



Sudan University of Science & Technology

Collage of Graduate Studies

**Spectroscopic Investigations of Dry Fermented
Leaves of Cassia Obtusifolia (Kawal)**

دراسة طيفية لأوراق نبات الكول الجافة المخمرة

*A Dissertation Submitted as Partial Fulfillment of the Requirements for the
Degree of Master of Science in Physics*

By:

Abdallah Yousif Adam Ahmed

Supervisor:

Dr. Ali Abdel Rahman Saeed Marouf

December 2016

DEDICATION

*To my family
For their everlasting love, care, hope and dedication
For their patience and perseverance
Assuring my every success.*

To my loyal teachers,

Past, present and future.

To all my friends.

With love and respect

abdallah

Acknowledgement

First I would like to thank Allah with whose will this work has been completed. Thereafter, I would like to express my sincere thanks and gratitude to Dr. Ali Abdu ElrahmanMarouf for his kind supervision, advice and directing views throughout my study. I should also thank all my teachers and members of the Department of physics Science, for their constant encouragement and unlimited help. I am also indebted and thankful to all my payments we have been working very hard and putting immense effort into getting the very best results.

Abstract

The Sicklepod (*Cassia obtusifolia*) leaves after fermentation and drying are commonly known as Kawal. Fermented leaves of *Cassia obtusifolia* are used as a substitute of meat by people of eastern of Chad and western of Sudan. In this study kawal was obtained from Forbranga western of Darfour as a powder. The objective of this study was identification the chemical compositions of the Kawal. FTIR spectrum and atomic absorption spectrophotometer methods were used. In FTIR experimental the sample was used as dried powder. While for the atomic absorption and inductively coupled plasma the sample was used after combussed for extracting the minerals (K, P, Na, Mg, Ca, Zn and Fe).

In FTIR method the investigations was showed that the Fermented cassia obtusifolia (Kawal) is consist of several types of molecular groups: alky halide, alkene, nitro function, aromatic, carbonyl, alcohol, esters, ether, and acid and amides.

Minerals were extracted from the samples by the inductively coupled plasma spectroscopy method: About 2.50% was found is presence of calcium and the other minerals as: Mg 0.66%, P 0.393%, Na 0.165% and K about 1.595%. and for extracting the Iron (Fe) and Zinc (Zn) minerals of fermented cassia leaves the absorption atomic spectrophotometer was used and found that Zn was 0.665mg/L and Fe consist of the value 15.6411mg/L

مستخلص :

يعرف ناتج عملية تخمير اوراق نبات (Sicklepod (cassia obtusifolia و تجفيفها بالكول. بحيث يستخدم كمادة غذائية يصاحب منتجات اللحوم مع العصيدة والتي يتواجد بصورة شائعة في شرق دولة تشاد وغرب السودان. تم الحصول على الكول من مدينة فوربرنقا ولاية غرب دارفور في شكل بودرة. كان الغرض من هذه الدراسة تحديد التراكيب الكيميائية ودراسة كميات بعض المعادن في الكول. استخدمت في هذا البحث مطيافية فورير للأشعة تحت الحمراء (FTIR) ومطيافية الامتصاص الذري. في اختبار (FTIR) استخدم العينة في شكل بودرة جافة بينما في العمليتين الاخيرتين استخدمت العينة بعد تجضيرها بالحرق للحصول على المعادن (كالسيوم، بوتاسيم، صوديوم، مغنيزيوم، فسفور، خارصين والحديد).

أظهرت نتائج العملية التي استخدم فيها مطيافية (FTIR) عدة تراكيب كيميائية: الكي هاليدات، الكينات، استرات، اثيرات، كحول، أحماض، رابطة نيترات، رابطة كربونيلية، أحماض أمينية ورابطة أروماتية.

اما المعادن فأظهرت نتائج التحاليل العملية التي استخدم فيها طريقة الحث البلازمي المزدوج أن العينة تحتوي العناصر الغذائية التالية: كالسيوم 2.50%، صوديوم 0.165%، مغنيزيوم 0.66%، فوسفور 0.393% و بوتاسيوم 1.595%. بينما للحصول على نسب عنصري الحديد والزنك تم استخدام طيف الامصاص الذري بعد تحضير العينة فكانت نسبة وجود الزنك 0.665 mg/L والحديد 15.6411 بوحدة قياس mg/L.

Contents

Topic	Page
Dedications	i.
Acknowledgements	ii.
Abstract	iii.
Arabic abstract	iv.
List of contents	v.
List of Tables	vi.
List of Figures	vii.
Chapter One	
Introduction	1
Objective	2
Preface	3
Chapter Two	
2.1-Spectroscopy	4
2.1.1-Electromagnetic radiation	4
2.1.2-Principle of atomic spectroscopy	5

2.1.3-Classification of spectra	5
2.1.4-IR frequency range and spectrum presentation	7
2.1.5-Spectrum obtained	8
2.1.6-Atomic spectroscopy	8
2.1.7-Mass spectroscopy	10
2.1.8-Origin of spectra lines	10
2.1.9-Elements identification	11
2.1.10-The range of IR radiation	12
2.1.11-Requirements of IR radiation absorption	13
2.1.12-Theory	14
2.1.13-origin of IR spectra	14
2.1.14-Selection rules	16
2.1.15-Molecular vibrations	16
2.1.16-Instrumentation	18
2.1.17-Working of double beam	25
2.1.18-Advance spectrophotometer	28

2.1.19-Fourier transform IR spectroscopy	28
2.1.20-Non-dispersive Photometer	29
2.2-Alkawal	30
2.2.1-Sicklepod Senna Obtusifolia	30
2.2.2-Kawal preparation	31
2.2.3-Nutritive value of cassia Obtusifolia	32
2.2.4-Kawal as meat substitute	33
2.2.5-Chemical compositions of Cassia	33
Chapter Three	
3.1-Material and Apparatus	34
3.2-Methods	35
Chapter four	
4.1-Results& Discussions	37
4.2-Conclusions	40
4.3-Recomondations	40
References	

List of Tables:

Table 2.1: Components of double beam IR spectrophotometer	19
Table 2.2: Some optical materials with their approximation transition limits	21
Table 4.4: Shows Ca, Na, K, Mg and P content of kawal	39
Table 4.5: Shows Iron and Zinc content	40

List of Figures:

Figure 2.1: Regions of electromagnetic spectrum	5
Figure 2.2: Types of vibration modes	17
Figure 2.3 Optical system of double beam Spectrophotometer	25
Figure 2.4: Cassia obtusifolia plant's leaves	30
Figure 3.5: Sun dried powder of kawal	34
Figure 3.6: Fourier transformer spectrophotometer device	35
Figure 4.7: Kawal FTIR spectrum	38

Chapter One
Introduction and Literature Review

Chapter One

Introduction and Literature Review

1-1- Introduction

Cassia obtusifolia (family leguminous) is a wild African plant found in wastelands in the rainy season. Its leaves can be fermented (named kawal) and is used by people from the western part of Sudan and the eastern part of chad as meat replacer or meat extender. The role of kawal and the like is in providing the sauces which make these staples palatable. During famine years, kawal, a protein source, probably protected many children against kwashiorkor. Then as today, kawal was shunned by the elite who consider it unfit for modern social life because of its repugnant, fetid odor that lingers on the fingers for hours (algadi and yousif 2015).

Algadi and yousif, (2015) studied the anti-nutritional factors of green leaves of cassia obtusifolia and kawal (the dry fermented leaves). They studied the effect of fermentation on anti-nutritional factors on the both samples. They found the in vitro protein digestibility is increased from 49.43 to 61.87%, while their used technique was wheeler and ferrel method. They used the same steps for making kawal as explained above in introduction. Their samples were as adry.

Nuha, Isam and Alfadil, (2010) studied the changes on the chemical composition, anti-nutrients and extractable minerals of cassia leaves, kawal and cooking. Their samples were as a dry powder. Then found the protein and ash contents increased after cooked, the total energy of the both samples were decreased after fermentation process and cooked too, anti-nutritional factors (tannin, phytate, total polyphenols) were also decreased on the created samples, and the total major minerals was increased. Total polyphenols by using spectrophotometer method by price and Bulter(1977), minerals composition by Chapman and pratt(1982) and HCL extractability by using Chauhan and Mahjan (1988).

Mutasim Othman (2004) evaluated the effect of fermentation on nutritional quality of cassia obtusifolia and kawal (chemical composition, protein fractions, in vitro protein digestability, amino acid contents, mineral content, phytic acid, tannin and total polyphenol of cassia leaves). And then found the changes as coming:

Fat content increased from 3.50 to 4.50‰, protein content from 24.81 to 35.13‰, ash content from 13.67 to 18.00‰, in vitro protein digestability from 49.43 to 61.87‰ and the fiber content decreased from 13.04 to 12.90‰. hence his techniques used dry and powder samples the his results as follow: crude fiber by method of A.O.A.C (1984), fat content by A.O.A.C method, protein content determination by using microkjeldahl technique, ash content by muffle furnace, in vitro protein digestability according to saunder et al(1973) method, total polyphenol by price Butter(1977) and phytic acid content by wheeler and ferrel(1971).

Mbailao, and Bessiere (2005) studied the chemical components of kawal and found aliphatic acids and they identified as hexanoic acid (27‰), butyric acid(10.4‰), and valeric acid(6.3‰) by using laser amounts of p-ethylphenol and p-methylphenol. While they found the protein fraction from leaves as 20.2‰ in the crude leaves and 12.9‰ in the fermented leaves (kawal). They used dried samples and used gas chromatography and gas chromatography-mass for analyzing volatiles and then identified the constituents of volatiles by using comparison of mass spectra with authentic compounds. For amino acid analysis they hydrolysed the kawal and leaves under nitrogen and HCL vapor by using pico-tag work. They used chromatography for Amino acids analysis. Their results as mineral contents as sodium and potassium by using flame spectrophotometer by using butane while for the calcium and magnesium with an atomic absorption spectrophotometer(spectro-varian 20Bo), and for iron determination they used colorimeter method.

1-4- The Objective:

The objectives of this study was Spectroscopic studies for the Dry fermented Cassia Obtusifolia leaves

1-6- Preface:

This research is consists of four chapters. Chapter one contains introduction and literature review while the theoretical back rounds and some physical concepts around spectroscopic, infrared spectroscopic and fermented kawal as material obtain in chapter two. Chapter three constructs of experimental part for a dry powdered material and the method of process. Finally there are result and discussions on chapter four

Chapter Two
Theoretical Back ground

Chapter Two

Theoretical Back ground

2-1 Spectroscopy

2-1-1- Electromagnetic radiation:

From electricity studies it is known that moving electrical charges induce magnetic fields and inversely that changes of the magnetic field create an electric field. Vibrating electrical changes therefore cause a periodic change of electromagnetic fields.

The visible part of electromagnetic spectrum is, by definition radiation visible to the human eye. Other detection systems reveal radiation beyond the visible regions of the spectrum and these are classified as radio wave, micro wave, infrared, ultraviolet, x-rays and Gama rays. These regions are illustrated in figure 1.1 together with the processes involved in the interaction of the radiation of these regions with matter.

The nature of the various radiations shown in figure 1.1 have been interpreted by Maxwell's classical theory of electro- and magnat0- dynamics-hence, the term of electromagnetic radiation.

According to this theory radiation is considered as two mutually perpendicular electric and magnetic fields, oscillating in single planes

At right angles to each other .these fields are in phase and are being propagated as a sine wave. the electromagnetic radiation was that the velocity of propagation in the vacuum is constant for all regions of the spectrum .and this is

known as the velocity of the light c and has the value $3 \times 10^8 \text{ m/s}$ (Helmut Gremlish 2002).

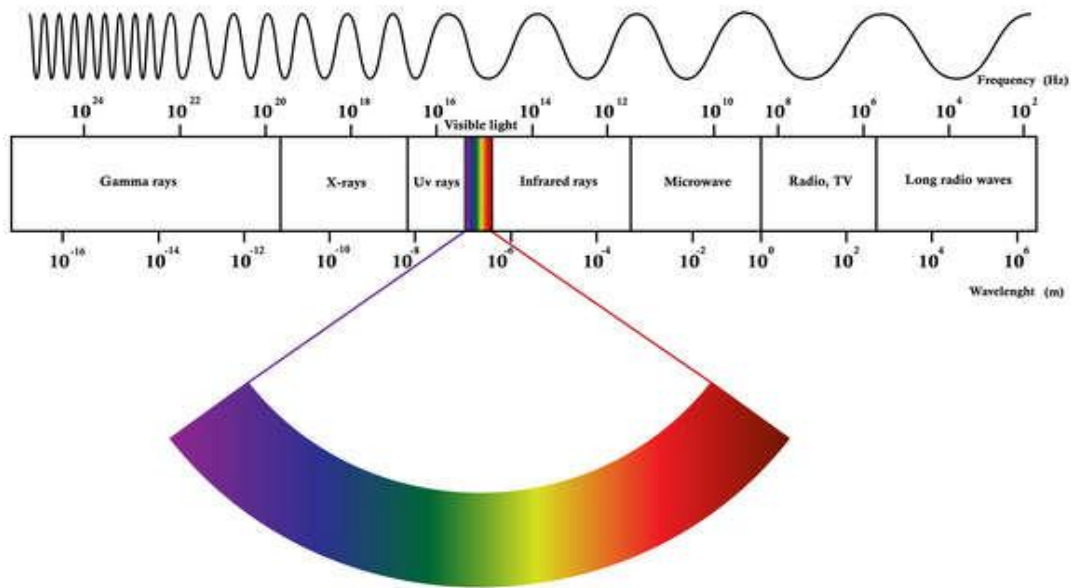


Figure 2.1: Explains the regions of electromagnetic spectrum.

2-1-2- Principle of atomic spectroscopy:

When a beam of polychromatic light is passed through a prism or grating, it splits up onto seven different colours. The set of the colours thus obtained is called a spectrum.

2-1-3- Classification of spectra:

There are two main classes of spectra namely emission spectrum and absorption spectrum.

Emission spectrum: when the light emitted by a substance is passed through a prism and examined directly with spectroscopy, the spectrum obtained is referred to as emission spectrum. Emission spectrum also classified according to their appearance as line, band and continuous spectrum.

-line or atomic spectrum:

This spectrum is obtained when the light emitting substance is in atomic state. Line spectrum consists of discrete wavelengths extended throughout the spectrum and is generally obtained from light sources.

-band spectrum:

Band spectrum arises when the emitter in the molecule state is excited (Hons 2001).

-continuous spectrum:

When the source emitting light is an incandescent solid, liquid or gas at a high temperature, then the spectrum so obtained is continuous. This type of spectrum is formed whenever the matter in a bulk is heated.

Absorption spectrum:

When a white light is passed through an absorbing substance and then observed through a spectroscopy, is found that certain colours are missing and dark lines appear at their places, the spectrum so obtained is called absorption spectrum,

these dark lines or bands depend upon the nature of absorbing material (Hons 2001).

Types of absorption spectrum:

-Line absorption spectrum:

In this type sharp dark lines will be observed when the absorbing substance is a vapour or gas.

-Band absorption spectrum: It is in the form of dark bands. While the third is a –

-continuous absorption spectrum which its spectrum arises when the absorbing material absorbs a continuous range of wavelengths.

2-1-4- IR frequency range and spectrum presentation

Infrared radiation spans a section of the electromagnetic spectrum having wavenumbers from roughly 13.000 to 10 cm, or wavelengths from 0.78 to 1000 micrometer. It is bound by the red end of the visible region at high frequencies and the microwave region at low frequencies

IR absorption positions are generally presented as either wavenumber or wavelengths. Wavenumber defines the number of waves per unit length .thus wavenumbers are directly proportional to frequencies. As well as the energy of the IR absorption. While the wavelengths are inversely proportional to frequencies and their associated energy.

2-1-5- Spectrum obtained

Spectrum can be used to obtain information about atomic and molecule energy levels, molecule geometries, chemical bonds, interactions of molecules and related processes often spectra are used to identify components of a sample and also used to measure the amount of materials in a sample (Hons 2001).

2-1-6- Atomic spectroscopy:

Atomic spectroscopy exploits different energetic transitions experienced by atoms that associated with either by absorption or emission of photons. When these transitions involve the excitation and relaxation of the valence shell electrons of metal atoms and ions, the corresponding photons have energies within the ultraviolet and visible regions of spectrum (Dr.Shardendu Kisala).

a - atomic absorption:

The energy absorbed to move an electron to more energetic level and or the energy emitted as the electron moves to less energetic energy level is in the form of photon. The wavelength of the emitted radiant energy is directly related to electronic transition which has occurred. Since every element has unique electronic structure, the wavelength of the light emitted is a unique property of each individual element. As the orbital configuration of a large atom may be complex, there are many electronic transitions which can occur, each transition resulting in the emission of the characteristic of the light.

If light of just the wright wavelength impinges on a free, ground state atom, the atom may absorb the light as it enters an excited in a process known as atomic absorption.

Atomic absorption measures the amount of the light at the resonant wavelength which is absorbed as it passes through a cloud of atoms. As the number of atoms in the light path increases, the amount of the light absorbed increases in a predictable way, by measuring the amount of light absorbed, a quantitative determination of the amount of analyte element present can be made. The use of special light sources and careful selection of the wavelength allow the specific quantitative determination of the individual elements in the presence of others .the atom cloud required for atomic absorption measurements is produced by supplying enough thermal energy to the sample to dissociate the chemical components into free atoms (Dr.Shardendu Kisala).

Atomic emission:

In atomic emission the sample is subjected to a high energy, the thermal environment in order to produce excited state atoms, capable of emitting light. The source energy can be an electrical, a flame, or more recently a plasma.

The emission spectrum of an element exposed to such an energy source consists of a collection of the allowable emission wavelengths, commonly called emission lines, because of the discrete nature of the emitted wavelengths. This emission spectrum can be used as a unique characteristic for qualitative identifications of the element.

Atomic emission using electrical arcs has been widely used in qualitative analysis. Emission techniques can also be used to determine how much of an element is present in a sample. For the quantitative analysis the intensity of the light emitted at the wavelength of the element to be determined is measured. The

emission intensity at this wavelength will be greater as the number of atoms of the analyte element increases.

Atomic fluorescence:

This technique incorporates aspects of both atomic absorption and atomic emission. Like atomic absorption ground state atoms created in a flame are excited by focusing a beam of light into the atomic vapor. Then the emission resulting from the decay of the atoms excited by the source of light is measured. The intensity of this fluorescence increases with increasing atom concentration providing the basis for quantitative determination.

The source lamp for atomic fluorescence is mounted at an angle to the rest of optical system, so that the light detector sees only the fluorescence in the flame and not the light from the lamp itself. While the atomic absorption is the most widely applied of the three techniques and usually offers several advantages over the other two, particular benefits may be gained with either emission or fluorescence in special analytical situations.

2-1-7- Mass spectroscopy:

Atomic mass spectroscopy is similar to other types of mass spectrometry in that it consists of an ion source, a mass analyser and detector.

Atoms identities are determined by their mass to charge ratio (mass analyser) and their concentrations are determined by the number of ions detected.

2-1-8- Origin of spectral lines:

It was necessary to explain how electrons are situated in atoms and why atoms are stable. The proposal of Bohr that the electron revolves around the nucleus of the atom with a definite fixed energy in a fixed path, without emitting or absorbing. The electron in the hydrogen atom exist only in certain definite levels. These energy levels called principal Quantum number n , denoted by the principal Quantum level $n = 1$ is closest to the nucleus of the atom and lowest energy. When the electron occupies the energy level of the lowest energy of the atom is said to be in its ground state. An atom can have only one ground state if the electron occupies one of the higher energy levels then the atom is in an excited state.

When a gaseous hydrogen atom in its ground state is excited by an input of energy, its electron is promote from the lowest energy level to one of the higher energy. Then the atom does not remain excited but re-emits energy as electromagnetic radiation. This is as result of an electron falling from a higher energy level to one of lower energy. This electron transition results in the release of a photon from the atom of the amount of energy ($E = h \nu$) equal to the difference in energy of the electronic energy levels involved in the transition. Then by using the prism can separate the emitted electromagnetic radiation into its component frequencies (wavelengths or energies) then these represented as spectral lines along an increasing frequency scale to form an atomic emission spectrum.

2-1-9- Element identification:

The energy levels in atoms and ions are the key to production and detection of light, energy levels or the shells exist for electrons in atoms and molecules .the colours of the dyes and other compounds results from electron jumps between

these shells. The colours of fireworks results of jumps of electrons from one shell to another. Observation of light emitted by the elements is also evidence for the existence of shells, subshells energy levels. The kinds of light that interact with atoms indicate the energy difference between shells and energy levels in quantum theory model of the atom.

The excited electron is still in the atom even in an excited. The valence electron will only scape the atom if the electron is given an amount of energy equal to the ionization energy for that atom.

Energy can be added to atoms many different ways. It can be in form of light, an electrical discharge or heat. This added energy is emitted when the excited electrons in the atoms give off light and fall back to lower shells. The light emitted has wavelengths and colours that depend on the amount of energy originally absorbed by atoms. Usually each individual excited atom will emit one type of light. Since we have billions of atoms we get billions of excitation and emissions.

All the atoms in a sample will absorb or be excited exactly the same. For example in hydrogen the ground state has the electron in $n = 1$ shell. The electron in hydrogen atoms may be excited into then $n = 2$ level. Other hydrogen atoms can have the electron excited into the $n = 4$. Different element emit different emission spectra when they are excited, because each type of element has unique energy shell or energy level system. Each element has a different set of emission colours because they have different energy level spacings. We will see the emission spectra or pattern of wavelengths emitted by six different elements in this lap. We will then identify an unknown element by comparing the colour of the unknown with the flame colour of our known (Kisala).

2-1-10- The range of infrared radiation:

The infrared radiation refers broadly to that part of electromagnetic spectrum which lies between the visible and microwave regions.

From instrument and infrared applications it divided onto:

- the photographic region: this range from visible to 0.8μ
- the near infrared regions: its ranges from 0.8 to 2.5μ
- the mid infrared regions: from 2.5 to 15
- the far infrared regions: from 15 to 200μ . (Hons 2001).

2-1-11- Requirements of infrared radiation absorption:

For a molecule to absorb infrared radiation it has fulfil certain requirements as fallow:

a- Correct wavelength of radiation:

a molecule absorb radiation only when the natural frequency of vibration of some part of molecule is the same as the frequency of the incident radiation. For instance the nature frequency of vibration of HCL molecule is about 2890 1/cm . when IR is passed through a sample of HCL and the transmitted radiation is analysed by IR spectrometer, it is observed that part of the radiation which has frequency 2890 1/cm has been absorbed by HCL molecule while the remaining frequencies of radiation has transmitted. Then after absorbing the correct wavelength of radiation the molecule vibrates at an increased amplitude (Hons 2001).

b-Electric dipole:

Molecule can absorb IR radiation its absorption causes a change in its electric dipole (dipole moment). Then when molecule having electric dipole is kept in electric field, this will exert forces on the electric charges in the molecule. Opposite charges will experience forces in opposite directions which tends to decrease or increase separation. When the charged atoms vibrate they absorb IR radiation from the radiation source, if the rate of the vibration of charged atoms in the molecule is fast, the absorption of radiation is intense and IR spectrum will have intense absorption bands. Symmetrical diatomic molecules do not have electric dipole so they cannot be excited by IR radiation (Hons 2001).

2-1-12- Theory:

Infrared technique is based upon the simple fact that a chemical substance shows marked selective absorption in the infrared region. The frequency or wavelength of absorption depends on the relative masses of atoms, the force constants of the bonds and the geometry of the atoms. Then the molecules of the chemical substance vibrate at many rates of vibration which give rise to close packed absorption bands (vibrational spectra appear as bands rather than lines because a single vibrational energy change is accompanied by a number of rotational energy changes). Various bands will be present in the IR spectrum which will correspond to the characteristic functional groups and bands present in a compound. Thus IR spectrum of a chemical substance is a fingerprint for its identification (Hons 2001).

2-1-13- Origin of infrared spectra:

The electrons contained in the atoms of a molecule exist in a number energy levels. When a molecule is placed in an electromagnetic field, a transfer of energy from electromagnetic field to the molecule occurs when: $\Delta E = h \nu$

Where the first term is the difference in energy between two states, h is plank's constant and ν is frequency of the light. When the molecule is excited. It absorbs energy from the lower energy state E' to higher energy state E'' and emits radiations of the same frequency when molecule reverts from the higher energy state to lower energy state

$\Delta E = E'' - E'$ in the case of absorption.

$\Delta E = E'' - E'$ in the case of emission.

The absorbed energy set a molecule as a whole rotating or causing vibration of the atoms within a molecule. The vibrational and rotational motions superimposed upon kinetic motion of the molecule. Not that the translational motion is dissipated as heat and is not involved in molecule spectra. Thus there are three changes in a molecule:

The first is change in energy which occurring due to transition of an electron from one orbit to another while the second is the change in energy of rotation of molecule as a whole and the third is change in energy of vibration of atom.

The energy required for exciting emission will be least for rotational and higher for vibrational and still higher for electronic transitions. Thus if the excitation energy is kept so small as to cause only transition from one rotational quantum level to another in a given vibrational level, the emission spectra observed

would correspond to changes in rotational quantum number. If the energy required for excitation is still higher to cause transition from one vibrational quantum level to another within a given electronic level emissions are observed corresponding to a change vibrational quantum number since change in vibrational level involves also change in rotational levels, the resulting spectrum is called vibrational rotational spectrum.

If the energies still higher are applied electronic transitions may take place which accompanied by vibrational level change. Each vibrational level change will be accompanied by rotational fine structure. The resulting spectrum will complex consisting of lines due electronic, vibrational and rotational transitions. Then now we can say there are three molecular or band spectra: rotational spectra, vibrational-rotational spectra and electronic band spectra (Hons 2001).

2-1-14- Selection rules:

Infrared light is absorbed when the oscillating dipole moment due to molecule vibration interacts with the oscillating electric vector of the infrared beam. For this absorption to occur it is necessary that the dipole moment at one extreme of the vibration.

The most important consequence of these selection rules is that in a molecule (with a center of symmetry) those vibrations which are symmetrical about the center of symmetry are inactive in infrared but active in RAMAN spectra while those vibrations which are not centrosymmetric are active in infrared and usually inactive in RAMAN. Thus these two type of spectrum are complementary, since most functional groups are not centrosymmetric, this technique is most informative

for identification of organic compounds. In solid state, the symmetrical properties of a molecule can be different from those of an isolated (Hons 2001).

2-1-15- Molecular Vibrations:

There are three type of vibration:

a-Stretching vibrations: a stretching vibration is a rhythmical movement along the bond axis such that the interatomic distance is increasing or decreasing. The symmetrical stretching vibration is inactive in infrared since it produces no change in dipole moment of the molecule. Stretching vibrations are two type: The first is Symmetric stretching: when the stretching and compression occur in a symmetrical fashion. While the second is asymmetric: is happen when one bond compressing and the other is stretching (Hons 2001).

b- Bending or deforming vibrations:

Bending vibration consist of a change in the bond angles between bonds with a common atom. Or the movement of a group of atoms with respect to the remainder of the molecule without movement of atoms in the group. Bending vibrations are four types:

-Scissoring: here are two atoms joined to a central atom move toward and away from each other with the deformation of the valence angle.

-Rocking: the structure unit swings back and forth in the plane of molecule.

-Wagging: the structural unit swings back and forth out of the plane of the molecule.

-Twisting: here the structural units rotate about the bond which joins to the rest of molecule (Hons 2001).

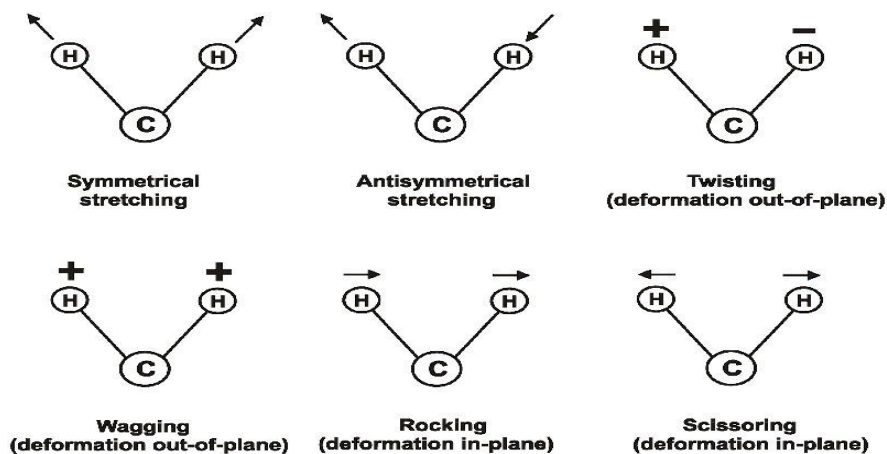


Figure 2.2: shows types of vibrations

2-1-16- instrumentation:

Three distinct types of instruments employed for IR absorption spectroscopy are considered in this section. Dispersive instruments with a monochromator are used in the mid-IR region for spectral scanning and quantitative analysis. Fourier transform IR systems are widely applied in the far- IR region and becoming quite popular for mid-IR spectroscopy. non dispersive instruments that use filters for wavelength selection or an infrared absorbing gas in the detection system are often used for gas analysis at specific wavelengths (James 1988).

a- Dispersive IR spectrophotometers:

Modern dispersive infrared spectrophotometers are in-variable double-beam instruments, but many allow single beam operation via a front-panel switch. Single

beam instruments are not very practical in the IR region because of the absorption of IR radiation atmospheric H₂O and CO₂. Double beam operation compensates for atmospheric absorption, for the wavelength dependence of the source spectral radiance, the optical efficiency of the mirrors and grating, and the detector responsive, and for the source and detector instability, which can be serious in the IR region. Also all solvents absorb in the infrared region, which makes single beam operation somewhat impractical even with storage of the reference spectrum (James 1988).

b- Dispersive spectrophotometer designs:

Modern dispersive infrared spectrophotometers are invariably double-beam instruments. But many allow single beam operation via a front panel switch. Single-beam instruments are not very practical in the IR region because of the absorption of IR radiation by atmospheric H₂O and CO₂.

Infrared spectrophotometers are either single beam or double beam. In single beam spectrometer the radiations emitted from the source are passed through a cell containing the sample and through the prism which disperses the light. Single beam spectrometers are simple, sensitive, accurate, versatile and are used to study fine details. But these instruments have two disadvantages:

- when the spectra of the solution is to be recorded, the absorption bands due to solvent are also obtained, thus making the interpretation of the bands and identification of the compounds more difficult (James D.ingle,jr, 1988).

- the base line, the line obtained without the use of the sample in the light path, slopes because the intensity of the source changes continuously with the wavelength (James D.ingle,jr, 1988).

c- Double beam spectrophotometers:

These are so designed that the light from the source is split into two beams of equal intensity. One passing through sample and the other through reference for (air or solvent) compensation. The two beams are recombined on to a common axis and are alternately focused on to the entrance slit of the monochromator. This removes the second disadvantage of the single beam spectrophotometers and horizontal base line is obtained.

Table 2.1: shows components of double beam infrared spectrophotometer

Radiation Source Condensing Optics and beam chopper	Sampling area	monochromator	detector	Amplifying system	Recorder
--	---------------	---------------	----------	-------------------	----------

d- Components of double beam infrared spectrophotometer:

Radiation source - monochromator and optical material -sampling area and detector

-radiation source:

Infrared radiation is produced by electrically heating source. Normally a Nernst filament or a Globar to 1000-1800 c. the Nernst filament is fabricated from a binder and oxides of thorium, cerium, zirconium and yttrium. The globar is small rod of silicon carbide. The maximum radiation for the globar occurs in the 5500-5000 1/cm region drops off by a factor of about 600 as the 600 1/cm the region is approached. Nichrome wire, carbon arc, rhodium wire and tungsten filament lamp are also used as light source. The most recent infrared source is the invention of KURT H. it operates at 2.8 volts and 30 watts. It develops an external temperature

of 1200 cm^{-1} and provides radiant infrared energy from 4000 $1/\text{cm}$ to 400 $1/\text{cm}$ (Hons 2001).

-monochromator and optical material:

The separation of desired frequencies can be achieved by means of monochromators. Prisms and gratings are used for this purpose. Most IR spectrophotometers use prism of alkali halides. Prisms of LiF or CaF_2 give more resolution in the region where the significant stretching vibrations are located. Recent made spectrometers are of grating type. Prisms, windows and cell faces are cut from crystals of these optical materials. The most common prism material is used NaCl for the entire region from 4000-650 $1/\text{cm}$, crystalline KBr and CsBr are satisfactory for the far infrared region while LiF provides prism material in the near infrared region (Hons 2001).

Table 1 lists some optical materials with their approximation transmission limits.

Table 2.2 Some Optical Materials

Material	Optimum range ($1/\text{cm}$)		Range for windows ($1/\text{cm}$)	
Potassium bromide	10 μ - 25 μ	1000 – 400	200 μ - 26 μ	50000 – 380
Cesium iodide	10 μ - 38 μ	1000 – 260	1 μ - 40 μ	1000 – 250
Lithium fluoride	600 μ - 6 μ	16666-1670	115 μ - 7 μ	87000 - 1430
Calcium fluoride	200 μ - 9 μ	50000-1100	125 μ - 10 μ	80000 - 1000
Barium fluoride	300 μ - 13 μ	33333-770	200 μ – 13.5 μ	50000 - 740
Silver chloride	----	-----	10 μ - 25 μ	1000 – 400
Fused silica	185 μ – 3.5 μ	5400-03140	185 μ - 4 μ	5400 - 2500
Glass	300 μ - 2 μ	333335-000	300 μ - 26 μ	33333 - 3850

(Hons 2001).

- Sample:

Compounds may be examined in the vapor phase as pure liquid, in solution and in solid state. Crystalline samples can be used for the study of infrared dichroism and information regarding molecular geometry and the manner of packing of the molecules in the lattice can be obtained (Hons 2001).

A- Gaseous samples:

The vapor is introduced into special cell. That can be placed directly in the path of one of the infrared beams. The end walls of the cell are usually made of sodium chloride, which is transparent to infrared. Most organic compounds have too low a vapor pressure for this phase to be useful. The low frequency vibrational changes in the gaseous phase often split the high frequency vibrational bands (Dr.H.KAUR and Hons 2001).

B-liquid samples:

Liquids are usually handled pure without solvent because all solvents have their own absorption spectra. The solvent must be dry and has least absorption in infrared region of interest. The cells are usually rinsed, filled and emptied with the aid of hypodermic cell. Highly viscous liquids are often simply sandwiched cell as a layer between two salt plates.

It is desirable to measure IR spectra of solution by filing a second cell of the same thickness as the sample cell with solvent and placing the second cell in the reference beam. This will eliminate the absorption bands of the solvents. Spectra taken in dilute solutions (1 to 50%) in non-polar solvents are most desirable because they are better resolved than spectra taken on solids and also because intermolecular forces which are strong in crystalline state are minimized. It may be noted

that stretching frequencies are lowered and bending frequencies are raised due to H-bonding (Hons 2001).

C-Solid samples:

A wide variety techniques is used for measurement of infrared absorption spectra of solid materials. Solid samples are used in the form of solution, powder, glassy film or pellet.

- Solid run in solution. If the solid is soluble in some suitable solvent, its solution can be made and in one of the cells for liquids. However suitable solvents are limited in number and none are totally transparent.

-Solid films: a morpous solid samples may be cased into films from solution on an infrared transmitting window or solid melted between salt plates and allowed to cool into a film. This technique is recommended for rapid qualitative analysis but not for quantitative analysis (Hons 2001).

- Mull technique:

The most convenient and routing method is called mulling. Mulls are prepared by thoroughly grinding 1mg of a solid in a smooth agate mortar. The powdered sample is mixed with little high boiling petroleum.

-Pressed pellet technique:

This technique depends on the fact that dry, powdered potassium bromide can be pressed under high pressure in vacuo to form transparent discs. The resulting transparent discs are inserted into a special holder of the spectrophotometer. The quality of the spectrum depends upon intimacy of mixing and the reduction of the suspended particles to 2μ or less. It is still well suitable and best general method

for running crystalline compounds is the solid state. In conjunction with mull method, it yield more information about the sample. Some polymers yield their best spectra in a KBR disc. The technique for handling polymers which are insoluble and difficult to grind because of their tough nature, consists of a making a low temperature KBR disc. The grinding is done at liquid nitrogen temperature that makes polymer brittle (Hons 2001).

-attenuated total reflection technique or internal reflection:

This recent technique developed by FAHRENFORT consist of a prism (45, 90)

The technique is based upon the fact that a beam of light that internally reflected from the surface of the transmitting medium passes a short distance beyond the reflecting boundary and returns to the transmitting medium as a part of process of reflection. If a sample of lower refractive index than the transmitting medium is brought in contact with the reflecting surface, the light passes through the material to the depth of a few microns, producing an absorption spectrum (Hons 2001).

-Detector:

Detector is a device that usually change the thermal radiant energy into electrical energy. In infrared detectors may be selective or non-selective. The selective detectors are those whose response is markedly dependent upon the wavelength of the incident radiation. Examples of this type are photocells, photographic plates, photoconductive cells and infrared phosphors. Photoconductive cells are has a rapid response and high sensitivity. When illuminated by infrared light these cells show an increase in conductivity. The non-selective detectors are those whose response is directly proportional to incident energy but relatively independent of

wavelength. Common examples include thermocouples, bolometers and pneumatic cell.

Thermocouple is a best detector for measuring infrared radiation, they are usually made by evaporating metals, such as bismuth, antimony, or semiconductor alloys on a thin film of cellulose nitrate or other supporting base. The radiant energy heats one of two bimetallic junctions.

While the bolometer is an excellent detector, it gives electrical signal as a result of the change in resistance of the metallic conductor with temperature. The main advantages of this type is faster response time because of small thermal capacity.

And the last is pneumatic detector cell, it was developed by GOLAY it is best suited when working at wavelength greater than 15μ . Its cells consist of two cells. The first three mm cell is filled with gas and connected to a smaller cell through a tube. One end of smaller tube is closed by a curved diaphragm which acts as a mirror. The radiation falls on the first cell and heats is conducted to the gas. Causing it to expand. Which in turn moves the mirror in the second cell. A steady beam of light from a lamp inside the detector can be focused upon diaphragm which reflect the light on to a photocell connected to an amplifier and recorder. Recently detectors are fabricated from crystals, these crystals take less time than other thermal detectors. Hence with these crystals radiation can be chopped at higher rate (KAUR 2001).

2-1-17-Working of the double beam spectrophotometer:

Two designs of double beam instrument, optical null system and electrical beam rotating system have been widely applied to mid infrared region. Optical system of double beam spectrophotometer is shown below:

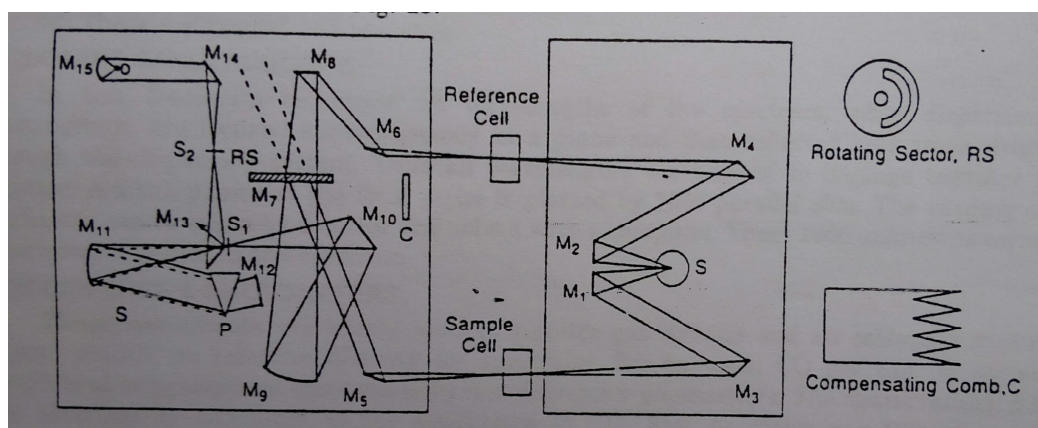


Figure 2.3: Shows optical system of double beam spectrophotometer (Dr.H.KAUR and Hons 2001).

Source and sampling area:

Infrared radiation is produced by electrically heating a Nernst filament or gas a Globar. The radiation from the source is divided into beams by mirrors M1 and M2. The two beams, reference and sample, are focused into the sample area by mirrors M3 and M4. These beams then pass through the reference cell and sample cell respectively. Opaque shutters mounted on the source housing permit blocking of either beam independently (Hons 2001).

-Photometer:

The reference beam passes through the attenuator and is reflected by mirrors M6 and M8 to the rotating sector mirror M7. Which alternately reflects the reference beam out of the optical system and transmits the beam to mirror M9. The reference beam

is now intermittent beam with a frequency of 8 and 13 cycles per sec. the beam is focused by mirror m10 on the slit s1. The sample beam is passes through the comp and is reflected by mirror m5 the rotating sector mirror m7, which alternately transmits the beam out of the optical system and reflects it to the mirror m9, m10 and slit s1. Then the reference beam and sample beam have been combined into a single beam of alternating segments. This establishes a switching frequency at the detector equal to the speed of the rotation of the m7. When the beams are of equal intensity, the instrument is at optical null. The comb in the sample beam permits balancing the beams. The recording pen is then at 100% transmittance when no sample is present. When the sample beam is absorbed by the sample the attenuator is driven into the reference beam until its intensity matches that of the sample beam (Hons 2001).

-Monochromator:

The combined beam passes through the monochromator entrance slit s1 to the mirror m11 which reflect through the prism to the LITTROW MIRROR m12. At this point the beam is dispersed by the prism over a range of frequencies. The dispersive is reflected back through the prism (to increase the dispersion) to mirror m11, then mirror m13 which focuses the beam on the exit slit s2. The width of the frequency range focused on the exit slit s2 is determined by the width of the entrance slit s1 and the dispersing power of the prism. The frequency range focused on s2 is determined by the angle of LITTROW mirror m12. Thus rotating m12 produces a scan of frequency ranges at the exit s2 and consequently at the detector. Maximum resulting is obtained by using prism or grating (Hons 2001).

-Detector:

After leaving the exit slit of the monochromator the beam is reflected by a flat mirror m14 to an ellipsoidal mirror m15. The foci of ellipsoidal are the exit slit s2 and the detector. The unbalanced signal can be amplified and recorded. The detector detects any change in the intensity of the radiation due to absorption. The amplified off null of the detector is used to position the optical attenuator so that the radiation from the reference and sample beam are kept at equal intensity. The amount of attenuation required is a direct measure of the absorption by sample (Hons 2001).

-Limitations:

Although double beam spectrophotometers are equipped with various refinements, such as higher precision and resolution, greater wavelength span. More versatility with respect to mode presentation of the spectrum, yet the most important limitations of these instruments is their inherent slow response. High speed instruments are required for kinetic studies and for on line identification of constituents of a flowing system. The obvious way to achieve the objective of increased speed is to gear up a conventional spectrophotometer to run faster (Hons 2001).

2-1-18-Advance spectrophotometers:

- Rapid-scan spectrophotometers:

It introduced with a cooled semiconductor or pyro electric detector. The instrument is so designed that it may be directly connected to a gas chromatograph. The carrier gas fills the reference cell and column effluent flows through the analytical cell. The resulting spectra possess resolution fully adequate for quantitative measurements of the chromatograph (Hons 2001).

2-1-19-Fourier transform infrared spectroscopy:

Recently a new method of taking an infrared spectrum involves the use of FTIR or interferometer. Light (covering the whole frequency range, typically 5000 – 400 cm^{-1}) is collimated by lens and beam splitter splits the light into two equal parts.

Either one beam passed through the sample or both are passed. But one beam is made to traverse a longer path than other. Recombination of two beams produces an interference pattern. By systematically changing the difference in the two paths. The interference patterns change to produce a detected signal varying with optical path difference. This pattern is known as the interferogram. However Fourier transformation of the interferogram using a computer converts it into a plot of absorption against wavenumber which resembles the usual spectrum obtained by traditional method. Interferograms can be interpreted well with electronic assistance or digital minicomputer.(HONS 2001).

There are several advantages to FTIR over the traditional method:

- by FTIR it is possible to measure the whole spectrum in a few seconds, because it is not necessary to scan each wave number successively.
- since it not depends upon slit and prism or grating, high resolution in FTIR is easier to obtain without sacrificing sensitivity.
- FTIR is especially useful for examining small samples (several scans can be added to gather) to taking the spectrum of the compounds produced in the out flow of a chromatograph. On the other hand conventional method takes several minutes and requires the collection of the samples first.

-the digital form in which the data are handled in the computer allows the spectrum of the pure compounds to be subtracted easily from that a mixture to reveal the spectrum of other compounds of the mixture (Hons 2001).

2-1-20-Non-dispersive photometers:

These instruments are widely used to monitor gas streams and air pollutant studies and depend mainly on selective filtering for specificity. For example CO gas can be successfully determined in presence of other gases by non-dispersive photometers (Hons 2001).

2-2- ALKAWAL:

Is a type of spice which used as one of the subcomponents for making salter, which uses with Sudanese porridge, specially in relative of DARFOUR county west of Sudan.

2-2-1 Sicklepod *Senna obtusifolia* (L.)

Also known as *coffeebean*, *Java bean*, *Cassia obtusifolia* (L.), *Cassia tora* (L.)

Classification and Description:

Sicklepod is a member of the *Fabaceae* (Leguminoseae) or bean (pea) family. Sicklepod is an annual, herbaceous to semi-woody plant that is thought to be native to the American tropics. It can be found throughout the southeastern United States. The cotyledons of sicklepod are rounded and have three to five distinct veins. The stems are erect, branched, lack hairs (glabrous) and can reach heights of 1 - 6 feet. Sicklepod has alternate leaves comprised of 4 - 6 leaflets that are egg-shaped and arranged pinnately compound, which means that the leaflets are opposite one

another. The pair of leaflets furthest from the main stem is the largest and the pair closest to the stem is the smallest. The egg-shaped leaflets can be 1 - 3.5 inches long. This weed reproduces by seed. The brownish seeds are housed in long (4 - 8 inches), sickle-shaped, hairless pods. Pods are a typical fruiting structure of a legume. Flowers have distinctive yellow petals and one or two arise from leaf axils (where stem meets leaf). Sicklepod has a stout taproot. This weed can be easily confused with coffee senna (*Cassia occidentalis*). However, the leaves of sicklepod are blunt while coffee senna's are pointed (Larry Steckel 2006).



Picture 2.4: cassia obtusifolia plant's leaves

Picture: appears leaves of cassia obtusifolia

Interesting Facts:

Though sicklepod is a legume, it does not fix nitrogen. *Senna* is from ancient Greek and means an aromatic plant.

This is in reference to sicklepod being rank smelling when crushed. The Latin *obtusifolia* refers to the shape of the

leaflets (*obtus*e blunt and *folia* leaves or blunt W125 leaves). Sicklepod seeds have had medicinal properties since 4000 B.C. when they were used as laxatives.

Sicklepod hosts several insects and diseases of agricultural importance, including Asian soybean rust. (*Larry Steckel 2006*)

2-2-2- Kawal preparation method:

kawal fermentation method was carried according to Dirar (1993), the green leaves are first freed of all extraneous matter, such as leaves of other plants, pods and flowers of the kawal plant itself, caterpillars and insectdamaged leaves. This process of sorting out the kawal leaves is strictly observed and in fact this part of the preparation procedure is the most tedious step as it takes hours of painstaking work. Green flower buds and delicate young pods may, processed with the green leaves. The unwashed, healthy green leaves, now clean from all adulteration are²⁵ beaten in a mortar-and -pestle to give a green paste. Pounding is done in such a way that the leaves are crushed without releasing their juice. In the final paste can be seen partially crushed leaves, twigs, mid-ribs and petioles. Meanwhile, a pit is dug in the ground in a shaded cool place. An earthenware pot (Burma) is fitted into the pit, leaving only the neck of the container above ground. The green paste is now packed into the pot by hand. Next, green sorghum leaves are folded onto the surface of the leaf paste in the Burma so that it is completely covered. Washed, dry stones are then placed on top of the sorghum leaves to weight them down. The mouth of the pot is then covered with some metal tray or dish and the whole sealed off with mud to prevent insect from entering. Every 3-4 days the jar is opened, the now yellow and dry sorghum leaves removed and the Burma thoroughly hand-mixed and repacked, this time a little loose. Fresh sorghum leaves are folded on the surface of the paste and weighted down as before. The Burma covered and sealed

off again. The paste is next molded into small, irregular balls or flattish cakes which are then sun dried for 3- 4 days. The duration of the fermentation is about 15 days for the supply of an average family.

2.2.3 Nutritive value of *Cassia obtusifolia*:

Cassia obtusifolia is not the only leguminous herb found widely in the Sudan. But in particular, it is the only one suitable for making kawal food due to the high nutritive value of the plant (Dirar, 1984). Regarding the nutritional value it has been reported that the plant is rich in calcium and iron, excellent source of vitamin B and in addition, it contains high levels of phosphorous, ash, riboflavin and ascorbic acid. Kawal plant contains high level of protein. The value of kawal leaf protein seems to lie in the quality rather than in the quantity, i.e., it contains high values of cysteine and methionine (Taweel, 2003). *Cassia obtusifolia* leaves are certainly among the richest plant materials with respect to various nutritional components of the human diet. Duke (1981) tabulated the chemical composition of the beans, leaves fruits, inflorescences, shoots and sprouts of over 30 leguminous plants. It can be seen from his tables , that the leaves of *Cassia obtusifolia* have the highest calcium and riboflavin of all the legumes listed. Moreover, the leaves of this plant rank second in iron and beta-carotene, and third with respect to ash and ascorbic acid. Further, if only the leaves are taken into consideration, those of *Cassia obtusifolia* have the highest phosphorous, calcium, riboflavin and ascorbic acid levels .out of some 16 legumes.

2.2.4 Kawal as meat substitute:

The traditional Sudanese fermented food ‘kawal’, defined as a meat substitute derived by fermentation of leaves of *Cassia obtusifolia* falls in the family of West African strong smelling flavor foods (Dirar, 1993). The major role of kawal in the diets is as a

meat substitute both in its capacity as source of high-quality protein and as a source of meaty flavor (Dirar *et al.*, 1985).

2.2.5 The chemical composition of *Cassia obtusifolia* leaves and

Kawal:

Proximate composition of plant material consists of determining the major classes of chemical components, which include moisture, crude fiber, fat content (ether extract), crude protein, ash and carbohydrates (by difference). Proximate composition provides good initial impression of relative nutritive value and utility of an agricultural product and allows basis of comparison between different species, plant parts and cultivation conditions (Abdel-Rahim, 2004).

Chapter Three
Material and Methods

Chapter Three

Material and Methods

3-1- Material and Apparatus:

The fermented leaves of *Cassia obtusifolia* or kawal was obtained from Forbranga (western Sudan).it was obtained in dry form after being sun dried and freed from foreign materials and powdered by hummer mill with same mesh size and was kept in clean bottles at room temperature for further use.



Picture 3.5: shows sun dried powder of kawal

Fourier transformer spectrometer device was used for analyzing the sample. The device model was (FTIR-8400s), SHIMADZU company.

For identification of the mineral contents: : Ca, Na, K, Mg, P, Zn and Fe the sample was used as a combussed dry that required for atomic spectroscopy. in

this instrumental the atomic absorption spectrophotometer model AA-6800, company SHIMADZU was used.



Picture 3.6: Fourier transformer spectrometer device

3-2- Method:

3-2-1-Method One FTIR Analysis:

In this experiment 100mg from Potassium bromide (KBr) was used. Additionally to 1mg from the sample which was used as a dry powder kawal. And then KBR and the sample were mixed until became mixture. The mixture samples were compressed after put them on piston then used the compressed mixture samples on Fourier transformer spectrometer device which connected with control process unit (CPU) and reading screen respectively. For detection of mineral contents of fermented cassia leaves:

3-2-2-Method Two digestion Method:

Ca, Na, K, Mg and P digestion method was used that the sample was digested in HNO₃/HCl₄ and elements were detected by inductively coupled

plasma spectroscopy (ICPS). Heater, test tube and flasks were used as apparatus. 2g of sample was weighed into test tubes, 8ml HNO₃ was added to the test tubes and covered them with glass ball and left it to stand for overnight, another HNO₃ produces fumes was added preferably was did it in chamber, 3ml of HClO₄ acid was added and heated slowly until almost dry. And then glass ball was removed and heated strongly until white fumes disappeared, tubes were cooled and 5ml of 0.1N HCl was added and boiled gently for 30 minutes, after left it for cooling the solution was transferred into 50ml flask. The tubes were rinsed with 10ml of 0.1N HCl and boiled gently for 10 minutes, the solution was poured into the previously flask and filled it to 50ml mark with 0.1N HCl, the ICP readings were record.

3-2-3-Method Three Atomic Spectroscopic Method:

For analyzing of Iron and Zn the atomic spectroscopy method was used after combussioned the sample, with the following details, hence the first values for Iron and the seconds for zinc respectively. the optics parameters: lamp current low(Am) (12 and 8), wavelength(nm) 248.3 and 213.9, slid width(nm) (0.2 and 0.5) and lamp mode BGC-D2. While the atomizer/gas flow set up: fuel gas flow rate(L/min) (2.2 and 2), flame type Air-C₂H₂, burner height(mm) (9 and 7), burner lateral pos(pulse) 0 and burner angle(degree) 0.and measurements parameters are: order first, zero intercept no, unit mg/L, repetition sequence SM-M-M-....., pre-spray time(sec) 3, integration time(sec) 5 and response time 1.

Chapter Four

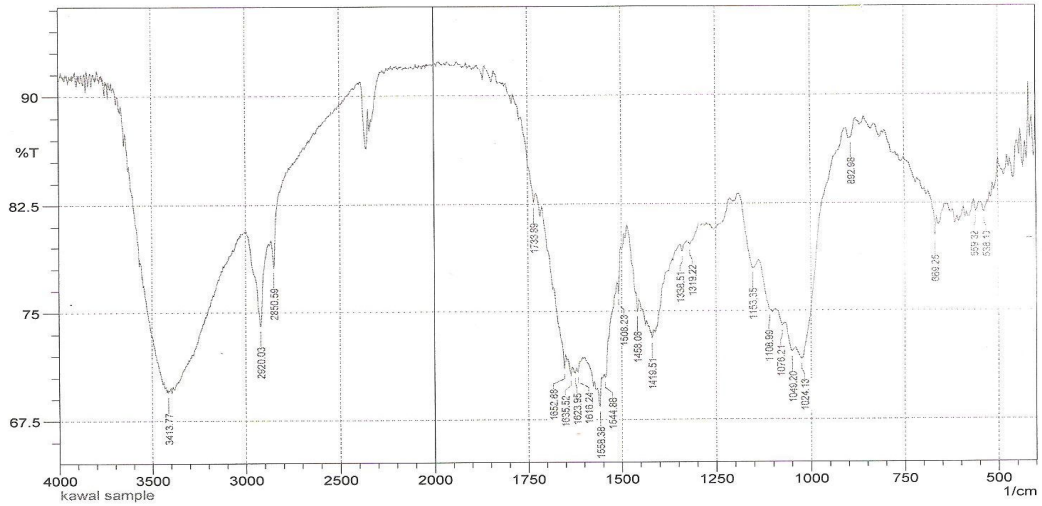
Results and Discussions

Chapter Four

Results and Discussions

4-1-Results & Discussion:

FTIR spectrum of fermented cassia obtusifolia leaves that obtained consist of several regions of frequencies that leads to identify of molecule group. Chemical compositions of fermented kawal is shown in figure 4.1 which is bellow. kawal was found consist of several types of molecular with three functional groups. On alky halide group function were found C-Br which has strong intensity vibration stretch, C-CL with strong intensity of stretching moves, and C-F molecular with the same prescriptions. While the alkene group function consist of C-H with bending strong mode of vibration, the two -C-H alkenes have bending mode of vibration but their intensity are variable, the three C=C alkenes have variable intensity with stretch motion and the two C-H with strong stretch mode of vibration. The absorption frequencies also was N-O which belong to nitro function group with stretch vibration mode and strong intensity. The C=C is aromatic molecular, stretch mode and its intensity is medium weak or multiple bands. C=O is carbonyl with strong stretch. O-H is alcohol structure which has stretch, H-bonded vibration and strong broad intensity. The two C-O are esters function group with stretch vibration mode and 2 bonds or more intensity. The ether C-O with the same descriptions of ester. The acid C-O stretch strong intensity of vibration. And lastly the two N-H amides have bending modes of vibrations and their intensity are medium (John Wiley and sons 1981).



	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	538.1	81.886	0.716	545.82	528.46	1.475	0.034
2	559.32	81.903	0.759	565.1	551.6	1.148	0.031
3	669.25	80.177	1.669	686.61	665.4	1.819	0.037
4	892.98	87.015	0.196	894.91	879.48	0.885	0.005
5	1024.13	71.664	2.695	1039.56	929.63	11.528	0.266
6	1049.2	72.192	0.852	1066.56	1039.56	3.726	0.08
7	1076.21	74.007	0.368	1095.49	1070.42	3.195	0.009
8	1108.99	75.032	0.231	1137.92	1107.06	3.555	0.011
9	1153.35	77.954	1.945	1190	1137.92	5.083	0.266
10	1319.22	79.599	0.524	1326.93	1294.15	3.147	0.05
11	1338.51	79.136	0.539	1344.29	1326.93	1.726	0.017
12	1419.51	73.143	0.428	1421.44	1413.72	1.035	0.009
13	1458.08	74.987	1.069	1461.94	1454.23	0.94	0.023
14	1508.23	76.159	1.64	1512.09	1500.52	1.257	0.021
15	1544.88	70.425	0.551	1546.8	1512.09	4.557	-0.037
16	1558.38	68.399	1.715	1566.09	1552.59	2.14	0.054
17	1616.24	70.792	0.536	1620.09	1612.38	1.143	0.011
18	1623.95	70.726	0.399	1631.67	1620.09	1.728	0.017
19	1635.52	70.569	0.771	1643.24	1631.67	1.716	0.025
20	1652.88	71.031	1.253	1681.81	1650.95	4.056	0.074
21	1733.89	82.593	1.191	1747.39	1728.1	1.492	0.045
22	2850.59	78.086	3.081	2866.02	2819.73	4.276	0.216
23	2920.03	74.036	6.043	2997.17	2877.6	13.024	1.574
24	3413.77	69.479	0.18	3417.63	3407.98	1.519	0.005

Comment;
kawal sample

Date/Time; 11/15/2016 10:49:57 AM
No. of Scans; 20
Resolution; 4 [1/cm]
Apodization; Happ-Genzel

Figure 4.1: kawal FTIR spectrum

The mineral (Calcium, Magnesium, Phosphorus, Sodium and potassium) of fermented kawal is reported on table below hence the raw for the mineral element and the another for amount of foundation %

Table 4.3: mineral elements contents of kawal

The element	Amount of kawal
Calcium	2.50%
Magnesium	0.66%
Phosphorus	0.396%
Sodium	0.165%
Potassium	1.595%

Minerals were extracted from the samples by the inductively coupled plasma spectroscopy method. About 2.50% was found is presence of calcium and the other minerals as: mg 0.66%, P 0.396%, Na 0.165% and K about 1.595%.

The fermented leaves of *C. obtusifolia* contain the major elements needed by human body (see Table 4.4). Other reports showed that the samples obtained from the Chad generally had higher quantity of elements compared to those from forbranga (Dirar et al., 1985). It appears that locations of Cassia species and their environments have effects on the chemical constituents. The mineral matter comprises large number of inorganic elements present in foodstuff with varying amounts. Parts of these are recognized to perform essential functions in human nutrition; therefore, they must be consensually supplied by soft stuffs. Accordingly, 14 different mineral elements are essentially required by human body for good health and growth (Wilson, 1967); Ca, P, K, Mg, Na, S, and Cl are needed in appreciable.

Table 4.5 which is below shows the content of Zn and Fe that extracted from the fermented cassia obtusifolia.

Table 4.5: shows the content of Zn and Fe of sample Mg/L

Mineral	Content
Zn	0.6655
Fe	15.6411

The values of Iron and Zinc obtained are shown on the table above that found the Iron content is 0.6655 and Zinc content is 15.6411.

4-2-Conclusions:

In conclusion, chemical compositions of dried fermented cassia obtusifolia leaves characterized using FTIR spectroscopy and it was found that this material consist of Alky halide, alkene and amines.

4-3-Recomondation:

- 1- The acute shortage of meat and animal proteins in developing countries has made it necessary for consumer to rely heavily on protein from legumes (especially kawal) which are rich in protein, B-vitamins, dietary fiber and mineral content.
- 2- Attention should be directed toward novel methods for kawal processing and utilization.
- 3- More research should be conducted for flavor components and the bad smelling constituents.
- 4- More detailed spectroscopic studies are needed to investigate the effect of kawal Fermentation on the changes in the physical attributes.

References:

Abdel-Rahim, (2004). *‘Effect of processing on anti-nutritional factors and in vitro protein digestibility of Faba bean (vicia faba)’* - university of Khartoum, Sudan.

Algadi yousif (2015). – *‘anti nutritional factors of green leaves of cassia obtusifolia and kawal’* – food science and technology Khartoum Sudan. 6-9.

Dr. H.KAUR and Dr. Hons (2001)-‘ Spectroscopy’ – K.K. Mittal

Dr. Shardendu Kisala- ‘Spectroscopy’.

H. A. Dirar, (1993). *‘The Indigenous Fermented Food of the Sudan. A study in African Food and Nutrition’* CAB International, Wallingford.

H. A. Dirar.; Harper, D. B. and Collins, M. A. (1985). *‘Biochemical and microbiological studies on Kawal, a meat substitute derived by fermentation of Cassia obtusifolia leaves’*- Journal of the Science of Food and Agriculture. 63: 881-892.

Helmt Gunzler, Dr. Hons Ulrich Gremlich (2002) – *‘Introduction to IR spectroscopy’* – Wiley-VCH Verlag GMBH

J. K. Duke, (1981). *‘Handbook of Legumes of World Economic Importance’* - Plenum Press, New York pp. 199-265

John Wiley and sons (1981) – *‘Identification of organic compounds’*– 4th.ed new York

Larry Steckel-UT Extenssion.(2006).

.Mbailao MBAIGUINAM, YAYA MAHMOUT, Minpereum TARKODJIEL, Merbard Delobel and Jean-Marie BESSIERE (2005).- *‘constituents of kawal, fermented cassia obtusifolia leaves*

atrational food chad' –African journal of Biotechnology VOL. 4(10). 1080-1083.

Mutasim Zean-Alabdeen (2014). – *'the effect of fermentation on nutritional quality of cassia obtusifolia leaves (kawal)'* – Omdurman Islamic university. Sudan.

Nuha.M.O, Isam.A.M.A and Alfadil.E.B. (2010).- *'chemical composition, antinutrients and extractable minerals of cassia leaves and kawal'* – international food research journal. 775-785.