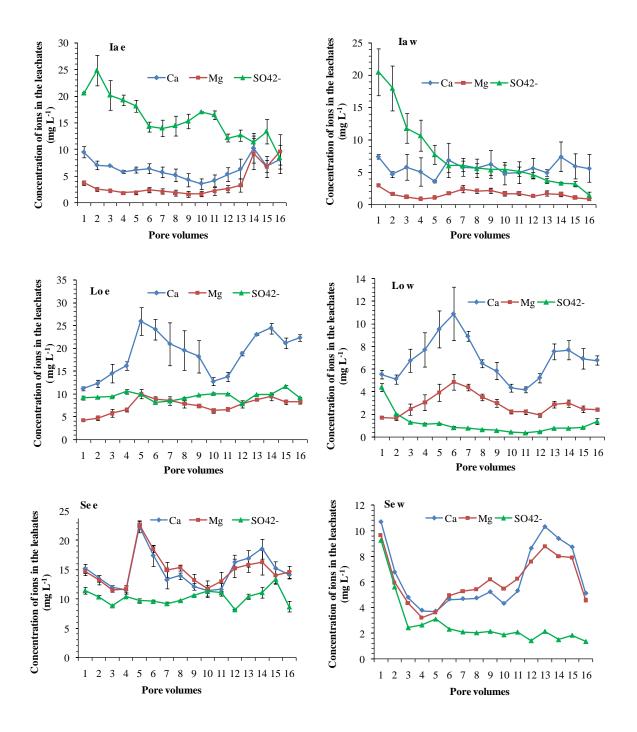
The concentrations of Ca and Mg in relation to Na are shown in Figures 3.3 a, b and c for the Inanda, Sepane and Longlands, respectively. The Na leached out of all soils unlike Ca and Mg in the effluent-leached columns. The soils showed a preferential adsorption of the divalent cations with Mg being retained more than Ca. The only exception was in the Lo where Ca was almost equal to the incoming effluent in the final pore volumes. It is unlikely that soil degradation would occur in these soils as most of the Na was leached out. Preferential adsorption of Mg rather than Ca is unusual in most soils. When Ca dominates the soil solution it limits Mg uptake on cation exchange sites (Howe and Wagner, 1999) suggesting that, in this case, the higher Mg (26.3 mg L<sup>-1</sup>) concentration in the incoming effluent compared to Ca (18.9 mg  $L^{-1}$ ) could have caused a reverse reaction resulting in the trend observed. Avers and Westcot (1985) reported that a Ca:Mg ratio of less than 1 in irrigation water would cause Mg retention in preference to Ca in soils, although this has not been extensively evaluated. Laurenson et al. (2010) carried out leaching studies in soil columns with soils from South Australia. They used municipal wastewater with a Ca:Mg ratio of 0.68:1 and found similar results which they attributed to redistribution of exchangeable and soluble cations to equilibrate the low Ca:Mg ratio in the wastewater.

The water-leached columns showed more Ca being leached than Mg but to a lesser extent then in the effluent-leached columns (Figures 3.3a, b and c). Intermittent leaching could contribute to the ion dynamics thus creating an abnormal leaching pattern. Ion release in a wet-dry situation tends to be in pulses rather than continuous promoting an irregular leaching pattern. In the water-leached columns most of the Na was leached out at the initial stages indicating again its rapid displacement from the soil.



**Figure 3.3** Concentration of calcium (Ca), magnesium (Mg) and sodium (Na) in leachates from (a) Inanda (b) Longlands and (c) Sepane soils leached with effluent (e) and (w) distilled water (± SE; n=3).

In comparing the Ca and Mg outflux to that of the  $SO_4^{2-}$  (Figure 3.4) there seemed to be no defined relationship between the Ca and Mg with the  $SO_4^{2-}$ . The amount of Ca and Mg in the leachates from the Se columns was similar with the leaching following the same trend both for the effluent and water columns.



**Figure 3.4** Concentrations of Ca, Mg and  $SO_4^{2-}$  in leachates from (Ia) Inanda, (Se) Sepane and (Lo) Longlands soils leached with (e) effluent and (w) distilled water.

The leaching of the sulphate ion from the effluent-leached columns showed that initial soil  $SO_4^{2-}$  was very high in the Ia compared to the Se and Lo. This was due to the acidic nature of the Ia resulting from organic matter in the Ia. The  $SO_4^{2-}$  concentration gradually decreased to approximately 8 mg L<sup>-1</sup>  $SO_4^{2-}$ , close to the concentration in the incoming effluent. This also shows that the leaching of  $SO_4^{2-}$  was not associated with Ca and Mg. In the water-leached columns, the  $SO_4^{2-}$  continuously decreased with most of it leached at the initial stages. The effluent contained more Cl<sup>-</sup> than  $SO_4^{2-}$  so Cl<sup>-</sup> was most likely the mobile anion associated with Na followed by the  $NO_3^{-}$ .

### 3.3.2.3 Elemental balances

The chemical balances of macro-elements of significant importance to plant growth are given in Table 3.3 reported as a function of the volume of incoming and outgoing solutions. Final concentrations gained or lost for each soil indicated that the effluent-leached columns gained nutrients while the water-leached columns had a negative balance resulting in loss of nutrients. The element which showed a remarkable gain was P and this could be attributed to its immobile nature in soil and its ability to be easily retained resulting in an accumulation (Chardon *et al.*, 2007). The amount of P gained was in the order Ia > Se > Lo. Availability of phosphorus varies with pH, clay type and/or amount and presence of sesquioxides. The Ia being an acidic soil contains high amounts of Fe and Al that easily fix P which explains its retention in this soil. The clay content of the Se soil was probably responsible for P retention. Retention of P by the Lo was unexpected considering the sandy nature of the soil (76.6%). Such retention could be due to a combination of the clay (10%) and the increase in the carbon content as a result of the addition from the leaching solution playing a role in the retention process. Carbon was lost from the Ia soil columns leached with both effluent and water. The C contribution from the effluent was negligible compared to the inherent C and with addition of the leaching solutions, the water soluble organic C was likely leached out. Translocation of organic carbon during leaching is achieved by degradation of insoluble organic to water soluble organic compounds, desorption or diffusion and convection flow (Cao et al., 1999). Comparing P and C leaching by pore volume it was observed that P in the leachates was very low and the C concentrations from both the water and effluent-leached columns had values close to one another at the initial stages of leaching. Final concentrations, however, show that there was a large gain in P compared to C for each soil except in the Ia where an inverse

relationship existed between P and C. In terms of nutrient requirements of maize (Table 3.3), the P gained from the effluent could sustain a maize crop provided it remained in the available form.

Inorganic-N showed a different trend with the gain decreasing in the order Se > Ia > Lo (Table 3.3). The gain was mainly driven by the NH<sub>4</sub>-N as most of the NO<sub>3</sub>-N was leached from columns. The NH<sub>4</sub>-N was totally absent in the leachate from both the effluent and the water in the Se columns. One of the possible fates of NH<sub>4</sub><sup>+</sup> in soils is its fixation by clays (Zhang *et al.*, 2007). This occurs by the replacement of NH<sub>4</sub><sup>+</sup> for interlayer cations in the expanded lattice of clay minerals. The fixed NH<sub>4</sub><sup>+</sup> can itself be replaced by cations which expand the lattice (Ca<sup>2+</sup>, Mg<sup>2+</sup>, Na<sup>+</sup>, H<sup>+</sup>) but not by those that contract it (K<sup>+</sup>, Rb<sup>+</sup>, Cs<sup>+</sup>). The cationic nature of NH<sub>4</sub><sup>+</sup> permits its sorption and retention by soil colloidal material. It is necessary for the soil to have a sufficiently high cation exchange capacity (Se soil) to retain the added NH<sub>4</sub><sup>+</sup> or it will be removed in percolating water. In the Se, isomorphous substitution could also account for the NH<sub>4</sub><sup>+</sup> retention in the vermiculite interlayer (Schaetzl and Anderson, 2005). The Lo soil also retained an appreciable amount of NH<sub>4</sub><sup>+</sup> as a proportion of input amount from the effluent. In meeting the N demands of maize, the inorganic-N retained would not be able to supply N adequately in the case of the Ia and Lo soils but could just meet the need with the Se soil.

		Inan	ıda	Longl	Longlands		Sepane		
		effluent	water	effluent	water	effluent	water		
	In-N	163	-378	35.4	-78.6	206	-76.5		
	Р	827	0.4	511	0.1	649	0.3		
Total loss or gain (kg ha <sup>-1</sup> )	К	42.3	-87.6	107	-29.5	245	-14		
-	Ca	543	-178	103	-130	230	-156		
	Mg	760	-53.2	398	-55.4	327	-154		
	total C	-938	-1112	106	-322	244	-412		
Nutrient	In-N	20	0	20	0	200	)		
requirements of irrigated maize	Р	20	)	80	)	60			
$(\text{kg ha}^{-1})^*$	Κ	20	5	10	0	10			

**Table 3.3**Inorganic-N (In -N), P, K, Ca and Mg and total C gain or loss in soils treated<br/>with either effluent or distilled water

\*Soil Fertility and Analytical Services Division (Department of Agriculture, Cedara, KwaZulu-Natal).

For potassium the gain was in the order Se > Lo > Ia. The same mechanism responsible for  $NH_4^+$  retention in the Se was attributed to that of K. The reversal in the Ia and Lo is perhaps due to the high organic matter content of the Ia since this has been found to decrease K fixation by inorganic colloids (Olk and Cassman, 1995). The clay fraction of the Ia is also dominated by highly weathered clays with limited sorption capacity.

# 3.4 Conclusions

The use of ABR effluent has the potential to improve the nutrient status of soil without adversely affecting soil properties. The effluent added P, N and K to all three of the soils studied although the increase in amount of each element varied depending on the properties of the particular soil. The heavy metal fraction of the effluent was below permissible limits for wastewater use for agricultural purposes. However, there is a need to monitor build-up in soil over time and also the plant's ability to absorb the elements retained in soils. As anticipated, the distilled water supplied very little nutrient input to the soils but rather leached out most of the nutrients. Irregular patterns were observed during the course of leaching showing that the ionic exchange processes in the soils followed an irregular time course. This indicates that the adsorption/leaching process occurs in pulses when leaching is not continuous (Nunez-Delgado et al., 1997). Leachate volumes collected were always about 10-15 mL less than the volumes of leaching solution added due to intermittent leaching which could account for cumulative build-up as leaching progressed. Major elements in effluent are greatly affected when applied to soil. Their concentrations in leachates are determined by soil type and also the ratio with respect to one another. Of utmost importance in the application of wastewater to soil is the Ca:Mg ratio. The greater concentration of Mg than Ca was shown to have resulted in more Mg than Ca being retained in soil which is contrary to the natural situation in soil. Excess Mg in irrigation water or soil can negatively affect soil infiltration and hydraulic conductivity. In the medium to long term, the preferential adsorption of Mg to exchange sites will have implications for the structural stability of irrigated soils (Vyshpolsky et al., 2010). The disposal of ABR effluent as a nutrient and water source may impact on soil properties although Ca can be added to mitigate the excess effects of Mg.

# Chapter 4

# EFFECT OF ANAEROBIC BAFFLED REACTOR EFFLUENT APPLICATION ON DISTRIBUTION AND FORMS OF MAJOR PLANT NUTRIENTS IN THREE CONTRASTING SOILS

# 4.1 Introduction

The use of anaerobic baffled reactor (ABR) effluent has been shown to improve the nutrient status of soils (Chapter 3). It is, however, important to investigate the fate of these nutrients in soil, to know the forms in which they are found and if they are readily available for plant uptake. It is also critical to assess the level in the column where accumulation takes place as this is indicative of nutrient movement and leaching through the soil. Availability and mobility of elements in a soil can depend on many factors amongst which are the soil surface characteristics and soil-metal interactions that affect sorption reactions (Sparks, 2003). Other interactions include complexation with either organic or inorganic species in soil (Vulkan *et al.*, 2002) while soil pH influences chemical speciation and solubility (Lindsay, 1979).

Environmental studies using soil analysis are often based on leaching and extraction procedures (single or sequential extractions) which enable broader forms or phases to be measured such as the bioavailable form which is critical for purposes of environmental policy (Rauret *et al.*, 1999).

The addition of nutrients by irrigating with ABR effluent necessitates therefore:

- $\triangleright$  an evaluation of the various fractions in terms of their availability; and
- $\rightarrow$  an assessment of the mobility and redistribution of nutrients within the soil column.

These are the primary aims of this part of the investigation.

## 4.2 Materials and methods

At the end of the leaching process described in Chapter 3, the columns were allowed to drain. The soil was pushed out and cut into 2 cm depth segments. Soil samples from the 0-2, 8-10 and 14-16 cm segments were taken for analysis to represent the top, middle and bottom parts of the column. Two columns per treatment were used and samples were analysed for pH and electrical conductivity (Section 3.2.1). A 2M KCl extraction was done (Rowell 1994) and extracts analysed by a continuous flow autoanalyser (TRAACS 2000) for inorganic-N (NH<sub>4</sub><sup>+</sup>-N and NO<sub>3</sub><sup>-</sup>-N). Fractionation for the various forms of P, Ca, Mg and K in the soil segments was done following an improvement of the Standard Measurements and Testing Programme (SM & T, formerly BCR) of the European Union (Rauret *et al.*, 1999). Analytical results were subjected to analysis of variance (ANOVA) using Genstat 12th edition and separation of means by the Tukeys procedure at the 5% level of significance.

# 4.2.1 Sequential extractions

Sequential extractions are procedures carried out to characterise pollution sources as well as to evaluate metal mobility and/or bioavailability (Filgueiras *et al.*, 2002). These extractions identify binding sites for assessing metal accumulation and transport mechanisms. The modified BCR 3-step extraction procedure involved acetic acid (step 1), acidified hydroxylamine-pH 2 (step 2), and hydrogen peroxide and ammonium acetate (step 3) corresponding to the acid soluble (AS), reducible (RE) and oxidizable (OX) fractions, respectively. An aqua regia digestion (step 4) is usually recommended to extract the residual (RS) fraction (Pueyo *et al.*, 2003) and was carried out in this study. The reagents used and extracting conditions are given in Table 4.1.

The acid soluble fraction contains elements which are precipitated or co-precipitated as carbonates (Clevenger, 1990). The exchangeable elements are also extracted within this fraction (Hernández-Moreno *et al.*, 2007). This fraction often constitutes a small percentage of total concentration of elements and can be significantly modified by moisture content (Bordas and Bourg, 1998; Emmerson *et al.*, 2000).

The reducible fraction is that bound to Al, Fe and Mn hydrous oxides (Stone and Droppo, 1996). The mechanisms by which this takes place are either one or a combination of the following: co-precipitation, adsorption, surface complex formation, ion exchange and penetration of the lattice.

The oxidizable fraction is that associated with organic matter. Degradation of organic matter under oxidising conditions might release metals bound to it (Clevenger, 1990). The hydrogen peroxide used does not totally destroy the organic matter and sulphides are only partially dissolved (Tessier *et al.*, 1979).

The residual fraction constitutes metals found in the crystalline lattice of primary and secondary minerals. They can only be destroyed by the use of strong acids, such as HF, HCl,  $HNO_3$  and  $HClO_4$  (Gleyzes *et al.*, 2002).

The acid soluble fraction is considered to be readily available to plants while the reducible and oxidizable fractions are relatively more stable if soil conditions remain unchanged.

	1		e	
Stop	Descenta	Volume	Temperature	Extraction time
Step	Reagents	(mL)	$(^{\circ}C)$	Extraction time
1	0.11 M CH <sub>3</sub> COOH	20	$22 \pm 5$	Shaking for 16 h (overnight)
2	0.5 M NH <sub>2</sub> OH.HCl	20	22 ± 5	Shaking for 16 h (overnight)
	acidified with 2 M HNO <sub>3</sub>	-	-	
3	8.8 M H <sub>2</sub> O <sub>2</sub>	5	$22 \pm 5$	Digest for 1 h (occasional
5	$0.0 \text{ WI } \Pi_2 O_2$	5	$22 \pm 5$	manual shaking)
		5	$85 \pm 2$	Digest for 1 h
	1 M NH <sub>4</sub> OAc pH 2	25	$22\pm5$	Shaking for 16 h (overnight)
4	Aqua regia (ISO 11466		$22 \pm 5$	Digest for 16 h
	protocol)		$22 \pm 5$	Digestion to h
	HCl (37%): HNO <sub>3</sub> (70%)	4	$130 \pm 2$	
	3:1	+	150 ± 2	Digest for 2 h (under reflux)
* D	(1000)			

**Table 4.1** Modified BCR protocol\* for extracting elements from soil

\* Rauret et al. (1999).

## 4.3 **Results and discussion**

#### 4.3.1 Plant nutrients retained from effluent and water

The difference in volume between the leaching solution and the leachate collected at each pore volume was considered as the volume of solution retained in the soil. The total nutrients retained were calculated as a function of this volume. Leaching occurred periodically and so a wetting and drying scenario characterised the leaching process and at the end the total amount of nutrients retained or lost was calculated (Table 4.2). The Ia soil had the highest retention for P, Ca and Mg. and the lowest for K. Leaching solutions were added as pore volumes (Section 3.2.2) and by calculation the total volume retained was 295 mL, 258 mL and 302 mL for the Ia, Lo and Se, respectively. In spite of the small difference in the volumes retained in the Se and Ia there were very large differences in nutrient composition showing the impact of the soil properties on the leaching solution. The hydraulic conductivity of the soils suggests that there should be small differences in solution retention especially between the Ia and Lo (Section 3.2.2) but the amounts of elements retained were dissimilar. The chemical and mineralogical compositions of the soils were different and were bound to affect the leaching solutions differently. For instance P was strongly bound by the sesquioxides while the Ca and Mg were used to raise pH in the Ia soil. The water-leached columns were generally negative and so nutrients were lost, not gained.

Element (ma $l_{a}^{-1}$ )	Inanc	la A		Longlar	nds E	Sepane A		
Element (mg kg <sup>-1</sup> )	effluent	water	_	effluent	water	effluent	water	
In-N	72.4	-168.2*	_	7.9	-17.7	60.9	-22.6	
Р	367.3	0.18		115.2	0.02	191.4	0.1	
K	18.8	-38.9		24	-6.6	72.3	-4.2	
Ca	241.4	-79.2		23.2	-29.4	67.8	-46.1	
Mg	337.7	-23.6		89.7	-12.5	96.4	-45.3	

 Table 4.2
 Inorganic-N (In-N), P, K, Ca and Mg amounts retained from the leaching solutions after sixteen pore volumes

\*negative values indicate amount lost from soil.

Table 4.2 is similar to Table 3.3 in that they both express elements retained. In Table 4.2, the amounts have been expressed per kilogram of soil and in Table 3.3 these amounts were

further converted into a per hectare basis in order to compare them with fertilizer recommendations.

# 4.3.2 pH, EC and inorganic-N in soil after leaching

There was a significant difference (p<0.05) between the pH<sub>H2O</sub> of the effluent and the water column segments for the Ia except the 14-16 cm segment of the effluent column which was not significantly different from the equivalent water column segment (Table 4.3). An increase of 1.28, 0.78 and 0.27 pH units was detected for the 0-2, 8-10 and 14-16 cm segments, respectively, for the Ia effluent columns as compared to the water columns. Within both effluent and water columns, there were no significant differences between the segments. For pH<sub>KCl</sub> (Table 4.3) there were no significant differences between the water column segments but the 0-2 cm segment was significantly higher than the other segments in the effluent columns and also from the water column segments in the Ia.

In the Lo, there were no significant differences between segments within the leaching solutions but the 0-2 cm segment for the effluent column was significantly higher than the top two segments of the water columns with a difference of 1.05 and 0.93 pH units, respectively, for the  $pH_{H2O}$ . For the  $pH_{KCl}$  the 14-16 cm segment was significantly different from the top two segments of the water columns while for the effluent columns, the 0-2 cm segment was significantly higher than the middle and bottom segments.

In the Se, there were no significant differences (p<0.05) between the effluent and water column segments with regards to  $pH_{H20}$ . With the  $pH_{KCl}$ , the effluent column segments were not significantly different from one another but a significant difference was noticed between the 0-2 cm segment of the effluent column and the two top segments of the water columns with a difference of 0.57 and 0.39 pH units, respectively.

The marked change in pH of the effluent columns compared to the water columns, especially in the Ia, suggests again the potential of the effluent as a liming agent. Generally for all soils, both  $pH_{H2O}$  and  $pH_{KCl}$  were lower in the top segment and higher in the bottom segment in the water columns while the reverse was the case in the effluent columns. This increase in pH in the top part of the soil shows that cations from the effluent may have caused displacement of  $Al^{3+}$  and  $H^+$  ions from exchange sites causing a reduction in exchangeable acidity or simply by an accumulation of calcium and magnesium from the effluent (Treder, 2005).

The EC showed an irregular pattern (Table 4.3) but was higher in the effluent than the water columns in all segments for all soil types. The 0-2 cm segment of the effluent columns was significantly different from all the water column segments except in the Lo soil. Within the effluent-leached columns, it was only the Ia 0-2 cm segment that was significantly different from the other segments. The nature of the Lo (sand) could promote downward movement of ions throughout the column accounting for the non-significance between segments and the even EC within the column.

There were no significant differences (p<0.05) in the inorganic-N (In-N) between the soil segments from both the water and effluent-leached columns in the Ia except in the 14-16 cm segment in the water-leached columns (Table 4.3). The In-N was below detection limits in the Se (0-2 cm) and all the Lo segments for the water-leached columns. There was equally no significant difference in In-N between the column segments both for water and effluent columns for Se and Lo (Table 4.3). This could be attributed to the rapid percolation of NO<sub>3</sub><sup>-</sup>-N and the conversion of NH<sub>4</sub><sup>+</sup>-N to nitrate which was shown by Egiarte *et al.* (2006) when using an anaerobic municipal sludge in an acid soil.

# 4.3.3 Fractionation of initial soils before leaching

Separation of the three soils into fractions (Table 4.4) showed that phosphorus generally followed a trend where values increased from the AS to the RS fractions for all soils. Potassium in the Ia soil showed a slightly different trend with AS fraction being high, decreasing considerably in the RE fraction and then increasing for the remaining fractions. In the Se the RE fraction was higher than the AS then it decreased in the OX fraction before increasing by an order of magnitude in the RS fraction. Potassium in the Lo had similar values for all fractions except the RS fraction where there was again an order of magnitude increase. Calcium was highest in the AS fraction of the Ia, decreased in the RE and OX fractions and increased in the RS. The same trend was followed in the Se and Lo except that the Se had much higher amounts of Ca in the AS and RE fractions compared to the OX and RS.

Soil form*	Soil depth (cm)				Leachi	ng solution			
			eff	luent				water	
		pH (H <sub>2</sub> O)	pH (1M KCl)	EC (dS m <sup>-1</sup> )	$\frac{\text{In-N}}{(\text{mg kg}^{-1})}$	pH (H <sub>2</sub> O)	pH (1M KCl)	$\frac{\text{EC}}{(\text{dS m}^{-1})}$	In-N $(mg kg^{-1})$
	0-2	5.57bcd	4.67def	0.27g	30.0cd	4.29a	3.82a	0.08abcde	26.1bcd
Inanda (Ia)	8-10	5.41bc	4.21bc	0.12cde	49.8d	4.63a	3.93ab	0.07abcd	28.3cd
	14-16	4.91ab	3.98ab	0.14ef	30.2cd	4.64a	3.92ab	0.07abcd	17.5abc
	0-2	6.84g	6.03k	0.08abcde	2.00ab	5.79cde	4.45cd	0.02a	bd**
Longlands (Lo)	8-10	6.30efg	5.15hi	0.05abc	1.40a	5.91cdef	4.61de	0.02a	bd
	14-16	6.29efg	5.05ghi	0.05abc	2.50ab	6.39efg	5.04fghi	0.02a	bd
	0-2	6.52fg	5.32ij	0.13de	6.70abc	6.13def	4.75defg	0.04ab	bd
Sepane (Se)	8-10	6.37efg	5.00fghi	0.08abcde	0.60a	6.31efg	4.93efgh	0.04ab	8.3abc
	14-16	6.54fg	5.22hi	0.09bcde	1.60ab	6.54fg	5.13hi	0.05abc	13.9abc

**Table 4.3**Mean values of pH, electrical conductivity (EC) and inorganic-N (In-N) for column segments after leaching (n=2)

\* Soil Classification Working Group (1991).

<sup>#</sup> Values within soils in column followed by the same letter are not significantly different (p<0.05).

\*\*bd- below detection.

Magnesium was higher in the AS fraction then decreased in the RE fraction before increasing in the last two fractions for Ia and Lo. In the Se, Mg continued to decrease into the OX fraction before increasing in the RS fraction.

From the above extractions, it can be observed that phosphorus is an element that is strongly retained in soil (Johnson *et al.*, 2003) since inorganic phosphate concentrations in the readily-extractable fractions were low. The main mechanism controlling P release in soil is the Fe and Al oxides especially at low pH and taking into account the mineralogy of the three soils (Table 3.1) it is most likely that less of the soluble form of P will be present in the Ia which was shown in the analysis (Table 4.4). The low K status of the Ia was reflected in the residual K which comes from the inability of the dominant kaolin in the Ia clay to fix K and also the higher soluble K compared to the Se and Lo as it could be easily leached out of the soil.

Soil form*	Fraction*		Element (	Element (mg kg <sup>-1</sup> )			
		Р	К	Ca	Mg		
	AS	3.07	113	338	60.3		
Inanda	RE	4.16	22.6	84.7	17.3		
(Ia)	OX	72.9	148	49.7	37.1		
	RS	225	183	154	146		
	AS	5.57	26.7	448	92.4		
Longlands	RE	15.3	25.2	85.2	26.5		
(Lo)	OX	272	26.3	36.8	35.2		
	RS	279	245	242	145		
	AS	3.92	72.15	1672	895		
Sepane	RE	3.86	102	1398	490		
(Se)	OX	86.2	60.2	96.8	135		
	RS	539	643	218	786		

**Table 4.4**Fractionation of soils used for the column leaching experiment

\* Soil Classification Working Group (1991)

<sup>#</sup> AS- acid soluble, RE- reducible, OX- oxidizable, RS- residual.

# 4.3.4.1 Acid soluble fraction

There were marked differences between the P concentrations in the soil segments for the effluent and water-leached columns (Figure 4.1a). In all soils, the 0-2 cm segment had a significantly (p<0.05) higher concentration of P than the middle and bottom sections for the effluent columns suggesting a build-up of P in the top section from the incoming effluent. This is not an unexpected occurrence in the Ia as the immobile nature of P in soils coupled with the presence of iron and aluminium oxides that tend to retain P explains the lack of movement down the soil column. The unusual build-up in the Lo which was almost equal in the 0-2 cm segment to that in the Ia was unpredicted due to its sandy texture. This accumulation could perhaps be attributed to the clay content and to a lesser extent to complexation. The coarser texture of the Lo soil. The P content in the 0-2 cm segment of the Se was mainly as a result of the higher clay content of the Se (34%) thus preventing its movement to the lower segments of the column. Contrary to the effluent columns, the water columns showed no significant differences between column segments for all soils as the P contents for the segments were very low compared to the effluent columns.

For K, the Ia and the Se (0-2 cm) showed significant differences between the effluent and water-leached columns (Figure 4.1b). Similar to P, the K content was higher in the top than the lower segments for Ia and Se in the effluent-leached columns. In the Lo the K trend was similar to inorganic-N which may be as a result of the low K contribution from the effluent coupled with the inherently low K status of the Lo compared to the Se soil. Potassium content in the water-leached columns was in the reverse order with lower segments having the higher content showing that K was being moved down the column since none was added with the water.

Calcium and Mg (Figures 4.1c and d) followed similar trends to the K for the water-leached columns. For the effluent-leached columns, Ca in the 0-2 cm segment was significantly greater than in the middle and bottom sections only in the Ia and in both Ia and Se for Mg. The greater response to Ca and Mg addition from the effluent in the Ia soil was reflected in

the pH change in the 0-2 cm segment. As a result the movement of these elements to the lower segments was minimal. The amounts of Ca and Mg in the water-leached columns were greater in the lower segments which is suggestive of their being lost from the column.

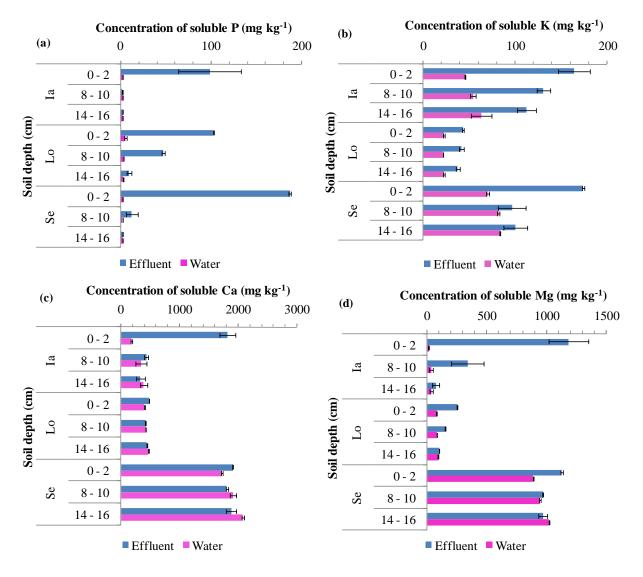


Figure 4.1 Concentrations of acid soluble (a) P, (b) K, (c) Ca and (d) Mg in Longlands (Lo), Inanda (Ia) and Sepane (Se) soil segments after leaching with effluent or distilled water.

## 4.3.4.2 Reducible fraction

There were no significant differences between the segments in the water-leached columns in terms of P, K, Ca, and Mg irrespective of soil type (Figure 4.2) except for a significant difference in the Se where the Ca and Mg concentrations in the 0-2 cm segment were higher

than in the middle and bottom segments (Figures 4.2c and d, respectively). In the effluentleached columns, the 0-2 cm segment was significantly greater than the two lower segments for Ca and Mg in all soils except the Lo. Phosphorus in the middle and bottom segments was negligible indicating that incoming P from the effluent was being bound to hydrous oxides of Fe, Al and perhaps Mn in the upper segment of the column (Figure 4.2a). The K concentration was not as high as other bases which meant that K was sparingly retained (Figure 4.2b). The Ia and Se accumulated more K in the 0-2 cm segment unlike the Lo which had an even distribution between segments.

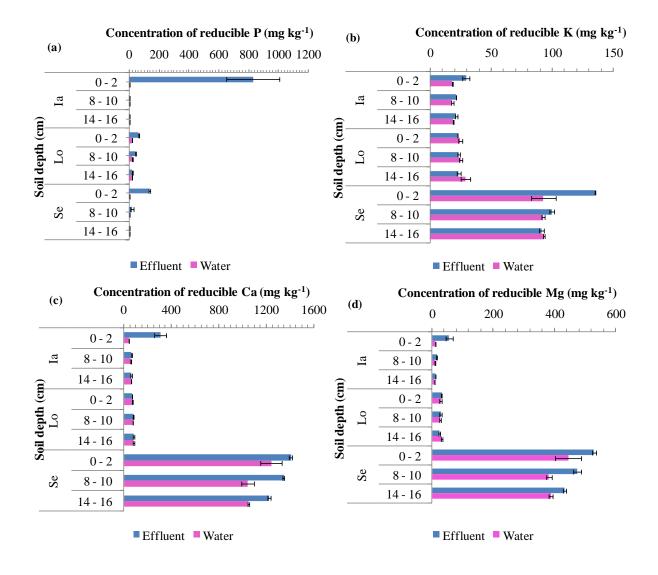


Figure 4.2 Concentrations of reducible (a) P, (b) K, (c) Ca and (d) Mg in Longlands (Lo),Inanda (Ia) and Sepane (Se) soil segments after leaching with effluent ordistilled water.

With regards to the water-leached columns, K was evenly distributed throughout. In the Ia soil, the Ca and Mg were bound in the upper segment. The bases in this fraction in the effluent-leached columns were almost the same as the initial concentrations in soil and lower in the water-leached columns. There is a possibility that what was extracted as reducible could actually be a leftover from the acid soluble extraction since bases will not bind to oxides of Fe and Al. Extractants used in sequential extractions lack selectivity and may extract species from other phases (Filgueiras *et al.*, 2002).

#### 4.3.4.3 Oxidizable fraction

The water-leached columns showed no significant differences between segments irrespective of soil type for the oxidizable fraction of P, K, Ca and Mg (Figure 4.3). Surprisingly a similar trend occurred in the effluent-leached columns except for P in the Ia (Figure 4.3a) where the 0-2 cm segment was significantly higher than the other segments of the column. This similarity between the water and effluent-leached segments down the column could be attributed to the lower organic matter status of the Se and Lo since the oxidizable fraction is considered bound to the organic matter in the soil.

In addition, the effluent being low in carbon (21.4 mg  $L^{-1}$ ) further explains this similarity between the effluent and water-leached segments for these two soils. The exception of the Ia was therefore as a result of the high organic matter content of the Ia. In instances where the oxidizable fraction of the Ia was low it might have been as a result of the ineffectiveness of the hydrogen peroxide in destroying the organic matter as found by Tessier *et al.* (1979).

## 4.3.4.4 Residual fraction

Primary and secondary minerals containing elements in the crystalline lattice constitute the bulk of this fraction. Presumably not much will be contributed in terms of availability of these elements to the soil at least in the short term. Also, this step involved a transfer of soil from the extraction tubes into the digestion tubes which may have resulted in some loss of material.

Nonetheless, this is indicative of how much of the particular element is still stored within the soil. Figure 4.4 shows that in most cases the concentrations of the various elements in the

effluent and the water-leached columns were not significantly different from each other signifying that the additions from the effluent had minimal influence on this fraction of the soil.

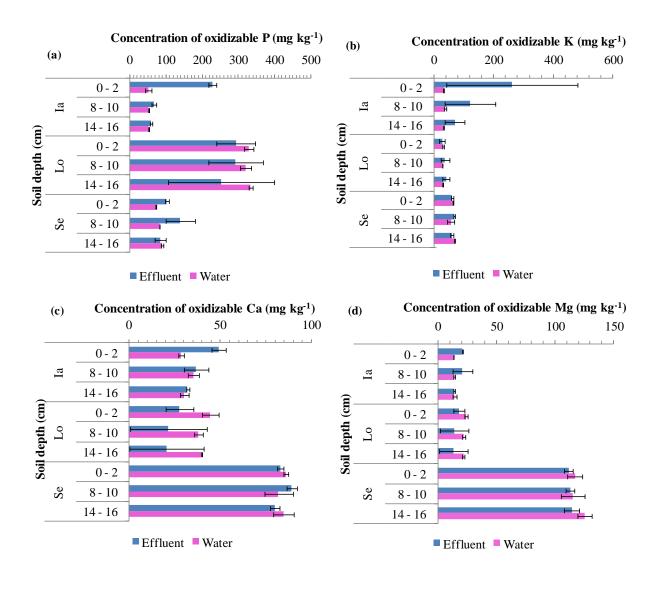


Figure 4.3 Concentrations of oxidizable (a) P, (b) K, (c) Ca and (d) Mg in Longlands(Lo), Inanda (Ia) and Sepane (Se) soil segments after leaching with effluent or distilled water.

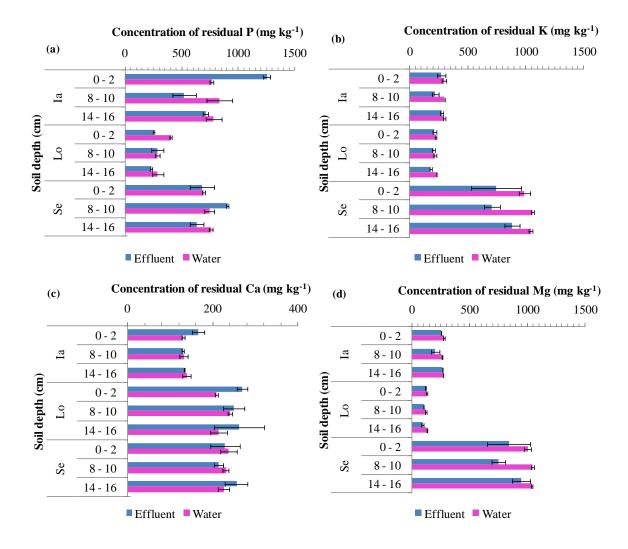


Figure 4.4 Concentrations of residual (a) P, (b) K, (c) Ca and (d) Mg in Longlands (Lo),Inanda (Ia) and Sepane (Se) soil segments after leaching with effluent ordistilled water.

#### 4.4 Conclusions

Phosphorus was the element that was most strongly influenced by the dynamics in the leaching process. The accumulation of elements, most especially P, in the upper layer during leaching is influenced by the nutrient loading in the leaching solution. This gives an opportunity for uptake by plants and also reduces the risk of downward movement. Alternatively, the P adsorbed to the Fe and Al oxides was high in the upper segment and could be potentially unavailable for plant uptake in the Ia soil. However, P in the surface layer may be susceptible to sediment transport by runoff which could pollute freshwater

sources. Elements such as Ca, Mg and K are not a threat to the environment but rather can be regarded as a resource to plants.

In comparison to the fractions in soil before leaching, concentration of nutrients was higher after leaching with effluent in the top segments except for Ca and Mg in the oxidizable fraction for all soils. There was greater mobility to the lower segments for the acid soluble forms of K, Ca and Mg in the water-leached segments of all soils showing movement of elements out of the soil. Additionally, the concentration of the elements in the water-leached columns was almost same as those in the initial soils for that fraction.

## Chapter 5

# EFFECT OF IRRIGATION WITH ANAEROBIC BAFFLED REACTOR EFFLUENT ON NUTRIENT AVAILABILITY AND SOIL PROPERTIES FOR MAIZE GROWTH

#### 5.1 Introduction

Treated sewage effluent has been successfully used for crop irrigation in several countries (Feigin et al., 1991; Fonseca et al., 2007). Soil application of treated wastewater as a water and nutrient source for agricultural irrigation represents a low cost alternative for wastewater treatment (Asano et al., 1996). The application of treated wastewater to the soil-plant system may mitigate the scarcity of water resources and the discharge of nutrients to water bodies by using soil and plants as natural filters (Pollice et al., 2004). Thus crop irrigation with treated wastewater constitutes an ecologically sound method for the disposal of effluent into the environment (Toze, 2006). The ability of soil to immobilize nutrients from anaerobic baffled reactor (ABR) effluent (Chapter 4) indicates the possibility that such nutrients can be used for crop growth. The concentrations of ions in soil are influenced by water movement, their concentrations in irrigation water and plant uptake (Heidarpour et al., 2007) which necessitates a study to assess the potential of plant growth. The substitution of conventional water with secondary treated sewage effluent has recorded success particularly on the maize crop (Al-Jaloud et al., 1995; Mohammad and Mazahreh, 2003). Pot experiments can assist in giving more insight on irrigating with this effluent and could eventually serve as guidance for irrigation at the field scale.

The objectives of this study were to:

- ➤ investigate the potential of ABR effluent to sustain crop growth;
- assess nutrient accumulation in plants (in particular N, P, K, Ca and Mg) from use of the effluent;
- > assess residual effects of plant nutrients in soil after effluent application; and
- ➤ investigate the liming capability of the effluent.

#### 5.2 Materials and methods

#### 5.2.1 Pot experiment 1

A pot experiment was carried out in a glasshouse at the University of KwaZulu-Natal (UKZN), Pietermaritzburg with maximum and minimum temperatures of 26°C and 16°C, respectively. Three contrasting soil types were used namely a Cartref E horizon (Cf; Typic Haplaquept), and the A horizons of an Inanda (Ia; Rhodic Hapludox) and Sepane (Se; Aquic Haplustalf) (Soil Classification Working Group 1991; Soil Survey Staff, 2010). These soils were a new batch of collection with the Ia and Se collected from the same locations as in Section 3.2.1 and the Cf from near Ottos Bluff near Pietermaritzburg under virgin veld. The soils were air dried, ground to pass a 2 mm sieve and physico-chemical properties determined following methods of The Non Affiliated Soil Analysis Work Committee (1990). Pots with inner diameter of 20 cm and a height of 17 cm were filled with 2 kg soil to approximate field bulk densities of 1.47 g cm<sup>-3</sup>, 0.77 g cm<sup>-3</sup>, and 1.21 g cm<sup>-3</sup> for Cf, Ia and Se soils, respectively. Fertilizer (N, P and K) was applied at the recommended rate, half the recommended rate and zero for maize on each soil. Ammonium nitrate, potassium dihydrogen phosphate and potassium nitrate were used to supply the fertilizer nutrients in solution at different rates before planting (0, 100, 200 kg N ha<sup>-1</sup> for all soils; 0, 40, 80 kg P ha<sup>-1</sup> and 0, 50, 100 kg K ha<sup>-1</sup> for the Cf; 0, 10, 20 kg P ha<sup>-1</sup> and 0, 102.5, 205 kg K ha<sup>-1</sup> for Ia; and 0, 30, 60 kg P ha<sup>-1</sup> and 0, 5, 10 kg K ha<sup>-1</sup> for Se). This was halved for the half fertilizer rate and no fertilizer was applied for the zero fertilizer rate. All pots were treated with a 5 mL aliquot of sodium molybdate (Na<sub>2</sub>MoO<sub>4</sub>) irrespective of fertilizer rate and irrigation solution. Magnesium sulphate (MgSO<sub>4</sub>-7H<sub>2</sub>O) was used to supplement Mg requirements of the Ia soil. Lime was applied to all Ia treatments as pure grade  $Ca(OH)_2$  at 5 g pot<sup>-1</sup> to achieve a quicker liming effect as opposed to commercial grade lime considering the growth duration of the experiment. The lime amount was calculated using the calcium carbonate equivalent of 136% for Ca(OH)<sub>2</sub> and an application rate of 10 tons ha<sup>-1</sup>. Eight maize seeds (PAN 4P-767BR) were planted per pot and later thinned to four plants two weeks after planting. Pots were watered with either tap water or ABR effluent with the latter sourced from the pilot plant in the School of Chemical Engineering, UKZN, Durban and kept in a cold chamber at about 4°C. An hour before irrigation, the required estimated quantity was taken out and left at room temperature. Each treatment was applied in triplicate (total of 54 pots) and the experiment was laid out in a randomized complete block design generated by Genstat 12.1. Plants were watered according to evapotranspiration demands and the total volume of solution added per pot was 43.3, 82.7 and 52.6 mm for the Cf, Ia and Se, respectively.

After six weeks of growth, plant height and number of leaves were measured. The plants were harvested at 1 cm above soil level, and dried at 70°C to determine dry matter yield. Dried samples were ground and stored for plant nutrient analyses. Total N was determined by Kjeldahl digestion (Rowell, 1994). Phosphorus, K, Ca, Mg, Zn, Mn and Cu were determined by inductively coupled plasma emission spectrometry (ICP, Varian 720-ES) after nitric acid digestion (Titshall, 2007). Nutrient concentrations were reported as nutrient accumulation rather than nutrient concentration as this gives a better understanding of the nutrient uptake. Data were analysed using Genstat 12.1 and the Student Newman Keul range test at 5% was used to determine differences between treatment means. Soil samples were collected from each pot and sent to the Soil Fertility and Analytical Services Division of the KwaZulu-Natal Department of Agriculture, Cedara for fertility analysis.

The pH and EC of the ABR effluent were measured on a Radiometer PHM 210 meter and a CDM 210 electrical conductivity meter, respectively. NH<sub>4</sub><sup>+</sup>-N and NO<sub>3</sub><sup>-</sup>-N were analysed with a TRAACS 2000 continuous flow auto-analyser. Total N was determined by steam distillation with magnesium oxide and Devarda's alloy (Rowell, 1994). Total carbon was measured with a Shidmadzu TOC analyser. The elemental composition of the ABR effluent was analysed by inductively coupled plasma emission spectrometry (ICP, Varian 720-ES). The *Escherichia coli* composition was determined using a modification of the plate count method (American Public Health Association, 1992) by plating dilutions on eosin methylene blue (EMB) agar plates and counting colonies formed after incubation at 35°C for 48 hours.

#### 5.2.2 Pot experiment 2

A second pot experiment was set-up with only the Ia soil. This was to investigate the ability of the effluent to act as a liming agent which could have been masked in pot experiment 1. The Ia soil was placed into similar pots as before and treated in the same manner except for the fact that they were neither fertilized nor limed. Maize was planted in the same way and the experiment was replicated three times for both the effluent and tap water pots giving a total of 6 pots. These were run concurrently with pot experiment 1. At six weeks after planting, the plants were harvested and treated as those from pot experiment 1. Soil samples were collected from each pot and analysed chemically and statistically as in pot experiment 1. This enabled a comparison between the limed Ia treatments in pot experiment 1 with the unlimed treatments in pot experiment 2.

#### 5.3 Results and discussion

#### 5.3.1 Pot experiment 1

#### 5.3.1.1 Soil and effluent characterisation

The chemical analyses, particle size distribution and mineralogy of the soils are given in Table 5.1. The differences shown as compared to Table 3.1 were as a result of the fact that these soils were a new batch of the same soils used for the column studies. The Lo was substituted by the Cf, which was very similar to the Lo but had slightly lower clay content. The fertility status of the soils shows a wide range of concentrations in terms of N, P, K, Ca and Mg and none met the fertilizer requirements for maize.

The effluent used was from the same batch as that used for the column studies and as shown in Table 5.2 only very slight changes had occurred over time. The effluent still belongs to salinity class C2S1 (medium-salinity water/low sodicity water) for irrigation water classification. The total amounts of N, P and K supplied by the effluent and the tap water during the course of the pot experiment are given in Table 5.3.

The mineralogical composition of the soils was the same as described in Section 3.3.1 except the Cf which was similar in composition to the Lo but with lesser amounts of Fe oxides and hydroxides.

		Sc	oil form <sup>a</sup> and	horizon
Parameter		Cf E	Ia A	Se A
	(H <sub>2</sub> O)	6.24	4.44	7.09
pH	(1M KCl)	4.95	4.06	5.92
Electrical conductivity (dS m <sup>-1</sup> )		0.02	0.09	0.10
Organic C (g 100g <sup>-1</sup> )		0.18	7.54	1.92
Total N (mg kg <sup>-1</sup> ) <sup>#</sup>		352	6234	2087
	Ca	1.11	0.56	8.23
Extractable base cations $(\text{cmol}_c \text{kg}^{-1})^{\#}$	Mg	0.45	0.21	7.39
	K	0.10	0.13	0.26
Exchangeable acidity (cmol <sub>c</sub> kg <sup>-1</sup> ) #		0.06	4.31	0.08
Total cations $(\text{cmol}_c \text{kg}^{-1})^{\#}$		1.73	5.21	16.0
Acid saturation (%) #		3.46	82.7	0.50
	Mn	3.52	6.49	9.57
Extractable metal cations $(mg kg^{-1})^{\#}$	Cu	0.70	1.95	2.61
	Zn	0.07	0.78	4.35
Extractable P (mg kg <sup>-1</sup> ) #		2.11	15.6	5.22
Particle size (%)				
Coarse sand (0.5-2 mm)		16.0	4.1	2.5
Medium sand (0.25-0.5 mm)		23.3	7.6	3.4
Fine sand (0.053-0.25 mm)		40.9	18.2	15.5
Silt (0.002-0.053 mm)		12.9	48.2	42.9
Clay (<0.002 mm)		6.9	21.9	35.7
Clay mineralogical composition (%)				
Vermiculite		*	*	**
Illite		*	-	*
Mixed-layer minerals		-	-	v-m **/ v-c
Kaolin		**	**	*
Quartz		**	tr	*
Feldspar		tr	tr	tr
Goethite		tr	*	tr
Anatase		tr	-	tr
Gibbsite		-	**	-

**Table 5.1**Some characteristics of the Cartref, Inanda and Sepane soils used for the pot<br/>experiments

<sup>a</sup> Cartref (Cf), Inanda (Ia), Sepane (Se) (Soil Classification Working Group, 1991).

<sup>#</sup> Analysis conducted by the Soil Fertility and Analytical Services Division (KwaZulu-Natal Department of

Agriculture, Cedara).

\*\* 20-60

\* 5-20

tr < 5

- not found

v – vermiculite

m – mica

c - chlorite

Parameter	Effluent	tap water
Electrical conductivity(dS m <sup>-1</sup> )	0.50	0.10
pH	6.68	6.62
Elements (mg L <sup>-1</sup> )		
Total N	9.70	1.30
Phosphorus	30.4	0.01
Potassium	10.5	3.51
Sulphur	7.24	0.73
Calcium	16.1	6.8
Magnesium	18.7	2.23
Sodium	27.2	3.53
Aluminium	0.06	0.05
Cadmium	0.01	0.01
Cobalt	*bd	bd
Chromium	0.01	0.01
Copper	0.04	0.06
Iron	0.20	0.11
Manganese	bd	bd
Molybdenum	bd	bd
Nickel	0.2	0.16
Lead	bd	bd
Selenium	bd	bd
Vanadium	0.14	0.08
Zinc	0.04	0.84
Boron	0.04	0.06
Bicarbonate	246	31.0
Chloride	31.0	9.00
Total carbon	20.7	1.50
<i>E.coli</i> count- colony forming unit (cfu mL <sup><math>-1</math></sup> )	$2.2 \text{ x} 10^4$	bd
Sodium adsorption ratio (SAR)	0.59	0.18

**Table 5.2**Chemical and *Escherichia coli* (*E. coli*) composition of the ABR effluent and<br/>tap water

\* bd – below detection.

Nutrient	*Cartref		Ina	anda	Sepane		
Nument	Effluent	Tap water	ater Effluent Tap water		Effluent	Tap water	
Ν	65	8	40	5	47	6.5	
Р	200	0.1	123	0.1	146	0.1	
K	70	22	42.5	14	50.3	17	

**Table 5.3**Total amounts of N, P, and K (kg ha<sup>-1</sup>) supplied to each soil by the irrigation<br/>solutions over the six week growing period

\* Soil Classification Working Group, 1991

The amount of nutrients supplied exclusively by the effluent could meet the nutrient requirements of an irrigated maize plant in terms of P for all soils and K for the Se but the N supply was inadequate for all the soils. This confirms the work of Campbell *et al.* (1983) who stated that a weekly application of 25 mm of municipal wastewater over two growing seasons was enough to supply 40-80% of the N and all of the P needed for a maize crop. The effluent was lower in N and P concentrations in comparison with the ABR effluent and was also void of heavy metals.

#### 5.3.1.2 Growth parameters and dry matter yields

At 6 weeks after planting there was a difference in plant height between water and effluent treatments and between zero and the full fertilizer rate for all soils (Plate 5.1). The Cf was significantly different (p<0.05) from the Ia and Se which were not significantly different from each other with regards to plant height for the effluent-irrigated pots irrespective of fertilizer rate (Table 5.4). A different scenario was noticed for the water-irrigated plants whereby the Cf was significantly different from the Ia and Se only at the full fertilizer rate and significant differences occurred between the Ia and Se also at the full fertilizer rate. It is notable that plant height was significantly higher in the control (unfertilized) pots for the effluent-irrigated than for the water-irrigated pots (Plate 5.2). The higher response to both effluent and fertilizer by the Cf is explained by the fact that it has a low cation exchange capacity and cannot absorb nutrients that thus remain readily available to the plants. This availability was

enhanced due to the fact that this was a closed system with no leaching taking place (Fonseca *et al.*, 2005a).

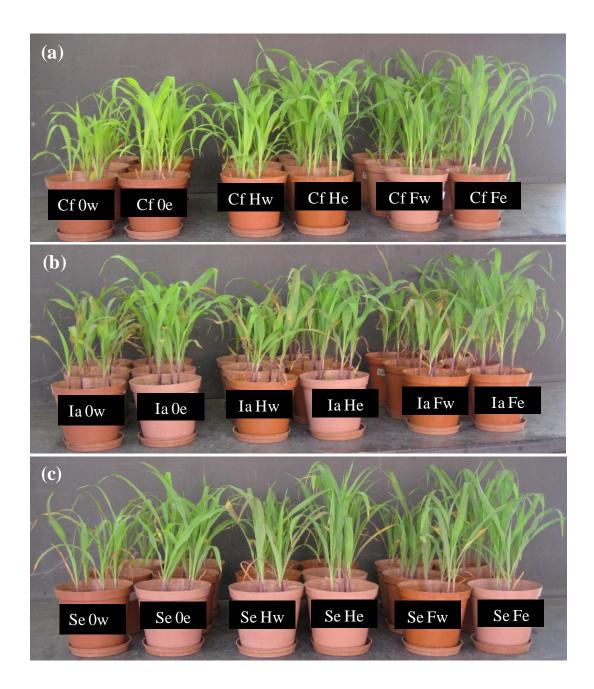


Plate 5.1Growth of maize (6 weeks) in (a) Cartref (Cf), (b) Inanda (Ia) and (c) Sepane<br/>(Se) soils irrigated with tap water (w) or effluent (e) treated with fertilizer at<br/>Zero (0), Half (H) and Full (F) recommended rates.

Table 5.4 further shows that the response in terms of number of leaves was significant in the Cf at the full fertilizer rate in both the effluent-irrigated pots and water-irrigated pots.

Most apparent was a P deficiency as observed by the purple colouration of the leaves, especially in the Ia and Se soils. Phosphorus deficiency was most obvious in the waterirrigated pots as compared with the effluent-irrigated pots especially in the pots with no fertilizer amendment (Plate 5.2).

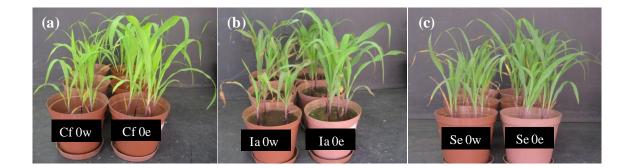


Plate 5.2 Growth of maize (6 weeks) in (a) Cartref (Cf), (b) Inanda (Ia) and (c) Sepane (Se) soils irrigated with tap water (w) or effluent (e) at zero fertilizer (control).

There was a significant (p<0.05) difference in dry matter yield between plants receiving the different irrigation solutions irrespective of fertilizer applied (Table 5.4). The highest dry matter yield (4.9 g pot<sup>-1</sup>) was measured in the Cf soil with full fertilizer application and irrigated with the effluent. It is also important to note that the dry matter yields of the Cf (2.27 g pot<sup>-1</sup>), Ia (2.17 g pot<sup>-1</sup>) and Se (2.13 g pot<sup>-1</sup>) pots irrigated with effluent with no fertilizer applied were almost double those from equivalent treatments irrigated with water (Table 5.4).

That this trend occurred irrespective of soil type indicates the potential that the effluent has for enhancing plant growth on soils with very different properties. The dry matter yields obtained from the effluent-irrigated pots with zero fertilizer were not significantly different from those from the water-irrigated pots at full fertilizer application for the Ia and Se. The Cf pots irrigated with effluent and with zero fertilizer were not significantly different from the water-irrigated pots at half fertilizer application further showing the ability of plants to source nutrients efficiently from the Cf soil in an unleached situation.

Irrigation	Soil	Fertilizer	Plant height	Number of leaves	Dry matter yield
solution	form*	rate <sup>α</sup>	(mm)	plant <sup>-1</sup>	$(g pot^{-1})$
		F	$640\pm6.3h^{\#}$	$6.0 \pm 0e$	$4.90\pm0.21j$
	Cf	Н	$599 \pm 10.8 g$	$5.8\pm0.2\text{de}$	$3.73 \pm 0.41 \text{hi}$
		0	$495 \pm 12.7 e$	$5.0 \pm 0$ abc	$2.27\pm0.03 def$
-		F	523 ± 11.6ef	6.1 ± 0.1e	$3.30\pm0.12 gh$
Effluent	Ia	Н	$509\pm6.6~e$	$6.0 \pm 0e$	$2.83\pm0.09 fg$
		0	$456 \pm 19.1 d$	$5.6 \pm 0.3$ cde	$2.17 \pm 0.20 cdef$
-		F	530 ± 3.0ef	5.3 ± 0.1abcd	$2.77\pm0.23 fg$
Se	Se	Н	$512\pm0.5e$	$5.1\pm0.1 abc$	$2.53 \pm 0.27 ef$
		0	$454 \pm 12.9 d$	$5.1 \pm 0.1 abc$	$2.13 \pm 0.15 bcdef$
		F	$552 \pm 5.3 \mathrm{f}$	$5.4 \pm 0.1$ bcd	$3.23\pm0.22 gh$
	Cf	Н	$462\pm4.3d$	$5.1 \pm 0.1$ abc	$2.40\pm0.15ef$
		0	$366 \pm 2.8a$	$4.7 \pm 0.1a$	$1.33\pm0.03ab$
-		F	$443 \pm 6.0d$	$5.6 \pm 0.2$ cde	1.77 ± 0.09abcde
Water	Ia	Н	$428 \pm 4.0 cd$	$5.3 \pm 0.1 abcd$	$1.33\pm0.18ab$
		0	$385 \pm 12ab$	$5.3 \pm 0.1 abcd$	$1.40 \pm 0.06 abc$
-		F	$403 \pm 10.7 bc$	5.1 ± 0.1abc	$1.57 \pm 0.03$ abcd
	Se	Н	$428\pm8.4cd$	$5.0 \pm 0$ abc	$1.87 \pm 0.09 abcde$
		0	367 ± 14.4a	$4.8\pm0.2ab$	$1.23\pm0.19a$

**Table 5.4**Mean growth parameters and dry matter yields (± SE; n=3) of maize as<br/>influenced by irrigation source and fertilization

\* Cartref (Cf), Inanda (Ia), Sepane (Se).

 $^{\alpha}$  F, H, 0 = full, half and zero fertilizer rates.

<sup>#</sup> Means followed by the same letter within each column are not significantly different (p<0.05).

### 5.3.1.3 Nutrient accumulation in plants

Maize above-ground nutrient concentrations were significantly higher (p<0.05) in the effluent-irrigated pots than in the equivalent treatment for water-irrigated pots (Table 5.5).

The N and P nutrient concentrations in the above-ground biomass were influenced positively by the effluent and the fertilizer application unlike the K where there was no consistent effect with the fertilizer and irrigation source.

Comparisons between the fully fertilized plants showed that nutrient concentrations in the effluent-irrigated plants were significantly higher than the water-irrigated plants indicating an additional input from the effluent. This is further shown in Table 5.5 where P content (5.25 mg pot<sup>-1</sup>) of plants from the Cf with no fertilizer application and irrigated with the effluent was about five times higher than that from the water-irrigated pots (0.99 mg pot<sup>-1</sup>). Likewise N followed the same trend as P with N content being higher (about three times) in the effluent-irrigated plants than in the water-irrigated plants (Table 5.5). These results are in agreement with the observations of Bielorai et al. (1984) but contrary to those of Fonseca et al. (2005a) who reported that the use of secondary treated sewage effluent on adequately fertilized maize plants did not increase plant N content. Feigin et al. (1981) observed no influence of the irrigation water quality (sewage effluent and deionized water) on N accumulation in maize plants. Phosphorus deficiency was evident in all treatments on the Ia and Se soils but not on the Cf and was more severe in the equivalent water-irrigated than in the effluent-irrigated pots. Despite this trend, the P content in the effluent-irrigated plants was higher than in the water-irrigated plants, irrespective of fertilizer use. The P deficiency in the Ia and Se soils was probably due to its non-availability to plant roots due to higher amounts of clay and iron and aluminum oxides in these soils. Plant K content was also higher in the fertilized, effluent-irrigated treatments than in the water-irrigated treatments, with plants on the Ia having the highest K.

In terms of Ca the fully fertilized, water-irrigated plants were not significantly different from the effluent-irrigated plants at half fertilizer application in the Cf (Table 5.5). Calcium was significantly higher in the effluent-irrigated plants than in the water-irrigated plants for all treatments on the Ia except in the unfertilized water-irrigated plants which were not significantly different from the effluent-irrigated plants at half fertilizer application (Table 5.5). This could possibly be indicative of an interaction effect between the lime, effluent and fertilizer which impacts on the ability of the plant to absorb Ca from the soil. In the Se the uptake was higher for effluent-irrigated than for the water-irrigated plants but this difference was not significant across the different fertilizer application rates (Table 5.5) due possibly to the initially high Ca status of the Se (Table 5.1).

Irrigation	Soil				Nutrient (mg pot <sup>-1</sup> )		
solution form*		Fertilizer rate <sup><math>\alpha</math></sup>	Ν	Р	K	Ca	Mg
		F	$80.3 \pm 0.69 g^{\#}$	$12.7\pm0.36g$	$55.6 \pm 2.61 \text{ef}$	$16.5 \pm 0.41$ de	$11.4\pm0.45i$
	Cf	Н	$59.4 \pm 2.52e$	$10.3\pm0.49 f$	$45.0 \pm 1.14 cde$	$13.1 \pm 0.35$ cd	$8.95\pm0.85h$
		0	$31.9 \pm 2.64 bc$	$5.25\pm0.15e$	$33.8 \pm 1.36 bcd$	$7.42 \pm 0.28 ab$	$4.86 \pm 0.07 cdef$
		F	81.0 ± 1.38g	$3.28\pm0.04d$	$83.2\pm0.38g$	$22.5 \pm 1.03 \text{fg}$	$7.39\pm0.43g$
Effluent	Ia	Н	$66.7\pm2.61\mathrm{f}$	$2.29\pm0.19bc$	$52.7\pm4.08e$	$19.8 \pm 2.79 efg$	$6.47 \pm 0.49 efg$
		0	$47.2\pm4.50d$	$2.05 \pm 0.25$ abc	32.1 ± 2.64abc	$23.3 \pm 1.66g$	$5.78 \pm 0.58 defg$
		F	$55.7 \pm 0.48e$	2.81 ± 0.18cd	47.4 ± 2.36cde	9.63 ± 0.54abc	$7.38\pm0.59g$
	Se	Н	$25.9 \pm 1.49 b$	$2.38\pm0.17c$	$44.1 \pm 4.62 cde$	$9.20 \pm 0.76$ abc	$6.81 \pm 0.69$ fg
		0	$37.2 \pm 0.80c$	1.91 ± 0.12abc	34.5 ±1.0bcd	$7.19 \pm 0.14ab$	$5.75 \pm 0.33 defg$
		F	57.1 ± 2.70e	$4.74 \pm 0.47e$	47.4 ± 5.98cde	$10.2 \pm 1.23$ bc	$6.0 \pm 0.49 defg$
	Cf	Н	$32.7 \pm 1.29 bc$	$2.40\pm0.1c$	37.6 ± 1.3bcd	$7.99 \pm 0.18$ ab	$4.53 \pm 0.37$ bcde
		0	$14.5 \pm 1.25a$	$0.99\pm0.04a$	$25.7\pm0.29ab$	$4.38\pm0.08a$	$2.27\pm0.06a$
		F	$45.8 \pm 4.10d$	1.2 ± 0.09a	48.8 ± 3.74de	13.7 ± 0.97cd	3.15 ± 0.12abc
Water	Ia	Н	$37.6 \pm 3.84c$	$1.03\pm0.14a$	$34.2 \pm 6.74 bcd$	$12.7 \pm 2.09$ cd	$2.77\pm0.4ab$
		0	$34.0\pm0.39 bc$	$1.15\pm0.09a$	$19.2\pm1.20a$	$18.7 \pm 1.42 ef$	$3.69 \pm 0.17 abc$
		F	19.1 ± 0.26a	$1.24 \pm 0.05a$	31.7 ± 1.63abc	5.8 ± 0.14ab	$4.19 \pm 0.11 bcd$
	Se	Н	$26.8 \pm 1.34 b$	$1.38 \pm 0.05 ab$	$37.3 \pm 0.79 bcd$	$6.8\pm0.42ab$	$4.98 \pm 0.18 cdef$
		0	$12.6 \pm 2.03a$	$0.97\pm0.08a$	25.7 ± 5.51ab	$4.8\pm0.60a$	3.33 ± 0.51abc

**Table 5.5**Effects of irrigation source and fertilization on mean nutrient accumulation (± SE; n =3) in above-ground biomass of maize

\*Cartref (Cf), Inanda (Ia), Sepane (Se).

<sup> $\alpha$ </sup> F, H, 0 = full, half and zero fertilizer rates.<sup>#</sup> Means followed by the same letter within each column are not significantly different (p<0.05).

Magnesium showed a slightly different trend from Ca. In the Cf, the plant Mg concentration was significantly different between the fertilizer rates for the effluent-irrigated plants but with the water-irrigated plants the full and half fertilizer rates were not significantly different from each other but both were significantly different from the unfertilized plants. In the Ia all the effluent-irrigated treatments were significantly different from all the water-irrigated treatments. In the Se, the plant Mg content showed no significant differences between the effluent-irrigated fully fertilized and all the water-irrigated treatments. The fertilized water-irrigated treatments were not significantly different from the unfertilized effluent-irrigated plants (Table 5.5) suggesting a stronger influence from the Mg concentration in the effluent than that of Ca, despite the high Ca relative to Mg concentration in the soil (Table 5.1).

Another observation from Table 5.5 is that the plant nutrient concentrations were in the order N > K > Ca > Mg > P irrespective of irrigation solution, with very few exceptions. This is different to Fonseca *et al.* (2005b) who found that when complete mineral fertilizer was present, irrigation with effluent resulted in less Ca and more K accumulation in maize than when deionized water was used. These differences could be due to the concentrations of these elements in the tap water used in the present study (Table 5.2). Potassium accumulation was more than Ca and Mg which conforms to results found in studies reporting that maize absorbs much more K than other cations (Fageria *et al.*, 1991).

#### 5.3.1.4 Post-harvest soil properties

#### pH and exchangeable acidity

Changes in soil pH occurred only in the Cf and Se water-irrigated pots as those with no fertilizer applied were significantly higher (p<0.05) than the full fertilizer rate (Table 5.6). This was the case in the Cf and the Se but not in the Ia probably due to the masking effect of the lime applied. This could also be the possible reason why the effluent-irrigated pots did not show significant differences across fertilizer rates due to the 'liming effect' of the effluent as indicated by its Ca and Mg concentrations (Table 5.6). The Cf soil responded positively to effluent irrigation as compared to water irrigation mirroring the ameliorating effect of secondary-treated sewage effluent on soil pH (Fonseca *et al.*, 2005b). Values for

exchangeable acidity were lower in treatments with higher pH values although the effect was significant only for the Ia (Table 5.6) due to its inherently high exchangeable acidity (Table 5.1). The effect of the effluent in ameliorating soil acidity could be evident in the case of the Ia treatments with no fertilizer application. Comparing the pH of the effluent with that of the water, it is unlikely that soil pH changes observed were influenced by the pH of the irrigation solutions (Bouwer and Idelovitch, 1987). Possible reasons for this increment could be the addition of exchangeable cations (Ca and Mg) originating from the effluent (Bouwer and Idelovitch, 1987) or the increased denitrification that occurs in effluent-irrigated soils (Friedel *et al.*, 2000). These findings agree with the results of Smith *et al.* (1996) and Al-Nakshabandi *et al.* (1997) that showed an increase in pH of less than 1 pH unit when irrigating with secondary-treated sewage effluent.

#### Electrical conductivity (EC)

There were no significant differences between the EC in the Cf for both effluent-irrigated and water-irrigated soils despite the fertilizer applied (Table 5.6). A significantly higher EC in the Ia effluent-irrigated pots than in the water-irrigated pots was observed in the unfertilized treatments. Within the effluent-irrigated soils, the fully fertilized Ia was significantly higher in EC than both the half and zero rates which were not significantly different from each other. In the case of the water-irrigated Ia all three fertilizer treatments were significantly different from each other (Table 5.6). In the Se there were no significant differences between fertilizer rates within irrigation solutions but the full and half fertilizer rates in the effluent-irrigated pots were significantly higher than all water-irrigated treatments. In general terms the soil EC after harvesting compared to that at the start of the experiment shows an increase irrespective of irrigation solution. This is in contrast to the work of Mohammad and Mazahreh (2003) where an increase in EC was observed only for wastewater-irrigated pots. The present results are in line with increases reported in EC by Mancino and Pepper (1992). The effluentirrigated pots experienced higher plant cation uptake (Table 5.5) and it was expected that soil solution could become dilute but this was counteracted by a continuous supply by the effluent that allowed for increases in EC values. In the water-irrigated pots there was a marked EC increase in the Ia due to less plant accumulation of cations and lime application.

Irrigation	Soil	Fertilizer	pН	EC	Exchangeable acidity	Ca	Mg
solution	form*	rate <sup>α</sup>	(1M KCl)	$(dS m^{-1})$	$(\text{cmol}_{c} \text{ kg}^{-1})$	(cmol <sub>c</sub>	$kg^{-1}$ )
		F	4.93f <sup>#</sup>	0.06ab	0.04a	1.24a	0.57a
	Cf	Н	4.98f	0.06ab	0.04a	1.20a	0.58a
		0	4.98f	0.06ab	0.05a	1.22a	0.57a
		F	4.33b	0.37f	0.78bc	6.12bc	0.59a
Effluent	Ia	Н	4.36b	0.32e	0.66b	5.99b	0.67a
		0	4.28ab	0.28e	0.87cd	6.46c	0.41a
		F	5.78g	0.19d	0.05a	8.89e	7.67d
	Se	Н	5.81gh	0.19d	0.06a	8.58de	7.11bc
		0	5.85h	0.15cd	0.06a	8.40d	6.93b
		F	4.57c	0.03a	0.06a	1.21a	0.39a
	Cf	Н	4.64d	0.03a	0.05a	1.11a	0.36a
		0	4.74e	0.03a	0.05a	1.15a	0.39a
		F	4.31b	0.41f	0.77bc	6.05bc	0.42a
Water	Ia	Н	4.35b	0.32e	0.71b	6.23bc	0.46a
		0	4.25a	0.20d	0.99e	6.43c	0.20a
		F	5.75g	0.11bc	0.05a	8.73de	7.02b
	Se	Н	5.77g	0.10bc	0.08a	8.74de	6.89b
		0	5.84h	0.11bc	0.06a	8.40d	6.66b
<b>C.V<sup>©</sup>(%)</b>			0.6	15.3	18.2	3.2	7.4

**Table 5.6**Effects of irrigation source and fertilization on mean (n=3) soil pH,<br/>exchangeable acidity, electrical conductivity (EC), calcium (Ca) and<br/>magnesium (Mg) of soil after harvest

\*Cartref (Cf), Inanda (Ia), Sepane (Se).

 $^{\alpha}$  F, H, O = full, half and zero fertilizer rates.

<sup>#</sup> Means followed by the same letter within each column are not significantly different

(p<0.05).

 $^{\odot}$ C.V = coefficient of variation.

Soil N concentrations were significantly higher in the unfertilized pots than in the fertilized pots irrespective of irrigation solution only in the Ia (Table 5.7) despite the fact that effluentirrigated plants had higher above-ground N concentrations than the water-irrigated plants in all soils. This phenomenon could be explained partially by the fact that N, being a mobile element in soil, led to vigorous uptake especially with the additions from the incoming effluent resulting in higher above-ground concentrations in the effluent-irrigated pots (Table 5.5) irrespective of fertilizer application and hence little or no residual effects in soil. The N concentration in the Ia with no fertilizer amendment after harvest is almost equal to the initial soil total N meaning very minimal plant uptake occurred in these treatments as evidenced by their above-ground N content (Table 5.5) even in the effluent-irrigated pots. This substantiates the fact that N added by effluent (Table 5.3) was insufficient and additional N fertilizer was required to boost plant N uptake.

Phosphorus, unlike N, was significantly higher in the effluent-irrigated pots than the waterirrigated pots for the Cf and Ia (Table 5.7). The increase in soil P as a result of added effluent influenced P in soil even for the unfertilized pots. In the Ia, a similar trend was observed as for N where the P value in the zero fertilized pots was equal to that in the original soil. However, P is an immobile nutrient in soil, thus long term field studies are necessary to understand the effluent-P dynamics in the soil (Bond 1998). This study disagrees with that of Fonseca *et al.* (2005a) where application of secondary-treated sewage effluent did not change soil P concentration.

Soil K showed no significant differences in the effluent-irrigated over the water-irrigated Cf across fertilizer rates (Table 5.7). Soil K was significantly higher in the water-irrigated soils than in the corresponding effluent-irrigated soils for the fertilized treatments in the Ia. There was more uptake from the effluent-irrigated than the water-irrigated soils which explains the greater K in the water-irrigated soils. In the Se, soil N, P and K were unaffected by either effluent or water irrigation as there were no significant differences between effluent and water-irrigated plants. These elements showed significant differences between the water and effluent irrigation in the plants (Table 5.5) which means the amounts supplied from the effluent could have been absorbed by the plants leaving the soils with their initial

Irrigation	Soil	Fertilizer	Ν	Р	Κ
solution	form*	$rate^{\alpha}$		g kg <sup>-1</sup> )	$(\operatorname{cmol}_{c} \operatorname{kg}^{-1})$
		F	355a <sup>#</sup>	19.7h	0.03a
	Cf	Н	356a	14.5fg	0.03a
		0	366a	11.8ef	0.03a
		F	5838c	11.4def	0.11c
Effluent	Ia	Н	5267c	10.9def	0.08b
		0	6788d	19.9h	0.08b
		F	2200b	5.13abc	0.18e
	Se	Н	2209b	5.70abc	0.18e
		0	2337b	5.38abc	0.18e
		F	393a	5.46abc	0.02a
	Cf	Н	505a	3.95ab	0.02a
_		0	418a	2.85a	0.02a
		F	5186c	9.04cde	0.14d
Water	Ia	Н	5298c	7.84bcd	0.10c
		0	6599d	16.20g	0.07b
		F	2201b	3.31a	0.18e
	Se	Н	2385b	4.33ab	0.19e
		0	2288b	3.66ab	0.18e
<b>C.V<sup>©</sup>. (%)</b>			11.2	18.1	11.1

**Table 5.7**Effects of irrigation source and fertilization on mean (n=3) nitrogen (N),<br/>phosphorus (P) and potassium (K) of soil after harvest

\*  $\overline{Cf = Cartref}$ ; Ia = Inanda; Se = Sepane.

 $^{\alpha}$  F, H, 0 = full, half and zero fertilizer rates.

<sup>#</sup> Means followed by the same letter within each column are not significantly different (p<0.05).

 $^{\odot}C.V = coefficient of variation.$ 

#### 5.3.2 Pot experiment 2

#### 5.3.2.1 Growth parameters

Plants watered with effluent and not limed showed vigorous growth and were significantly different from the water treatments but the same as the limed treatment with the effluent (pot experiment 1) (Table 5.8). The average number of leaves per plant was greater in the effluent unlimed treatments than in the limed treatments but was significantly different only from the water unlimed treatment. The implications of this are that more leaves result in more biomass and thus would impact on the uptake of nutrients and subsequently on the yield.

Visually, plants from the effluent unlimed treatments were more vigorous in growth than the equivalent water-irrigated plants as well as all the limed treatments. Phosphorus deficiency symptoms were minor in the unlimed treatments compared to the limed treatments (Plate 5.3). Stunted growth characterised the unlimed water-irrigated treatments as was expected due to the lack of any form of amendments and again confirms the effluent as a nutrient source for plant growth.

# **Table 5.8**Effects of lime and irrigation source on mean (± SE; n =3) growth of maize in<br/>the Inanda (Ia) soil

Treatments*	Plant height (mm)	Number of leaves plant <sup>-1</sup>
Ia w-L	$385 \pm 12.0b^{\#}$	5.3 ± 0.1ab
Ia e-L	$456 \pm 19.1c$	$5.6 \pm 0.3$ ab
Ia w-no L	$293 \pm 4.06a$	$5.0 \pm 0a$
Ia e-no L	$455\pm7.01c$	$5.9\pm0.1b$
$\mathbf{C.V}^{\mathbb{G}}$ (%)	5.2	5.5

\*Irrigated with water, limed (Ia w-L).

Irrigated with effluent, limed (Ia e-L).

Irrigated with water, not limed (Ia w-no L).

Irrigated with effluent, not limed (Ia e-no L).

<sup>#</sup>Means followed by the same letter within each column are not significantly different

(p<0.05).

 $C.V^{\odot} = coefficient of variation.$ 

#### 5.3.2.2 Dry matter yield and nutrient accumulation in plants

Lime application did not significantly influence dry matter yield of the water-irrigated pots as opposed to the effluent-irrigated pots that recorded a significantly higher dry matter yield for the unlimed pots (Table 5.9). Nitrogen content of plants was significantly different between the unlimed water and effluent-irrigated plants but not between the limed treatments.

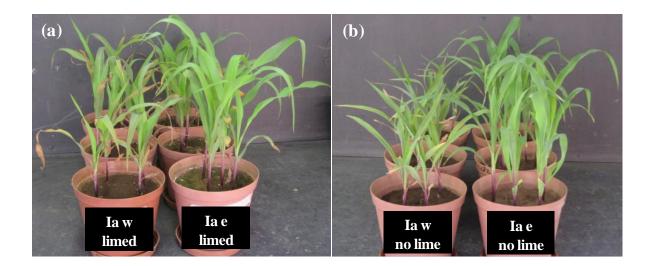


Plate 5.3 Growth of maize (6 weeks) in Inanda soil irrigated with (a) water (w); effluent(e) with lime application and (b) water (w); effluent (e) without lime application.

Furthermore, the effluent-irrigated plants in the unlimed treatments had a higher N content than in the limed soils with the reverse being the case in the water-irrigated plants showing an interaction effect between the lime and the irrigation solutions (Table 5.9). This indicates that N uptake was mainly driven by the effluent and that liming exacerbated N uptake. In the case of P, the effluent-irrigated plants had a significantly higher P accumulation than the unlimed water-irrigated plants and the limed plants irrespective of the irrigation source. It is worthwhile noting that liming did not impact on the plant P content as much as effluent application. Potassium showed a different trend whereby there were significant differences between the limed water and effluent treatments and unlimed water and effluent treatments; the limed water-irrigated plants were not significantly different from the unlimed effluent-irrigated plants.

 Table 5.9
 Effects of lime and irrigation source on mean dry matter yields and nutrient uptake (± SE; n =3) in above-ground biomass of maize at 6 weeks after planting in Inanda (Ia) soil

 Dry matter

Treatment*	Dry matter yield (g pot <sup>-1</sup> )		1	Nutrient (mg pot <sup>-1</sup>	)	
		N	Р	K	Ca	Mg
Ia w-L	$1.40\pm0.06a^{\#}$	$34.0\pm0.39b$	$1.15\pm0.09a$	$19.2 \pm 1.20 \text{b}$	$18.7 \pm 1.42c$	$3.69\pm0.17\text{b}$
Ia e-L	$2.17{\pm}0.20b$	$47.2\pm4.50b$	$2.05\pm0.25b$	$32.1\pm2.64 d$	$23.3 \pm 1.66 \text{d}$	$5.78\pm0.58c$
Ia w-no L	$1.10 \pm 0.06a$	19.6 ± 0.26a	$0.86 \pm 0.04a$	$5.60\pm0.47a$	$4.51\pm0.25a$	$2.30\pm0.14a$
Ia e-no L	$2.67\pm0.09c$	54.6 ± 1.21c	$3.17 \pm 0.31c$	$20.8 \pm 0.43 bc$	$8.61 \pm 0.48 b$	$7.19 \pm 0.22 d$
C.V <sup>©</sup> (%)	11.1	10.5	19.7	13.2	14.2	12.0

\*Irrigated with water, limed (Ia w-L).

Irrigated with effluent, limed (Ia e-L).

Irrigated with water, not limed (Ia w-no L).

Irrigated with effluent, not limed (Ia e-no L).

<sup>#</sup>Means followed by the same letter within each column are not significantly different

(p<0.05).

 $^{\odot}$ C.V = coefficient of variation.

Calcium and Mg showed a similar pattern to one another where the plant accumulation of each element was significantly different across treatments. The higher Ca content in the limed plants was expected due to the Ca additions that made more available for uptake. Potassium recorded the highest values (as in Table 5.5) owing to the fact that maize takes up much more K compared to other cationic elements (Fageria *et al.*, 1991) despite the relatively low K compared to Ca and Mg in sewage effluent (Feigin *et al.*, 1991; Emongor and Ramolemana, 2004; Arienzo *et al.*, 2009). Potassium concentrations in sewage effluents were reported in the range of 24 mg L<sup>-1</sup> in Israel (Feigin *et al.*, 1991), and 25 mg L<sup>-1</sup> in Botswana (Emongor and Ramolemana, 2004). Despite these values being more than those recorded for K in ABR effluent, maize was still able to accumulate more K. Plants accumulated up to about 4, 2 and 3 times more K, Ca and Mg, respectively, in unlimed soils irrigated with effluent than with water showing the potential contained in this effluent (Table 5.9).

#### pH and exchangeable acidity

The pH of the limed soils was significantly higher than that of the unlimed soils regardless of the irrigation solutions (Table 5.10). This was expected as the  $Ca(OH)_2$  had a stronger neutralising effect than the effluent. On the other hand exchangeable acidity of the soil decreased in the limed soils and was significantly lower in the effluent-irrigated than in the water-irrigated treatments for the unlimed soils (Table 5.10). This is because soil pH changes more as a function of the cations in soil solution than the pH of the irrigation solution (Bouwer and Idelovitch, 1987). Falkiner and Smith (1997) also observed this inverse relationship between soil pH and exchangeable acidity after sewage effluent application.

Treatment*	pH (KCl)	EC (dS m <sup>-1</sup> )	Exchangeable acidity (cmolc kg <sup>-1</sup> )	N (mg ł	P (xg <sup>-1</sup> )	K ((	Ca cmol <sub>c</sub> kg <sup>-1</sup>	Mg
Ia w-L	4.25b <sup>#</sup>	0.20ab	0.99a	6599a	16.2b	0.07a	6.43b	0.20b
Ia e-L	4.28b	0.28c	0.87a	6788a	19.9b	0.08a	6.46b	0.41d
Ia w-no L	3.90a	0.16a	3.10c	5803a	9.74a	0.07a	0.59a	0.14a
Ia e-no L	3.91a	0.19a	2.89b	6360a	17.6b	0.07a	0.70a	0.31c
<b>C.V<sup>©</sup> (%)</b>	0.7	14.6	5.5	7.3	15.6	13.2	2.4	11.3
Initial values	4.06	0.09	4.31	6234	15.6	0.13	0.56	0.21

Table 5.10 Effects of lime and irrigation source on mean (± SE; n =3) soil pH, electrical conductivity (EC), exchangeable acidity and major elements 6 weeks after planting in Inanda (Ia) soil

\*Irrigated with water, limed (Ia w-L).

Irrigated with effluent, limed (Ia e-L).

Irrigated with water, not limed (Ia w-no L).

Irrigated with effluent, not limed (Ia e-no L).

<sup>#</sup>Means followed by the same letter within each column are not significantly different (p<0.05).

<sup> $\odot$ </sup> C.V = coefficient of variation.

#### Electrical conductivity (EC)

The EC was significantly higher in the effluent-irrigated and limed soils than in other treatments. There was no significant difference between the Ia w-L, Ia w no-L and Ia e no-L. Addition of lime contributed to the higher EC values as evident in the high Ca content of the soil (Table 5.10), more so in the effluent-irrigated pots as compared with the unlimed soils. Although the soil Ca content of the Ia w-L was not significantly different from the Ia e-L, the effect on EC could be attributed to other cations such as Mg.

#### Soil nutrient concentrations

Concentrations of major plant nutrients in soil after harvest (Table 5.10) generally show that concentrations of N, P and Mg were in the order Ia e-L > Ia e no-L > Ia w-L > Ia w no-L. There was no significant difference between the treatments with respect to N. This could arise from the already high N status of the Ia such that additional N did not have further effects in soil.

A significant increase in P on the limed water-irrigated soil showed that liming encouraged P release from retention sites but the pH increase was not enough to cause appreciable P release into solution. On the effluent-irrigated soils, the P status in the unlimed soils was not significantly different from that of the limed soils. Judging from the pH increase (Table 5.10) more P was released from the limed and effluent-irrigated soils but with the non-significant effects observed, there is evidence of an inhibition of P from the effluent by liming. As with P, effluent irrigation improved soil Mg with the Ia e-L having the highest soil Mg probably from the Mg application on the limed soils at planting. Phosphorus and Mg concentrations doubled when irrigating with effluent as opposed to water in the unlimed soils. The opposing effect due to liming was absent in this case, so P and Mg could be the most affected by the lime application. As expected, the lime application tremendously increased the Ca concentrations to an extent that uptake by the plant could not deplete the soil concentration and resulted in the significantly higher concentrations in the limed soils. Soil K was unaffected by the treatments most likely as a result of the low K fixing capacity of kaolin (Talibudeen and Goulding, 1983; Sardi and Csitari, 1998) which is the predominant clay mineral in the Ia. In addition to this, the Ia was limiting in K for maize growth and thus all available K either released due to liming or from the effluent was taken up by the plants so the effects could have been masked in soil.

#### 5.3.3 Effluent–lime interactions

From the above observations there is an apparent interaction between lime and the irrigation solution in terms of certain parameters because plants irrigated with effluent and not limed did not show P deficiency symptoms as seen in the plants growing in the limed soils irrigated with either solution (Plate 5.3). It could possibly be that the effluent has liming capabilities emanating from its Ca and Mg content and the further addition of a liming material triggers an antagonistic reaction between the effluent and lime where the action of one is inhibited by the other. These interaction effects on various plant parameters are shown in Figure 5.1. Significant interaction effects (p<0.05) were observed except for Ca and K (Figures 5.1d and e) concentrations. Liming resulted in higher plant Ca and K content than in the unlimed soil for both irrigation solutions showing a main effect of irrigation solution and liming but no interaction. High Ca addition from lime application and maize being a high K uptake crop could partially explain why irrigating with either solution could not suppress effects manifested by liming on the concentrations of these two elements in plants. In the case of other parameters (Figures 5.1a, b, c, f), there was an interaction as well as a main effect of irrigation solution.

Phosphorus removal from sewage effluent by adsorption and chemical precipitation are the most common and cheapest techniques that have been employed (Oguz, 2004; Can and Yildiz, 2006). Lime is one of the materials used to achieve this process which may suggest that application of lime to the Ia resulted in a chemical reaction between the effluent and lime that made P to be unavailable to plants. Although P in soil was not significantly different between the Ia e-L and Ia e-no L (Table 5.10), the former was significantly higher than the latter in the plants (Table 5.9). This shows that there was interference in P uptake by plants in the limed soils and explains the P deficiency symptoms manifested by plants growing in the limed soil as opposed to those on the unlimed soil (Plate 5.3).

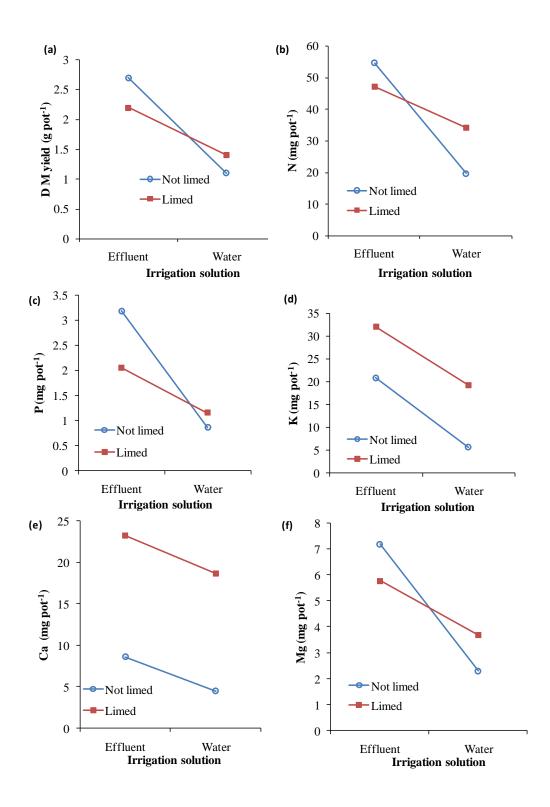


Figure 5.1 Interaction between lime and effluent on (a) dry matter yield (DM), (b) N,(c)P, (d) K, (e) Ca and (f) Mg concentrations in above-ground biomass of maize at 6 weeks after planting, irrigated with effluent or water in the Inanda soil.

#### 5.4 Conclusions

The ABR effluent used to irrigate maize improved dry matter yields and nutrient concentrations compared to similar treatments irrigated with water. The unfertilized effluentirrigated plants were equivalent to the water-irrigated plants with half the recommended fertilizer rate. This shows that the effluent can supplement fertilizer use for maize thus reducing cost of fertilizer application. Effluent N cannot substitute for the N requirements for maize and thus supplementary N is required. Comparisons within the unlimed treatments show the ability of the effluent to sustain plant growth as opposed to the water treatments reinforcing the fertigation component of the effluent. Lime application increased the Ca concentrations in soil after harvest but this was not influenced by the irrigation. Residual effects on soil were quite substantial for P and Mg concentrations which could have implications for the subsequent crop that is planted. The P status after harvest suggests that P supplied by the effluent could match that being released as a consequence of lime application. Although liming and irrigation solutions had masking effects in the soil, the effects were manifested on nutrient uptake by the plants. The response shown by the sandy soil clearly indicates that the soil properties will determine how much nutrients will be available for uptake by plants. The fact that plants grown on the sandy soil produced more biomass does not mean that it will be able to sustain growth during later maturation stages and as such this finding is inconclusive and requires more investigation.

The potential of the effluent as a 'liming agent' lies in its Ca and Mg content and not in its pH. The interaction effect manifested on certain plant parameters (dry matter yield, N, P and Mg) suggests that the lime and effluent interact to a point where the action of one is being inhibited by the other in the Ia soil and this is further investigated in Chapter 6.

# Chapter 6

# CHARACTERISATION OF ANAEROBIC BAFFLED REACTOR EFFLUENT AND ITS EFFECTS ON SOILS AND GROWTH OF MAIZE IN LEACHING COLUMNS

#### 6.1 Introduction

The anaerobic baffled reactor (ABR) effluent used so far has demonstrated its capacity for use as an irrigation and fertilizer source. The leaching columns (Chapter 4) showed soil's ability to immobilize some elements from the effluent while maize was able to take up some of these elements (Chapter 5). The effluent was void of heavy metals but there was a need for partitioning into the liquid and solid fractions to establish an understanding of their availability. The immobilization of major plant nutrients in the effluent by the sandy soil called for more research to gain insight into the likely mechanisms responsible. Also, a real life irrigation of plants would occur concurrently with water leaching down the soil profile. These investigations were therefore to:

- compare a new batch of effluent with that previously collected in order to ascertain the stability of effluent as well as availability of elements;
- vevaluate the changes in leachate characteristics after ABR effluent application to the Cartref (same as used in Chapter 5) soil to the Longlands soil previously used in Chapter 3; and
- assess uptake of plant nutrients from effluent by maize along with the leaching process.

#### 6.2 Materials and methods

#### 6.2.1 Effluent and distilled water collection and characterisation

A new batch of effluent was collected from the same pilot plant in the School of Chemical Engineering, UKZN, Durban in 25 L plastic containers and transported to Pietermaritzburg.

Aliquots of the effluent were analysed directly on unfiltered samples. An aliquot was centrifuged at 25364g for 15 minutes to separate the residue. One litre of centrifuged effluent gave approximately 0.5 g of wet residue. The residue was subjected to two different treatments. 0.3 g was digested with nitric acid for determination of elements while 0.25 g was used for Kjeldahl digestion as in Section 5.2.1 for determination of total N. The different fractions of the effluent and distilled water were analysed as follows. The pH and EC were measured on a Radiometer PHM 210 meter and a CDM 210 electrical conductivity meter, respectively.  $NH_4^+$ -N and  $NO_3^-$ -N were analysed with a TRAACS 2000 continuous flow auto-analyser. Total N was determined by vapour distillation with magnesium oxide and Devarda's alloy (Rowell, 1994). Total carbon was measured with a Shidmadzu carbon analyser. The elemental composition of the effluent was analysed by inductively coupled plasma emission spectrometry (ICP, Varian 720-ES). The *Escherichia coli* composition was determined using a modification of the effluent on eosin methylene blue (EMB) agar plates and counting colonies formed after incubation at 35°C for 48 hours.

#### 6.2.2 Soil leaching column experiment-Cartref (Cf) soil

The same PVC columns used in Section 3.2.2 were filled with the Cf soil and uniformly tapped on the bench top to achieve a bulk density of 1.47 g cm<sup>-3</sup>. Columns were leached with either ABR effluent or distilled water in triplicate (total of 6 columns). Prior to leaching the columns were saturated with distilled water by capillary wetting. With an assumed particle density of 2.65 g cm<sup>-3</sup>, a pore volume for the Cf was calculated as 168 mL (Rowell, 1994). Each leaching event comprised of a drip flow from the top into the columns according to the hydraulic properties of the Cf soil resulting in a flow rate of 11.7 cm hr<sup>-1</sup>. Columns were leached with a total of 16 pore volumes over a period of 23 weeks with each leaching event taking place once every 7 days. After pore volume 12, columns were allowed to equilibrate for 4 weeks thereafter leaching commenced again at 2 weeks intervals except for pore volume 16 that was carried out after 3 weeks. Leachate samples from each leaching event were collected and analyzed immediately for  $NH_4^+$ -N,  $NO_3^-$ -N, pH and EC. Meanwhile aliquots of 100 mL from each column were acidified with 0.5 mL nitric acid and stored at 4°C for determination of elemental composition.

Despite results from earlier investigations showing immobilization of some elements by the soil and their uptake by maize plants, these processes were, however, investigated independently from one another. It is thus of interest to have both leaching and plant growth taking place together.

The columns consisted of polyvinyl chloride 360 mm long columns (inner diameter = 100 mm) with a fine stainless-steel mesh attached to the base. Glass wool was placed on the mesh in order to minimize soil loss during leaching and a funnel fitted over the base to channel the leachate for collection. The Cartref (Cf), Inanda (Ia) and Sepane (Se) soils (the same as used for the pot trials in Chapter 5) were placed in the columns to a height of 350 mm by constantly tapping on the bench to achieve bulk densities of 1.47 g cm<sup>-3</sup>, 0.77 g cm<sup>-3</sup> and 1.21 g cm<sup>-3</sup>, respectively. No fertilizer amendment was used and maize variety PAN 4P-767BR was planted at 8 seeds per pot and thinned to 4 seeds 2 weeks after germination. This was done in triplicate for each soil giving a total of 9 columns. The columns were mounted on tripod stands and placed as a completely randomized design in the glasshouse at the University of KwaZulu-Natal (UKZN), Pietermaritzburg with maximum and minimum temperatures of 26°C and 16°C, respectively, as shown in Plate 6.1.

Soil in each column was moistened with about 1200 mL, 1900 mL and 1500 mL of the effluent (Section 6.2.1) for the Cf, Ia and Se, respectively, calculated as a pore volume of the mass of soil in each column. Afterwards the columns were irrigated with the same effluent following evapotranspiration needs, recording volume of effluent added at every irrigation time and making sure that no loss of leachate occurred. Leaching of columns commenced at two weeks after planting and this was done once per week at 3, 4, 5 and 6 weeks by adding 100 mL of effluent to each column until the required amount had leached through which was usually between 100-150 mL of leachate within a duration of 4-5 hours. Leachates were collected in glass bottles and an aliquot used for pH, EC,  $NH_4^+$ -N and  $NO_3^-$ -N determination immediately while about 50 mL was acidified and stored at 4°C for elemental determination as before (Section 6.2.1).

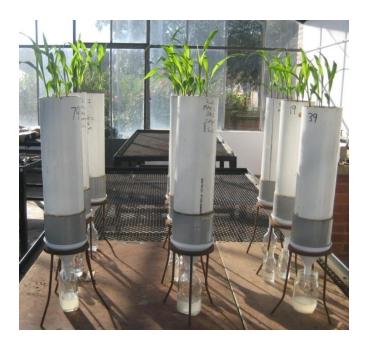


Plate 6.1The plant-soil leaching column experimental set-up at two weeks after<br/>planting.

Maize was harvested at 6 weeks after planting and analysis of the above-ground dry matter yield was performed (Section 5.2.1).

## 6.3 Results and discussion

#### 6.3.1 Effluent characterisation

Characterisation of the effluent (Table 6.1) shows various attributes of the effluent when unfiltered, filtered and in the residue. Comparatively this batch of effluent was similar to that collected for the previous investigations (Chapters 3 and 5) with regards to heavy metal composition as these were below permissible levels according to the South African Department of Water Affairs and Forestry (DWAF) (1996) and Food and Agricultural Organisation (Ayers and Westcot, 1985; Pescod 1992) quality limits for irrigation water. However, the heavy metals were higher in the residue than in solution confirming results from studies showing that the heavy metals tend to stay within the biosolid fraction of sewage sludge (Cameron *et al.*, 1997; Behbahaninia *et al.*, 2010).

Parameter	Effluent ± SE	Centrifuged supernatant ± SE	Digested residue ± SE	Distilled water	South African irrigation water quality <sup>a</sup>	FAO <sup>b</sup>
Electrical conductivity(dS m <sup>-1</sup> )	0.64 ± 0 (3)*	0.64 ± 0 (3)	nd	0.004 ± 0 (16)	<0.4	0-3
рН	$7.40 \pm 0.01$ (3)	$7.38 \pm 0.01$ (3)	nd	$6.14 \pm 0.13$ (16)	6.5-8.4	6.5-8.4
Total dissolved solids (TDS) (mg $L^{-1}$ )	442 ± 33.5 (3)	-	-	-	-	<2000
Elements	${ m mg}~{ m L}^{-1}$	$mg L^{-1}$	g kg <sup>-1#</sup>	$mg L^{-1}$	mg L <sup>-1</sup>	mg L <sup>-1</sup>
Nitrate-Nitrogen	bd	nd	nd	bd		0-10
Ammonium- Nitrogen	$13.7 \pm 0.02$ (2)	nd	nd	bd		0-5
Phosphorus	$38.7 \pm 0.14$ (5)	$39.9 \pm 0.46$ (3)	8.57 ± 0.08 (2)	bd	-	<2
Potassium	$16.3 \pm 0.06$ (5)	$16.6 \pm 0.30$ (3)	$1.34 \pm 0.02$ (2)	bd	-	<2
Sulphur	$11.1 \pm 0.08$ (5)	$11.4 \pm 0.14$ (3)	13.3 ± 0 (2)	bd	-	<960
Calcium	$24.9 \pm 0.21$ (5)	$25.4 \pm 0.46$ (3)	19.42± 0.07 (2)	bd	-	<400
Magnesium	$33.3 \pm 0.29$ (5)	$33.8 \pm 0.68$ (3)	$4.23 \pm 0.05$ (2)	bd	-	<61
Sodium	$33.5 \pm 0.18$ (5)	35.1 ± 0.53 (3)	$1.89 \pm 0.01$ (2)	bd	<70	0-69
Aluminium	bd	bd	$6.27 \pm 0.09$ (2)	bd	<5	5
Carbon	$24.2 \pm 0.21$ (2)	nd	nd	bd	-	-
Cadmium	bd	bd	bd	bd	< 0.01	0.01
Cobalt	bd	bd	bd	bd	0.05	0.05
Chromium	0.01 ± 0 (5)	0.01 ± 0 (3)	0.14 ± 0 (2)	bd	0.1	0.1
Copper	bd	bd	$0.49 \pm 0.01$ (2)	bd	<0.2	0.2
Iron	$0.33 \pm 0.1$ (5)	0.21 ± 0 (3)	$5.44 \pm 0.07$ (2)	bd	<5	5

**Table 6.1**Properties of ABR effluent and distilled water compared with irrigation water quality guidelines

		Centrifuged	Digested residue	Distilled	South Africa	
Parameter	Effluent ± SE	supernatant $\pm$	± SE	water	irrigation	FAO <sup>b</sup>
		SE	± SE	water	water quality <sup>a</sup>	
Manganese	$0.06 \pm 0$ (5)	0.06 ± 0 (3)	0.54 ± 0.01 (2)	bd	< 0.02	0.2
Molybdenum	$0.04 \pm 0.01$ (5)	$0.03 \pm 0$ (3)	$0.04 \pm 0$ (2)	bd	< 0.01	0.01
Nickel	$0.001 \pm 0(5)$	0.01 ± 0 (3)	0.09 ± 0 (2)	bd	< 0.2	0.2
Lead	bd	$0.02 \pm 0.01$ (3)	0.01 ± 0 (2)	bd	< 0.2	5
Selenium	$0.02 \pm 0.01$ (5)	$0.01 \pm 0$ (3)	$0.17 \pm 0.01$ (2)	bd	< 0.02	0.02
Vanadium	$0.02 \pm 0$ (5)	$0.02 \pm 0$ (3)	$0.04 \pm 0$ (2)	bd	< 0.1	0.1
Zinc	$0.01 \pm 0$ (5)	bd	$1.55 \pm 0.02$ (2)	bd	<1	2
Boron	$0.07 \pm 0$ (5)	$0.05 \pm 0$ (3)	$0.08 \pm 0.03$ (2)	bd	<0.5	<0.7
Bicarbonate	245.6 ± 1.52 (2)	nd	nd	-	-	<610
Chloride	48.6 ± 3.5 (2)	nd	nd	-	<100	<350
<i>E.coli</i> count- colony forming unit (cfu ml <sup>-1</sup> )	$2.8 \times 10^4 (2)$	$0.2x \ 10^4 \ (2)$	nd	bd	<1	<100
Sodium adsorption ratio (SAR)	1.03 (5)	nd	nd	-	<2	<9

Table 6.1 cont'd Properties of ABR effluent and distilled water	compared with irrigation water	quality guidelines
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\* n in brackets; ±SE- standard error of mean

bd below detection

nd not determined

Dash means no standard developed

<sup>#</sup> Equivalent oven dry residue

<sup>a</sup> Department of Water Affairs and Forestry (DWAF), 1996

<sup>b</sup>Food and Agricultural Organisation (Ayers and Westcot, 1985, Pescod, 1992).

The 0.5 g of residue obtained from 1 L of unfiltered effluent translates to about 0.04 g equivalent dry weight. This implies that to attain the concentrations mentioned in Table 6.1, about 23 kL of effluent must have been generated. The major elements, namely P, K, S, Ca and Mg, had higher concentrations than in the previously used effluent with K and S concentrations double those in the previous effluent. Consistency was shown in the Ca:Mg ratio (1:1.3) with Na, NO<sub>3</sub><sup>-</sup>-N and NH<sub>4</sub><sup>+</sup>-N unchanged.

The EC of the effluent, though meeting the FAO standards, were above the DWAF guidelines but these recommend that a 95% relative yield of moderately salt sensitive crops can be achieved using a low frequency irrigation system.

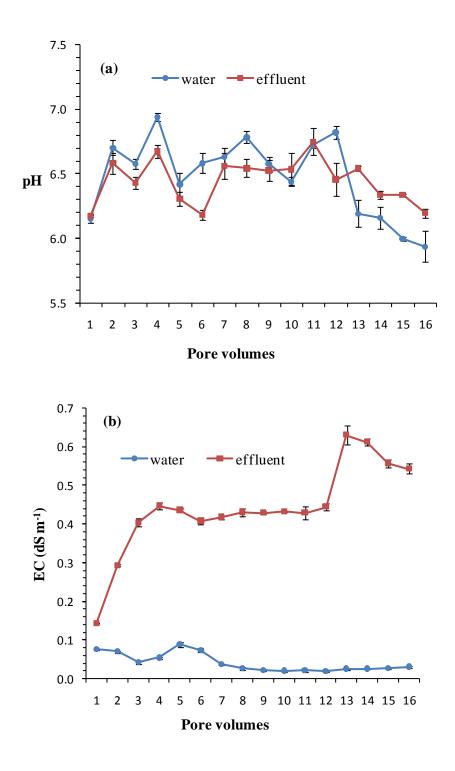
Analysis of the distilled water showed that pH and EC ranges were below those of the effluent with all elements being below detection levels (Table 6.1).

#### 6.3.2 Soil leaching column experiment-Cartref (Cf) soil

#### 6.3.2.1 pH and electrical conductivity (EC)

The pH of leachates from both the effluent-leached and water-leached columns showed very slight differences with leachates from the water having slightly higher pH values than those from the effluent. At pore volume (PV) 13 the reverse occurs but not significantly (Figure 6.1a). Possible reasons could be that the slightly acidic Cf, does not contain enough buffering capacity to impact on the pH of the effluent and water. However, after considerable leaching, the soil had minimal impact on the leaching solutions.

The water leachates had consistently low EC values close to zero (Figure 6.1b), due to the soil solution becoming more dilute as ions were leached out of the soil. The EC of the effluent leachates increased from PV 1-4 then stabilised between PV 6 and 12. The EC of leachates from the effluent treated columns had a characteristic peak at PV 13 then decreased to about 0.5 dS m<sup>-1</sup>. The two week break in leaching allowed the soil to equilibrate resulting in leachate EC (0.63 dS m<sup>-1</sup>) close to that of the incoming effluent. This supports the fact that the soil had minimal impact on the EC of the leaching solutions after considerable leaching.



**Figure 6.1** The (a) pH and (b) electrical conductivity (EC) (± SE; n=3) of Cartref (Cf) soil leached with water or ABR effluent.

#### 6.3.2.2 Leachate concentrations of major elements

Nitrate-N and  $NH_4^+$ -N represented the inorganic nitrogen fraction. In the effluent-leached columns,  $NO_3^-$ -N in the leachates increased with each leaching event up to PV 4 then

decreased steadily to PV 11 before increasing and stabilising at PV 13 (Figure 6.2a). Ammonium-N concentration in the leachates was lower than  $NO_3^-$ -N and by PV 6 it was in trace amount in the leachates (Figure 6.2a). A secondary maximum peak occurred at PV 13 before stabilising near zero at PV 15. The fact that the columns were subjected to longer intervals (2 weeks) between leaching events towards the end of the experiment could explain this trend.

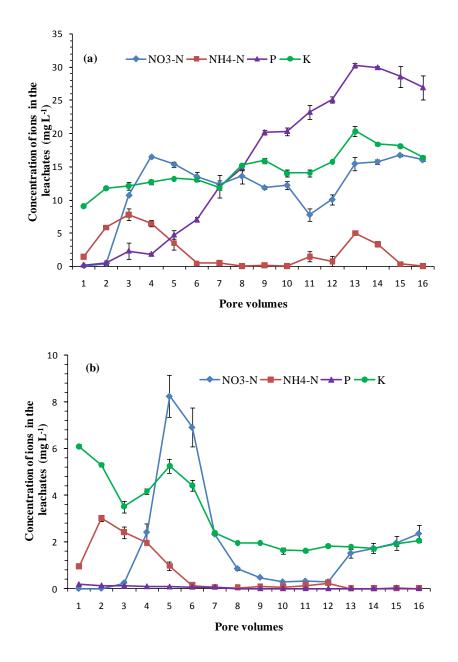
This pattern of leaching could be as a result of the wetting and drying progress which triggered release of ions into solution. Cui and Caldwell (1997) reported that wetting of a dry soil could trigger a pulse of available soil nitrate in a field plot, apparently within a short duration. The concentration of  $NO_3^-$ -N in the leachates at the end of leaching was almost equal to the  $NH_4^+$ -N concentration in the effluent at the start of leaching. At the beginning of leaching,  $NO_3^-$ -N was absent from the incoming effluent but as leaching progressed, the  $NO_3^-$ -N concentration of the leachates gradually increased while that of  $NH_4^+$ -N decreased. It could be that with time,  $NH_4^+$ -N in the effluent was gradually converting to the nitrate form contributing to the trend shown in the effluent-leached soils.

This was not the case with the water-leached columns where the highest  $NO_3^{-}N$  concentration in the leachate was 8.2 mg L<sup>-1</sup> at PV 5 as opposed to 16.7 mg L<sup>-1</sup> in the effluent at PV 4 (Figure 6.2b). Ammonium-N in the water leachates was apparent at the initial PVs with PV 2 having the highest amount and after PV 6 there was no  $NH_4^+$ -N effect. Most of the  $NO_3^-$ -N was leached between PV 3 and 8 as the  $NO_3^-$ -N stabilised at PV 9-12 before increasing at PV 13. The same reason for the sudden increase in  $NO_3^-$ -N in the effluent leachates could also be used to explain this but the differences lie in the intensity and amount released in such a process.

Phosphorus concentration in leachates from the effluent-leached columns was low initially and with every leaching event the concentration increased almost equating with that of the incoming effluent at PV 13 (Figure 6.2a). The amount of P leached from the water-leached columns was negligible (Figure 6.2b) which shows that most of the P leached from the effluent-leached columns was that from the incoming effluent. The P concentration in the Cf soil was very low (Table 5.1) further explaining the low P in leachates from the water-leached columns.

Potassium in leachates from the effluent gradually increased with each leaching event and also peaked at PV 13 like the  $NO_3^--N$ ,  $NH_4^+-N$  and P with the trend being more like that of

the  $NO_3$ -N (Figure 6.2a). At PV 16, the K concentration was similar to that in the incoming effluent.



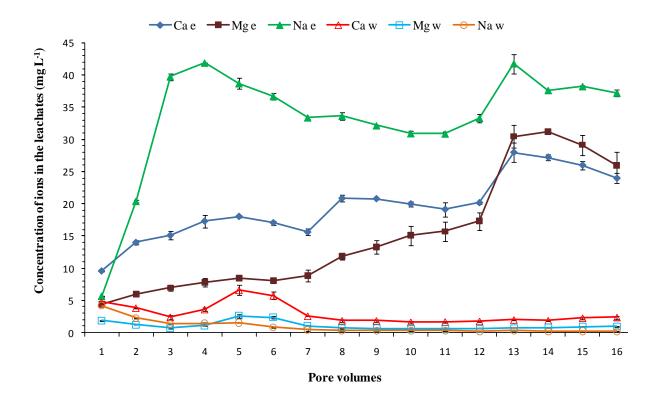
**Figure 6.2** Leachate concentrations of  $NO_3^--N$ ,  $NH_4^+-N$ , P and K ( $\pm$  SE; n=3) leached with (a) effluent or (b) water from Cartref soil columns.

In the water-leached columns the K concentration trend was also similar to that of the  $NO_3$ -N, declining from PV 5 and remaining quite constant from PV 10 to PV 16 indicating a

steady loss of K from the soil as the amounts in the distilled water were below detection (Figure 6.2b).

In effluent irrigation, the concentration of exchangeable cations in relation to each other is very critical because of the impact on soil structure. Concentrations of Ca, Mg and Na in the leachates (Figure 6.3) show the ability of Na to flow through the columns thus preventing its accumulation in soil. The Na concentration was higher than in the incoming effluent towards the end of leaching. In the water-leached columns most Na was leached out of the columns in the initial pore volumes and became less in the later stages of leaching.

Magnesium was retained preferentially to Ca with leachates having higher Ca than Mg concentrations from both the effluent and water-leached columns (Figure 6.3). In the effluentleached columns this could be attributed to relatively higher Mg than Ca in the effluent resulting in more Mg retained on exchange sites than Ca causing the latter to remain in solution. In the water-leached columns since ions were being lost from the soil, the higher soil Ca relative to Mg promoted Ca being leached faster than Mg. However, at pore volume 13 the reverse occurred in the effluent-leached columns with more Mg in the leachates than Ca (Figure 6.3). This suggests that the concentrations of these two cations in the effluent could possibly be the driving force behind the changes. At inception of leaching, soil Ca:Mg ratio was about 3:1(Table 5.1; Section 5.2.1) so additional calcium from the effluent could no longer be retained resulting in most of it being leached out in solution. In the course of leaching, there was a build-up of Mg resulting in a similar situation as seen for the Ca and with the concentration of Mg being higher than Ca in the effluent more Mg was then leached through. This change occurred after the columns were allowed to equilibrate for 4 weeks further supporting the fact that the effluent was the driving force in these changes for this was not the case with the water-leached columns. In addition after the 4 week break in leaching, the concentrations of Ca and Mg in the leachates at PV 13 were almost equal to each other. Thereafter concentrations of both elements decreased with Ca to a value close to that of the incoming effluent. The concentration of Mg in the effluent at PV 16 was below that of the incoming effluent suggesting continued preferential retention of Mg over Ca in the soil column.



**Figure 6.3** Concentrations of Ca, Mg and Na ( $\pm$  SE; n=3) in leachates from Cartref soil columns leached with effluent (e) or water (w).

#### 6.3.2.3 Elemental balance

An elemental balance shows that while there was a gain of elements in the effluent-leached columns, the water-leached columns experienced a loss of elements (Table 6.2). The highest gain was that of P. Although at the end of leaching P in the leachates was close to that in the incoming effluent, the gain that occurred at the beginning of leaching accounted for the overall gain in P. It was expected that the Cf would not gain P due to its sandy texture but this was not the case. The amount gained was almost equal to the P gained in the Lo (Section 3.3.2.3). Phosphorus retention exceeded recommended levels for maize in the Cf thus a maize crop in this soil would not likely benefit from further P input. Considering the sandy texture of the soil, maize roots could readily source the P. High retention of P in the Cf could be explained by the immobile nature of P even in this sandy soil. However, at the end of leaching the amount of P being retained was very low compared to that in the incoming effluent which attests to the relatively low sorption capacity the Cf soil has for P over a long leaching period. There seems to be an inverse relationship between P and total C in the leachates (Figure 6.4) especially in the early stages (to PV 7). There was also a loss of C from

effluent-leached soil but relatively less than from the water-leached soil (Table 6.2). The water-leached columns had a negative balance because of no addition of elements from the distilled water which rather leached elements out of the soil. When compared to the Lo soil used in Chapter 3 (Table 3.4) the trends are very similar and in the case of the effluent-treated columns similar values were obtained (Table 6.2). The main difference is in the total C value which in the Lo showed a gain (105.8 kg ha<sup>-1</sup>) but in the Cf showed a loss (60.8 kg ha<sup>-1</sup>). This could be due to the coarser texture of the Cf (greater amount of coarse sand and less clay) allowing for less C retention.

# **Table 6.2**Inorganic-N (In-N), P, K, Ca, Mg and total C gain or loss in Cartref soiltreated with either effluent or distilled water

		Effluent	Water
	In-N	34.50	-48.45#
	Р	535.46	-1.0
Total loss or gain (kg ha <sup>-1</sup> )	Κ	68.61	-60.23
	Ca	155.36	-59.01
	Mg	428.43	-23.03
	Total C	-60.8	-198.59
Nutriant maninements of	N	200	
Nutrient requirements of	Р	80	
irrigated maize (kg ha <sup>-1</sup> )*	K	100	

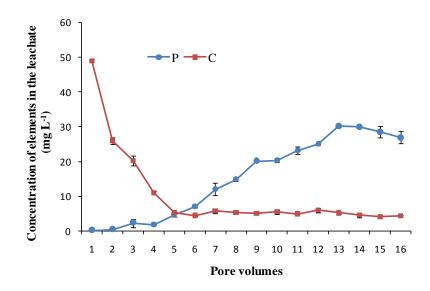
<sup>†</sup> negative values mean loss from soil.

\*Soil Fertility and Analytical Services Division (Department of Agriculture, Cedara, KwaZulu-Natal).

The inorganic-N gain in the soil was determined by  $NH_4^+$ -N as it was held by the negatively charged soil colloids unlike the  $NO_3^-$ -N. Despite the inorganic-N retention the N requirement for irrigated maize was not met (Table 6.2) and thus supplementary N application would be needed to satisfy N demand.

Potassium gain was less compared to the other elements and this gain cannot support a maize crop considering the fact that maize has an aggressive demand for K. Calcium and Mg were

both retained in large amounts though as indicated above (Section 6.3.2.2) Mg was retained preferentially to Ca.



**Figure 6.4** Concentrations of P and C ( $\pm$  SE; n=3) in leachates from Cf soil columns after effluent application.

#### 6.3.3 Plant-soil leaching column experiment

#### 6.3.3.1 Soil leachate variables

pH and electrical conductivity (EC)

The pH of the leachates was not considerably altered with progress in leaching and plant growth for the Cf and Se soils (Figure 6.5a). The Ia soil, however, had a gradual rise in pH with the leachates finally reaching a pH of 5 at the final leaching event. There was a significant impact on the acidity in the Ia as a result of the effluent addition.

The EC of the leachates consistently decreased initially (Figure 6.5b). After effluent application there was likely an adsorption of cations by the soils and these were available for uptake by the maize plants. Continuous application of effluent reached a point where the soil exchange complex became saturated and also the input of nutrients exceeded plant uptake. At

this point the EC of the leachates stabilised or started to increase as evident in the Se and Cf but not in the Ia (Figure 6.5b). In the Ia the Ca and Mg from the effluent was used to satisfy the liming needs of this soil and with progress in leaching less of these elements were leached from the soil. The EC correlates highly with the Ca and Mg leaching from the soil as evident in Figure 6.6 with low EC values corresponding to low Ca and Mg concentrations in the leachates and *vice versa*. The Ca concentration in the leachates was consistently higher than Mg as was the case with the laboratory leached columns (Section 6.3.2.2).

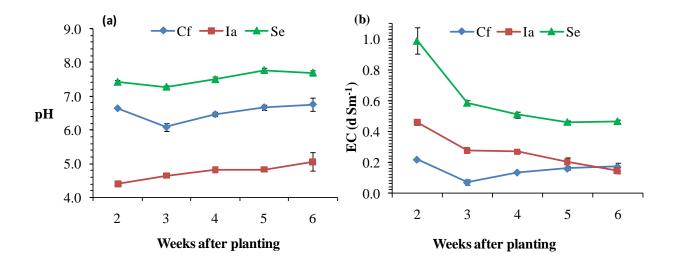


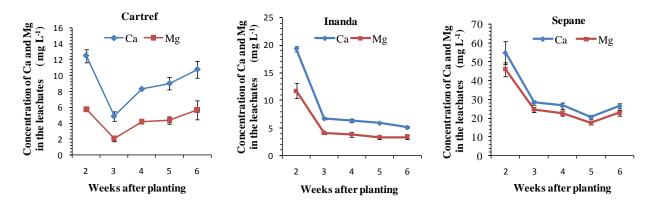
Figure 6.5The (a) pH and (b) electrical conductivity (EC) (± SE; n=3) of leachates from<br/>Cartref (Cf), Inanda (Ia) and Sepane (Se) soil columns planted with maize.

Leaching of major plant nutrients

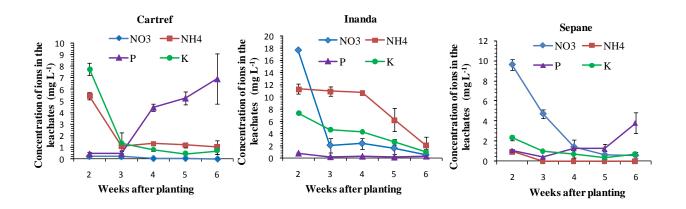
It was expected that concentrations of plant nutrients in the leachates would decrease with progressive leaching as a result of plant uptake but P did not adhere to this trend in the Cf and Se soils (Figures 6.7a and c). This expectation was met by leachates from the Ia soil with P almost not detectable in the leachates. Phosphorus behaviour in the Cf and Se could be explained partly by the supply of P from the effluent being more than the plants could absorb within the growth period. The Cf being unable to retain such amounts resulted in P leaching out of the soil. The low adsorption capacity of the Cf as opposed to the Se and Ia could partly be responsible for P leaching. Comparatively, P leaching from the planted columns (Figure 6.7) showed similar trends to the laboratory columns (Figures 6.2 and 3.2) with the exception

of the Lo which was not used in the planted columns. The presence of plants changed the nutrient dynamics of  $NO_3^--N$ ,  $NH_4^+-N$  and K within the columns resulting in uptake of these ions thereby minimising the amount of nutrients leached out of the columns (Figure 6.7). The inorganic N forms were readily available for maize uptake and the high affinity of maize for K changed the leaching pattern from that obtained in the laboratory leached columns (Figures 6.2 and 3.2).

At harvest an elemental balance was done as a function of volume of effluent added minus leachate collected (Table 6.3). Despite plant uptake, there was a gain in major plant nutrients recorded in all soils. The P gain was again higher than all other elements probably due to the high P content in the effluent and the same reason could be advanced for the Mg gain being higher than Ca for each soil.



**Figure 6.6** Concentrations of Ca and Mg (± SE; n=3) in leachates from soil columns planted with maize and leached with effluent.



**Figure 6.7** Concentrations of NO3--N, NH4+-N, P and K (± SE; n=3) in leachates from soil columns planted with maize and leached with effluent.

The elemental gain was similar in the Ia and Se soils for the major elements except for Ca and Mg where there was a slightly higher gain in the Ia than in the Se. These similarities depict the retention ability of these soils although from different causes (Section 3.3.2.3). Amounts gained in the Cf soil were all lower than in the other soils, reflecting the coarser texture of this soil. The effluent retained was approximately 2.5 L, 3.3 L and 3.1 L in the Cf, Ia and Se, respectively. This translates to 2709 L, 3600 L and 3382 L for the Cf, Ia and Se, respectively, on a per hectare basis. The total pore volumes added were 2.7, 2.1 and 2.6 for the Cf, Ia and Se, respectively.

		Cartref	Inanda	Sepane
	In-N	48.1	51.1	56.5
	Р	133	166	161
Total gain (kg ha <sup>-1</sup> )	K	55.4	67.1	67.5
	Ca	80.6	101	80.5
	Mg	113	139	119
	Total C	56.9	67.1	68.9
Nutrient requirements of	In-N	200	200	200
-	Р	80	20	60
irrigated maize $(kg ha^{-1})^{\#}$	Κ	100	205	10

**Table 6.3**Inorganic-N (In-N), P, K, Ca, Mg and total C gain in soil leaching columnsplanted with maize and irrigated with effluent

\* Cartref (Cf), Inanda (Ia), Sepane (Se) (Soil Classification Working Group, 1991).
<sup>#</sup>Soil Fertility and Analytical Services Division (Department of Agriculture, Cedara, KwaZulu-Natal)

#### 6.3.3.2 Plant variables

Growth parameters and dry matter yields

Growth was vigorous on the Ia and Se as opposed to the Cf as evidenced by the plant height and number of leaves (Table 6.4). This eventually resulted in significantly higher (p<0.05) biomass production in the Ia and Se than in the Cf. Visually growth was slow in the Cf and at 6 weeks after planting the plants from the Cf were stunted and showing N deficiency symptoms characterised by the yellowing of older leaves (Plate 6.2). This yellowing was also shown by plants in the Se but not on plants grown on the Ia (Plate 6.2).

**Table 6.4**Mean growth parameters and dry matter yields (± SE; n=3) of maize grown in<br/>soil leaching columns irrigated with effluent

Soil form*	Plant height (mm)	Number of leaves plant <sup>-1</sup>	Dry matter yield (g pot <sup>-1</sup> )
Cf	$575 \pm 9.38a^{\#}$	$7.00 \pm 0a$	$5.40 \pm 0.15a$
Ia	$629 \pm 10.7 ab$	$7.40\pm0.08b$	$7.80\pm0.3b$
Se	$652 \pm 12.6b$	$7.30\pm0b$	$8.20\pm0.3b$

\* Cartref (Cf), Inanda (Ia), Sepane (Se) (Soil Classification Working Group, 1991).

<sup>#</sup> Significant differences at p< 0.05 level indicated by different letters within the column.



Plate 6.2Growth of maize (6 weeks) in Cartref (Cf), Inanda (Ia) and Sepane (Se) soilsin leaching columns irrigated with effluent.

#### Plant nutrient uptake

Maize uptake of major plant nutrients was significantly different between the soils for most elements (Table 6.5). Yellowing of leaves in the Cf and Se was as a result of significantly

lower N concentration in the plants from these soils than in plants from the Ia (Table 6.5). This further explains the absence of N deficiency symptoms in the Ia plants. In terms of meeting the N requirements of maize the N gained from the effluent was limiting (Table 6.3), thus plants thrived on the inherent N of the soil. The Cf soil being low in N readily showed N deficiency symptoms. The N content in the Se was also inadequate to satisfy the maize N needs.

Although there was more P gained in the Se and Ia soils than in the Cf (Table 6.3) the plant P accumulation was not significantly different between the soils (Table 6.5). The Cf soil does not affect P availability unlike the Ia and Se. The high P fixing capacity of the Ia caused by the presence of Al and Fe oxides and the high clay content of the Se affected optimum P uptake by the plants. Despite the relatively lower P gained by the Cf from the effluent it was available to the plants.

Plant K content (Table 6.5) was significantly different among the soils and was in the order Se > Cf > Ia. The inherent K in the soils played a major role in the uptake as the amounts gained from the effluent were similar to one another. The low uptake of K in the Ia soil was caused by the inherently low K status of the Ia soil attributed to the kaolin type clay which has an inability to retain K (Talibudeen and Goulding, 1983; Sardi and Csitari, 1998).

Plant Ca accumulation was significantly lower in the Cf and Ia than in the Se. It is possible that the Ca retained from the effluent was used to alter acidity of the Ia soil while most of the Ca was leached out of the Cf soil. Uptake of Mg was significantly greater in the Ia and Se than in the Cf although its retention trend was similar to that of the Ca.

An observation was the higher uptake of nutrients from the plant-soil leaching columns when compared to the uptake in pot experiment 2 for the Ia (Table 5.9) considering that the amount of soil was almost the same. The length of the column could have given the roots an advantage in downward growth creating more room for the maize plant to source for nutrients.

Soil		N	utrient (mg pot <sup>-1</sup> )	)	
form*	Ν	Р	К	Ca	Mg
Cf	$48.4 \pm 1.48a^{\#}$	$14.7\pm0.42a$	$85.0\pm2.84b$	21.1 ± 1.97a	18.8 ± 0.51a
Ia	$102 \pm 5.17c$	$14.0\pm1.23a$	$52.0\pm2.70a$	19.5 ± 1.26a	$31.1 \pm 1.65 b$
Se	$76.2 \pm 2.49b$	$11.0 \pm 0.36a$	$159 \pm 6.48c$	$26.6\pm0.38b$	$34.3 \pm 1.45b$

**Table 6.5**Mean nutrient uptake (± SE; n=3) in above-ground maize biomass from<br/>leaching columns irrigated with effluent

\* Cartref (Cf), Inanda (Ia), Sepane (Se) (Soil Classification Working Group, 1991).

<sup>#</sup> Significant differences at p < 0.05 level indicated by different letters within the column.

#### 6.3.3.3 Soil properties at harvest

Soil properties varied after irrigation and leaching compared to the soil properties before planting (Table 6.6). There were slight changes in pH and EC before and after harvest in all soils and a larger difference in exchangeable acidity in the Ia soil. The N status of the Cf and Se soils improved slightly at harvest but a small decrease in soil N for the Ia soil suggested high N uptake as shown by the absence of N deficiency in plants grown on the Ia. The effluent contributed strongly in raising soil P for all soils especially in the Cf. In spite of P leaching towards the end of the experiment the soil was able to immobilize part of it. Compared to the initial soil concentration, K was lower in all soils confirming the high K uptake by maize. In the Ia and Cf, Ca and Mg remained unchanged from initial values but Mg was slightly lower and Ca higher than the initial values in the Se.

An evaluation of the soil properties after harvest for the Ia columns did not show marked differences from those of the same soil after harvest in pot experiment 2 (Section 5.3.2.3, Table 5.10). In pot experiment 2, about 3.5 L of effluent was used for irrigation against a volume of about 3.3 L retained in the plant-soil leaching columns. As such there were similarities in the soil properties between Table 5.10 and Table 6.6 suggesting that even under leaching conditions the soil can retain plant nutrients from the effluent.

	, ui		kets) at hai vest					
Soil form*	pH (KCl)	EC (dS m <sup>-1</sup> )	Exchangeable acidity (cmolc kg <sup>-1</sup> )	N	Р	K	Ca	Mg
				mg l	kg <sup>-1</sup>	(	cmolc kg <sup>-</sup>	1
Cf	5.68	0.05	0.05	478	12.6	0.02	1.11	0.51
	(4.62)	(0.02)	(0.06)	(352)	(2.11)	(0.10)	(1.11)	(0.45)
Ia	4.56	0.08	2.87	5845	20.5	0.06	0.58	0.18
	(4.06)	(0.09)	(4.31)	(6234)	(15.6)	(0.13)	(0.56)	(0.21)
Se	6.63	0.11	0.06	2142	9.16	0.18	8.68	6.80
	(5.92)	(0.10)	(0.08)	(2087)	(5.22)	(0.26)	(8.23)	(7.39)
LSD <sub>5%</sub>	0.17	0.01	0.05	455	8.42	0.01	0.21	0.07

**Table 6.6**Effects of irrigation and leaching on mean  $(\pm SE; n=3)$  soil properties (initial<br/>values in brackets) at harvest

\* Cartref (Cf), Inanda (Ia), Sepane (Se) (Soil Classification Working Group, 1991).

#### 6.4 Conclusions

These results indicate that the major plant elements are in soluble form as they are very low in the residue from the effluent while the reverse is the case for trace elements. The amount of effluent used will determine the rate of residue build-up in soil. This accumulation is indicative of how much effluent has to be applied to the soil before the elements exceed their permissible levels assuming no uptake by plants. The hydraulic conductivity of the Cf enhanced greater flow of mobile elements explaining the lower retention of inorganic-N and K by the soil. The leaching process resulted in water being retained in soil and it is by this retention that most elements were gained in soil. The wetting and drying process between leaching events therefore played a major role in retention and release of elements in soil. Leaching in the presence of plants gave similar leachate characteristics as leaching with no plants (laboratory soil leaching columns). The similarities were in the trends shown by the elements but in terms of amounts leached, the presence of plants reduced the elemental loss. The nutrient balance indicated that there could be an N deficiency due to the low N status of the effluent but this is inconclusive as leaching aided in N being removed from the root zone.

It could be that the rate of N uptake was slower than the supply by the effluent coupled with the mobile nature of N and the stage of growth. In a field situation where water demand depends on the crop stage, the N balance could turn out to be different. The concentrations of major plant nutrients in soil at harvest were a reflection of plant uptake especially in the plant growth and simultaneous leaching results obtained could be used to project the amount of effluent needed to grow a maize crop in the field. On the basis of pore volumes added it could be complex in comparing the laboratory and the planted columns. These were run separately and with controlled conditions for the laboratory columns. Additionally, the planted columns had a crop factor included and so irrigation was not based on pore volumes as was the case in the laboratory leaching columns.

### Chapter 7

# EFFECT OF LIMING AND ANAEROBIC BAFFLED REACTOR EFFLUENT IRRIGATION ON SOIL PROPERTIES AND GROWTH OF MAIZE ON TWO STRONGLY ACID SOILS

#### 7.1 Introduction

Wastewater treatment plants have often used lime to remove P from water in a bid to avoid P build-up that leads to eutrophication of water bodies. The removal of P from wastewaters depends on the purpose for reuse. Irrigation with anaerobic baffled reactor (ABR) effluent was seen to lower exchangeable acidity in acid soils suggesting that the effluent possesses liming properties (Chapter 5). However, such irrigation on limed soils resulted in plants showing P deficiency symptoms (Chapter 5). Lime application on acid soils could bind P and inhibit its supply from the effluent thus depriving the plants of this nutrient. This phenomenon implies there is an antagonistic interaction between the effluent and the lime which needs to be investigated.

Research on the liming capabilities of wastewaters is very limited. Soil pH increments of about half a pH unit have been reported in Australia (Falkiner and Smith, 1997), New Zealand (Schipper *et al.*, 1996) and the USA (Kim and Burger, 1997) using secondary treated sewage effluent. Sparling *et al.* (2001) have reported a soil pH increase of 1.8 units after 22 years of irrigation on New Zealand volcanic ash and pumice soils.

The unavailability of P for uptake by plants could be due to adsorption and chemical precipitation by lime as highlighted in Chapter 5. Chemical precipitations with compounds of aluminium, calcium and iron have been used for P removal from wastewater (Tchobanoglous *et al.*, 2005).

The objectives of this chapter are therefore to;

- establish effects of lime rate and type on maize grown on different soils, irrigated with ABR effluent; and
- > evaluate changes in soil chemical properties following irrigation with ABR effluent.

#### 7.2 Materials and methods

#### 7.2.1 Soils

Two soil types were used in this investigation. The A horizons of an Avalon (Av) form (Soil Classification Working Group, 1991); Plinthic Paleudult (Soil Survey Staff, 2010) collected from Geluksberg, KwaZulu-Natal and an Inanda (the same used in the previous investigations reported in Chapter 5). The reason for choice of soils was mainly their acidity and also differences in certain characteristics, especially their organic carbon content, were considered.

#### 7.2.2 Pot experiment

One kilogram of each soil which had been air-dried, milled and sieved to pass a 2 mm sieve was weighed into pots (inner diameter of 18 cm, height of 14.5 cm). Lime was applied from two sources, either dolomite (CaMg(CO<sub>3</sub>)<sub>2</sub> ground and sieved to pass a 250 $\mu$ m sieve) or calcium hydroxide (Ca(OH)<sub>2</sub>) at rates of 0%, 25%, 50%, 75% and 100% of the recommended rate for these soils. In the Ia these rates corresponded to 0, 1.1, 2.2, 3.2 and 4.3 g pot<sup>-1</sup> for dolomite and 0, 0.6, 1.2, 1.8 and 2.4 g pot<sup>-1</sup> for calcium hydroxide. In the Av soil application rates were 0, 0.7, 1.3, 2.0 and 2.6 g pot<sup>-1</sup> for dolomite and 0, 0.4, 0.8, 1.1 and 1.5 g pot<sup>-1</sup> for calcium hydroxide. Fertilizer was applied to all pots to meet the recommendation of 200 kg N ha<sup>-1</sup>, 20 kg P ha<sup>-1</sup> and 205 kg K ha<sup>-1</sup> for Ia; 200 kg N ha<sup>-1</sup> and 20 kg P ha<sup>-1</sup> for Av to avoid nutrient supply becoming a limiting factor. The Av soil met the K recommendation for maize so no K fertilizer was applied. A total of 54 pots were placed in the glasshouse in a randomized complete block design with three replicates.

Maize (PAN 4P-767BR) was planted at eight seeds per pot later thinned to four plants two weeks after planting. Watering of pots was done with ABR effluent (same batch as used in

Chapter 6) following evapotranspiration needs. After 6 weeks of growth, plant height and number of leaves were recorded and the experiment was terminated by cutting the plants at about 1 cm above the soil surface. Plant samples were oven dried at 70°C in a forced draught oven and thereafter weighed to get the above-ground dry matter yield. Soil samples were collected form each pot, air dried, sieved to less than 2mm and sent to the Soil Fertility and Analytical Services Division of the KwaZulu-Natal Department of Agriculture, Cedara for chemical analysis. Dried plant samples were ground for total N determination by Kjeldahl digestion (Rowell, 1994). Phosphorus, K, Ca and Mg were determined by inductively coupled plasma emission spectrometry (ICP, Varian 720-ES) after nitric acid digestion (Titshall, 2007). Data were analysed using Genstat 12.1 and the Student Newman Keul range test at 5% was used to determine differences between treatment means.

#### 7.3 Results and discussion

#### 7.3.1 Soils

Physicochemical analysis of the two soils (Table 7.1) showed that they were very similar in pH and EC but differed mainly in their organic carbon, total N, trace elements and texture. The trace elements in Ia were much higher than in the Av, while the Ia was a loam and the Av a sandy clay loam. The mineralogy of the Ia is described in Section 5.3.1. The Av is a highly weathered soil dominated by kaolinite but with less Fe oxides (Farina *et al.*, 2000) as shown in Table 7.1.

#### 7.3.2 Plant growth and dry matter yields

Non-significant effects were recorded for plant height, number of leaves and dry matter yields regardless of lime source and rate (Table 7.2). This implies that in the soils that had no lime application, plant growth and dry matter yields were not significantly different from soils to which lime had been applied regardless of liming rate. This again shows that the effluent has liming capabilities which could sustain plant growth in acidic soils. Although most plants grown on limed soils recorded higher plant height, these were not significantly different from plants on the unlimed soils. Above-ground biomass as demonstrated by dry matter yields (Table 7.2) further showed non-significant effects due to lime type and rate.

		Soil form	and horizon
Parameter		Av A	Ia A
pH	(H <sub>2</sub> O)	4.13	4.44
pm	(1M KCl)	3.84	4.06
Electrical conductivity (dS m <sup>-1</sup> )		0.08	0.09
Organic C (g 100g <sup>-1</sup> )		1.68	7.54
Total N (mg kg <sup>-1</sup> ) <sup>#</sup>		909	6234
	Ca	0.40	0.56
Extractable base cations $(\text{cmol}_{c} \text{ kg}^{-1})^{\#}$	Mg	0.21	0.21
	K	0.43	0.13
Exchangeable acidity (cmol <sub>c</sub> kg <sup>-1</sup> ) #		2.78	4.31
Total exchangeable cations $(\text{cmol}_c \text{kg}^{-1})^{\#}$		3.82	5.21
Acid saturation (%) #		72.8	82.7
	Mn	4.96	6.49
Extractable metal cations $(mg kg^{-1})^{\#}$	Cu	1.49	1.95
	Zn	0.91	0.78
Extractable P (mg kg <sup>-1</sup> ) #		18.2	15.6
Particle size (%)			
Coarse sand (0.5-2 mm)		2.1	4.1
Medium sand (0.25-0.5 mm)		5.7	7.6
Fine sand (0.053-0.25 mm)		38.7	18.2
Silt (0.002-0.053 mm)		18.2	48.2
Clay (<0.002 mm)		35.3	21.9
Clay mineralogical composition (%) <sup>b</sup>			
Vermiculite		*	*
Mixed-layer minerals		**	-
Mica		*	-
Kaolin		***	**
Quartz		-	tr
Feldspar		-	tr
Goethite		-	*
Gibbsite		-	**

 Table 7.1
 Some characteristics of the Avalon and Inanda A horizons used in the pot experiments

<sup>a</sup>Avalon (Av), Inanda (Ia) (Soil Classification Working Group, 1991).

<sup>b</sup>Clay minerological composition of the Avalon-Farina *et al.* (2000).

<sup>#</sup> Analysis conducted by the Soil Fertility and Analytical Services Division (KwaZulu-Natal Department of

Agriculture, Cedara).

\*\*\* > 60

\*\* 20-60

\* 5-20

tr < 5

- not found

Table 7.2	Growth parameters and dry matter yields at 6 weeks after planting ( $\pm$ SE; n=3)
	of maize grown on Inanda (Ia) and Avalon (Av) as affected by lime type and
	rate

Soil form*	Lime type <sup>α</sup>	Lime rate (%)	Plant height (mm)	Number of leaves plant <sup>-1</sup>	Dry matter yield g pot <sup>-1</sup>
	control	0	$321 \pm 8.30 ab^{\#}$	6.00 ± 0a	$1.63 \pm 0.03a$
		25	312 ± 19.7a	$6.00 \pm 0a$	$1.33\pm0.20a$
	D	50	351 ± 15.6abc	$6.00 \pm 0a$	$1.80 \pm 0.21 a$
	D	75	$340 \pm 15.4 abc$	$6.00 \pm 0a$	$1.66 \pm 0.19a$
Ia		100	355 ± 22.5abc	6.00 ± 0a	$1.60\pm0.25a$
	С	25	$325\pm 6.00 ab$	$6.00 \pm 0a$	$1.26\pm0.12a$
		50	$348 \pm 10.0 abc$	$6.00 \pm 0a$	$1.63\pm0.12a$
		75	$340 \pm 12.2abc$	$6.00 \pm 0a$	$1.23\pm0.07a$
		100	381 ± 26.6abc	6.10 ± 0.10a	$2.03 \pm 0.44a$
	control	0	341 ± 18.2abc	5.53 ± 0.25a	$1.46 \pm 0.23a$
		25	$344 \pm 7.88 abc$	$5.43\pm0.22a$	$1.33\pm0.09a$
	D	50	$358 \pm 9.29 abc$	$5.77\pm0.14a$	$1.33\pm0.03a$
	D	75	$310 \pm 4.26 abc$	$5.77\pm0.14a$	$1.60\pm0.06a$
Av		100	380 ± 9.22abc	6.00 ± 0a	$1.66\pm0.09a$
		25	$359 \pm 8.97 abc$	$5.83\pm0.17a$	$1.36\pm0.09a$
	С	50	$363 \pm 6.00 abc$	$5.70\pm0.08a$	$1.33\pm0.07a$
	C	75	$403 \pm 7.31c$	$5.70\pm0.08a$	$1.86 \pm 0.12a$
		100	$388 \pm 16.3bc$	$5.93 \pm 0.08a$	$1.76 \pm 0.20a$

\* Avalon (Av), Inanda (Ia) (Soil Classification Working Group, 1991).

<sup> $\alpha$ </sup> D- dolomite, C- Ca(OH)<sub>2</sub>.

<sup>#</sup> Significant differences between treatments at p < 0.05 level indicated by different letters in the column within soils.

#### 7.3.3 Nutrient uptake in plants

There was no significant difference in N accumulation in plants as affected by lime type and rate within each soil (Table 7.3). Lime application had no effect on N uptake as the control was not significantly different from the limed soils both for lime type and rate within each soil. The significantly higher N accumulation from the Ia control treatment than from the Av control treatment confirms the higher initial N in the Ia soil which was about six times that in the Av. Liming, however, enhanced N uptake in the Avalon to the extent that the dominance of the Ia over the Av in supplying N was masked (Table 7.3).

Phosphorus accumulation was unaffected by lime type and rate with non-significant effects across soil type. This shows that the effluent alone was able to impact on P availability and further liming did not trigger any response to P. Although liming had non-significant effects, the dolomitic lime applied to the Ia showed higher P uptake in the control than at the 100% rate. Evidence of this was shown by the P deficiency symptoms in plants across all lime type and rates (Plate 7.1).

Plant K accumulation was non-significant from the control to the highest rate for both lime types in the Ia but significant effects were recorded in the Av soil between the control and the 75% Ca(OH)<sub>2</sub> application. It is important to note that at planting the Av soil did not receive K supplementation as the soil analysis showed adequate K for maize growth. Liming of soils increases the Ca concentration in soil solution which depresses K uptake (Uexkull, 1986). With more Ca being supplied from both the effluent and the lime its concentration probably suppressed that of the K from the effluent. Also maize has an aggressive uptake for K so the K was absorbed faster from the soil again leaving more Ca in the soil solution.

Calcium uptake was significantly higher in the 50%, 75% and 100% than the control for dolomite and between the 100% and control for  $Ca(OH)_2$  in the Ia In the Av significant effects occurred only between the control and the 75% and 100%  $Ca(OH)_2$  application. Within the limed treatments there was no significant increase in Ca uptake with increase in lime rate. The Ca both from lime addition and effluent rather suppressed soil acidity than being available for uptake. There was a significantly higher uptake in the Ca(OH)<sub>2</sub> than in the dolomite treated soils at the 100% application rate for the Ia soil. The greater neutralising influence of the Ca(OH)<sub>2</sub> could be responsible for this effect

Concentration of Mg in plants was highly variable with application rate, lime type and soil type. Dolomite application increased Mg uptake compared to  $Ca(OH)_2$  in the Ia. With the additional Mg in dolomite (Section 7.2.2) it was expected that this would boost Mg accumulation in plants. In the Av, although an increase occurred, this was not significant. Magnesium uptake was significantly different between the lower and higher application rates for dolomitic lime as opposed to the non-significant effects across the Ca(OH)<sub>2</sub> application rates in both soils. The control was not significantly different from the limed treatments for each soil.

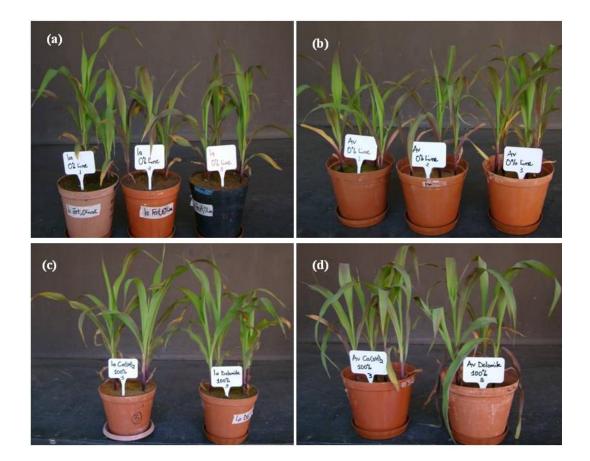


Plate 7.1 Growth of maize (6 weeks) on Inanda (Ia); (a) not limed (c) 100% limed and Avalon(Av); (b) not limed and (d) 100% limed with dolomite or calcium hydroxide.

Soil	Lime	Lime	N	Р	K	Ca	Mg				
form <sup>*</sup>	* 0		rate (%) Nutrient (mg pot <sup>-1</sup> )								
	control	0	$45.0 \pm 1.63b^{\#}$	$1.36 \pm 0.05a$	33.8 ± 0.30ab	$4.06 \pm 0.40a$	$5.56 \pm 0.42$ abc				
		25	$40.8\pm6.54ab$	$1.22 \pm 0.28a$	$29.5\pm5.33a$	$5.35\pm0.87ab$	$4.15\pm0.73ab$				
	D	50	$49.7\pm8.91ab$	$1.59\pm0.17a$	$39.5 \pm 3.60 abc$	$11.0 \pm 1.41$ bcd	$7.93 \pm 1.25c$				
	D	75	$52.2 \pm 3.22ab$	$1.55 \pm 0.15a$	$37.5 \pm 2.13$ abc	$10.9 \pm 0.34 bcd$	$7.65\pm0.68c$				
Ia		100	51.4 ± 8.96ab	$1.30 \pm 0.22a$	$35.2 \pm 4.86ab$	$11.0 \pm 2.00$ bcd	7.89 ± 1.63c				
		25	$41.3 \pm 4.10$ ab	$1.07\pm0.10a$	$29.4 \pm 3.16a$	$7.66 \pm 0.79$ abcd	$3.89 \pm 0.38ab$				
	С	50	55.1 ± 1.39ab	$1.45 \pm 0.11a$	$38.9 \pm 3.20 abc$	$12.9 \pm 0.67$ de	$4.76\pm0.38ab$				
		75	41.9 ± 1.16ab	$1.07\pm0.04a$	$29.5 \pm 1.71a$	$10.6 \pm 0.59 bcd$	$3.68 \pm 0.30$ ab				
		100	64.1 ± 10.6b	$1.85 \pm 0.34a$	$46.6 \pm 7.64$ abc	$17.6 \pm 3.88 f$	$6.47 \pm 1.64 bc$				
	control	0	31.5 ± 4.49a	1.14 ± 0.17a	35.0 ± 4.95ab	3.21 ± 0.80a	$2.86\pm0.32a$				
		25	$29.9 \pm 1.23a$	$1.09\pm0.10a$	$36.4 \pm 3.58 abc$	$3.69 \pm 3.69a$	$2.29\pm0.21a$				
	D	50	$35.2 \pm 0.52a$	$1.02 \pm 0.04a$	$38.4 \pm 0.74$ abc	$4.82 \pm 0.15a$	$2.59\pm0.03a$				
	D	75	$35.8 \pm 2.61a$	$1.26\pm0.07a$	$48.6 \pm 1.18 abc$	$6.99 \pm 0.21$ abc	$3.67\pm0.12ab$				
Av		100	$40.3 \pm 2.81a$	$1.32 \pm 0.15a$	$51.8 \pm 3.22 bc$	8.39 ± 0.51abcd	$4.23 \pm 0.27$ ab				
		25	33.9 ± 2.41a	1.11 ± 0.10a	39.0 ± 2.47abc	5.83 ± 0.32abc	$2.56\pm0.20a$				
	С	50	$33.4 \pm 2.15a$	$1.00\pm0.10a$	41.6 ± 3.81abc	$6.94 \pm 0.65 abc$	$2.58\pm0.27ab$				
	C	75	$42.8\pm3.58ab$	$1.52\pm0.21a$	$55.1 \pm 3.79c$	$11.1 \pm 1.02 bcd$	$3.29\pm0.13ab$				
		100	$41.3 \pm 3.43 ab$	$1.40 \pm 0.16a$	$53.1 \pm 5.77 bc$	$11.8 \pm 1.54$ cd	$3.26\pm0.43ab$				

**Table 7.3**Nutrient accumulation at 6 weeks after planting (± SE; n=3) in above-ground biomass of maize grown on Inanda (Ia) and<br/>Avalon (Av) as affected by lime type and lime rate

<sup>\*</sup> Avalon (Av), Inanda (Ia) (Soil Classification Working Group, 1991).

<sup> $\alpha$ </sup> D- dolomite, C- Ca(OH)<sub>2</sub>.

<sup>#</sup> Significant differences between treatments at p < 0.05 level indicated by different letters in the column within soils.

#### 7.3.4 Acidity and extractable elements in soils

An increase from the initial pH was apparent only on limed soils with a significant increase as lime application rate increased (Table 7.4). This trend was clearly distinct in the Ia but in the Av the pH of control soils was not significantly different from that of the lower lime application rates. The alteration in soil acidity was obvious in the exchangeable acidity and acid saturation of the soils. In the Ia soil, exchangeable acidity and acid saturation was suppressed with an increase in amount of dolomite application but after the 75% application of calcium hydroxide, there was no significant difference in these soil properties. In the Av these properties increased significantly for both lime types to the highest lime application rate except for the exchangeable acidity with the 50% and 75% application rates which were not significantly different from each other. The higher percentage calcium carbonate equivalence of calcium hydroxide (136%) is evident in its ability to lower acidity more than the equivalent rate of dolomite. This explains the non-significant change in acidity in the Ia soil above the 75% application rate. The control treatments of both soils witnessed a reduction in exchangeable acidity and acid saturation than the levels measured at planting although this was not comparable to the decrease in the limed soils. Although the 100% application rate for both liming materials achieved less than 20% acid saturation this did not enhance uptake of elements.

Liming had almost no effect on soil N as there were no significant differences between the control and the limed soils across lime rates or lime type within each soil (Table 7.4). The same trend was shown in the N uptake as that in the soil N at harvest.

Phosphorus in soil generally decreased with increase in lime rate and this trend was more clearly defined for the calcium hydroxide than the dolomite, although the effects were non-significant (Table 7.4). This decrease indicates that as pH of the soil increases due to lime application, exchangeable P tends to decrease (Naidu *et al.*, 1990).

Potassium followed a similar trend to P for the Ia with an irregular trend observed for the Av soil.

Soil form*	Lime type <sup>α</sup>	Lime rate (%)	pH (1M KCl)	Exchangeable acidity (cmol <sub>c</sub> kg-1)	Acid saturation (%)	N	Р	K	Ca	Mg
						(mg	kg <sup>-1</sup> )		(cmolc kg	1)
	control	0	3.92b	2.82h	61.5h	5989bc	19.4ab	0.17ab	0.75a	0.59abc
		25	4.00c	2.12g	45.7g	6116bc	19.4ab	0.16ab	1.57b	0.81d
	D	50	4.09 de	1.75f	32.3de	6125bc	15.0a	0.14ab	2.37c	1.10e
	D	75	4.19f	1.33e	22.3bc	6470c	20.4ab	0.16ab	3.10d	1.40f
Ia		100	4.29i	0.88bc	12.7a	6042bc	22.5ab	0.16ab	4.01e	1.72g
		25	4.04cd	2.06g	38.0ef	5875bc	20.8ab	0.18b	2.54c	0.71bcd
	C	50	4.20fg	1.26de	21.0bc	5662b	17.4ab	0.14ab	4.06e	0.61abc
	С	75	4.27gh	0.98bcd	13.7a	6042bc	17.5ab	0.14ab	5.26f	0.65abcd
		100	4.36j	0.69ab	9.00a	6234bc	17.6ab	0.11a	6.48g	0.59abc
	control	0	3.79a	1.99g	56.0h	841a	27.8b	0.35f	0.60a	0.60abc
		25	3.87ab	1.66f	47.7g	780a	19.4ab	0.33ef	0.97a	0.54ab
	D	50	3.94b	1.30de	35.3e	799a	21.9ab	0.28cde	1.40b	0.69abcd
	D	75	4.03cd	1.05cde	27.0cd	888a	20.4ab	0.25c	1.80b	0.80cd
Av		100	4.14ef	0.69ab	16.3ab	889a	21.2ab	0.26cd	2.22c	1.0e
		25	3.85ab	1.61f	43.7fg	770a	23.4ab	0.28cde	1.33b	0.49ab
	C	50	3.92ab	1.28de	33.7de	771a	25.4ab	0.30cde	1.72b	0.53ab
	С	75	4.02cd	1.07cde	24.7c	849a	23.5ab	0.25c	2.47c	0.55ab
		100	4.20fg	0.51a	11.0a	776a	18.4ab	0.32def	3.41d	0.47a
<sup>©</sup> C.V (%)			0.9	9.1	10.5	7.8	18.6	9.8	9.2	10.3

**Table 7.4** Chemical analysis of the soil samples from the pots at 6 weeks after planting as affected by lime type and lime rate

\*Avalon (Av), Inanda (Ia) (Soil Classification Working Group, 1991).

<sup> $\alpha$ </sup> D- dolomite, C- Ca(OH)<sub>2</sub>.

<sup>#</sup> Significant differences between treatments at p < 0.05 level indicated by different letters.

<sup>©</sup>C.V =.coefficient of variation.

The Av soil had sufficient K before planting which might mask the effects of additional K from the effluent.

Increased soil Ca followed the increase in lime rate for both lime types and soils. This was expected as Ca from lime application greatly influenced the Ca soil content. Soil Mg recorded significant differences between lime rates only for the dolomite. This was because the dolomite application contained Mg that was lacking in the calcium hydroxide.

#### 7.4 Conclusions

There was a greater response at lower lime application rates for the Ia than for the Av especially with calcium hydroxide owing to the stronger neutralising power of calcium hydroxide compared to dolomite. Phosphorus accumulation in plants and soils was not significantly affected by lime rate and type although P decreased with higher lime application. The fact that the effluent on its own (no liming) was able to impact positively on P availability suggests that liming could be suppressing the ability of effluent to supply P for uptake. Phosphorus in soil at harvest in some cases was lower in the lime treatments than in the control suggesting that liming tends to precipitate P from the effluent which affects its uptake. This explains the inability of lime application to raise the pH sufficiently to counteract plant P uptake and also the similarities between the limed and unlimed soils. Liming of soils for maize growth is supposed to be below 20% acid saturation which was not achieved by the effluent alone. The effluent alone was able to suppress acid saturation to about 60% for both soils but despite the fact that this did not attain the 20% requirement for maize its effects were similar to those of limed soils on uptake. Increasing the lime application rate raised the Ca in soil but these increments were not reflected in plant tissue. The benefits from such increments might only ensue on a long term basis. The findings on N and K show that liming has minimal effects on the availability of these elements for uptake.

## Chapter 8

# **GENERAL DISCUSSION AND CONCLUSIONS**

#### 8.1 Introduction

Wastewater reuse as a means of water recycling and reducing pressure on freshwater is becoming increasingly popular. Sewage effluents have been shown in many cases to result in improvement of soil physical conditions and chemical properties. Their significantly higher nutrient concentrations such as N and P and their organic matter make them a valuable fertilizer material. The application of some of these effluents to soil has also resulted in adverse effects such as heavy metal accumulation, high pH changes, pathogen loadings and risk of groundwater contamination. Most effluents generated from treatment works have highly variable chemical and biological properties due to treatment method which could be aerobic, anaerobic or a combination of the two. The success of effluent reuse lies in how its physical, chemical and biological properties can be assimilated through the soil/plant system. The diversity in properties of the effluents makes it difficult and inappropriate to transfer the use of one type of effluent to another. Different soils will assimilate nutrients differently depending on their properties can be tolerated by that crop. This investigation has addressed the following questions;

- Can anaerobic baffled reactor (ABR) effluent application affect soil properties?
- Is ABR effluent able to supply the major plant nutrients through the soil/plant system for uptake by maize?
- Is there a possibility of loss of nutrients through leaching when irrigating with this effluent?
- Does the effluent suppress soil acidity?

This Chapter is a synthesis of findings of the earlier chapters set in the broader sense of soil/ plant/effluent interaction.

The ABR effluent met the requirements for reuse options from an agricultural perspective as its elemental composition represented elements essential to plant growth. It did not contain heavy metals which characterise most wastewaters even from domestic origin. It, however, did not conform to the microbiological standards as the *Escherichia coli* count was above recommended levels for wastewater reuse. This then restricts the use of such an effluent on crops eaten raw.

#### 8.2 Effects of effluent on soil properties

Retention of major plant nutrients occurred when leaching the effluent through the soil both without (Chapters 3, 4 and 6) and with plants (Chapter 5 and 6). This was quantified in the volume of effluent that leached through the soil. Despite differences in soil properties, retention occurred in all soils but the amounts retained were a function of the soil properties. The addition of effluent to soil columns resulted in lesser concentrations of major elements in the leachates. This was different when a simultaneous leaching was carried out with distilled water. More elements were found in the leachates and considering that none was added there was a net negative balance. Elemental retention from the effluent followed an irregular trend with P being the most strongly retained element. Kellman (2002) reported that the existence of Fe and Al coatings on soil particle surfaces increases the ability of soils to suppress rapid leaching of nutrient loads. This observation was confirmed in this study and attributed to the mineralogical complexity of the highly weathered soil (Ia). Although P was the most retained element even in the sandy soils the leaching pattern showed that at the later stages of leaching more P as well as other major elements were being lost from soil. This implies that there is a limit to retention and once the soil complex is saturated there is tendency for less adsorption. This could be an explanation for the increase in pH and EC at the later stages of leaching especially in the acidic soil as its leachates gradually increased towards the pH and EC of the effluent. Intermittent wetting and drying led to retention of small amounts of effluent in the soil after every leaching event which cumulatively could account for the build-up observed in the soils. An unexpected result was the preferential retention of Mg to Ca. A peculiarity of the effluent used in this study was the higher Mg than Ca concentration unlike in most wastewaters.

Deterioration of soil structure is one of the attributes associated with wastewater reuse. Although an assessment of this soil physical property was not carried out as part of this study, which has focused on soil chemical attributes, it can be indicated from the Na accumulation in the soils. This study eliminated possibilities of soil structure degradation as all the sodium was leached out of the soil. In essence this was the only element that showed breakthrough over the timespan of the experiments.

As leaching progressed, the  $NH_4^+$  in the effluent decreased with time while the  $NO_3^-$  increased in the collected leachate. The final amounts retained for each of these N species could either be coming from the change before leaching or from the changes that occurred within the soil

Elemental distribution within the column can further explain retention capacity and quantitative analysis of the form in which the elements were found. Sequential extractions on the top, middle and bottom segments of the columns (Chapter 4) were used to assess availability of the retained elements. The greater amounts of acid soluble elements in the upper segment of the columns were due to elemental loadings from the effluent in available form. This implies that there is a possibility of uptake by plants. Despite the fact that the effluent supplied more to the acid soluble fraction, this remained in the upper part of the column indicating lesser mobility down the column. The non-selectivity of extractants may increase fractions from the former phases at the later stages of extraction (Filgueiras *et al.*, 2002).

Retention of elements from the effluent was again evaluated in a plant-soil leaching situation (Chapter 6). This assessment was critical mindful of the fact that uptake of elements had to be maintained with the rate of leaching. The trend of leaching indicated less leaching of  $NO_3^-N$ ,  $NH_4^+-N$ , Ca, Mg and K which was understandable from a plant uptake perspective. In this particular situation, P was easily leached from the Cf soil. Regardless of elemental loss through leaching and plant uptake the soil was able to retain major plant nutrients.

Soil accumulation of major plant nutrients was equally observed in irrigation in closed systems (pot experiments). After harvest the soils showed an increase in major plant nutrients more than the retention found in the leaching columns (Section 6.3.3). Soil acidity improvement on effluent-irrigated soils could be attributed to the high Ca accumulation which was not the same on water-irrigated soils (Fonseca *et al.*, 2005b). The ability of the effluent to raise soil pH (investigated in the Ia soil) provided evidence that the effluent on its own could raise soil pH. Soil pH in other cases has been found to decrease after irrigation

with treated domestic sewage effluent on alkaline soils (Hussein et al., 2004). There were noticeable adverse effects on maize, especially P deficiency, when limed soils were irrigated with effluent suggesting an interaction effect between the lime and effluent which hinders the uptake of certain plant elements. Tchobanoglous et al. (2005) found that P could be precipitated out of solution by compounds of Al, Fe and Ca. Further investigations by using two liming materials at different rates compared to the effluent found that the effluent was limited in its ability to suppress soil acidity. Despite lime application soil P was not enhanced by lime addition even at higher rates as this was still not different from the unlimed soil. Phosphorus particularly was the most affected element as evident in the P deficiency symptoms shown by plants. The mechanisms responsible for P unavailability from the effluent are linked to P removal from wastewater by lime precipitation (Malhortra et al., 1964; Marani et al., 1997; Vanotti et al., 2002; Pastor et al., 2008). Calcium phosphates will be formed depending on the pH, phosphate concentration, calcium ion concentration, bicarbonate alkalinity, and reaction temperature (Jang and Kang, 2002). Vanotti et al. (2002) reported that calcium phosphate starts to form by pH 7 and by pH 9 it then precipitates out of solution. In the present study with an effluent pH of about 7.4 the possibility of P being precipitated out of the effluent cannot be ruled out. The pH of leachates from the Ia soil in the column experiment could be indicative of the change in effluent pH but no lime was added to the Ia soil during leaching.

#### 8.3 Effects of effluent on maize growth

Use of sewage effluent has become popular over the years and irrigation has been carried out with effluents of diverse characteristics. The ABR effluent has been found to improve growth, dry matter yield and nutrient uptake in maize. Fonseca *et al.* (2005a) observed that irrigation with secondary treated sewage effluent could partly substitute the nutrients required for maize growth. Likewise, this study has found that the use of effluent for irrigation at half the fertilizer application rate could be comparable to the recommended dose of fertilizer application in meeting the nutrient demands of a maize crop. Effluent irrigation has been carried out with wastewaters of lower (Fonseca *et al.*, 2005a; Singh *et al.*, 2012) and higher (Mohammad and Mazareh, 2003; Hassanli *et al.*, 2009) nutrient compositions when compared to the effluent used in this study. Notwithstanding the variability in effluent properties and in the different crop types the results in this study are comparable with those

obtained in other studies (Singh *et al.*, 2012). Maize grown on soils receiving effluent was able to gain access to the nutrients retained and immobilize them in plant tissue.

Comparisons between the pot trials and the plant leaching columns show that plant nutrient uptake increased between the soils in the same order for N and P. There was an inverse relationship in uptake between the two trials in the Ca and Mg accumulation as well as in the dry matter yields. It could be suggested that the leaching of plant nutrients affected the uptake which was not the case in the pot experiment but this is inconclusive as the two experiments were not run concurrently. Moreover, the irrigation was done with effluents from separate batches that had slightly different properties.

The liming component of the effluent was assessed along with that of liming agents and addition of lime did not increase N, P, K and Mg uptake more than the effluent (Chapter 7). The benefits gained from liming did not translate to plant nutrient uptake for these elements. Calcium uptake was the only element that was significantly influenced by liming which was definitely triggered by the increased amounts in soil.

In conclusion, the research presented in this thesis has provided an understanding of the issues surrounding ABR effluent irrigation and implications for use within the soil and the soil/crop interrelationship. The leaching column experiments showed how different soils were able to retain major elements namely N, P, K, Ca, Mg and SO<sub>4</sub><sup>2-</sup> from the effluent applied to them and their availability and depth within the column for possible uptake by plants or leaching into the environment. The pot experiments provided information on how much of the plant nutrients retained could be absorbed by maize within a specified time. Irrigating with ABR effluent could be considered as an alternative to treatment aimed at achieving the stringent standards for wastewater disposal into watercourses. It allows for the soil's contribution in accommodating pollutants harmful to water bodies which is an aspect that is not factored into most guidelines on wastewater utilisation for agriculture. The ABR effluent serves as a nutrient source for plants and this has implications for the amounts of fertilizers needed for field crops. Supplementing fertilizer application could have financial benefits especially for poor subsistence farmers who have to deal with the ever increasing prices of fertilizers. The water component gives an opportunity for dry season cropping especially in agricultural areas that depend on rainfall. From this study there is an indication that the sandy soil has minimal impact on the effluent and thus better crop response but only

in a closed system. This finding however is inconclusive as mentioned earlier and forms part of the future work.

These studies, however, have raised new questions that warrant additional investigations.

- 1. Leaching columns concurrently with different irrigating regimes with and without plants would give a clearer understanding of element retention. In this study leaching was done once a week and the results obtained were used to set-up the plant/soil leaching columns. Moreover, in leaching where plant growth is involved it would be of importance to equally leach similar columns with no plants. As such there is a platform for direct comparison on absolute concentrations rather than on a trend basis.
- The mechanisms responsible for the lime and effluent interaction have to be investigated further and this could be done by leaching columns of acidic soils with lime and fertilizer amendments.
- 3. Given that maize was planted for just six weeks and that nutrient deficiencies might have been aggravated by the plant population (Hughes *et al.*, 2005), it is essential to have appropriate plant populations over longer duration to assess the ability of the effluent to sustain nutrient uptake. A crop with regenerative growth like perennial rye grass could also be used.
- 4. A field assessment in order to ratify the reliability of nutrient supply would be obligatory. Considering that crops have stages of critical nutrient uptake and that an irrigation regime is to be followed there is a need to synchronise these processes. This will also involve monitoring element retention with nutrient uptake along with potential environmental problems. The trace elements in ABR effluent were found to be within acceptable limits but if the effluent is to be used extensively on a larger scale then a monitoring of build-up over time coupled with issues of salinity must be investigated.
- 5. In terms of response of less treated water to soil/crop, the use of a wetland to further treat the water is possible and if safe future work could compare the more treated and the less treated water in laboratory and field experiments.

- 6. The long term impact of adding Mg to soils needs further study especially in susceptible soils such as dryland soils which are prone to salinization and structural degradation.
- 7. The effect of effluent addition on the surface charge characteristics of soils by undertaking surface charge fingerprints could be undertaken. This will show the impact of effluent on increasing the capacity of the soil to retain cations through the development of variable charge.

A final note that may be of interest to the reader is that as this thesis was nearing completion, so too was the field-placed ABR at Newlands-Mashu. The effluent from this ABR, which has very similar characteristics to those used in this study, will be used in field experiments growing maize (*Zea mays*), taro (*Colocasia esculenta*), dry beans (*Phaseolus vulgaris*) and Swiss chard (*Beta vulgaris*) as from mid 2012.

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