بسم الله الرحمن الرحيم

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صدق الله العظيم

سورة طه الآية (114)

Dedication

To

The soul of my father,

My dear mother

Acknowledgements

With gratitude I express my appreciation to all people whose interest and encouragement have helped me in bringing this thesis to completion and in particular to, my supervisor Prof. Elmugdad Ahmed Ali for his guidance, excellent advice and constant encouragement throughout the course of this work, and my co–supervisor Dr. Mohamed Elmukhtar Abdel Aziz for his guidance, active participation, continuous support, imaginative supervision and enthusiasm during the execution of this research.

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Abstract

Two Sudanese mica samples were collected from Shereik, River Nile State and Red Sea State. A systematic series of analysis were performed to characterize the micas and to study effects of impurities on physical, mechanical, thermal, and electrical properties.

The classical methods namely gravimetric and volumetric analysis were used for the determination of the major and impurities constituents, SiO_2 was 40.03% - 39.02%, Al_2O_3 was 35.54% - 36.01%, K_2O was 18.16% - 18.05% , Na_2O was 0.37% - 0.54%, while by Inductively, Coupled Plasma Optical Emission Spectroscopy (ICPOES) SiO_2 was 40.49% - 39.90%, Al_2O_3 was 37.53% - 35.78%, K_2O was 19.51% - 20.09%, Na_2O was 0.50% - 0.38%. Using Flame Photometry Emission (FE) K_2O was 16.76% - 18.00%, Na_2O was 0.40% - 0.34% and Atomic Absorption Spectroscopy (AAS) K_2O was 31.06% - 29.24%, Na_2O was 0.55% - 0.61%.

The supplementary techniques, Energy Dispersive X–ray Fluorescence (EDXRF) SiO_2 was 36.01% - 35.49%, $Al_2O_3$was 33.79% - 35.97%, K_2O was 27.93% - 26.40%.

The X – ray diffraction (XRD) revealed that mica samples were composed of mica minerals in addition to quartz and kaolin impurities.

Thermogravimetry (TG) curves evident that a slight loss of weight (0.15%) was observed at temperature range 100^0C–105^0C.

Infrared technique showed very strong absorption band at 3622 cm^{-1} is an indication of free hydroxyl water , strong band at 762 cm^{-1} most of the frequencies assigned to the Si – O vibration.

Mechanical studies, showed that mica samples had hardness between gypsum and calcite (2 – 3 Mohs), tensile strength 1734 Kg/cm^2 and 1718 Kg/cm^2, for samples Modulus of elasticity were between $1380x10^{-3}$ – $2015x10^{-3}$ Kg/cm^2, and between $1392x10^{-3}$ – $2070x10^{-3}$ Kg/cm^2, respectively.

Electrical study showed that mica samples had dielectric constant 2.8 – 4.7 kV/mm.

المستخلص

عينتان من المايكا السودانية تم جمعهما من منطقة الشريق ولاية نهر النيل , ولايةالبحرالاحمر. أجرى عليهما تحليل كيميائي تقليدى وتحاليل آلية للتعرف علي التركيب المعدني للمايكا ثم دراسة أثر الشوائب علي الخواص الفيزيائيةوالميكانيكية والحرارية والكهربية.

استخدمت طرق التحليل الكيميائي التقليدي (الوزني والحجمي) لتحديد المكونات الرئيسة والشوائب وكانت النسب كالاتي اكسيد السيليكون 40,03% - 39,02%, اكسيد الالمنيوم 35,54% - 36,01%, اكسيد البوتاسيوم 18,16% - 18,05% واكسيد الصوديوم 0,37% - 0,54% وكذلك استخدمت مطيافية الانبعاث الحثي للبلازما وكان اكسيد السيليكون 40,49% - 39,90% , اكسيد الالمنيوم 37,53% - 35,78%, اكسيد البوتاسيوم 19,51% - 20,09%, اكسيد الصوديوم0,50% - 0,38% ,مطيافيةالانبعاث الذري ومضوائية اللهب اكسيد البوتاسيوم 16,76% - 18,00%, اكسيد الصوديوم 0,40% - 0,34%, مطيافية الامتصاص الذري اكسيد البوتاسيوم 31,06% - 29,24%, اكسيد الصوديوم 0,55% - 0,61%.

فلورة الاشعة السينية اكسيد السيلكيون 36,01% - 35,49%, اكسيد الالمنيوم 33,79 – 35,97%, اكسيد البوتاسيوم 27,93% - 26,40%.

أظهرت نتائج حيود الاشعة السينية ان المكون الرئيسفى عينتي المايكا هو معدن المايكا إضافة لذلك وجود الكوارتز والكاولين.

بجانب التقنيات أعلاه استخدمت تقنيات التحليل الحراري الوزنى حيث أوضحت ان معدن المايكا يفقد الماء الممتص فوق 100 درجة مئوية

والاشعة تحت الحمراء لتشخيص عينتالمايكا اوضحت حزمة الامتصاص القوية جدا عند 3622سم$^{-1}$ يدل علي وجود هيدروكسيل الماء حر, وحزمة الامتصاص القوية عند 762 سم$^{-1}$ وهي تشير الي تردد ذبذبات رابطة السيليكون والاكسجين.

الدراسة الميكانيكية بواسطة مقياس موهز للصلابة اظهرت ان عينتي المايكا لهما صلابة بين صلابةالجبصوالكالسيت (2 – 3 موهز). وقوة الشدة مقاسة بواسطة جهاز مقياس الشد بالنسبة لعينة المايكا (1) 1734 كغ/سم^2 وعينة المايكا (2) 1718 كغ/سم^2 . معامل المرونة للعينة (1)- $1380x10^{-3}$ $2015x10^{-3}$ وللعينة (2) بين $1392x10^{-3}$ - $2070x10^{-3}$كغ/سم^2.

الدراسة الكهربية بواسطة مقياس الممانعة أظهرت ان عينتي المايكا لهما ثابت عزل 2.8 – 4.7 كفولت/مم^2.

List of Abbreviations

1. A^0: Angstrom = 10^{-8} meter.
2. SMC: Sudanese Mining Corporation.
3. NW: Northwestern.
4. NE: Northeastern.
5. N – S: North – South.
6. TG: Thermogravimetry
7. ICPOES: Inductively Coupled Plasma Optical Emission Spectroscopy
8. EDXRF: Energy Dispersive X – Ray Fluorescence.
9. EDS: Energy Dispersive Spectroscopy.
10. EDX: Energy Dispersive X – ray.
11. SEM: Scanning Electron Microscopy.
12. XRD: X – Ray Diffraction.
13. UTS: Ultimate Tensile Strength.
14. DNA: Deoxyribonucleic acid.
15. AFM: Atomic Force Microscopy
16. FE: Flame emission.
17. IRCC: Industrial Research and Consultancy Center.

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Chapter One

1. Introduction and Literature review

1.1 Introduction

Mica is mineral present in igneous rocks and occurs in massive form in some localities. Muscovite Mica is a valuable by–product in the production of china clay, because it is largely unaltered by the hydrothermal processes which formed Kaolinite; the comparatively large plate–like grains of Mica along with quartz particles are, easily,sediment from the, finely, divided clay mineral. The separation of the quartz is effected by sieving and screening. Mica is valuable raw material for the electrical industry, but is rarely used as a constituent of glazes. Mica rich in alkalis (Grimshaw, 1971). Micas may be regarded as a natural mineral following the montmorillonites as their structure is also based on those of pyrophyllite or talc. The layers are not electrically balanced and require the presence of extra cations in inter–lamellar positions (Ames, and Sand,1958).

There is one important difference, however, which results in a fundamental change in the physical character. The layers of Mica have a greater deficiency of charge and the balancing cations are held more strongly; in fact, there is ionic bonding, linking successive sheets through these additional cations. Unlike montmorillonite type minerals the layers of micas cannot expand by water, nor can the cations between them be exchanged or removed in any way other than by decomposition of the mineral (Noll, 1936).

Micas are primary igneous minerals; they crystalline in large platelike masses which are easily cleaved along their basal section. They were the first layer–lattice silicates to be examined by X–rays and their structure, as defined by Pauling (Pauling, 1930) has formed the basis for other minerals of similar layer construction.

The essential difference between montmorillonite and the Mica minerals is that whereas the former has its charge–deficiency in the central octahedral layer, the deficiency in micas is entirely in the outer tetrahedral networks. From this it is to be expected that the cations in micas would be held more firmly than those in the montmorillonite minerals. The average net residual negative charge on a unit of montmorillonite is 0.33 whilst in mica it is about 1.00 (Roy, and Osborn, 1954).

The structural formula of muscovite mica can be expressed as

$K_{1.00}$

$\uparrow$

$Al_2 (Al\ Si_3)\ O_{10}\ (OH)_2$

Potassium cations in muscovite are, symmetrically, placed over each hexagonal ring in the external sheets of the layers. They are linked to six oxygen atoms of one sheet and six in the adjacent layer, making twelve in all. The interlayer distance is about $9.8A^0$.

Micas are also of complex chemical composition being, mainly, hydrated alumino–silicates but always containing notable proportions of alkalies, alkaline earths, iron and sometimes fluorine.

The structural formula of a particular mica, may be calculated from the chemical analysis as for montmorillonites. Within the mica group there is a further subdivision into a dioctahedral and trioctahedral series based on pyrophyllite and talc respectively.Muscovite mica is a true dioctahedral mica and has the chemical composition $K_2O.3Al_2O_3.6SiO_2.2H_2O$.

One silicon atom in every four in the outermost tetrahedral positions of each layer is replaced by an aluminum atom, thereby causing a unit deficiency of charge.This is not a general rule, however, for isomorphous replacements in the octahedral layer may also cause at least part of the negative charge.

There are three important groups of micas (Grimshaw Rex, 1971).

(a) A dioctahedral series when trivalent cations occupy the octahedral positions and the structure is based on pyrophyllite.

(b) A trioctaheral series, with trivalent and monovalent cations (lithium Li^+) in octahedral positions.

(c) A normal trioctahedral series with divalent cations arranged as in talc.

1.2 Objective

The objectives of this research are

- To determine the composition of the samples of the Mica ore.

- To determine the mineral impurities of the ore samples.

- To asses the effect of mineral impurities on the physical properties of the samples.

- To compare and contrast the methods used to analyse Mica ore.

1.3 Literature Review

1.3.1. Silicate Minerals

The silicate minerals can be considered inorganic polymers based on two monomer structure. These are the tetrahedron and octahedron. Many of the silicates can be pictured as the configurations made by joining of such tetrahedron and octahedron within themselves and to each other in three dimensions. These involve the sharing of corners, edges, and faces in numerous configurations. The possible geometric permutations are further modified by chemical substitutions within the structure, which usually depend on how well a metal ion will fit among closed–packed oxygen ions. This is largely a matter of relative ionic radii. Given an O^{2-} ionic radius of 1.40Å. The preferred (most stable) coordination of cations common in industrial silicate minerals has been calculated and expressedin terms of ionic radius ratio (Carr,1994).

Mineralogical purity of industrial minerals is a factor distinct from chemical purity for some end uses. Commercially exploitable ore deposits are rarely mono - mineral. A substantial component of the value – added cost of many industrial mineral products is the expense incurred by the producer in reducing mineral impurities by screening, air classification, washing, flotation, centrifuging, magnetic separation, heavy media separation, electrostatic separation (Deer, Howie and Zussman,1978).

1.3.1.1 Quartz

Quartz: SiO_2 , is the fundamental structure unit of silicate minerals is silica tetrahedron. Quartz is just a densely packed arrangementof thesetetrahedron. Extended in three dimensions, this structure provided the characteristic hardness and inertness of quartz. The different forms of crystalline silica –most commonly quartz. Cristobalite, and Tridymite–differ mainly in the relative orientation of adjacent tetrahedral and the shape of voids created within a given plane (Grim,1953). Natural silica products are crystalline and are classified for most of their uses under the term sand and gravel. Finely ground natural silica with high mineralogical and chemical purity is nevertheless produced in substantial quantities. It is generally characterized as off – white to white and abrasive, with low surface area, low binder demand, and low cost. Amorphous forms of natural silica exist as diatomaceous earth, volcanic glass, and non commercialopaline mineral (Grim,1962). Synthetic silica are, generally, characterized as amorphous and white, with high purity, high surface area, high liquid absorption, and ultrafine particle size. These include silica gel and precipitated silica, also known as hydrated silica, and fumed silica, also known as pyrogenic or anhydrous silica. Hydrated silica are made in aqueous media, which results in a relatively high density of surface silanol (- Si–OH) groups. They therefore absorb water readily and are typically sold with 5 to 6% free moisture. The pyrogenic process used to produce fumed silica leavesmost surface silanols condensed to siloxane bridge (- Si– O–Si -) so that silanol surface density is only about 25% of that of hydrated silica. There is consequently less tendency to absorb water, and the products typically contain less than 2% free moisture (Grim Shaw,1980).

1.3.1.2 Feldspars

Feldspars: (Anorthite $CaAl_2Si_2O_3$, Albite $NaALSi_3O_8$, Orthoclase $KAlSi_3O_8$): are the most widespread mineral group. Comprising approximately 60% of the earth's crust. Feldspar minerals are sodium, potassium, and calcium aluminosilicates. Their ores are commonly associated with quartz and mica, and may also contain spodumene (a lithium aluminosilicate), kaolin, garnet, or iron minerals depending upon the type of deposit. Most commercial feldspars are produced by flotation and magnetic separation followed by milling. Air classification is used for the finest grades. Products typically contain quartz– low levels in high grade feldspars, high levels in feldspar/quartz mixtures fold for glassmaking. Feldspar ores and products generally contain mixtures sold for glassmaking. Feldspar ores and products generally contain mixtures of feldspar minerals rather than one mineralogical distinct feldspar type (Grim Shaw, 1980).

1.3.1.3 Kaolinite

Kaolinite: $Al_2Si_2O_5(OH)_4$, commercial grades of kaolin are composed primarily of the mineral kaolinite, a sheet silicate, and may contain greater or lesser quantities of related sheet silicates (mica, illite, chlorite, smectite) and quartz. An individual kaolinite particle has the shape of a hexagonal plate. In nature these plate occur in stacks or books that exhibit varying degrees of stacking regularity. Kaolin is hydrophilic (readily water dispersible), for non aqueous applications matrixcompatibility can be improved by surface treatment (Hurbut, and Klain, 1977).

1.3.1.4 Talc

Talc: $Mg_3Si_4O_{10}(OH)_2$, if a sheet of silica rings is attached to the magnesia side of Chrysotile, the bending tendencies on either side of octahedral layer negate each other. The mineral structure remains planar, and the laminar trioctahedral an alogue of pyrophyllite results. As with pyrophyllite, individual talc lamina are held together by weak Van der Waals forces. Sliding and delamination are relatively easy, giving talc its characteristic soft, slippery feels.Commercial talc is composed primarily of the mineral talc, a sheet silicate, but may contain related sheet silicates such as chlorite and serpentine, plus prismatic tremolite, anthophyllite , and carbonates such as magnesite dolomite and calcite (Newman,1987).

Talc particles are characteristically platy in morphology and are oleophilic/hydrophobic, they are wetted by oil instead of water. Talc's reinforcing and pigmenting properties, together with good color, make it desirable as a functional filler in both aqueous and non aqueous applications. For non aqueous uses its naturally good matrix compatibility can be further enhanced by surface treatment (Van Olphan,1977).

1.3.1.5 Vermiculite

Vermiculite: $Mg_3(Mg,Fe)_3(Al,Si)_4O_{10}(OH)_4.8H_2O$, is a platy sheet silicate that is similar in appearance to the mica from which it was altered and similar in ion exchange properties to the trioctahedral. Vermiculite is the basic talc structure, differs from talc primarily in its substitution of Al^{3+} for tetrahedral Si^{4+} and the presence of two oriented layers of two between individual laminae (Bureau of Mines, 1991).

1.9.1.6 Phyllosilicates

Phyllosilicates: Silica tetrahedral also can join into rings. Phyllosilicates are characterized in part by an indefinitely extended sheet of rings, with three of the tetrahedral oxygen shared and the fourth (apical) oxygen in each case pointing in the same direction. Another characteristic of most phyllosilicate minerals is the presence of a hydroxyl group central to the apical oxygen. This configuration is achieved through bonding of the silica sheet to a continuous sheet of octahedral, with each octahedron tilted onto one of its triangular sides (Bureau of Mines, 1992).

1.3.1.7Wollastonite

Wollastonite: Among the industrial minerals high tetrahedral density and consequent hardness are also found in chain silicates. The chains formed by these silica tetrahedral are connected by calcium in octahedral coordination. Because of this chain structure wollastonite can occur as acicular crystals, in some cases of macroscopic dimensions. This acicular particle shape is important in certain uses as a functional mineral filler (Bureau of Mines, 1993).

1.3.1.8 Chlorite

Chlorite : is an accessory mineralin some ore. It is laminar and composed of alternating talc and brucite sheets. The chlorite structure includes upper silica layer of an adjoining platelet. Unlike talc, chlorite accommodates appreciable substitution of both tetrahedral and octahedral cations (Carr,1994).

1.3.1.9 Pyrophyllite

Pyrophyllite : $Al_2Si_4O_{10}(OH)_2$, in pure or near pure form is rare in nature and in commerce. It can be viewed as an aluminum analogue of talc, and a pure, platy specimen would share many of talc's physical characteristics. In addition to the platy morphology, pyrophyllite occurs in nature as massive aggregates of small crystals and as large needle–like crystals. In Japan and Korea, where pyrophyllite is abundant, have been used in applications traditionally reserved elsewhere for kaolin and talc (Harben, and Bates,1990). The requirements for competing in a world economy, however, have more recently moved Pacific Rim countries towards wider use of imported kaolin and talc. In other producing countries a relatively low value is assigned pyrophyllite, so that many natural mineral mixtures (some even devoid of this mineral) are sold under its name, generally at low prices. In many cases the lack of purity has use to advantage. The major current uses, actually, exploit the unique and desirable features of the mineral blends that constitute commercial grades (Lewis,1988) . The mineral, most commonly, associated with commercial pyrophyllite are the related sheet silicates (mica, kaolinite, chlorite, smectite, illite) plus diasporeandalusite and quartz (Milewski and Katz,1987).

1.3.1.10. Smectites

Smectites, (Montmorillonite $(Al,Mg)_2Si_4O_{10}(OH)_2$, Hectorite $(Mg,Li)_3Si_4O_{10}(OH,F)_2$, Saponite $Mg_3(Si,Al)_4O_{10}(OH)_2$, are water swellable clays that have a platy structure. Smectite is the mineralogical term for a group of clays. Most smectite are, more commonly, known under the geological term bentonite. By convention, bentonite is understood to be an ore or product with a substantial smectite content (Weiss,1985). The range of possible chemical variations in the basic smectitetrilayer lattice start with montmorillonite, the high aluminum end member.Montmorillonite is composed of a central alumina octahedral layer sandwiched between tetrahedral silica layers. This is identical to the dioctahedralpyrophyllite structure expect for small substitutions of Mg^{2+} for Al^{3+} in octahedral positions and Al^{3+} for Si^{4+} in tetrahedral positions. The resulting charge imbalance is compensated by exchangeable alkali and alkaline earth cations, which contribute to the ability of the clay to swell (Dannenberg,1987) When the exchangeable cations are, predominately sodium, the individual platelets separate to produce a colloidal structure in water. At the other end of the smectite series are the high magnesium members, hectorite and saponite. These clays possess a talc structure, with a trioctahedral magnesia layer sandwiched between the silica layers. Swellability results from minor substitution of aluminum for silicon in saponite or lithium for magnesium in hectorite.As with montmorillonite, the type of exchangeable cation determines the degree of swelling. Hectorite also has partial substitution of lattice hydroxyl by fluorine. Each macroscopic smectite particle is composed of thousands of submicroscopic platelets stacked in sandwich fashion with exchangeable cations and oriented water between each. The platelet faces carry a negative charge from lattice substitutions, while edges have a slight positive charge from broken bonds and cation adsorption (Goodman,1995). When smectite and water mixed, water penetrates the area between the platelets forcing them farther apart. With calcium as the major exchange ion the platelets will swell in this fashion but have limited ability to, completely, delaminate. Whensodium is the predominant exchange ion, the platelets separate further apart and the exchange ions begin to diffuse away from the platelet faces. Further penetration of water between the platelets then proceeds in an osmotic manner until they are, completely, separated. The presence of dissolved substances in the water will prolong hydration time by inhibiting this osmotic swelling (Griffiths,1990). Once the smectite platelets are separated, weak positive platelet edges are attacked to the negatively charged platelet faces. The resulting three dimensional structure, often referred to as a house of cards imparts thixotropy, pseudo plasticity, and yield value. Most smectites are processed by drying, crushing, and milling to a 200 mesh powder, with mineralogical purity determined by ore selection. Granular grades are produced for absorbent uses. A relatively small quantity of white or light colored smectitehydro classification to produce products of sufficient mineralogical purity for pharmaceutical, cosmetic, and the more demanding industrial uses. The impurities, most commonly, associated with commercial smectite are silica, feldspar, zeolites, and carbonate minerals (Joslyn,1986).

1.4. Chemical Analysis of clay (silicate minerals)

1.4.1. Infra–Red Spectroscopy

The infrared (IR) spectroscopic technique has been utilized in mineralogy and particular in clay mineralogy in the wavelength range 2.25–25 µm. (Nyquist and Kagel,1971). It was established that infrared spectroscopy could be used in mineral analysis. It can be used to identify minerals of different (but constant) chemical composition, minerals of constant composition but different crystal structures (Gadsden,1975). IR can also allow distinction between minerals that exhibit arrangement of composition (Smith,1999). It can also help to distinguish between minerals that vary in both crystal structure and chemical composition.

The IR Spectra of clay minerals have been investigated by many workers (Lyon,1960), (Newnham,1961), (Serratosa,1962), (Wolff,1963), (Fripiat and Tosaint. 1963), and (Leddoux and White,1964).

Gadsden (Gadsden, 1975) and collaborators showed that certain groupings which are common to many minerals give characteristic adsorption bands. Buswell and Dudenbostel have shown that the unbonded OH group which absorbs at about 3700 cm^{-1} and 1700 cm^{-1} is believed to be due to adsorbed water, while the absorption at about 900 cm^{-1} is believed to be due to Al–Olinkage. The octahedral alumina sheet is probably responsible of the absorption at 1050 cm^{-1}.

Workers (Serratosa,1962), (Wolff,1993), (Fripiat and Tosaint, 1963), (Ledoux and White,1964) on IR spectrum of Mica concluded that each sharp hydroxyl band corresponds to a distinct type of hydroxyl. The sharp bands at 3697 cm^{-1} arises from OH group perpendicular to Mica sheets, while the bands at 3670 cm^{-1}, 3652 cm^{-1}, and 3620 cm^{-1} arisefrom OH group at 3620 cm^{-1} assigned to the fourth hydroxyl group. Farmer and Russell (Farmer and Russell,1964) established that the OH groups in the dioctahedral silicates absorb in the range 3750–3700 cm^{-1}. However, Farmer and Russell (Farmer and

Russell,1967) reported that the substitution by Al for Si causes broadening in OH absorption band at 3620 cm^{-1}. Farmer and Russell (Farmerand Russell,1964) established that the OH stretching and bending vibrations are largely independent of the vibration of both silicon and oxygen in the rest of the lattice. They showed that the Si–O band vibrates at 1027 cm^{-1} and 1022 cm^{-1} with oxygen being apical. Bands at 1110 cm^{-1} , 1027 cm^{-1} and 1002 cm^{-1} were caused also by Si–O stretching. Other workers (Wolff,1963), (Fripiat andTosaint,1963), (Fripiat and Toussaint,1960) determine the spectral bands caused by the silica layer in Mica Si – O from SiO_4in average wave number 1012 cm^{-1} - 1114 cm^{-} sharp, and medium bands at average wave number 794 cm^{-1} - 693 cm^{-1} and 430 cm^{-1} - 370 cm^{-1}. Percival and Duncan (Percival and Duncan,1974) concluded that condensed SiO_4 in spinel absorb at 1050 cm^{-} and 1150 cm^{-} , the bands at 1062 cm^{-} - 1124 cm^{-} ascribed for Al – Si in spinel. Heller (Heller,1978) confirmed the findings of Percival and Duncan concerning the Si–O and Al–O vibration. Serratosa and Bradly (Serratosa and Bradly,1958) observed that among Mica, trioctahedral compositions exhibits an OH band axis normal to the cleavage flake with ν 3700 cm^{-} , but dioctahedral compositions exhibit OH band axes near the plane of cleavage and of less absorption frequency.

IR Spectroscopy is becoming of paramount importance when dealing with amorphous material for which X–ray diffraction are tenuous. IR can serve as a very useful supplementary technique to the X–ray diffraction in mineral identification.

1.4.2 Inductively Coupled Plasma Optical Emission Spectroscopy

Inductively Coupled Plasma is one method of optical emission spectroscopy. When plasma energy is given to an analysis sample from outside, the component elements (atoms) are excited. When the excited atoms return to low energy position, emission rays (spectrum rays) are released and the emission rays that correspond to the photons wavelength are measured. The element type is determined based on the position of the photon rays, and the content of each element is determined based on the rays intensity.

To generate plasma, first, argon gas is supplied to torch coil, and high frequency electric current is applied to the work coil at the tip of the torch tube. Using the electromagnetic field created in the torch tube by the high frequency current, argon gas is ionized and plasma is generated. This plasma has high electron density and temperature (10000K) and this energy is used in excitation–emission of the sample . Solution samples are introduced into the plasma in an atomized state through the narrow tube in the centre of the torch tube.

Equipment for ICP optical emission spectroscopy consists of a light source unit, a spectrophotometer, a detector and a data processing unit. There are several types of equipment based on differences in the spectrophotometer and the detector.

1.4.3Atomic Absorption Spectroscopy

Atomic Absorption Spectroscopy (AAS) is a sensitive technique for quantitative analysis. Generally it can be used for the determination of major or trace elements in silicate minerals(Skoog,1992).

Ultra–violet and visible atomic spectra are obtained by converting the components of sample solution into gaseous atoms or elementary ions by a suitable heat treatment. Absorption or emission from the resulting gaseous mixtures serves for quantitative determination of the elements present in the sample (Skoog,1992).

Basically the atomic absorption instrument consists of four basic components. A light source (almost exclusively a hollow cathode lamp) an absorption cell in which the atoms of the sample are produced (Flame, graphite furnace, etc.) a monochromator and detector.

The method used was that described by Vogel (Vogel,1959). To 0.5 g of each sample 10 ml HF and 5 ml Conc. HNO_3 were added, and the resulting solution was evaporated to dryness on steam bath. 1 ml of 10% HCl was added and the resulting solution was transferred quantitatively to a 200 ml volumetric flask and a few ml of lanthanum chloride was added to eliminate the chemical interferences. Lanthanum can act not only as a releasing agent but as ionization suppressor. The resulting solution was completed to the mark with deionized water. A blank solution was prepared in the same way.

To calibrate the instrument a standard solution (1000 ppm) and a series of dilution were prepared for magnesium, iron, potassium, manganese, and sodium. The absorption of the standard and sample for each element were measured. A calibration curve were constructed for the standard from which the concentration of each element was determined.

1.5.Mechanical Properties

1.5.1 Measurement of Hardness

One of the most important tests for identifying minerals specimens is Mohs Hardness Test. This test compares the resistance of a mineral to being scratched by ten reference minerals known as the Mohs Hardness Scale.

The test is useful because most specimens of a given minerals are very close to the same hardness. This makes hardness a reliable diagnostic property for most minerals.

Friedrich Mohs, a German mineralogist, developed the Scale in 1812. He selected ten minerals of distinctly different hardness that ranged from a very soft mineral (Talc) to a very hard mineral (diamond), with the exception of diamond, the minerals are all relatively common and easy or inexpensive to obtain.Making Hardness Comparisons: " Hardness" is the resistance of a materials to being scratched. The Test is conducted by placing a sharp point of one specimen on an unmarked surface of another specimen and attempting to produce a scratch. Here are four situations that you might observe when comparing the hardness of two specimens:

1. If specimen A can scratch specimen B, then specimen A is harder than specimen B or vice versa.

2. If specimen A does not scratch specimen B, then specimen B is harder than specimen A.

3. If the two specimens are equal in hardness then they will be relatively ineffective at scratching one another. Small scratches might be produced or it might be difficult to determine if a scratch was produced.

4. If specimen A can be scratched by specimen B but it can't scratched by specimen C, then the hardness of specimen A is between the hardness of specimen B and specimen C.

1.5.1.1 Mohs Hardness Testing Procedure:

1. Begin by locating a smooth, unscratched surface for testing.2. With one hand, hold the specimen of unknown hardness firmly that the surface to be tested is exposed and accessible.

3. Hold one of the standard hardness specimens in the other hand and place a point of the specimen against the selected flat surface of the unknown specimen.

4. Firmly press the point of the standard specimen against the unknown specimen, and firmly drag the point of the standard specimen across the surface of the unknown specimen.

5. Examine the surface of the unknown specimen with a finger, brush away any mineral fragments or powder that was produced. Be careful not to confuse mineral powder or residue with scratchA scratch will be a distinct groove cut in the mineral surface, not a mark on the surface that wipes away.

6. Conduct the Test a second time to confirm the result (Geoscience News and Information 2017).

1.5.2 Measure of Tensile Strength and Modulus of Elasticity

A tensile test, also known as tension test, is probably the most fundamental type of mechanical test you can perform on material. Tensile tests are simple, relatively inexpensive, and fully standardized. By pulling on something, you will very quickly determine how the material will react to forces being applied in tension, as the material is being pulled, you will find its strength along with how much it will elongate. (Courtney,2000).

You can learn a lot about a substance from tensile testing. As you continue to pull on the material until it breaks, you will obtain a good, complete tensile profile. A curve will result showing how it reacted to the forces being applied. The point of failure is of much interest and is typically called it "Ultimate Tensile Strength" or UTS on the chart.(Davis, 2004).

Hooke's law

For most tensile testing of materials, it is notice that the relationship between the applied force, or load, and the elongation the specimen exhibits is linear. In this linear region, the line obeys the relationship defined as "Hooke's law" where the ratio of stress to strain is a constant, or

$$\sigma / \varepsilon = E$$

E is the slope of the line in this region where stress (σ) is proportional to strain (ε) and is called the "Modulus of Elasticity" or "Young's Modulus".

The Modulus of Elasticity is a measure of the stiffness of the material, but it only applied in the linear region of the curve, if a specimen is loaded within this linear region, the material will return to its exact same condition if the load is removed. At the point that the curve is no longer linear and deviates from the straight–line relationship, Hooke's law no longer applied and some permanent deformation occurs in the specimen. This point is called the "elastic , or proportional limit". From this point on the tensile test, the material reacts plastically to any further increase in load or stress. It will not return to its original, unstressed condition if the load were removed. A value called " Yield Strength" of a material (e.g. metals and plastics), the departure from the linear elastic region cannot be easily identified. Therefore, an offset method to determine the Yield Strength of the material tested is allowed. These methods are discussed in ASTM E8 (metals) and D638 (plastic). An offset is specified as α% of strain(for metals, usually 0.2% E8 and sometimes for plastics α value 2% is used). The stress (R) that is determined from the intersection point "r" when the line of the linear elastic region (with slope equal to Modulus of Elasticity) is drawn from the offset "m" becomes the Yield Strength by the offset methods.(Dieter,1986).

Alternative Moduli: The tensile curves of some materials do not have a very well – defined linear region. In these cases, ATSM Standard EIII provides for alternative methods for determining the Modulus of a material, as well as Young's Modulus. (Boyer, 2002).

These Alternative Moduli are the Secant Modulus and Tangent Modulus.

Strain: You will also be able to find the amount of stretch or elongation the specimen undergoes during tensile testing this can be expressed as an absolute measurement in the change in length or as α relative measurement called "Strain". Strain itself can be expressed in two different ways, as "engineering strain" and "true strain". Engineering strain is probably the easiest and the most common expression of strain used. It is the ratio of the change in length to the original length (Budinski and Budinski, 2005).

$e = (L - L_0)/L_0 = \Delta L/L$

Whereas, the true strain is similar but based on the instantaneous length of the specimen as the test progresses.

$\varepsilon = \ln (L_i/L_0)$

Where L_i is the instantaneous length and L_0 the initial length.

Ultimate Tensile Strength: One of the properties you can determine about a material is its Ultimate Tensile Strength (UTS). This is the maximum load the specimen sustains during the test. The UTS may or may not equate to the strength at break. This all depends on what type of material you are testing brittle, ductile or a substance that even exhibits both properties.(Ashby,2002).

1.6 Electrical Properties

1.6.1 Measurement of Dielectric Constant

A dielectric is a material having low electrical conductivity in comparison to that of a metal. It is characterized by its dielectric constant. Dielectric constant is measured as the ratio of the capacitance C of an electrical condenser filled with the dielectric to the capacitance C_o of evacuated condenser i.e.,

$$\varepsilon = C/C_o$$

1.7. Thermal Properties

1.7.1 Thermogravimetry

In thermogravimetry (TG)generally reports refers records the weight of a substance when heated through a selected rate of temperature. The weight loss is plotted versus temperature. The record obtained called thermogravimetric curve which provides information concerning the thermal stability and composition of the sample.

1.8. Mica

Mica is a natural valuable mineral product applied to a group of a complex aluminosilicate minerals having a sheet or a plate like structure with different chemical compositions and physical properties. All micas, natural or synthetic crystallize in the form of flat, six sided monoclinic crystals (Milford, 1962).

Micas are silicates sheets historically significant for their ability to be split into large, thin sheets that are uniquely useful for their electrical, thermal, and mechanical properties. They have high electrical and thermal insulating properties, they are resistant to chemical attack,they can be split into transparent or optically flat films, and they can be cut or stamped to shape. Most mica used today, however, is in ground forms, although the mineral's platy nature and inertness are still primary attributes (Rajgarhia,1951).

1.8.1 Names of mica

Other names of mica (Browning, Millsaps and Bennett,1965).

i. Cat – gold.

ii. Cat – sliver.

iii. Glimmer.

iv. Glist.

v. Katen – silber.

vi. Katzen – silber.

vii. Katzengold.

viii. Or des chats.

ix. Rhomboidal mica.

1.8.2 Classification of Mica

1.8.2.1 Chemical classification

(Browningand Bennett,1965).

Table:1.1 Types of Mica, chemical formula's and classes

Type of Mica	Chemical formula	Class
Muscovite	$KAl_2(AlSi_3O)O_{10}(OH)_2$	Dioctahedral
Paragonite	$NaAl_2(AlSiO_3)O_{10}(OH)_2$	Dioctahedral
Biotite	$KMg_3(AlSiO_3)O_{10}(OH)_2$	Trioctahedral
Phlogopite	$K(MgFe)_3(AlSi_3)O_{10}(OH)_2$	Trioctahedral
Margarite	$CaAl_2(Al_2Si_2)O_{10}(OH)_2$	Trioctahedral
Paragonite	$NaAl_2(AlSiO_3)O_{10}(OH)_2$	Trioctahedral

1.8.2.2 Classification of Mica in terms of their traditional production

Mica products are often classified in terms of their traditional production in sheet form as opposed to the more recent filler forms (Feld, McVay, Gilmoreand Clemmons,1966).

1.8.2.2.1 Mica Sheet

Mica sheet consists of blocks of mica laminae and are classified according to thickness, purity, and maximum usable area that can be cut or stamped. These criteria dictate the subcategories, in order of decreasing thickness of block, thins film, and splitting. Film mica is split from block and thins. Splitting are very thin but have, relatively, small useable areas, and they are used, mostly, to fabricate built – up mica. The ASTM designates 13 quality groups for sheets mica based on color and visible imperfections and 12 additional groups based on maximum usable rectangle. Although its overall volume is minor compared to ground mica, sheet mica is still, widely, used in the electrical and electronics industry for its combination of thermal and electrical insulating properties and high mechanical strength (ASTM Electrical Insulating Materials, 1966).

1.8.2.2.2 Built–up Mica

Also known as micanite, built – up mica is a fabricated sheet of desired thickness made by over lapping, irregularly shaped splitting and then binding them together with an organic or inorganic binder, heat, and pressure. Some products are reinforced with a paper, fiberglass, or textile backing. Built – up mica is produced as an alternative to natural sheet mica for electrical insulation applications (Standards for Manufacture Mica, 1956).

1.8.2.2.3 Mica Paper

Mica paper is a fabricated alternative to natural sheet and built–up mica products. Scrap mica is form through a combination of thermal, chemical , and mechanical treatments. Mica pulp is then processed on a Paper making machine into a continuous, homogenous sheet of uniform thickness(Clark, 1924).

1.8.2.2.4 Scrap Mica

The term scrap is hold over from the days when sheet was the major mica product. It is basically, mica that is insufficient in size or quality to qualify as sheet. Scrap is produced at mines and at sheet mica factories. Scrap mica can be byproduct of sheet mica mining or of the recovery of other minerals or the sole product of a mine. Factory scrap is the trimmings from sheet mica. Scrap is the source for ground mica products, which today account for more than 95% of all mica sold worldwide (Doelter, 1888).

1.8.2.2.5 Flake Mica

Flake mica is essentially non factory generated scrap mica. It is recovered by flotation from mica ores, from which quartz and feldspar are also generally separated and recovered, or as a floated byproduct of feldspar, kaolin,or lithium–bearing ores (Grigoriev,1934).

1.8.2.2.6 Wet–ground Mica

Flake mica concentrates from flotation are ground wet in mills designed to delaminate as well as to grind. Such mills provide products having a higher aspect ratio, sheen, and slip compared to dry–ground mica. Trimmings from good–quality sheet mica are also used as feed for wet – grinding plants (Eitel,1946).

1.8.2.2.7 Dry–ground Mica

Flake mica flotation concentrates are at least partially dried and then ground in mills appropriate for the particle size desired (>100 mesh) are processed with hammer mills and screens or air separators. Fine–ground products, -100 mesh to -325 mesh in particle size, are processed in fluid energy mills, usually with supper heated air (Tokiti Noda, 1955).

1.8.2.2.8 Micronized mica

This is dry–ground mica milled to -20 or -10 μm in fluid energy mills using supper heated steam. Some is calcinedforusein cosmetics (Hatch, Humphrey, Eitel, and Comeforo,1957).

1.8.2.3 Classification of Natural Mica according to their Commercial and Industrial purposes

For commercial and industrial purpose natural mica are mainly divided into three categories (Shell and Ivey,1967).

1.8.2.3.1 Processed Mica

Is fundamentally a natural sheet mica of an irregular size and polygonal shapes that is relatively flat and free from physical defects and structural imperfections having a minimum usable area of 4.8 sq. cm. (0.75 sq. inch), suitable to be cut, punched or stamped into specific size and shape chiefly for use by the electronic and electrical industries (Shell and Warwick,1962).

1.8.2.3.2 Fabricated Mica

Is basically a natural sheet mica cut, stamped or punched to specified size, shape and thickness for industrial and uses, such as, discs, washers, cut–films or sheets, joints, backing plates, spacers, mica formers for irons, toasters, rice cookers, etc., (Shell, 1960).

1.8.2.3.3 Manufactured Mica

Means mica–based products manufactured from natural mica, such as, micanite or built up mica , mica paper, mica heating elements, mica capacitors, mica flakes and power, mica bricks, etc., (Johnson and Shell,1963).

1.8.3 Occurrence and Geology of Mica

Mica is, widely, distributed as small flakes in many masochists and kaolin contain sufficient muscovite for it to be recovered for use as scrap mica. Muscovite sheet mica is recovered only from pegmatite deposits. Zones pegmatite are the principle source. Concentrations or shoots mica are distributed various sizes and thicknesses. These crystals, usually roughly hexagonal and tabular, range from less than an inch to several feet in maximum dimension. It is not possible to identify reliably a potentially productive within a productive dike except by close samplingLarge crystals of phlogpite usually are found in pyroxenite dikes in association with calcite and apatite (Kirkpatrich, 1983).

Throughout the world the occurrence of higher–quality and larger–grade size of sheet mica is irregular and spotty. Even in the famous Bihar mica field of India, only two or three veins in a hundred are said to be rich enough to permit development on an extensive scale. In all areas, low–and high–quality mica are found together (Anthony, Bideaux, Bladh and Nichols,1990).

1.8.3.1 Occurrence and Geology of SudaneseMica

Muscovite – bearing pegmatites are erratically distributed along the western side of the river Nile within the triangle Berber – Abu Hamad – Elkab and extend for more than 65 Km westwards into Bayuda desert. Those deposits which have been worked and are of potential economic importance, are in Jebel Rahaba area west of Shereik. Attention was first drawn to this area in 1940S when a number of small firms and individuals mined mica and continued to do so, intermittently, for 30 years. Mining was primitive and only easily–worked surface deposits, were exploited. Most of the mica derived from these workings, was stained and decayed and had to be sold as scrap. Only one producer tried to grade and classify mica for sales reached some 700 tons (Mgeed,1998).

Mining had, virtually, ceased when a joint survey was carried out by Sudan Geological Survey and United Nations Development Programme, which led to restarting mining in 1974. In 1977, the concession was transferred to the former

SMC which mined the Rahaba I and II pegmatites by underground methods up to the end of 1984, when all operations were suspended due to a combination of factors such as prevailing low prices, increase in failure rates of their aging equipment plus mining difficulties (Mgeed,1998).

Following an upturn in international demand for mica of all grades and better prices, coupled with increasing buyer interest, SMC (Sudan Mining Companies) carried out a new survey of the area in 1990 with a view of resuming operations within a reasonable period. Consequently to a general survey of the pervious workings and the unexploited pegmatite outcrops, geological surface mapping was carried out for four of the most promising bodies, Rahaba II, Rahaba III, Absol, and Seih Al Abid. The general parameters of those pagmetites with mica potential are (Mgeed,(1998)

* Dips between 45^0 and 55^0

* Thickness between 1m and 7m

* Strike lengths between 50m to 150m

* Hanging–wall competent

Underground mining in the past has shown that mica quality improves with depth. The present Rahaba I underground workings, show that mica to be low–grade and well–disseminated throughout the intrusion. Although the richest part can be seen underground in Rahaba II, SMC was unwillingto resume underground operations in the beginning because of the extra capital required, higher operating costs and the need for, comparatively, large number of skilled miners. From work done in 1970, the underground mica potential for Rahaba I, II and Abaza I, is good in that these deposits should meet the standards regarding total content of Mica, sheet mica from crude Mica(Mgeed,1998). Out of the four bodies mapped by SMC, Absol is considered the most amenable to open pit working (rather than underground mining) until a down dip depth of 25m from the surface, their calculated waste to ore ratios increase after a depth of 10m from an average of 0.67:1 to 1.56:1 and then to 2.23:1 which could make open pit mining at the mining at the lower depths uneconomical(Mgeed,1998).

There is evidence that sheet mica is of good grade and meets international specifications. However, development of sheet mica production is laborintensive in that material has to be hand mined and graded. It seems that a more appropriate strategy would be the development of an integrated feldspar and flake scrap mica open pit deposit, the products beings utilized in ceramic production and exported for paint and filler usage respectively. It is possible that rare earth minerals in these pegmatite can be recovered as byproducts(Mgeed,1998).

1.8.3.2 Regional Geology

Most of Bayuda desert is made up of crystalline rocks of the basement complex. This complex can be subdivided into four rocks series as follows: (Mgeed,1998).

The Abu Harik series comprises highly migmatitic, partly granitisedgneisses and amphibolites which have reached the amphibolitesfaces of regional metamorphism. it is overlain by the Kurmut series, a sequence of metasediments, quartzite's, schist's, marbles, calcsiticate rocks, gneisses and amphibolites, all metamorphosed into the amphibolites faces. Near the Nile Valley and north of Barber, shearing and catalysis caused retrograde metamorphism of parts of the Kurmut series which led to the formation of greenchistfacies rock (Mgeed,1998).The acid quartzo – feldspathic gneisses, biotite garnet gneisses and amphibolites of Rahaba series conformably overlie the metasediments of the Kurmut series. They make up most the western and central parts of Bayuda desert. Large portions of the series are migmatised or even granitised. Pegmatoid veins in the quartzo – feldspathic gneisses contain minable Mica (Mgeed,1998).

The Kyanite – garent– staurolite schist's of the Absol series, which contain layers of amphibolitesand small lenses of meta– calcareous and psmmitic rocks, form a narrow synclinal belt in the northeastern part ofBayuda desert. They overlie the gneisses of Rahaba series(Mgeed,1998).All rocks are intensely folded. The strike is mainly NE but turns to the NW in the northern part of the area. Shearing and shear folding with axes striking N–S affected the rocks near the Nile valley, north of Berber and south of Abu Hamad(Mgeed,1998).Post– orogenic igneous complexes (mainly alkali granite and alkali syenite) intruded all rock series of the basement. They were accompanied by numerous swarms of rhyolitic, felsitic and trachytic dykes, some of them forming ring dykes around the plutonic masses. Alkali rhyolitic and alkali trachytic lava and pyroclastics form part of some of the ring complexes. Radiometric age determinations on rocks of the ring complexes carried out by the Federal Institute for Geosciences and Natural Resources of Germany yielded the following results (BGR 1975 – 1978) (Mgeed,1998).

Mica pegmatite are a typically constituent of the Rahaba series. They occur occasionally in Absol series. Rahaba I and Rahaba II Tables 1.2, 1.3 and 1.4 were first operated by the Sudan Geology Survey as experimental mines, where a drilling program was carried out. According to the former SMC, a total of 147.378 ton of crude mica is available in Rahaba mines (Mgeed,1998).

Table: 1.2 Mica in Rahaba I mines

Proved ore	46000 ton
Possible ore	31000 ton
Probable ore	31000 ton
Total	108000 ton

Table: 1.3 Mica in Rahaba IIA mines

Proved ore	9890 ton
Possible ore	6594 ton
Probable ore	6594 ton
Total	23078 ton

Table: 1.4 Mica in Rahaba IIB mines

Proved ore	4650 ton
Possible ore	4650 ton
Probable ore	25000 ton
Total	343000 ton
Total Rahaba II	57378 ton

Total reserve of crude mica (in form of book) in Rahaba mines 165378 ton. The annual production was stated to be approximately 36 ton. In addition, manual mining was carried out by the natives on a, fairly, small scale at a series of small occurrences. Mica mined by them was sold to SMC (Mgeed,1998).

1.8.4 Mining and Preparation of Mica

The unique properties of mica and the erratic character of its natural occurrences are such that methods employed in mining and processing other types of minerals are applicable only to a limited extent in the development of mica deposits. As a result, mining methods at sheet mica mines tend to be rather primitive. In fact, it has been stated that micamining is not a business, but a disease." Some mines, operating chiefly for scrap, are more mechanized. A flotation process developed by the U.S. Bureau of Mines effectively separates mica from other minerals (Brown and Norrish,1952).

Although the nature, extent, and quality of many ores and minerals can be forecast from geologic evidence, sheet mica may be distributed unevenly in pegmatite that it is virtually impossible to estimate either the quantity or quality present. Most metallic and nonmetallic minerals can be beneficiated and either reduced or chemically treated to yield highly purified marketable products. The value of sheet mica, however, depends on size, structure, and purity of the sheets that can be obtained by splitting and trimming the natural mica books. None of these important characteristics of sheet mica be improved by either metallurgical or chemical processes (Green–Kelly,1973).

The crude books, comprising only 2–7 % of the volume of rock broken, are picked out of the broken rock by hand and cobbled to remove adhering rock and dirt. After sorting out those that are valuable only as scrap the remaining books are roughly splits into sheets or plates, usually less than 1/16 in. thick. The products are (Bear, 1964).

i. untrimmed book or mixed sizes.

ii. punch and washer stock.

iii. scrap.

The sheets are then trimmed to remove edge cracks and imperfections. In India, all mica is "full–trimmed" that is all imperfections are eliminated from the edges, and the edges are beveled so as to facilitate subsequent splitting into thinner sheets or films. In the United States,where labor rates are high and skilled workers fewer, full–trim corresponding to the Indian preparation is uncommon. "Thumb–trimming" the usual preparation of punch mica, is confined to breaking off ragged edges with fingers.

"Knife–trimmed" block is trimmed closer. "half–trim" mica is trimmed on two adjacent edges, and in "three–quarter trim" the imperfections are cut from three sides. Close trimming may waste good mica. Part–trimmed small mica not only has the extra areas permitting the fabricator to get a larger pattern, but also affords the press operator more to hold onto in punching small shapes (Bailey,1966).

Block mica is sheet mica 0.007 in. or greater in thickness, with a minimum Film mica is a thin sheet or sheets split from the better qualities of block mica. Thickness specifications for film mica range from 0.00125 to 0.004 in., with tolerance of 0.00025 in. the films usually split from good–stained or processing known as scaling, cleaning, or chillas.usable area of one square in. Splitting are thin pieces of irregularly shaped mica with a maximum thickness of 0.0012 in. the most desirable thickness are between 0.0007 and 0.001 in. splitting are usually, although not necessarily, made from lower qualities and smaller sizes of mica, both muscovite and phlogopite (Bailey,1975).

1.8.5 Colors Stains, and Associated Secondary minerals

Muscovite is often called white or ruby mica to distinguish it from phlogopite, which is often called amber mica. In crystals or in thick plates, the colors of muscovite range through varying shapes of red and green, depending primarily on the iron content but sometimes on the amount and charge of other ions. Color, in fact, is a basis for industrial classification of muscovite into white (including yellow), ruby (amber, rum, red, brown) and green. When split into thin films, both muscovite and phlogopite are nearly colorless and transparent, although when viewed against a very white background the green variety can, usually,be picked out. Staining and visible mineral (clay or other) or vegetable inclusions, generally, reduce the value of commercial mica (Danna,1993).

Inclusions and intergrowths of other minerals in mica books are sometimes broadly referred to as stains. Some minerals, such as magnetite and hematite, are parallel with the cleavage, but others, such as quartz and zircon, penetrate cleavage planes and produce pinholes (Sordon,1984).

Stains are classified as primary or secondary. Primary stains are those formed during crystallization of the mica and include (Marshall,1964).

i. Mottling

ii. A kind of stain referred to as vegetable although it is inorganic

iii. Mineral inclusions and intergrowths.

Secondary stains are those formed between the cleavage planes, chiefly, through the action of circulating groundwater and include air creep or air stain, clay, iron, and manganese stains and true vegetable stains caused by organic matter. Air stain is gas occluded in pockets between the sheets, there is doubt as to its origin. Primary stains occur as likely to increase with depth of the mine as to decrease, but secondary stains occur only at or near the ground surface and are absent from those parts of the deposit beneath the oxidized zone (Fagan and Garrett,2006).

Minerals stain is the most serious of the primary impurities. It comprises intergrowths and inclusions of recognizable crystals. Magnetite and hematite are the most common inclusion minerals have been identified within books of muscovite. Whereas magnetite inclusions may seriously impair the splitting qualities of muscovite, hematite inclusions appear to have little effect as far as commercial preparation is concerned. Both magnetite and hematite, however seriously increase the conductance of mica in which they occur, as thus lower its value (Brindleyand De Kimpe,1961).

1.8.6 Grades Qualities, and Classification of sheet mica

From the foregoing, it is evident that to the uninitiated, the accurate sorting of the natural mica into homogenous groups is something of a nightmare. A rule of thumb, however, is simply that if mica looks good and is low in iron, it will perform satisfactory.Mica is classified according to the following properties to (Nelson and Roy,1958)

1. Extent of preparation–crudehand–cobbled, thumb–trimmed, half–trimmed (or 2 sides), full–trimmed.

2. Thickness – block, film, splitting.

3. Size. Table 1.5.

4. Electrical quality. Table 1.6.

5. Visual quality (for muscovite only). Factors considered include color, flatness, stain, cracks, air inclusions, waviness, herringbone, sandblast, hardness, and others.

6. Thermal stability (for phlogopite only). High heat phlogopite must withstand 750^{0}C for 30 min. with less than 25% increase in thickness after cooling, and with no evident transformation such as silvery or friable stains. Regular quality pholgopite fails this test. Clear ruby muscovite has the highest quality of any natural mica, and is also the most reliable as regards performance. Green and dark–color mica are more variable, particularly as regards dissipation factor, but may be equally valuable provided they pass the necessary laboratory tests for electrical properties. Iron (both ferric and ferrous ions substituting of Mg is the principal constituent of mica giving rise to inferior electrical properties. A useful but destructive test for rapid indication of iron concentration is to heat the sample in a muffle furnace, in an oxidizing atmosphere, at 800–1000^{0}C for 5–10 min. a silvery color shows low iron, tan to brown, increasing quantities of iron. Sorting sheet mica for size (grade) and quality is a costly hand operation requiring much skill and experience. The world standards were established in India and are rarely carried to the same degree of precision elsewhere(Stresse and Hofmann,1941)

In United Stated, the size gradation is often expressed in inches the smallest size, punch is untrimmed mica large enough to yield a 1.5 in. circle, the next size circle is untrimmed mica that will yield up to a 2 in. circle. The poorest quality of punch (cross–grained or heavy–stained), only slightly better than scrap, is sometimes called washer mica (Norrish and Brown,1952).

Table:1.5 Muscovite Block and Film Mica, Grade by Size

Area of minimum Rectangle, in.^{2}	ASTM and India	U.S. domestically produce	Malagasy Republic	Brazil	Min. dimensions on one side in.
100	OOEE Special	8x12	OOOO	OOE Special	4
80	OEE Special	8x10	OOO	EE Special	4
60	EE Special	6x10	OO	E Special	4
48	E Special	6x8	O	Special	4
36	ASTM-AI Special India-Special	6x6	AI	AI	31/2
24	1	4x6	1	1	3
15	2	3x5	2	2	2
10	3	3x3	3	3	2
6	4	2x3	4	4	11/2
3	5	11/2x2	5	5	1
21/2	51/2	Circle	6	51/2	7/8
1	6	Punch	6	6	¾

Legend: O = over ; E = extra

Table 1.5 gives the grades of muscovite block and film mica according to sizes. These range from 1 in.^{2} punch mica to 8x12 in.^{2} or larger books.

Muscovite block mica is classified by ASTM specifications, based on the Bengal India system, into twelve quality groups according to color and appearance as regards stains, waves, cracks, inclusions, etc. (Grimshaw, Sadler and Roberts,1962).

A resonant–circuit test set, known as the Q–meter, was developed by Bell Telephone Laboratories for testing mica and other insulation. Results of the test are Q units, and for the best mica should be about 2500.

Table: 1.6 Q Values for Electrical Quality Group (1)

Film		2500	1000
E – 1	Block	2500	....
E – 1	Film	2500	500
E – 2	Block	1500	……..
E – 2	Film	1500	200
E – 3	Block	200	……..
E – 3	Film	200	100

Since Q is in reality the reciprocal of the dissipation or power factor (Q = 1/power factor), apparatus that will actually determine the dissipation factor may be used to determine Q.

table 1.6 give the Q values for the electrical quality groups. Experience has shown that the Q–value range of ruby and white mica is 80–90% of the E-1 grade, while the Q values of light–green, dark–green, greenish–brown, and rum–colored block mica is 45–90% E–1 (Grim, Bray and Bradley,1937).

1.8.7 Properties of Mica

Mica is invaluable in the electrical industry because of its unique combination of physical, chemical, and thermal properties, low power loss factor, dielectric strength. Dielectric strength is the ability to withstand high voltage without puncturing. Common specifications are 1000 volts and even 1500 volts per millimeter of thickness without puncturing, and mica provides a high factor of safety at these figures. Dielectric constant may be defined as the capacity for momentary storing electrostatic energy. The properties like low power loss factor and dielectric constant make mica ideal for use in condensers, the basic function of which is to store electrostatic energy in the dielectric field momentarily perhaps one millionth part of a sec. and then to redeliver it with the minimum possible loss (Clifford,1957).

No other natural substance has been found to possess properties equal to those of mica. Muscovite mica can be split into flexible and transparent films as thin as 0.00025 in. which give added advantage in making built – up mica, tapes and films that can be used in any shape and size.Of all known varieties of mica only muscovite and phlogopite are of commercial importance and valued in the electrical industry. Muscovite finds the largest use while phlogopite has a limited application. Phlogopite does not possess the split ability and flexibility of muscovite. On the other hand phlogopite is superior to muscovite in heat resistance. Muscovite can withstand temperatures up to 700^0C, and phlogopite up to about 1000^0C. phlogopite is therefore, preferred where a high temperature is required. Other mica have no use except for lepidolite which is a source of lithium (Crow. and Max,1975).

The quality of mica for commercial purposes depends, largely, on the amount of the staining, air inclusions, the degree of flatness, and the color. The staining is caused by mineral inclusions which occur intergrown with muscovite or between cleavage planes. The most common minerals which occurs as inclusions are biotite, quartz, magnetite, hematite, garnet, plagioclase, apatite, clay minerals and the alteration products of biotite and iron oxides (Bear,1964).

Muscovite which does not split up into even cleavage has a lower market value. It can be sold only as scrap mica. Such mica is called buckled mica. Buckling effect in mica results. Other features possessed by muscovite, which greatly lower its value are A–structure and wedge–structure. Such structures originated at the time of crystallization. A–structure refers to cleavage imperfections called reeves or ridges that intersect at an angle of about 60^0. This feature results from the winning phenomenon, crystals with A–structure which are thicker at one end than the other are said to possess a wedge–structure.

The presence of A or wedge structure greatly reduces the yield of sheet mica. Normally most of the muscovite in an individual body is of one habits, it either does or does not possess these structures (Young and Hewat,1988).

1.8.7.1Chemical Properties of Mica

Mica is a complex hydrous silicate of aluminum, lithium and also traces of several other elements. It is stable and completely inert to the action of water, acids (except hydro–fluoric acid and concentrated sulfuric acid) alkalis conventional solvents, oil and virtually unaffected by atmospheric action (Potts,1987).

Table:1.7 Chemical Analysis of Mica Sillimanites Schist

SiO_2	61.50%
Al_2O_3	21.11%
Fe_2O_3	1.02%
FeO	3.34%
CaO	5.77%
MgO	1.54%
Na_2O	3.64%

K_2O	0.01%
TiO_2	0.20%
MnO	0.02%
P_2O_5	0.07%
Loss on ignition	0.74%

Table:1.8 Chemical Analysis of MicaeousAmphibolites

SiO_2	47.32%
Al_2O_3	3.66%
Fe_2O_3	5.75%
FeO	5.66%
CaO	8.65%
MgO	23.18%
Na_2O	0.14%
K_2O	1.02%
TiO_2	0.05%
MnO	0.08%
Loss on ignition	4.46%

1.8.7.1.1 Chemical Analysis of Muscovite Mica

$K_2Al_2(Si_3AlO_{10})(OH,F)_2$(www.tedpella.com)

Table: 1.9 Chemical Analysis of muscovite Mica

Silica (SiO_2)	45.57%
Alumina (Al_2O_3)	33.1%
Potassium oxide (k_2O)	9.87%
Ferric oxide (Fe_2O_3)	2.48%

Sodium oxide (Na_2O)	0.62%
Titanium oxide (TiO_2)	Traces
Calcium oxide (CaO)	0.21%
Magnesia (MgO)	0.38%
Moisture at 100^0C	0.25%
Phosphorus (P)	0.03%
Sulfur (S)	0.01%
Graphitic Carbon	0.44%
Loss on ignition (H_2O)	2.74%

1.8.7.2 Physical Properties of Mica

Physically Mica is transparent, optically flat, easily split into thin films along its cleavage, colorless in thin sheets, resilient and incompressible.(Potts,1987).

Table: 1.10 Physical Properties of Mica

Characteristic	Unit	Phlogopite
Color	-	Amber/Yellow
Density	g/cm^3 Ib/in^3	2.6 – 3.2 0.095-0.116
Specific Heat	-	0.21
Hardness	Moh Scale Shore Test	2.3 – 3.0 70 – 100
Optic Axial Angle	-	5 – 25
Tensile Strength	Kgf/cm^2 Ibf/in^2	about 1000 about15000
Shear Strength	Kgf/cm^2	1000 – 13000

	Ibf/in^2	14000 – 19000
Compression strength	Kgf/cm^2 Ibf/in^2	……. ……
Modulus of Elasticity	Kgf/cm^2 x10^{-3} Ibf/in^2 x10^{-6}	1400 – 2100 20 – 30
Coefficient of expansion per^{0}C perpendicular to cleavage plan	-	30x10^{-6} – 60x10^{-6}
Calcining Temperature	C F	900 – 1000 1650 – 1830
Maximum Operating temperature	C F	800 – 900 1470 – 1650
Thermal conductivity	-	-
Perpendicular to cleavage planes	Gm.cal/sec/cm^2/C/cm BTU/hr/ft^2/of/ft	about 0.0001 about 0.24
Parallel to cleavage planes	Gm.cal/sec/cm^2/C/cm BTU/hr/ft^2/of/ft	about 0.012 about 3.0
Water of constitution %	-	3.0
Moisture absorption	-	Very low
Apparent electric strength	0.001 to 0.003 KV/mm Thick volts per 0.001	……
at 15^{0}C (60^{0}F)	0.01 to 0.05 KV/mm Thick volts per 0.001	30 – 60 750 – 1500
Permittivity at 15^{0}C (60^{0}F)	-	5 – 6
Power factor (loss tangent)	at 15^{0}C (60^{0}F)	0.001 – 0.005
Volume resistivity	25^{0}C (77^{0}F)ohm cm	1x10^{12} – 1x10^{12}
Acid reaction	-	Affected by sulphuric acid

1.8.7.2.1 Physical Properties of muscovite mica

Table: 1.11 Physical properties of muscovite mica

(www.tedpella.com)

Characteristic	Unit	Muscovite
Color	-	Ruby/Green
Density	g/cm^3 Ib/in^3	2.6 – 3.2 0.095-0.116
Specific Heat	-	0.21
Hardness	Moh Scale Shore Test	2.8 – 3.2 80 – 105
Optic Axial Angle	-	55 – 75
Tensile Strength	Kg/cm^2 Ibf/in^2	about 1750 about 25000
Shear Strength	Kg/cm^2 Ibf/in^2	2200 – 2700 31000 – 38000
Compression strength	Kg/cm^2 Ibf/in^2	1900 – 2850 27000 –32000
Modulus of Elasticity	Kg/cm^2 $x10^{-3}$ Ibf/in^2 $x10^{-6}$	1400 – 2100 20 – 30
Coefficient of expansion perpendicular to cleavage plan	-	$9x10^{-6}$ - $36\ x10^{-6}$
Calcining Temperature	C F	700 – 800 1290 – 1470
Maximum Operating temperature	C F	500 – 600 930 – 1110
Thermal conductivity	-	-

Perpendicular to cleavage planes	cal/sec/cm^2/C/cm hr/ft^2/of/ft	about 0.0013 about 0.31
Parallel to cleavage planes	cal/sec/cm^2/C/cm BTU/hr/ft^2/of/ft	- -
Water of constitution %	-	4.5
Moisture absorption	-	Very low
Apparent electric strength	0.001 to 0.003 KV/mm Thick volts per 0.001	120 – 200 3000 – 5000
at 15^{0}C (60^{0}F)	0.01 to 0.05 KV/mm Thick volts per 0.001	40 – 80 1000 – 2000
Permittivity at 15^{0}C (60^{0}F)	-	6 – 7
Power factor (loss tangent)	at 15^{0}C (60^{0}F)	0.0001–0.0004
Volume resistivity	25^{0}C (77^{0}F)ohm cm	40x10^{13} – 2x10^{17}
Acid reaction	-	Affected by hydrofluoric acid

1.8.7.3 Mechanical Properties of Mica

Mica is relatibilitysoft and can be hand–cut, machined or die–punched. It is flexible , elastic and tough, having high tensile strength. It can withstand great mechanical pressure perpendicular to plane but the lamination have weak cleavage and can be easily split into very thin leaves (Fagan and Garrett, 2006).

1.8.7.4 Thermal Properties of Mica

Mica is fireproof, infusible, incombustible and non–flammable and can resist temperatures of 600^0C to 900^0C (1112^0F to 1652^0F) depending on the type of mica. It has low heat conductivity, excellent thermal stability and may be exposed to high temperatures without noticeable effect (Corradiand Leonelli,1996).

1.8.7.5 Electrical Properties of Mica

Mica has the unique combination of great dielectric strength, uniform dielectric constant and capacitance stability, low power loss (high Q factor), high electrical resistivity and low temperature efficient and capacitance. It is noted for its resistance to arc and corona discharge with no permanent injury (Gualtieri and Bellotto, 1998).

The theoretical dielectric strength of a material is an intrinsic property of the bulk material and is dependent on the configuration of the material or the electrodes with which the field is applied. At breakdown, the electric field frees bound electrons. If the applied electric field is sufficiently high, free electrons may become accelerated to velocities that can liberate additional electrons during collisions with neutral atoms or molecules in a process called avalanche breakdown. Breakdown occur quite abruptly (typically in nanosecond), resulting in the formation of an electrically conductive path and a disruptive discharge through the material. For solid materials, a breakdown event severely degrades, or even destroys its insulating capability (Gualtier and Bellotto,1998).

1.8.7.5.1 Factors affecting Dielectric Strength

i. It increases with the increase in thickness of the specimen. (directly proportional).

ii. It decreases with increase in operating temperature. (inversely proportional).

iii. It decreases with increase in frequency. (inversely proportional).

iv. It decrease with the increase in humidity. (inversely proportional).

1.8.8 Uses of Natural Mica Sheet

1.8.8.1 Electrical Apparatus

Armature coils, commentators, heat sinks, invertors, rectifiers, rotating field coils, transformers (Massoit and Omine,1974).

1.8.8.2 Electric Heating Appliances

Cigarette lighters, coffee percolators, curling irons, flat–irons, hair dryers, hot plates, immersion heathers, rice cookers, and boilers, permanent wave machines, soldering irons, space heaters, steam iron presses, toasters, vibrators,waterheaters, waffle irons, portable dryers (Russell and Farmer,1969).

1.8.8.3 Electrical Control

Electrical and thermal regulators, grid resistances boxes, industrial motors starters, motor control panel, pyrometers, relays, rheostat rods and terminals, starting boxes of trolley cars(Simopoulos, et al., 1975).

1.8.8.4 Electrical Lighting Equipment

Arc lamps, dimmer switches, flashers, large incandescent lamp shades, liners for lamp shades, neon–lights (Joseph and Antonl, 1991).

1.8.8.5 Industrial Electric Heating Appliances

Branding irons, glue pots, laundry irons, lead pots, localized heating units Miscellaneous heating elements, thermostats (Gam,1965).

1.8.8.6 Mechanical uses

Compass cards, diaphragms for hearing aids, quided missiles, Mommsen lungs, non breakable goggles, oxygen breathing apparatus, petromax chimney, quarter wave plates for optical instruments, shields for liquid level gauges of high pressure steam boiler,stove and furnace inspection windows, synthetic optical crystals(Anthony, et al, 1995).

1.8.8.7 Miscellaneous Electrical uses

Covers for fuse plugs, insulators, spark plugs for heavy duty high compression engines, washers (Elwakeel and Riley,1961).

1.8.9 Industrial uses of Mica

The United States is the leading supplier of mica, with about 46% of the world's production. The U.S. produces only ground muscovite. The only significant production of phlogopite is in Canada and Finland, which together account for about 15% of total worldwide mica output. India produces is used in wallboard joint cements. Others major end users are coatings, plastics, and well drilling fluids(Mark,1971).

1.8.9.1 Joint Cements

Fine dry–ground (fluid energy–milled) muscovite is used in drywall joint compounds, where it contributes to consistency and work ability,smooth surface finish, and resistance to shrinkage and cracking(Asselin, et al, 1990).

1.8.9.2 Coatings

Fine–ground, - 325 mesh and micronized mica grades are used in paint as a paint extender and for dry film reinforcement. The inert, platy mica improves suspension stability, controls film checking, chalking, shrinkage, and blistering, improves resistance to weathering, chemicals, and water penetration, and improves adhesion to most surfaces. Coarser grinds are used in textured paints, and wet–ground mica is used in high quality exterior house paints. High aspect ratio grades are preferred for porous surface sealers to seal pores, control penetration, and reduce sagging and film cracking. Automotive paints use high aspect ratio mica to achieve a metallic effect either as is, or after conversion to pearlescent pigments by surface coating with metal oxides (Brady and Clauser,1977).

1.8.9.3Plastics

Finely ground, -325 mesh and micronized mica are used in plastics to improve electrical, thermal, and insulating properties. Mica is considered the most effective mineral for reducing war page and increasing stiffness and heat deflection temperature in plastics. In general, mica reinforces crystalline better than amorphous polymers. Best results are obtained with non polar polymers used in both thermoplastics and thermo sets. Its largest single used is in polyolefin, even though it requires stabilizers to prevent degradation of polypropylene. Both muscovite and phlogopite mica are used in plastics, with high aspect ratio grades preferred for their superior reinforcement properties(Eirich,1978).

1.8.9.4 Drilling fluids

Coarse, hammer milled (+10 mesh) mica is used in water based oil well drilling fluids to prevent fluid loss into porous rock formations. The coarse mica flakes bridge openings and seal porous sections of the drill hole against loss of circulation. Mica's platy nature also aids in the suspension of drilling fluid solids and cuttings (Mark, et al, 1971).

1.8.9.5 Other uses of Mica

Ground mica is used as an asbestos substitute in certain thermal boards, brake linings, gaskets, and cement pipes, as a filler and nonstick surface coating for roll roofing and asphalt shingles, as a mold lubricant and release agent in the manufacture of tires and other molded rubber goods, as a flux coating on welding rods, and as a pearlescent pigment in wall papers(Dick,1987).

1.8.10 Industrial Applications of Mica

Sheet mica is used in a number of electrical and electronic appliances in different shapes and sizes. As an insulating material it is used in equipment like condensers, transformers, sheostats, radio and electronic tubes and radar circuits. It is used in the form of washer, discs, tubes and plates.(Flick,1989). Nowadays mica is finding increasing use in equipment that encounters very high temperatures like rockets, missiles, and jet engine ignition system. It is reported that in the manufacture of Telstar transmission satellites by the USA, good use of mica has been made. The success of space research both in USA and USSR is to some extent due to the usefulness of mica in the fields of communication and insulation. Phlogopite is used in spark plugs. Sheet mica, however, is not always available in required size as demanded by the industry.

Great progress has been achieved in making built–up mica called micanite. Mica films are placed with alternate layers of binding materials like shellac, alkyl, or silicon resin and then pressed and baked. Micanite is in common use now. It is convenient to cut or punch micanite according to requirement.

In the electric–field, natural mica is mainly used in the manufacture of capacitors such as bridge–resistance and low–loss properties at high frequencies are required. In such delicate equipment, mica of thickness varying from 0.015 in. and below are used. The mica of thickness in the range of 0.007 to 0.015 in. is used in bridge–spacers. Even thinner films, between 0.004 to 0.006 in. are used as backing plates for capacitors and further thinner films in the range of 0.0007 to 0.002 in. as dielectric. The splitting of such fine thickness are made with the help of pin and knife only.

Scrap mica obtained during the processing of crude mica and in the factories while punching is utilized in the manufacture of mica bricks for heat insulation, mica power use as filler in rubber goods, lubricant and to some extent in plastic industries. It is also used manufacture of roofing material, welding rod, wall–paper, lamp chimneys, shades etc.Waste or scarp mica is used invariable in the form of ground mica. The uses of ground mica depend, largely, upon its appearance and lubricating properties. Both of these characteristics are affected by the methods of grinding a well as the purity and nature of the scrap.

Muscovite has many different industrial uses depending on its condition, whether that be ground or sheet. Ground mica is often used in paint because of its light weight and flat shape. It holds the paint together, protecting the paint film and keeping it from becoming chalky or shrinking. It also prevents the paint from weather and water.

Muscovite possesses great resistance to the passage of electricity and heat and it is therefore used as an electronic insulator.

Muscovite was used as a glass because of its transparent layers that peel in thin sheets. It was also used in furnace doors, wallpaper, glitter, makeup, fireproofing, fillers, and a lubricant in oils.

1.8.11 Investigation of DNA adsorption on chlorite and other layered minerals

Muscovite mica is, commonly, used to immobilize DNA molecules onto a flat surface. This method , however, requires either the use of divalent cations in the buffer solution or the chemical modification of the surface, as both the surface and DNA molecules are negatively charged. DNA molecules have different binding affinities and assume different conformations when adsorbed to different layered minerals. Using atomic force microscopy (AFM) it has been, experimentally, confirmed that biotite, talc and brucite have a much higher affinity than muscovite, with -7, -20, and -25fold more volume of DNA deposited, respectively.

The deposition of DNA onto chlorite presents two distinct areas. Brucite–like regions have high DNA coverage, and mica–like regions where DNA molecules are almost absent. In some cases, DNA molecules become stretched across these mica–like regions. The stretching is driven by the surface potential gradient between the two regions.

Differences in the structure and chemistry of the mineral substrates are responsible for a variety of DNA adsorption mechanisms. The active stretching of DNA on chlorite is a clear indication of the technological potential of this mineral. Work is being out to utilize this property to gain understanding of the adsorption mechanisms. Nanolithography is also implemented to create deposition arrays, along with using the surface to investigate DNA–protein interactions.

Chapter Two

Materials and Methods

2. Materials and Methods

2.1. Materials

Hydrofluoric acid HF

Hydrochloric acid HCl

Zinc (dust) Zn.

Disodium hydrogen phosphate Na_2HPO_4

Oxalic acid. $H_2C_2O_4$

Perchloric acid $HClO_4$

Nitric acid. HNO_3

Potassium bromide KBr

Sodium carbonate Na_2CO_3

Potassium carbonate K_2CO_3

Methyl red.

Sodium diphenyl amino sulphate.

2.1.1. Solvents

i. Ammonia solution NH_4OH

ii. Concentrated ammonia NH_3

iii. Ammonium nitrate NH_4NO_3

2.1.2. Instruments used

1. Flame photometer. AFP 100.
2. Infra red spectrophotometer; FTIR – 8400S, Shimadzu.
3. Furnace Carbolite CWF 1200
4. Oven BTL BS 2648
5. Thermogravimetery TGA PT 1000 Linseis.
6. Energy Dispersive X – Ray Fluorescence Spectrophotometer, ShimadzuEDX–8000
7. Inductively Coupled Plasma ICP – OES 725 ES
8. Perkin Elmer 3110 atomic absorption spectrometer.
9. XRD – 7000 X – Ray Diffractometer maxima- Shimadzu.
10. Mohs Test Scale.
11. Tensile Strength Tester.

1. Impedance Analyzer Quad tech 1920 Precision LCR Meter.

2.2. Methods

2.2.1 Sampling

Two samples of Mica were collected with the aid of a geological research team from Bayuda desert Jebel Rahaba area west of Shereik (River Nile State), and East Sudan (Red Sea State). The samples were designated as follows:

Table: 2.1 Mica Samples Locations

Sample	Area
Numbre1	Shereik(River Nile State)
Number 2	(Red Sea State)

2.2.1.1. Preparation of Samples

Representative samples were obtained by quartering process. A portion of each sample was crushed, pulverized, milled and made to pass a 300 BS, mesh sieve (63 μm).

2.2.1.2 Preparation of Sample for Chemical Analysis

For chemical analysis smaller particle size is used because, it enhances the fusion technique and consequently a clear solution for the analysis is obtained. However, most of naturally occurring silicates are insoluble in hydrochloric acid, hence, their decomposition is carried out by fusion with mixture of sodium carbonate and potassium carbonate in platinum crucible. The residue of fusion is then dissolved in hydrochloric acid. Clay may also be fused with sodium carbonate, or even sodium hydroxide. Decomposition can be accomplished by fusion withlithium metaborate ($LiBO_2$) followed by treatment with acid (Newman,1987).

2.2.1.3 Preparation of Samples (1 and 2) for EDXRF

For Energy Dispersive X – ray Fluorescence the sample of Mica powder placed on glass slide and dried.

2.2.1.4 Preparation of Sample for XRD

The sample of Mica powder was prepared for X–ray Diffraction by placing the sample on glass slide and dried.

2.2.1.5 Preparation of Sample for ICPOES

0.5011g of powdered mica sample was weighed into platinum dish, 7 ml of HCl 37%, 1 ml of HNO_3 65%, and 2 ml of HF 40% were added. The dish was covered with a lid. The reaction, was allowed to proceed for at least 15 min, then the lid was removed and the mixture evaporated to dryness on a sand bath in a fume cupboard, taking care to avoid spurting. Cooled, and 200 ml of deionized water were added. 10 ml of the above solution was diluted to 100 ml to identify (K, Ca, and Mg) multiply by dilution factor.

2.3. Methodology (Experimental)Techniques

2.3.1 Chemical Analysis

The Chemical Analysis was carried out using the Classical Methods of Analysis namely Gravimetric and Volumetric Analysis for the major constituents. However, for trace elements Instrumental Methods were used such as Inductively Coupled Plasma Optical Emission Spectroscopy(ICP OES),Atomic Absorption Spectroscopy (AAS), Flame Photometer, X–ray Diffraction (XRD), and Energy Dispersive X–ray Fluorescence (EDXRF).

2.3.1.1. Loss on Ignition

1.015g of powdered mica sample was heated for 15 min. and weighed covered platinum crucible (a porcelain crucible may also be used) of 20 to 25 ml capacity by placing it in a muffle furnace at temperature of 900–1000^{0}C, cooled and reweighed, percent loss in weight was recorded as the loss on ignition and was calculated (Vogel,1959).

2.3.1.2.Moisture

1.021g of powdered mica sample was heated for one hour in a weighed and covered platinum crucible in oven at temperature 105^{0}C, cooled and are weighed. The loss in weight as moisture was recorded and calculated as weight percent. (Vogel, 1959).

2.3.1.3. Determination of Silica

Silica as a major constituent of Mica was determined Gravimetrically according to the Jeffery's method(Jeffery, 1975) 0.5021g of each powered Mica sample, 5.0 g of sodium carbonate and potassium carbonate fusion mixture (1:1 w:w) were fused in platinum crucible. The melt was dissolved in (1:1) HCl, then evaporated on a steam bath and baked in an oven at 105^{0}C for one hour.

The residue was dissolved in hot water and dilute HCl. The silica was filtered through ashless filter paper No. 41 Whatman, ignited in platinum crucible at 1000^{0}C ,cooled, and weighed. As confirmation the residue was mixed with H_2SO_4 and HF, evaporated, ignited and the crucible finally was weighed (Vogel,1959). The difference between the two weights represent the weight of silica.

2.3.1.4. Determination of Combined Oxides

In a suitable aliquot of the stock filtrate from silica determination 100 ml of the solution was treated with ammonia solution (1:1) and ammonium chloride. the filtrate and washing collected in a 250 ml volumetric flask for determination of aluminum oxide (Al_2O_3). From the stock solution 100 ml were taken using a pipette in a 250 ml beaker (acidic solution), add methyl orange and heat, add dropwise conc. ammonia solution till just alkaline, $Al(OH)_3$ will form as a gelatinous, ppt. heat to co–ogulate and filter. Ignite and weigh the ppt. as Al_2O_3.

2.3.1.5. Determination of Iron

In a suitable aliquot of the stock of silica determination, total iron can be determined volumetrically (Vogel, 1959) method of reduction with stannous chloride or zinc granules, followed by titration versus potassium dichromate or permanganate

50 ml of the filtrate was boiled with zinc granules, the resulting solution was cooled, 5 ml of (1:1) H_3PO_4 and 5 ml of (2M) H_2SO_4 were added followed by 3 drops of diphenylamine indicator. The solution was titrated against standard potassium dichromate. Total iron was calculated from the difference of combined oxides (Vogel,1959).

2.3.1.6. Determination of Calcium Oxide

100 ml from filtrate combined oxide solution was diluted to 350 ml by distilled water on 500 ml beaker, was heated in Bunsen burner, and 2.004g of oxalic acid was added the color was changed to pink, the dropwise of diluted ammonia was added to precipitate in alkaline media.(Vogel,1959).

2.3.1.7. Determination of Magnesia

100 ml of filtrate solution was heated, 10 ml of disodium hydrogen phosphate was added, then the solution was turned in cool water bath and 30–40 ml of concentrated ammonia was added with continuous stirring for 30 min. the solution let overnight, then the solution was filtrated, burned, and ignited at about 800 – 900^{0}C for 30 min. (Vogel,1959).

2.3.1. 8. Determination of Potassium and Sodium

Accurately 1.013g of powered mica sample was weighed in platinum dish, 5 ml of concentrated nitric acid, 5 ml of perchloric acid, and about 10 ml of hydrochloric acid were added, the dish was covered with a lid. The reaction was allowed to proceed for 15 min, was removed the lid the mixture dryness on a sand bath in a fume cupboard, taking care to avoid spurting. 5 ml of perchloric acid were added, the lid and sides of the dish were rinsed with water and carefully evaporated to dryness. To the cool, dry residue 2 ml of concentrated hydrochloric acid and 15 ml of deionized water were added.

Digested on a steam bath for 10 min, cooled, filtered, if necessary. The solution was diluted with deionized water to 250 ml in a volumetric flask, and Stored in a polythene bottle (Vogel, 1959).

2.3.1.9. Instrumental Methods of Analysis

2.3.1.9.1 Flame Photometry

Flame Photometry is one of the most sensitive and precise methods for the determination of alkali metals. If a solution of such metals is aspirated into a suitable flame, optical emission lines are observed from the elements, these lines are detected at a characteristic wavelength and their intensity is proportional to the concentration of the element in solution (Vogel, 1959).

2.3.1.9.2 Inductively, Coupled Plasma Optical Emission Spectroscopy (ICP–OES)

A prepared solution containing analyte elements is aspirated into the plasma generated by inductively coupled plasma source; the atomized elements produce characteristic emission spectral lines, which are separated by a simultaneous optical spectrometer. The intensity of spectral line of an element is proportional to its concentration.

Equipment of ICPOES are Inductively, Argon Plasma – Atomic Emission Spectrometer (ICP–AES), Varian, Vista–MPX–CCD, Simultaneous, with background correction, auto–fit multi–

calibration curve fitting, Signal to Background Ratio (SBR), and signal to root background ratio.

2.3.1.9.3 Atomic Absorption Spectrophotometer (AAS)

Atomic Absorption Spectroscopy (AAS) Fig3 is a sensitive technique for quantitative analysis. Generally it can be used for the determination of major or trace elements in silicate minerals (Skoog,1992).

In this investigation atomic absorption runs were carried out using Perkin Elmer 3110 atomic absorption spectrometer. (Vogel,1959). To 0.5020g of each sample 10 ml HF and 5 ml Conc. HNO_3 were added, and the resulting solution was evaporated to dryness on steam bath. 1 ml of 10% HCl was added and the resulting solution was quantitatively transferred to a 200 ml volumetric flask and a few ml of lanthanum chloride was added to eliminate chemical interferences. Lanthanum can act not only as a releasing agent but as ionization suppressor. The resulting solution was completed to the mark with deionized water. A blank solution was prepared in the same way.

To calibrate the instrument a standard solution (1000 ppm) and a series of dilutions were prepared for magnesium, iron, potassium, manganese, and sodium. The absorption of the standard and sample for each element were measured. A calibration curve was constructed for the standard from which the concentration of each element was determined.

2.3.1.9.4 Infra–Red Spectrophotometer (IR)

Infrared runs were carried out using Perkin Elmer, FT – IR spectrophotometer spectrum 1000. The radiation source is placed within the instrument, where mirrors are used to produce two parallel beams (referred to as reference and sample beams respectively). A KBr disc (blank) is placed in the reference and a disc of KBr containing the sample is placed in the path of the sample beam.

After passing through the KBr disc the two beams are recombined alternating beam, which then passes through diffraction grating, one particular wavelength being selected by an exit slit. The grating is slowly rotated to scan a range of wavelengths by the scan motor, which also rotates the recording drum. As long as the intensities of the sample beams change with the change in wavelength, infrared spectrum of the sample was recorded on the chart mounted on the recording drum. The scanning limit is 4000–200 cm^{-1} at a medium speed (Elzamzami, 1979).

2.3.1.9.5 X – ray Diffraction

In clay mineralogy, X–ray diffraction (XRD) is used to study the fine structures of minerals (Maurice, 1988). A diffractometer is an instrument for studying materials by measurement of the way in which they diffract X–ray of a known wavelength. In a diffractometer the intensity of a diffracted beam is measured directly either by means of ionization it produces in a gas, or the fluorescence it produces in a solid.

The diffractometer consists of: Goniometer, X–ray tube, tube stand, slit system carrier and detector. A monochromatic radiation from an X–ray tube is diffracted by the specimen to form convergent diffracted beam which comes to a focus at a slit and then enters the counter. The receiving slit and the counter are supported on a carriage which may be rotated around the diffractometer axis whose angular position 2θ may read on graduated scale (D8 Advanced/D8 Discover X–ray diffractometerUsers Manual, 1999).

The diffraction pattern may be obtained either continuously or intermittently. In the former, the counter is set near 2θ = 0 and connected to a counting rate meter which is then driven at constant angular velocity through increasing values of 2θ unit, the whole angular range is scanned. At the same time, the proper chart on the recorder moves at a constant speed, so the distance along the length of the chart is proportional to 2θ.

In the latter the counter is connected to a scaler and set at fixed values of 2θ for a time sufficient to make an accurate count. The counter is then moved to a new angular position and the operation is repeated. The whole range of 2θis converted in this fashion and a curve of intensity versus 2θ is finally plotted (Chung and Smith, 2000).

AD8 advanced X–ray diffractometer from Bruker Analytical X –ray System was used. It was operated at a generated voltage of 40Kv and 40 mA current using copper filter CuK α radiation. The samples were scanned at a limit 2θ range $4^0 - 70^0$, $\Delta\theta = 0.02$, step time 3 sec/point (Buhrke et al., 1998).

53.3.1.9.6 Energy Dispersive X–ray Fluorescence

Energy Dispersive X–ray Spectroscopy (EDS or EDX) is a chemical microanalysis technique used in conjunction with scanning electron microscopy (SEM). The EDS technique detects X–rays emitted from the sample during bombardment by an electron beam to characterize the elemental composition of the analyzed volume. Features or phases as small as 1 µm or less can be analyzed (Indian Standard, 1972).

When the sample is bombarded by the SEM's electron beam, electrons are ejected from the atoms comprising the sample's surface. The resulting electrons vacancies are filled by electrons from a higher state, and an X–ray is emitted to balance the energy difference between the two electrons states. The X–ray energy is characteristic of the element from which it was emitted.(Leonard, 1977).

The EDS X–ray detector measures the relative abundance of emitted X–rays versus their energy. The detector is typically a lithium–drifted silicon, solid–state device. When an incident X–ray strikes the detector, it creates a charge pulse that is proportional to the energy of the X–ray. The energy pulse is converted to a voltage pulse (which remains proportional to the X–ray energy) by a charge – sensitive preamplifier. The signal is then sent to a multichannel analyzer where the pulses are sorted by voltage. The energy, as determined from the voltage measurement, for each incident X–ray is sent to a computer for display and further data evaluation. The spectrum of X–ray energy versus counts is evaluated to determine the elemental composition of the sampled volume.(Norrish and Huttopn,1967 and Corradi and Leonelli,1996).

XRF and EDXRF Spectrometers are the elemental analysis tools of choice, for many applications, in that they are smaller, simpler in design and cost less to operate than other technologies like Inductively, Coupled Plasma Optical Emission Spectroscopy (ICP–OES) and Atomic Absorption Spectroscopy (AAS). Examples of some common EDXRF applications are: Cement and raw meal: sulfur, iron, calcium, silicon, aluminum, magnesium, etc., Kaolin clay: titanium, iron, aluminum, silicon, etc., Granular catalysts: palladium, platinum, rhodium, ruthenium, etc., Ores: copper, tin, gold, silver, etc., Cement and mortar fillers: sulfur in ash.

2.3.1.9.7 Thermogravimerty

In thermogravimetry (TG) one generally reports or records the weight of a substance as it is heated through a selected rate of temperature. The weight loss is plotted versus temperature. The record obtained is called thermogravimetric curve which provides information concerning the thermal stability and composition of the sample.

Basically the thermogravimetric apparatus consists of recording balance, sample holder, furnace and a programme controller that records heat of the sample at a uniform rate.

(Dollimore and Keattch,1963). Method in which a few mg of the sample was introduced into platinum crucible. The run was carried out in air using heating rate of 20^0C/min. the weight change and temperature rise were recorded automatically on a chart. The weight changes over the temperature range up to 1000^0C were plotted as percentage weight loss over this temperature range.(Wendlandt,1986).

2.3.2 Mechanical Properties

2.3.2.1 Testing of Hardness of Mica Samples by Mohs Hardness Scale

One of the most important tests for identifying minerals specimens is Mohs Hardness Test. This test compares the resistance of a mineral to being scratched by ten reference minerals known as the Mohs Hardness Scale. (Chandler, 2000).

Table: 2.2Mohs Hardness Scale

Mineral	Hardness (Mohs)
Talc	1
Gypsum	2
Calcite	3
Fluorite	4
Apatite	5
Orthoclase	6
Quartz	7
Topaz	8
Corundum	9
Diamond	10

2.3.2.2 Measurement of Tensile Strength and Modulus of Elasticity of Mica Samples

A tensile test, also known as tension test, is probably the most fundamental type of mechanical test you can perform on material. Tensile tests are simple, relatively inexpensive, and fully standardized. By pulling on something, you will very quickly determine how the material will react to forces being applied in tension, as the material is being pulled, you will find its strength along with how much it will elongate. (Courtney,2000).

You can learn a lot about a substance from tensile testing. As you continue to pull on the material until it breaks, you will obtain a good, complete tensile profile. A curve will result showing how it reacted to the forces being applied. The point of failure is of much interest and is typically called it "Ultimate Tensile Strength" or UTS on the chart.

Hooke's law

For most tensile testing of materials, you will notice that in the initial portion of the test, the relationship between the applied force, or load, and the elongation the specimen exhibits is linear. In this linear region, the line obeys the relationship defined as "Hooke's law" where the ratio of stress to strain is a constant, or

$$E = \sigma / \varepsilon$$

E is the slope of the line in this region where stress (σ) is proportional to strain (ε) and is called the "Modulus of Elasticity" or "Young's Modulus". (Davis,2004).

The Modulus of Elasticity is a measure of the stiffness of the material, but it only applied in the linear region of the curve, if a specimen is loaded within this linear region, the material will return to its exact same condition if the load is removed. At the point that the curve is no longer linear and deviates from the straight – line relationship, Hooke's law no longer applied and some permanent deformation occurs in the specimen. This point is called the "elastic , or proportional limit". From this point on the tensile test, the material reacts plastically to any further increase in load or stress. It will not return to its original, unstressed condition if the load were removed. A value called " Yield Strength" of a material (e.g. metals and plastics), the departure from the linear elastic region cannot be easily identified. Therefore, an offset method to determine the Yield Strength of the material tested is allowed. These methods are discussed in ASTM E8 (metals) and D638 (plastic). An offset is specified as α% of strain(for metals, usually 0.2% E8 and sometimes for plastics α value 2% is used). The stress (R) that is determined from the intersection point "r" when the line of the linear elastic region (with slope equal to Modulus of Elasticity) is drawn from the offset "m" becomes the Yield Strength by the offset methods. (Dieter,1986).

Alternative Moduli: The tensile curves of some materials do not have a very well–defined linear region. In these cases, ATSM Standard EIII provides for alternative methods for determining the Modulus of a material, as well as Young's Modulus. These Alternative Moduli are the Secant Modulus and Tangent Modulus.(Dowling,1998).

Strain, You will also be able to find the amount of stretch or elongation of the specimen undergoes during tensile testing this can be expressed as an absolute measurement in the change in length or as α relative measurement called "Strain". Strain itself can be expressed in two different ways, as "engineering strain" and "true strain". Engineering strain is probably the easiest and the most common expression of strain used. It is the ratioof the change in length to the original length.(McClintock and Argon1966).

$e = (L – L_0)/L_0 = \Delta L/L$

Whereas, the true strain is similar but based on the instantaneous length of the specimen as the test progresses:

$\varepsilon = \ln (L_i/L_0)$

Where L_i is the instantaneous length and L_0 the initial length.

Ultimate Tensile Strength: One of the properties you can determine about a material is its Ultimate Tensile Strength (UTS). This is the maximum load the specimen sustains during the test. The UTS may or may not equate to the strength at break. This all depends on what type of material you are testing brittle, ductile or a substance that even exhibits both properties. Sometimes α material may be ductile when tested in a lab, but, when placed in service and exposed to extreme cold temperatures it may transition to brittle behavior.

2.3.3 Electrical Properties

2.3.3.1 Measurement of Dielectric Constant

A dielectric is a material having low electrical conductivity in comparison to that of a metal. It is characterized by its dielectric constant. Dielectric constant is measured as the ratio of the capacitance C of an electrical condenser filled with the dielectric to the capacitance C_o of evacuated condenser i.e.,

$$\varepsilon = C/C_0$$

Chapter Three

Results and Discussions

Chapter Three

3. Results and Discussions

Table 3.1 shows the percentage composition of oxides in samples (1and 2) moisture and loss on ignition, the results show almost, identical composition of the Mica ore samples. The results obtained by the classical methods such as volumetric and gravimetric were used to determine the major and impurities constituents were found to be more or less consistent with those obtained by ICPOES, AAS, FE, EDXRF.

Table 3.2 Results of flame emission shows that no significant difference in oxides percent of K and Na of the Mica ore sample

Table: 3.1 Results of Chemical Analysis of Elements and their Oxides, Moisture and Loss on Ignition in samples (1 and 2)

Constituent of mica	Percentage sample(1)	Percentage sample(2)
SiO_2	40.03%	39.02%
Al_2O_3	35.54%	36.01%
Fe_2O_3	2.12%	2.11%
CaO	0.73%	0.67%
MgO	0.32%	0.32%
K_2O	18.16%	18.05%
Na_2O	0.37%	0.54%
Moisture	0.36 %	0.28%
Loss on Ignition	4.64%	4.48%

Table: 3.2Results of Flame Photometer Analysis of Elements and their Oxides in samples (1 and 2)

Sample	Element	Concentration (ppm)	%Oxide composition
Sample (1)	K	20.2 ppm	16.76%
Sample (1)	Na	0.55 ppm	0.40%
Sample (2)	K	21.7 ppm	18.00%
Sample (2)	Na	0.46 ppm	0.34%

Table 3.3 shows the results of analysis of mica samples (1and 2) by Inductively, Coupled Plasma Optical Emission Spectrophotometer.

Table: 3.3 Results of ICPOES Analysis of Elements and their Oxides in samples (1 and 2)

Element	Concentration (ppm) sample(1)	Concentration (ppm) sample(2)	% Oxide composition sample (1)	% Oxide composition sample (2)
Al	51.05ppm	49.98ppm	37.53%	35.78%
Si	52.51ppm	54.79ppm	40.49%	39.90%
K	33.91ppm	32.06ppm	19.51%	20.09%
Fe	3.98ppm	4.41ppm	2.27%	2.52%
Mg	0.92ppm	0.67ppm	0.62%	0.33%
Mn	0.13ppm	0.11ppm	0.067%	0.056%
Ca	1.64ppm	1.34ppm	0.92%	0.75%
Na	1.12ppm	0.09ppm	0.60%	0.05%

Table 3.4 shows the results of analysis of elements and their oxides of mica samples byAAS.

Tables 3.5 and 3.6 shows the results of Analytical Results of elements and their oxides of Mica Samples (1and2) by EDXRF respectively.

Table: 3.4 Results of AASAnalysisof Elements and their Oxides in samples (1 and 2)

Element	Concentration (ppm) sample(1)	Concentration (ppm) sample(2)	% Oxide composition sample (1)	% Oxide composition sample (2)
Fe	10.77 ppm	9.49 ppm	2.15%	1.99%
K	30.98 ppm	28.35 ppm	31.06%	29.24%
Mn	0.045 ppm	0.054 ppm	0.023%	0.027%
Mg	0.58 ppm	0.39 ppm	0.39%	0.26%
Na	0. 11ppm	0.13 ppm	0.06%	0.07%

Table: 3.5 Results of EDXRF Analysis of Elements and their Oxides in sample (1)

Element	Result	% Oxide composition	3σ
K	33.364%	27.93%	0.151
Si	31.733%	36.01%	0.526
Al	18.702%	33.79%	0.561
Fe	12.268%	2.02%	0.048
Ca	1.286%	0.79%	0.085
Ti	1.064%	0.02%	0.036
S	0.535%	0.11%	0.130

Rb	0.518%	0.02%	0.007
Mn	0.185%	0.04%	0.006
Zn	0.137%	0.03%	0.004
Ga	0.096%	0.02%	0.003
Nb	0.058%	0.01%	0.004
Cu	0.049%	0.03%	0.008
Pb	0.007%	Traces	0.001

Table: 3.6 Results of EDXRF Analysis of Elements and their Oxides in sample (2)

Element	Result	% Oxides composition	3σ
K	30.031%	26.40%	0.152
Si	29.109%	36.49%	0.544
Fe	20.619%	1.76%	0.066
Ca	0.000%	0.000%	0.000
Al	17.137%	34.97%	0.603
Rb	1.147%	0.01%	0.012
Ti	0.485%	0.02%	0.022
Sn	0.364%	0.013%	0.025
Nb	0.350%	0.02%	0.006
Mn	0.280%	0.06%	0.008
Zn	0.267%	0.06%	0.006
Ga	0.159%	0.01%	0.004
Cu	0.045%	0.05%	0.008
Ti	0.007%	0.02%	0.003

Table: 3.7 Hardness of Mica Samples (1and 2)

Sample	Hardness
Sample(1)	2 – 3 Mohs
Sample(2)	2 – 3 Mohs

Table 3.7 shows Hardness of Mica samples (1 and 2)

Table: 3.8 shows the Tensile Strength and Modulus Elasticity of sample Mica (1and2) by Tensile Strength Tester

Table: 3.8 Tensile Strength and Modulus of Elasticity of Mica samples (1and 2)

Sample	Tensile Strength	Modulus of Elasticity
Sample(1)	1734 Kg/cm^2	$1380x10^{-3}$ – $2015x10^{-3}$ Kg/cm^2
Sample(2)	1718 Kg/cm^2	$1392x10^{-3}$ - $2070x10^{-3}$ Kg/cm^2

Table: 3.9 shows Dielectric Constant of Mica samples(1and 2).

Table: 3.9 Dielectric Constant of Mica Samples (1and 2)

Sample	Dielectric Constant
Sample(1)	2.8 – 4.7 kV/mm
Sample(2)	2.8 – 4.7 kV/mm

Tables 3.10 and 3.11 shows oxides percent of impurities by different techniques were found to be more or less consistent with those obtained with gravimetric and volumetric analysis.

Table: 3.10 Analysis of sample (1) oxides (impurities) by different techniques

Impurities	FE	ICPOES	AAS	EDXRF
Fe_2O_3	-	2.27%	2.15%	2.02%
Na_2O	0.40%	0.50%	0.55%	-
CaO	-	0.92%	-	0.79%
TiO	-	-	-	0.01%
CuO	-	-	-	0.03%
MnO	-	0.07%	0.05%	0.04%
Rb_2O	-	-	-	0.02%
ZnO	-	-	-	0.04%
MgO	-	0.62%	0.60%	0.56%

Table: 3.11 Analysis of sample (2) oxides (impurities) by different techniques

Impurities	FE	ICPOES	AAS	EDXRF
Fe_2O_3	-	2.52%	1.99%	2.00%
Na_2O	0.34%	0.38%	0.61%	-
CaO	-	0.75%	-	-
TiO	-	-	-	0.02%
CuO	-	-	-	0.05%
MnO	-	0.06%	0.04%	0.06%
Rb_2O	-	-	-	0.01%
ZnO	-	-	-	0.06%
MgO	-	0.33%	0.56%	0.49%

3.1 Characterization

3.1.1 Chemical Analysis Results

The results of chemical analysis using different methods are shown in Table 3.1. It can be seen that the major constituents in the mica are the silica (SiO_2) and alumina (Al_2O_3). The constituents of silica and alumina are determined by the classical methods, for sample (1) from River Nile State SiO_2 40.03%, Al_2O_3 35.54% and for sample (2) from Red Sea State SiO_2 39.02%, Al_2O_3 36.01%.

The same constituents SiO_2, Al_2O_3 when determined by ICPOES for sample (1) are 40.49% and 37.53% respectively, Table 3.3 and for sample (2) are 39.90% and 35.78% respectively, Table 3.3 . Also the constituents SiO_2 and Al_2O_3 when determined by EDXRF technique gave results for sample (1) 36.01% and 33.79% respectively Table 3.5 and for sample (2) 36.49. %, 34.97% respectively Table 3.6.

However, a minor difference is shown when the results were compared with those obtained in Table1.10 (www.tedpella .com).

As it is seldom, if ever to find pure Mica in nature ; Fe_2O_3, Na_2O, CaO, MgO, TiO_2 and MnO are always presents as impurities. The iron content (Fe_2O_3) in samples (1and 2) was 2.12% and 2.11% respectively.

3.1.2 X – ray Diffraction Results

By using the XRD technique, it is possible to determine the mineralogical composition of samples (1and 2) of mica since each mineral has its characteristic feature in the XRD pattern. In contrast the chemical analysis, does not give the necessary information regarding the mineralogical composition of the mica i.e., in chemical analysis it is difficult to determine whether the reported silica content (SiO_2%) comes only from the mica mineral or from kaolinite or quartz.

3.1.3 Thermogravimetry Result

Thermogravimetry(TG) curves for the studied mica samples (1and2) shown in appendix (3and4). It is evident that a slight loss of weight (0.15%) was observed at the temperature range 100^0C – 105^0C. No loss on weight was observed at the temperature 110^0C – 400^0C, but the weight loss remained almost constant above 520^0C.

3.1.4 Infra – red Spectroscopy Result

The principal infra – red absorption bands of mica samples (1and 2) are shown in appendix (1and2). The mica samples (1and 2) showed very strong absorption band at 3622 cm^{-1}. Strong band at 762 cm^{-1}, while two medium bands appeared at 800 cm^{-1} and 831 cm^{-1}.

The appearance of very strong band at 3622 $cm^{-1}$is an indication of free hydroxyl water, which is presumably associated with aluminum in the $(Al – O(OH)_3^-$ octahedral.

Most of the frequencies assigned to the Si – O vibration is strong at 762 cm^{-1} and it is consistent with those reported for the reference samples and literature values (Tuddenham, and Lyon,1951).

3.1.5 ICPOES Results

The impurities in mica samples (1and2) identify by ICPOES are Fe_2O_3 2.27%, 2.52%, Na_2O 0.50% , 0.38%, CaO 0.92% ,0.75%, MnO 0.07%, 0.62%, 0.06% and MgO 0.33% are correlates very well with those obtained in the previous studies Table 1.9.

3.1.6 EDXRF Results

The impurities in mica samples (1 and 2) identify by EDXRF are Fe_2O_3 2.02% , 2.00%, CaO 0.79%, 0.00%,TiO 0.01% , 0.02%, CuO 0.03% , 0.05%, MnO 0.56%, 0.04%, Rb_2O 0.02%, 0.01%, ZnO 0.04%, 0.06%, MgO 0.56%, 0.04% are correlated very well with those obtained in table 1.9.

3.1.7 AAS Results

The impurities in mica samples (1 and 2) measured by AAS Fe_2O_3 2.15%, 1.99%, Na_2O 0.55%, 0.61%, MnO 0.05%, 0.04%, MgO 0.06%, 0.56% are correlated with those obtained in Table 1.9.

3.1.8 Flame photometry Results

The value of Na_2O which measured by flame emission in mica samples (1 and 2) 0.04%, 0.34% were nearest to the ones obtained in Table 1.11.

3.1.9 Hardness Result

Hardness of mica samples (1 and 2) measured by Mohs hardness scale found between gypsum and calcite, are correlated very well with those obtained in Table 1.11

3.1.10 Tensile Strength and Modulus of Elasticity Result

Tensile strength of mica samples (1 and 2) measured by Tensile Strength Tester are 1734 Kg/cm^2, 1718 Kg/cm^2which are correlated very well with those obtained in Table 1.11.

Modulus Elasticity of mica samples (1 and 2) which also measured by Tensile Strength Tester are between 1380×10^{-3} – 2015×10^{-3} Kg/cm^2, 1392×10^{-3} – 2070×10^{-3} Kg/cm^2 are nearest to those obtained in Table 1.11

3.1.11 Dielectric Constant Result

Dielectric Constant of mica samples (1 and 2) which measured by Impedance Analyzer are 2.8 – 4.7 KV/mm are nearest with those obtained in Table 1.11.

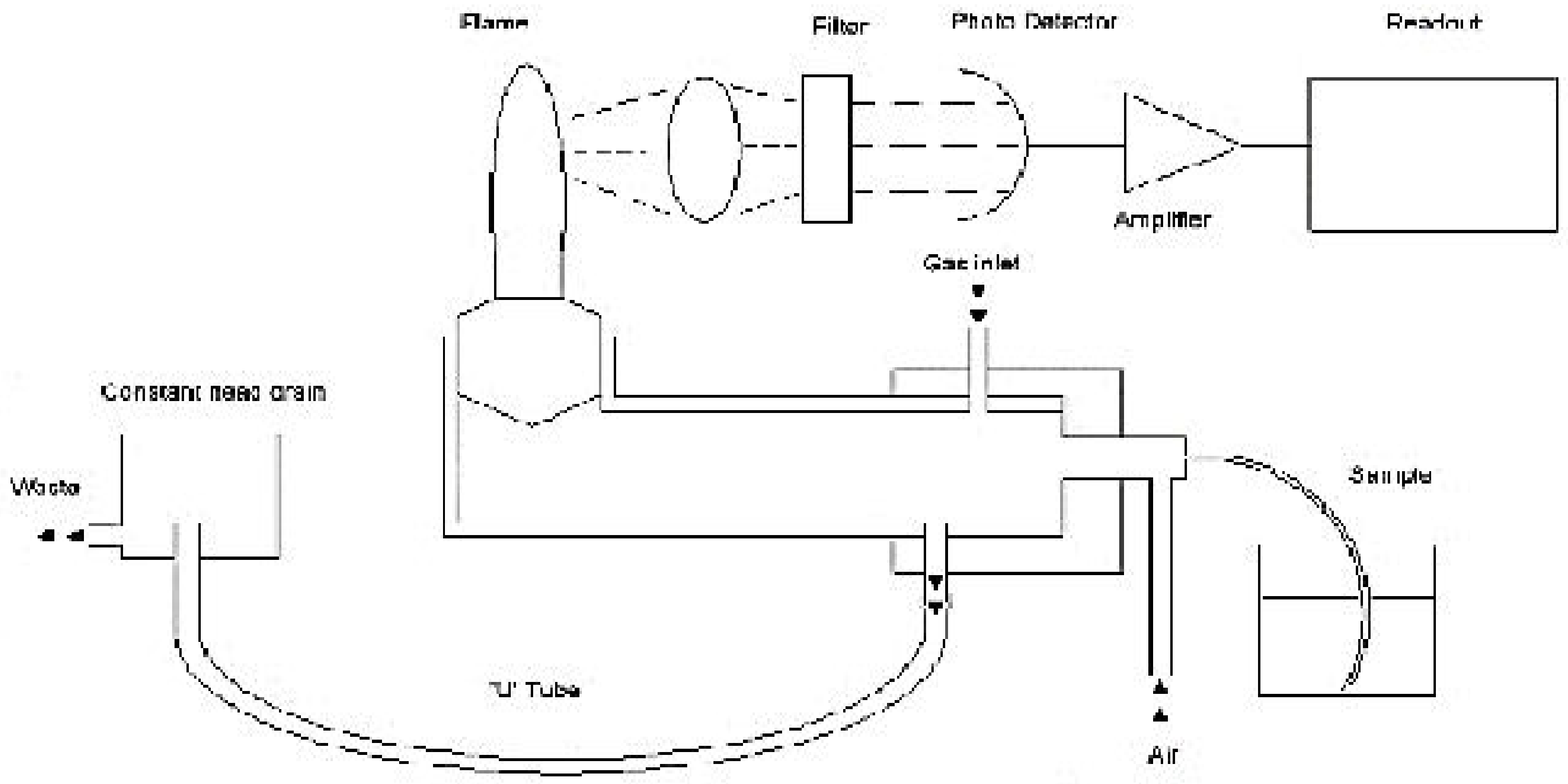


Figure No. 1 Basic Components of a simple Flame Photometer

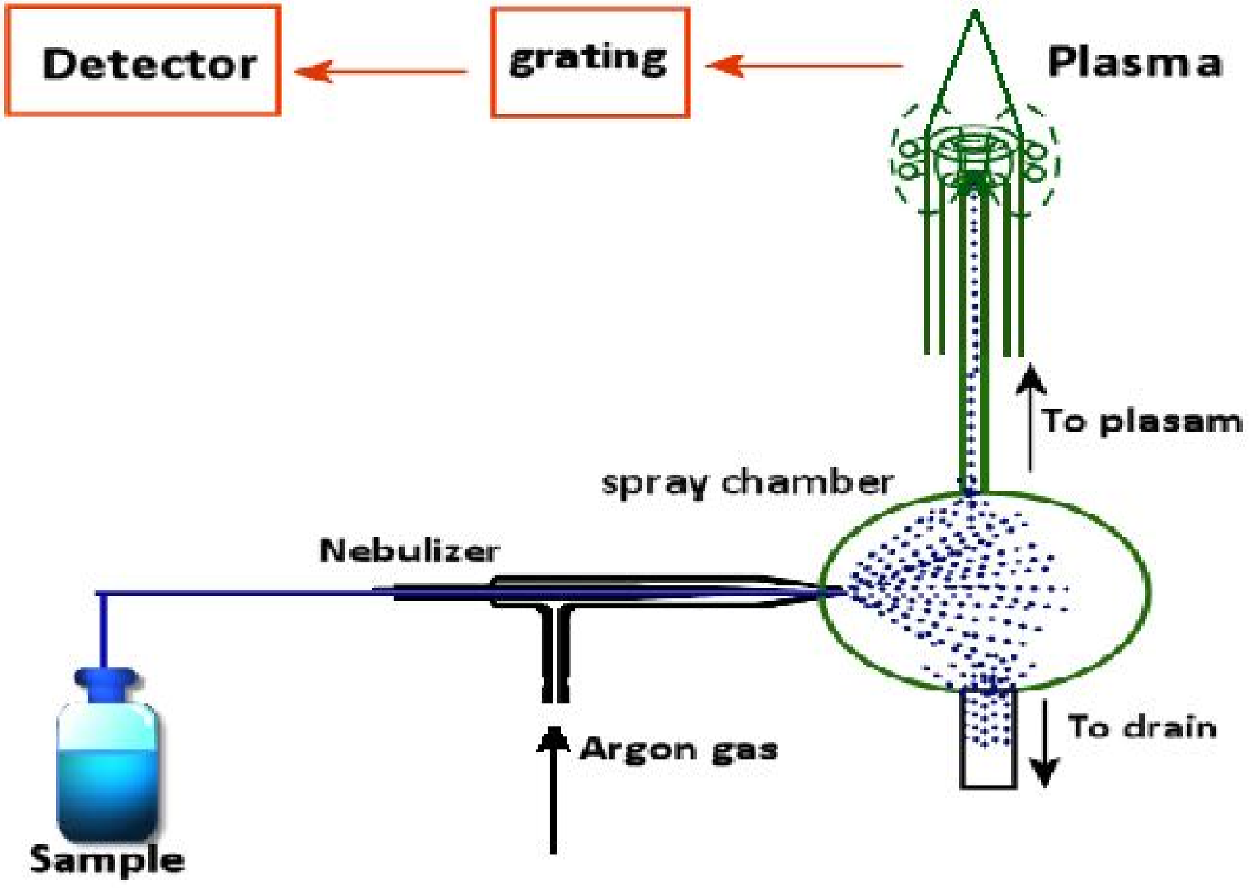


Figure No. 2 Schematic Diagram of Inductively Coupled Plasma Optical Emission Spectroscopy Apparatus

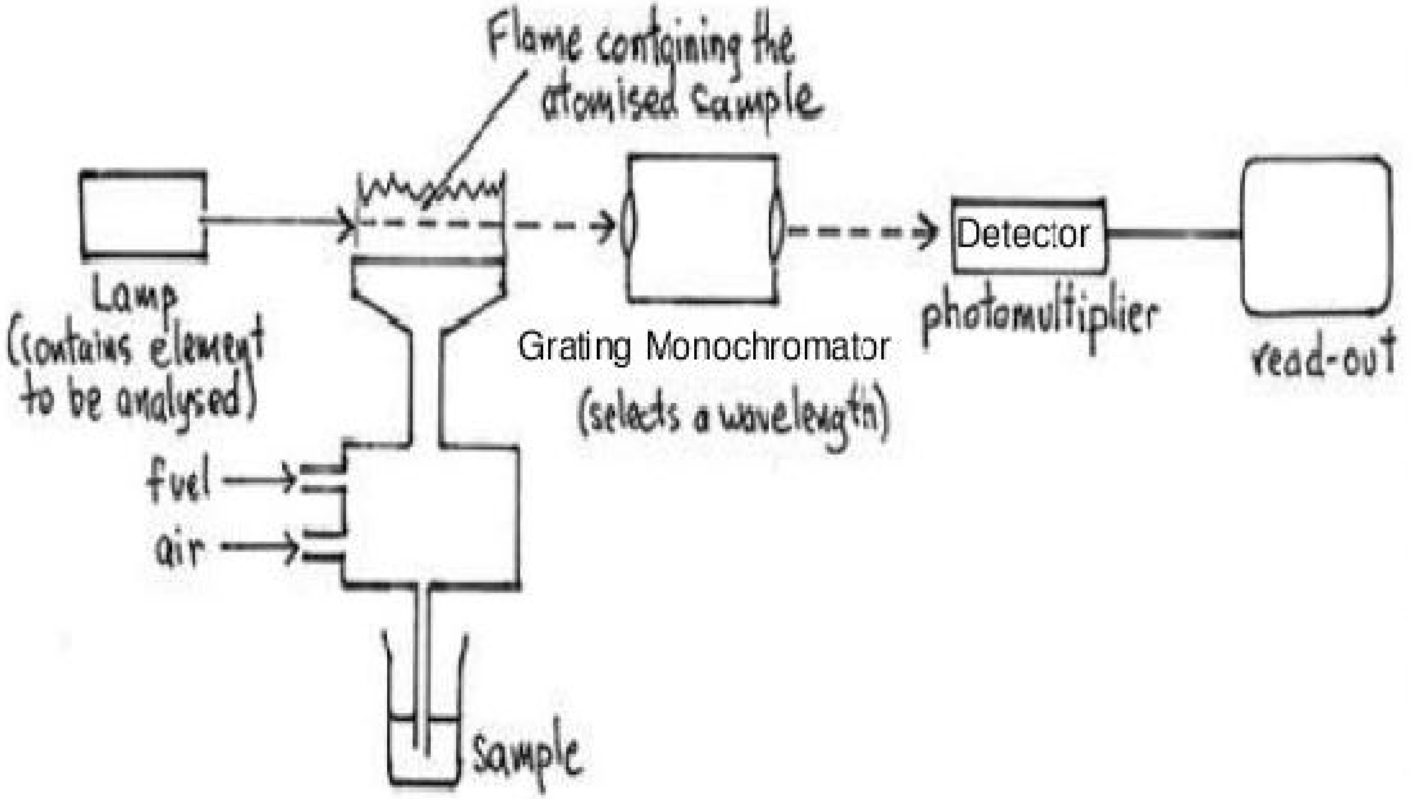


Figure No. 3 Schematic Diagram of Atomic Absorption Spectroscopy

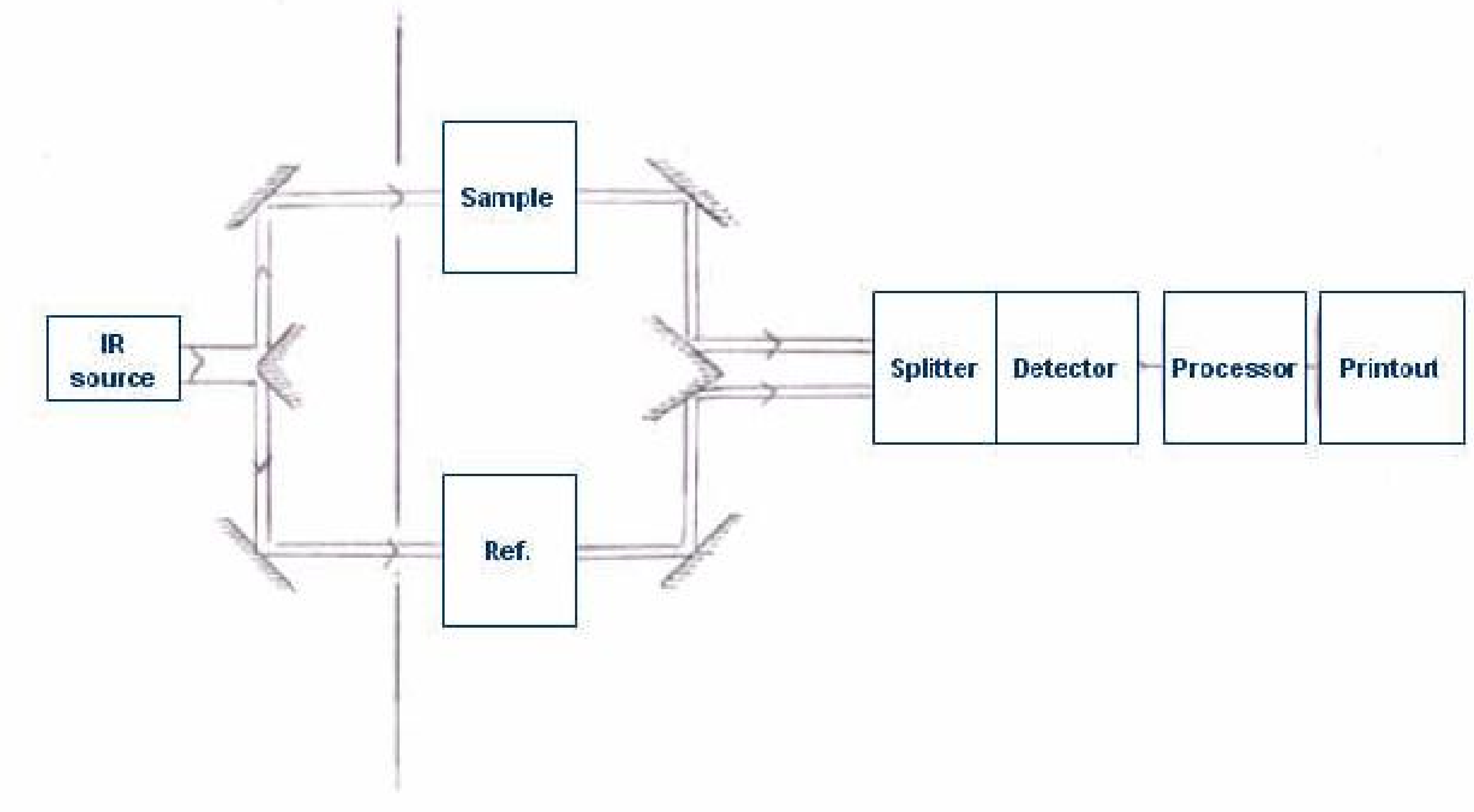


Figure No. 4 Schematic Diagram of Infra-red Spectroscopy

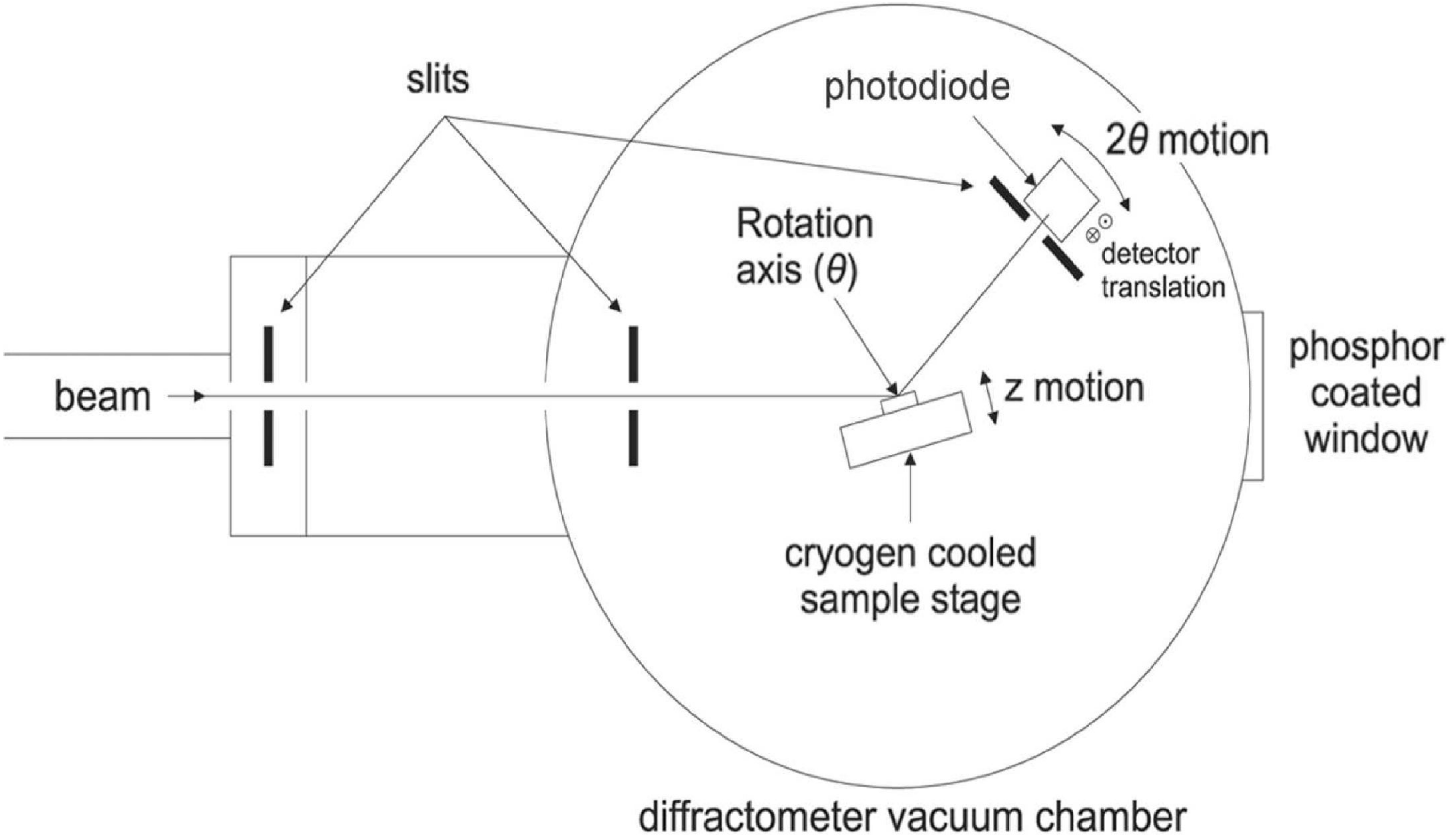


Figure No. 5 Components of D8 Advanced X-ray Diffractometer

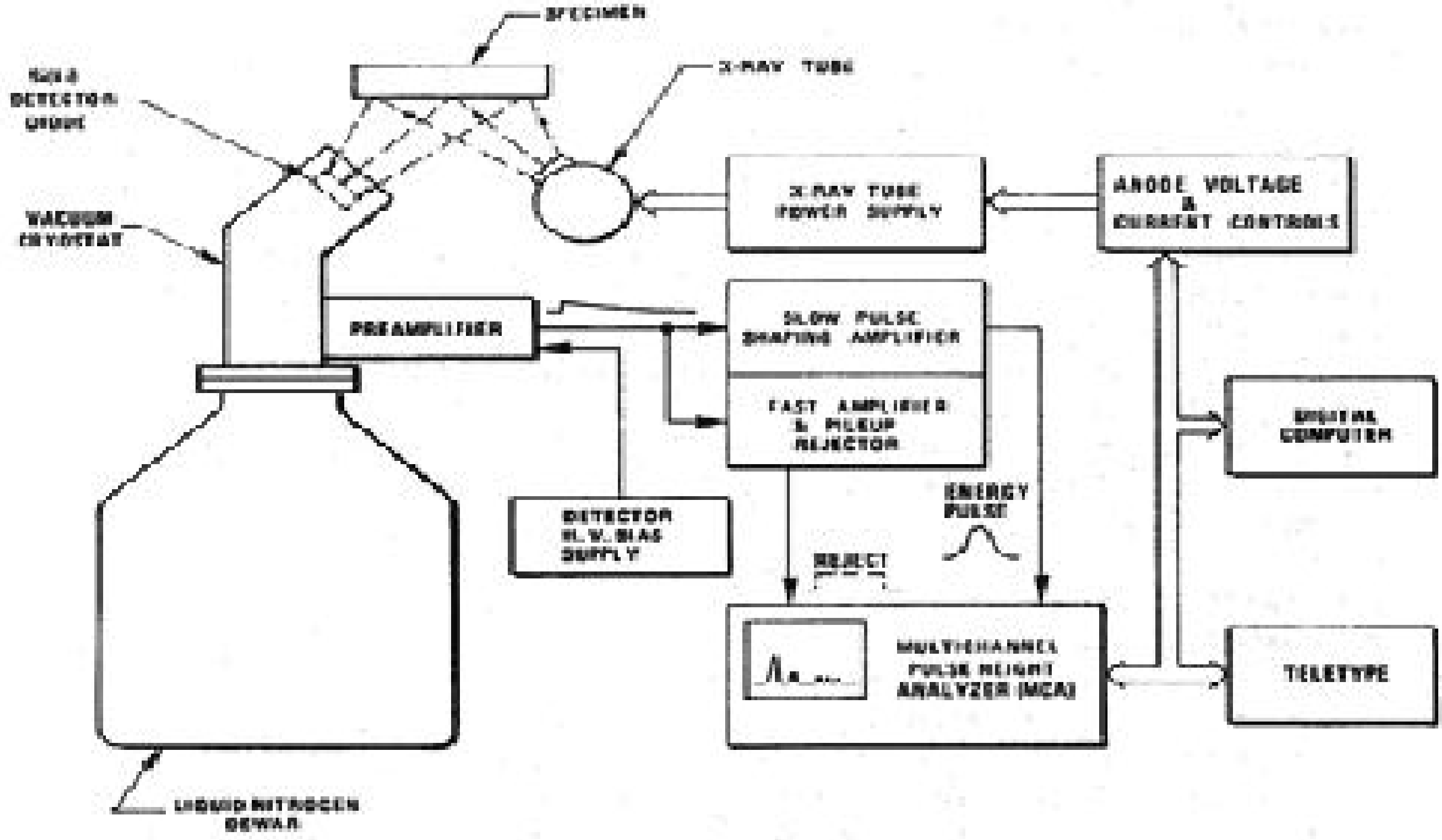


Figure No. 6 Schematic Diagram of Energy Dispersive X-ray Spectroscopy

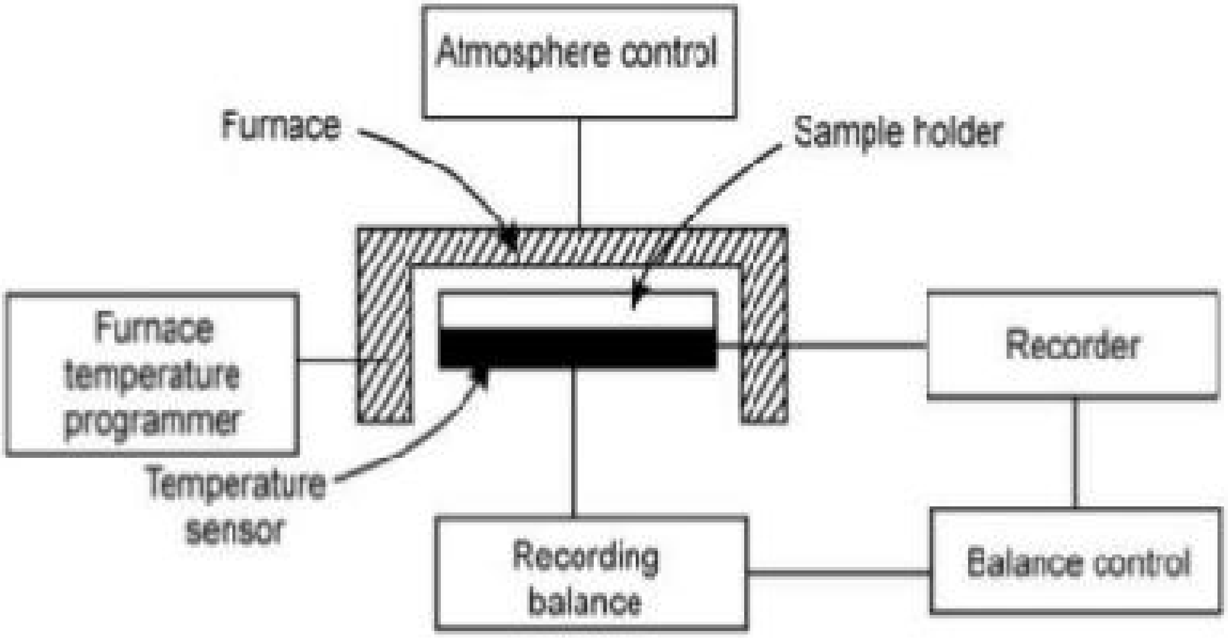


Figure No. 7 Thermogravimetric Analysis instrumentation/ Block Diagram of TGA

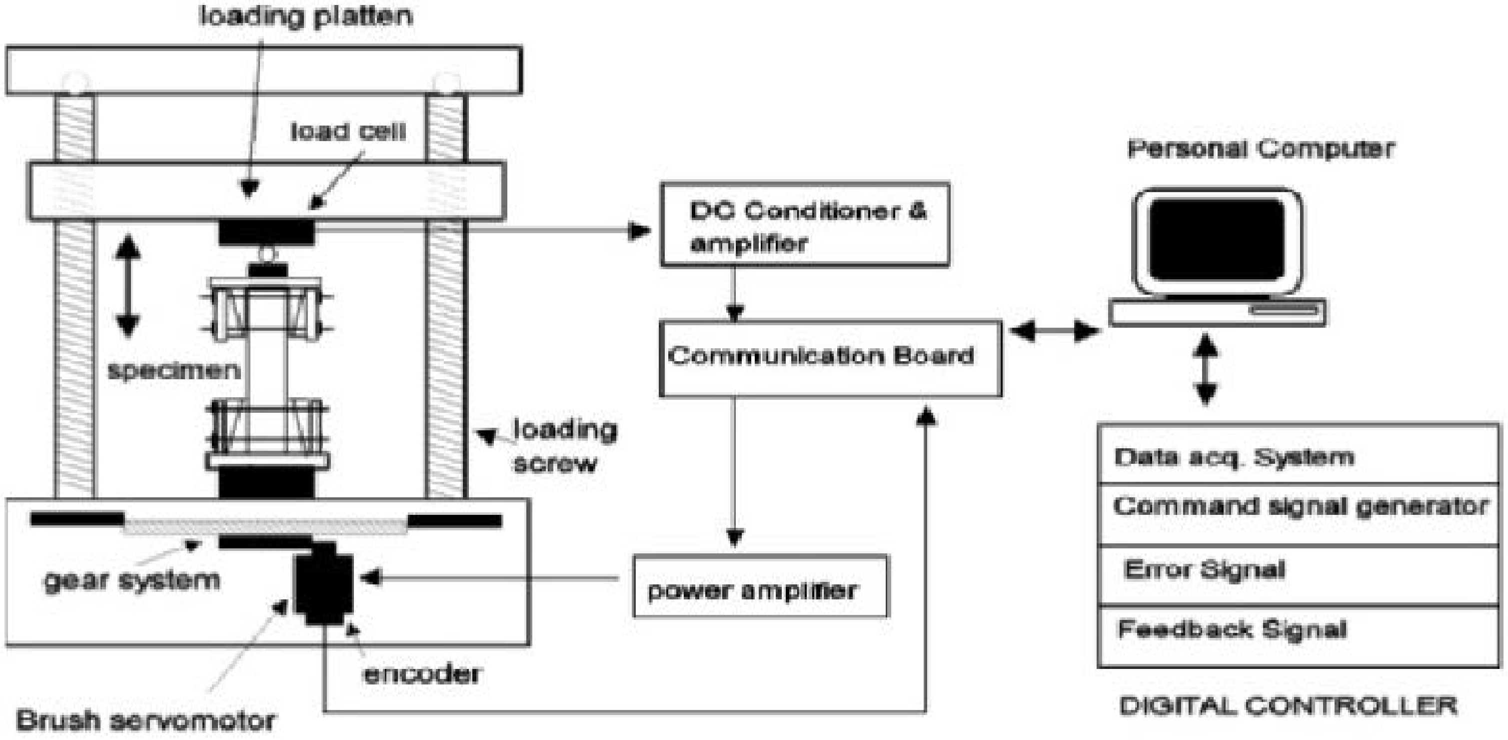
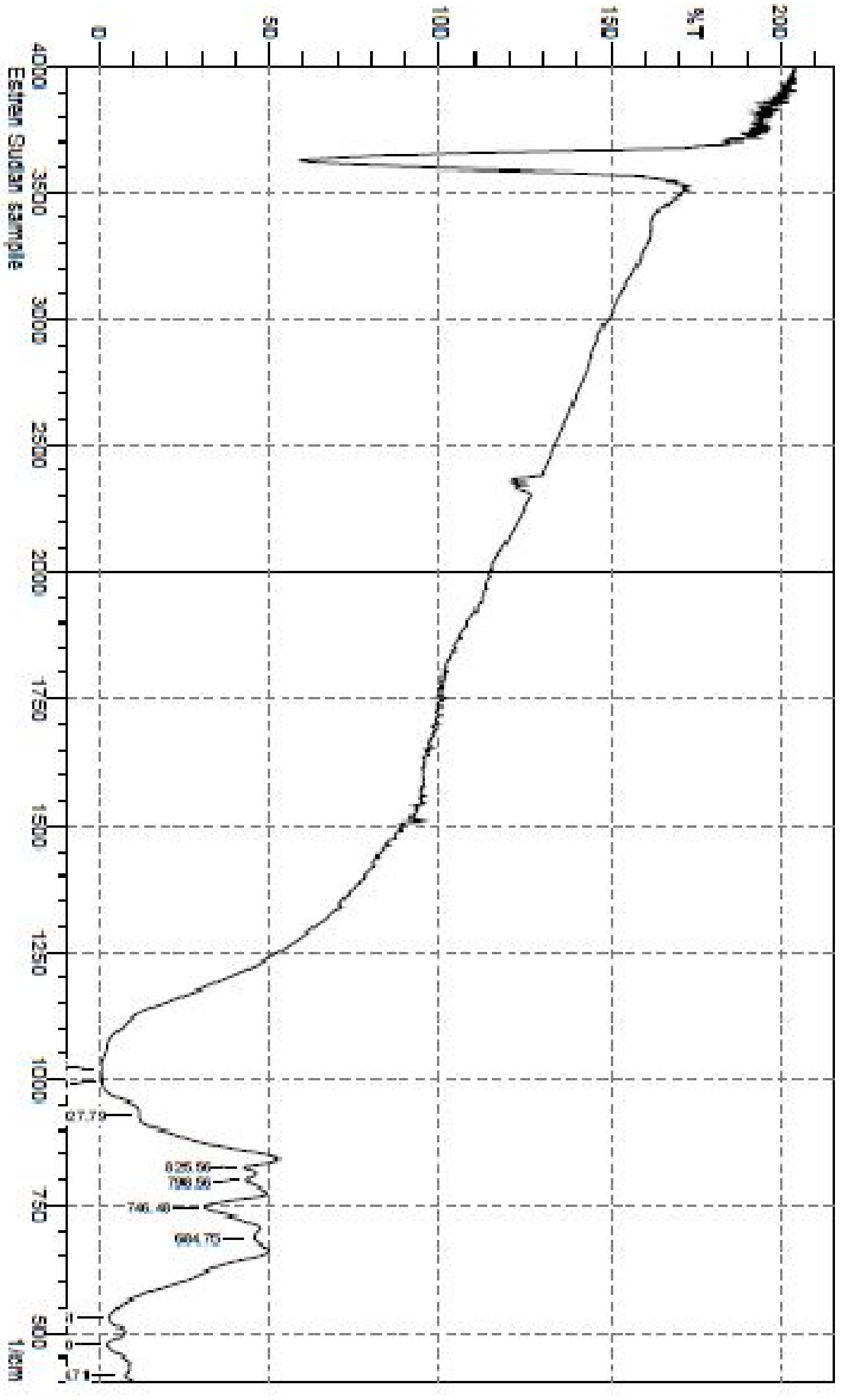
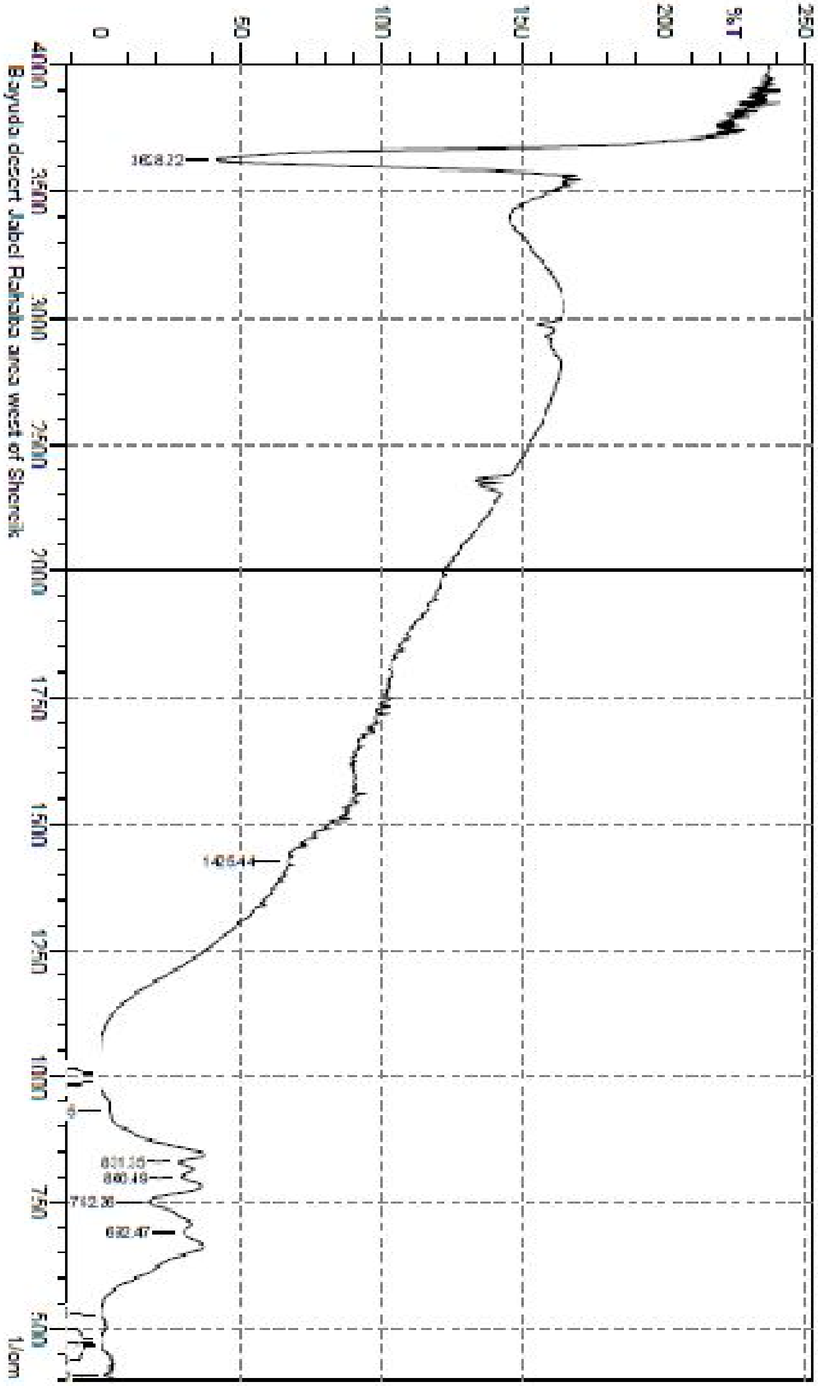


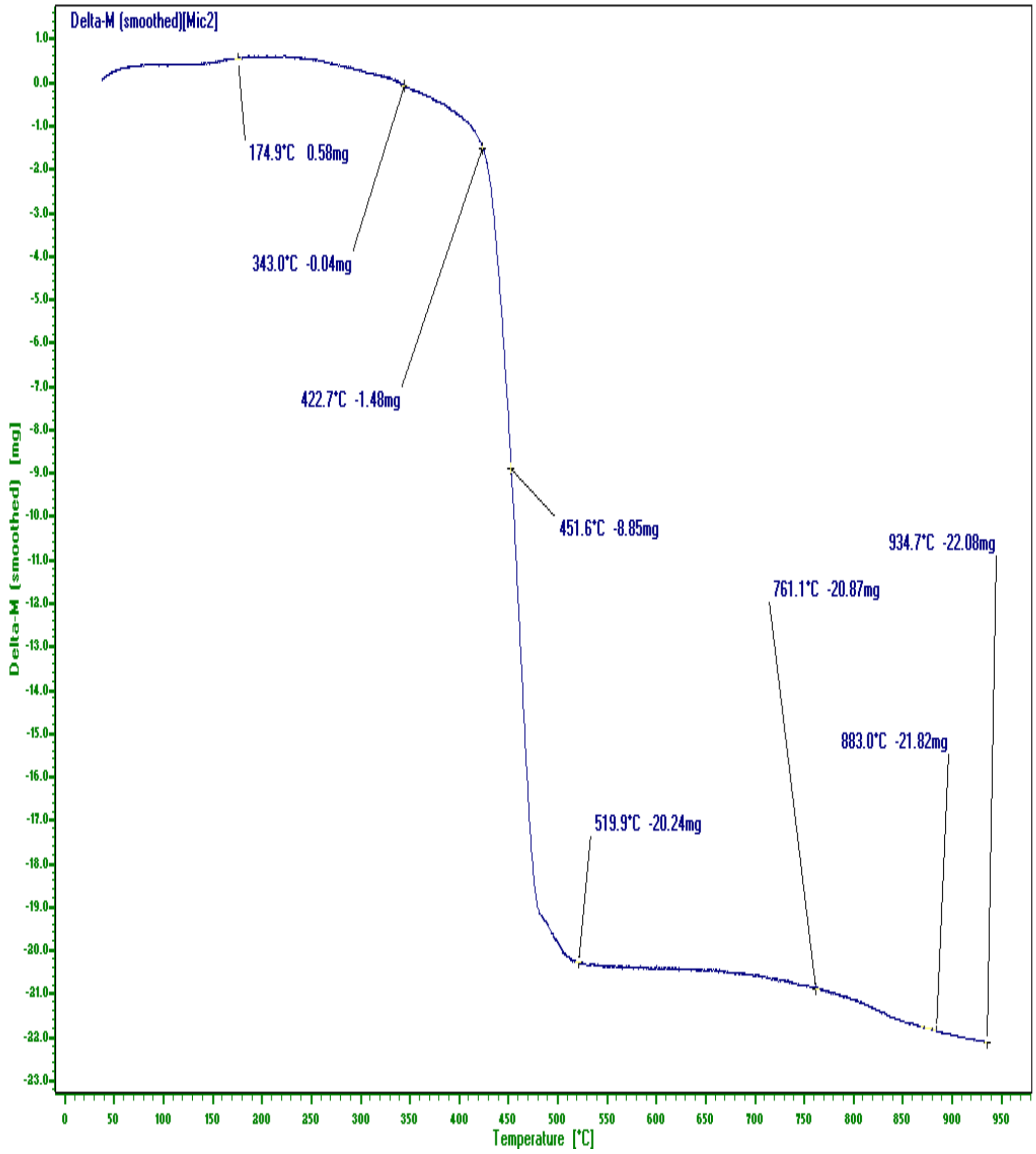
Figure No. 8 Schematic Diagram of Tensile Strength Tester



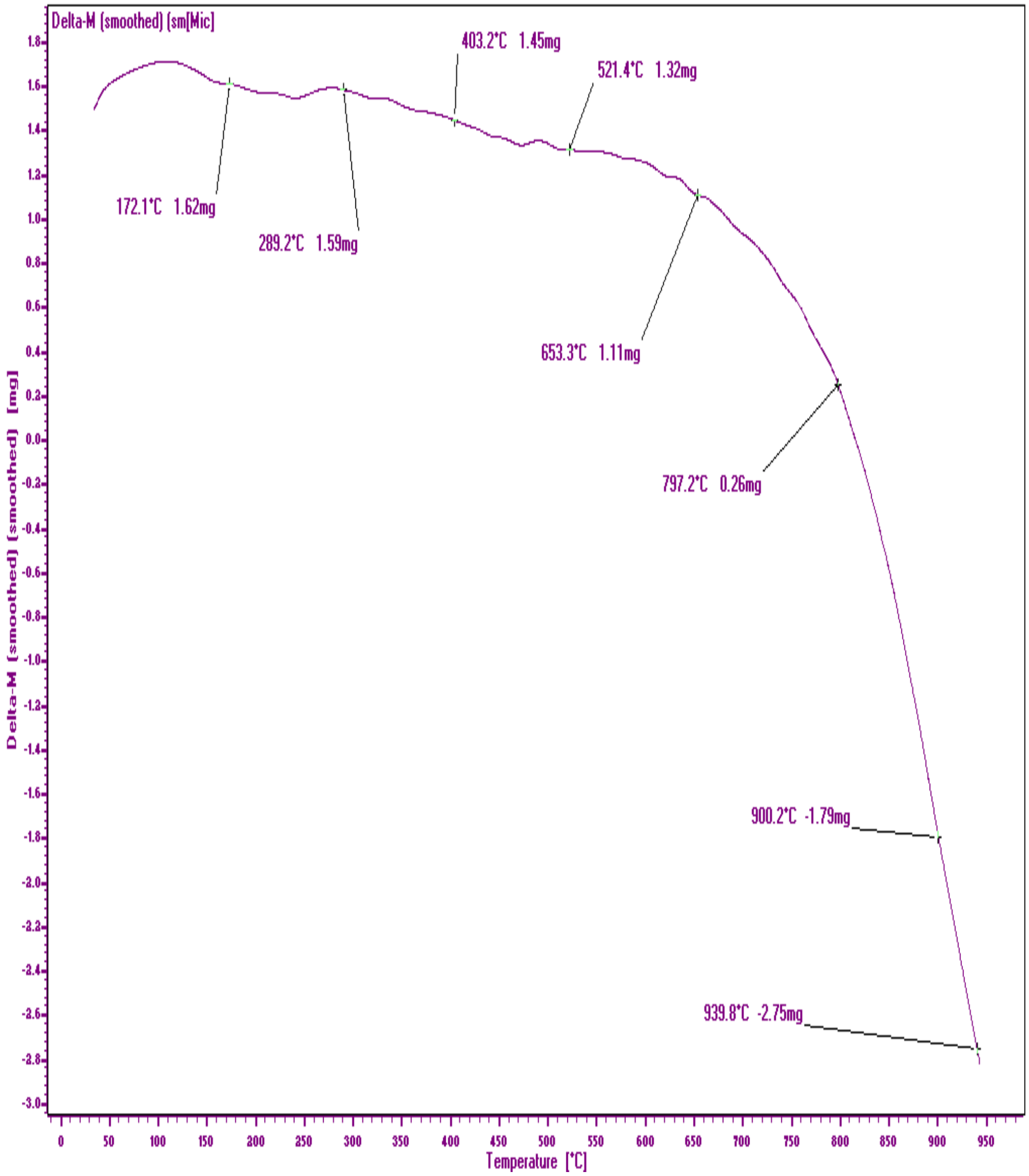
Appendix. 1 Infra–red Spectra of Mica sample (1)



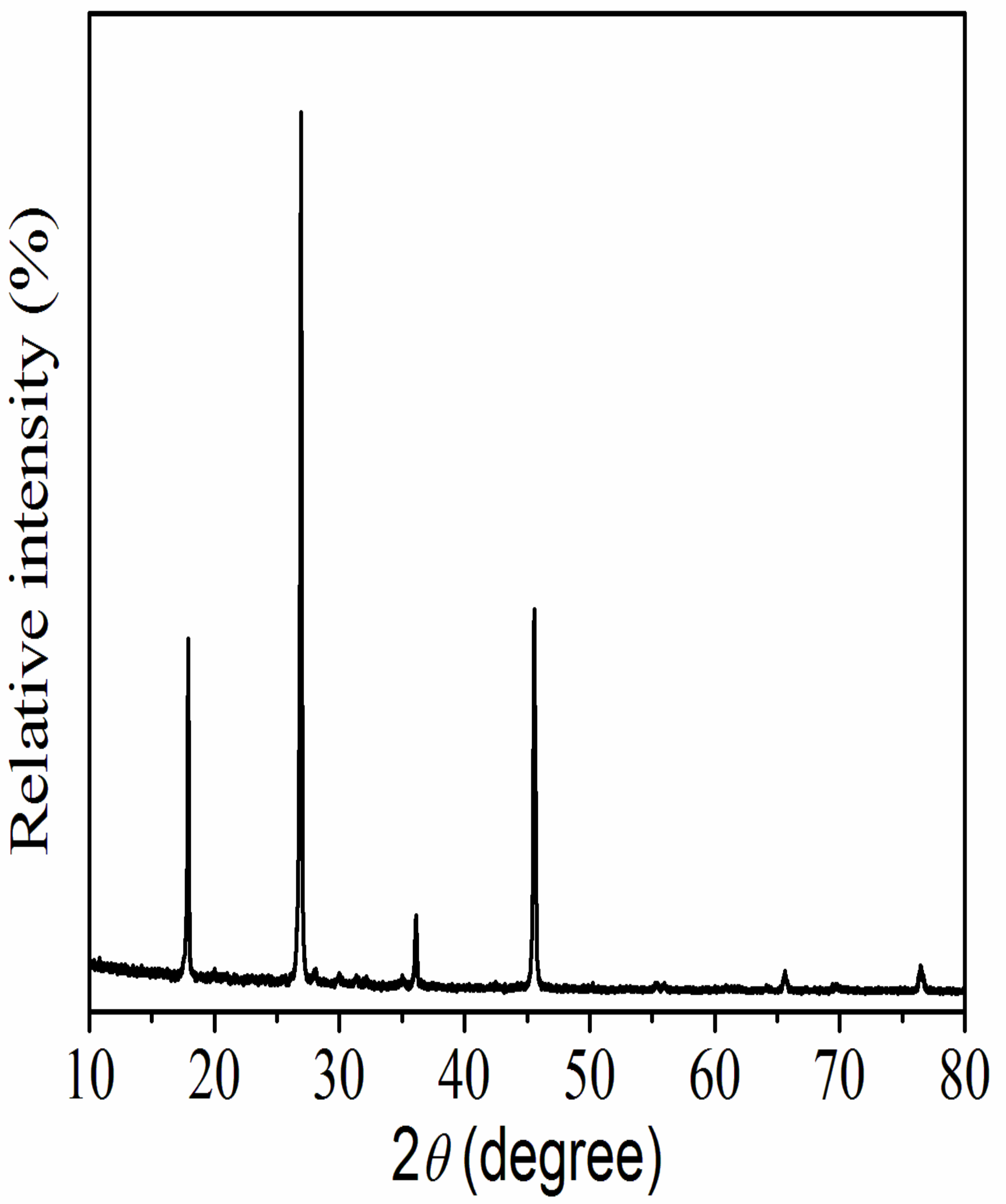
Appendix. 2 Infra–red Spectra of Mica sample (2)



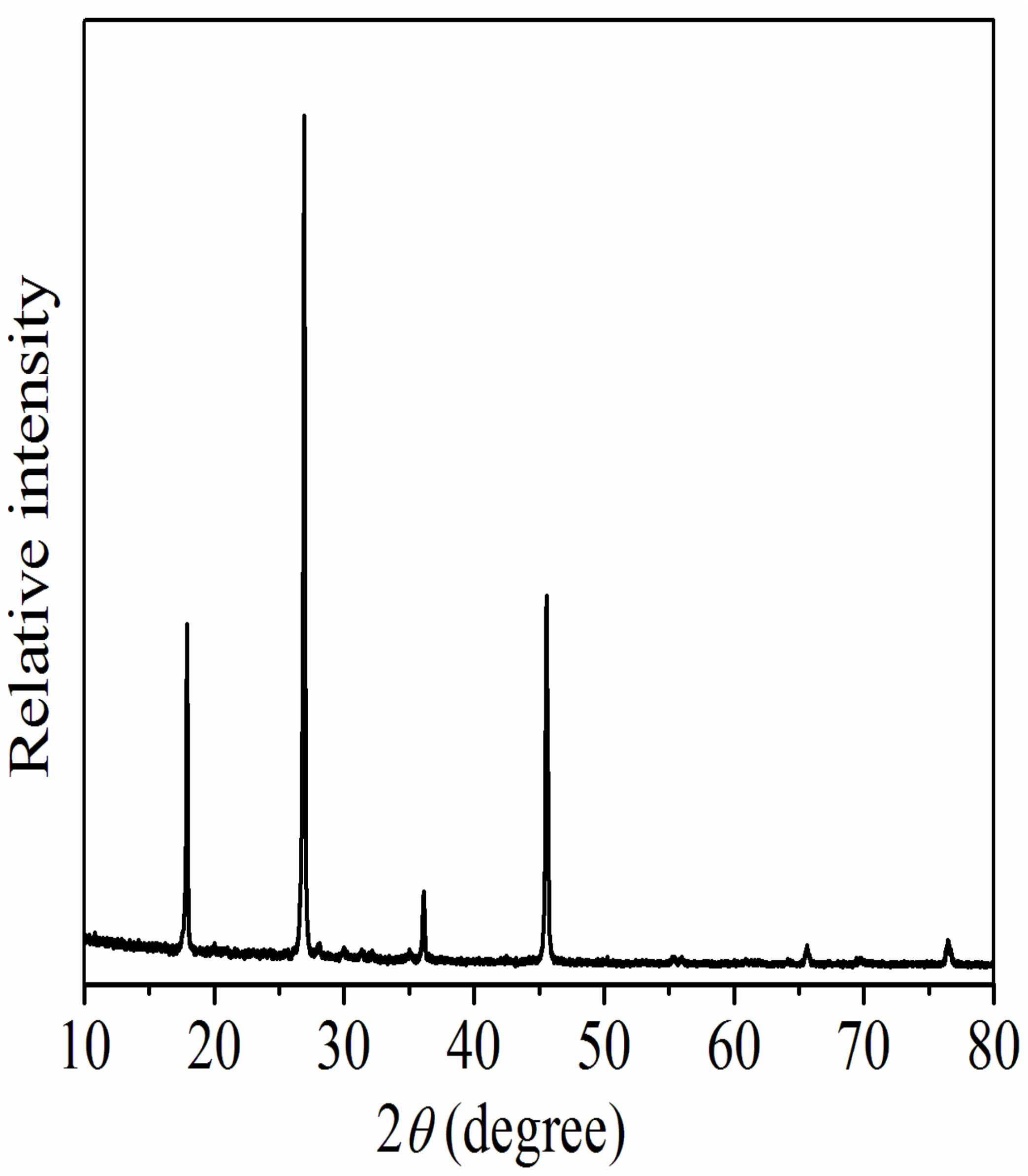
Appendix. 3 Thermogravimetric of Mica sample (1)



Appendix. 4 Thermogravimetric of Mica sample (2)



Appendix. 5 XRD Spectra of Mica sample (1)



Appendix. 6 XRD Spectra of Mica sample (2)

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