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Title by:

**Study of Physical and Chemical Characteristics for
Hibiscus sabdariffa L Using Different Techniques**

دراسة بعض الخصائص الفيزيائية والكيميائية لنبات الكركدي باستخدام
تقنيات مختلفة

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بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

الآية

قال تعالى:

{اللَّهُ نُورُ السَّمَاوَاتِ وَالْأَرْضِ ۚ مَثَلُ نُورِهِ كَمِشْكَاةٍ فِيهَا
مِصْبَاحٌ ۚ الْمِصْبَاحُ فِي زُجَاجَةٍ ۚ الزُّجَاجَةُ كَأَنَّهَا كَوْكَبٌ
دُرِّيٌّ يُوقَدُ مِنْ شَجَرَةٍ مُبَارَكَةٍ زَيْتُونَةٍ لَا شَرْقِيَّةٍ وَلَا غَرْبِيَّةٍ
يَكَادُ زَيْتُهَا يُضِيءُ وَلَوْ لَمْ تَمْسَسْهُ نَارٌ ۚ نُورٌ عَلَى نُورٍ ۗ
يَهْدِي اللَّهُ لِنُورِهِ مَنْ يَشَاءُ ۚ وَيَضْرِبُ اللَّهُ الْأَمْثَالَ لِلنَّاسِ ۚ
وَاللَّهُ بِكُلِّ شَيْءٍ عَلِيمٌ}

صدق الله العظيم

سورة النور الآية 35

Dedication

*To my mother, who has been
sacrificing throughout the whole of
our educational journey .loyalty*

*To my father, may Almighty Allah please and
forgive him, who died before my education
journey began.*

*To my brother, who have been supportive to me
Throughout.*

*To my candy beloved daughter "Donia" and
my husband "Yasir"*

*To all who supported me during my
educational stages.*

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Abstract

Two types of *hibiscussabdariffa L* were prepared (red and white) in the form of powder respectively and the samples were exposed to the XRF meter to identify elements and their concentrations of the samples. Also the samples were exposed to X-ray to study the crystal structure. It was found that the material was amorphous. Also some of the samples were dissolved in cold and hot water in the form of a solution and were characterized by UV-VIS spectrometer to study the absorbance spectrum of the samples and determine the maximum wavelength. Also some of the samples were dissolved with distilled water and exposed to EC meter to find conductivity which was found equal to 3.2 Siemens per meter (s/m) at 26.7c, 3.08 Siemens per meter (s/m) at 26.5c respectively and used the pH meter to measures the difference in [electrical potential](#) between a pH electrode and a reference electrode and acidity and it was observed to have the same acidity as 3.93.

المستخلص

تم تجهيز نوعان من نبات الكردي (الأحمر والأبيض) على التوالي، في شكل مسحوق (بودرة)، وعرضت العينات لجهاز XRF لمعرفة أنواع وتركيز العناصر في العينات، وأيضاً عرضت العينات لجهاز X-ray لدراسة التركيب البلوري حيث وجد أن المادة غير متبلورة. أيضاً تمت إذابة جزء من العينات في الماء البارد والساخن في شكل محلول وعرضت لجهاز ال UV-VIS لدراسة طيف إمتصاصية العينات وتحديد أقصى طول موجي، وأيضاً تمت إذابة جزء من العينات بالماء المقطر وعرضت لجهاز ال EC لإيجاد الموصلية لهما ووجد أنها تساوي 3.2 s/m في درجة حرارة 26.7 درجة مئوية 3.08 s/m في درجة حرارة 26.5 درجة مئوية على التوالي، وأستخدم جهاز pH لقياس فرق الجهد بين القطب الكهربائي لـ pH والقطب الكهربائي المرجعي والحمضية ولاحظ أن لهما نفس الحمضية وتساوي 3.93.

List of Contents

Title	Page
البسمة	I
Holly Quran Verse	II
Dedication	III
Acknowledgement	IV
Abstract	V
Arabic Abstract	VI
List of Contents	VII
List of Tables	X
List of Figures	XI
Chapter One	
Introduction and Literature Review	
1.1 Prelude	1
1.2 The problem	2
1.3 Objectives	2
1.4 Literature Review	3
1.5 Thesis layout	6
Chapter Two	
Theoretical Background	
2.1 HistoryOfMaterialScience	7
2. 2 Classification of Materials	8
2.3 Properties of materials	14
2.3.1 Mechanical Properties	15

2.3.2 Thermal Properties	15
2.3.3 Electrical Properties	18
2.3.4 Magnetic Properties	19
2.3.5 Chemical Properties	19
2.3.6 Optical Properties	19
2.4 Material Structure	21
2.5 Applications of Materials	22
2.6 Conductivity	23
2.7 Types of Conductivity	24
2.8 Spectroscopy	26
2.8.1 Types of Spectroscopy	26
2.6.2 Types of Instruments	30
Chapter Three <i>Hibiscus sabdariffa L</i>	
3.1 Introduction	35
3.2 Types of <i>Hibiscus sabdariffa L</i>	36
3.3 Importance and Uses of <i>Hibiscus sabdariffa L</i>	36
3.4 Medicinal and Industrial Applications	37
3.5 Phytochemicals Content	38
Chapter Four Experiment and Methodology	
4.1 Equipment and Materials	39
4.3 Methodology	43
4.4 Results	44

4.5 Discussion	46
<p style="text-align: center;">Chapter Five</p> <p style="text-align: center;">Conclusion and Recommendation</p>	
5.1 Conclusion	47
5.2 Recommendation	47
References	48

List of Tables

Title	Page
Table (3.5) Chemical analysis of dried <i>Hibiscus sabdariffa</i> Lcalyces (red and white)	38
Table (4.1) shows the reading of XRF red <i>Hibiscus sabdariffa</i> Lpowder	44
Table (4.2) shows the reading of XRF red <i>Hibiscus sabdariffa</i> L	44
Table (4.3) shows the reading of XRF white <i>Hibiscus sabdariffa</i> Lpowder	45
Table (4.4) shows the reading of XRF white <i>Hibiscus sabdariffa</i> L	45
Table (4.5) shows the reading of UV Spectrometer wavelength and Absorbance	46
Table (4.6) shows the reading of Conductivity Meter	46

List of Figures

Title	Page
Figure (3.1) Red <i>Hibiscus sabdariffa</i> L powder	34
Figure (3.2) White <i>Hibiscus sabdariffa</i> L powder	35
Figure (4.1) XRF Meter	38
Figure (4.2) pH Meter	39
Figure (4.3) Conductivity Meter	40
Figure (4.4) UV Spectrometer	40
Figure (4.5) X-ray Detector	41
Figure (4.6) shows the reading of XRF red <i>Hibiscus sabdariffa</i> L powder	42
Figure (4.7) shows the reading of XRF red <i>Hibiscus sabdariffa</i> L	43
Figure (4.8) shows the reading of XRF white <i>Hibiscus sabdariffa</i> L powder	44
Figure (4.9) shows the reading of XRF white <i>Hibiscus sabdariffa</i> L	44

CHAPTER ONE

CHAPTER ONE

INTRODUCTION

1-1: Prelude

A material is defined as a substance (most often a solid, but other condensed phases can be included) that is intended to be used for certain applications[1]. There are a myriad of materials around us they can be found in anything from buildings to spacecraft. Materials can generally be further divided into two classes: crystalline and non-crystalline. The traditional examples of materials are metals, semiconductors, ceramics and polymers. New and advanced materials that are being developed include nanomaterials, biomaterials and energy materials to name a few[2].

An increase in a solution's temperature will cause a decrease in its viscosity and an increase in the mobility of the ions in solution. An increase in temperature may also cause an increase in the number of ions in solution due to dissociation of molecules. As the conductivity of a solution is dependent on these factors then an increase in the solution's temperature will lead to an increase in its conductivity [3]. Thermal conductivity is important in building insulation and related fields. However, materials used in such trades are rarely subjected to chemical purity standards [4].

Spectroscopy is the study of the interaction between matter and electromagnetic radiation. Historically, spectroscopy originated through the study of visible light dispersed according to its wavelength by a prism. Later the concept was expanded greatly to comprise any interaction with radiative energy as a function of its wavelength or frequency. Spectroscopic data is often represented by a spectrum, a plot of the response of interest as a function of wavelength or frequency, there are many types of spectroscopy as

IN,UV-VIS,NIR, MIR, Raman and Fluorescencespectroscopy etc, there are many types of instruments as IN,UV-VIS, NIR, MIR, Raman and Fluorescence spectrometers etc[5] .

1-2: The Problem

Karkade has a great benefits and uses in different fields of life, so it becomes a main subject for many studies and this is one of them:

Study of physical and chemical characteristics for karkade by using different techniques.

1-3: The Objective

1. Study spectrum of red and white *Hibiscus sabdariffa* L powder to determine wave length maximum and higher absorbance.
2. Determine ratio element consist of the samples.
3. Measurement conductivity and acidity of the samples.

1-4: Literature Review

Many studies were conducted in this fields:

Karkade calyces samples (variety Rahad) collected from different local sites were spray dried and the powder samples were analyzed physically, chemically and microbiologically. The particle size of the various karkade powder samples ranged from 11.8 to 14.1 μm , while the flowability (angle of repose) ranged from 41° to $43^\circ 20'$. The reconstitution properties slightly varied when the various karkade samples were compared. The ranges of wettability, sinkability and dispersibility were 320–370 s, 800–850 s and 0.04–0.05 OD, respectively. The bulk density ranged from 0.57 to 0.65 g/ml, and the yield of karkade powder of the various samples ranged between 31.4 and 34.3%. The spray-dried samples from different sites were comparable in most of the chemical components and they contained relatively good amount of

macro- and micronutrients and high nutritive value. Virtually, all the spray-dried karkade powder samples were devoid of coliform, staphylococci and Salmonella cells [6].

More than 300 species of *Hibiscus* are grown over the world. It is an annual herbaceous shrub belonging to the family Malvaceae. Sudan is considered as the country in which *Roselle* originated, particularly in the Kordofan and Darfur areas. Roselle is known as karkade in the Sudan and other Arab countries. It is mainly grown for its fleshy calyx (sepals), which is the commercially valuable part of the plant. The color of the calyx plays an important role in determining the quality of karkade. The plant has some medicinal uses in Europe, it is used in food preparation in sauces, jams, juices, jellies, syrups ,flavoring , as coloring agent for food and drinks. This paper is a review of the applications and production of roselle plants and points out that roselle is a promising crop for medicinal uses, which is an aspect that has not been widely studied to date [7].

This study dealt with calyces and leaves of karkade (*Hibiscus sabdariffa* L.) to compare the effect of fermentation on its chemical composition and antinutritional factors. The calyces of karkade were obtained from El-Nasr *Hibiscus* Factory, Khartoum North and the leaves for the same cultivar were collected from the experimental field of Khartoum University farm, Faculty of Agriculture. Calyces and leaves of karkade were fermented traditionally for 3, 5 and 7 days and compared with unfermented (control) sample. The moisture contents of calyces and leaves had dropped by 10% and 25% respectively, protein content increased in calyces from 8.53% to 10.57% and decreased from 8.75% to 7.07% in the leaves. The fermentation time increased the fiber content by 37% in calyces and slightly affected that of leaves. There was no significant difference in the oil content of calyces, with increasing of 36% in the content of leaves. The ash content increased significantly ($p < 0.05$) from 8.57% to 10.67% while that of leaves was not

significantly affected ($p>0.05$). Carbohydrate content decreased by 16% in the calyces, but it was increased 6% in the leaves. Mineral contents of karkade calyces and leaves were found to be generally dropped throughout the fermentation time. Fermentation generally tend to decrease the amounts of antinutritional factors in calyces and leaves. The effect was clearly observed on phytic acid ($p<0.05$) which decreased from 522.50 mg/100g in the beginning to 356.40 mg/100g at the end of fermentation and continued by the same way in the leaves. This effect also happened in the contents of polyphenols and tannins [8].

Growth plant is the result of transfer of solar radiation in to the photosynthetic processes of green leaf and transmission of photosynthates into increased biomass. Recently, many researchers have manipulated plant row-spacing and direction as well as plant populations within the row, to increase crop production through more efficient use of solar energy. A field experiment was conducted in North Kordofan State of Sudan, to investigate the effect of intra – row spacing (10, 20, 30 and 40cm) and planting stand (two and three plants per stand) on yield and yield components of *Hibiscussabdariffa* L. The results showed that, plant population had a significant effect on most of the attributes measured. Closer spacing increased the number of branches per plant, days to 50% flowering, days to 95% physiological maturity, number of calyces per plant, calyces yield (g/plant) and decreased final calyces yield (t/ha). Spacing of 20 cm and three plants per stand gave highest calyces yield (t/ha)[9].

Roselle (*Hibiscus sabdariffa*) were evaluated at Shambat (Sudan) for two consecutive seasons to assess genetic variability and heritability of different characters. Field experiment was conducted at Shambat Demonstration Farm in a randomized complete block design with three replications. Data were collected on thirteen plant attributes. Analysis of variance indicated significant differences among the sixteen genotypes for most of the characters

studied. In general, the morphological characters had larger environmental variances than their respective genotypic ones. The highest genotypic coefficient of variation was exhibited by the seed yield/plant in both seasons. The highest genetic advance, 46.63 % in the first season and 170.13 % in the second season were obtained both for calyx x yield/unit area. High values of heritability estimates (> 0.70) were recorded for days to 50 % flowering over the two seasons. On the other hand, the yield components showed low heritability estimates (< 0.50) except for fruit weight in the second season [10].

Fields experiments were conducted at North Kordofan state, Sudan, on naturally infested fields within the same area, using three similar fields during 2008/2009 rainy season, to determine optimal weeding frequency for weeding management in two widely used cultivated varieties of *Hibiscus sabdariffa* L, (Elrahad and Elfashir). The weeding (hand hoeing) treatments consisted of four levels (no weeding, weeding once (at 2 weeks), weeding twice (at 2 and 3 weeks) and weeding three times (at 2, 4 and 6 weeks after sowing). Weeding three times at 2, 4 and 6 weeks after sowing was optimal for plant height, leaf area index, number of branches, number of calyces per plant, calyx diameter and calyces yield per unit area. Elfashir variety was superior in all frequencies of weeding. The land used was dominated by *Cenchrus biflorus* L [11].

The antioxidant properties of the water extract of *Hibiscus sabdariffa* L. as well as their aroma profile were investigated. The samples analyzed were three red (Al-Rahad, Al-Fashir, Al-Gezira) and one white varieties. Total phenolic content, total flavonoid content, total anthocyanin content, ferric reducing antioxidant power and radical scavenging activity (RSA %) were measured spectrophotometrically. The red varieties of Karkade showed better overall antioxidant properties and aroma profile compared to the white variety. Conversely, the white *H. sabdariffa* might also be used in

antioxidants applications, as its extract exhibited the highest RSA % compared to other varieties. Nineteen volatile compounds were detected in the H. sabdariffa extracts, including aldehydes, alcohols, ketones, esters and phenol. Partial least squares regression (PLSR) was applied to determine the relationship between sensory descriptors and instrumental data. PLSR result showed a good relationship between sensory analysis and instrumental data (t_1 and $t_2 = 94.63\%$) [12].

1-5: Thesis layout

This research includes five chapters, chapter one introduction, chapter two theoretical background, chapter three material, chapter four experiment and methodology and chapter five conclusion and recommendation.

CHAPTER TWO

CHAPTER TWO

THEORETICAL BACKGROUND

2-1: History of Material Science

Materials are probably more deep-seated in our culture than most of us realize. Transportation, housing, clothing, communication, recreation and food production virtually every segment of our everyday lives is influenced to one degree or another by materials. Historically, the development and advancement of societies have been intimately tied to the members' ability to produce and manipulate materials to fill their needs. In fact, early civilizations have been designated by the level of their materials development (Stone Age, Bronze Age, Iron Age)[13]. The earliest humans had access to only a very limited number of materials, those that occur naturally: stone, wood, clay, skins and so on. With time they discovered techniques for producing materials that had properties superior to those of the natural ones, these new materials included pottery and various metals. Furthermore, it was discovered that the properties of a material could be altered by heat treatments and by the addition of other substances. At this point, materials utilization was totally a selection process that involved deciding from a given, rather limited set of materials the one best suited for an application by virtue of its characteristics. It was not until relatively recent times that scientists came to understand the relationships between the structural elements of materials and their properties. This knowledge, acquired over approximately the past 100 years, has empowered them to fashion, to a large degree, the characteristics of materials. Thus, tens of thousands of different materials have evolved with rather specialized characteristics that meet the needs of our modern and complex society; these include metals, plastics, glasses and fibers. The

development of many technologies that make our existence so comfortable has been intimately associated with the accessibility of suitable materials.

An advancement in the understanding of a material type is often the forerunner to the stepwise progression of a technology. For example, automobiles would not have been possible without the availability of inexpensive steel or some other comparable substitute. In our contemporary era, sophisticated electronic devices rely on components that are made from what are called semiconducting materials[14].The field has since broadened to include every class of materials, including ceramics,polymers,semiconductors,magnetic materials,medicalimplant materials, biological materials,nanomaterials, with modern materials classed within 3 distinct groups: Ceramic, Metal or Polymer. The prominent change in materials science during the last two decades is active usage of computer simulation methods to find new compounds, predict various properties and as a result design new materials at a much greater rate than previous years [15].

2-2: Classification of Materials

Solid materials have been conveniently grouped into three basic categories: metals, ceramics and polymers. This scheme is based primarily on chemical makeup , atomic structureand most materials fall into one distinct grouping or another. In addition,there are the composites, which are engineered combinations of two or more different materials. A brief explanation of these material classifications and representative characteristics is offered next. Another category is advanced materialsthose used in high-technology applications,such as semiconductors,biomaterials, smart materials and Nano engineered materials classified as follows :

1. Metals

Materials in this group are composed of one or more metallic elements (e.g iron, aluminum, copper, titanium, gold and nickel) and often also nonmetallic elements (e.g carbon, nitrogen and oxygen) in relatively small amounts. Atoms in metals and their alloys are arranged in a very orderly manner and in comparison to the ceramics and polymers, are relatively dense. With regard to mechanical characteristics, these materials are relatively stiff and strong, yet are ductile (i.e., capable of large amounts of deformation without fracture) and are resistant to fracture, which accounts for their widespread use in structural applications. Metallic materials have large numbers of nonlocalized electrons, that is, these electrons are not bound to particular atoms. Many properties of metals are directly attributable to these electrons. For example, metals are extremely good conductors of electricity and heat and are not transparent to visible light, a polished metal surface has a lustrous appearance. In addition, some of the metals (i.e Fe, Co and Ni) have desirable magnetic properties [14].

2. Ceramics

Are compounds between metallic and nonmetallic elements, they are most frequently oxides, nitrides and carbides. For example, common ceramic materials include aluminum oxide (or alumina, Al_2O_3), silicon dioxide (or silica, SiO_2), silicon carbide (SiC), silicon nitride (Si_3N_4), in addition, what some refer to as the traditional ceramics those composed of clay minerals (i.e., porcelain), as well as cement and glass. With regard to mechanical behavior, ceramic materials are relatively stiff and strong, stiffnesses and strengths are comparable to those of the metals. In addition, they are typically very hard. Historically, ceramics have exhibited extreme brittleness (lack of ductility) and are highly susceptible to fracture. However, newer ceramics are being engineered to have improved resistance to fracture, these materials are

used for cookware, cutlery and even automobile engine parts. Furthermore, ceramic materials are typically insulative to the passage of heat and electricity (i.e. have low electrical conductivities) and are more resistant to high temperatures and harsh environments than metals and polymers. With regard to optical characteristics, ceramics may be transparent, translucent or opaque and some of the oxide ceramics (e.g. Fe_3O_4) exhibit magnetic behavior.

3. Polymers

Polymers include the familiar plastic and rubber materials. Many of them are organic compounds that are chemically based on carbon, hydrogen and other nonmetallic elements (i.e. O, N and Si). Furthermore, they have very large molecular structures, often chainlike in nature, that often have a backbone of carbon atoms. Some of the common and familiar polymers are polyethylene (PE), nylon, poly (vinyl chloride) (PVC), polycarbonate (PC), polystyrene (PS) and silicone rubber. These materials typically have low densities, whereas their mechanical characteristics are generally dissimilar to the metallic and ceramic materials; they are not as stiff nor as strong as these other material types. However, on the basis of their low densities, many times their stiffnesses and strengths on a per-mass basis are comparable to the metals and ceramics. In addition, many of the polymers are extremely ductile and pliable (i.e. plastic), which means they are easily formed into complex shapes. In general, they are relatively inert chemically and unreactive in a large number of environments. One major drawback to the polymers is their tendency to soften or decompose at modest temperatures, which in some instances, limits their use. Furthermore, they have low electrical conductivities and are nonmagnetic [14].

4. Composites

A composite is composed of two (or more) individual materials, which come from the categories previously discussed: metals, ceramics and polymers. The design goal of a composite is to achieve a combination of properties that is not displayed by any single material and also to incorporate the best characteristics of each of the component materials. A large number of composite types are represented by different combinations of metals, ceramics and polymers. Furthermore, some naturally occurring materials are composites; for example, wood and bone. However, most of those we consider in our discussions are synthetic (or human-made) composites. One of the most common and familiar composites is fiberglass, in which small glass fibers are embedded within a polymeric material (normally an epoxy or polyester). The glass fibers are relatively strong and stiff (but also brittle), whereas the polymer is more flexible. Thus, fiberglass is relatively stiff, strong and flexible. In addition, it has a low density. Another technologically important material is the carbon fiber-reinforced polymer (CFRP) composite: carbon fibers that are embedded within a polymer. These materials are stiffer and stronger than glass fiber-reinforced materials, but more expensive. CFRP composites are used in some aircraft and aerospace applications, as well as high-tech sporting equipment (e.g. bicycles, golf clubs, tennis rackets, skis/snowboards) and recently in automobile bumpers. The new Boeing 787 fuselage is primarily made from such CFRP composites [14].

5. Advanced Materials

Materials that are utilized in high technology (high-tech) applications are sometimes termed advanced materials. By high technology we mean a device or product that operates or functions using relatively intricate and sophisticated principles; examples include electronic equipment (camcorders, CD/DVD players, etc.), computers, fiber-optic systems, spacecraft, aircraft and

military rocketry. These advanced materials are typically traditional materials whose properties have been enhanced and also newly developed, high-performance materials. Furthermore, they may be of all material types (e.g. metals, ceramics, polymers) and are normally expensive. Advanced materials include semiconductors, biomaterials and what we may term “materials of the future” (that is smart materials and nanoengineered materials). The properties and applications of a number of these advanced materials for example, materials that are used for lasers, integrated circuits, magnetic information storage, liquid crystal displays (LCDs) and fiber optics.

a) Semiconductors

Semiconductors have electrical properties that are intermediate between the electrical conductors (i.e. metals and metal alloys) and insulators (i.e. ceramics and polymers). Furthermore, the electrical characteristics of these materials are extremely sensitive to the presence of minute concentrations of impurity atoms, for which the concentrations may be controlled over very small spatial regions. Semiconductors have made possible the advent of integrated circuitry that has totally revolutionized the electronics and computer industries over the past three decades.

b) Bio Materials

Biomaterials are employed in components implanted into the human body to replace diseased or damaged body parts. These materials must not produce toxic substances and must be compatible with body tissues (i.e. must not cause adverse biological reactions). All of the preceding materials metals, ceramics, polymers, composites and semiconductors may be used as biomaterials [14].

c) Smart Materials

Smart (intelligent) materials are a group of new and state-of-the-art materials now being developed that will have a significant influence on many of our technologies. The adjective smart implies that these materials are able to sense

changes in their environment and then respond to these changes in predetermined mannerstraits that are also found in living organisms. In addition, this “smart” concept is being extended to rather sophisticated systems that consist of both smart and traditional materials. Components of a smart material (system) include some type of sensor (that detects an input signal) and an actuator (that performs a responsive and adaptivefunction). Actuators may be called upon to change shape, position, natural frequency, or mechanical characteristics in response to changes in temperature, electric fields and magnetic fields. Four types of materials are commonly used for actuators: shape memory alloys, piezoelectric ceramics,magnetostrictive materials and electrorheological magnetorheological fluids. Shape memory alloys are metals that, after having been deformed, revert back to their original shape when temperature is changed.Piezoelectric ceramics expand and contract in response to an applied electric field (or voltage), conversely, they also generate an electric field when their dimensions are altered. The behavior of magnetostrictive materials is analogous to that of the piezoelectrics, except that they are responsive to magnetic fields. Also,electrorheological and magnetorheological fluids are liquids that experience dramatic changes in viscosity upon the application of electric and magnetic fields, respectively. Materialsdevices employed as sensors include optical fibers,piezoelectric materials (including some polymers)and microelectromechanical systems (MEMS). For example, one type of smart system is used in helicopters to reduce aerodynamic cockpit noise that is created by the rotating rotor blades. Piezoelectric sensors inserted into the blades monitor blade stresses and deformations,feedback signals from these sensors are fed into a computer-controlled adaptive device,which generates noisecancelingantinoise [14] .

d) Nano Materials

One new material class that has fascinating properties and tremendous technological promise is the nanomaterials. Nanomaterials may be any one of the four basic types metals, ceramics, polymers and composites. However, unlike these other materials, they are not distinguished on the basis of their chemistry but rather size the nanoprefix denotes that the dimensions of these structural entities are on the order of a nanometer (10^{-9} m) as a rule, less than 100 nanometers (equivalent to approximately 500 atom diameters). Prior to the advent of nanomaterials, the general procedure scientists used to understand the chemistry and physics of materials was to begin by studying large and complex structures, then to investigate the fundamental building blocks of these structures that are smaller and simpler. This approach is sometimes termed “top-down” science[14]. On the other hand, with the development of scanning probe microscopes, which permit observation of individual atoms and molecules, it has become possible to design and build new structures from their atomic level constituents, one atom or molecule at a time (i.e. “materials by design”). This ability to carefully arrange atoms provides opportunities to develop mechanical, electrical, magnetic and other properties that are not otherwise possible. We call this the “bottom-up” approach and the study of the properties of these materials is termed nanotechnology. Some of the physical and chemical characteristics exhibited by matter may experience dramatic changes as particle size approaches atomic dimensions. For example, materials that are opaque in the macroscopic domain may become transparent on the nanoscale, some solids become liquids, chemically stable materials become combustible and electrical insulators become conductors. Furthermore, properties may depend on size in this nanoscale domain. Some of these effects are quantum mechanical in origin, others are related to surface phenomena the proportion of atoms

located on surface sites of a particle increases dramatically as its size decreases [14].

2-3: Properties of Materials

Different materials possess different properties to meet the various requirement for engineering purposes [16]. The properties of materials which dictate the selection are as follows:

2-3-1: Mechanical Properties

The important mechanical properties affecting the selection of material are:

- i. **Tensile Strength:** This enable the material to resist the application of tensile force .To withstand the tensile force , the internal structure of the material provides the internal resistance .
- ii. **Hardness:** It is the degree of resistance to indentation or scratching , abrasion and wear . Alloying techniques and heat treatment help to achieve the same.
- iii. **Ductility:** This is the property of a metal by virtue of which it can be drawn into wires or elongated before rupture takes place. It depends upon the grain size of the metal crystals.
- iv. **Impact strength:** It is energy required per unit cross-sectional area to fracture a specimen, i.e it is a measure of the response of a material to shock loading.
- v. **Wear Resistance:** The ability of a material to resist friction wear under particular conditions, i.e to maintain its physical dimensions when in sliding or rolling contact with a second member .
- vi. **Corrosion Resistance:** Those metals and alloys which can withstand the corrosive action of a medium , i.e corrosion processes proceed in them at a relatively low rate are termed corrosion- resistant .
- vii. **Density :** This is an important factor of a material where weight and thus the mass is critical ,i.e aircraft components .

2-3-2: Thermal Properties

The characteristics of a material, which are function of temperature are termed as thermal properties. One can predict the performance of machine components during normal operation, thermal properties are specific heat, latent heat, thermal conductivity, thermal expansion, thermal stresses, thermal fatigue, etc [16].

- i. Specific Heat (c): It is heat capacity of a unit mass of a homogeneous body, specific heat is given by:

$$C = C_H / M \quad (2.1)$$

where C_H is heat capacity and M is the mass of the body. Its units are cal/g/°C. Thermal Conductivity (k): This represents the amount of heat conducted per unit time through a unit area perpendicular to the direction of heat conduction when the temperature gradient across the heat conducting elements is one unit [16]. Copper and aluminium are good conductors of heat and therefore extensively used whenever transfer of heat is desired. Bakelite is a poor conductor of heat and hence used as heat insulator. Its unit is W/m/K, the thermal conductivity (k) is given by:

$$k = Q \cdot x / (A(\theta_1 - \theta_2)t) \quad (2.2)$$

Where Q is flow of heat, A is face area, t is time, θ_1 and θ_2 are temperatures of hot and cold side of the material and x is the distance between two faces.

The thermal conductivity of metal can be expressed as:

$$K = \frac{\pi^2}{3} \left(\frac{k}{e} \right)^2 T n e^2 \lambda / 2 m v_0 \quad (2.3)$$

Where λ mean free path, k Boltzmann constant , m electron mass , e electronic charge , v_0 initial velocity of the electron .The ratio of heat and electrical conductivity (k and σ respectively) is given by:

$$K/ \sigma = \pi^2/3 (k/e)^2 T \quad (2.4)$$

- ii. **Thermal Expansion:** All solids expand on heating and contract on cooling. Thermal expansion may take place either as linear, circumferential or cubical. A solid which expands equally in three mutually orthogonal directions is termed as thermally isotropic. The increase in any linear dimension of a solid , e.g length, width ,height on heating is termed as linear expansion .The coefficient of linear expansion is increase in length per unit length per degree rise in temperature . The increase in volume of a solid on heating is called cubical expansion .The thermal expansion of a solid has its origin in the lattice vibration and lattice vibrations increases with the rise in temperature [16].
- iii. **Thermal Resistance (R_T):** It is the resistance offered by the conductor when heat flow due to temperature difference between two points of a conductor. It is given by:

$$R_{th(j-a)} = (T_{j \text{ Max}} - T_a) / P_{\text{Max}} \quad (2.5)$$

Where: $R_{th(j-a)}$: Junction-to-ambient thermal resistance K/W

$T_{j \text{ Max}}$: Maximum junction temperature K

T_a : Ambient temperature (temperature condition of P_{Max}) K

P_{Max} : Maximum power dissipation / W [17].

- iv. **Thermal Diffusivity (h) :** It is given by:

$$h = k / (C_p) (\rho) \text{ cm}^2/\text{s} \quad (2.6)$$

Where: k thermal conductivity, C_p heat capacity and ρ density.

A material having high heat requirement per unit volume possesses a low thermal diffusivity because more heat must be added to or removed from the material for effecting a temperature change.

- v. Thermal fatigue: This is mechanical effect of repeated thermal stresses caused by repeated heating and cooling.

The thermal stresses can be very large , involving considerable plastic flow . We can see that fatigue failures can occur after relatively few cycles. The effect of the high part of the temperature cycle on the strength of material plays an important factor in reducing its life under thermal fatigue.

2-3-3: Electrical Properties

Conductivity ,resistivity, dielectric strength are few important electrical properties of a material .A material which offers little resistance to the passage of an electric current is said to be a good conductor of electricity .The electrical resistance of material depends on its dimensions and is given by:

$$\text{Resistance} = \text{Resistivity} \times (\text{length} / \text{cross-section area}) \quad (2.7)$$

Usually resistivity of a material is quoted in the literature, unit of resistivity is ohm-meter. On the basis of electrical resistivity materials are divided as:

- Conductor
- Semiconductor
- Insulators

In general metals are good conductors .Insulators have very high resistivity .Ceramic insulators are most common examples and are used on automobile spark plugs , Bakelite handles for electric iron ,plastic coverings on cables in domestic wiring. When a large number of metals and alloys are sufficiently

cooled below transition temperature, T_c , enter the state of superconductivity in which the do resistivity goes to zero. The estimates of the resistivity in the super-conducting phase place it at less than $4 \times 10^{-25} \Omega.m$, which is essentially zero for all practical purposes. The highest value of T_c up to 133k has been reached mercury cuprate [16].

2-3-4: Magnetic Properties

Material which a state of magnetism can be induced are termed magnetic materials. There are five classes into which magnetic material may be grouped:

- i. Diamagnetic
- ii. Paramagnetic
- iii. Ferromagnetic
- iv. Antiferromagnetic
- v. Ferrimagnetic

Iron, Cobalt, Nickel and some of their alloys and compounds possess spontaneous magnetization[16]. Magnetic oxides like ferrites and garnets could be used at high frequencies. Because of their excellent magnetic properties along with their high electrical resistivity these material today find use in a variety of applications like magnetic recording tapes, inductor and transformers, memory elements, microwave device, bubble domain devices, recording hard cores, etc. Hysteresis, permeability and coercive force are some of the magnetic properties of magnetic substances which are to be considered for the manufacture of transformers and other electronic components.

2-3-5: Chemical Properties

These properties includes atomic weight , molecular weight ,atomic number ,valency, chemical composition, acidity ,alkalinity ,etc. These properties govern the selection of materials particularly in chemical plant .

2-3-6: Optical Properties

Theoptical propertiesof materials ,e.g refractive index , reflectivity and absorption coefficient etc, affect the light reflection and transmission.

2-4: Material Structure

Inrecent years , the number and variety of materials , which are of particular interest to an engineer have increased tremendously .Each type of material has specific composition possessing specific properties for specific use .

It is not possible for one to explain the properties of all types of these materials. Material structure can be classified as:

- a) Macro Structure: Macrostructure of a material is examined by low–power magnification or naked eye. It deals with the shape ,size and atomic arrangement in a crystalline material. In case of some crystals ,e.g quartz ,external form of the crystal may reflect the internal symmetry of atoms. Macrostructure may be observed directly on a fracture surface or on a forging specimen . The individual crystal of a crystalline material can be visible ,e.g in brass doorknob by the constant polishing and etching action of a human hand and sweat . Macrostructure can reveal flaws , segregation , cracks etc, by using proper techniques and one can save much expenses by rejecting defective materials at an early stage[16].
- b) Micro Structure: This generally refers to the structure of the material observed under optical microscope . Optical microscopes can magnify a structure about 1500 to 3000 times linear , without loss to resolution of details of the material structure . We may note that optical microscopes

can resolve two lines separately when their difference of separation is 10^{-7} m (0.1 μ m). Cracks, porosity, non-metallic inclusions within materials can be revealed by examining them under powerful optical microscope.

- c) Sub Structure: When crystal imperfections such as dislocations in a structure are to be examined, a special microscope having higher magnification and resolution than the optical microscope is used. Electron microscope with magnification 10^5 are used for this purpose. Another important modern microscope is field ion microscope, which can produce images of individual atoms as well as defects in atomic arrangements.
- d) Crystal Structure: This reveals the atomic arrangement within a crystal. X-ray diffraction method are commonly used for studying crystal structure. It is usually sufficient to study the arrangement of atoms within a unit cell. The crystal is formed by a very large number of unit cells forming regularly repeating pattern in space.
- e) Electronic Structure: This refers to the electrons in the outermost shells of individual atoms that form the solid. Spectroscopic techniques are commonly used for determining the electronic structure.
- f) Nuclear Structure: This is studied by nuclear Spectroscopic techniques, e.g. nuclear magnetic resonance (NMR) and Mossbauer Spectroscopy [16].
- g) Nano Structure: Nanostructure deals with objects and structures that are in the 1—100 nm range [18]. In many materials, atoms or molecules agglomerate together to form objects at the nanoscale. This causes many interesting electrical, magnetic, optical and mechanical properties. Nanotextured surfaces have one dimension on the nanoscale, i.e. only the thickness of the surface of an object is between 0.1 and 100 nm.

2-5: Applications of Materials

There are many applications of the material as follows:

- ❖ Materials are commonly used for actuators shape memory alloys, piezoelectric ceramics, magnetostrictive materials and

electrorheological magnetorheological fluids. Shape memory alloys are metals that, after having been deformed, revert back to their original shape when temperature is changed. A smart system is used in helicopters to reduce aerodynamic cockpit noise that is created by the rotating rotor blades.

- ❖ Composites are used in some aircraft and aerospace applications, as well as high-tech sporting equipment (e.g. bicycles, golf clubs, tennis rackets, skis/snowboards) and recently in automobile bumpers. The new Boeing 787 fuselage is primarily made from such CFRP composites [14].
- ❖ Ceramics are used for cookware, cutlery and even automobile engine parts. Furthermore, ceramic materials are typically insulative to the passage of heat and electricity (i.e. have low electrical conductivities) and are more resistant to high temperatures and harsh environments than metals and polymers.
- ❖ Semiconductors have made possible the advent of integrated circuitry that has totally revolutionized the electronics and computer industries over the past three decades.
- ❖ Advanced materials are used in electronic equipment (camcorders, CD/DVD players, etc.), computers, fiber optic systems, spacecraft, aircraft and military rocketry are used for lasers, integrated circuits, magnetic information storage, liquid crystal displays (LCDs) and fiber optics [14].

2-6: Conductivity

Electrical conductivity is one of the physical properties of a material which depends on the structure of that material and hence on the bonds that hold the material together. In order to choose a material for fabrication and suitability in use we should consider building blocks & properties of the material.

interaction of that material with environment, other materials surrounding it and cost of the material[19].In physics, thermal conductivity is the property of a material's ability to conduct heat. It appears primarily in Fourier's Law for heat conduction. Thermal conductivity is measured in watts per kelvin-meter ($W/(K \cdot m)$) or in IP units ($Btu/(hr \cdot ft \cdot F)$). Multiplied by a temperature difference (K) and an area (m^2) and divided by a thickness (m), the thermal conductivity predicts the rate of energy loss (W) through a piece of material. In the window building industry "thermal conductivity" is expressed as the U-Factor [20].

2-7: Types of Conductivity

a) Electrical Conductivity

Electrical Conductivity is the ability of a material to carry an electrical current. The term conductivity can also be used in other contexts (e.g thermal conductivity).Electrical conductivity (EC) is a useful indicator of total dissolved solids (TDS) because the conduction of current in an electrolyte solution is primarily dependenton the concentration of ionic species [21].

An increase in a solution's temperature will cause a decrease in its viscosity and an increase in the mobility of the ions in solution. An increase in temperature may also cause an increase in the number of ions in solution due to dissociation of molecules. As the conductivity of a solution is dependent on these factors then an increase in the solution's temperature will lead to an increase in its conductivity [22].

Ohm's law defines the resistance of a sub stance to the pas sage of an electrical current as:

$$R = V/I \quad (2.8)$$

Where R is the electrical resistance (Ohms), V is electrical potential (Volts), and I is current (Amperes). The resistivity of a sub stance is defined by the

resistance to electrical current passing between the faces of a unit cube of the substance. The SI units of resistivity are Ohm·m. For fluids, the inverse of resistivity or conductivity, is conventionally used to characterize their ability to transmit electrical currents. The SI units of EC are thus 1/(Ohm·m). The units for the inverse of ohms were, in the past, often designated as “mho”, but the preferred unit is siemen (abbreviated “S”).

Sometimes, electrical conductivity σ is used to specify the electrical character of a material [23]. It is simply the reciprocal of the resistivity, given by:

$$\sigma = 1/\rho \quad (2.9)$$

Where σ is Conductivity and ρ is resistivity ($\Omega \cdot m^{-1}$)

b) Thermal Conductivity:

For general scientific use, thermal conductance is the quantity of heat that passes in unit time through a plate of particular area and thickness when its opposite faces differ in temperature by one kelvin. For a plate of thermal conductivity k , area A and thickness L this is kA/L , measured in $W \cdot K^{-1}$ (equivalent to: $W/^\circ C$). Thermal conductivity and conductance are analogous to electrical conductivity ($A \cdot m^{-1} \cdot V^{-1}$) and electrical conductance ($A \cdot V^{-1}$). There is also a measure known as heat transfer coefficient: the quantity of heat that passes in unit time through unit area of a plate of particular thickness when its opposite faces differ in temperature by one Kelvin [24].

Thermal conductivity depends on many properties of a material, notably its structure and temperature. For instance, pure crystalline substances exhibit very different thermal conductivities along different crystal axes, due to differences in phonon coupling along a given crystal axis. Sapphire is a notable example of variable thermal conductivity based on orientation and temperature. Thermal conductivity is important in building insulation and related fields. However, materials used in such trades are rarely subjected

tochemical purity standards [25]. Temperature gradients in a solid produce a heat flux as follows:

$$q = -\lambda \, dT / dx \quad (2.10)$$

Where q is the heat flux density (thermal energy per unit area and time), T is the temperature and λ is the thermal conductivity. The magnitude of λ determines whether a material is a good or a poor thermal conductor. Usually excellent electrical conductors, i.e. metal are also good thermal conductors and vice versa, electrical insulators usually also have a low thermal conductivity.

2-8: Spectroscopy

Spectroscopy is the study of the interaction between matter and electromagnetic radiation. Historically, spectroscopy originated through the study of visible light dispersed according to its wavelength, by a prism. Later the concept was expanded greatly to comprise any interaction with radiative energy as a function of its wavelength or frequency.

2-8-1: Types of Spectroscopy

The Types of Spectroscopy as follows:

1. Infrared Spectroscopy

The mid- (fundamental) infrared region (IR or MIR) extends from 4000 cm^{-1} ($\lambda = 2.5 \mu\text{m}$) to 400 cm^{-1} ($25 \mu\text{m}$). It is surrounded by the far-IR region (FIR) from 400 cm^{-1} ($25 \mu\text{m}$) to 10 cm^{-1} (1 mm) and the very important near-IR region (NIR) from 12500 cm^{-1} (800 nm) to 4000 cm^{-1} ($2.5 \mu\text{m}$). Infrared spectroscopy is the most commonly used spectroscopic method. There are a number of reasons for its great success and dissemination [26]. The method is rapid, sensitive, easy to handle and provides many different sampling techniques for gases, liquid and solids. Important aspects are the convenient qualitative and quantitative evaluation of the spectra. The standard format of an IR spectrum is transmittance [%T] versus wavenumber [cm^{-1}]. According

to IUPAC recommendations the values of the wavenumber axis decrease towards its right-hand end. The features of an IR spectrum (number of infrared absorption bands, their intensities and their shapes) are directly related to the molecular structure of a compound. The IR spectrum is a unique physical property of an individual compound, its molecular fingerprint. The IR region comprises fundamental vibrations of bound atoms. Whenever such bound atoms vibrate, they absorb infrared energy, i.e. they exhibit IR absorption bands.

2. Raman Spectroscopy

Raman spectroscopy is a spectroscopic method, which is complementary to IR spectroscopy. It offers various advantages over MIR and NIR spectroscopy. Since water is a weak scatterer in the VIS range, no special accessories are needed for measuring aqueous solutions. Furthermore, atmospheric gases are very weak scatterers, therefore purging of the Raman instrument is not needed. Ordinary glass is transparent in the visible and near-infrared spectral regions, where Raman spectra are excited so inexpensive liquid sample cells made from glass can be used for most Raman measurements. For remote analysis glass fiber optics can be used. The standard Raman spectral range extends down to 10 cm^{-1} , so the technique is ideal for both organic and inorganic samples. The limitations of Raman spectroscopy in comparison to IR are sensitivity and undesired fluorescence. Relatively expensive and sophisticated instrumentation also should be taken into account [26, 27].

3. UV/VIS Spectroscopy

Absorptions in the UV/VIS are associated with electronic transitions from the ground level to an excited state. The strongest transitions are $\sigma\text{-}\sigma^*$, they are observed below 200 nm (vacuum UV). Typical examples are C-C and C-H

bonds. Because all molecules include σ electrons, σ - σ^* transitions constitute the short-wavelength cut-off of the routine UV/VIS range. Saturated compound with pairs of free electrons exhibit n - π^* transitions in a wavelength range from 150 to 250 nm, their absorption coefficients do not exceed $1000 \text{ l cm}^{-1} \text{ mol}^{-1}$ [26].

Most UV/VIS investigations are based on n - π^* or π - π^* transitions, which occur across the UV/VIS range and result from unsaturated groups. Typical absorption coefficients of n - π^* transitions are below $100 \text{ l cm}^{-1} \text{ mol}^{-1}$, while absorption coefficients of π - π^* transitions exceed $1000 \text{ l cm}^{-1} \text{ mol}^{-1}$. Absorptions of transition metal ions are caused by their 3d and 4d electrons, whereas 4f and 5f electrons are excited in lanthanide and actinide ions. Absorption bands of d and f electrons are sharper than those of most chromophores because the inner orbitals are largely shielded from external influences. Transitions of donor electrons to an acceptor orbital (charge transfer complexes) originate in inorganic as well as organic compounds, their absorption coefficients usually exceed $10000 \text{ l cm}^{-1} \text{ mol}^{-1}$. Band transitions in solids also lead to UV/VIS absorptions. Such transitions may occur between valence and conduction bands or between a band and a localized energy level in the forbidden zone. Such conditions may occur for instance in the case of lattice defects or point defects [26].

4. Near-Infrared Spectroscopy

The near infrared (NIR) spans the range from 12500 - 4000 cm^{-1} (800 - 2500 nm) and is dominated by overtones and combinations of O-H, N-H, C-H and C-O vibrations. Overtone and combination bands are rather weak. Band intensities usually drop by a factor between 10 and 100 from excitation level to excitation level. The low absorbance of overtones and combinations usually restricts the application range of NIR spectroscopy to liquids and solids. Absorption regions for some important groups and vibrations. In

addition, the short wavelength NIR range covers lowest-energy electronic transitions. Fermi and other resonances occur in the region as well

[5, 26]. NIR is increasingly used in process and environmental analysis, the food industry, agriculture, the pharmaceutical industry and polymer analysis. In-line measurement with fiber optics and rapid multi-component quantification are the most important advantages of NIR spectroscopy. In comparison to mid-infrared, NIR analysis is much faster and more versatile. Most samples are analysed in one minute or less.

5. Mid-Infrared (MIR) Spectroscopy

In the MIR spectral region we are dealing with transitions between various vibrational energy levels of molecules. Gaseous samples are a special case, because rotational fine-splitting of spectral bands can be observed. Fine-splitting is caused by simultaneous excitation of rotational and vibrational transitions. The MIR spectral range extends from 4000 to 400 cm^{-1} . Transitions can be observed by absorption or emission measurements. For analytical purposes, absorption measurements are usually preferred. The decision about an optimal sampling technique is very much dependent on the aggregate state of the sample under investigation [5].

6. Fluorescence Spectroscopy

After the appearance of the first book on fluorescence in 1951 [28], fluorescence spectroscopy became a widely used scientific tool in biochemistry, biophysics and in material science. In the last few years, however, several new applications based on fluorescence have been developed, promoting fluorescence spectroscopy from a primarily scientific to a more routine method. The phenomena of fluorescence is for example exploited in simple analytical assays in environmental science and clinical

chemistry, in cell identification and sorting in flow cytometry and in imaging of single cells in medicine. The analyte, whose light emission is investigated, is often called a “dye”. Fluorescence measurements give information about the photophysical properties of the dye as well as about the chemical and physical nature of the surroundings of the dye [27].

2-8-2: Types of Instruments

There are a few basic types of instruments which are used in optical spectroscopy for the determination of absorption, fluorescence or Raman spectra of condensed and gaseous samples. These basic types are monochromators, interferometers and polychromators.

The wavelength range of optical spectroscopy extends from 200 nm (UV) to 500 μm (FIR). It is impossible to build one single spectral instrument capable of covering the region completely and providing information about the different processes of absorption, emission and scattering of light. Light sources, detectors and other optical components have limited operational ranges, caused by the underlying physical work principles. The choice of the appropriate instrument type depends on the application. The interaction process of light with the material and the spectral interval of interest have to be taken into account. Traditionally, spectrometers for absorption measurements are optimized for (UV/VIS) (175-750 nm), (NIR) (0.8-2.5 μm), (MIR) (2.5-25 μm) and (FIR) (25-1000 μm) ranges. Some commercial spectrometers are capable of covering neighboring spectral regions (for instance, UV/VIS/NIR or MIR/ FIR). Spectrometers for investigation of scattering and emission of light belong to different classes of instruments. Raman and fluorescence spectrometers belong in this group [28].

❖ NIR Spectrometers

Absorption of electromagnetic radiation in the NIR region is caused by overtone and combination vibrations. Polyatomic molecules exhibit many

overtone and combination vibrations, their spectral bands overlap and make typical NIR bands look very broad and featureless. Nevertheless, NIR spectra contain molecular information about the sample and this information can be extracted by means of chemometric methods. A prerequisite for chemometric evaluations is high quality of the collected spectral data. Therefore, wavelength precision, resolution, photometric precision and signal-to-noise ratio are important criteria for the selection of an NIR spectrometer. Among all optical spectroscopic methods, NIR offers the greatest diversity of instrumentation principles and the market for commercially available instruments is undergoing continuous change and growth. NIR has an enormous variety of applications, e.g. in agriculture, in food processing, in medical and in pharmaceutical applications, in polymer and plastics processing, in environmental analysis, in material recycling and in satellites or aircraft for remote sensing [29, 30]. Commercial NIR spectrometers vary remarkably with respect to cost, size and portability, measurement time and environmental conditions for on-line applications in industry. According to their measurement principles, NIR spectrometers fall into one of six categories:

1. Fourier-Transform spectrometers
2. Scanning-Grating spectrometers
3. Diode array spectrometers (fixed-grating spectrometers)
4. Filter spectrometers
5. LED (light-emitting diode) spectrometers
6. AOTF (acousto-optical tuneable filter) spectrometers

❖ Raman Spectrometers

Raman spectrometers are used to analyze light scattered by molecules [26, 31]. A major advantage of Raman spectroscopy is the high spatial resolution that can be obtained, typically of the order of 1 μm (compared to approx. 10 μm with FT-IR). In conventional Raman experiments the sample is illuminated by monochromatic light. The registration of low intensity Raman scattering in the presence of strong Tyndall and Rayleigh scattering implies special requirements for Raman spectrometers. A Raman spectrometer has to combine very good filter characteristics for eliminating Rayleigh and Tyndall scattering with high sensitivity for detecting very weak Raman bands. Currently, there are three types of Raman instruments available on the market:

1. Raman grating spectrometer with single channel detector
2. FT-Raman spectrometer with near infrared excitation
3. Raman grating polychromator with multichannel detector

❖ MIR Spectrometers

There are two types of MIR spectrometers, dispersive and Fourier-transform (FT) spectrometers. Today FT spectrometers are used predominantly. The most significant advantage of FT spectrometers is that radiation from all wavelengths is measured simultaneously, whereas in dispersive spectrometers all wavelengths are measured consecutively. Therefore, a FT spectrometer is much faster and more sensitive. Dispersive MIR spectrometers are no longer available on the market, but they are still in use in laboratories. Information about the absorption of infrared radiation in the sample is obtained by measuring the intensity ratio of the radiation “before” and “after” the sample. In order to obtain this ratio with sufficient accuracy, infrared absorption spectrometers should be double channel instruments [32, 33].

❖ UV/VIS Spectrometers

The UV/VIS spectral region extends from 190 to 400 nm (UV range) and from 400 to 780 nm (visible range). In order to obtain complete spectra in the UV/VIS range, dual beam dispersive scanning instruments or dispersive multi-channel instruments are employed. UV/VIS scanning spectrometers consist of a light source, a monochromator, a chopper (rotating sector mirror or rotating sector disc) to generate a sample and a reference beam as well as to recombine them, a sample and reference compartment and a detector. Spectrometers, which permit a synchronous measurement of sample and reference beams are denoted double beam instruments. Note the position of sample and reference after the monochromator in this type of UV/VIS spectrometer. There they are substantially less exposed to the high-energy UV radiation than directly after the source. The disadvantage of this optical layout is its sensitivity to ambient stray light, which may directly reach the detector if the sample chamber is not fully covered. A multi-channel spectrometer has a light source with shutter, a sample and reference compartment, a grating polychromator and a diode array detector. All spectral elements are recorded simultaneously by the array detector, i.e the measuring time with the shutter in its open position is very short. The short illumination time permits the sample and reference positions to be positioned immediately after the light source. Multi-channel spectrometers may also be constructed as double beam instruments. For special measurements, e.g rapid kinetic investigations, when the chopper frequency is too low with respect to the rate of the process under investigation, double beam instruments with two separate detectors are used. UV/VIS absorption spectra may also be obtained with single beam instruments. In single beam spectrometers the background and sample spectra are measured one after the other. Since a chopper and reference chamber are not needed, single beam instruments are usually cheaper than double beam instrument [34, 35].

❖ Fluorescence Spectrometers

Basically, instruments for measuring fluorescence and phosphorescence spectra have similar construction and should be called luminescence spectrometers. However the group of molecules that exhibit fluorescence is by far larger than that exhibiting phosphorescence, hence the term fluorescence spectrometer is used. The main spectral features of luminescence are: spectral distribution, polarization and radiation lifetime. For analytical purposes spectral distribution and polarization are mainly used. Measuring the lifetimes requires a rather sophisticated time-resolved spectroscopic technique. It is very seldom used for analytical purposes and will not be discussed in this chapter. Two basic types of spectra can be produced by a conventional fluorescence spectrometer. In the emission spectrum, the wavelength of the exciting radiation is held constant (at an absorption wavelength of the analyte) and the spectral distribution of the emitted radiation is measured. In the excitation spectrum, the fluorescence signal is measured at a fixed wavelength of the emission selector as the wavelength of the exciting radiation is varied. An analyte can fluoresce only after it has absorbed radiation and an excitation spectrum identifies the wavelengths of light that the analyte is able to absorb. Thus, the excitation spectrum of a molecule should be the same as its UV/VIS absorption spectrum [35].

CHAPTER THREE

CHAPTER THREE

HIBISCUS SABDARIFFA L

3-1: Introduction

Roselle (*Hibiscus sabdariffa L.*) an important base fiber crop is a member of the malvaceae family. Roselle is properly a native of West Africa and is now widely distributed throughout the tropics, it has been in Asia for at least three centuries and was taken early to the new world by the slave trade. It is known in Sudan by the colloquial name “karkade”. Two botanical varieties were recognized : variety sabdariffa, branched with red or green stems and red or pale yellow calyces, variety altissima, a tall un branched plant with fibrous spiny inedible calyces, grown for fiber. The main types of karkade calyces locally known in the Sudan as EL Rahad and EL Fashir[6].

Hibiscus sabdariffa L. belongs to the family Malvaceae and is an annual shrub, well known in Sudan with the name karkade and used to make beverages. The calyces are used to make beverages and have been used in folk medicines and claimed effective as diuretics, stomachic, aphrodisiac, antiseptic, astringent, digestive, sedative, laxative, antimicrobial or as remedy for pyrexia, abscesses, heart ailments and hypertension [36].

Hibiscus sabdariffa L. is grown in various parts of the Sudan, particularly Kordofan and Darfur. It is one of the cash crops cultivated by traditional farmers in Kordofan and Darfur States under rain-fed conditions, where large quantities are produced both for local consumption and for export. The total area under cultivation was estimated at 290,000 feddans (approximately 121,800 ha) in the 2000/2001 season, compared with 22,300 to 78,444 feddans (approx. 9370–32,950 ha) in the 1970s and 47998 to 59882 feddans (approx.

20,160–25,160 ha) in the 1980s. The increased area raised production from 454 tons in the 1960s to 26,000 tons in the 1999/2000 season [37].

Roselle is an important cash crop and a source of income for small farmers in western Sudan, especially in North Kordofan State. The crop is grown mainly by traditional farming methods, exclusively under rain-fed conditions [11]. China and Thailand are also major producers and control much of the world's supply. Thailand has invested heavily in roselle production and their product is of superior quality, whereas China's product, with less stringent quality control practices is less reliable and reputable. The world's best roselle comes from the Sudan, but the quantity is low and poor. Processing hampers quality. Mexico, Egypt, Senegal, Tanzania, Mali and Jamaica are also important suppliers but production is mostly used domestically [38].

3-2: Types of *Hibiscus sabdariffa* L

There are two types of *Hibiscus sabdariffa* L:

1. Red *Hibiscus Sabdariffa* L



Figure (3.1) Red *Hibiscus Sabdariffa* L powder

2. White *Hibiscus Sabdariffa L*



Figure (3.2) White *Hibiscus Sabdariffa L* Powder

3-3: Importance and Uses of *Hibiscus Sabdariffa L*

The importance of *Hibiscus sabdariffa Lat* the local and international level as following:

- 1- The benefits of *Hibiscus sabdariffa Las* food, making juices, sweets, jellies and ice preparing.
- 2- *Hibiscus sabdariffa L* juice is of many benefit e.g. promotes digestion, hypotensive, broncho dilator, cardiac muscles control, nervous relaxation, laxative and wound detergent.
- 3- Roots powder can be used as laxative because of its tartaric acid content.
- 4- The newly grown leaves and buds can be used as salads, as medicine for dental caries and body swellings.
- 5- Stems can be used for making threads and sacks or as source of fuel like wood.

6- Its normal dye is of benefit in dying clothes and medicine manufacture.

7- *Hibiscus sabdariffa* L is of high cosmetics value.

8- In Sudan it is used as cold and hot drinks and during Al Mahadia time, its roots were used as alternative to coffee beans.

9- *Hibiscus sabdariffa* L is used for making tars which is used in scabs in animals especially camels. It is also used to make animal skins sacks.

10- *Hibiscus sabdariffa* L seeds contain 16% oil, which is used in human food. This oil is low in unsaturated fatty acids.

11- The seeds are considered excellent feed for chickens. The residue after oil extraction is valued as cattle feed when available in quantity [6].

3-4: Medicinal and Industrial Applications

Many medicinal applications of the roselle plant have been developed around the world. In China it is used to treat hypertension, pyrexia and liver damage and in ayurvedic medicine [38]. Recently the sepal extract has been used as an effective treatment against leukemia due to its high content in polyphenols, particularly protocatechuic acid [39]. Roselle seeds which until now have not had any commercial applications, are a source of a vegetable oil that is low in cholesterol and rich in other phytosterols and tocopherols, particularly β -sitosterol and γ -tocopherol. The overall characteristics of roselle seed oil allow for important industrial applications and represent added value for its cultivation [40]. Roselle also has certain therapeutic properties, the reported benefits of taking it internally in the form of herbal tea include: soothing colds, clearing a blocked nose, clearing mucous, as an astringent, promoting kidney function, aiding digestion, as a general tonic, as a diuretic and helping to reduce fever [41]. Taken as a drink made from the calyx, it is a mild diuretic and purgative, among many other effects. The drink is said to be a folk remedy for cancer. Restored roselle drink has no bacterial isolate [42].

3-5: Phytochemicals Content

Roselle is rich in anthocyanins and protocatechuic acid. The dried calyces contain the flavonoids gossypetine, hibiscetine and sabdaretine. The major pigment, formerly reported as hibiscine, has been identified as daphniphylline. Small amounts of myrtillin (delphinidin 3-monoglucoside) chrysanthenin (cyaniding 3-monoglucoside) and delphinidin are also present .

Roselle seed are a good source of lipid-soluble antioxidants , particularly γ tocopherol [40] .

Table(3.5) : Chemical analysis of dried *Hibiscus Sabdariffa L* calyces (red and white)[43]

Component	Red <i>Hibiscus sabdariffa L</i>	White <i>Hibiscus sabdariffa L</i>
Moisture	11.00	9.30
Crude protein	7.88	7.53
Crude fiber	13.20	12.00
Crude fat	0.16	0.12
Ash	10.60	9.50
Total carbohydrates	57.16	61.55
Ascorbic acid (mg/100g)	11.00	15.50
Titrate acidity (mg/100g)	9.00	11.00
Total soluble solids(%)	5.00	5.50
Calcium (mg/100g)	60.00	50.00
Iron (mg/100g)	25.00	20.00

CHAPTER FOUR

CHAPTER FOUR

EXPERIMENT AND METHODOLOGY

4-1: Equipment and Materials

4-1-1: X-ray Fluorescence Meter (X-MET— Oxford Company - model 5000)

XRF is a technique utilized in a routine analysis of various materials such as minerals powders and fluids in a non-destructive manner. Other phrases for XRF instruments include: XRF analyzer, XRF spectrometer, XRF analysis, X-ray fluorescence analyzer, x-ray fluorescence spectrometry, fluorescence x-rays spectrometer de fluorescence X, Rontgen fluorescence analyze, fluorescence de rays X, Rontgen fluorescence, Rontgen fluorescent, Rontgen fluorescence as shown in Figure (4.1).



Figure (4.1) XRF Meter

4-1-2: pH Meter (pH meter— Company JENWAY-model 3505)

A pH meter is a scientific instrument that measures the hydrogen-ion activity in water-based solutions, indicating its acidity or alkalinity expressed as pH. The pH meter measures the difference in electrical potential between a pH electrode and a reference electrode and so the pH meter is sometimes referred to as a "potentiometric pH meter". The difference in electrical potential relates to the acidity or pH of the solution as shown in figure (4.2).



Figure (4.2) pH meter

4-1-3: Electrical Conductivity Meter (Conductivity meter– Company JENWAY-model 4320)

An apparatus called a conductivity meter that consists of a conductivity cell and a meter measures conductivity. The conductivity cell consists of two electrodes (platinum plates) rigidly held at a constant distance from each other and are connected by cables to the meter. The meter consists of a Wheatstone bridge circuit. The source of electric current in the meter applies a potential to the plates and the meter measures the electrical resistance of the solution. In order to avoid change of apparent resistance with time due to chemical reactions (polarisation effect at the electrodes) alternating current is used. Some meters read resistance (ohm) while others read in units of conductivity (milli-Siemens per meter), as shown in figure (4.3).



Figure (4.3) Electrical Conductivity meter

4-2-4: UV/VIS Spectrometer (UV Spectrometer – Company JENWAY-model 6505)

The UV/VIS spectral region extends from 190 to 400 nm (UV range) and from 400 to 780 nm (visible range). In order to obtain complete spectra in the UV/VIS range, dual beam dispersive scanning instruments or dispersive multi-channel instruments are employed. UV/VIS scanning spectrometers consist of a light source, a monochromator, a chopper (rotating sector mirror or rotating sector disc) to generate a sample and a reference beam as well as to recombine them, a sample and reference compartment and a detector, shown in figure (4.4).



Figure (4.4) UV Spectrometer

4-2-5: X-ray Detector (X –ray – Company Leybold - model 55481)

Most X-ray devices emit electrons from cathode, accelerate them with a voltage (vacuum) and let them bombard an target (anode). As a result of interactions of atoms of target elements and electrons, x-ray is Produced .The energy of the x-ray shows different distribution depending on the target material .During the process, the device emits two different types of radiation shown in figure (4.5).



Figure (4.5) X-ray Detector

4-2: Methodology

Two samples of *Hibiscus sabdariffa L* (red and white) in to different shapes (solid and powder) were prepared , the samples were tested using the XRF device (X-MET 5000) respectively and the elements consistent in each sample were recorded in tables (4.1),(4.2),(4.3),(4.4) Below, the samples were solved in cold and hot water respectively, then the solution were tested using UV(6505 UV-VIS Spectrometer)to determine the spectrum and to find the maximum value of the wavelength of each sample and absorbance of them were found as shown in table (4.5). 2gram of red *Hibiscus sabdariffa L* were solved in 100 ml of distilled water, then the conductivity and acidity were measured ,the same thing was done for 2 gram of the white *Hibiscus sabdariffa L* and the results shown in table (4.6).

4-3: Results

Table (4.1) shows the reading of **XRF** red *Hibiscus sabdariffa L* powder

Elements	Concentration
Cr	0.03%
Mn	0.14%
Fe	0.18%
Ni	0.01%
Cu	0.00%
Zn	0.01%
Pb	0.01%

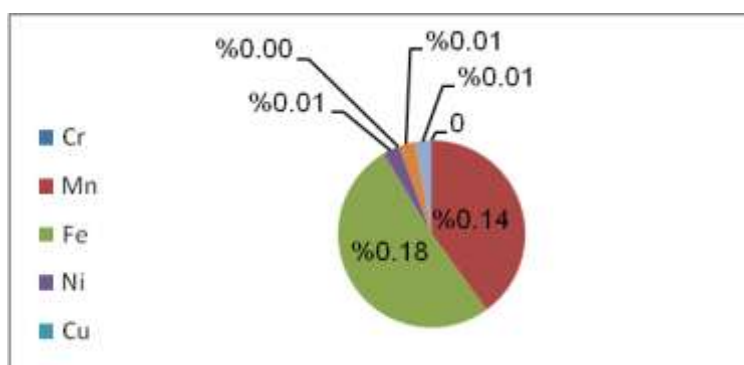


Figure (4.6) shows the reading of **XRF** red *Hibiscus sabdariffa L* powder

Table (4.2) shows the reading of **XRF** red *Hibiscus sabdariffa L*

Elements	Concentration
Cr	0.02%
Mn	0.06%
Fe	0.20%
Ni	0.01%
Cu	0.00%
Zn	0.04%
Pb	0.02%

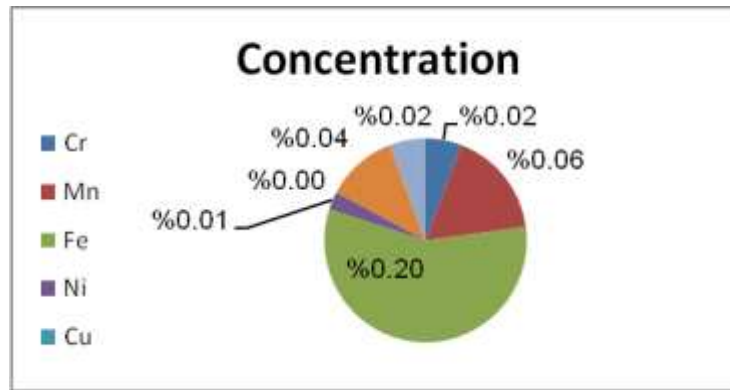


Figure (4.7) shows the reading of **XRF** red *Hibiscus sabdariffa L*

Table (4.3) shows the reading of **XRF** white *Hibiscus sabdariffa L* powder

Elements	Concentration
Cr	0.02%
Mn	0.06%
Fe	0.20%
Ni	0.01%
Cu	0.00%
Zn	0.04%
Pb	0.02%

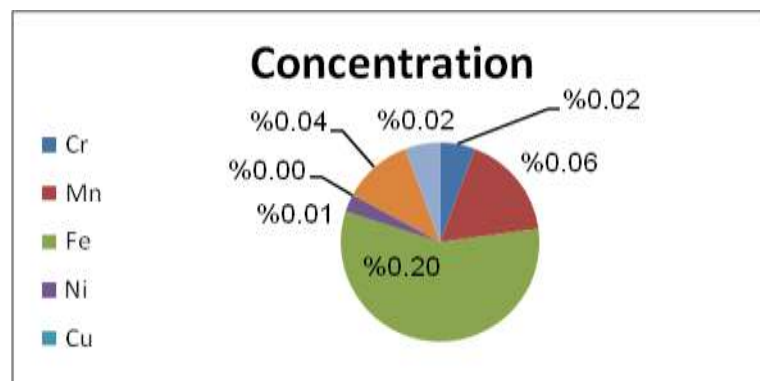


Figure (4.8) shows the reading of **XRF** white *Hibiscus sabdariffa L* powder

Table (4.4) shows the reading of **XRF** white *Hibiscus sabdariffa L*

Elements	Concentration
Cr	0.01%
Mn	0.34%
Fe	0.30%
Ni	<0.00%
Cu	0.00%
Zn	0.00%
Pb	0.01%

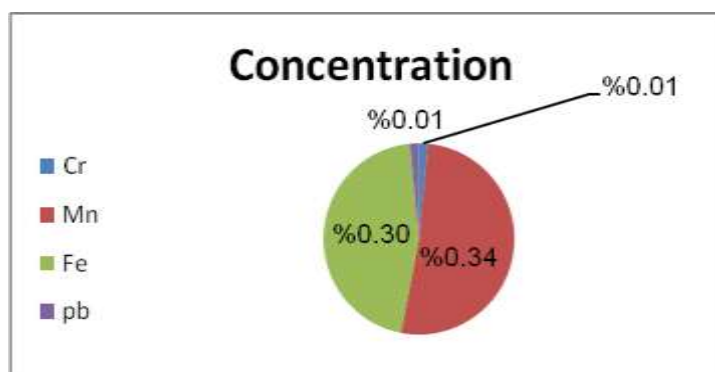


Figure (4.9) show the reading of **XRF** white *Hibiscus sabdariffa L*

Table (4.5) shows the reading of UV device wavelength and Absorbance

Material	Wavelength/nm	Absorbance
hot red <i>Hibiscus sabdariffa L</i>	510.0	1.105
Cold red <i>Hibiscus sabdariffa L</i>	515.0	0.775
hot white <i>Hibiscus sabdariffa L</i>	215.0	1.278
cold white <i>Hibiscus sabdariffa L</i>	210.0	1.343

Table (4.6) shows the reading of Conductivity device

Type	Quantity/g	Acidity	Conductivity/ms	Temperature/c
Red	2	3.93	3.21	26.7
White	2	3.93	3.08	26.5

4-5: Discussion

In tables (4.1), (4.3) (Red and White *Hibiscus sabdariffa L* samples powder) were studied, both were found to be consist of different elements with various concentrations which increases in some and decreases in others we as follows:

The samples contains Chromium (Cr) of 0.03% in powder which is decreases to 0.02% in solid sample, the magnesium (mg) decreases from 0.14% in powder to 0.06 % in white powder , in addition other elements found to be increases in their percentage such as (Fe) which increases from 0.18% in the powder sample to 0.20% in the white powder sample, also the Nicole (Ni) has the same percentage of 0.01% in both samples, while the Zn and Pb increases from 0.01% to 0.025 respectively, lastly, the element of copper was not detected in any of the samples investigated.

Table (4.5) shows the relation between absorbance and maximum wavelength for the solution of red *Hibiscus sabdariffa L* (solved in warm and cold water), the wavelength varies from 510 nm to 515 nm, while the absorption varies from 0.775 to 1.105 respectively. Also the temperature found to linearly proportional to absorption and inversely proportional to the wavelength.

Table (4.6) shows the conductivity and the acidity of both samples of *Hibiscus sabdariffa L* solved with distilled water, denoted that both samples have the same acidity of 3.93 and their conductivity varies from 3.08 at 26.5 c to 3.21 at 26.7 c.

CHAPTER FIVE

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5-1: Conclusion

Two types of *Hibiscus sabdariffa L* red and white were crushed into small sized and used as a solution in hot and cold water, UV-VIS Spectrometer used to study the absorbance and its wave length. Also X-ray detector used to study the crystal structure, it found the spectrum of *Hibiscus sabdariffa L* in range (200-800 nm) and it found to be amorphous material and the interstitial distance were not according to the Bragg's law. Also XRF Meter used to study the element exist on those samples of *Hibiscus sabdariffa L*. Also EC Meter used to study the conductivity of two solution red and white to be 3.21 s/m at 26.7°C and 3.08 s/m at 26.5°C respectively. Also pH Meter used to study acidity for the two types of solutions red and white *Hibiscus sabdariffa L* they have the same acidity 3.93.

5-2: Recommendation

1. In this thesis the sample used in XRF met is different in method of preparation than that used for UV-VIS spectrometer, pH meter, X-ray detector and conductivity meter, so in the future work the sample for different techniques must be prepared by the same way.
2. In this research was used to make solution for all techniques, but it convenient to take other solutions in consideration.

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