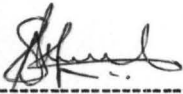


DECLARATION

I hereby declare that this project work is a record of a research work that was undertaken and written by me. It has not been presented before for any degree or diploma or certificate at any university or institution. Information derived from personal communications, published and unpublished work were duly referenced in the text.



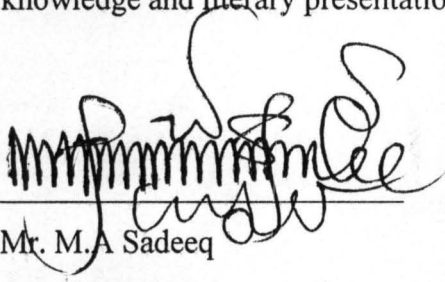
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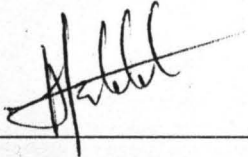
CERTIFICATION

This project entitled "Effect of Direct Wastewater Use on Spinach Cultivation (A Case Study of Soje Irrigation Scheme kpagungu, Minna, Niger State) by Awarun .O. Adekunle meets the regulations governing the award of the degree of Bachelor of Engineering (B.ENG.) of the Federal University of Technology, Minna, and it is approved for its contribution to scientific knowledge and literary presentation.



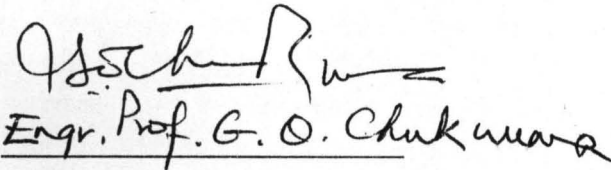
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External Examiner

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DEDICATION

This project work is dedicated to the almighty God by whose grace I am able to see this day.

ACKNOWLEDGEMENTS

This project will be incomplete if I had to express my heartfelt gratitude to God who in his infinite mercies protected and gave me the understanding during my studies at the university.

To my wonderful supervisor, Mr. Sadeeq Muhammad, whose intelligent suggestions and contributions have made this piece of work a dream come true.

To my Head of Department Engr. Dr. A.A. Balami and the entire lecturers, I extend my bountiful appreciation for their concerted efforts towards impacting knowledge in me.

To my dear parents A.V.M (rtd) and Mrs. Awarun for their financial and moral support my beloved sisters and brother, Nike, Yinka and Goke. I must say thank you for always being there for me.

I must not forget Mallam Jamil, and the staff of the Upper Niger River Basin Authority for their contributions in making this project work a reality

I am highly indebted to my friends and colleagues, Adetoye Oluwaseun, Bianca Madubuko, Hashim Mohammed, Taufiq Oyeniran, Bashir Mohammed, Tope, Quartey Seyi and Haruna Muhammed. You have been Wonderful. My course mate and pals you are not left out, I appreciate your encouragement and I say a big thank you.

ABSTRACT

This study investigates the impact of wastewater use on spinach cultivation in terms of the nutrient and phyt-available metal contents. An investigation was carried out at pilot scale to test the feasibility of using the effluent of wastewater generated from a settlement in Niger state.

The objective of the study was to carefully monitor the impact of wastewater irrigation on the nutrient value and phyt-available metal content of the crop. Experimental plots with spinach were irrigated with fresh and wastewater. The physiochemical properties of the wastewater were analyzed along with the nutrient and metal content levels of the spinach. In the case of the wastewater irrigated spinach, higher compositions of metals and lower proximate values were obtained. In the wastewater spinach, Potassium, Phosphorus, Magnesium and Calcium recorded values of 571.70mg, 50.88mg, 80.89mg and 101.20mg respectively while fresh water spinach recorded 558.70mg, 48.58mg, 79.79mg and 99.20mg respectively. When compared with the USDA standards, Carbohydrates and ash levels recorded at 5.46g and 2.45g with the maximum USDA standard value of 3.36g and 1.75g respectively. This emphasizes the need for proper analysis of water used for irrigation of agricultural produce. The greatest benefits for farmers using wastewater was reliable wastewater supply which allowed them to grow high yield vegetable crops.

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EFFECT OF DIRECT WASTEWATER USE ON SPINACH CULTIVATION

(A Case Study of Soje Irrigation Scheme Kpagungu, Minna, Niger State)

BY

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FEBRUARY, 2010

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CHAPTER ONE

1. INTRODUCTION

Water is the second most abundant free gift to nature but human activities resulting from industrial, domestic, agro-chemicals, etc. contributes immensely to water pollutants and contaminants. Nearly all waters contain metals, dissolved salts and trace elements, many of which result from the natural weathering of the earth's surface, in addition, drainage waters from irrigated lands and effluent from city sewage and industrial waste, all impact water quality. Water is one of the most abundant compounds found in nature, however, a number of trace elements are found in water which can limit its use for irrigation. Inadequate handlings of urban waste and high intensity agricultural cultivation in peri-urban areas are having effects on the environment and farmlands, Brook & Davila (2000). The direct use of wastewater may have some impacts or effects on its intended use depending on the composition of its constituents hence the effects of potentially harmful contamination are usually described as a hazard to the intended use usually when in excess, Janssen *et al.* (2000). Therefore excessive mineral and trace metals may pose risk to man and his environment. In most irrigation situations, the primary water quality concern is minerals and salinity levels, since these can affect both the soil structure and crop yield.

The use of industrial or municipal wastewater in agriculture is a common practice in many parts of the world, Ensink *et al.* (2002) and is becoming more important due to increasing water scarcity in arid and semi-arid regions of the world, including countries like Nigeria, therefore the standards for wastewater reuse in many countries have been influenced by the WHO (2006) Health Guidelines and the USDA (2005) Guidelines. Since then, epidemiological studies have

been carried out by London School of Hygiene and Tropical Medicine, with colleagues in Mexico and Indonesia, and microbiological studies of crops irrigated with treated wastewater have been carried out by Leeds University, with colleagues in Brazil and Portugal, to assess the validity of these guidelines. Water use in Africa has also increased rapidly during the last decade especially in agriculture; this increase is attributed to increased farming, population growth, urbanization, and economic development. In the Africa, if no additional water resources are developed, severe water scarcity is expected to begin in the nearest future. One possible new source of water is reclaimed wastewater.

The use of industrial or municipal wastewater in agriculture is a common practice in many parts of the world (WHO 2006). Wastewater can be said the spent water of a community, a combination of the liquid and water-carried waste from residences, commercial buildings together with any ground water, surface water and storm water that may be present. Water we use never really goes away. In fact, there never will anymore or any less water on earth than there is right now, which means that all of the wastewater generated by our communities or environment each day from our homes, farms, businesses and factories eventually returns to the environment to be used again, either through the direct re-use of this wastewater or a process known as the hydrological cycle. Rough estimates indicate that at least 20 million hectares in 50 countries are irrigated with raw or partially treated wastewater. Scott et al (2004).

1.1 Soje Irrigation Scheme

This study is performed in the Soje settlement located in Minna, the Niger State capital. Small scale faming is practiced. The irrigated farm plots are adjacent to a wastewater flow.

Farmers have constructed small dams and weirs in the wastewater to divert water through hand constructed canals to the lower areas of the farm plots, while centrifugal pumps are used to

irrigate crops at higher elevations of the farm plots. With a typical plot size is 60 x 20 meters farmers grow vegetables which are mainly grown for the local market. Irrigation and other farm work are usually carried out by members of the family. The use of water from the wastewater for irrigation is informal. Although residential areas are situated upstream of the wastewater flow, slums are located on the slopes above. Waste and wastewater from residences are dumped onto the streets and into natural drainage channels from where they find their way into the water channel.

Human activities influences the presence of wastewater in any environment, therefore, the type of activities and practices has great impact on the quantity of the wastewaters' constituents and consequently defines the type. The wastewater body in Soje settlement is contained by numerous pollutants such as; sewage and domestic waste Agricultural discharges, toxic metals, Siltation, fertilizers and detergents. These pollutants have great influence on the water constituents and properties which are contained in various concentrations.

1.1.2 Spinach Cultivation

Spinach, (*Spinacia oleracea*) is one of the annual crops cultivated in the settlement. Spinach (is an edible flowering plant in the family of Amaranthaceae. It is native to central and southwestern Asia. It is an annual plant (rarely biennial), which grows to a height of up to 30 cm. The leaves are alternate, simple, and ovate to triangular-based, very variable in size from about 2-30 cm long and 1-15 cm broad, with larger leaves at the base of the plant and small leaves higher on the flowering stem. Spinach is a dark green leafy vegetable whose leaves can be eaten raw or cooked, the smooth seeded form that is used today in commercial production. It is grown for its nutritious and savory leaves. It contains Iron, vitamin A, and vitamin B2 (riboflavin). Spinach leaves are picked from the immature plants when in the form of rosette, close to the ground. It is a fast growing crop which produces harvestable leaves in about 40 days after seeding. The

cultivation of such food crops has to be carefully monitored for safety in terms of quality standards and nutriment values since it is produced to be directly consumed, hence, to prevent any negative outbreaks and epidemic.

1.3 Statement of Problem

In September 2006, the United States suffered a major outbreak of *E. coli* O157:H7, causing hundreds of reported injuries and several deaths and resulting in spinach recall (FDA 2006). Most farmers practice irrigation as long as there is a constant water source. With the persistence in untreated wastewater irrigation especially on vegetables, the practice suggests that the irrigators believe the benefits outweigh any perception of risks of food toxicity, which this study stands to clarify. However, this indicates the importance of safety especially in crop produce and knowledge in aspects of crop quality standards.

1.4 Scope of Study

The study focuses on a range of metals such as Magnesium, Chromium, Potassium, Calcium, Iron and Phosphorus composition and the impacts on vegetables cultivation in the Soje irrigation scheme.

1.5 Objective of Study

The objectives of this study include:

- To determine the common metals available within the wastewater and its effect on the nutriment value of spinach cultivated in the area.
- To determine the total and phyt-available contents of mineral metals (Mg, Fe, K, Ca and P) in spinach cultivated in the area.

1.6 Justification of Study

From experimental studies, it could be seen that wastewater in general contains various soluble and insoluble chemical and biological constituents contained in varying proportions, with some of these constituents having favorable and unfavorable effects when directly re-used in crop cultivation. Therefore, there is an urgent need to increase understanding of the effects of untreated wastewater and bio solids for agricultural cultivation in terms of nutrient value and potential risks. Predictive modeling tools combined with experimental data addressing the effect of waste products and trace metal content in wastewater irrigation are necessary for appropriate use of such on agricultural lands and for the improvement of consumers security.

The knowledge of the various constituents contained in the wastewater bodies used for irrigation of crops (spinach) in the Soje irrigation scheme can be checked with the irrigation water quality Standards. Hence the knock-on effect of the wastewater use on crops cultivation could be identified

CHAPTER TWO

2. LITERATURE REVIEW

Droughts, explosive population growth in arid areas of the country, and the continuing view that water is an infinite resource are some of the reasons for water shortages in many areas across the nation. In response to this problem, some wastewater professionals are reusing treated wastewater and have found it to be a reliable alternative water source. In addition to conserving highly treated, expensive drinking water, wastewater reuse reduces the release of nutrient-rich wastewater into environmentally stressed streams and rivers. Wastewater can be viewed as a resource, fresh water containing plant nutrients (nitrogen, phosphorus, and potassium), in the groundwater, these nutrients are a pollutant, but on a growing crop or turf, they are a resource. When wastewater is reused, it is not available to pollute the groundwater supply.

2.1 Wastewater

The spent water of a community, a combination of the liquid and water-carried waste from residences, commercial buildings together with any ground water, surface water and storm water that may be present. It is any water that has been affected in quality by anthropogenic influence. It encompasses a wide range of potential contaminants and concentrations. In the most usage it refers to the municipal wastewater that contains a broad spectrum of contaminants resulting from the mixing of wastewater from different sources.

Chand (2002) reported that nearly all waters contain dissolved salts and trace elements, many of which result from the natural weathering of the earth's surface. In addition, drainage waters from irrigated lands and effluent from city sewage and industrial waste water can impact water quality. In most irrigation situations, the primary water quality concern is salinity levels, since

salts can affect both the soil structure and crop yield. However, a number of trace elements are found in water which can limit its use for irrigation.

2.2 Wastewater Origins

The wastewater body used for irrigation in the Soje settlement originates from several sources as studied, such as: Human waste, usually from lavatories (feces, used toilet papers, urine and other body fluids) also known as black water, Cesspit leakages, septic tank discharge, washing water (dishes, floors, cloths) also known as gray water, Ground water infiltration into sewerage, runoffs and industrial wastes.

Chand (2002) simplified wastewater origins as residential and non-residential. They emphasized on the word 'sewage' used to describe all types of wastewater generated from every room in a house, when compared to the variety of wastewater flow generated by different non-residential sources. Household wastewater shares many similar characteristics overall since they are two types of domestic sewage wastewater from toilets and gray water, which is water from all sources except toilets. Black water and gray water have different characteristics, but both contain pollutants and disease causing agents. Non-residential waste water on the other hand in small communities as that of Soje as having such diverse sources as businesses offices, departmental stores, hospitals, farms, manufacturers and other commercial and institutional entities, including storm water which carries trash and other objects

2.3 Types of Wastewater

Wastewater can be grouped into residential and non-residential wastewater.

Residential wastewater: although the word "sewage" usually used to describe all types of wastewater generated from every room in a house when compared to the variety of wastewater flow generated by different non-residential sources, household wastewater shares many similar

characteristics overall. There are two types of residential wastewater; Black water which is wastewater from toilets and Gray wastewater from all sources from homes apart from toilets. Black water and gray water have different characteristics with both containing pollutants and disease causing agents.

Non-residential wastewater: non residential wastewater in small communities as Soje has diverse sources such as offices, departmental stores, schools, hospitals, farms and other commercial institutional entities.

2.4 Wastewater Use

The term wastewater reuse is often used synonymously with the terms wastewater recycling and wastewater reclamation. Because the general public often does not understand the quality difference between treated and untreated wastewater, many communities have shortened the term to water reuse, which creates a more positive image. The U.S. Environmental Protection Agency (EPA) defines wastewater reuse as, "using wastewater or reclaimed water from one application for another application. The deliberate use of reclaimed water or wastewater must be in compliance with applicable rules for a beneficial purpose (landscape irrigation, agricultural irrigation, aesthetic uses, ground water recharge, industrial uses, and fire protection). A common type of recycled water is water that has been reclaimed from municipal wastewater (sewage)" EPA (2003). This practice is likely to continue in the foreseeable future due to the high investment cost associated with the installation of treatment facilities.

Ensink et al. (2002) in a case study concluded; in order to systematically document the advantages and disadvantages of using untreated urban wastewater, a case study was undertaken in Haroonabad, which is a small town in the southern Punjab in Pakistan. Information on costs and benefits associated with wastewater use was obtained by monitoring a group of 20

wastewater farmers and a group of 20 non-wastewater farmers over a one-year period. Water and nutrient applications and the quality of groundwater and soil were investigated in nine fields, of which some were irrigated with wastewater and others with regular canal water. To assess the human health impacts, a comparison was done between a settlement where wastewater irrigation was practiced and one where regular canal water was used. The greatest benefit for farmers using wastewater was the reliable water supply, which allowed them to grow high-value vegetable crops. However, water and nutrient applications to wastewater-irrigated fields were excessive in relation to the recommended values. From this we can deduce that, with the improved distribution of wastewater, more farmers could benefit from the water and the nutrients it contains. Although there were signs of accumulation of heavy metals in wastewater-irrigated soils, the values did not exceed internationally recommended standards. However, there were negative health impacts, especially in the form of an increased prevalence of hookworm infections among wastewater farmer

2.4.1 Reasons for Wastewater Use

The most common reason for establishing a wastewater reuse program is to identify new water source for increased water demand and to find economical ways to meet increasingly more stringent discharge standards.

2.4.2 Guidelines and Regulations

Alberta (MPDB 2000) in a practical guide to **Municipal Wastewater Irrigation - Discussions and Guidelines** emphasized that municipal wastewater must be collected and treated to meet quality standards before its authorized release into the environment, that is, whether it is discharged directly to a water body or disposed on land. Disposal of the treated effluent on land through wastewater irrigation, while affording a unique opportunity to avoid discharges of nutrient-rich

wastewater irrigation, while affording a unique opportunity to avoid discharges of nutrient-rich wastewater to surface waters, can also impose certain risks to public health and the environment. Not all treated municipal wastewater meets a quality that would enable unrestricted use for irrigation. Elevated nutrient, salt, sodium, or other biological or chemical constituents are often present at concentrations that could influence the rate and/or frequency of application or restrict the type of crop to be grown. In certain cases, treated municipal wastewater has been found to contain salt or sodium levels that would completely exclude consideration of its use for irrigation due to the harmful effects it would cause to the land and the crops to be grown. Wastewater loading is ultimately based on the consumptive water needs of the specific crop grown. This wastewater loading value, however, must also consider issues of varying rainfall, seasonal moisture deficiencies, application efficiencies, and other considerations related to leaching and crop nutrient utilization factors. The primary objectives should, therefore, be the long-term enhancement of crop production, while minimizing associated health and environmental risks.

Thus, when evaluating wastewater for irrigation use, it is important to:

- Identify physical, biological, and chemical constituents that may be a potential environmental or health-based concern
- Ensure wastewaters, considered for irrigation application, have been appropriately tested.

Other climatic, social, management, and land base considerations that should also receive close evaluation and assessment include:

- development of appropriate protocols for determining land areas that are suitable to receive municipal wastewater for irrigation
- determination of climatic, crop, land use, or easement restrictions that may apply

- **determination of the appropriate annual wastewater loading rates, the frequency, duration and method of application, as well as the period over which such applications can occur and**
- **Inclusion of a process that will ensure there is appropriate technical review of valid neighborhood stakeholder concerns.**

These above noted factors must be investigated and reported before any municipal wastewater irrigation is authorized. Guidelines are therefore necessary to facilitate such an authorization process.

2.5 Wastewater Irrigation

Irrigation can be defined as the artificial watering of land to sustain plant growth. The use of wastewater for irrigation is increasingly being considered as a technical solution to minimize soil degradation and to restore nutrient content of the soils. The aim of the study was to increase fertility and minimize degradation of the soils irrigated with wastewater. A field experiment was conducted to investigate the effects of the controlled plot and that irrigated with wastewater. Wastewater irrigation significantly affected soil chemical properties especially at 0 – 30cm soil depth and plant nutrient contents after one year. Application of wastewater increased soil salinity, organic matter, exchangeable Na, K, Ca, Mg, plant – available P, and micro – elements and decreases soil pH. Wastewater increased also yield and N, P, K, Fe, Mn, Zn, Cu, B and Mo content of cabbage plants. Undesirable side effects were not observed in plant heavy – metals content, due to salinity and toxic concentration of metals from the application of wastewater to the soil. Sites irrigated with wastewater for 10, 5, and 2 years and site not irrigated were sampled for soil and plant chemical analysis to evaluate its long term effect. Long term wastewater irrigation increased salts, organic matter and plant nutrients in the soil. Soil pH was not

consistently affected. Soil Cu was not affected by wastewater application while Zn, Fe and Mn were not consistently affected. Wastewater irrigation had no significant effect on soil heavy metals (Pb and Cd) regardless of duration of wastewater irrigation. The barley biomass increased with added wastewater and nutrients provided with the wastewater.

However, longer period of wastewater application (10 years) resulted in lower biomass production but remained higher than that of the control plants. Plant essential nutrients (Total N, NO₃, P, and K) were higher in plants grown in soils irrigated with wastewater. Plant Cu, Zn, Fe, Mn increased with 2 years of wastewater irrigation, and then reduced with longer period. Plant Pb and Cd increased with wastewater irrigation and their levels were higher the longer the period of wastewater irrigation. Based on these results, it can be concluded that proper management of wastewater irrigation and periodic monitoring of soil and plant quality parameters are required to ensure successful, safe, long-term wastewater irrigation.

From studies on wastewater irrigation, reports presence of high levels of total dissolved solids which includes sodium-100mg/l, phosphorous-50mg/l, and nitrogen-200mg/l is sufficient to cause significant long term damage some sites and soil and ground water pollution if excessive application rates are used.

2.5.1 Irrigation Schedule

This involves the plan of irrigation to be done, showing the order in which irrigation is to be carried out and the amounts of water allocated to them. A crops water requirement determines the schedule of irrigation. Since nutrients are always present in wastewater, then any amount of irrigation water above the crop water requirement may create a problem. The problem could be an environmental or an agronomic one or both. It should be noted that with wastewater irrigation

an environmental or an agronomic one or both. It should be noted that with wastewater irrigation it is even more important to follow proper scheduling of irrigation than that with water of good quality.

2.5.1.1 Amount of water:

For all practical purposes, water needed by crop plants is equal to their evapotranspiration requirement. This amount might be exceeded due to leaching fraction if required. Crops must be irrigated according to their crop water requirements. It could be stressed that since the amount of irrigation water is different from place to place due to climatic conditions, the nutrients in wastewater could be in excess or inadequate for the same crop under the same soil fertility conditions in different places. Similarly, wastewater of the same quality could have adverse environmental impact in one place but be very safe in another place. Because of this, it is difficult to give absolute numbers for fertilization with wastewater, while with fresh water it is different case.

2.5.1.1 Frequency Of Irrigation.

This refers to the intervals in between successive irrigations. Irrigators should be also aware that with crops at full growth stage, the amount of water per irrigation must be always the same to reach a certain soil depth where the active roots are concentrated. However, since the absolute amount of irrigation water varies with climatic conditions the frequency of irrigation should be changed. The amount of water per irrigation must remain the same.

2.6 Wastewater Quality Characterization

As water quality standards for municipal wastewater discharging to surface water bodies become more stringent, the associated treatment costs correspondingly escalate. Irrigation is therefore becoming a more desired alternative for wastewater disposal for many communities. However,

since different water quality variables need to be considered when evaluating wastewater effluents as a potential irrigation water source than those considered for its direct discharge into a receiving stream, a specific set of wastewater quality reporting requirements must be outlined and defined. In this overall treatise it is therefore important to first evaluate restrictions that may apply to the use of standard sources of irrigation water and then consider what supplemental evaluations would apply to wastewater irrigation use.

2.7 Natural Irrigation Water Quality Characterization

Alberta, MPDB (2000). Simplified the use of waters for irrigation application, it normally involves evaluation of two major water quality parameters:

Electrical conductivity (EC): is a reliable indicator of the total dissolved solids (salts) content of the water. The addition of irrigation water to soils adds to the concentration of salt in the soil. Concentration of these salts will result in an increase in osmotic potential in the soil solution interfering with extraction of water by the plants. Toxic effects may also result with an increase in salinity. EC is measured in dS m⁻¹. For specific values on acceptable EC levels in waters used for irrigation are in Table 2.1

Sodium Adsorption Ratio (SAR): is an indicator of the sodium hazard of water. Excess sodium in relation to calcium and magnesium concentrations in soils destroys soil structure that reduces permeability of the soil to water and air. Sodium may be toxic to some crops.

$$\text{SAR} = \frac{\text{Na}^+}{\sqrt{\text{Ca}^{2+} + \text{Mg}^{2+} / 2}} \quad \text{and} \quad \text{SAR} = \frac{\text{Na}^+}{\sqrt{\text{Ca}^{2+} + \text{Mg}^{2+}}}$$

For concentration s in mmoles/L

Cations are expressed in equivalent of charge per litre or millimoles of charge per litre.

Table 2:1 Irrigation Water Quality Standards

Parameter	Safe	Possibly Safe	Hazardous
EC (dS m ⁻¹)	< 1.0	1.0 - 2.5	> 2.5
SAR	< 4	4 - 9	> 9

Adapted from A practical guide to Municipal Wastewater Irrigation, Alberta Environment 2002

EC = Electrical conductivity

SAR = Sodium Adsorption Ratio

2.8 Other Water Quality Aspects

Other water quality aspects to be included in the comprehensive wastewater characterization assessment prior to the development of a wastewater irrigation system are included in the sections to follow.

2.8.1 General Chemical Parameters

The general parameters are those that are analyzed to assess the effectiveness of the wastewater treatment process and to evaluate variability in the quality of the wastewater prior to its release to the environment. They also represent water quality values that, if exceeded, can often restrict treated wastewater sources from being considered for irrigation purposes.

2.8.1.1 Biochemical Oxygen Demand (BOD)

The BOD₅ is the amount of oxygen used over a five-day period by microorganisms as they decompose the organic matter in sewage at a temperature of 20° C (68° F) typically ranges from 10 to 20 mg/L for most municipal wastewaters. Values below 100 mg/L pose no restriction to irrigation use.

2.8.1.2 Total Suspended Solids (TSS)

These could be organic or inorganic materials in suspension but not in solution. Typically it ranges from 10 to 20 mg/L for most municipal wastewaters. Values below 100 mg/L pose no restriction to irrigation use.

2.8.1.3 Chemical Oxygen Demand (COD)

Is the amount of oxygen required to oxidize the organic matter by use of dichromate in an acid solution and to convert it to carbon dioxide and water. The value of COD is always higher than that of BOD₅ because many organic substances can be oxidized chemically but cannot oxidize biologically. Typically it ranges from 25 to 50 mg/L for most municipal wastewaters. Values below 150 mg/L pose no restriction to irrigation use.

2.8.1.4 pH

The measure of the acidity or alkalinity of a solution, typically, ranges from 6.5 to 8.5 for most municipal wastewaters. These values are comparable to most natural surface waters and are considered to pose no restriction to irrigation use. A continued long-term use of waters outside this pH range could eventually alter naturally occurring pH levels in surface soils to which they are applied and therefore could possibly lead to micro nutrient imbalances and potential future crop production and fertility problems

2.8.1.5 Electrical Conductivity (EC)

These values range widely within municipal wastewater and like some natural water sources exceed levels that would be recommended for irrigation. Those municipal wastewaters with EC values less than 1.0 dS/m are considered of good quality and should pose no problems for irrigation use, unless the sodium adsorption ratio (SAR) of the wastewater is greater than 4.

2.8.1.6 Total Organic Carbon

This is the measure by the catalytic conversion of organic carbon in wastewater to carbon dioxide

2.8.2 General Physical Parameters

The general physical parameters are those that are analyzed to evaluate variability in the quality of the wastewater prior to its release to the environment.

2.8.2.1 Turbidity

Turbidity in water mainly arises from colloidal matter, fine suspended particles and soil erosion. Generally the greater the turbidity, stronger is the effluent concentration and worse the effect.

2.8.2.2 Odour

Odour in water is caused both by chemical agents like (H_2S , Cl_2 , NH_3) and biological agents (such as algae, fungi, microorganisms). Lower pH, higher will be the amount of H_2S produced and greater the odor nuisance

2.8.2.3 Thermal Pollutants

It is mainly arises by the discharge of unutilized heat produced in various thermal plants. The heated waters have reduced the amount of dissolved oxygen content due to which organic substance degrades faster

2.8.2.4 Color

Color change is not harmful unless it is associated with a toxic chemical, but it may affect the quality of the sunlight that penetrates to a given depth inhibiting plants metabolism

2.8.3 Nutrients

One of the main believed advantages of using wastewater irrigation is that it can often enhance the fertility of the lands to which it is applied. This can add considerably to potential crop yield

and therefore the associated agricultural resource value. Nutrient loading rates, while significant, are seldom at levels that would present a concern when using municipal wastewater for irrigation. Most nutrient levels are well within the range that can be assimilated by plants if the wastewater is applied at a rate and frequency that conforms to active crop growth. Potential contamination of groundwater would only be a concern under extremely shallow groundwater levels, unsuitable soil conditions, or gross mismanagement of the applied wastewater. Since all these factors are carefully considered as part of the guidelines, potential contamination of the groundwater should not present a concern. The following nutrients should be analyzed and reported as part of the comprehensive wastewater quality characterization process:

- Nitrogen can be evaluated in a number of different forms. Evaluation of nitrogen by analyzing for $\text{NO}_3 - \text{N}$, $\text{NH}_4 - \text{N}$ and $\text{NO}_2 - \text{N}$ should be conducted. The typical range for total nitrogen of most municipal wastewater is 10 to 20 mg/L. This means that if 30 cm of wastewater were applied, an N loading of 30 to 60 kg/ha/yr. would be applied to the land base. Providing wastewater is not applied in quantities that exceed the field moisture capacity during periods of wastewater applications, and is applied during the active crop growing season, such loadings can be easily assimilated by the growing crop without harmful health or environmental concerns developing.
- Phosphorus is to be evaluated as total dissolved phosphorus. The typical range of total dissolved phosphorus in municipal wastewater is between 2 and 6 mg/L. If 30 cm of wastewater were applied, this would translate to a P loading of 6 to 18 kg/ha/yr. Since these levels are considered to be reasonably low and phosphorus is effectively immobilized in most soils at shallow depths, the potential for adverse impacts on groundwater quality is remote. Care must be exercised, however, to ensure wastewater

applications are applied at rates that do not exceed the infiltration capacity of the soils as high phosphorus levels in surface runoff and erosion sediments can create significant environmental concern if washed into neighboring lakes, streams or other surface water bodies.

- Potassium is another major nutrient present in wastewater of value for crop production that should be evaluated. The typical range for potassium in most municipal wastewaters is 5 to 40 mg/L. If 30 cm of wastewater were applied this would translate to a K loading of 15 to 120 kg/ha/yr. Such levels are readily assimilated by the actively growing crops and are thus not considered to be of an environmental or health risk.

2.9 Wastewater Treatment

FAO Cairo (2003) pointed out that the main objective of treatment is to produce treated effluents of suitable and acceptable level of risk for human health and the environment. In this respect, the most appropriate wastewater treatment is that which provides and secures effluents with the chemical and microbiological quality required for a certain specific use at low cost and minimal operational and maintenance requirements. Wastewater treatment plants, regardless of the type treatment, reduce organic and suspended solids, remove wastewater chemical constituents that may be toxic to crops as well as biological constituents (i.e. pathogens) which are of main concern to public health in general. The different degrees of conventional treatment are:

- Preliminary: Removal of coarse solids and other large fragments from the raw wastewater.
- Primary: Removal of submerged organic and inorganic solids as well as floating materials.
- Secondary: Removal of the residual organic and suspended solids from primary treated wastewater.
- Tertiary and/or advanced: Removals of specific wastewater constituents like nutrients and heavy metals, which are not removed by secondary treatment. Disinfection, usually with chlorine, is used to reduce microbiological constituents.

2.9.1 Trace Elements and Heavy Metals

They are the main problem with both untreated and treated wastewater reuse in countries with light or heavy industry. The metals that may be present in sewage {Cadmium (Cd), copper (Cu), molybdenum (Mo), nickel (Ni) and zinc (Zn)} can pose significant health hazards to humans and animals and also may affect the irrigated crops. These metals in most cases are accumulated in the crop, and could adversely affect humans or domestic animals, feeding on these crops. Because of this, many developed countries have set maximum permissible cumulative loading of metals to agricultural lands.

2.9.2 Salinity

In most areas of the country the water used for municipal supply is the best water quality available or afforded and the salinity level not taken into consideration. However, under water scarcity conditions salinity may be a problem. The quantity and kind of salts present are important to assess the suitability of treated or untreated wastewater for irrigation. Potential problems are related to the total salt content, to the type of salt or to excessive concentration of one or more elements, Ayers and Westcot. (1997).

To overcome the problem of salinity, at farmer's level, more emphasis must be given to the following approaches:

- Select crops tolerant to the wastewater salinity and still be on the profitable site, the appropriate cropping pattern depending on the salinity of wastewater and salt tolerance of crops. With salinity less than 3 dS/m, and good management, most fruits and vegetables can be produced. As salinity rises the selection of crops becomes difficult and, except for certain vegetables, the choice is mostly restricted to fodder crops.

- Select salt tolerant crops with the ability to absorb high amounts of salts without particular toxicity effects (salt harvesting crops). In case of irrigation with untreated wastewater of high salinity, particularly in areas with limited rain and natural leaching, for long term sustainable reuse salt harvesting crops may help reduce salinity build-up in the soil. Some recommended crops are sorghum and barley.
- Select irrigation system with uniform application, high efficiency and providing the possibility for more frequent irrigation.
- Leaching is a common approach by farmers but not the best probable solution in the case of water scarcity, inadequate drainage or shallow water table. In a long term the total quantity of salt applied in the soil with the wastewater (salt in) and the rate at which salt is removed by leaching and crop uptake (salt out) should be approximately the same
- Drainage is one of the measures necessary to prevent irrigation-induced water logging and salinization in arid and semi-arid regions is the installation of drainage facilities. Drainage, in combination with adequate irrigation scheduling, allows for the leaching of excess salts from the plant root zone.

2.9.3 Alkalinity

Dispersion of soil colloidal phase, stability of aggregates, soil structure and permeability for water, are very sensitive to the type of exchangeable ions present in irrigation water. The increase in soil alkalinity, which may occur with treated wastewater due to high sodium concentration even though leaching is allowed, reduces soil permeability for water, particularly at the soil surface, since soil clays tend to disperse and swell from the increased level of exchangeable sodium ion (Na^+). However, at certain sodium adsorption ratio (SAR) the infiltration rate increases or decreases with the salinity level.

2.9.4 Specific Ion Toxicity

The most toxic ions generally occurring in treated wastewater are sodium (Na), chloride (Ca), and boron (B) which is causing most prevalent toxicity cases. Boron is one of the essential elements to plants; hence, Boron concentrations exceeding 0.5 mg/l could be toxic to sensitive crops.

2.10 Crop Nutrient and Fertilizer Requirement

The selection of a sound fertilization programme is influenced by the knowledge of the nutrient requirement of the crop, the nutrient supplying power of both soil and water, the efficiency of nutrient uptake under different irrigation, fertilization methods, and the expected yield. When the soil and irrigation water do not furnish adequate quantities of elements necessary for normal growth of plants, it is essential that the required amounts be applied.

2.10.1 Removal of nutrients by crops from soil.

The quantities of nutrients removed by a crop from the soil are good information, which can be used to optimize soil fertility level needed for various crops. Part of nutrients removed by crop is used for vegetative growth (canopy) and the other for fruit production. The amount of nutrients removed by crop must be present in soil irrespective of the fact that part of them may remain or return to the soil by the end of the growing season as canopy and roots. An important key is to have enough nutrients in the right proportions in the soil to supply crop needs during the entire growing season. In crops from which just the fruits are removed much lower quantities of nutrients are lost than if the entire above ground portion were harvested. Uptake will vary considerably, depending on a number of factors, including yield level, nutrient supply of the soil, fertilization and irrigation method. In addition, they are an indication of the rate at which the reserve or "storehouse" nutrient in the soil is depleted by certain crops.

Certain amounts of nutrients are needed to be available in soil. However, not all of the nutrients should necessarily come from fertilizer. Part of them is supplied from the soil and part from wastewater. In this respect, estimations of amount of nutrients which may be available to the crop from soil (nutrient supplying power of soil) and wastewater (nutrient supplying capacity of wastewater), are important. These amounts are subtracted from the overall amount, which should be supplied by fertilizer applications.

2.10.2 Nutrients Coming From Soil

A soil test can help for estimating the nutrient-supplying power of a soil for P, K, Ca, Mg and other nutrients. Soluble N leaches readily during the rainy season or with the irrigation water, so there may be little carry-over. Because of this, soil testing is not practiced as a tool for soil N fertility. Nevertheless, nitrification may build up the supply of nitrate in soil as the season advances, in case that organic matter is adequate. The soil test measures a part of the total nutrient present in the soil assumed or experimentally found to be well correlated with the available nutrient. The values of soil analysis are of little use in themselves. To employ such a measurement in predicting nutrient needs of crops should use those chemical methods, which have been tested and calibrated against nutrient uptake rate experiments. The results of the soil test could be reliable only if they are based on representative samples. In order to estimate the nutrient supplying capacity of a soil the following parameters are needed:

- depth of the crop rooting system;
- percent (%) of soil occupied by the root system under different irrigation systems; and
- Soil Bulk Density (Bd).

These parameters are needed in order to:

- Calculate the weight of soil of a certain area to a depth where the active root zone of the crop is developed; and
- Estimate the reserves or storehouse available nutrients for the crop in a soil.

2.11 Irrigation Methods

2.11.1 Surface (Traditional) Methods

These methods account for about 95% of the world's irrigated area and dominate the world's irrigation systems. They are of low cost, simple to understand and implement. They are suitable for many developing countries particularly if water is not the limiting factor for agricultural production. They are:

- Flood irrigation (by border or basin): wetting almost all the land surface
- Hose-basin irrigation: The water is delivered by hose
- Furrow irrigation: wetting only part of the ground surface.

2.11.2 Pressurized Irrigation Methods

Sprinklers: (Sprinklers of high capacity, ordinary mini-sprinklers, and sprayers). Crops and soil are wetted the same way as rain.

Drip: (point or localized irrigation system): The main characteristics of the system are:

- High application efficiency.
- It is probably, if it is used properly, the best method for irrigation in places where scarcity of water is a problem.
- Appropriate method to cope with problems associated with saline irrigation, water and soil alkalinity.
- This method is safe and in general might be the most promising for irrigation with wastewater, particularly if purification is such to prevent extensive clogging.

- Contact of wastewater with both the farmers and the irrigated crops is minimized.
- No aerosols are formed and, therefore, no pollution of the atmosphere and of the area nearby to the irrigated fields occurs

2.12 Selection of Irrigation System

Selection of the appropriate irrigation system depends on the quality of wastewater, crop, tradition, background, skill, ability of the farmers to manage the different methods, and the potential risk to the health of farmers, public and the environment. Suitability of common irrigation methods, namely, border, furrow, sprinkler and drip in relation to brackish water is also valid for treated wastewater, particularly concerning its salinity content.

Clogging problems with the sprinkler, mini sprinkler, drip, and subsurface irrigation systems might be a serious problem. Growths (slimes, bacteria, etc.) in the sprinkler head, emitter orifice or supply line causes plugging. Salts and suspended solids may also create clogging. The most serious clogging occurs with drip irrigation, which is considered ideal as far as health protection and plant contamination but might be difficult to be used for irrigation with wastewater high in suspended solids (SS).

2.13 Irrigation of Agricultural Crops

There are guidelines for the safe use of wastewater in agriculture taking into account the type of crops and source of wastewater. The oldest and largest reuse of wastewater is for irrigation of agricultural crops. Potential constraints that bring about restriction in wastewater irrigation application are:

- surface and groundwater pollution, if poorly planned and managed,
- marketability of crops and public acceptance,
- effect of water quality on soil, and crops,

- Public health concerns related to pathogens.

2.14 Nutrients and Spinach

2.14.1 Iron

Spinach is considered a rich source of iron and calcium. The bioavailability of iron is dependent on its absorption. This is influenced by a number of factors. Iron enters the body in two forms: non-heme iron and heme iron. All of the iron in grains and vegetables, and about three fifths of the iron in animal food sources (meats), is non-heme iron. The much smaller remaining portion from meats is heme iron. Thus, the iron in spinach is poorly absorbed by the body unless eaten with vitamin C. The type of iron found in spinach is non-blood (non-heme), a plant iron, which the body does not absorb as efficiently as blood (heme) iron, found in meat. However, spinach contains iron absorption inhibiting substances, including high levels of oxalate which can bind to the iron to form ferrous oxalate, which renders much of the iron in spinach unusable by the body.

2.14.2 Calcium

Spinach also has high calcium content. However, the oxalate content in spinach also binds with calcium decreasing its absorption. Calcium and zinc also limit iron absorption. The calcium in spinach is the least bio-available of calcium sources. By way of comparison, the body can absorb about half of the calcium present in broccoli, yet only around 5% of the calcium in spinach. Oxalate is one of a number of factors that can contribute to gout and kidney stones.

2.14.3 Other Nutrients

Spinach has a high nutritional value and is extremely rich in antioxidants, especially when fresh, steamed, or quickly boiled. It is a rich source of vitamin A (and lutein), vitamin C, vitamin E, vitamin K, magnesium, manganese, foliate, iron, vitamin B2, calcium, potassium, vitamin B6, folic acid, copper, protein, phosphorus, zinc, niacin, selenium and omega-3 fatty acids. Recently,

opioid peptides called rubiscolins have also been found in spinach. It is a source of folic acid (Vitamin B₉), and this vitamin was first purified from spinach. To benefit from the folate in spinach, it is better to steam it than to boil it. Boiling spinach for four minutes can halve the level of folate.

2.15 Types of Spinach

A distinction can be made between older varieties of spinach and more modern varieties. Older varieties tend to bolt too early in warm conditions. Newer varieties tend to grow more rapidly but have less of an inclination to run up to seed. The older varieties have narrower leaves and tend to have a stronger and bitterer taste. Most new varieties have broader leaves and round seeds.

There are three basic types of spinach:

2.15.1 Savoy Spinach

It has dark green, crinkly and curly leaves. It is the type sold in fresh bunches in most supermarkets. One heirloom variety of Savoy is Bloomsdale, which is somewhat resistant to bolting.

2.15.2 Flat/Smooth Leaf Spinach

It has broad smooth leaves that are easier to clean than Savoy. This type is often grown for canned and frozen spinach, as well as soups, baby foods, and processed foods.

2.15.3 Semi-Savoy Spinach

It is a hybrid variety with slightly crinkled leaves. It has the same texture as Savoy, but it is not as difficult to clean. It is grown for both fresh market and processing. Five star is a widely grown variety and has good resistance to running up to seed.

2.16 Spinach Outbreaks

- 2007 United States salmonella outbreak: On August 30, 2007, 8,000 cartons of spinach (from Metz Fresh, a King City-based grower and shipper, Salinas Valley, California) were recalled after salmonella was discovered upon routine test. Consumer advocates and some lawmakers complained it exposed big gaps in food safety, even if 90% of suspect vegetable didn't reach the shelves
- In September 2006, there was an outbreak of disease caused by the E. coli strain O157:H7 in 21 U.S. states. Over a hundred cases were reported, including five deaths. The E. coli was linked to bags of fresh spinach, after which the FDA issued a warning not to eat uncooked fresh spinach or products containing it. The U.S. Food and Drug Administration issued a press release updating the available information. According to the FDA release as on 2006-10-4, 192 cases of E. coli O157:H7 infection has been reported to the Centers for Disease Control and Prevention (CDC) including 30 cases of Hemolytic Uremic Syndrome; there was one death and 98 hospitalizations. The infection affected 26 states. By early 2007, there were 206 illnesses and three deaths attributed to E. coli-tainted spinach.

CHAPTER THREE

3. METHODOLOGY

3.1 Study Area

The project study area is located in Soje few kilometers from the Minna railway station and 300m from the west of Morris Fertilizer Company. The municipal/domestic wastewater derives its source from Minna Township. The wastewater flows through an unlined channel, land owners (farmers) linear to the flow takes advantage of its continuous flow for irrigation

Figure 3.1 shows the position of the project study area on a map (abridged Niger State map). Niger state is situated in the middle belt of the federal republic of Nigeria. It lies in the savanna zone of the tropics between latitude ($9^{\circ} 34'N$) and longitude ($6^{\circ} 33'E$). Its climate is influenced mainly by the rain-bearing South West monsoon winds from the oceans and the dry dusty or harmattan North East winds (air masses) from the Sahara desert. There are mainly the rainy and the dry seasons. The rainy season begins in April and ends in October and the dry season starts in November and ends in March. Vegetables are the principal crops grown in the area and locally consumed by residents or marketed. The wastewater and its sludge are used directly to irrigate the crops. The spinach farms are irrigated with pipes connected with pipes connected to pumps drawing the water directly from the wastewater channel. The use of this untreated wastewater with its high BOD forms a thin layer of black residue on the soil surface.

Practically, from the above weather/seasons descriptions, this study was undertaken during the dry season between the month of March and May.

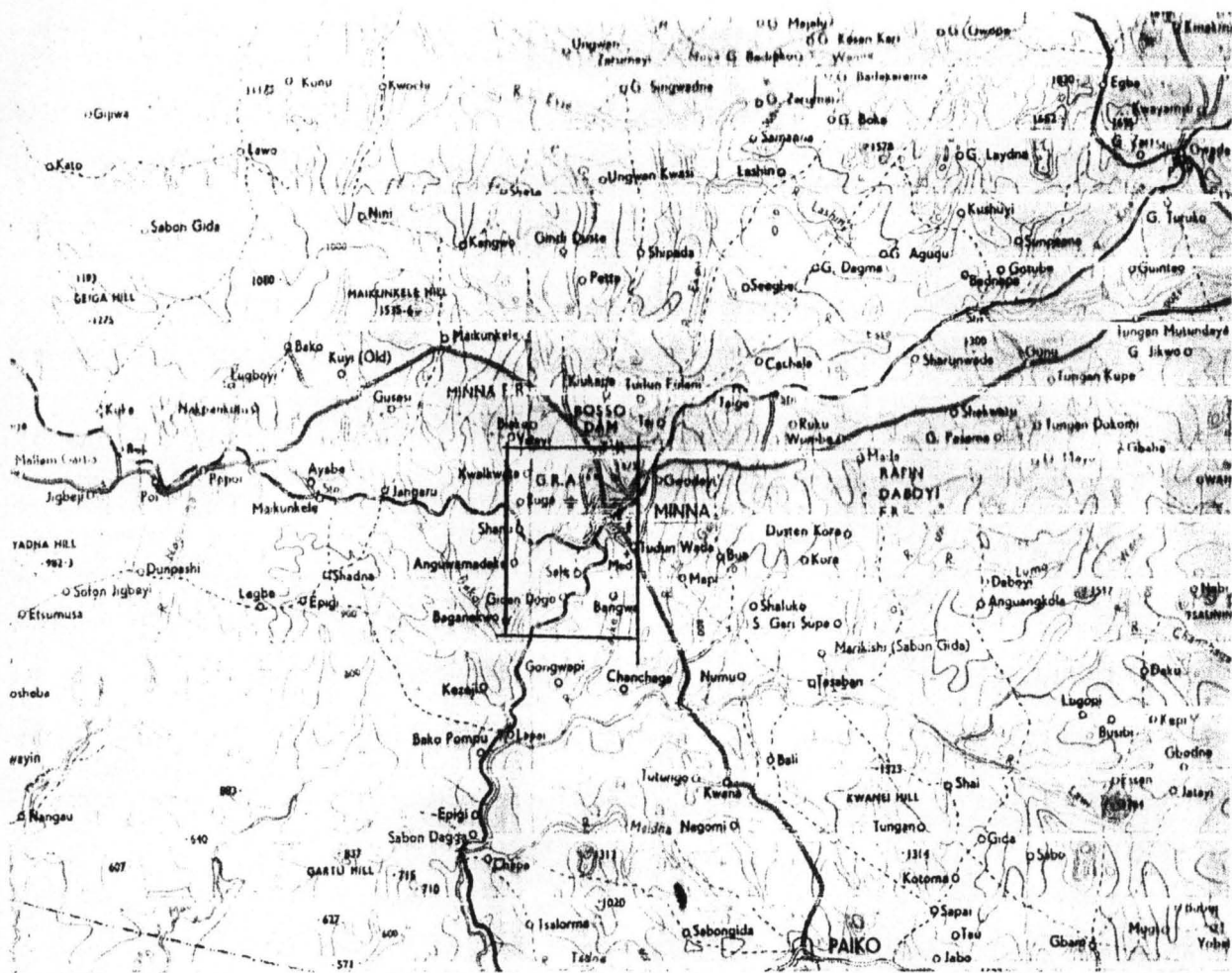


Fig 3.1: Map of Niger State Showing the Study Site

3.2 Water and Crop Sampling

The wastewater effluent sample (upstream, middle stream and downstream) was collected during April 2009. The effluent sample was collected in well cleaned polythene bottle. Before collection each bottle was washed with fresh water and then tightly closed and the physiochemical properties tested.

Two different sites were used for spinach cultivation. The first site (A), was irrigated with wastewater, while the second site (B), was irrigated with fresh water (borehole). Crop (spinach) sampling was performed June 2009 during the early maturity period fifty-five days after

planting. 10 crop samples were collected in the field by means of random sampling method on each site and tested for its nutrient and metal content.

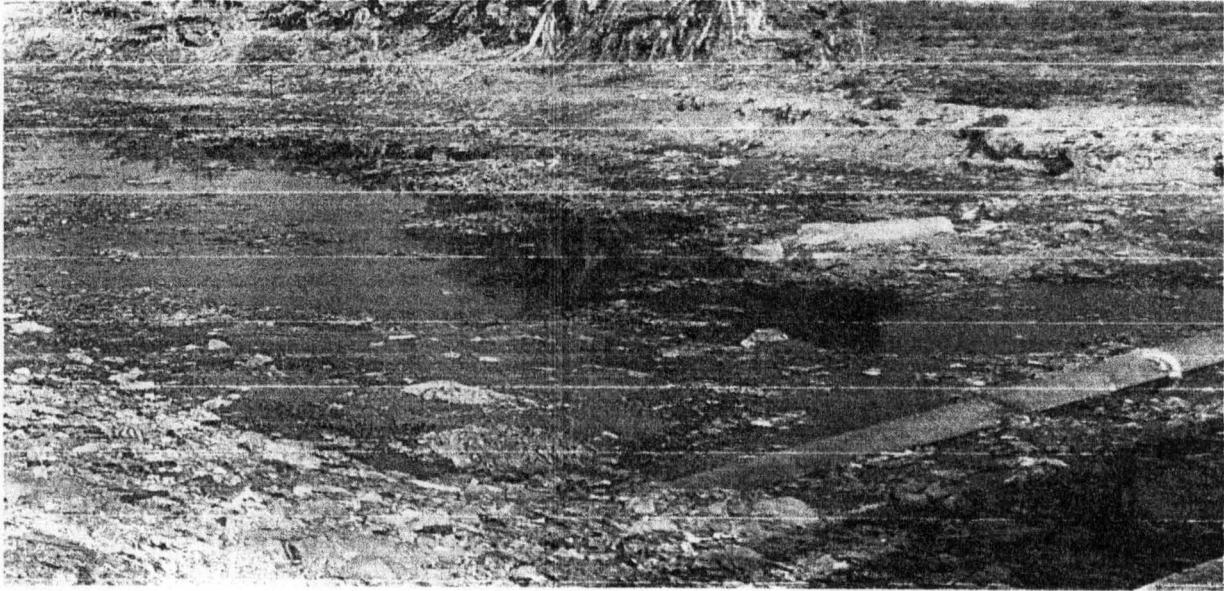


Plate 3.1: Wastewater Body at Middle Stream

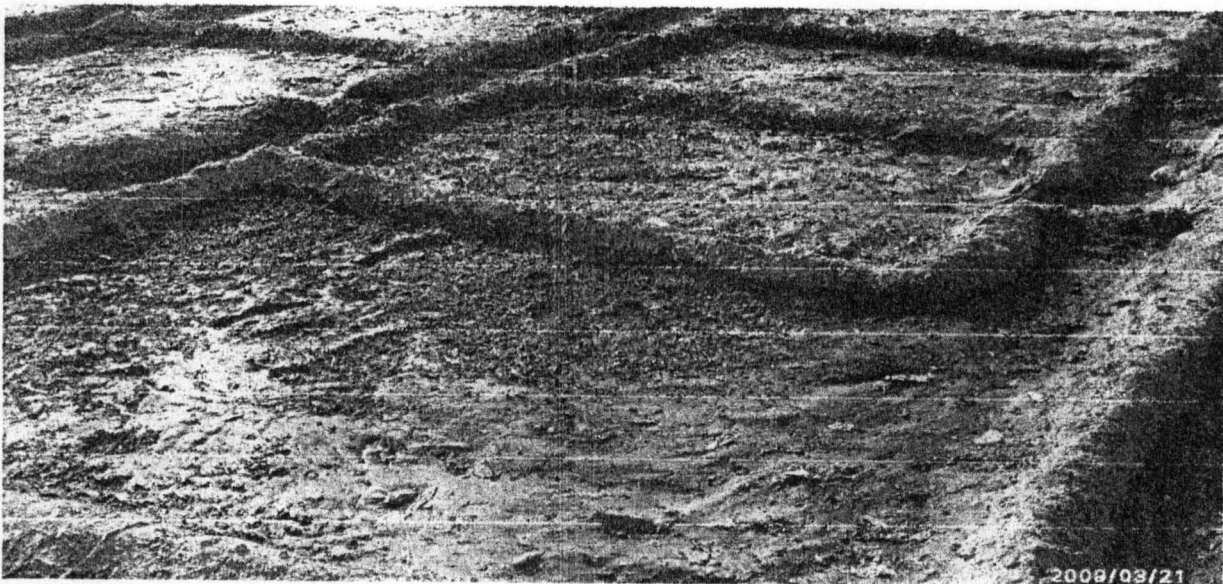


Plate 3.2: Site Location before Germination

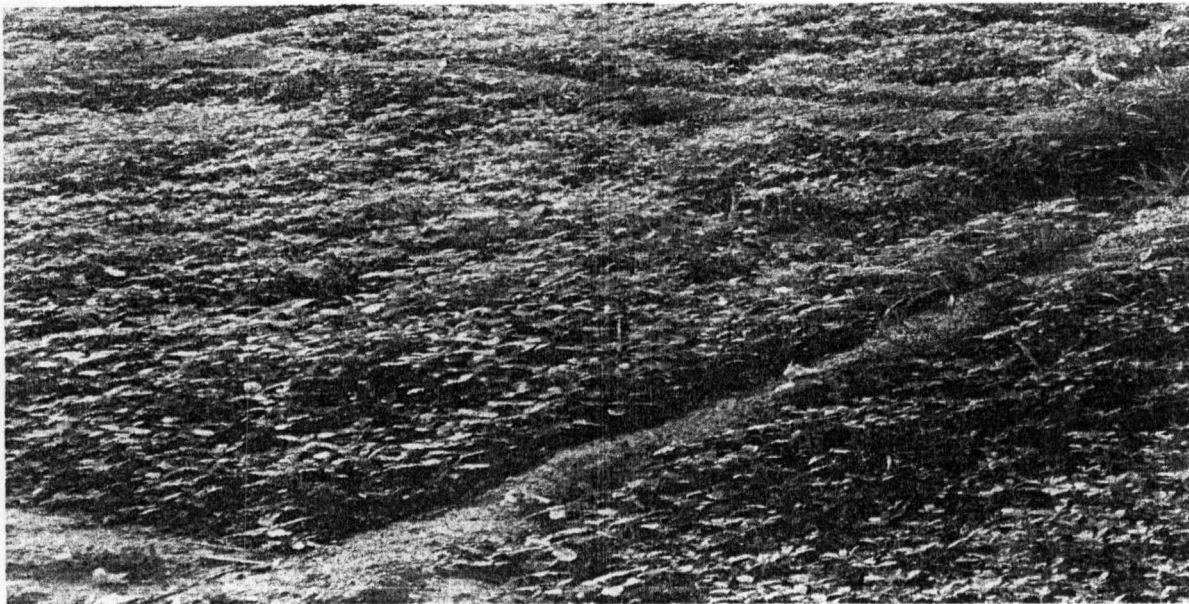


Plate 3.3: Clean water Irrigated Spinach Thirty Days after Planting



Plate 3.4: Wastewater Irrigated Spinach Thirty Days after Planting

3.3 Effluent Analysis

Effluent sample was collected in well cleaned polythene bottle which was washed with fresh water and carried for testing in less than 3 hours.

3.3.1 Calcium Hardness

Reagents: Hydroxide solution, 56.1g of KOH or 40g of NaOH in distilled water and dilute to 1 liter, Calver II Calcium indicator (manufactured by the Hach Company). Muroxide indicator (ammonium purpurate) – changes from pink to purple, prepare by dissolving 150mg dye in 100g absolute ethylene glycol. Water solutions of the dye are not stable for longer than 1 day. A ground mixture of dye powder and sodium chloride (NaCl provides a stable form of the indicator). Prepare by mixing 200mg muroxide with 100g solid NaCl and grinding the mixture to 40 to 50 mesh.

Procedure:

1. Measure a 50ml sample into a 125ml Erlenmeyer flask.
2. Add 2ml of the hydroxide solution (to produce a pH of 12-13 in the 50ml sample). Add 0.1 to 0.2g of calver II calcium indicator or muroxide indicator
3. Titrate slowly with disodium salt solution (0.01M) until the colour changes to blue for calver II and pink for muroxide.
4. Hardness is then calculated as shown in appendix A

3.3.2. Iron (Total) Phenanthroline Method

Reagents:

1. Concentrated HCl, containing less than 0.00005% iron.
2. Hydroxylamine Hydrochloride solution: dissolve 10g $\text{NH}_2\text{OH}\cdot\text{HCl}$ in 100ml water.
3. Ammonium acetate buffer solution: dissolve 125g $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$ in 75ml water. Add 700ml conc. (glacial) acetic acid.

4. Phenanthroline solution: dissolved 100mg 1, 10-phenanthroline monohydrate $C_{12}H_8N_2H_2O$, in 100ml water by stirring and heating to $80^\circ C$. Do not boil. Discard the solution if it darkens. Heating is unnecessary if two drops of conc. HCl are added to the water. 1ml of this reagent is sufficient for no more than 100mg Fe.
5. Stock iron solution: slowly add 20ml conc. H_2SO_4 to 50 ml distilled water and dissolve 1.404g $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$. Add 0.1 mol^{-1} potassium permanganate ($KMnO_4$) drop by drop until a faint pink colour persists. Dilute to 1L with distilled water and mix.
6. A standard iron solution: pipette 50.00ml stock solution into 1l volumetric flask and dilute to mark with distilled water.
7. Pipette 5.00ml of stock solution into a 1l volumetric flask and dilute to the mark with distilled water $1 \text{ ml} = 1.00 \text{ mg}$ of Fe.

Preparation of calibration graph

1. Prepare a blank and a series of standards to a range of 100-400 using 100ml volumetric flask or 50-200 range using 50ml volumetric flask using 1cm cell.
2. Treat the blank and standards as indicated in the procedure. Zero the instrument against distilled water and measure the absorbance of the standard and the blank.
3. Subtract the absorbance of the blank from the absorbance of the standards to obtain the net absorbance. Prepare a calibration graph relating net absorbance to the amount of iron.

Procedure:

1. Mix the sample thoroughly and measure 50.0ml into a 125ml Erlenmeyer flask. If the sample contains more than $200 \mu\text{g}$ iron, use a smaller accurately measured portion and dilute to 50ml and add 2ml of concentrated HCl and 1ml of hydroxylamine hydrochloride solution.

2. Drop in a few glass beads and boil until the volume is reduced to 10-20ml. Cool to room temperature.
3. Transfer to 50ml or 100ml volumetric flask. Add 10ml ammonium acetate buffer solution. Add 2ml of phenanthroline solution and dilute to the mark with distilled water. Mix thoroughly and set aside for 10-15 minutes for full colour development.
4. Measure the colour absorbance intensity photo metrically at 510nm. Subtract the absorbance of the blank from that of the sample to determine the net absorbance.

Iron concentration Calculation in Appendix A.

3.3.3 Phosphate – Phosphorous (PO_4^{3-} - P), Ascorbic Acid Method

Reagents

1. Sulphuric acid, H_2SO_4 : Dilute 70 ml Concentrated H_2SO_4 to 500ml with distilled water.
2. Potassium antimony tartrate solution: dissolve 1.3715g $\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6 \cdot 1/2\text{H}_2\text{O}$ in a 500 ml volumetric flask and dilute to volume, Store in a glass Stoppard bottle.
3. Ammonium molybdate solution: dissolve 20g $(\text{NH}_4)_6\text{MoO}_{24} \cdot 4\text{H}_2\text{O}$ in 500ml distilled water, store in a glass Stoppard bottle.
4. Ascorbic acid, 0.1M: Dissolve 1.76g ascorbic acid in 100ml distilled water. The solution is stable for about 1 week at 4°C..
5. Stock Phosphate solution: dissolve in distilled water 219.5mg anhydrous KH_2PO_4 and dilute to 1000ml; 1.00ml = 50.0 μg PO_4^{3-} - P.
6. Standard Phosphate solution: dilute 50ml stock phosphate solution to 1000ml with distilled water; 1.00ml = 2.50 μg PO_4^{3-} - P.

Procedure:

Pipette 50ml sample into a clean, dry test tube or 125ml Erlenmeyer's flask. Add 1 drop of phenolphthalein indicator. If a red colour develops, add H_2SO_4 solution drop wise to just discharge the colour. Add 8ml combined reagent and mix thoroughly. After at least 10min. but no more than 30minutes, measure absorbance of each sample at 880nm, using reagent blank as the reference solution.

Preparation of calibration curve – prepare individual calibration curves from a series of six standards within the phosphate ranges from 0-6 μ g. plot absorbance against phosphate concentration.

3.3.4 Potassium, Sodium and Lithium Using Flame Photometer

Principle:

When metal ions in solution are aspirated into a low temperature flame (in an aerosol form) the electrons of the ion are excited to higher energy states. When these electrons return to the ground state, they lose the excitation energy and a discrete wavelength of visible light is emitted. This light wavelength can be isolated from other light wavelength by an optical filter and the amount of light emitted can be detected with suitable photo-detector. The amount of light emitted is proportional (for low concentrations of metal ion only) to the number of ions in the flame and hence the number of ions in solution. The electrical signal from the photo-detector is amplified and displaced on a digital readout.

Procedure:

1. Turn on the fuel at the source. Switch on the air compressor.

2. Depress the power switch to switch on the flame photometer. The Light Emitting Diode (LED) will be illuminated and an ignition cycle will commence.
3. If the flame on LED is not illuminated at the end of the ignition cycle, check the setting of the fuel control.
4. Set the filter selector to the required position.
5. Insert the nebulizer inlet tube in a beaker containing 100ml of diluents and allow 15 minutes for the operating temperature to stabilize. This will ensure a stable burner temperature when solutions are aspirated, after the warm up period.
6. During the warm up period prepare a set of calibration solutions to cover the required measurement range. To obtain maximum linearity, Sherwood Scientific recommend that the highest standard concentration does not exceed 30 mg/L for Sodium, 10mg/L for Potassium and 10mg/L for Lithium.
7. While aspirating diluents, adjust the blank control so that the display read 0.0
8. Aspirate the highest concentration standard.
9. Allow 20 seconds for a stable reading and then adjust coarse and fine controls for a convenient reading e.g. 20mg/L of Sodium can be set to read 20 on the display.
10. Remove the standard solution, wait 10 seconds, then aspirate a blank solution of diluents for 20seconds. Adjust the blank control for a 0.0 reading. Remove the blank solution and wait 10 seconds.

11. Repeat paragraph 8,9,10 until the blank reading is 0.0 (within ± 0.2) and calibration reading is within $\pm 1\%$. If a chart recorder is being used set zero on the blank solution and set span while aspirating the calibration curve.

12. Aspirate each of the remaining calibration standards for 20 seconds (starting with the lowest concentration to avoid carry over) again allowing 10 seconds between measurements. Note the value of each standard and plot the results on a graph against standard concentration on linear graph paper.

13. Check calibration standards and blank readings.

14. Dilute the unknown solutions with diluents to give a concentration of the element under test within the range of the calibration standards. Several attempts might be necessary to determine the correct dilution ratio.

15. Aspirate each of the diluted unknowns for 20 seconds, and then note the readings. The concentration of the element in the unknown sample can be calculated by reading the sample concentration from the calibration curve and multiplying it by the dilution factor.

3.4 Crop Analysis (phyt-available metals)

3.4.1 Materials

10 samples of each of the crop samples from site A and B were taken and tested.

3.4.2 Preparation of Samples

3.4.2.1 Dry Ashing

High-temperature oxidation destroys the organic material. The plant sample is ashed at 500–600 °C by placing a suitable weight (0.5–1.0g) of the sample in a silica crucible and heating it in a

muffle furnace for 4–6 hours. The ash residue is dissolved in dilute HNO₃ or HCl, filtered through acid-washed filter paper in a 50/100-ml volumetric flask, and the volume is made up to the mark. The estimation of K, Ca, Mg and micronutrients (including B and Mo) is carried out in the dry-ashed sample solution. Dry ashing is a preferred method for the analysis of P, K, Ca, Mg and trace elements, especially B and Mo. It is a relatively simple method and requires very little operational attention. It does not involve the use of perchloric acid. It also avoids the use of boiling acids. However, at times, incomplete recovery of some elements may be caused by volatilization of elements such as S (also Se and halogens).

To avoid loss of S, Mg (NO₃)₂ should be mixed with plant samples while dry ashing.

Retention of elements such as Cu on the walls of silica crucibles, hence, platinum crucible should be used.

Formation of compounds that are not completely soluble in the acid is used for digestion.

A blank should always be carried out to account for any contamination through the acids used in the digestion.

3.4.3 Nitrogen

Total N in plants is estimated by the Kjeldahl method. In plants, N is present in protein form, and digestion of the sample with H₂SO₄ containing digestion mixture (10 parts potassium sulphate and 1 part copper sulphate) is required for estimation. Sample size may be 0.5–1.0 g depending on the type of crop and the plant part.

The procedure for sample digestion, distillation and estimation of N is the same as for total N estimation in soil.

3.4.4 Phosphorus

The Phosphorus content of the plant sample is converted to orthophosphates by digestion with an

acid mixture (di-acid or tri-acid). The digested sample is used for Phosphorus estimation. When orthophosphates are made to react with molybdate and vanadate, a yellow-coloured vanadomolybdophosphoric heteropoly complex is formed. The intensity of the yellow colour is directly proportional to the concentration of Phosphorus present in the sample, which can be read on the spectrophotometer.

The apparatus required consists of: A digestion block, a spectrophotometer, beakers/flasks.

The reagents required are:

1. Ammonium molybdate – ammonium vanadate in HNO_3 (vanadomolybdate): Dissolve 22.5 g of $(\text{NH}_4)_6\text{MO}_7\text{O}_2 \cdot 4\text{H}_2\text{O}$ in 400 ml of distilled water. Dissolve 1.25 g of ammonium vanadate in 300 ml of boiling distilled water. Add the vanadate solution to the molybdate solution and cool to room temperature. Add 250 ml of concentrated HNO_3 and dilute to 1 litre.
2. Standard phosphate solution: Dissolve 0.2195 g of analytical-grade KH_2PO_4 and dilute to 1 litre. This solution contains 50 μg P/ml.

Procedure:

1. Preparation of the standard curve: Put 0, 1, 2, 3, 4, 5 and 10 ml of standard solution (50 μg P/ml) in 50-ml volumetric flasks. Add 10 ml of vanadomolybdate reagent to each flask and make up the volume. The P contents in these flasks are 0, 1, 2, 3, 4, 5 and 10 μg P/ml, respectively.

The standard curve is prepared by measuring these concentrations on a spectrophotometer (420 nm) and recording the corresponding absorbance.

2. Take 1g of plant sample and digest as per the wet digestion method, and make the volume up to 100 ml.

3. Put 5ml of digest in a 50ml volumetric flask, and add 10 ml of vanadomolybdate reagent.

4. Make up the volume with distilled water, and shake thoroughly. Keep for 30 minutes.
5. A yellow colour develops which is stable for days and is read at 420 in spectrophotometer.
6. For the observed absorbance, determine the P content from the standard curve.

The relevant calculation in Appendix (B₄)

3.4.5 Potassium

The acid-digested or dry-ashed plant sample is used for determining K.

The reagents required are; Di-acid/tri-acid digestion mixture and KCl (AR-grade) standard solution:

Procedure:

1. Set up the AAS and standardize. The relevant parameters for K estimation on an AAS are:
Lamp current = 6 mA, Wavelength = 766.5 nm, Linear range = 0.4–1.5 µg/ml, Slit width = 0.5 nm, Integration time = 2 seconds, Flame = air acetylene.
2. Preparation of the standard curve: Prepare the standard curve using 0, 5, 10, 15 and 20 µg K/ml. The curve will show a linear relationship between the concentration of K and absorbance on a specific wavelength as read from the AAS.
3. Acid-digest 1 g of plant sample and make up to 100 ml. Keep the sample for estimation in the range 5–10 mg K/kg (5–10 µg K/ml) by further diluting as appropriate.
4. Prepare a blank in the same way without adding plant digested material.
5. Take an aliquot of 5 ml for estimation and make up to 100 ml. Atomize on the calibrated AAS, on which the standard curve has also been prepared.
6. Record the absorbance against each sample.
7. From the standard curve, note the concentration of K for the particular absorbance observed for the sample.

Relevant calculation in Appendix A

3.4.6 Calcium

Estimation by AAS is described here. However, Ca estimation in the acid digest can also be done by the EDTA (ethylene diamine tetra-acetate) titration method.

The apparatus required consists of: An AAS, Some volumetric flasks, a fume hood, a hotplate; a muffle furnace (when dry ashing has to be done).

Reagent:

Standard Ca solution: Take 0.2247 g of primary standard CaCO_3 and add 5 ml of deionized water. Add about 10 ml of HCl to ensure complete dissolution of CaCO_3 . Dilute to 1 litre with deionized water. This will give Ca solution of 100 μg Ca/ml. Dilute 10 ml of this solution to 100 ml to obtain 10 μg Ca/ml.

Procedure:

1. Take 1 g of prepared plant sample. Digest in di-acid, and make the volume up to 100 ml.
2. Dilute the sample solution to 10–20 times depending on expected content of Ca, which can be estimated from the standard curve prepared for the purpose.
3. Set up and calibrate the AAS using the relevant parameters: lamp current = 10 mA; Wavelength = 422.7 nm; Linear range = 1–4 $\mu\text{g}/\text{ml}$; Slit width = 0.5 nm; Integration time = 2 seconds; Flame = nitrous oxide acetylene.
4. After setting the AAS, atomize the standard solutions of different concentrations of Ca and record the absorbance for the respective concentrations of Ca. Plot the concentration of Ca on the x-axis and the corresponding absorbance on the y-axis in order to prepare the standard curve.

5. Put 5 ml of the sample solution in a 100-ml volumetric flask and make up the volume, atomize, and observe the absorbance. Note the corresponding concentration for the absorbance recorded that represents the content of Ca in the sample solution.

The relevant calculation of Ca contents in Appendix A

3.4.7 Magnesium

Estimation by AAS is described here. However, Mg estimation in the acid digest can also be done by the EDTA titration method.

Reagent:

Standard Mg solution: Dissolve 10.141 g of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ in 250 ml of deionized water, and make the volume up to 1 litre. This will give 1 000 μg Mg/ml solution. Under this procedure, the preparation of the standard curve, the estimation and the calculation procedure are the same as described for Ca estimation (above). The relevant parameters for estimation by AAS are:

Lamp current = 3 mA, Wavelength = 285.2 nm, Linear range = 0-0.5 $\mu\text{g}/\text{ml}$, Slit width = 0.5 nm, Integration time = 2 seconds, Flame = air acetylene.

The procedure is done by the EDTA titration method

3.5 Crop Analysis (Proximate)

The samples were analyzed for Crude protein, crude fat, lipid content, Ash, Moisture content and Carbohydrates using the official methods of the Association of Official Analytical Chemists (A.O.A.C., 1980).

3.5.1 Reagents

The reagents used for this analysis are:

- Potassium sulfate (K_2SO_4)

- Copper sulfate (CuSO_4)
- Concentrated sulfuric acid (H_2SO_4)
- Sodium hydroxide (NaOH)
- Methyl red indicator solution
- Petroleum ether (C_6H_6)
- 60-62% Perchloric acid (HClO_4)
- Concentrated nitric acid (HNO_3)
- Boric acid

3.5.2 Apparatus

- | | |
|---------------------|-------------------------------|
| •Crucible | •Desiccators |
| •Muffle furnace | •Volumetric flasks |
| •Beakers | •Measuring cylinder |
| •Conical flask | •Spatula |
| •Filter paper | •Muslin cloth |
| •Condenser | •Soxhlet Extraction Apparatus |
| •Round bottom flask | •Extraction thimble |
| •Heating mantle | •Kjeldahl flask |

- Burettes
- Petri dish
- Water bath
- Macro kjeldahl Nitrogen Digestion and Distillation Apparatus
- Pipette
- Filter paper
- Oven

3.5.3 Ash

This is the residue of incineration at 550-600°C. This analysis was carried out using standard analytical method A.O.A.C, (1980).

Procedure

The crucible was washed and dried in the oven at a temperature of 100°C for six hours; it was removed from the oven and kept in a desiccator to cool. It was weighed and the weight was recorded as W_1 . 2g of the yellow sample was weighed into the crucible. The crucible was reweighed and the new weight was recorded as W_2 . The crucible was kept in a muffle furnace at a temperature of 600°C for 2 hours. It was removed from the muffle furnace and kept in the desiccators to cool. It was then weighed and the weight was recorded as W_3 . This process was repeated twice. The procedure was also carried out for brown sample in triplicates respectively.

%Ash was calculated from the weights gotten from the procedure as follows;

$$\%Ash = (W_3 - W_1 / W_2 - W_1)100$$

Where,

W_1 = weight of crucible

W_2 = weight of crucible and sample before ash

W_3 = weight of crucible and sample after ash

3.5.4 Crude Fat

Fat are glyceride of fatty acids which are soluble in non polar organic solvents. Extraction is carried out with soxhlet apparatus with petroleum ether. The usual method is continuous heating at 40-60°C. The ether extraction method is based on the principle that non polar components of the sample are easily dissolved in ether. Direct extraction gives the proportion of free fat but gives no clue to the particular fatty acids. A.O.A.C, (1980) was used to carry out this analysis.

Procedure

Glass wares were washed with distilled water and dried in an oven. The thimble was washed and kept in an oven to dry and its weight was taken as W_1 . 2g of moisture free plant sample was added; the weight of thimble plus sample was taken and recorded as W_2 . A fat free 500ml round bottom flask was then weighed and the weight was recorded as W_3 . The flask was then filled with two third of its volume with petroleum ether. A soxhlet extractor with a reflux condenser was fitted together; heat was applied using a heating mantle at a temperature of 60°C so that the solvent boiled gently, it was left to siphon for a period of 8 hours. The condenser was detached and the thimble was removed. The flask containing fat residue was dried on a water bath. It was kept in desiccators to cool, it was weighed and the weight was recorded as W_4 . The fat content of the plant was calculated as follows;

$$\% \text{ Crude fat} = (W_4 - W_3 / W_2 - W_1) 100$$

Where,

W_1 = weight of empty thimble

W_2 = weight of the thimble and plant sample

W_3 = weight of fat free empty flask

W_4 = weight of flask containing oil residue

W_4 = weight of flask containing oil residue

3.5.5 Crude Protein

The central basis used in this procedure is the oxidation of the organic compound using strong sulfuric acid. As the organic material is oxidized the carbon it contains is converted to carbon dioxide and the hydrogen is converted into water. The nitrogen, from the amine groups found in the peptide bonds of the polypeptide chains, is converted to ammonium ion, which dissolves in the oxidizing solution, and can later be converted to ammonia gas. The Kjeldahl method of nitrogen analysis is the worldwide standard for calculating the protein content in a wide variety of materials.

Procedure

Step 1: Digestion

12ml concentrated H_2SO_4 was measured using a 100ml measuring cylinder into a kjeldahl flask. One digestion tablet was added. 1g of plant sample was weighed and added to the mixture. The mixture was weighed and recorded as W_1 . Heat was applied using a kjeldahl digestion block at a low temperature for 15 minutes; the temperature was increased to medium for 30 minutes and was later increased to the highest temperature until a grey white coloration was noticed. Heating

continued for 3 more minutes to complete digestion. The digest was left to cool and filtered. The filtrate was made up to 50ml (V_1) and transferred into a round bottom flask. A blank digestion was carried out using the method above.

Step 2: Distillation

The distillation apparatus was set up. A few pumic stone was added to the filtrate from step. 15 ml of boric acid was added into a 100ml Erlenmeyer flask, 3 drops of indicator was added. It was carefully placed below the condenser with the receiver tube dipped in the acid. 10ml of 40% NaOH was poured carefully into the flask. The flask was quickly connected to the distillation system so as to avoid the loss of nitrogen. The volume of the distillate was measured as V_2 . A blank distillation was carried out using this method.

Crude protein is obtained by multiplying the % Nitrogen by 6.25 for food samples. The factor 6.25 owes its origin to the assumption that all food protein contains 16% nitrogen and that all the nitrogen in feed are present as protein.

Therefore,

$$\% \text{ Crude Protein} = \text{NFE} \times \text{Factor}$$

Where NFE = Nitrogen Free Extract.

3.5.6 Carbohydrate

Carbohydrates are generally referred to as the energy giving portion of food material; the end product of digestion of carbohydrate is glucose which is stored in plants as starch and in animals as glycogen. According to the procedure outlined by AOAC, (1980), for the determination of

carbohydrate content, the percentage carbohydrate content is mathematically obtained from the expression below.

$$\% \text{Carbohydrate} = 100\% - (\% \text{protein} + \% \text{lipid} + \% \text{ash} + \% \text{ fibre})$$

Since milk generally has no fibre content (Anderson *et al*; 2007)

Therefore;

$$\% \text{Carbohydrate for milk} = (\% \text{protein} + \% \text{lipid} + \% \text{ash})$$

CHAPTER FOUR

4. RESULTS AND DISCUSSION

4.1 Wastewater Analysis

The upstream, midstream and downstream of the wastewater samples were collected and tested.

The results of wastewater analysis are presented in Table 4.1 below:

Table 4.1: Upstream Wastewater Analysis Result

Parameters	Unit	Measured Value	FAO Standard
Dissolved oxygen	mg/L	1.00	-
Temperature	°C	24.8	24-32
Conductivity	ds/cm	993	10-1000
Turbidity	NTU	43.9	5
Chloride	mg/l	110	250
Sodium chloride	mg/l	176	-
Nitrate	mg/l	0.05	3
Ammonia	mg/l	10.91	0-5
Calcium	mg/l	41.68	20
Magnesium	mg/l	4.66	5

Parameters	Unit	Measured Value	FAO Standard
Total hardness	mg/l	410	250
Calcium hardness	mg/l	104	-
Magnesium hardness	mg/l	306	-
Alkalinity	mg/l	230	-
Manganese	mg/l	4.3	2.0
Aluminum	mg/l	0.265	1.0
Chromium	mg/l	10	1.0
Phosphate	100ml	1.14	0

The upstream sample taken from a location Morisferterlizer around Tunga in Minna is found to be high in composition of heavy metals such as manganese and chromium which is above the FAO irrigation water standard. These would have long term effect on the soil in the area and can define the type of water in the middle stream where the study area is located. The sample was also high in ammonia and calcium. Chromium is not generally recognized as essential growth element. Conservative limits recommended due to lack of knowledge on toxicity to plants.

Table 4.2: Midstream Wastewater Analysis Result

Parameters	Unit	Measured Value	FAO Standard
Dissolved oxygen	mg/l	7.17	-
Temperature	°C	28.5	24-32
Conductivity	ds/cm	504	10-1000
Turbidity	NTU	5.66	5
Chloride	mg/l	33	250
Sodium chloride	mg/l	52.8	-
Nitrate	mg/l	0.05	3
Ammonia	mg/l	4.38	0-5
Calcium	mg/l	34.07	20
Magnesium	mg/l	3.66	5
Total hardness	mg/l	100	250
Calcium hardness	mg/l	85	-
Magnesium hardness	mg/l	15	-
Alkalinity	mg/l	16	-
Manganese	mg/l	0.5	2.0

Parameters	Unit	Measured Value	FAO Standard
Aluminum	mg/l	0.004	1.0
Chromium	mg/l	10	1.0
Phosphate	100ml	0.192	0

The middle stream sample collected at the site of cultivation where it is directly used for irrigation also contains high levels of chromium and calcium. Calcium which is required by plants as macronutrients is required in small quantities and not in excess. Levels of aluminum, magnesium and manganese are within the standard range.

Table 4.3: Downstream Wastewater Analysis Result

Parameters	Unit	Measured Value	FAO Standard
Dissolved oxygen	mg/L	7.14	-
Temperature	°C	28.2	24-32
Conductivity	ds/cm	418	10-1000
Turbidity	NTU	4.41	5
Chloride	mg/l	27.5	250
Sodium chloride	mg/l	44	-
Nitrate	mg/l	0.05	3
Ammonia	mg/l	4.36	0-5

Parameters	Unit	Measured Value	FAO Standard
Calcium	mg/l	34.07	20
Magnesium	mg/l	0.0	5
Total hardness	mg/l	85	250
Calcium hardness	mg/l	85	-
Magnesium hardness	mg/l	15	-
Alkalinity	mg/l	16	-
Manganese	mg/l	0.0	2.0
Aluminum	mg/l	0.036	1.0
Chromium	mg/l	10	1.0
Phosphate	100ml	0.132	0

The downstream sample taken from a location southwest of the site location called Sauka Kwaut also high in chromium and calcium. These may be as a result of depositions at the area

4.1.1 Physiochemical Analysis

Total Hardness: This is due primarily to calcium and secondary to magnesium carbonates bicarbonates. Thus, wastewater to be used for irrigation, the hardness must not exceed 150mg/l.

The municipal wastewater total hardness result of 106mg/l satisfies the total hardness standard of irrigation water but away from total hardness, there are so many other analytical parameter in their standards that will jointly tells if irrigation water is safe to use.

Chloride (Cl), Fluoride (F), and Sodium (Na):- These are toxic ions. Irrigation water that contains these ions at above tolerance value can cause plant toxicity problems, Such as impaired growth, reduced yield, changes in morphology of plant and even death. For safe, chlorine and sodium should be present in irrigation in the range (0 – 30mg/L) and (0 – 40mg/L) respectively (FAO, 2003). From the wastewater analysis, chlorine (23.98mg/L) and sodium (31.5mg/L) are within the safe range.

Nitrates: This represents the final product of the biochemical oxidation of ammonia. In water, the presence of nitrate is probably due to the presence of nitrogen organic matter and to some extent, of vegetable origin, for only small quantities are naturally present in water. Nitrate content may be considered toxic if it exceeds 10mg/L (FAO, 2003). From the municipal wastewater analysis, the nitrate content is very high (182.45mg/L) which poses great threat (toxicity) on plant nutritional value.

4.2 Plant (Spinach) Nutrient Analysis

Through photosynthesis, green plants manufacture their own organic food, using carbon dioxide and oxygen as raw materials. The nutrients usually supplied to plants by soil are almost entirely mineral salts. Plant physiologists have discovered that plants require carbon, hydrogen, oxygen, nitrogen, phosphorus, potassium, magnesium, sulfur, calcium, iron, manganese, boron, zinc, copper, and probably molybdenum. Carbon, hydrogen, and oxygen are obtained in large quantities from water and air, but the remaining elements are ordinarily supplied as salts by the soil. The relative amount of each of these elements required for normal growth is different in each plant, but all plants require relatively large proportions of nitrogen, phosphorus, potassium, magnesium, sulfur, and calcium. Iron, manganese, boron, zinc, copper, and molybdenum are

supplied in minute quantities, and are called micronutrients or trace metals. Plant nutrient analysis in Table 4.4 shows proximate analysis, mineral analysis and vitamin analysis.

Table 4.4 Plant (Spinach) Nutrient Analysis Result

Nutrient	Unit	WWIS	PWIS
PROXIMATE:			
Water	g	89.07	90.80
Protein	g	2.70	2.79
Fat/Oil	g	0.36	0.31
Ash	g	2.41	2.50
Carbohydrate	g	5.46	3.60
Fiber	g	1.78	2.00
MINERALS:			
Calcium, Ca	mg	95.00	97.00
Iron, Fe	mg	1.93	1.21
Phosphorus, P	mg	45.10	47.40
Potassium, K	mg	530.00	543.00

Nutrients	Unit	WWIS	PWIS
Vitamin C	mg	25.4	24.1

Sixteen elements are absolutely necessary for normal plant growth. Many of these elements are the same as those required by humans. In addition to carbon, hydrogen, and oxygen, which the plant gets from the air and water, another thirteen elements are required by plants, which they obtain from the soil. These are usually divided into three classes: primary nutrients, secondary nutrients, and micronutrients. Functions of elements in plant metabolism and symptoms are related to their deficiencies. Based on soil test, fertilizers are applied to provide plants with some of these essential nutrients for optimal growth. Examples are enlisted above with their respective evaluated composition per 100g of edible portion in spinach

4.3 Evaluation of Plant (Spinach) Analysis Result

The results of the plant analysis can be compared with the standard nutritional values of spinach from the sourced from the USDA National nutrient database, 2004 (see Appendix B). Table B, shows the standards of the analyzed nutritional levels of spinach and comparisons of both WWIS and PWIS to those of the USDA standard values having considered the standard error involved.

Table 4.5 Comparison of Plant (Spinach) Analysis Result

NUTRIENT	Unit	USDA	(±) Std Error	WWIS	WWIS*	PWIS	PWIS*
PROXIMATE:							
Water	g	91.40	0	89.07	87.07	90.80	90.80
Protein	g	2.86	0.112	2.70	2.81	2.79	2.90
Fat/Oil	g	0.39	0.032	0.36	0.39	0.31	0.34
Ash	g	1.72	0.035	2.41	2.45	2.50	2.54
Carbohydrate	g	3.63	0	5.46	5.46	3.60	3.60
Fibre	g	2.2	0	1.78	1.78	2.00	2.00
METALS:							
Calcium, Ca	mg	99	4.996	97.00	101.2	95.00	99.2
Iron, Fe	mg	2.71	0.522	1.21	1.73	1.93	2.45
Magnesium, Mg	mg	79	4.794	76.10	80.89	75.00	79.79
Phosphorus, P	mg	49	3.479	47.40	50.88	45.10	48.58
Potassium, K	mcg	558	28.703	543.00	571.70	530.00	558.70
VITAMINS:							
Vitamin C	mg	28.1	4.129	25.4	29.53	24.1	28.22

Values are in per 100g of edible portion

WWIS* – waste water irrigated spinach having compared with the USDA

PWIS* –potable water irrigated spinach having compared with the USDA's

As seen from the above table, the nutritional level of the potable water irrigated spinach PWIS is generally closer to the USDA standard nutritional level for spinach with exception of fat/oils and carbohydrates. It also has slightly higher values in the nutriment values when compared with the wastewater irrigated spinach WWIS. In the case of the carbohydrate proximate for WWIS, having considered the standard error, the reason for the large difference being that the wastewater irrigation readily and excessively gives up mineral nutrient to the plant that nurtures the growth of fat/oil and carbohydrates in plants. This shows that toxicity (excess of plant mineral nutrient) problem occurs in wastewater irrigation having exceeded the USDA's standard. This could be detrimental to human health.

But from all indication, as it relates to this study, considering the relative impact of the wastewater on the respective plant (spinach) nutrient, to show visible variations in their impacts on plant nutrient considering the descriptive nature of Table above, wastewater irrigated spinach have a larger variation in the proximate, metals and vitamins compared with the portable irrigated spinach, with lower values in protein, fiber, calcium, iron, magnesium, phosphorus, potassium and vitamin C.

Farming communities in water-scarce regions increasingly practice the use of urban wastewater in agriculture. Untreated urban wastewater is generally considered unacceptable for direct use because of potential health risks. However, in many parts of the world, poor farmers in peri-urban areas use untreated wastewater.

CHAPTER FIVE

5. CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

From the observed parameters in this study, the composition of metals in the wastewater are found to have effects on spinach cultivated in the area, with the WWIS having higher composition of metals and lower proximate values which is non compliance with the acceptable limits set by FAO. The results from this study highlight the economic value of untreated urban wastewater in crop cultivation. The most important benefit to farmers in this semi-arid country is the reliable supply of wastewater, which allows them to grow high-value vegetable crops.

5.2 Recommendations

The research work points out some effect of using untreated wastewater for irrigation and suggests that proper analysis and treatment of waste should be performed before depositions into water bodies as issued by the Nigeria Land Use Act (1978).

Water hyacinth with proper control measures could be used for local purification in such areas. Todd and Josephson (1996) reported that the plant is extremely tolerant towards, and of high capacity of up taking heavy metals, such as Cd, Cr, Co, Ni, Pb and Hg, hence could be utilized for the bio-cleaning of industrial wastewater. Not only the heavy metals, it can also remove toxins such as cyanide.

In further studies, wider range of heavy metals should be used as basis for sampling and testing, including soil tests

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APPENDICES

Appendix A

Determination of Effluent Analysis

Calcium hardness as CaCO_3

$$\text{Mg CaCO}_3/\text{L} = \frac{(A-B) \times D \times 100}{\text{ml of sample}}$$

$$\text{Calcium as Ca}^{2+}/\text{L} = \frac{(A-B) \times D \times 400.8}{\text{ml sample (100)}}$$

Magnesium hardness ($\text{mg CaCO}_3/\text{l}$) = total hardness – calcium hardness

Calculated magnesium as mg^{2+}

$$\text{mg}^{2+}/\text{l} = \text{magnesium hardness as mg CaCO}_3/\text{l} \times 0.244 = 34.07\text{mg/l}$$

Calculation for manganese in water:

When the entire original sample is taken for analysis,

$$\text{Mg Mn}/\text{L} = \frac{\text{Mg Mn (in 100ml final volume)}}{\text{ml sample}}$$

$$\text{Normality of KMnO}_4 = \frac{\text{g Na}_2\text{C}_2\text{O}_4}{(A-B) \times 0.06701}$$

Where A is ml Titrant for sample and B is ml Titrant for blank.

Average results for several titrations. Calculate volume of this solution necessary to prepare

1 Liter of solution so that 1 ml = $50\mu\text{gMn}$ as follows:

$$\text{ml KMnO}_4 = \frac{4.55}{\text{normality KMnO}_4}$$

Calculation for Phosphorus

content in 100g of sample

$$P \text{ content (g) in 100g samples (\%P)} = \frac{C \times df \times 100}{1000000} = \frac{C \times 1000 \times 100}{1000000} = \frac{C}{10}$$

Where:

C = concentration of P ($\mu\text{g/ml}$) as read from the standard curve;

df = dilution factor, which is $100 \times 10 = 1\,000$, as calculated below:

- 1 g of sample made to 100 ml (100 times);
- 5 ml of sample solution made to 50 ml (10 times).

1 000 000 = factor for converting μg to g.

Calculation for Potassium

content in 100g of sample

$$K \text{ content (g) in 100g samples (\%K)} = \frac{C \times df \times 100}{1000000} = \frac{C \times 2000 \times 100}{1000000} = \frac{C}{5}$$

Where:

C = concentration of K ($\mu\text{g/ml}$) as read from the standard curve;

df = dilution factor, which is $100 \times 20 = 2\,000$, as calculated below:

- 1 g of sample made to 100 ml (100 times);
- 5 ml of sample solution made to 100 ml (20 times).

1 000 000 = factor for converting μg to g.

Calculation for Calcium

$$Ca \text{ content (g) in 100g samples (\%Ca)} = \frac{C \times df \times 100}{1000000} = \frac{C \times 2000 \times 100}{1000000} = \frac{C}{5}$$

Where:

C = concentration of Ca ($\mu\text{g/ml}$) as read from the standard curve;

df = dilution factor, which is $100 \times 20 = 2\ 000$, as calculated below:

- 1 g of sample made to 100 ml (100 times);
- 5 ml of sample solution made to 100 ml (20 times).

1 000 000 = factor for converting μg to g.

APPENDIX B

Table B: Standard Nutritional Level of Spinach

Nutrients	Units	Value Per 100 Gram Of Edible Portion	Number of data point	Std Error
PROXIMATES:				
Water	g	91.40	1	0
Energy	kcal	23	0	0
Energy	kJ	97	0	0
Protein	g	2.86	9	0.112
Total lipid (fat)	g	0.39	7	0.032
Ash	g	1.72	8	0.035
Carbohydrate, by difference	g	3.63	0	0
Fibre, total dietary	g	2.2	1	0
Sugars, total	g	0.42	0	0
Sucrose	g	0.07	8	0.036
Glucose (dextrose)	g	0.11	8	0.032
Fructose	g	0.15	8	0.07
Lactose	g	0.00	1	0
Maltose	g	0.00	1	0
Galactose	g	0.10	1	0
MINERALS:				
Calcium, Ca	mg	99	9	4.996
Iron, Fe	mg	2.71	10	0.522
Magnesium, Mg	mg	79	7	4.794
Phosphorus, P	mg	49	7	3.479
Potassium, K	mg	558	10	28.703

Adapted from the USDA National nutrient database, 2004

Name/Location of Sampling Point: Soje (Kpakungu)
 Date Sample Collected: 27/4/2009
 Date / Time Sample Delivered To the Laboratory: 27/4/2009
 Source of Water Point: Waste Water (Middle stream)
 State/Local Government of Sampling Point: Minna L.GA / Niger
 Sample Analyzed by: Laboratory Analysts

PARAMETER	UNIT	MEASURED VALUE	WHO GUIDE LEVEL	REMARK
Dissolved Oxygen	mg/L	7.17	-	O.K
Color	Pt-Co	-	15	O.K
Temperature	^o C	28.5	-	O.K
Conductivity	µs/cm	504	10-1000	O.K
Turbidity	NTU	5.66	5	high
Total Dissolved Solid	mg/L	337.68	1000	O.K
Chloride	mg/L	33	250	O.K
Sodium Chloride	mg/L	52.8	-	O.K
Ammonia	mg/L	4.36	-	O.K
Hydroxide (OH ⁻)	mg/L	0	250	O.K
Nitrate-Nitrogen (NO ₃ -N)	mg/L	19.30	-	O.K
Calcium ²⁺	mg/L	34.07	-	O.K
Magnesium ²⁺	mg/L	3.66	-	O.K
Total Hardness	mg/L	100	250	O.K
Calcium Hardness	mg/L	85	-	O.K
Carbonate (CO ₃)	mg/L	0	-	O.K
Magnesium Hardness	mg/L	15	-	O.K
Alkalinity	mg/L	16	-	O.K
Manganese	mg/L	0.5	-	O.K
Aluminum	mg/L	0.004	-	O.K
Chromium	mg/L	<10	-	O.K
Phosphate (PO ₄ -P)	/100mL	0.192	0	O.K
Bicarbonate (HCO ₃ ⁻)	/100mL	16	0	O.K

REGIONAL WATER QUALITY
 LAB MINNA
 SIGNATURE: [Signature]
 DATE: 20/09

Name/Location of Sampling Point: Soje (Kpakungu)

Date Sample Collected: 27/4/2009

Date / Time Sample Delivered To the Laboratory: 27/4/2009

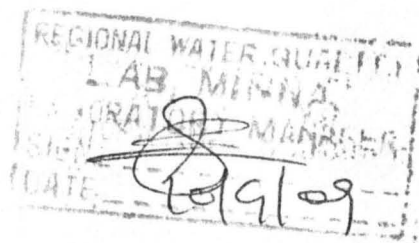
Source of Water Point: Waste Water (Upstream)

State/Local Government of Sampling Point: Minna LGA / Niger

Sample Analyzed by: Laboratory Analysts

PARAMETER	UNIT	MEASURED VALUE	WHO GUIDE LEVEL	REMARK
Dissolved Oxygen	mg/L	7.41	-	O.K
Color	Pt-Co	-	15	O.K
Temperature	°C	28.2	-	O.K
Conductivity	µs/cm	418	10-1000	O.K
Turbidity	NTU	4.41	5	O.K
Total Dissolved Solid	mg/L	280.06	1000	O.K
Chloride	mg/L	27.5	250	O.K
Sodium Chloride	mg/L	44	-	O.K
Ammonia	mg/L	4.36	-	O.K
Hydroxide (OH ⁻)	mg/L	0	250	O.K
Nitrate-Nitrogen (NO ₃ ⁻ -N)	mg/L	19.30	-	O.K
Calcium ²⁺	mg/L	34.07	-	O.K
Magnesium ²⁺	mg/L	0.0	-	O.K
Total Hardness	mg/L	85	250	O.K
Calcium Hardness	mg/L	85	-	O.K
Carbonate (CO ₃)	mg/L	0	-	O.K
Magnesium Hardness	mg/L	0	-	O.K
Alkalinity	mg/L	16	-	O.K
Manganese	mg/L	0.4	-	O.K
Aluminum	mg/L	0.036	-	O.K
Chromium	mg/L	<10	-	O.K
Phosphate (PO ₄ ³⁻ -P)	/100mL	0.132	0	O.K
Bicarbonate (HCO ₃ ⁻)	/100mL	16	0	O.K

JAMILU HABU
LABORATORY MANAGER



CENTRAL SERVICES LABORATORY

NATIONAL CEREALS RESEARCH INSTITUTE

BADEGGI

P.M.B. 8

RESULT OF PROXIMATE ANALYSIS

Lab No	Sample Description	% Crude protein	% Crude fibre	% Fat/oil	% Moisture content	% Ash	C/Difference
50/51	A	2.29	2.0	0.31	90.9	2.5	3.6
53	B	2.70	1.78	0.36	89.07	2.41	5.46

Compiled by *M. M. M. M. M.*

Date: *19-6-2009*

Sign

OFFICE OF THE HEAD
 Central Services Laboratory
 National Cereals Research Institute
 Badeggi, Niger State
 Date: *19/6/09*

Lab.

