



Sudan University of Science and Technology

College of Science – chemistry department

Chemical Analysis of Grewia Tenax Fruits

تحليل وتقدير محتوى بعض العناصر الكيميائية في نبات القضم

**A dissertation submitted for Fulfillment of the
requirements of the Degree of B.Sc in Chemistry
(Honors)**

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بِسْمِ اللّٰهِ الرَّحْمٰنِ الرَّحِیْمِ

قال تعالى:

(وَقُلْ اَعْمَلُوا فَسَيَرَى اللّٰهُ عَمَلَكُمْ وَرَسُولُهُ وَالْمُؤْمِنُونَ وَسَتُرَدُّونَ

اِلَى عَالَمِ الْغَيْبِ وَالشَّهَادَةِ فَيُنَبِّئُكُمْ بِمَا كُنْتُمْ تَعْمَلُونَ)

صدق الله العظيم

سورة التوبة الآية (105)

Dedication

To our beloved mother, father , sisters and brothers

And friends with love.

Acknowledgements

First and foremost profound thanks to Allah for the providing us with unfailing support, good health and wellbeing throughout our study and through the process of researching and writing this thesis.

We would like to express our sincere gratitude to our Supervisor Ustaz Shams Eddeen Omer Mohammed for his guidance, patience and continuous support throughout the period of this study.

Abstract

In the present study the nutritional evaluation of guddaim fruits, was carried out, The sample was ashes (4.04%) and treated with hydrochloric acid, the resulting solution was used to determine the calcium, iron, sodium and potassium, The content of iron was determined using spectrophotometer which was found to be 0.22%.

Calcium, sodium and potassium determined using flame photometer which were found to be 0.433%, , 0.0073% and 1.036% respectively.

The protein content was determined using kjedahl digestion method and was found to be 14.5%.

المستخلص

في هذه الدراسة تم دراسة القيمة الغذائية لثمار القزيم، حيث تم حرق العينة وكانت نسبة الرماد الناتج تساوي 4.04%، وتمت معالجته بواسطة حمض الهيدروكلوريك، والمحلول المتحصل عليه تم استخدامه في تحديد الكالسيوم، الحديد، الصوديوم والبوتاسيوم.

تم تحليل الحديد باستخدام جهاز التحليل الطيفي الجزيئي (إسبكتروفوتوميتر) وكانت نسبته 0.22% كما تم تحليل كل من الكالسيوم، الصوديوم والبوتاسيوم باستخدام جهاز مطيافية اللهب وكانت النسب 0.433%، 0.0073% ، 1.036% على التوالي.

تم تحديد البروتين باستخدام طريقة كلدان وكانت نسبته 14.5%.

List of content:

Contents	Page
الآية	i
Dedication.	ii
Acknowledgements.	iii
Abstract (English).	iv
المستخلص	v
List of content	vi
List of tables.	ix
List of figures	x
List of Abbreviation and nomenclature	xi
Chapter one:	
Introduction and literature review	
1.1 Introduction	1
1.2 plant profile	3
1.3 Classification	3
1.4 Chemical Composition	4
1.5 Geographical distribution	4
1.6 Products of Economic Importance	4
1.7 Medicinal importance	5
1.8 Sodium	5
1.8.1 Chemistry	6
1.8.2 Compounds:	6
1.8.3 Occurrence	7

1.9 Calcium	7
1.9.1 Chemistry and compound	8
1.9.2 Occurrence	9
1.10 Iron	10
1.10.1 Occurrence	10
1.10.2 Chemistry and compounds	11
1.11 Potassium	12
1.11.1 Chemistry	13
1.11.2 Compounds	14
1.11.3 Occurrence	15
1.12 Methods of analysis of element	15
1.12.1 Quantitative analysis	15
1.12.2 Qualitative analysis	16
1.12.3 Spectrophotometry	16
1.12.4 Flame emission	17
1.12.4.1 Atomic emission – the flame test	17
1.12.4.2 Flame photometry	19
1.13 Objective of this work	21
Chapter two	
Materials and methods	
2.1 Materials and equipments	22
2.1.1 Chemical	22
2.1.2 Apparatus	22
2.2 Methods	23
2.2.1 Sample preparation	23
2.2.2 Chemical analysis of the sample	23

2.2.2.1 Ash content	23
2.2.2.2 Determination of protein	23
2.2.2.3 Determination of metals	24
2.2.2.3.1 Sample preparation for metals analysis	24
Determination of iron	25
Determination of sodium	27
Determination of potassium	28
Determination of calcium	29
Chapter three	
Discussion and recommendation	31
References.	33

List of tables:

Table	Description	Page
Table 2.1	The weight of ash/g	23
Table 2.2	Determination of Fe ⁺³	25
Table 2.3	The absorbance of Fe ⁺³ and concentration/ppm	25
Table 2.4	The emission of sodium and concentration/ppm	27
Table 2.5	The emission of potassium and concentration/ppm	28
Table 2.6	The emission of calcium and concentration/ppm	29

s

List of figures :

figures	Description	Page
Figure 1.1	The flame test observation of the flame color (a) without colored glass and (b)	18
figure 1.2	The sequence of events in the flame test.	18
figure 1.3	A schematic diagram of a flame photometer.	19
figure 1.4	A plot of intensity (I) VS concentration (C) representing quantitative analysis.	20
figure 2.1	Absorbance of Fe^{3+} against concentration.	26
Figure 2.2	Emission of sodium against concentration.	27
Figure 2.3	Emission of potassium against concentration.	28
Figure 2.4	Emission of calcium against concentration.	30

List of Abbreviations and Nomenclature:-

Abbreviations:

- 1.g: grame.
2. ppm: part per million.
3. nm: nano meter (10^{-9} of a meter).
4. conc: concentration.

Chapter One

Introduction and literature review

Chapter one

Introduction and literature review

1.1 Introduction:

Thousands of indigenous plants have been used by man from prehistoric times on all continents for relieving and curing ailments. In spite of tremendous development in the field of allopathy medicinal plants and their derivatives still remain one of the major sources of drugs in modern and traditional systems throughout the world playing a major role in medicinal therapy. In India about 7300 plant species are used in traditional health care systems. 90% of the medicinal plants come from natural habitats. There are many medicinal plants which find place in day-to-day uses, many of these, are used as herbal remedies. The expanding domestic and global demand of herbal products has put the native medicinal plant resources under significant stress. The plant genus "Grewia" is one of the best examples of multipurpose fruit species which are useful source of food, fodder, fiber, fuelwood, timber and a range of traditional medicines which cure a number of diseases. The genus was named by Carolus Linnaeus in honor of Nehemiah Grew, an English vegetable anatomist and physiologist, very famously known as the "Father of Plant Physiology"(1641-1712) from England.

This paper is focused on an important Grewia species *G. tenax* (Frosk.) Fiori. The plant is commonly known as Gangeran. The species is known for its edible fruits which are nutritionally balanced and rich in iron and calcium. The drupes also contain amino acids, mineral elements (K, Ca, Mn, Fe, Cu and Zn), tannin and pectic substances.

The plant is not only adapted to high temperatures and dry conditions, but has deep roots which stabilize sand dunes(Saied, A, *etal*, 2007). The shrub plays effectively for rehabilitation of wastelands if grown along the trees.

The plant has high medicinal values and is widely used for the treatment of various common diseases. *G. tenax* Fiori is reputed to cure upset of stomachs, some skin and intestinal infections, cough, fever, diarrhoea, dysentery, jaundice, rheumatism and have mild antibiotic properties. The plant preparations are used for the treatment of bone fracture and for bone strengthening. Its root and fruits are well known household remedy for the treatment of osteoporosis, tissue and wound healing. Leaves and twigs of *G. tenax* are an important component of folk medicine for the treatment of trachoma, tonsillitis, infections and are used as a poultice to treat swelling. *Grewia*'s extracts are also supposed to be helpful in curing hepatitis and other such diseases. The plant species has free radical scavenging activities which may be responsible for the therapeutic action against tissue damage. The plant gum was found to improve the fluidity of paracetamol granulation and could be a useful substitute binder in paracetamol tablet formulations (Martins, E, Christiana, I & Olobayo, K 2008). The potential of *Grewia* gum as a film coating agent was investigated using praziquantel tablets⁷. The plant gum may serve as a good suspending agent for Ibuprofen pediatric formulation, requiring no further aid in suspension redispersibility. Some other *Grewia* species are also known for their enormous medicinal values. *G. hirsuta* is used in the ayurvedic management of menopause and included in the class of drugs called "Rasayanas" that have an overall anti-aging effect in the body. The gum mucilage isolated from *G. optiva* had comparable binding ability and appears suitable for use as a pharmaceutical binder. Some *Grewia* species have been found to have anti emetic activity (Tijani, AY et al. 2008). In the experimental investigation some *Grewia* species (*G. sapida* Roxb., *G. pinnata* Roxb. and *G. nervosa* Lour.) were found to have 65 to 97% antioxidant activities. *G. hirsuta* Vahl and *G. tenax* Forsk. are mentioned in different classical texts as medicinal plants of wound healing. Root and fruits of *G. damine* Gaertn. are used in powdered form and employed in prevention of the

osteoporosis. Herbal preparation of *G. hirsuta* Vahl is given for the ayurvedic management of menopause. The plant drug Nagbala has properties to pacify provocation of Vata and Pitta humor. Drug is useful as nervine tonic, brain tonic, demulcent, anti-acidic, expectorant, antipyretic, diuretic, aphrodisiac, carminative and cardiac tonic. This herbal medicine Nagbala is used to boost the immune system of the body and maximize its potential in fighting the tumor. Ayurvedic medicine has the potential for a substantial contribution in the management of both early and advanced cancer (Pereira, J V et al. 2002).

1.2 Plant Profile

Common name : White Crossberry, Phalsa Cherry, Raisin bush, Gangara, Gangu, kanger.

Hindi name : Gondni, Gangeran

Synonyms : *Chadaratenax* Forssk, *G. populifolia* Vahl.

1.3 Classification

Kingdom : Plantae

Division : Angiospermae

Sub-division : Dicotyledons

Class : Polypetalae

Series : Thalamiflorae

Order : Malvales

Family : Tiliaceae

Genus : *Grewia*

Species : *Tenax*

1.4 Chemical Composition

The stem bark and leaves of *G. populifolia* are reported to constitute some phytochemicals like triacontan-1-ol, α - amyrin, β -amyrin, β -sitosterol, lupenne, erythrodiol, betulin and tetratriacont-21-ol-12-one. (Dash, VB&Materiamedica1987).The plant has been found to contain grewinol,tetratricontane-22-ol-13-one. The seeds contain 5% of bright-yellow oil containing palmitic acid, stearic acid, oleic acid, linoleic acid and unsaponifiabl (Morton.1987). In preliminary phytochemical studies plant extracts in different solvents were found to contain diterpines, glycosides.

1.5 Geographical distribution

The plant is native of Algeria, Botswana, Chad, Djibouti, Ethiopia,Iran, Kenya, Mali, Mauritania, Morocco, Namibia, Niger, Nigeria, Saudi Arabia, Senegal, Somalia, South Africa, Sudan, Tanzania, Uganda, Zimbabwe. The plant species is exotic to India and Pakistan.

In India it is spread over Andhra Pradesh, Bihar, Gujarat, Haryana, Karnataka, Kerala, Madhya Pradesh, Maharashtra, Punjab, Rajasthan, Tamil Nadu and West Bengal.

1.6Products of Economic Importance

Food: The fruits consumed by man and animals contain a large amount of iron and can be made into a refreshing drink. Fruit storage can be extended by drying. The dead leaves are eaten, but only while they remain on the plant. Its fruits are thirst quencher in summer season. A drink is prepared by soaking the fruit overnight, hand-pressing, sieving, and sweetening.

Fodder: Young leaves are consumed by livestock, they are slightly palatable at the end of dry seasons, and have fairly good feed value.

Fuel: The branches are used as firewood, and can be used in charcoal making.

Fiber: Ligno-Cellulosic Fibre with good tensile strength is made by the bark, which is used to make ropes and for binding purposes in house construction (Prasad cv,etal. 2010)

Timber: G. tenax wood is used in making weapons such as clubs, bows, arrows and for other general purposes.

Poison: A mucilaginous bark preparation is used by women against.

1.7 Medicinal importance

Leaves and twigs of G. tenax are important components of folk medicine for the treatment of trachoma, tonsillitis, infections and are used as a poultice to treat swelling. Because of its high iron contents, fruits of G.tenaxare often used in special diets for pregnant women and anemic children. G. tenaxplant is used for the treatment and prevention of iron deficiency anaemia. Porridge,calledNesha, is prepared by boiling fruit pulp of G. tenax and millet flour given to lactating mothers. (Aschers, & Schwf.2004). Ointment of whole plant extract applied locally for hard tissue repair and bark paste of G. tenax can be applied as plaster .(Jaiswal S, Singh SV, Singh B, & Singh HN.2004). A preparation of G. tenaxfruit powder mixed with milk is given for the treatment of bone fracture and swelling(Shekhawat D,& BatraA.2006).

1.8 Sodium:

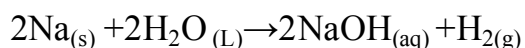
Sodium is metallic element found in the first group of the periodic table. As the sixth most abundant element in the Earth's crust, sodium compounds are commonly found dissolved in the oceans, in minerals, and even in our body. Sodium was discovered and isolated in 1807 by Sir Humphry Davy. In its pure form it is silvery white and soft enough to cut with a knife. Occurring in large amounts in both (sea) water and soil in various mineral compounds, the most common of which is sodium chloride.

1.8.1 Chemistry:

Sodium is highly reactive and is never found pure in nature. It tarnishes easily and has a low melting point and density. Therefore, sodium is usually stored in mineral oil or kerosene. Chemically, sodium is highly reactive with the halogen family to form ionic salts. For example, sodium is commonly found combined with chloride to form NaCl, known to us as table salt. It has one electron in the outermost electron shell and thus wants to give up one electron to a highly electronegative element.

Within the alkali metals, sodium is more reactive than lithium but less reactive than potassium. Its ionization energy is higher than potassium but lower than lithium. Sodium is also more metallic than lithium. When exposed to air, sodium oxidizes immediately. When burned, sodium forms sodium peroxide (Na_2O_2) or oxide (NaO_2). If burned in oxygen under pressure, sodium superoxide (NaO_2) is formed.

Sodium reacts exothermically with water, releasing heat when in contact with water. The chemical equation:



1.8.2 Compounds:

Sodium is frequently reacted with other elements to form useful compounds. Some important sodium compounds include table salt (NaCl), baking soda (NaHCO_3), sodium nitrate (NaNO_3), and sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_2 \cdot 5\text{H}_2\text{O}$). These compounds are used in the chemical, glass, metal, paper, petroleum, soap, and textile industries.

Sodium tends to form water-soluble compounds, such as halides, sulfate, nitrate, carboxylates and carbonates. But examples of many insoluble sodium compounds such as sodium bismuthate (NaBiO_3), sodium octamolybdate ($\text{Na}_2\text{Mo}_8\text{O}_{25} \cdot 4\text{H}_2\text{O}$), sodium thioplatinate ($\text{Na}_4\text{Pt}_3\text{S}_6$), and sodium uranate (Na_2UO_4) is found in nature.

1.8.3 Occurrence:

Sodium is found as a compound in nature due to its high reactivity. It is found in a variety of minerals, but the most common is table salt (NaCl), largely found in seawater and in solid deposits. Other compounds include amphibole, cryolite, soda niter and zeolite. Within the Earth's crust, sodium is the sixth most abundant element overall and the most abundant alkali metal. It makes up about 2.6% of the Earth's crust by weight.

Sodium is also abundant in stars, as the D spectral lines of this element are the most prominent in star light. Its presence in the atmosphere of planet Mercury was detected by ground telescopes and confirmed by spacecraft Mariner 10 and MESSENGER because of its high abundance and intense spectral lines (Eagleson, 1994).

1.9 Calcium:

Calcium is a chemical element with symbol Ca and atomic number 20. Calcium is a soft gray Group 2 alkaline earth metal, fifth-most-abundant element by mass in the Earth's crust. The ion Ca^{2+} is also the fifth-most-abundant dissolved ion in seawater by both molarity and mass, after sodium, chloride, magnesium, and sulfate (Dickson, 1994). Free calcium metal is too reactive to occur in nature. Calcium is produced in supernova nucleosynthesis.

Calcium is essential for living organisms, particularly in cell physiology where movement of the calcium ion into and out of the cytoplasm functions as a signal for many cellular processes. As a major material used in mineralization of bone, teeth and shells, calcium is the most abundant metal by mass in many animals.

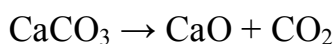
1.9.1 Chemistry and compound:

Most calcium salts are colorless. As with magnesium salts and other alkaline earth metal salts, to some degree soluble in water. Calcium is the main problematic ion in hard water, and reacts with soap to form soap scum and clogs plumbing with various minerals.

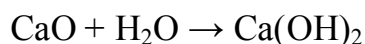
Calcium chemistry is almost exclusively that of Ca^{2+} salts (Earnshaw & Alan, 1997). Ca^{2+} is a "hard cation", that is, it characteristically favors oxide ligands. Hence the abundance of carbonates, nitrates, phosphates, and sulfates. Most of these species crystallize with water. Because it is generally nontoxic and abundant, calcium is found in many useful materials and minerals.

Combined with phosphate, calcium forms hydroxylapatite ($\text{Ca}_5(\text{PO}_4)_3(\text{OH})$), the mineral portion of animal bones, teeth, and some corals (Hu *et al*, 2001) Large-scale chemical processes are involved in the conversion of calcium phosphate minerals into fertilizer.

Combined with carbonate, calcium forms calcium carbonate (CaCO_3), which occurs naturally as limestone and chalk. When water percolates through limestone or other calcium-containing rocks, it partially dissolves the rock. The slow re-precipitation of this dissolved calcium leads to formation of stalactites and stalagmites. Upon heating above $825\text{ }^\circ\text{C}$, calcium carbonate converts calcium oxide (CaO), also known as quicklime:



When added to water, quicklime vigorously reacts (hence its name) to form calcium hydroxide:



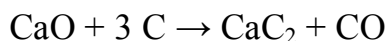
Also known as slaked lime, $\text{Ca}(\text{OH})_2$ is an inexpensive base material used throughout the chemical industry. When mixed with sand, it hardens into a mortar and is turned into plaster by carbon dioxide uptake. Mixed with other compounds, lime forms an important part of Portland cement.

Combined with sulfate, calcium forms the mineral gypsum. When heated to about $300\text{ }^\circ\text{F}$ ($150\text{ }^\circ\text{C}$), it undergoes partial dehydration (.Franz, 2012).

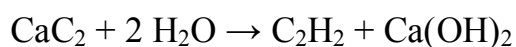


The resulting powder, when mixed with water, forms a stiff but workable paste that hardens to give Plaster of Paris.

Organocalcium compounds, those containing Ca-C bonds are known, but generally of specialized interest in the research laboratory. One major exception is calcium carbide, which arises from heating calcium compounds with coal or other carbon-rich reducing agents.



With the formula CaC_2 , it was historically important precursor to acetylene.



Other important calcium compounds are calcium nitrate used in fertilizers, calcium

chloride used as for deicing roads, calcium cyanamide, and calcium hypochlorite, used for bleaching.

Focusing on chemical structure, Ca^{2+} is a relatively large ion that tends to adopt a high coordination number. In CaF_2 , the mineral fluorite, each Ca^{2+} ion is surrounded by eight F^- ligands.

1.9.2 Occurrence:

Calcium is not naturally found in its elemental state. Calcium occurs most commonly in sedimentary rocks in the minerals calcite, dolomite, and gypsum. It also occurs in igneous and metamorphic rocks chiefly in the silicate minerals: plagioclases, amphiboles, pyroxenes, and garnets.

1.10 Iron:

Iron is a chemical element with symbol Fe (from Latin: ferrum, ultimately from ferre to bear or carry) and atomic number 26. It is a metal in the first transition series. It is by mass the most common element on Earth, forming much of Earth's outer and inner core. It is the fourth most common element in the Earth's crust. Its abundance in rocky planets like Earth is due to its abundant production by fusion in high-mass stars.

Iron plays an important role in biology, forming complexes with molecular oxygen in hemoglobin and myoglobin; these two compounds are common oxygen transport proteins in vertebrates. Iron is also the metal at the active site of many important redox enzymes dealing with cellular respiration and oxidation and reduction in plants and animals. A human male of average height has about 4

grams of iron in his body, a female about 3.5 grams. This iron is distributed throughout the body in hemoglobin, tissues, muscles, bone marrow, blood proteins, enzymes, ferritin, hemosiderin, and transport in plasma (Kohl & Walter, 1995).

1.10.1 Occurrence:

Iron is the sixth most abundant element in the Universe, and the most common refractory element (Dyson & Freeman, 1979). It is formed as the final exothermic stage of stellar nucleosynthesis, by silicon fusion in massive stars (Woosley & Janka, 2006). Metallic or native iron is rarely found on the surface of the Earth because it tends to oxidize, but its oxides are pervasive and represent the primary ores. While it makes up about 5% of the Earth's crust, both the Earth's inner and outer core are believed to consist largely of an iron-nickel alloy constituting 35% of the mass of the Earth as a whole. Iron is consequently the most abundant element on Earth, but only the fourth most abundant element in the Earth's crust (John *et al*, 1980). Most of the iron in the crust is found combined with oxygen as iron oxide minerals such as hematite (Fe_2O_3) and magnetite (Fe_3O_4). Large deposits of iron are found in banded iron formations. These geological formations are a type of rock consisting of repeated thin layers of iron oxides alternating with bands of iron-poor shale and chert. The banded iron formations were laid down in the time between 3,700 million years ago and 1,800 million years ago (Lyons & Reinhard 2009).

1.10.2 Chemistry and compounds:

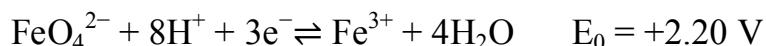
Iron forms compounds mainly in the +2 and +3 oxidation states. Traditionally, iron (II) compounds are called ferrous and iron (III) compounds ferric. Iron also occurs

in higher oxidation states, an example being the purple potassium ferrate (K_2FeO_4) which contains iron in its +6 oxidation state, although this is very easily reduced. Iron (IV) is a common intermediate in many biochemical oxidation reactions. Numerous organometallic compounds contain formal oxidation states of +1, 0, -1, or even -2. The oxidation states and other bonding properties are often assessed using the technique of Mössbauerspectroscopy. There are also many mixed valence compounds that contain both iron(II) and iron(III) centers, such as magnetite and Prussian blue ($Fe_4(Fe[CN]_6)_3$). The latter is used as the traditional "blue" in blueprints (Nam & Wonwoo, 2007).

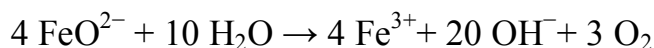
Iron is the first of the transition metals that cannot reach its group oxidation state of +8, although its heavier congeners ruthenium and osmium can, with ruthenium having more difficulty than osmium (Holleman *et al*, 1985). While iron's most common oxidation states are +2 and +3, ruthenium's is +3 and osmium's is +4. Iron also commonly forms aqueous cations in the +2 and +3 oxidation states, which is possible for ruthenium but not osmium. Some canary-yellow powder sits, mostly in lumps, on a laboratory watch glass.

Hydrated iron(III) chloride, also known as ferric chloride The iron compounds produced on the largest scale in industry are iron(II) sulfate ($FeSO_4 \cdot 7H_2O$) and iron(III) chloride ($FeCl_3$). The former is one of the most readily available sources of iron(II), but is less stable to aerial oxidation than Mohr's salt ($(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$). Iron(II) compounds tend to be oxidized to iron(III) compounds in the air. Unlike many other metals, iron does not form amalgams with mercury. As a result, mercury is traded in standardized 76 pound flasks (34 kg) made of iron (Reiffet *al*, 1984). Iron is by far the most reactive element in its group; it is pyrophoric when finely divided and dissolves easily in dilute acids, giving Fe^{2+} . However, it does not react with concentrated nitric acid and other oxidizing acids due to the formation of an impervious oxide layer, which can nevertheless

react with hydrochloric acid (Holleman et al, 1985). The standard reduction potentials in acidic aqueous solution for some common iron ions are given below (Holleman et al, 1985):



The red-purple ferrate (VI) anion is such a strong oxidizing agent that it oxidizes nitrogen and ammonia at room temperature, and even water itself in acidic or neutral solutions (Ware & Mike (1999):



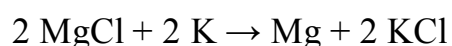
1.11 Potassium:

Potassium is a chemical element with symbol K (derived from Neo-Latin, kalium) and atomic number 19. It was first isolated from potash, the ashes of plants, from which its name derives. In the Periodic table, potassium is one of seven elements in column (group) 1 (alkali metals). All elements in Group 1 have a single valence electron in the outer electron shell, which is easily removed to create an ion with a positive charge – a cation, which combines with anions to form salts. Potassium in nature occurs only in ionic salts. Elemental potassium is a soft silvery-white alkali metal that oxidizes rapidly in air and reacts vigorously with water, generating sufficient heat to ignite hydrogen emitted in the reaction and burning with a lilac-colored flame. It is found dissolved in sea water (which is 0.04% potassium by weight (Webb 1939).

1.11.1 Chemistry:

Potassium is an extremely active metal that reacts violently with oxygen and water in air. With oxygen it forms potassium peroxide, and with water potassium forms

potassium hydroxide. The reaction of potassium with water is dangerous because of its violent exothermic character and the production of hydrogen gas. Hydrogen reacts again with atmospheric oxygen, producing water, which reacts with the remaining potassium. This reaction requires only traces of water; because of this, potassium and the liquid sodium-potassium — NaK — are potent desiccants that can be used to dry solvents prior to distillation. Because of the sensitivity of potassium to water and air, reactions with other elements are possible only in an inert atmosphere such as argon gas using air-free techniques. Potassium does not react with most hydrocarbons such as mineral oil or kerosene (Holleman *et al*, 1985). It readily dissolves in liquid ammonia, up to 480 g per 1000 g of ammonia at 0 °C. Depending on the concentration, the ammonia solutions are blue to yellow, and their electrical conductivity is similar to that of liquid metals. In a pure solution, potassium slowly reacts with ammonia to form KNH₂, but this reaction is accelerated by minute amounts of transition metal salts. Because it can reduce the salts to the metal, potassium is often used as the reductant in the preparation of finely divided metals from their salts by the Rieke method (Rieke, (1989). For example, the preparation of magnesium by this method employs potassium as the reductant:



1.11.2 Compounds

The only common oxidation state for potassium is +1. Potassium metal is a powerful reducing agent that is easily oxidized to the monocation, K⁺. Once oxidized, it is very stable and difficult to reduce back to the metal (Dye, 1979). Potassium hydroxide reacts readily with carbon dioxide to produce potassium carbonate, and is used to remove traces of the gas from air. In general, potassium compounds have excellent water solubility, owing to the high hydration

energy of the K^+ ion. The potassium ion is colorless in water and is very difficult to precipitate; possible precipitation methods include reactions with sodium tetraphenylborate, hexachloroplatinic acid, and sodium cobaltinitrite (Holleman *et al*, 1985). Potassium oxidizes faster than most metals and forms oxides with oxygen-oxygen bonds, as do all alkali metals except lithium. Three species are formed during the reaction: potassium oxide, potassium peroxide, and potassium superoxide (Lide & David, 1998). formed of three different oxygen-based ions: oxide (O^{2-}), peroxide (O_2^{2-}), and superoxide (O_2^-). The last two species, especially the superoxide, are rare and are formed only in reaction with very electropositive metals; these species contain oxygen-oxygen bonds. All potassium-oxygen binary compounds are known to react with water violently, forming potassium hydroxide. This compound is a very strong alkali, and 1.21 kg of it can dissolve in as little as a liter of water (Lide, (2005). Potassium compounds are typically highly ionic and thus most of them are soluble in water. The main species in water solution are the aquated complexes $[K(H_2O)_n]^+$ where $n = 6$ and 7 . Some of the few poorly soluble potassium salts include potassium tetraphenylborate, potassium hexachloroplatinate, and potassium cobaltinitrite (Holleman *et al*, 1985).

1.11.3 Occurrence

The element is widely distributed in Nature, being the seventh in abundance order. The ocean water contains about 0,07% of potassium chloride, and it is necessary to evaporate 98% of this to make potassium salts start to crystallize. Potassium can be found in many volcanic rocks, which by disintegration supply the element to the soils and the water. The silicate leucite and glauconite are the commercial sources of potassium. The clays can absorb potassium retaining it in the soil and allowing the plants to use it. The main potassium ores are: the sylvite (KCl), the carnalite ($KCl \cdot MgCl_2 \cdot 6H_2O$), the langbeinite ($K_2SO_4 \cdot 2MgSO_4$), and the polyhalite ($K_2SO_4 \cdot MgSO_4 \cdot 2CaSO_4 \cdot 2H_2O$) that can be found in Germany, USA, Canada,

Russia, Palestine, Congo, Ethiopia, Brazil and Bolivia.

1.12 Methods of analysis of element:

The most common form of elemental analysis, CHN analysis, is accomplished by combustion analysis. In this technique, a sample is burned in an excess of oxygen and various traps, collecting the combustion products: carbon dioxide, water, and nitric oxide. The masses of these combustion products can be used to calculate the composition of the unknown sample. Modern elemental analyzers are also capable of simultaneous determination of sulfur along with CHN in the same measurement run (Sahu *et al*, 2011).

1.12.1 Quantitative analysis:

Quantitative analysis is the determination of the mass of each element or compound present. Other quantitative methods include Gravimetry, where the sample is dissolved and then the element of interest is precipitated and its mass measured or the element of interest is volatilized and the mass loss is measured. Optical atomic spectroscopy, such as flame atomic absorption, graphite furnace atomic absorption, and inductively coupled plasma atomic emission spectroscopy, which probe the outer electronic structure of atoms. Neutron activation analysis which involves the activation of a sample matrix through the process of neutron capture. The resulting radioactive target nuclei of the sample begin to decay, emitting gamma rays of specific energies that identify the radioisotopes present in the sample. The concentration of each analyte can be determined by comparison to an irradiated standard with known concentrations of each analyte.

1.12.2 Qualitative analysis:

To qualitatively determine which elements exist in a sample, the methods are:

spectrometric atomic spectroscopy, such as inductively coupled plasma mass spectrometry, which probes the mass of atoms. Other spectroscopy which probes the inner electronic structure of atoms such as X-ray fluorescence, particle-induced X-ray emission, X-ray photoelectron spectroscopy, and Auger electron spectroscopy.

1.12.3 Spectrophotometry

Spectrophotometry is the quantitative measurement of the reflection or transmission properties of a material as a function of wavelength (Allen, 2010). It is more specific than the general term electromagnetic spectroscopy in that spectrophotometry deals with visible light, near-ultraviolet, and near-infrared, but does not cover time-resolved spectroscopic techniques. Spectrophotometry uses photometers that can measure a light beam's intensity as a function of its color (wavelength) known as spectrophotometers. Important features of spectrophotometers are spectral bandwidth (the range of colors it can transmit through the test sample), the percentage of sample-transmission, the logarithmic range of sample-absorption, and sometimes a percentage of reflectance measurement. A spectrophotometer is commonly used for the measurement of transmittance or reflectance of solutions, transparent or opaque solids, such as polished glass, or gases. However they can also be designed to measure the [diffusivity](#) on any of the listed light ranges that usually cover around 200 nm - 2500 nm using different controls and [calibrations](#) (Allen, 2010). Within these ranges of light, calibrations are needed on the machine using standards that vary in type depending on the [wavelength](#) of the photometric determination (Schwedt & Georg, 1997). Atomic spectroscopic methods are used for the qualitative and quantitative determination of more than 70 elements. Typically, these methods can detect parts-per-million to parts-per-billion amounts, and in some cases, even smaller concentrations. Atomic spectroscopic methods are also rapid, convenient,

and usually of high selectivity. These methods can be divided into two groups; optical atomic spectrometry¹ and atomic mass spectrometry. Spectroscopic determination of atomic species can only be performed on a gaseous medium in which the individual atoms or elementary ions, such as Fe¹, Mg¹, or Al¹, are well separated from one another. Consequently, the first step in all atomic spectroscopic procedures is atomization, a process in which a sample is volatilized and decomposed in such a way as to produce gas-phase atoms and ions. The efficiency and reproducibility of the atomization step can have a large influence on the sensitivity, precision, and accuracy of the method. In short, atomization is a critical step in atomic spectroscopy (skooget *al*, 2014).

1.12.4 Flame emission

1.12.4.1 ATOMIC EMISSION-THE FLAME TEST

When a small amount of a solution of a metal ion is placed in the flame of a Bunsen burner, the flame turns a color that is characteristic of the metal ion. A sodium solution gives a yellow color, a potassium solution results in a violet color, a copper solution gives a green color, etc. Such an experiment, called the flame test, has been used in conjunction with other tests in many qualitative analysis schemes for metal ions. Whatever color our eye perceives indicates what metal ion is present. When more than one metal ion is present, viewing the flame through a colored glass filter can help mask any interference. Figure 1.1 shows this experiment.

The phenomenon just described is an "atomic emission" phenomenon. This statement may seem inappropriate, since it is a solution of metal ions (and not atoms) that is tested. The reason for calling it atomic emission lies in the process occurring in the flame. One of the steps of the process is an atomization step. That is, the flame converts the metal ions into atoms. When a solution of sodium chloride is placed in a flame, for example, the solvent evaporates, leaving behind

solid crystalline sodium chloride. This evaporation is then followed by the dissociation of the sodium chloride crystals into individual ground state atoms - a process that is termed atomization. Thus sodium atoms are actually present in the flame at this point rather than sodium ions, and the process of light emission actually involves these atoms rather than the ions .

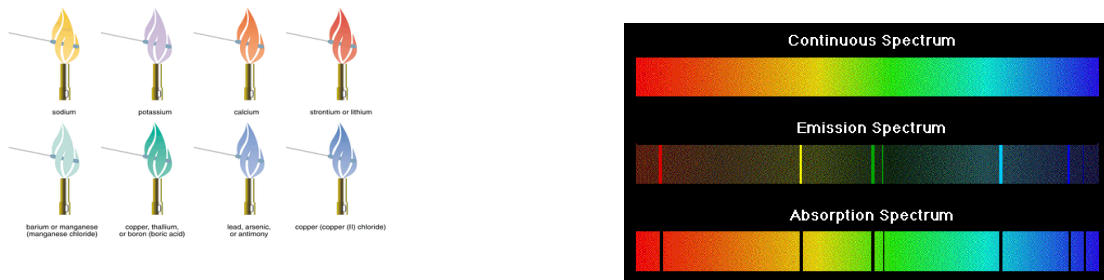


Figure 1.1 The flame test: observation of the flame color (a) without colored glass and (b) through prism

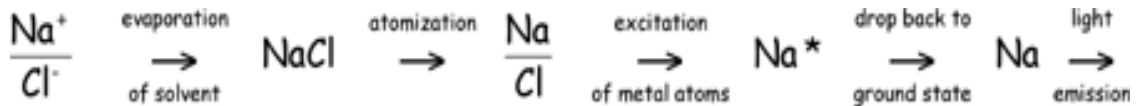


Figure 1.2 . The sequence of events occurring in the flame test.

As with fluorescence, the atomic emission is a result of electrons dropping from an excited state to lower states. The difference is that (1) atoms are involved here, rather than molecules, and (2) light is not absorbed prior to this atomic emission. Following atomization, a small percentage of the atoms absorb sufficient energy from the flame (as opposed to a light beam) so as to be promoted to an excited state. As with molecules in fluorescence, these atoms quickly return to a lower state, and light corresponding to the energy that is lost in the process is generated. It is this light that our eye perceives. The complete sequence of events is depicted in Figures 2 and 3.

1.12.4.2 FLAME PHOTOMETRY

The transition from the simple flame test to an instrumental technique is easily described. In the instrumental technique of flame photometry (FP), a monochromator replaces the colored glass filter, and a photocell detector/readout replaces our eye. Also, the burner design is more sophisticated in that the sample is continuously fed into the flame by aspiration. A schematic of this design is shown in Figure 1.3.

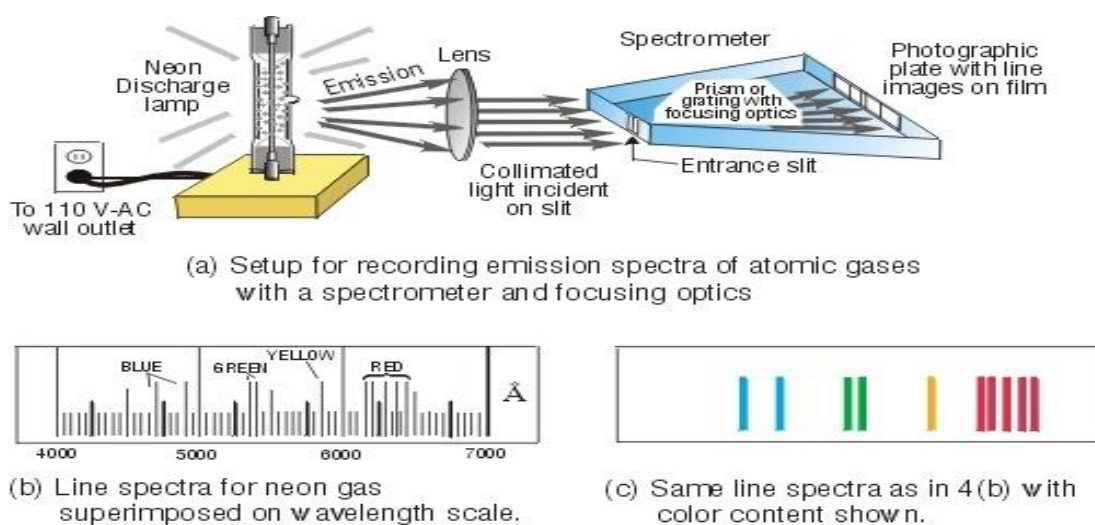


Figure 1.3 A schematic diagram of a flame photometer.

Since each element emits its own characteristic line spectrum, qualitative analysis can be performed here by observing what wavelengths are emitted and comparing these with various standards. However, since the detector is capable of measuring light intensity, quantitative analysis, as well as qualitative analysis, is possible. The intensity of the emitted light increases with concentration, and the relationship is usually linear:

$$I = K c$$

Thus, unknown concentrations can be determined by comparison with one or a series of standards in the same manner as described for the molecular techniques in UV-Vis Absorption spectroscopy.

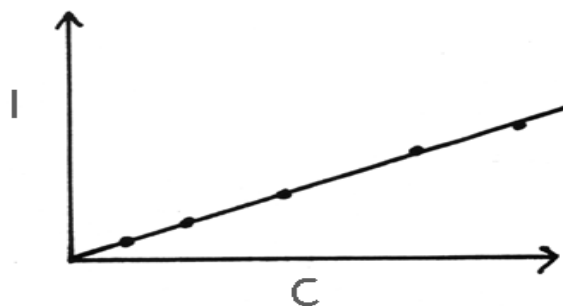


figure 1.4 A plot of intensity (I) vs concentration (C) representing quantitative analysis.

In short, flame photometry (FP) is an atomic technique which measures the wavelength and intensity of light emitted by atoms in a flame resulting from the drop from the excited state (formed due to absorption of energy from the flame) to lower states. No light source is required since the energy imparted to the atoms comes from the flame. Thus, FP is different from atomic absorption spectroscopy (AA), which is described in the next section.

1.13 Objectives of this work:

The objectives of this work are:

- * To determine some elements that occurs in GrewiaTenax including the sample preparation using dry ashing method.
- * Getting through spectrophotometric methods of analysis using advance instruments such as Flame photometer, spectrophotometer and some others instruments.

Chapter Two

Materials and methods

2. Materials and methods

2.1 Materials and equipments:

2.1.1 Materials

Concentrate Sulfuric acid 98%.

Sodium hydroxide 40%.

Boric acid 2%.

Methyl red indicator,

Coppersulphate.sodiumsulphate (1:9).

Potassium permanganate,

Concentrate hydrochloric acid.

Nitric acid.

Ptassiumthiocyanate.

2.1.2 Apparatus

Spectrophotometer (UK, Genway 6505).

Flame photometer (UK, Enway, pfp7).

Fume cupboard.

Markham distillation.

Kjedahl flask.

Oven.

Burette.

Pasture pipette.

Measuring cylinder.

Beakers (100).

volumetricflask (100ml).

Analytical balance (Japan A&D INSTRUMENT GR120).

2.2 Methods:

2.2.1 Sample preparation:

Preparation of raw materials: Guddaim fruits were collected from a valley local market in Khartoum. The experiment was carried out during the year 2016.

2.2.2 Chemical analysis of the sample:

2.2.2.1 Ash content:

15.083g of the sample was burned in oven at 600° C , until removed all carbon from organic matter in form of carbon dioxide, water which produced from this heat and remaining of burning were weighted of ash . The percent of ash was found to be as shown below:

Table 2.1

Weight of crucible/g	Weight of crucible and ash/g	Weight of ash/g
44.668	45.277	0.609

$$ash = \frac{w \text{ of g of ash}}{w \text{ of g of sample}} 100$$

$$ash = \frac{0.609}{15.083} 100 = 4.04\%$$

2.2.2.2 Determination of protein:

Crude protein determined by measuring the nitrogen content of the feed multiplying it by a factor of 6.25 this factor is based on the fact that most protein contain 16% nitrogen.

Method Involve: digestion , distillation and titration .

Digestion:

0.2113g of sample was weighted into kjedahl flask ,3ml of sulfuric acid conc. added, 0.1g of coppersulphate, and 0.9g of sodium sulphate were added ,heated in a fume cupboard for 15 min .until the digesta become clear pale green, cooled and rapidly 100ml distilled water added .

Distillation:

The distillation apparatus steam up ,3ml of digest added into apparatus via a funnel and allowed it to boil , 10ml sodium hydroxide added by measuring cylinder (to not loss ammonia) ,distilled into 50ml of 2% boric acid containing screened M.R indicator .

Titration :

The alkaline ammonium borate titrated directly with 0.1M HCL, the volume recorded.

Result =3.5ml

Calculation:

$$\%N = \left(\frac{TF \times 0.014 \times 6.25 \times 0.1}{W_{\text{(of sample /g)}}} \right) \times 100$$

TF ≡ titration figure ,0.014 : nitrogen and 6.25 constant factor for protein

$$\%N = \left(\frac{TF \times 0.014 \times 6.25 \times 0.1}{0.2113} \right) \times 100 = 14.5\%$$

2.2.2.3 Determination of metals:

2.2.2.3.1 Sample preparation for metals analysis:

The ash obtained from step 2.2.2.1 was dissolved with hydrochloric acid, converted to 100ml volumetric flask and completed up to mark with distilled water.

1) Determination of iron:

0.076g of ammonium ferrous sulphate was weighted into 100ml beaker, dissolved with small portion of water and sulfuric acid and oxidized to Fe^{+3} using potassium permanganate converted to 100ml volumetric flask and completed to mark with distilled water. The solving solutions were prepared using Solution of sample, KSCN and HNO_3 , then completed up to the mark with distilled water as shown in table 2.2:

Table (2.2)

C/Ppm	0	2	4	6	8	10
Fe^{+3}	0	1	2	3	4	5
HNO_3	5	5	5	5	5	5
SCN	3	3	3	3	3	3
Sample	1	1	1	1	1	1

And the adsorption was measured at 480 nm. The results was recorded at Table 2.3

Table (2.3)

Concentration ppm	absorption
0	0.001
2	0.109

4	0.127
6	0.151
8	0.179
10	0.181

$$\%Fe = \left(\frac{\text{ppm} \times v \times df \times 10^{-4}}{15.083} \right) \times 100 = 0.22\%$$

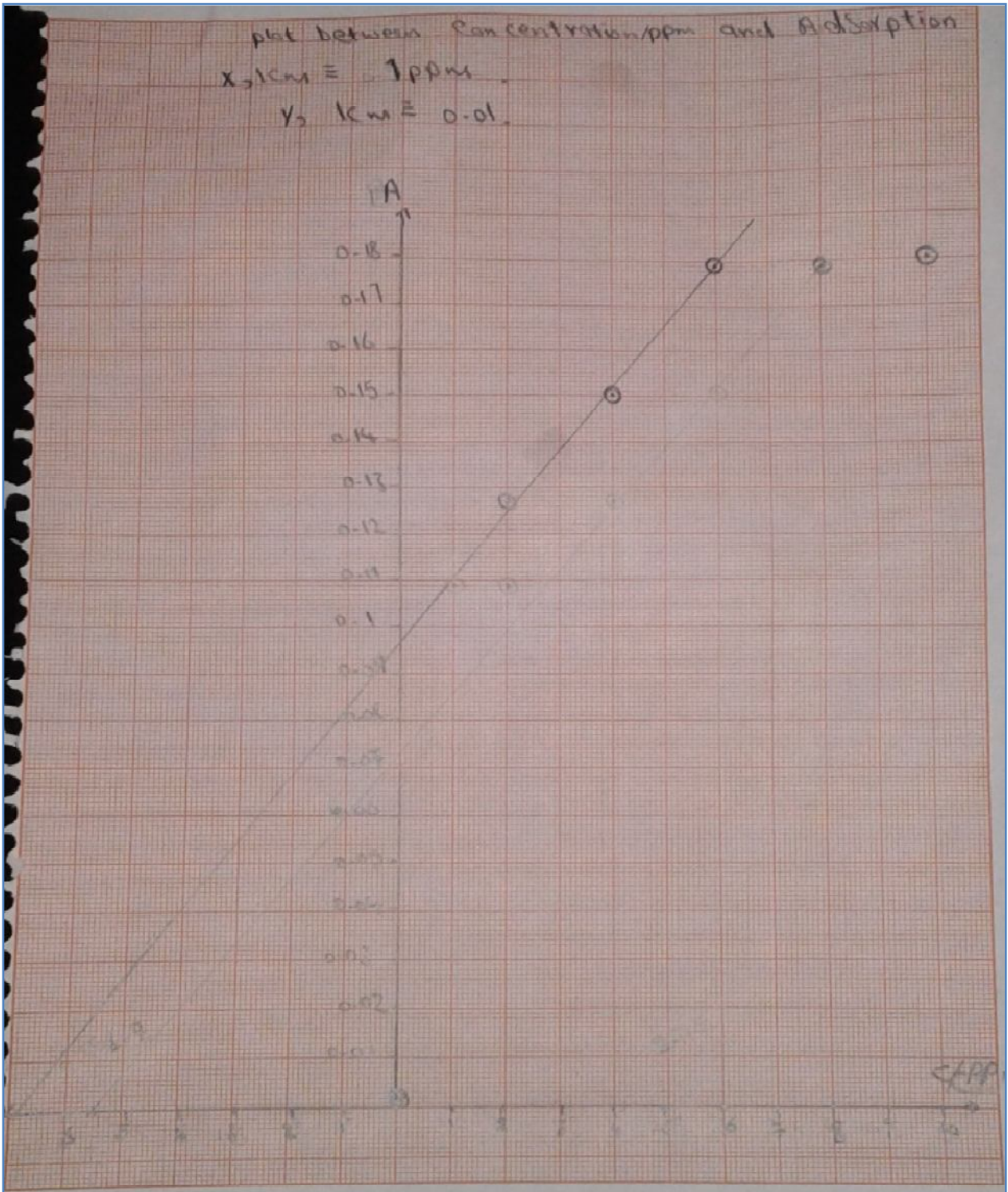


Figure (2.1) absorbance of Fe^{+3} against concentration

2. Determination of sodium:

1000ppm of solution of sodium was prepared, from which 0,2,4,6,8 and 10 ppm were prepared in 50 ml volumetric flask, the emission was measured, 5ml of the sample was transferred to 10 ml volumetric flask filled up to the mark with D.W , the emission was measured and the results were recorded at Table 2.4.

Table (2.4)

C/ppm	Emission
0	0
2	11
4	21
6	32
8	39
10	50
Sample	28

$$\%Na = \left(\frac{5.5079 \times 100 \times 2 \times 10^{-4}}{15.083} \right) \times 100 = 0.0073\%$$

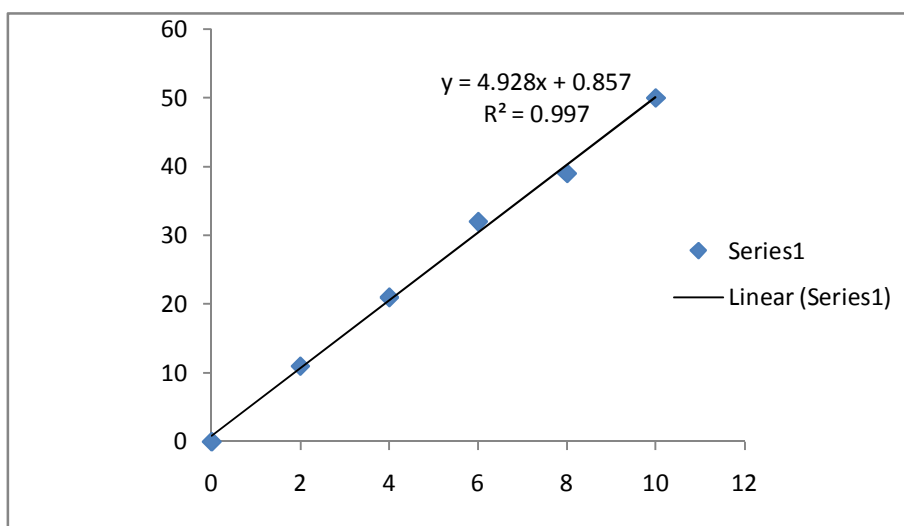


Figure (2.2) Emission of sodium against concentration

3)Determination of potassium:

1000ppm of solution of potassium was prepared, from which 0,2,4,6,8 and 10 ppm were prepared in 50 ml volumetric flask, the emission was measured, 1 ml of the sample was transferred to 100 ml volumetric flask filled up to the mark with D.W , 2 ml of this solution was diluted to 10 ml, the emission was measured and the results were recorded at Table 2.5

Table (2.5)

C/ppm	Emission
0	0
2	10
4	20
6	31
8	40
10	51
Sample	16

$$\% K = \left(\frac{3.127 \times 100 \times 500 \times 10^{-4}}{15.083} \right) \times 100 = 1.036\%$$

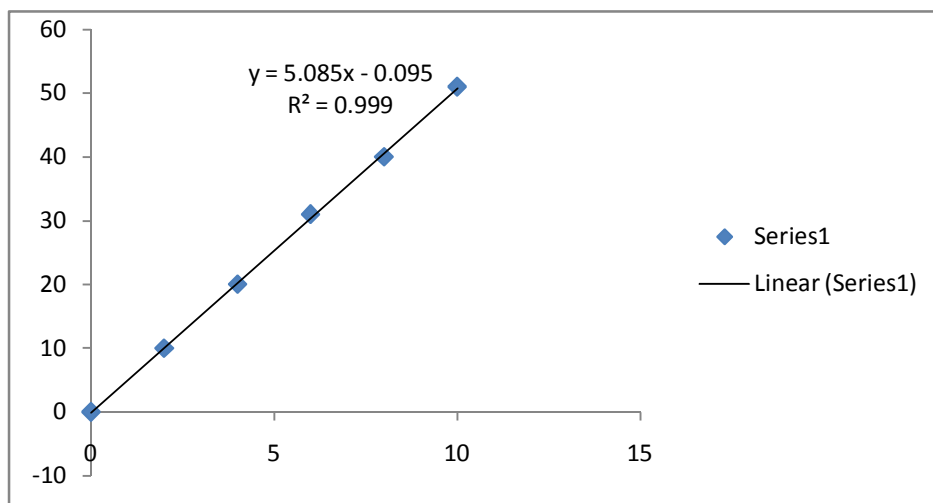


Figure (2.3) Emission of potassium against concentration

4) Determination of calcium:

1000ppm of solution of sodium was prepared, from which 0,2,4,6,8 and 10 ppm were prepared in 50 ml volumetric flask, the emission was measured, 5ml of the sample was transferred to 10 ml volumetric flask filled up to the mark with D.W , the emission was measured and the results were recorded at Table 2.6.

Table (2.6)

C/ppm	Emission
0	0
10	10
20	21
30	31
40	40
50	50
Sample	33

$$\% \text{Ca} = \left(\frac{32.667 \times 100 \times 20 \times 10^{-4}}{15.083} \right) \times 100 = 0.433\%$$

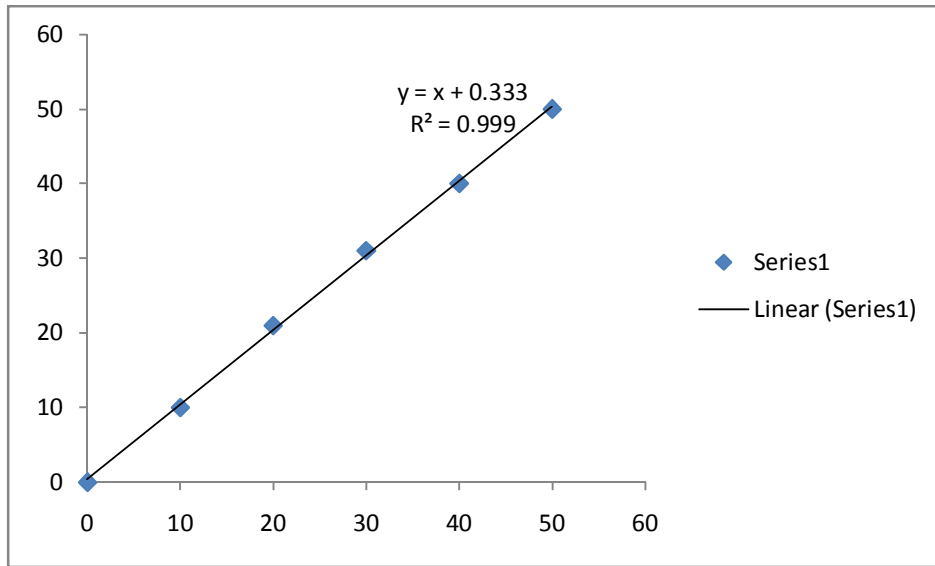


Figure (2.4) Emission of calcium against concentration.

Chapter Three

Discussion and recommendation

3. Discussion and Recommendation

Discussion:

The approximate chemical analysis indicated that guddaim fruits contained high amounts of crude protein, ash, sodium, potassium, calcium and iron. So that it use to treats many disease such as diarrhea, dysentery, rheumatism and anemia.

The sample of grewiateanax was prepared using dry ashing method in order to determine the nutrients of it such as protin and some of elements.

The percentages of ash, protein, calcium, iron, sodium and potassium in Grewia Tenax were found to be 4.04%, 14.5%, 0.433%, 0.22%, 0.0073% and 1.036% respectively.

The results shows that gewiateanax contains a high levels of Iron, calcium and potassium, so grewatenaks fruits may be used to treat the lake of this elements in human body.

Recommendation

Since the guddium fruit contains high amounts of, sodium, potassium, calcium and iron we recommended that may be used to by human to treat the deficits of these elements.

Calcium: The most important are that aids in maintaining bone health and dental health as well as prevention of colon cancer

Potassium: The health benefits of potassium include relief from stroke, blood pressure, kidney disorders, anxiety and stress. And iron for Anemia.

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