CONCENTRATION OF IRON IN MURRAM (LATERITE) FROM RUIRU AREA, THIKA DISTRICT, KENYA TO ASSES ECONOMIC VIABILITY.

BY

KERU GODFREY KAMITHA (B.Ed Sci.)

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DECLARATION

I declare that this thesis is my original work and has not been presented for a degree or any other award in any other university

Signed: ....Date..

KERU GODFREY KAMITHA (B.Ed. Sc)

This thesis has been submitted with our approval as the University Supervisors

Signed: .... Date: ....

PROF NAFTALI T. MURIITHI

Chemistry Department,

Kenyatta University

Signed: .... Date: ....

Dr. JACKSON WACHIRA MUTHENGIA

Chemistry Department,

Kenyatta University.

DEDICATION

To my children Chris Maina, Teddy Keru and Debra Wangari for whom I believe this work will be a source of inspiration in their lives.

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ABBREVIATIONS AND ACRONYMS

KNBS	Kenya National Bureau of Statistics
XRD	X-Ray Diffraction
XRFS	X-ray Fluorescence Spectroscopy
AAS	Atomic Absorption Spectroscopy
EDTA	Ethylenediamine Tetra Acetic Acid
NESC	National Economic and Social Council of Kenya
USGS	United States geological survey
MRISA	Minerals Resource Information of South Australia
CCMET	Canadian Centre for Mineral and Energy Technology
SPSS	Statistical Program for Social Scientists.
ANOVA	Analysis of Variance

ABSTRACT

A study on possibility of concentrating iron in laterite (murram) has been carried out. This was done using froth floatation where oleic acid was used as collector and cresylic acid as frother. Magnetic separation was also used. The study has shown that the level of iron in the original sample could be increased from 33% to 55%. Mineral content was determined using X-Ray diffraction which identified iron mineral present as goethite. Chemical analysis was also carried out on raw and concentrated material using Atomic Absorption Spectroscopy (AAS), X-Ray Fluorescence Spectroscopy (XRFS) and titrimetric analysis using ethylenediamine tetra acetic acid (EDTA). Results have also shown that iron present in these laterite, goethite were converted to magnetite by mixing laterite with charcoal and then heating the mixture using charcoal as the source of energy. Magnetite so formed was then concentrated using magnetic separation. It has also been shown that froth flotation can be viably used to concentrate iron in laterite. This method is affordable because the chemicals used for flotation were recovered by washing the concentrate with 1:1 diethyl ether, filtered using vacuum filtration. Diethyl ether was allowed to evaporate and chemicals recovered recycled. Magnetic separation increased amount of iron by 17.98 percent and froth flotation by11.74 percent. The data was analysed using student's t- test and analysis of variance (ANOVA).

CHAPTER ONE

INTRODUCTION

1.1 Background

Elemental iron is ranked fourth in the abundance in the Earth’s crust and is a major constituent of the Earth core (Kirk - Othmer, 2004). It rarely occurs in nature as a native metal. It combines with oxygen and sulphur to form oxides and sulphides. The two common oxidation states are +2 and +3 (Alan, 1995). Pure iron is silvery-white, very ductile, and strongly magnetic and melts at 1528 °C (Kirk - Othmer, 2004).

Availability of iron ore is fundamental to the development and maintenance of industrial processes (Snell and Ettre, 1972). Iron accounts for approximately 95 percent of the metals used by modern industrial society. Iron is the world's most commonly used metal (Mineral Resource Institute South Australia [M.R.I.S.A.], 2006). It is used primarily in structural engineering applications and in maritime purposes, automobiles and general industrial applications (machinery). Other well known uses of iron compounds include the manufacture of iron sulphate which is used as fungicide, oxalate of iron used as pigment in photographic development; limonite, goethite, hematite used as a pigments and abrasives and magnetite used in production of industrial electrodes. Iron chloride and iron nitrate are used as mordent and industrial reagents in production of several types of inks. Iron carbonyl catalyzes many chemical reactions. Micaceous hematite is used as a protective paint on steel superstructures (Mineral Resource Institute South Australia [M.R.I.S.A.], 2006).

Several studies have shown that vast amounts of iron reserves exist in several locations in Kenya. These areas include Taita, Embu, Lolgarien, Funyula, Macalder, Ikutha, Mugundu and Marimante (Dubois and Walsh, 1971). These deposits have not attracted any commercial interest so far. This could be attributed to the problems of ore beneficiation, location of the ore relative to the market, the cost of building rail infrastructure and energy cost requirements (Sirengo, 2009).

Kenya continues to rely on imported iron for its industrial consumption. Approximately 540,024 tons was imported in 2007. This was worth Kshs.26.5 billion and 472,404 tons in 2008 worth Kshs.35.5 billion (Kenya National Bureau of Statistics [KNBS] 2009). Decreased amount of iron imported could be attributed to increased cost. The amount imported as finished products is not factored in the above estimates. For example, automobiles, if considered the costs would exceed Kshs.60 billion (National Economic Social Council [NESC], 2010). If iron ore resources available in Kenya are exploited, the country would not need to use enormous amount of money importing iron and iron products.

The major iron ores of commercial interest include hematite, magnetite, goethite and ilmenite. It is estimated that world wide there are 800 billion tons of iron ore reserves, containing more than 230 billion tons of iron (Mineral Resource Institute South Australia [M.R.I.S.A.], 2006). It is also estimated that the United States has 110 billion tons of iron ores. Largest iron ore producers include Russia, Brazil, China, Australia, India and the United States. In the year 2006 about 1690 million tons of iron ore were produced (U.S. Geological Survey, 2006). Alternative sources of iron should be explored. laterite which are rich in iron and aluminium and owe their brown colour to iron oxides. Laterite would provide a good alternative to existing iron ores or supplement existing deposits. These are present and are distributed throughout the lowlands of the world (Aleva, 1994).

Iron ores can be exploited economically from ores with 25 percent iron and higher after some ore concentration. It has been shown that laterite found in many parts of Kenya contain between 20 to 45 percent iron (Muriithi, 1985). Ore concentration is carried out before any mineral is processed to make the whole process economical (Snell and Ettre, 1972).

The main methods used to concentrate iron minerals are jigging, magnetic separation and flotation. The method used depends on the mineral being processed, for example the mineral magnetite is readily concentrated using magnetic separation. Hematite is principally concentrated using froth flotation after use of frothing agents. This study is aimed at investigating the possibility of concentrating iron minerals in laterite from selected regions in Ruiru division of Thika District in the Republic of Kenya. This is to be done using affordable methods and locally available technology that would require less energy.

1.2 Statement of the Problem

Laterite (murram) deposits in Kenya are known to contain sufficient iron levels for commercial extraction. Known concentration methods require large per capital input and advanced technology they are thus expensive making iron extraction from the ores uneconomical and unviable. Roasting a mixture of laterite and carbon (charcoal) then using ordinary charcoal as source of energy converts iron minerals present to magnetite. This may provide an alternative route and perhaps affordable that can be used for separation of the iron from the gangue.

1.3 Hypothesis

Laterite which are widely distributed in selected lowland areas of Kenya can be concentrated for commercial exploitation of iron.

1.4 Objectives of the Study

1.4.1 General Objective

To develop a cheap method for concentrating iron minerals in laterite (murram) deposits in selected regions of Ruiru Division in Thika District, Kenya.

1.4.2 Specific Objectives

1. To determine the mineral composition in laterite using X-Ray Diffraction spectrometry (XRD).
2. To determine iron content in laterite using X-ray Fluorescence spectroscopy (XRF), Atomic absorption spectroscopy (AAS) and titrimetric methods.
3. Concentrate iron minerals in laterite using magnetic separation and froth flotation.
4. To mix the ground laterite with carbon (charcoal), heat in the temperature range 300 – 700 ºC, cool then investigate the effectiveness of magnetic concentration and separation of the treated laterite.
5. To determine the amount of iron in the concentrate using XRF, AAS and Titration Methods.

1.5 Research Justification

Geological survey has established that Kenya has many economical iron ore deposits (Dubois and Walsh, 1970). Currently no company is exploiting this iron. Even laterite which have not been known as iron ores contain levels of iron which can be exploited economically. Muriithi (1985), while doing chemical analysis of some samples of black murram and red from various localities in Kenya, observed that their iron concentration varies between 31.96 to 68.0 percent depending on source.

These materials which are used for surfacing roads contains comparable amount of iron to materials used in other countries to produce iron. Currently Kenya doesn’t manufacture any iron but consumption of iron and iron products is high and continues to increase. The present study is aimed at finding out whether iron levels in laterite can be concentrated to levels where iron extraction can be economical. However, concentration methods that are affordable with lower per capita investments are necessary especially in developing countries like Kenya. This will save government expenditure used to import iron and iron products.

Developing countries will continue to rely on developed countries for technological and machinery investments. This can be greatly reduced or reversed if local/ indigenous technology and available methods are explored and viably utilized. The possibility of beneficiating and/or concentrating the available laterite in Kenya may help establish local technology and industries. More so, this will help our country to boost its export levels from the sales of the extracted iron whether in processed or unprocessed form.

In the Vision 2030, the Kenyan Development Blue Print relies heavily on the establishment of local industries and attraction of foreign investors (National Economic and Social Council, 2008). Vision 2030 seeks to transform Kenya into a newly industrialized middle- income country by the year 2030.

1.6 Scope and Limitation of the Study

The study was aimed at collecting samples from selected sites in Ruiru Division in Thika District in the Republic of Kenya. Only iron metal is of major interest in the study.

CHAPTER TWO

LITERATURE REVIEW

2.1 Laterite

Laterite are found in most regions in Kenya (Muriithi, 1985). They are products of intensive and long period of tropical rock weathering. This is accelerated by high rainfall and elevated temperatures. Formation of most laterite started in the Tertiary Period, about 65 million years ago (Aleva, 1994). Laterite formation is controlled by chemical reactions between the rocks and the infiltrated rainfall (Schellmann, 1983). These reactions are controlled by mineral composition and their physical properties (cleavage and porosity) which favour the access of water to the rock. The other factors that favour formation of laterite are the properties of the reacting water which include dissolved constituents, temperature and acidity (pH). These properties are themselves controlled by climate, vegetation and morphology of the landscape (Schellmann, 1994).

Tropical and sub-tropical areas show a rather high annual precipitation but its temporal distribution varies from country to country. Some countries have pronounced long lasting dry spells while others with equatorial climate have continuous precipitation. Chemical weathering slows down in the dry seasons, at least above the fluctuating water table where there is no water. Aqueous dissolution of minerals proceeds when the chemical equilibrium is disturbed. For example, when the dissolved constituents are removed from the water. The chemical reactions are determined by the activity of water which is equal to the one in freely moving water but lowered within small pores in the soil (Aleva, 1994). Stability and reaction rate vary from mineral to mineral, for example, quartz is more stable than feldspar. There are minerals which may have the same chemical composition but belong to different crystal system for example calcite and aragonite. Strong alteration proceeds at the surface of the parent rock whereas it is slower in the regolith (soils or loose rock particles) above the bed rock (Bardossy and Aleva, 1990).

The chemical and mineralogical analysis results have shown that the primary minerals are not fully dissolved in rain water, but partially transformed into secondary minerals which are more stable under intensive weathering conditions. The elements in the primary rock minerals are released and show different reactions in the aqueous solution. Sodium, potassium, magnesium and calcium do not react with other elements and are removed by percolating water. The initial weathering is promoted by the high acidity of water (Aleva, 1994). A high percentage of the dissolved silicon is equally removed by percolating water and the remaining amount reacts with dissolved aluminium to form the clay mineral, kaolinite. The aluminium hydroxide, gibbsite, is formed if the concentration of dissolved silicon is extremely low due to very strong drainage. Iron that is dissolved in water is very reactive with hydroxyl ions to form goethite and hematite after oxidation which cause the red-brown colour of laterite.

The dominant process of laterite formation is the residual enrichment of iron and aluminium by removal of silica, alkalis and alkaline earth metals (Schellmann, 1994). Some of the minerals formed by chemical alterations of primary minerals and form the bulk of laterite are goethite ($FeO.OH$), hematite (Fe_2O_3), kaolinite [$Al_4Si_4O_{10}(OH)_8$] and gibbsites ($Al(OH)_4$), (Aleva, 1994)

Laterites cover has a thickness of a few meters but occasionally can be much thicker. Their formation is favoured by slight relief which prevents erosion of the surface cover. laterite can either be soft and friable or firm and physically resistant. Indurate varieties are sometimes cut into blocks used as brick stones for house building. Hardened laterite varieties are applied for construction of roads as shown in Plate 2.1. Nowadays, solid lateritic gravel is put in aquaria where it favours growth of tropical plants (Bardossy and Aleva, 1990).

Plate 2. 1 Laterite being used in construction of the Thika Super Highway

Laterite are economically important for ore deposits such as bauxite (aluminium-rich laterite variety), lateritic nickel ores, namely, nickel limonite, which contain 1-2% of nickel in goethite and nickel silicate (Golightly, 1981).

2.2 Iron Ores

An ore is a material from which particular element(s) can be extracted economically. Iron ore is a mineral substance which, when heated in the presence of a reductant, will yield metallic iron. The iron ores are mostly dark grey and rusty red in colour. They have a high specific density for example; magnetite has a density of 5.4 g/cm^3. The chief impurities in iron ores are silica, alumina, titanium, sulphur, and phosphorous. Titanium occurs in limonite or rutile. Phosphorous occurs principally as apatite and sulphur as pyrite. Some iron ores contain manganese, chromium and nickel (Kirk - Othmer, 2004).

Iron ores occur in deposits of all geological ages. Most of the world supply is obtained from Precambrian and Jurassic rocks (Snell and Ettre, 1972). Principal types of deposits yielding iron ores are bedded ores, igneous segregation, contact metamorphic deposits, vein deposits and superficial residues. The bedded ores are the most important and constitute a large proportion of world iron ore reserves (Luttrell, 1975). Chief iron bearing minerals are given in Table 2.1

Table 2. 1.Iron Bearing Minerals **(**Snell and Ettre, 1972).

Class	**Mineralogical name**	**Chemical formula**	**Common designation**
Oxides	Magnetite Hematite Ilmenite Limonite (goethite)	Fe_3O_4 Fe_2O_3 $FeTiO_3$ $HFeO_2$	Ferrous-Ferric oxide Ferric oxide Iron-Titanium oxide Hydrous Iron oxide
Carbonates	Siderites	$FeCO_3$	Iron carbonate
Silicate	Chamosite Silomelane Greenalite Minesotaite Gruenerite	All are often complex	Iron silicates
Sulphides	Pyrite Marcasite Pyrrhotite	FeS_2 FeS_2 FeS	Iron sulphides

Iron rich rocks are common worldwide, but ore grade for commercial mining operations are dominated by the countries shown in the Table 2.2. The table also contains their production in millions of metric tons.

Table 2. 2 World iron ore production (source (U.S. Geological survey, 2006).

Country	**Production (Millions of metric tonnes)**
China	520
Brazil	300
Australia	270
India	150
Russia	105
Ukraine	73
United States	54
South Africa	40
Canada	33
Sweden	24
Venezuela	20
Iran	20
Kazakhstan	15
Mauritania	11
Others	43
Total	**1690**

2.3 Beneficiation of Iron Ores

Very low grade iron ore (with less than 60 percent iron) cannot be used in metallurgical plants and needs to be upgraded to increase the iron content and reduce the gangue content (Sharma, 2004). Beneficiation is a general term for the processes used to upgrade iron ores. The ore is upgraded to higher iron content through concentration. Iron ores are beneficiated to meet the quality required for iron and steel industries (Kirk - Othmer, 2004). However, each source of the ore has its own peculiar mineralogical characteristics and requires specific beneficiation and metallurgical treatment. The choice of the beneficiation treatment depends on the nature of the gangue present and its association with the iron ore. Several techniques are used. They include washing, jigging, magnetic separation, flotation and advanced gravity separation (Sharma, 2004). Only a moderate proportion of iron ore, as mined, is suitable for direct reduction in the blast furnace. This is due to the presence of impurities which make the mining process expensive. A lot of energy is consumed and output of pig iron reduced (Roe, 1957). Figure 2.1 shows a general beneficiation process of iron ore.

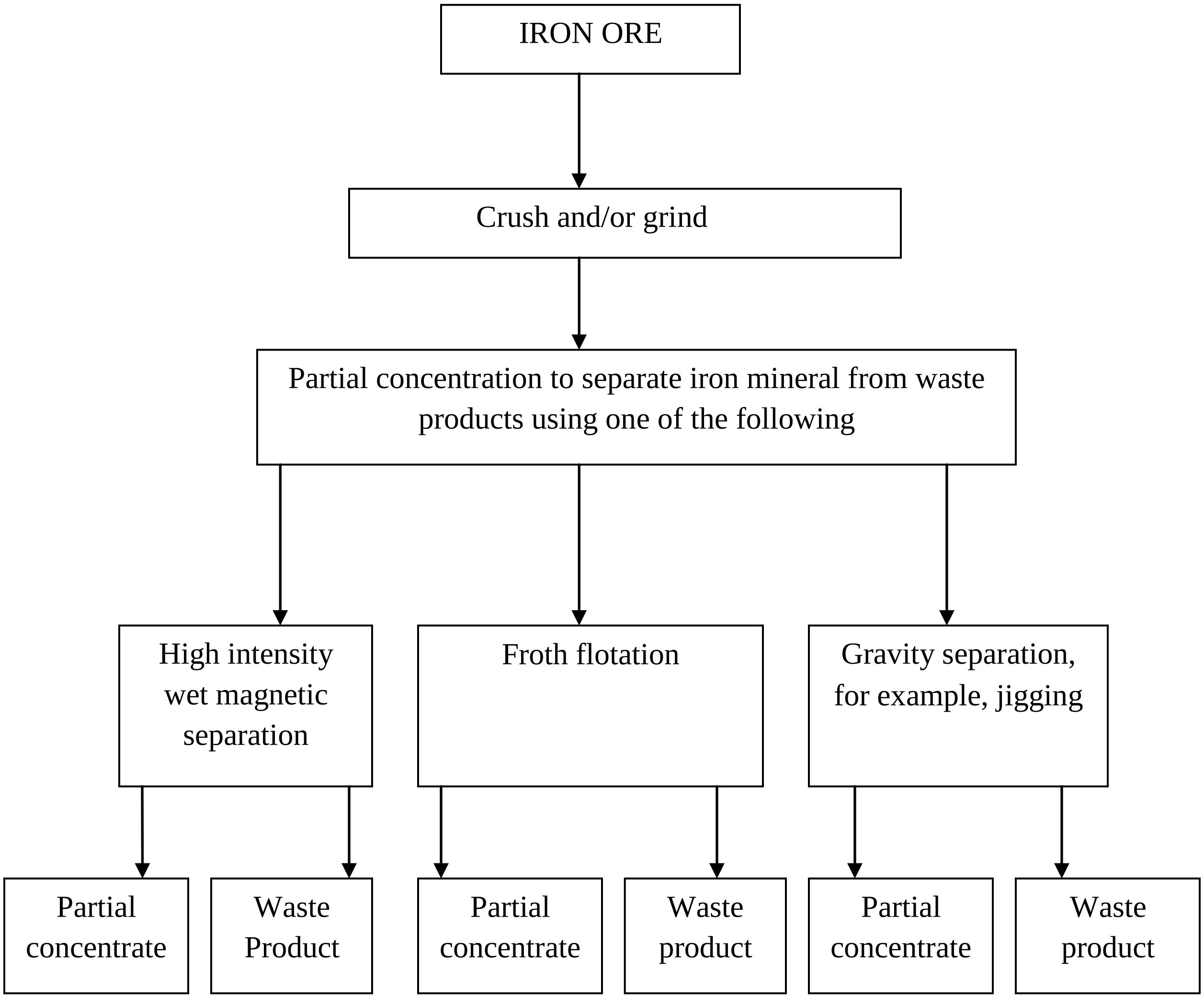

Figure 2. 1 Beneficiation process (Lawver , 1976).

2.3.1 Jigging

Jigging is hindered settling consisting of stratification of particles into layers of different densities. This is caused by repeated upward and downward current of fluid to a thick suspension of mixed particles to settle or fall for short period of time (Jain, 2001). It is one of the oldest methods of gravity concentration. It is used to concentrate relatively coarse material. When the feed is fairly close sized, it is easy to achieve a good separation of fairly narrow range in specific gravity of minerals. When specific gravity difference is large, good concentration is possible with a wider size range (Prackash *et al.*, 2007). The separation of minerals of different specific gravities is done in a designed bed. The bed is rendered fluid by a pulsating current of water to produce stratification. The aim is to dilate the bed of material being treated and control the dilation so that heavier, smaller particles fall under conditions similar to hindered settling (Lymann, 1992; Olubambi and Potgietez, 2005).

In a single movement in series (pulsion stroke), the bed is normally lifted as a mass. As the velocity decreases it tends to dilate. The bottom particles falling first until the whole bed is loosened. On suction stroke, it closes slowly. This is repeated in a number of strokes. The frequency of pulse ranges from 55 – 330 counts per minute. Fine particles pass through the interstices while large ones are immobilized. The motion can be obtained by using a fixed sieve jig, pulsating water or employing a moving sieve as done in simple hand-jig (Barry, 1997).

The size of particles is very important in jigging action. The highest recovery of iron ore is achieved on 600 µm size (Barry, 1997). Lower separation efficiency in fine particles is believed to be caused by the negligible mass associated with these size particles (Barry, 1997). Lower recovery of iron at large particle size is due to lower possibility of large particles passing through the jig screen. A coarse particle will have lower chances of passing through the jig screen and is lost as overflow in the tailing (Mohanty and Ram, 2002).

2.3.2 Froth Flotation

Froth flotation is a highly versatile method for physically separating particles. It is based on the ability of the air bubbles to selectively adhere to specific mineral surfaces in mineral/water slurry (Barry, 1997). The particles with attached air bubbles are then carried to the surface and removed. Particles that do not have air bubbles are dense and sink. Froth flotation can be adapted to a broad range of mineral separation. This is possible because chemical treatment selectively alters mineral surfaces so that they have the necessary properties for the separation (Cruizer, 1984; Nagaraj, 1994; Ranny, 1980). It is currently in use for diverse applications which include, separation of sulphides from silica gangue, separating potassium chloride (sylvite) from sodium chloride (halite), separating coal from ash-forming minerals, separating silicate minerals from iron ores and separating phosphate minerals from silicates. It is particularly useful for concentrating fine grained ores that are not amenable to conventional gravity concentration (Kawatra and Eisele, 1987).

The basis of froth flotation is the difference in wet-abilities of different minerals. Particles range from those that are easily wet-able by water (hydrophilic) to those that are water repellent (hydrophobic). If a mixture of hydrophobic and hydrophilic particles are suspended in water and air is bubbled through the suspension, air bubbles will attach to the hydrophobic particles and float to the surface as illustrated in Figure 2.2.

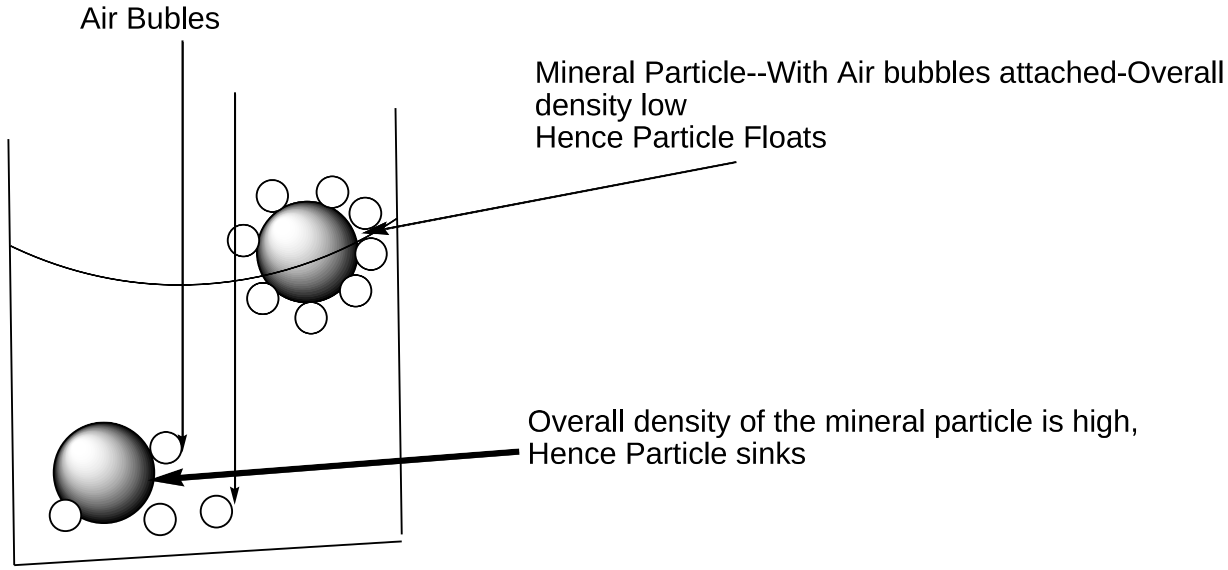

Figure 2. 2 Air bubble attached to mineral particles

The froth layer that forms on the surface consists of hydrophobic minerals and can be removed as the concentrate. Hydrophilic particles are left in solution as tailing. Particles can either be naturally hydrophobic or hydrophobicity can be induced by chemical treatment (Kawatra and Eisele, 1987). These chemicals selectively coat mineral surfaces with monolayer of non- polar oils known as flotation reagents (Nagaraj, 1994).

The attachment of the bubbles to the surface is determined by the interfacial energies between the solid, liquid and gases. This is determined by the Young/ Dupre equation shown in Equation 2.1 (Kawatra and Eisele, 1992)

$$\gamma_{lv}\cos\theta = (\gamma_{sv} - \gamma_{sl}) \text{ -- } 2.1$$

Where

γ_{lv} = Surface energy of the the liquid \ vapour interface

γ_{sv} = Surface energy of the solid \ vapour interface

γ_{sl} = Surface energy of the solid \ Liquid interface

θ = Contact angle

The junction formed between vapour, solid and liquid phases is shown in Figure 2.3

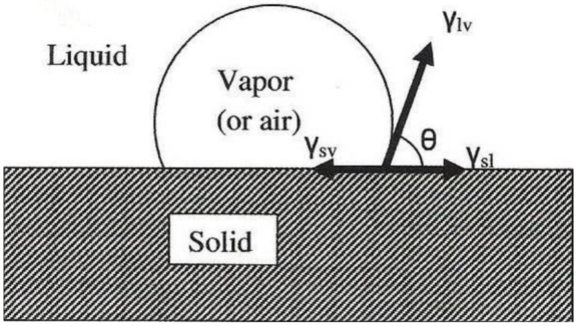

Figure 2. 3 Contact angle between mineral particle and air bubble(Kawatra and Eisele, 1992)

If the contact angle is very small, then the bubble does not attach to the surface, while a very large contact angle result in a very strong bubble attachment. Minerals with a high contact angle are said to be aerophilic. A contact angle near 90 ^{0} is effective for froth flotation (Sivakumar and Somasundaran, 1988).

Once the particles are rendered hydrophobic, they must be brought into contact with gas bubbles so that gas can attach to the surface. If the bubble and mineral surfaces do not come into contact, then no flotation occurs. Contact between particle and bubble can be accomplished in a flotation cell as the one shown in Figure 2.4(Kawatra and Eisele, 1992)

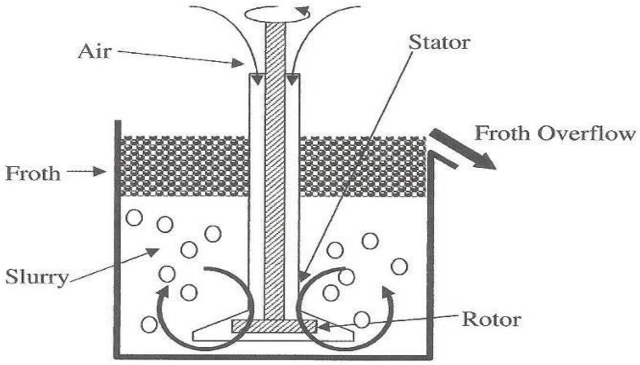

Figure 2.4 A Flotation cell

The rotor draws slurry through the stator and expels it to the sides, creating a suction that draws air down the shaft of the stator. The air is then dispersed as air bubbles through the slurry and comes in contact with particles in the slurry that is drawn through the stator (Kawatra and Eisele, 1992)

When sufficient numbers of bubbles attach themselves to the mineral particle, they lower its density and the mineral floats. The particle and the bubbles must remain attached when they move up the froth layer at the top of the cell. The froth layer must persist long enough to either flow over the discharge lip of the cell by gravity or to be removed by mechanical froth scrappers. If the froth is insufficiently stable, the bubbles will break and drop the hydrophobic particle back into the slurry prematurely (Ahmed and Jameson, 1989*)*.

The surface area of the bubbles in the froth is also important. Since the particles are carried into the froth by attachment to bubble surfaces, increasing the amount of bubble surface area allows more rapid flotation rate of particles. At the same time, increased surface area also carries more water into the froth as a film between the bubbles. Excessive amount of water in the froth can result in significant contamination of the product with gangue minerals (Kawatra and Eisele, 1992)

There are minerals which do not attract air bubbles as naturally mined. However when treated with certain chemicals called collectors, they acquire this property. There are many types of collectors as shown in Figure 2.5. Collectors can either be anionic or cationic (Barry, 1997).

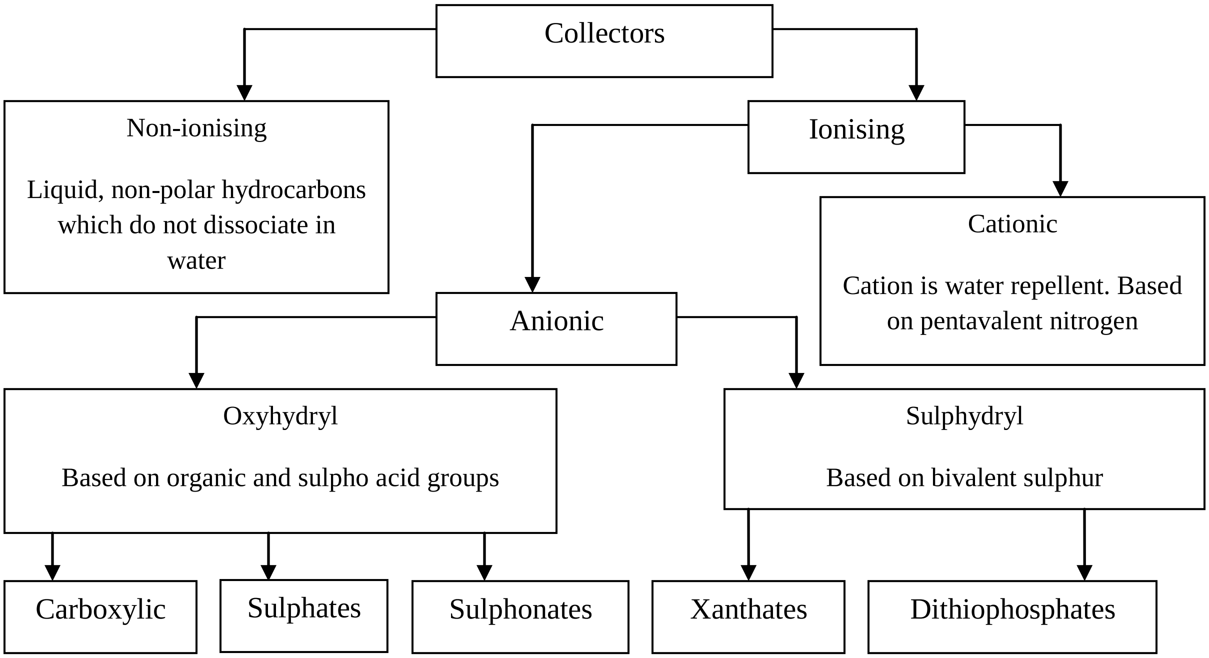

Figure 2. 5 Types of collectors (Barry, 1992)

Frothers, stabilize air bubbles so that they remain well dispersed in the slurry. They include methyl isobutyl carbinor (MIBC), pine oil and cresylic acid (Klimpel, 1995). Modifiers, influence the way the collectors attach to the mineral surface. They either increase the adsorption of the collector on a given mineral (activators) or prevent collection from adsorbing onto a mineral (depressants). Activators enhance the ability of the collector to adsorb onto surfaces they could not normally attach to. An example of an activator is copper sulphate which acts as an activator for sphalerite (ZnS) flotation with xanthate collectors (Fuerstenau *et al.*, 1985). Depressants prevent collectors from adsorbing onto particular mineral surfaces. Their typical use is to increase selectivity by preventing one mineral from floating while allowing another mineral to float.

2.3.3 Magnetic Separation

Magnetic separation exploits the difference in magnetic properties of the ore minerals. It is used to separate valuable minerals from non-magnetic gangue. Magnetite, for example, is separated using this technique from quartz (Svoboda, 1994). All materials are affected by magnetic field but to different extents. Those materials with unpaired electrons are attracted more strongly than with no unpaired electrons. The materials goethite, hematite and magnetite fall in this category with magnetite being several times more imperative than the others. An ore with magnetite can therefore be readily concentrated using a strong magnet. This study describes a technique developed to convert goethite or hematite to magnetite.

The unit of measurement of magnetic flux density or magnetic induction (B), which is the number of lines of force passing through a unit area of material, is tesla (T). The magnetising force, which induces the line of force through a material, is called the field intensity (H). The intensity of magnetism or the magnetisation (M) of a material relates to the magnetisation induced in the material as shown in Equation 2.2 (Svoboda, 1987)

$$B = \mu_0 (H + M) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (2.2)$$

Where μ_0 = Permeability of free space

In a vacuum, M = 0 and it is extremely low in air, therefore Equation 2.2 reduces to Equation 2.3

$$B = \mu_0 H \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (2.3)$$

The capacity of a magnet to lift a particle is not only dependent on field intensity value but also field gradient. Paramagnetic minerals have higher magnetic permeability than surrounding medium. They concentrate the line of force of an external magnetic field. The higher the magnetic susceptibility, the higher the field intensity in the particle. Diamagnetic minerals have lower magnetic susceptibility than their surrounding medium and hence expel the lines of force of the external magnetic field (Cohen, 1986). To generate a given lifting force, there are an infinite number of combinations of field and gradient which will give the same effect. Production of high field gradient as well as high intensity is therefore an important aspect of separator design. Types of magnetic separators include low-intensity magnetic separators which are used to separate coarse particles (up to 100 mm in size). They are dusty, expensive and have low capacity (Wasmuth and Unkelbach, 1991). High-intensity separators are used to concentrate very weakly paramagnetic minerals and high-tension separators which utilise the difference in electrical conductivity between various minerals in the ore feed (Svabodo, 1987).

Haematite and goethite minerals belong to a group of hard magnetic materials which need high magnetic field in order to reach magnetic saturation (Maher *et al*., 2004). They can be converted to magnetite through heating and reduction. This is done by roasting at 500 – 550 °C. Siderite can be converted to magnetite by roasting in atmosphere at 700 – 775 °C. In both cases, ores must be cooled in air-free atmosphere to 100 °C. The roasted ore is easier to crush and grind than unroasted ore. It can also be concentrated by low magnetic separators (Kirk - Othmer, 2004).

2.4 Analytical Techniques

Four analytical techniques were used in the present study. These are X-ray diffraction spectrometry (XRD), X-ray fluorescence spectroscopy (XRFS), Atomic Absorption Spectroscopy (AAS) and Ethylenediamine-tetra-acetic acid (EDTA) titration.

2.4.1 X-Ray Diffraction Spectrometry (XRD)

X-ray diffraction spectrometry (XRD) is a rapid analytical technique used for phase identification of minerals. X- ray diffraction spectrometry has a direct application in determination of minerals, which are, by nature, naturally occurring crystalline substances whose constituent particles are inorganic in nature. The interaction of the incident ray with the sample produces constructive interference (and diffraction) when conditions satisfy Bragg's law shown in Equation 2.6 and Figure 2.6

$$n\lambda = 2d\sin\theta \qquad \text{………………………………………………………} \quad (2.6)$$

Where, n – is an integer,

λ – The wavelength of incident ray

d – The spacing between the planes in the atomic lattice

θ – The angle between the incident ray and scattering plane.

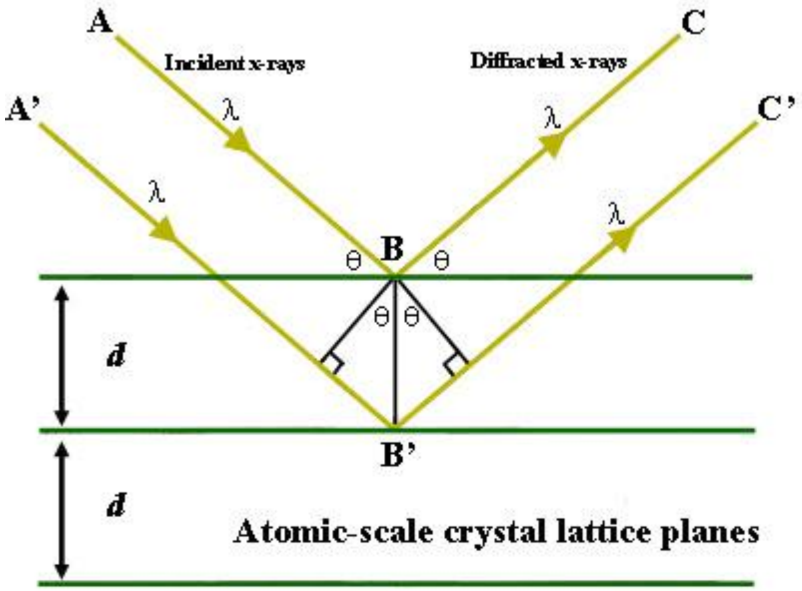

Figure 2. 6 Bragg's law reflection (Eby, 2004)

The Bragg's law relates the wavelength of electromagnetic radiation to the diffraction angle and the lattice spacing in a crystalline sample. The diffracted x-rays are detected, processed and counted. Conversions of the diffraction peaks to d-spacing allow the identification of the mineral because each mineral has a set of unique d-spacings. This is achieved by comparing the d-spacing with standard reference patterns (Moore *et al.*, 1997).

2.4.2 X-Ray Fluorescence Spectroscopy (XRFS)

XRFS involves secondary X-rays. Here, primary X-rays actually knock off electrons from the K and L shells of atom when a chemical element is subjected to a beam of X-rays. The wavelengths and intensity of emissions measurement are used for qualitative and quantitative analysis respectively (Fified and Kealy, 1995). The primary X-rays are directed onto a secondary target, where a proportion of the incident ray is absorbed. This absorption leads to ejection of inner K and L electrons from the atom of the sample. Subsequently, the excited atom relaxes to ground state. During relaxation, many excited atoms will lose their excess energy in form of secondary X-ray photons as electrons from high energy orbital's drop into 'holes' in the K and L shells (Skoog and Leavy, 1992). For reasons of sensitivity and versatility, the combination of high powered sealed X-ray tube and wavelength dispersion, selected crystals remain the most practical and preferred technique for quantitative X-ray fluorescence analysis (Schenk *et al*., 1981).

2.4.3 Atomic Absorption Spectroscopy (AAS)

Atomic absorption is a physical process that involves the absorption (by free ions of an element) of light at a wavelength specific to that element. This causes an atom or ion to move from one steady state to a higher energy state (Van loon, 1980).

If these states are O for the ground state and P for excited state with energies E_0 and E_P, where $E_P > E_0$. then the transition $0 \rightarrow P$ results in absorption of energy and $P \rightarrow O$ results in emission of energy. The $0 \rightarrow P$ absorption transition is always stimulated by external radiation. This phenomenon forms the integral part of Atomic Absorption Spectroscopy (McLerman and Kolwasiki, 1995). For an unexcited atom, each electron is in the ground state, otherwise it is excited. The proportion of excited to ground state atoms in a population at a given temperature, is given by the general statement of the Maxwell-Boltzmann law shown in Equation 2.8

$$\frac{N_p}{N_o} = \frac{g_v}{g_o} exp\left[E_o\_E_p / KT \right] \ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{(2.8)}$$

Where, N – the number of atoms in state 0 and P,

g –the statistical weights for states 0 and P,

T –the temperature,

K – The Boltzmann constant,

E – Energies.

The wavelength at which an atom with its valence electrons in the ground state can absorb radiation is called resonance wavelength (Skoog and Leavy., 1992).

To calculate how much light is absorbed by a cloud of atoms, parallel beams of light at the resonance wavelength for the atoms concerned are considered when striking a cell containing N atoms. If light of intensity I_0 enters the cell, the intensity remaining after absorption I_1 is given by Equation 2.9.

$$I_1 = I_o exp[kL] \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{(2.9)}$$

Where L is the cell length and k is the absorption coefficient (i.e. the fraction of energy absorbed per unit length).Taking logarithms of both sides, we get Equation 2.10

$$kL = \log\frac{I_0}{I_1} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{(2.10)}$$

The expression log I_0/I_1 is defined as absorbance.

Since the product kL is proportional to the number of atoms in the cell, so is the absorbance. For this reason, absorption is the preferred readout mode of modern atomic absorption spectrometer, giving a linear relationship between absorbance and concentration. This principal has been used for quantitative determination of various elements in the minerals.

2.4.4 Ethylenediamine-tetra-acetic Acid Titrations (EDTA)

The EDTA anion (Y^{4-}) has the widest general application in analysis because of the spatial structure of its anion which has six ligand atoms, thus corresponding with the co-ordination number most frequently encountered among the metals. In addition, it forms strain-free five-member rings on chelation (Vogel, 2004), as shown in Figure 2.7.

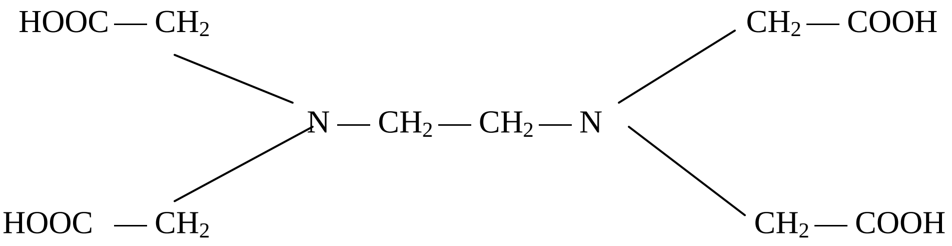

Figure 2. 7 Structure of EDTA

The efficiency of metal-EDTA complex formation is determined by the concentration of the Y^{4-} ion, the pH of the solution, the solubility of the metal hydroxide and its solubility constant. Thus, the titration of each metal with EDTA is governed by the pH in which its complex is most stable. Hence, due to its great versatility arising from its inherent potency as a complexion agent and from the availability of the numerous metal-ion indicators, with each indicator having a range of pH values for different elements, EDTA is the most useful titrant in analysis of metals (Hesse, 1972). It has the advantage that it is cheap and hence, can be used to give scientifically very important data even when more expensive equipment such as XRF or AAS is not available.

CHAPTER THREE

MATERIALS AND METHODS

3.1 Sample Collection

Sampling was done from selected sites of Ruiru division in Thika district. These sites were Mchana, Tassia, Mugutha, Miti Kenda, Kwihota, Theta, Ruiru Prison, Githunguri Ranching, Githurai Kimbo, Mwihoko, Kahawa Sukari, Murera Coffee, Gatong'ora and Tatu. The area covered is in the attached map of Thika District in the Republic of Kenya. Within a given site, samples were collected as follows; the surface materials up to 20cm deep were removed because they contained a lot of organic matter. Samples picked from this depth were labelled level O. A hole was dug to a depth of one meter and samples collected labelled level 1. Where laterite (murram) is being harvested, samples were picked from a depth of about 40cm and another at a depth of one meter then labelled level 0 and 1 respectively. A total of three samples were collected from each depth and site.

3.2 Apparatus

All glassware used was cleaned by soaking it in 1:1 nitric acid overnight. It was then cleaned using detergent and rinsed with distilled water. They were dried in an oven at 105 °C and stored in desiccators. Platinum crucibles were cleaned by heating $KHSO_4$ in them for two hours in a furnace. They were then cleaned using appropriate detergent, rinsed with distilled water and dried at 105 °C in an oven. Plastic containers were washed with

1:1 nitric acid, followed by appropriate detergent and rinsed with distilled water. They were dried in an oven at 50 °C.

3.3 Chemical Reagents

Analytical grade reagents were used. They were all prepared using standard methods as highlighted in Vogel's text book of quantitative inorganic analysis (Vogel, 2004). Distilled water was used throughout the procedures. Chemicals were prepared as follows; Aqua- regia – 375 ml of concentrated hydrochloric acid was put in a 500 ml volumetric flask. Concentrated nitric acid was added up to the mark (ratio of HCl to HNO_3 is 3:1). The solution was then transferred into a plastic container. Ethylenediamine-tetra acetic anion (EDTA) was prepared by dissolving 37.29 grams of di-sodium ethylenediamine-tetra acetic dehydrate in 500 ml of water in a 1000 ml volumetric flask then diluted to the mark. Conditioning reagent was prepared by dissolving 3.04g of iron (II) sulphate in a 500 ml of distilled water and making it to a litre. Sodium silicate was prepared by dissolving 6.1 g in 500 ml of water then made to a litre. 100 ml of iron (II) sulphate prepared before was slowly added to 100 ml of sodium silicate to form a suspension. Boric acid 320.0 g were dissolved in 2.5 litres of water, the solution was warmed, and then about 9.0 g of CsCl added. The mixture was allowed to cool to room temperature.

4 Instruments and their settings

The following instruments were used in this study.

3.4.1 pH Meter

All pH measurements were done using a microprocessor pH meter, model 211, from Hannah Instruments. The meter was calibrated using pH buffer Tablets of pH 4 and 7.

3.4.2 Weighing Balance

All weighing of small quantities but not exceeding 160 g were done using analytical balance model Mettler AJ150 model GWB. Large quantities exceeding 160g was done with electronic balance, type UX6200H from Shimadzu Corporation.

3.4.3 Calibration of Instruments

The AAS was calibrated using SY-3 rock Standards for all analysis. The SY-3 rocks standards were obtained from Canadian Centre for Mineral Technology (CCMT) through the Department of Mines and Geology, Ministry of Environment and Mineral Resources of Kenya. The instrumental condition for each parameter was set as indicate in Table 3.1

Table 3. 1Instrumental condition for AAS analysis (CCMET, 1991).

Element	**Lamp current (Amp)**	**Slit width (nm)**	**Wavelength (nm)**	**Acetylene flow**	**Photomultiplier voltage (V)**	**Flame**
Si	7	0.2	251.6	4.5	359.3	N_2O-acetylene
Al	6	0.5	309.3	4.5	287.4	N_2O-acetylene
Fe	8	0.2	248.3	1.5	340.5	Air-acetylene
Mg	3	0.5	285.2	1.5	318.6	Air-acetylene
Ca	None	0.5	422.7	4.5	231.1	N_2O-acetylene
Na	none	0.5	589.0	1.5	365.5	Air-acetylene
K	None	1.0	766.5	1.5	353.5	Air-acetylene
Mn	5	0.2	279.5	1.5	349.9	Air-acetylene
Ti	10	0.5	364.3	4.5	303.0	N_2O-acetylene

The XRFS Minipal Software was used with internal standards. These internal standards were prepared with standard reference material. This standard reference material is manufactured by the U.S. Geological Survey Geochemical reference materials and certificate.

For XRD internal standards were used with Pro Analytical X’pert high score plus software. The internal standards are from International Centre for diffraction Data,. X’pert Pro model that is supplied by Pan Analytical Company limited was used.

3.5 Sample Pre-treatment and Analysis Procedures

Laterite samples were transferred from plastic bags to paper bags, dried in an oven at 105 °C for 8 hours and then allowed to cool at room temperature in desiccators. They were then pulverized to 300 microns (150 meshes). The major minerals in the laterite were determined using X-ray diffractometric technology. The instrument was connected to computer software which gave direct reading of the minerals present. Chemical composition was quantified using XRF, AAS and EDTA titration for various oxide compositions.

3.5.1 X-Ray Diffraction (XRD) Analysis

Pulverised laterite samples were put in sample holders. They were loaded for analysis of minerals using data collector software. Results of analysis of minerals present in each sample were given using X’pert High Score Plus software. The examples of output are shown in Appendices 7 and 8

3.5.2 X-Ray Fluorescence Spectrometer (XRFS) Analysis

About 10.0 g of pulverized sample was weighed; about 5.0 g of flux starch added and the mixture mixed in a motor and pestle. The resulting mixture was made into pellets using Herzog hydrolyric jack pelleting machine with a minimum load capacity of 170 Kilo Newtons (kN). The pellets were loaded into sample holder cups. Sequential X-ray fluorescence analysis was done using Minipal-2 version 4 Panalytical Model. The results were recorded in terms of the oxides of the elements as shown in Appendix 9.

3.5.3 Atomic Absorption Spectroscopy (AAS)

Pulverized laterite samples weighing about 0.1 g were put in a plastic bottle. About 1 ml of aqua-regia (mixture of concentrated HCl and HNO_3 in the ratio 3:1) was added. This was followed by 3.0 ml of hydrofluoric acid. The container was stoppered and the mixture allowed to digest overnight. About 50.0 ml boric acid was added and allowed to stand for 1 hour. About 46.0 ml of distilled water was added to make the total mixture 100.0 ml. SY-3 rock standards were also digested following the same procedure used to digest the samples. Dilutions of the samples were made by putting 5.0 ml in 100 ml volumetric flask. The solution was made up to the mark using 1:1 boric acid solution. The samples and the standards were analysed using AAS instrument (Varian AA.10 model).

3.5.4 EDTA Titration

About 1.0 g of the ground laterite was fused with 10.0 g of sodium carbonate in a platinum crucible at 1000 °C in a furnace for 60 minutes. After allowing the crucible to cool, it was put in a 100 ml beaker and submerged in distilled water. About 1 ml concentrated HNO_3 and 1 ml concentrated HCl were added. The mixture was heated on a hot plate until the solid residue content of the crucible disintegrated. The mixture was filtered through Whatman filter paper number 541 into a 250 ml volumetric flask. The residue was washed three times with hot distilled water at 60 ^{o}C. The filtrate was topped to the mark with distilled water and kept for further analysis. Aliquot portion of 25.0ml were measured using a 25.0 ml bulb pipette into 250- ml beaker. The pH was adjusted to between 2 and 3 using 1:1 hydrochloric acid. On addition of about 0.1 g crystals of potassium thiocyanate indicator, the colour changed to red. This solution was titrated with 0.1M EDTA solution. The titration was done in triplicate. The average titre was used to calculate the amount of iron using Equations 3.2

$$M_{l\,EDTA} X M_{EDTA} X Atomic\ Weight\ of\ Fe = mg\ of\ Fe \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3.1)$$

Where $M_{l=}\ Volume\ of\ EDTA$

$M_{EDTA} = Molarity\ of\ EDTA$

A.W=55.847 for iron

3.6 Concentration of Iron in Laterite

3.6.1 Heating a Mixture of Laterite and Carbon (Charcoal).

About 20.0 g of laterite sample were weighed and put in a fire clay crucible. Each sample was mixed with 3.0 g of powdered charcoal. Ordinary charcoal was put in a charcoal burner shown in Plate 3.1

Plate 3. 1 Charcoal burner.

The adjustable top was lowered to the lowest level. Fire clay crucibles containing the mixture of laterite and charcoal were heated in open air for 4 hours. The fire was monitored by looking at the amount of charcoal consumed. Charcoal was added into the burner every time they reduced. After four hours, the roasted samples were allowed to cool to room temperature. The sample from Tassia site was weighed twice. One sample was mixed with carbon (charcoal) then heated and the other was heated without mixing with carbon to act as a control. Cooled samples from Tassia were analysed using XRD to get the difference in their mineral contents. Heated samples were concentrated using magnetic separators shown as Plate 3.2

Plate 3. 2 Laboratory Magnetic Separator at the lapidary Laboratory Mines and Geology Department of Ministry of Environment and Mineral Resources.

Mass of the concentrate from each sample was determined. The percent of laterite picked by magnet was calculated as shown in Equation 3.2

$$\frac{Mass\ of\ laterite\ picked\ by\ magnet}{Original\ mass} \times 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \; (3.2)$$

Both the concentrate and the tailing were analysed using XRFS, AAS and EDTA titrations as detailed in Section 3.6.

3.6.2 Froth Flotation

About 500.0 g of ground laterite were weighed and put in a 1000 ml beaker. About 500.0 ml distilled water were added to make 1:1 slurry. The mixture was put in a flotation cell shown in Plate 3.3 and agitated for 5.0 minutes to make the slurry. About 5.0 ml of conditioning reagent was added. The mixture was agitated for 5.0 minutes after adding conditioning reagent. More water was added to the mixture to make slurry with about 30% solid. The pH of slurry was adjusted to between 8 and 9 using sodium hydroxide and 1:1 hydrochloric acid solutions. About 30.0 ml of oleic acid was added and the mixture agitated for 10.0 minutes. About 3.0 ml of cresylic acid was added and mixture agitated for 3.0 minutes. Air was bubbled through and froth collected in plastic containers. Flotation was done for 10.0 minutes.

Plate 3. 3 Laboratory flotation equipment

Both the froth and tailings were separately filtered using vacuum filtration as shown in figure 3.1. A Buchner funnel was placed on the heavy side arm filtering flask. A rubber adaptor was used to seal flask when under vacuum. A filter paper was placed in a Buchner funnel, wetted with a little water and then mixture was poured on. The vacuum source was then turned on (Paria, 2005).

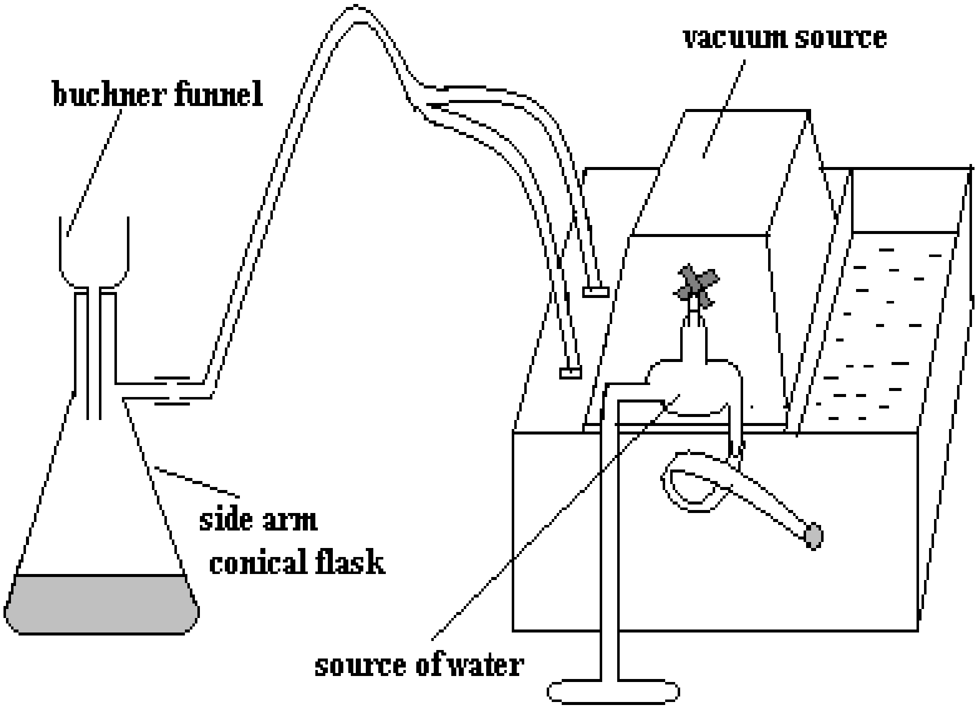

Figure 3. 1 Vacuum filtration

The solid residue on the filter paper was rinsed with 50.0 ml of a mixture of water and diethyl ether in the ratio 1:1 and finally twice with water. Diethyl ether was used to dissolve oleic acid and cresylic acid in order to recover them. The filter paper and the residue were carefully removed from the Buchner funnel and dried in an oven at 105 ^{o}C for 6.0 hours. The dry residue was then pulverised to 300 microns for analysis using the XRFS, AAS and EDTA titration as explained in Section 3.6.

3.8 Data Analysis

The results were obtained in triplicate then averages and the means calculated. Graphical representation of the data was done using statistical programme for social scientists (SPSS). This included results for AAS, XRFS and EDTA titration analysis. Significance tests for the analytical methods were done using t-test and one way analysis of variance (ANOVA) [Miller and Miller, 1988]. Standard errors were calculated using Equation 3.3

$$S.E = \frac{S.d}{\sqrt{n}} \quad \text{...} \ (3.3)$$

Where

S.E - Standard error

S.d - Standard deviation

n - Population

While comparing means using significance test (t-test), of methods of analysis and methods of beneficiation, the assumption that the population standard deviation is the same were proved not true. This was done using F-test two tailed (Miller and Miller, 1988) as in Equation 3.4

$$F = \frac{S_1^2}{S_2^2} \quad \text{...} \ (3.4)$$

Where

S^2 = Variance of population in set 1 and 2

T- Calculated was obtained using equation 3.5

$$t = (\bar{x}_1 - \bar{x}_2)/\sqrt{(s_1^2/n_1 + s_2^2/n_2)}$$ …………………….. (3.5)

The degree of freedom was calculated from the equation 3.6 (Miller and Miller, 1988)

$$\text{degrees of freedom} = \left\{ \frac{(s_1^2/n_1 + s_2^2/n_2)^2}{\dfrac{(s_1^2/n_1)^2}{n_1 + 1} + \dfrac{(s_2^2/n_2)^2}{n_2 + 1}} \right\} - 2$$ ……………… (3.6)

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Mineral and Chemical Composition of Raw laterite

Laterite samples from identified sampling sites in Ruiru Division of Thika District (Appendix 1) namely Mchana, Mugutha, Miti Kenda, Tatu, Kwihota, Tassia, Ruiru Prison, Githurai Kimbo, Gatong'ora, Githunguri ranching, Mwihoko, Kahawa Sukari, Murera coffee and Theta were analyzed for their mineral content using X-ray diffraction technique. The results are given in Table 4.1.

Table 4. 1 Mineral Content of Raw laterite from Selected Sites in Ruiru Division, Thika District.

Sampling Site	Compound name	Chemical Formula
Mchana	Goethite Quartz Aluminium phosphate (V)	$Fe_2O_3.H_2O$ SiO_2 $AlPO_4$
Mugutha	Quartz Aluminium phosphate(V) Goethite Goethite, Syn	SiO_2 $AlPO_4$ $Fe_2O_3.H_2O$ FeO (OH)
Miti Kenda	Quartz Goethite, Syn Rutile HP Aluminium phosphate(V)	SiO_2 FeO (OH) TiO_2 $AlPO_4$
Tatu	Quartz Berlinite	SiO_2 $AlPO_4$

	Rutile HP Goethite Groutellite	TiO_2 $Fe_2O_3.H_2O$ MnO_2
Kwihota	Quartz Aluminium phosphate(V) Goethite Goethite, Syn	SiO_2 $AlPO_4$ $Fe_2O_3.H_2O$ FeO (OH)
Theta	Quartz Berlinite Rostile Goethite	SiO_2 $AlPO_4$ (Al,Fe)(SO4)(F,OH). 5H2O $Fe_2O_3.H_2O$
Tassia	Quartz Aluminium phosphate(V) Goethite Goethite, Syn	SiO_2 $AlPO_4$ $Fe_2O_3.H_2O$ FeO (OH)
Ruiru Prisons	Quartz Aluminium phosphate(V) Goethite Groutellite	SiO_2 $AlPO_4$ $Fe_2O_3.H_2O$ MnO_2
Githurai Kimbo	Goethite Quartz Unnamed Mesoporous Groutellite	$Fe_2O_3.H_2O$ SiO_2 $Si_{11.4}TiO_{24.8}$ MnO_2

Gatong'ora	Quartz Berlinite Rostile Goethite	SiO_2 $AlPO_4$ (Al,Fe)(SO4)(F,OH). 5H2O $Fe_2O_3.H_2O$
Murera Coffee	Quartz Berlinite Rutile HP Goethite Brounite-IQ	SiO_2 $AlPO_4$ TiO_2 $Fe_2O_3.H_2O$ $(Mn_2O_3)3Mn\ SiO_3$
Githunguri Ranching	Quartz Berlinite Rutile HP Goethite	SiO_2 $AlPO_4$ TiO_2 $Fe_2O_3.H_2O$
Kahawa Sukari	Quartz Berlinite Rostile Goethite	SiO_2 $AlPO_4$ (Al,Fe)(SO4)(F,OH). 5H2O $Fe_2O_3.H_2O$
Mwihoko	Quartz Aluminium phosphate(V) Goethite Goethite, Syn	SiO_2 $AlPO_4$ $Fe_2O_3.H_2O$ FeO (OH)

The results show that the iron mineral present in laterite is Goethite which corresponds to the formula, $Fe_2O_3 \cdot H_2O$, and FeO (OH). The other minerals present include quatz (SiO_2), aluminium phosphate (V) ($AlPO_4$). Groutellite, syn (MnO_2) and rutile HP (TiO_2) are also present in a few areas. Aleva (1994) and Schellmann (1983) observed that laterite in the world contain heavily altered materials, rich in secondary iron or alumina oxides or both. They consist of minerals goethite, hematite, kaolinite and gibbsite.

Results of mean chemical analysis and statistical analysis of the two analytical methods used, AAS and XRF, are given in Table 4.2 for level O. Table 4.3 represent result for chemical analysis for level 1

Table 4. 2 Mean Chemical Composition of Raw laterite in Level O and Statistical Comparison of AAS and XRF

Compound	AAS± SE	XRF± SE	df	tcal	tcrit
Al_2O_3	12.68±0.51	20.09±0.58	81.00	9.534	0.960
SiO_2	22.78±0.91	23.03±0.57	69.00	0.820	0.960
Fe_2O_3	50.50±1.19	51.47±1.29	82.00	0.580	0.960
Fe	32.33±0.97	31.91±0.92	82.00	0.765	0.960

Each value is mean ± SE of 3 replicates

Table 4. 3 Mean Chemical Composition of Raw laterite in Level 1 and Statistical Comparison of AAS and XRF

Compound	AAS± SE	XRF± SE	df	t- cal	t- crit
Al_2O_3	14.341±0.439	19.152±0.520	82.000	1.234225	0.960
SiO_2	22.334±0.808	23.606±0.672	82.000	0.229712	0.960
Fe_2O_3	52.358±1.045	52.647±1.204	82.000	0.856635	0.960
Fe	33.746±0.723	33.164±0.761	82.000	0.609354	0.960

Each value is mean ± SE of 3 replicates

From the results there was no significance difference between the means of SiO_2, Fe_2O_3 and Fe. their t_{cal} was less than t_{crit}. .For Al_2O_3 there was significance difference between AAS and XRF t_{cal} was greater than t_{crit}. This can be attributed to the fact that XRF exaggerates amount of aluminium (Frank and Hafida, 2003). Full results of analysis by AAS are given in Appendix 2 and XRF Appendix 3.

Amount of iron oxide was also determined using EDTA titration. The amount obtained was used to calculate percentage of iron. Results are shown in Table 4.4 for level O and Table 4.5 for level 1.

Table 4. 4 Percentage Composition of Iron (III) Oxide and Iron in Raw laterite Determined by EDTA Titration Level O.

	iron oxide	%iron
SITE	Mean ±SE	Mean ±SE
Gatong'ora	42.55±0.05	26.79±0.01
Githunguri R.	41.04±0.04	25.82±0.01
Githurai Kimbo	50.65±0.05	31.82±0.00
Kahawa sukari	51.65±0.05	32.52±0.01
Kwihota	48.50±0.00	30.55±0.00
Miti Kenda	60.00±0.00	37.81±0.01
Mchana	54.00±0.00	34.01±0.00
Mugutha	62.00±0.00	39.07±0.00
Murera Coffee	56.20±0.00	35.42±0.01
Mwihoko	60.36±0.06	38.00±0.00
Ruiru prisons	40.04±0.04	25.21±0.01
Tassia	48.65±0.05	30.63±0.00
Tatu	50.65±0.05	31.89±0.01
Theta	38.04±0.04	23.95±0.00

Each value is Mean ±SE of 3 replicates

Table 4. 5 Percentage Composition of Iron (III) Oxide and Iron in Raw laterite Determined by EDTA Titration Level 1.

SITE	Iron oxide Mean ±SE	% iron Mean ±SE
Gatong'ora	36.04±0.04	22.67±0.01
Githunguri R.	52.86±0.05	33.26±0.00
Githurai Kimbo	45.05±0.05	28.33±0.03
Kahawa sukari	51.65±0.05	32.52±0.01
Kwihota	48.25±0.25	30.25±0.01
Miti Kenda	50.80±0.00	32.01±0.00
Muchana	55.40±0.00	34.91±0.00
Mugutha	52.00±0.00	32.77±0.01
Murera Coffee	48.60±0.00	30.63±0.00
Mwihoko	60.06±0.06	37.81±0.01
Ruiru prisons	54.06±0.05	34.03±0.00
Tassia	58.86±0.06	37.05±0.00
Tatu	52.25±0.05	32.90±0.00
Theta	62.67±0.06	39.45±0.01

Each value is Mean ±SE of 3 replicates

Results of mean average percent of iron was used to statistically compare the three methods of analysis, AAS, XRF and EDTA titration at P = 0.05. Table 4.6 gives this comparison.

Table 4. 6 Statistical Comparison of percentage of Iron (all sites) for AAS, XRF and EDTA titration Method

level	**AAS±S.E**	**XRF±S.E**	**EDTA±S.E**	**P-value**
LEVEL 0	31.82±0.91	31.91±0.92	31.67±0.91	0.984
LEVEL 1	33.04 ±0.88	33.16 ±0.76	32.75 ±0.78	0.933

One-way ANOVA show no significant difference between the three methods ($p>0.05$ at $\alpha = 0.05$)

The results of analysis of raw laterite show that the iron content of laterite from Ruiru Division is in the range 22 to 40 percent. This amount of iron compares favourably with that in iron ores in other countries (Boltz, 1970. Olubambi, *et al.*, 2005). In France, India, USA, and Nigeria ores are used for production of metallic iron and iron products after concentration. For example USA in the Masebi Range uses an ore with 34.0% iron, France in Sancy District uses an ore with 32.0% iron, United Kingdom in Frodigham district uses an ore with between 18-24% irons, and Nigeria in Itakpe region uses an ore with 34.0% iron. In all examples above the ore is used for production of metallic iron and iron products after concentration. laterite in Ruiru contain higher percentage of iron than some of these ores

The Kenyan laterite found in Ruiru Division in Thika District are therefore iron ore grade. They contain more than 25.0 percent iron, this is the level considered economical for ore exploitation (Kirk - Othmer, 2004). Geological survey reports show that laterite are widespread in Kenya (Dubois and Walsh, 1970). But the ores would require beneficiation before smelting can be done (Johnstone and Johnstone, 1961).

4.2 Concentration of Iron by Heating a Mixture of Laterite with Carbon (charcoal*).*

The raw laterite was heated in mixture with carbon (charcoal) and using charcoal as fuel. Magnetic separation was then used to concentrate iron from the heated mix. The amount of heated laterite picked by magnet expressed as percentage shown in Table 4.7 with respect to region of sampling.

Table 4. 7 Percent of Heated Laterite Mix Picked by Magnet in Level O and Level 1.

Site	% of heated laterite mix picked by magnet level O Mean±SE	% of heated laterite mix picked by magnet Level 1 Mean±SE
Gatong'ora	52.03±0.03	77.13±0.07
Githunguri R.	77.57±0.07	68.03±0.03
Githurai Kimbo	58.03±0.03	77.63±0.07
Kahawa sukari	61.03±0.03	78.07±0.07
Kwihota	65.10±0.03	65.10±0.06
Miti Kenda	55.03±0.03	60.10±0.06
Muchana	68.10±0.06	65.03±0.03
Mugutha	54.07±0.03	63.07±0.03
Murera Coffee	63.03±0.03	74.53±0.03
Mwihoko	60.03±0.03	62.53±0.03
Ruiru prisons	52.57±0.03	51.07±0.03
Tassia	57.53±0.03	70.10±0.06
Tatu	67.60±0.06	62.53±0.03
Theta	60.07±0.03	73.10±0.06

Each value is Mean ±SE of 3 replicates

XRD results the minerals picked by magnet showed it contained mainly magnetite with chemical formulae $Fe_{2.46}MgO_{0.42}AlTi0_3O_4$, Fe_3O4, $Fe_{2.886}O_4$ and quartz. The results are shown in Table 4.8.

Table 4. 8 Mineral Content of Heated Mixture of Laterite and Carbon (charcoal)

Compound name	Chemical formula	Quantification (% by mass)
Quartz	SiO_2	30.3
Magnetite	$Fe_{2.46}MgO_{0.42}AlTi_{.03}O_4$	15.2
Iron diiron (III) Oxide, Magnetite	Fe_3O_4	41.4
Magnetite low	$Fe_{2.886}O_4$	13.1

When laterite were heated using charcoal but not in mix with the charcoal, the percent conversion of goethite to magnetite was about 2% clearly, magnetic separation cannot concentrate such product. Prizepiera and Prizepiera (2003) observed that heating Goethite in the temperature range of 200-400 ^oC converts it to hematite. The reaction that took place is given in Equation 4.1.

$$Fe_2O_3.H_2O \xrightarrow{\Delta} Fe_2O_3 + H_2O \quad ....(4.1)$$

Results of current study show that, when a reducing agent was included, all the goethite present was converted to magnetite. The same was observed by Purwanto *et al.* (2003) while working with laterite from Indonesia. He noted that when laterite were heated in presence of a reducing agent, Goethite was converted to magnetite. The reducing agent used was a mixture CO/ CO_2 in the ratio 1:3. The reactions that take place are given in Equations 4.2 and 4.3 (Purwanto *et al.*, 2003)

$Fe_2O_3.H_2O \xrightarrow{673K} Fe_2O_3 + H_2O$.. (4.2)

$3Fe_2O_3 + CO \xrightarrow{973K} 2Fe_3O_4 + CO_2$ ……………………………..….... (4.3)

The amount of iron in the concentrate was determined using XRF. It was then used to calculate increased iron content compared to that in raw laterite. Table 4.9 gives iron content of level O concentrate and Table 4.10 for level 1

Table 4. 9 Iron Content in Concentrate after Magnetic Separation Level 0

	Site	% Fe in raw laterite ± SE	% Fe after magnetic separation± SE
1	Gatong'ora	21.17± 0.03	46.12±0.00
2	Githunguri R.	26.16± 0.01	44.50±0.00
3	Githurai Kimbo	30.88± 0.01	50.10±0.00
4	Kahawa sukari	34.67±0.01	54.40±0.00
5	Kwihota	30.63±0.01	40.40±0.00
6	Miti Kenda	38.51±0.01	56.91±0.00
7	Muchana	35.32±0.01	46.71±0.00
8	Mugutha	39.32±0.01	58.07±0.00
9	Murera Coffee	35.17±0.01	49.53±0.00
10	Mwihoko	41.16±0.01	61.55±0.00
11	Ruiru prisons	26.51±0.01	39.68±0.00
12	Tassia	30.63±0.01	54.02±0.01
13	Tatu	33.91±0.01	49.53±0.00
14	Theta	22.69±0.01	37.95±0.01
	Mean average Increase		**17.93±0.03**

Each value is mean ± SE of 3 replicates

Table 4. 10 iron Content in Concentrate after Magnetic Separation Level 1

	Site	% Fe in raw laterite ± SE	% Fe after magnetic separation ± SE
1	Gatong'ora	21.81±0.01	35.11±0.00
2	Githunguri R.	33.91±0.01	49.53±0.00
3	Githurai Kimbo	27.41±0.01	44.27±0.03
4	Kahawa sukari	33.73±0.01	49.51±0.01
5	Kwihota	30.63±0.01	51.92±0.01
6	Miti Kenda	35.80±0.01	54.17±0.01
7	Muchana	37.00±0.01	56.60±0.00
8	Mugutha	34.52±0.01	54.60±0.00
9	Murera Coffee	33.10±0.01	49.51±0.00
10	Mwihoko	37.19±0.01	56.05±0.00
11	Ruiru prisons	26.51±0.01	54.82±0.00
12	Tassia	37.95±0.01	58.58±0.00
13	Tatu	34.17±0.01	53.66±0.00
14	Theta	40.59±0.01	56.77±0.01
	The mean average increase		**17.98±0.03**

Each value is mean ± SE of 3 replicates

The amount of iron increased by an average of 17.98% across the samples. This increase compares well with increase that takes place where iron ore is being concentrated for commercial exploitation. Purwanto *et al.* (2003) found out that magnetic separation increased amount of iron in laterite from Sebuku Island (Indonesia) from 45 to 55 percent. Youssef and Morsi (1998) also observed that heating oxidised iron ore from Aswan locality, Egypt, then reducing using either CO or H_2 increased percent of iron from 45.25 to 59.6. This is possible because goethite in laterite is magnetized by heating and reduction. According to Purwanto *et al.* (2003) magnetite formed could be concentrated by magnetic separation. The amount of gangue reduced thereby increasing the amount of iron. Like haematite, goethite belongs to the group of hard magnetic materials which need high magnetic fields in order to reach magnetic saturation. For this reason they cannot be concentrated using magnetic separation. According to France and Oldfield (2000) some samples containing goethite could not reach their magnetic saturation at fields of 10 Tesler. It was possible to concentrate magnetite using magnetic separation because the gangue is classified as paramagnetic or diamagnetic that has low magnetic susceptibilities (Resende *et al.,* 1986). The relatively high iron content in the magnetic separation concentrate indicated a potential use as raw material for iron smelting.

4.3 Concentration of Iron in laterite by Froth Flotation

After concentration of laterite by froth flotation the amount of iron in concentrate was determined by XRF. The amount was used to calculate increased iron. Table 4.11 gives results of iron content in concentrate and increment value level O and Table 4.12 gives results of level 1.

Table 4. 11 Iron Content in Concentrate after Froth Flotation Level O

Site	% Fe in raw laterite ± SE	% Fe after Froth flotation± SE
Gatong'ora	21.17±0.01	40.15±0.05
Githunguri R.	26.16±0.01	38.70±0.00
Githurai Kimbo	30.88±0.01	43.20±0.00
Kahawa Sukari	34.67±0.01	49.10±0.00
Kwihota	30.63±0.01	37.10±0.00
Miti Kenda	38.51±0.01	47.51±0.00
Muchana	35.32±0.01	41.91±0.00
Mugutha	39.32±0.01	49.31±0.01
Murera Coffee	35.17±0.01	43.11±0.00
Mwihoko	41.16±0.01	51.01±0.00
Ruiru prisons	26.51±0.01	35.01±0.00
Tassia	30.63±0.01	47.61±0.00
Tatu	33.91±0.01	44.31±0.01
Theta	22.69±0.01	34.91±0.00
The mean average increase		**11.74±0.10**

Each value is mean ± SE of 3 replicates

Table 4. 12 Iron Content in Concentrate after Froth Flotation Level 1

Site	% Fe in raw laterite ± SE	% Fe after Froth flotation± SE
Gatong'ora	21.81±0.01	31.95±0.05
Githunguri R.	33.91±0.01	46.50±0.00
Githurai Kimbo	27.41±0.01	37.85±0.05
Kahawa Sukari	33.73±0.01	44.51±0.01
Kwihota	30.63±0.01	46.21±0.00
Miti Kenda	35.80±0.01	47.41±0.00
Muchana	37.00±0.01	48.81±0.01
Mugutha	34.52±0.01	45.71±0.00
Murera Coffee	33.10±0.01	44.21±0.00
Mwihoko	37.19±0.01	48.81±0.01
Ruiru prisons	26.51±0.01	47.61±0.00
Tassia	37.95±0.01	51.41±0.00
Tatu	34.17±0.01	48.21±0.00
Theta	40.59±0.01	47.71±0.00
The mean average increase		**11.74±0.10**

Each value is mean ± SE of 3 replicates

From the results it was observed that the level of iron was enriched by an average of 11.74 %. Ola *et al.* (2009) while working with iron ores of Itakpe Nigeria were able to enrich iron content from 35 to 63.4 percent by froth flotation. Ajaka (2009) was also able to upgrade iron in the tailing of Itakpe iron ore process, Nigeria from 22 to 43 percent. Viana, *et al.* (1991) while testing flotation columns observed that iron content increased from 56.87 to 67.84 percent. In all the above examples the increase is higher than that of the present study apart from that of Viana, *et al.* (1991) but it is important to note that concentrate is collected then re-floated several times as shown in Figure 4.1

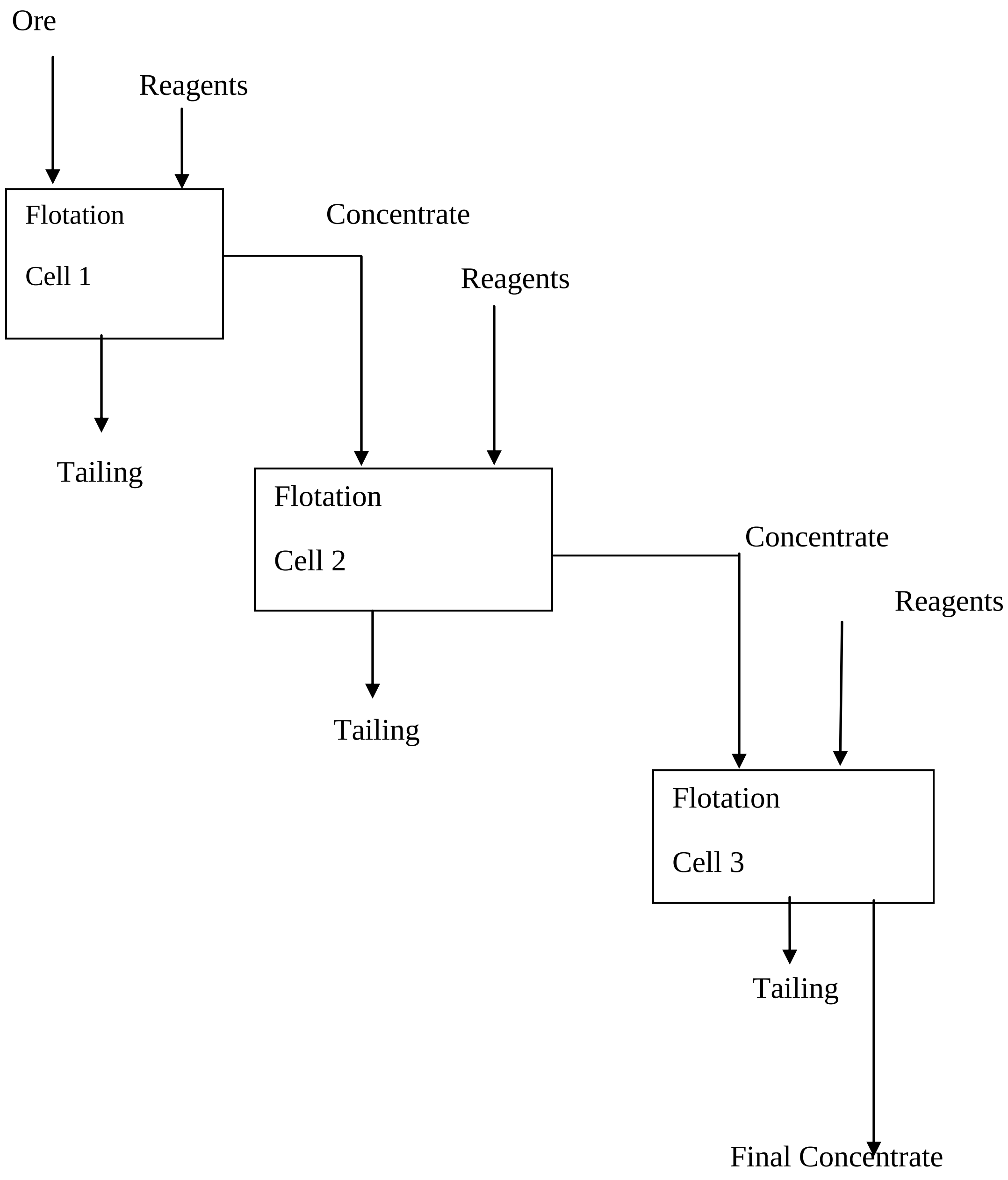

Figure 4. 1 flotation scheme.

4.4 Statistical t-test Analyses for Methods of Concentration (Magnetic Separation and Froth Flotation)

Then two methods of concentration were statistically analyzed using paired t-test at P= 0.05 and 26 degrees of freedom. The mean average increase of iron was used to compare the two methods. t-cal was found to be 16.78. t-crit was 2.06; t-cal was greater than t-crit, the two methods are significantly different. The significance difference between the two can be attributed to the iron minerals in the concentrate. The percent of iron in minerals are different; Magnetite contains 72% iron, hematite 70% iron and goethite 63% iron. Magnetic separation is a better method of concentrating iron than froth flotation because the mean average increase was higher.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSIONS

From the results obtained and analysis of this study the following conclusions can be drawn

1) Iron content in laterite from Ruiru Division of Thika District is in the range between 22 to 40 percent. These content are of ore grade because it is more than 25 percent which is considered economical for exploitation

   .

2) The amount of iron in laterite from Ruiru Division of Thika District is of low grade and beneficiation is necessary before it can be used as a feed in the blast furnace.

3) The laterite when mixed with charcoal heated converts goethite to magnetite. Magnetic separation can then be used for concentration. Magnetic separation increased percent of iron by 17.98%.

4) Froth flotation can also be used to concentrate iron minerals in laterite. This method increased percent of iron by 1174%.

5.2 Recommendation for Further Research.

Only laterite found in Ruiru Division were used in this research. It is, therefore, important for iron content in Murram (laterite) from other areas of the country to be analysed. These can provide quantitative information of iron in laterite deposits in Kenya.

Silicon (IV) oxide and aluminium oxide are the major contents of the tailing after laterite concentration. It is necessary to determine whether the tailing can be used as raw material for cement /ceramics manufacture.

The study was limited to laboratory analysis therefore, it is necessary to set up a pilot plant to get iron from laterite of Ruiru Division and hence carry out an economical survey.

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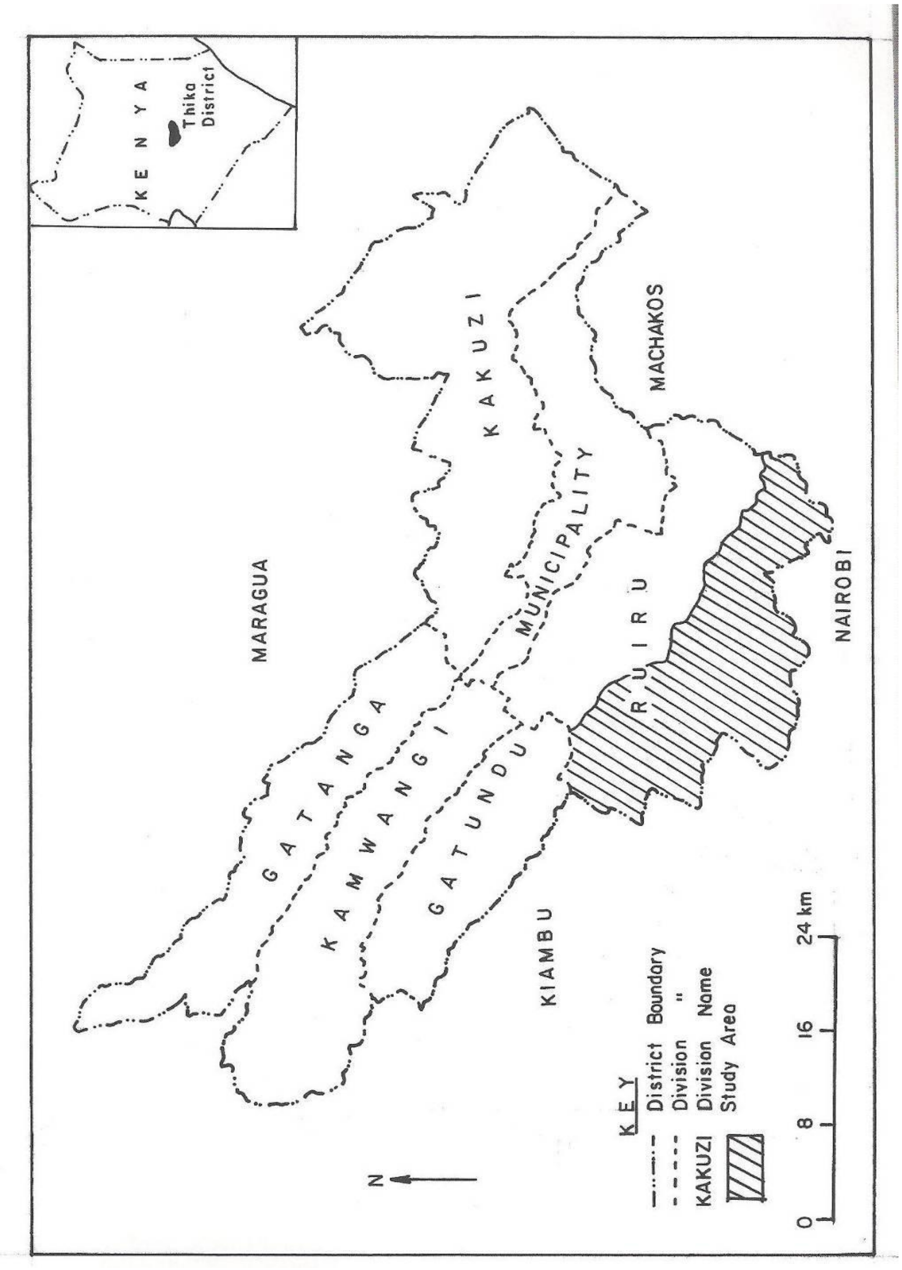
Appendices

Appendix 1

Map of Ruiru Division Thika District, Kenya



Appendix 2

Percentage chemical composition of raw laterite from Ruiru analysed by AAS level O

site	Al_2O_3 % Mean ±SE	SiO_2 % Mean ±SE	Fe_2O_3 % Mean ±SE	Fe % Mean ±SE
Gatong’ora	12.40±0.00	30.52±0.02	43.79±0.02	27.59±0.01
Githunguri R.	11.55±0.02	34.12±0.02	40.79±0.02	25.70±0.00
Githurai Kimbo	12.05±0.02	26.72±0.02	46.79±0.02	29.48±0.00
Kahawa sukari	12.42±0.02	19.31±0.01	54.79±0.01	34.52±0.00
Kwihota	13.60±0.00	24.32±0.02	48.00±0.02	30.24±0.00
Miti Kenda	12.25±0.02	14.31±0.01	60.52±0.01	38.13±0.01
Mchana	10.60±0.00	18.61±0.01	55.20±0.01	34.79±0.00
Mugutha	10.40±0.00	21.71±0.01	60.52±0.01	38.13±0.01
Murera Coffee	13.60±0.00	15.11±0.01	56.22±0.01	35.42±0.01
Mwihoko	9.62±0.02	15.71±0.01	60.52±0.01	38.13±0.01
Ruiru prisons	10.40±0.00	28.82±0.02	45.02±0.02	28.36±0.00
Tassia	12.02±0.02	24.12±0.02	49.31±0.02	31.07±0.01
Tatu	12.80±0.00	19.61±0.01	50.20±0.01	31.63±0.00
Theta	23.81±0.01	25.92±0.02	35.52±0.02	22.38±0.00

Each value is Mean ±SE of 3 replicates

Appendix 2 (continued)

Percentage chemical composition of raw laterite from Ruiru analysed by AAS level 1

site	Al_2O_3 Mean ±SE	SiO_2 Mean ±SE	Fe_2O_3 Mean ±SE	Fe Mean ±SE
Gatong'ora	21.84±0.04	30.02±0.02	34.03±0.03	21.43±0.01
Githunguri R.	13.83±0.03	17.32±0.02	53.55±0.05	33.72±0.00
Githurai Kimbo	11.42±0.02	32.52±0.02	41.81±0.01	26.33±0.00
Kahawa sukari	11.85±0.02	28.74±0.02	55.56±0.06	35.98±0.01
Kwihota	14.83±0.03	17.81±0.01	50.62±0.02	31.88±0.00
Miti Kenda	15.13±0.03	21.41±0.01	54.92±0.02	34.60±0.00
Mchana	14.86±0.03	22.01±0.01	54.65±0.05	34.41±0.00
Mugutha	18.07±0.04	20.01±0.01	52.85±0.05	33.27±0.01
Murera Coffee	13.65±0.03	27.94±0.02	53.55±0.05	33.72±0.00
Mwihoko	15.97±0.03	14.92±0.01	56.56±0.06	35.61±0.00
Ruiru prisons	12.02±0.02	22.31±0.01	50.62±0.02	31.89±0.01
Tassia	10.62±0.02	20.71±0.01	58.22±0.02	36.67±0.01
Tatu	13.05±0.03	18.53±0.01	54.05±0.05	34.03±0.00
Theta	13.63±0.03	18.41±0.01	62.02±0.02	39.07±0.00

Each value is Mean ±SE of 3 replicates

Appendix 3

Percentage chemical composition of raw laterite from Ruiru analysed by XRF level O

site	Al_2O_3 Mean ±SE	SiO_2 Mean ±SE	Fe_2O_3 Mean ±SE	Fe Mean ±SE
Gatong'ora	20.01±0.01	28.43±0.03	44.73±0.03	21.17±0.01
Githunguri R.	25.02±0.02	29.03±0.03	41.53±0.03	26.16±0.01
Githurai Kimbo	22.06±0.04	24.02±0.02	49.40±0.00	30.88±0.01
Kahawa sukari	18.15±0.03	20.02±0.02	55.00±0.00	34.67±0.01
Kwihota	24.06±0.04	24.05±0.02	48.63±0.03	30.63±0.01
Miti Kenda	15.04±0.03	20.02±0.02	61.10±0.00	38.51±0.01
Mchana	21.06±0.04	18.03±0.02	56.04±0.04	35.32±0.01
Mugutha	15.01±0.01	19.04±0.02	62.44±0.04	39.32±0.01
Murera Coffee	18.15±0.03	22.02±0.02	55.80±0.00	35.17±0.01
Mwihoko	13.54±0.02	17.52±0.02	65.30±0.00	41.16±0.01
Ruiru prisons	24.02±0.02	25.05±0.03	42.03±0.03	26.51±0.01
Tassia	22.06±0.04	24.02±0.02	48.60±0.00	30.63±0.01
Tatu	18.05±0.03	23.05±0.02	53.84±0.04	33.91±0.01
Theta	25.02±0.02	28.05±0.03	36.12±0.02	22.69±0.01

Each value is Mean ±SE of 3 replicates

Appendix 3(continued)

Percentage chemical composition of raw laterite from Ruiru analysed by XRF level 1

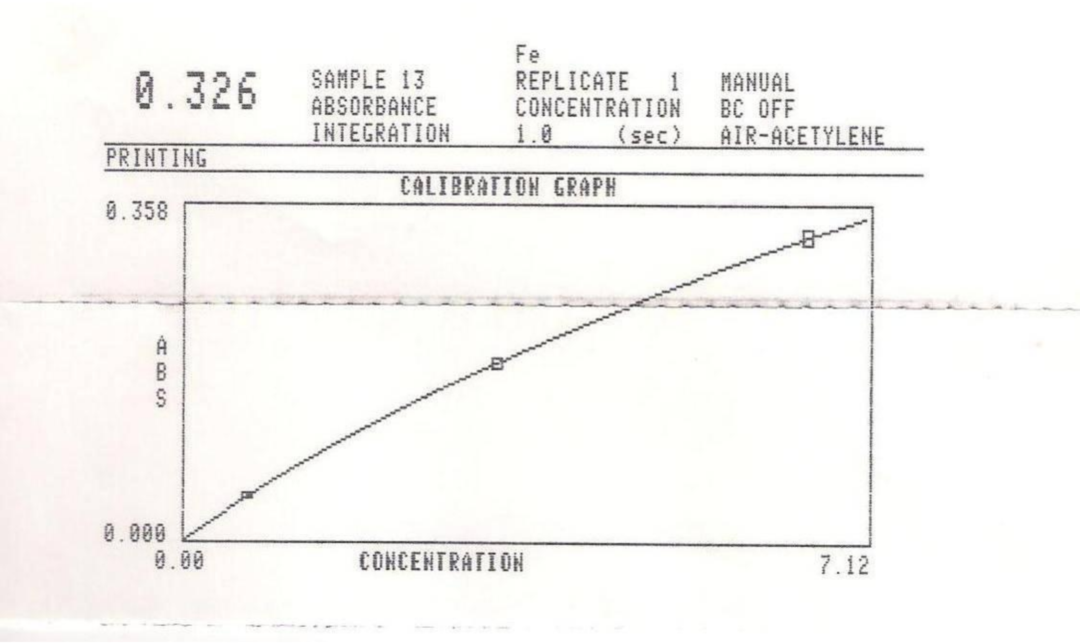
site	Al_2O_3 Mean ±SE	SiO_2 Mean ±SE	Fe_2O_3 Mean ±SE	Fe Mean ±SE
Gatong'ora	26.02±0.02	35.04±0.04	34.80±0.00	21.81±0.01
Githunguri R.	17.51±0.01	23.12±0.02	53.80±0.00	33.91±0.01
Githurai Kimbo	20.01±0.01	25.05±0.03	43.53±0.03	27.41±0.01
Kahawa sukari	19.65±0.03	24.42±0.02	53.50±0.00	33.73±0.01
Kwihota	20.01±0.01	28.05±0.03	48.63±0.03	30.63±0.01
Miti Kenda	19.01±0.01	21.04±0.02	56.84±0.04	35.80±0.01
Mchana	15.01±0.01	19.04±0.02	58.74±0.04	37.00±0.01
Mugutha	20.01±0.01	22.05±0.02	54.84±0.04	34.52±0.01
Murera Coffee	20.05±0.04	24.22±0.02	52.50±0.00	33.10±0.01
Mwihoko	12.03±0.02	26.03±0.03	59.00±0.00	37.19±0.01
Ruiru prisons	24.02±0.02	25.03±0.03	42.00±0.00	26.51±0.01
Tassia	19.01±0.01	18.03±0.02	60.24±0.04	37.95±0.01
Tatu	19.75±0.03	21.32±0.02	54.20±0.00	34.17±0.01
Theta	16.01±0.01	18.03±0.02	64.44±0.04	40.59±0.01

Each value is Mean ±SE of 3 replicates

Appendix 4

Calibration curve for Fe

Method: AAS

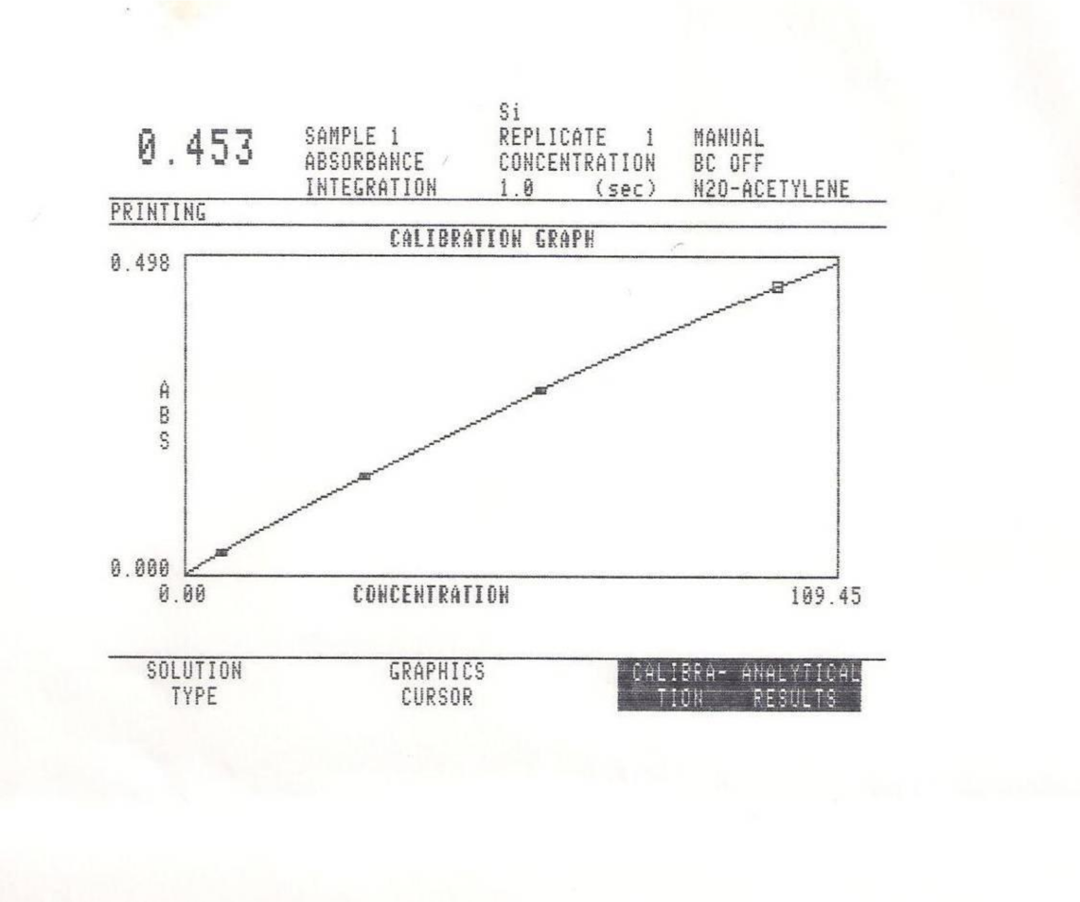


```
                          Fe
0.326   SAMPLE 13         REPLICATE   1   MANUAL
        ABSORBANCE        CONCENTRATION   BC OFF
        INTEGRATION       1.0     (sec)   AIR-ACETYLENE
PRINTING
```

Appendix 5

Calibration curve for Si

Method: AAS

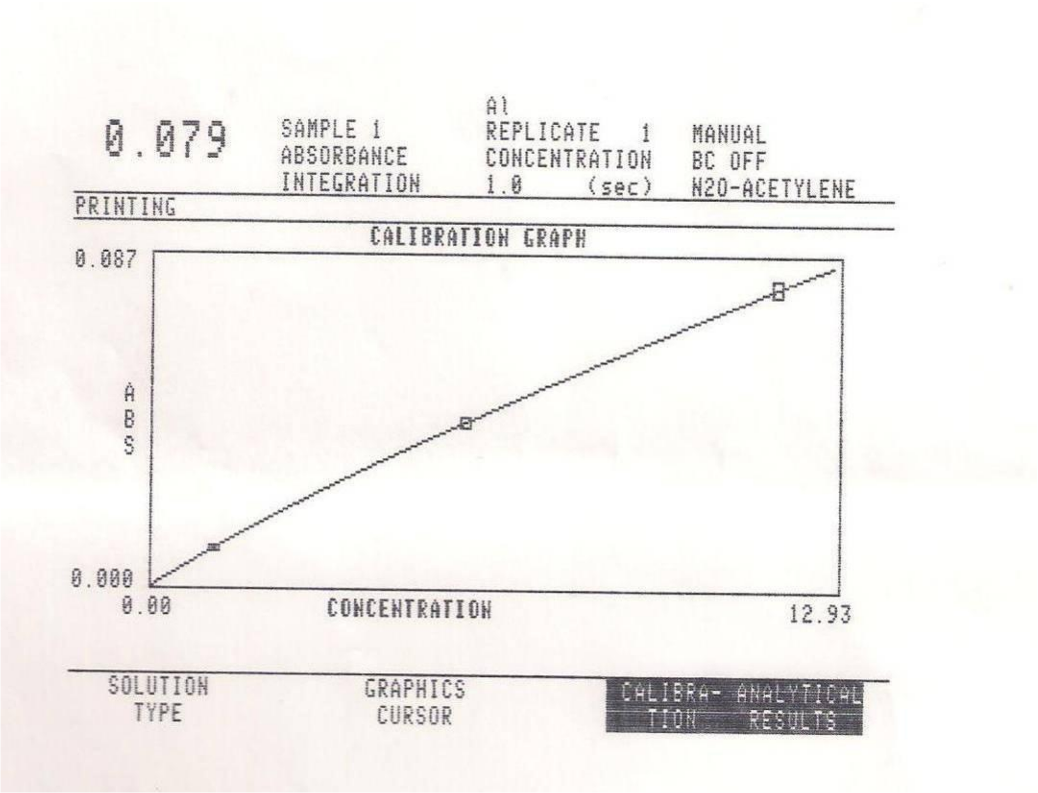


```
                          Si
0.453      SAMPLE 1       REPLICATE   1    MANUAL
           ABSORBANCE     CONCENTRATION    BC OFF
           INTEGRATION    1.0     (sec)    N2O-ACETYLENE
PRINTING
```

Appendix 6

Calibration curve for Al

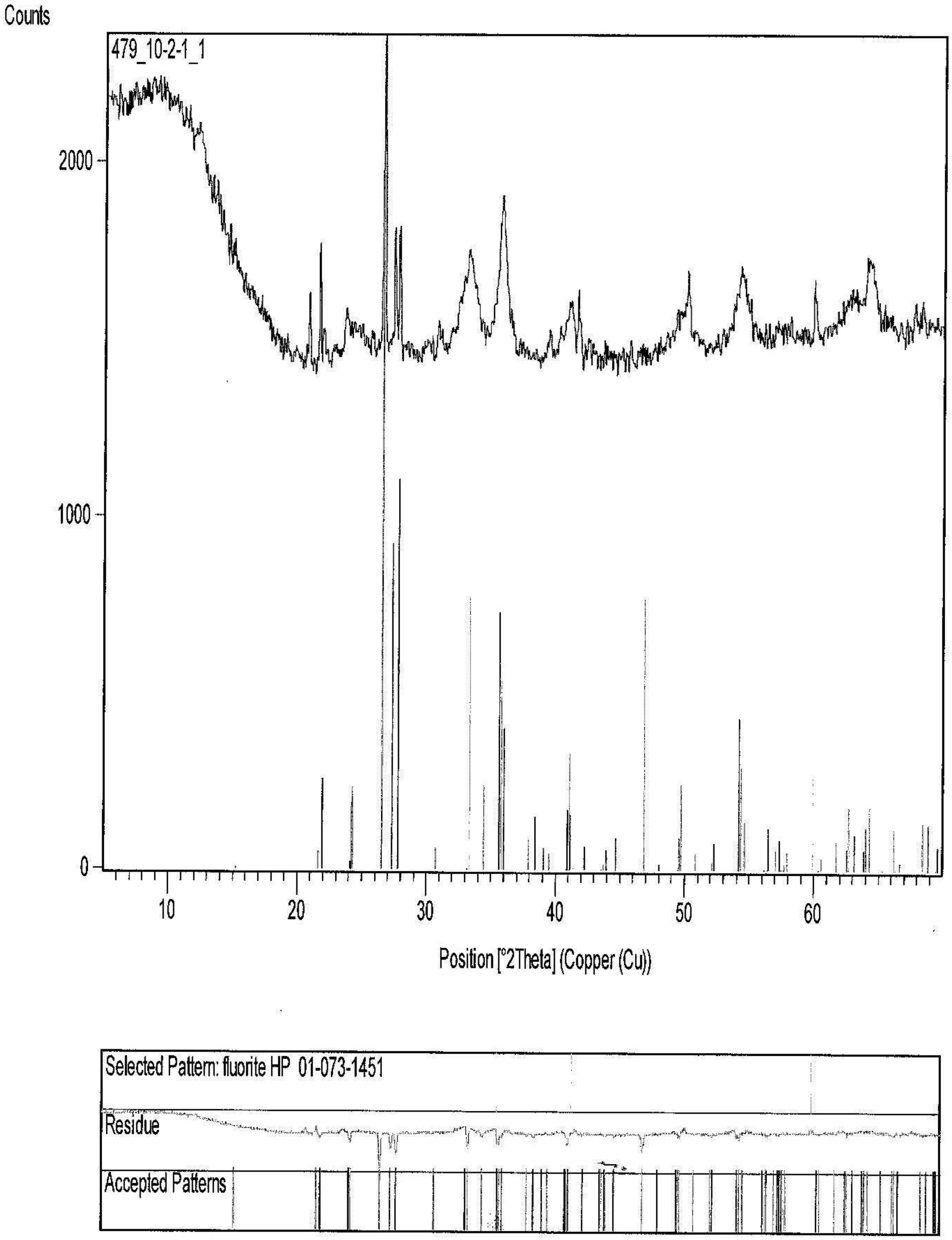
Method: AAS



```
                                   Al
  0.079    SAMPLE 1                REPLICATE    1    MANUAL
           ABSORBANCE              CONCENTRATION     BC OFF
           INTEGRATION             1.0      (sec)    N2O-ACETYLENE
PRINTING
                         CALIBRATION GRAPH
```

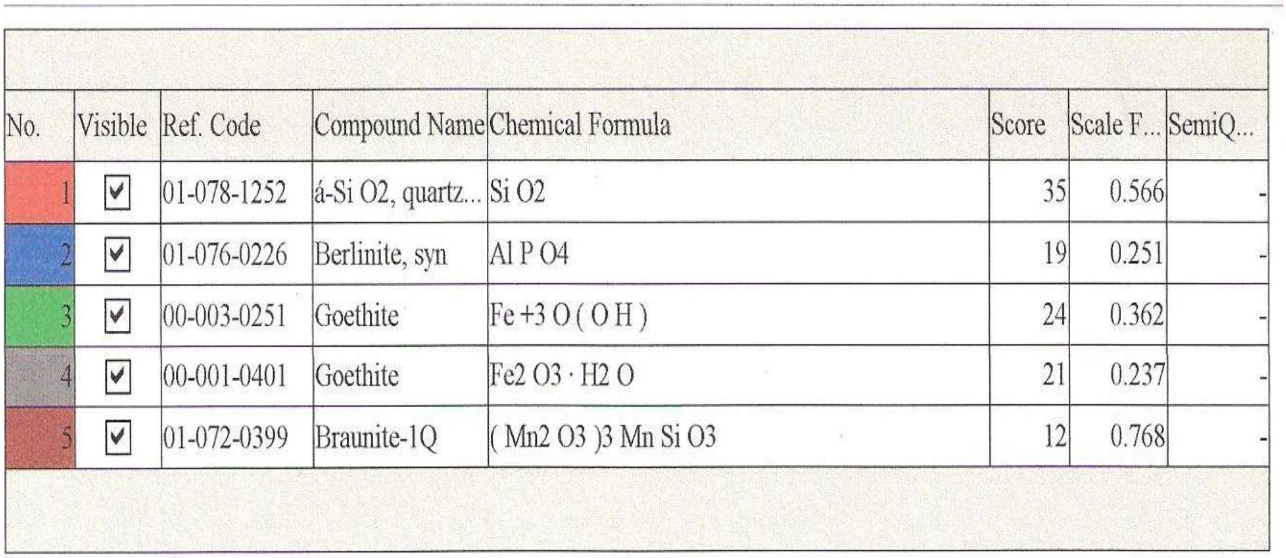
Appendix 7

XRD peaks for Tassia level O sample



Appendix 8

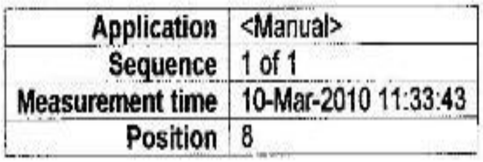
XRD results for Tassia level O sample



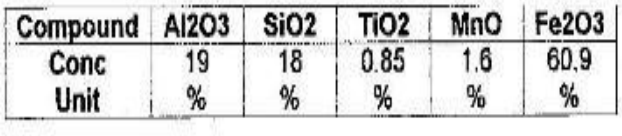
No.	Visible	Ref. Code	Compound Name	Chemical Formula	Score	Scale F...	SemiQ...
1	☑	01-078-1252	á-Si O2, quartz...	Si O2	35	0.566	-
2	☑	01-076-0226	Berlinite, syn	Al P O4	19	0.251	-
3	☑	00-003-0251	Goethite	Fe +3 O (O H)	24	0.362	-
4	☑	00-001-0401	Goethite	Fe2 O3 · H2 O	21	0.237	-
5	☑	01-072-0399	Braunite-1Q	(Mn2 O3)3 Mn Si O3	12	0.768	-

Appendix 9

XRF result for Tassia level 1sample



Application	<Manual>
Sequence	1 of 1
Measurement time	10-Mar-2010 11:33:43
Position	8



Compound	Al2O3	SiO2	TiO2	MnO	Fe2O3
Conc	19	18	0.85	1.6	60.9
Unit	%	%	%	%	%