



كلية الدراسات العليا

Sudan University of Science and Technology
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A Comparative Analysis of Mica Samples in Shereik Area

دراسة مقارنة لعينات المايكا بمنطقة الشريك

**A Thesis Submitted in Partial Fulfillment for the Requirements
of M.Sc. in chemistry**

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Dedication

This work is dedicated to my parents, brother, sisters, friends, and everyone who helped me.

Acknowledgements

Firstly thanks to Allah Almighty for given me the strength to complete this work, special and great thanks to my supervisor Dr. Elmugdad Ahmed for the support and follow up during all stage of this research.

My thanks are also extended to all the academic teacher of the chemistry department and all workers in the faculty of science (SUST).

Abstract

This work was directed toward the study and analysis of mica in Shereik area which in River Nile State (Sudan), it is Muscovite and occur in pegmatite which is the main source rock. The goal is to determine the content of this kind of mica by several methods (classical analysis, flame photometer, EDX and SEM) and compare them.

The flakes of mica were taken from the west side of river Nile near Algareeb village in Shereik area, were crushed until became homogeneous powder, were measured by above mentioned methods and the results by classical analysis were : moisture 4.10%, organic compounds (loss of ignition) 2.41%, SiO₂ 44.57%, Al₂O₃ 36.21%, CaO 0%, MgO 0% , Fe₂O₃0.7%. By flame photometer were : Na 0%, K 11.9%. By EDX were: K 35.814%, Si 30.489%, Al 19.115%, Fe 12.543%, Ti 0.656%, Rb 0.597%, Cl 0.324%, Zn 0.147%, Mn 0.141%. By SEM were: O 45.6%, Si 20.4%, Al 18.2%, K 9.4%, In 3.7%, Fe 2.6%.

The study was Compared these types of analysis and arrived that: the EDX & SEM is more accurate in measurement of elements (especially gases) than classical & flame photometer method, but EDX & SEM are subject to prior contamination problem and they are expensive. SEM is more accurate than EDX because it uses nanotechnology, but when scanning big area by SEM the scanning will take several time than small area.

Generally the study recommended to use EDX & SEM in analysis because they give accurate result and saving time.

The results prove that the analyzed mica samples are of high quality & it could be exported or utilized in different industries.

المستخلص

يقوم هذا العمل بدراسة وتحليل المايكا في منطقة الشريك بولاية نهر النيل (السودان), هذه المايكا من نوع المسكوفاييت وتوجد في صخور البجماتيت كمصدر رئيسي لها. الهدف الرئيسي من البحث هو تحديد مكونات هذه المايكا بعدة طرق تحليل والمقارنة بينها (تحليل تقليدي, تحليل بواسطة جهاز مطياف اللهب, تحليل بواسطة جهاز مطيافية تشتت الطاقة بالأشعة السينية, تحليل بواسطة المجهر الماسح الالكتروني) والمقارنة بينهم .

تم أخذ رقائق المايكا من الضفة الغربية لنهر النيل بالقرب من قرية الغريب بمنطقة الشريك, ثم طحنت حتى أصبحت بكرة متجانسة, ثم قيست بواسطة الطرق المذكورة أعلاه وكانت النتائج بالنسبة للتحليل التقليدي كالآتي: الرطوبة 4.10%, المواد العضوية 2.41%, السيليكات 44.57%, أكسيد الألمونيوم 36.21%, أكسيد الكالسيوم 0%, أكسيد الماغنسيوم 0%, أكسيد الحديد 0.7%. بالنسبة للتحليل بواسطة جهاز مطياف اللهب كالآتي: البوتاسيوم 11.9%, الصوديوم 0%. بالنسبة للتحليل بواسطة جهاز مطيافية تشتت الطاقة بالأشعة السينية كالآتي: البوتاسيوم 35.814%, السيليكون 30.489%, الألمونيوم 19.115%, الحديد 12.543%, التيتانيوم 0.656%, الربيديوم 0.597%, الكلور 0.324%, الزنك 0.147%, المنجنيز 0.141%. بالنسبة للتحليل بواسطة المجهر الماسح الالكتروني كالآتي: الأكسجين 45.6%, السيليكون 20.4%, الألمونيوم 18.2%, البوتاسيوم 9.4%, الانديوم 3.7%, الحديد 2.6%.

في هذه الدراسة تم المقارنة بين هذه الطرق وتم التوصل للآتي: جهاز مطيافية تشتت الطاقة بالأشعة السينية وجهاز المجهر الماسح الالكتروني أكثر دقة في قياس العناصر (خصوصا الغازات) من التحليل التقليدي و جهاز مطياف اللهب لكن جهاز مطيافية تشتت الطاقة بالأشعة السينية وجهاز المجهر الماسح الالكتروني لديهما مشكلة وهي تلوث في غرفة العينة والتحليل بهما مكلف جدا. جهاز المجهر الماسح الالكتروني أكثر دقة من جهاز مطيافية تشتت الطاقة بالأشعة السينية لأنه يستخدم تقنية النانو لكن عند القياس بواسطة جهاز المجهر الماسح الالكتروني مسح مساحة كبيرة من العينة يأخذ زمن أطول في القياس عنه عند القياس للمساحة الصغيرة.

عموما الدراسة توصي بإستخدام جهاز مطيافية تشتت الطاقة بالأشعة السينية وجهاز المجهر الماسح الالكتروني عند التحليل لأنهما يتميزان بالدقة ويوفران الجهد والوقت.

النتائج أثبتت أن عينات المايكا المحللة ذات جودة عالية ويمكن استخدامها في الصناعات المختلفة.

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List of abbreviations

1. EDX= Energy-dispersive X-ray spectroscopy
2. SEM= scanning electron microscope
3. Mohs = Unit of mineral hardness.
4. Ohm = Unit of measures electrical resistance.
5. KV= Kilovolt.
6. °C = Centigrade.
7. °F = Fahrenheit .
8. Kg = Kilogram.
9. g = gram.
- 10.cm = Centimeter.
- 11.mm = Millimeter.
- 12.sec= Second.
- 13.cal= Calorie.
- 14.USSR= Union of Soviet Socialist Republics.
- 15.U.N.D.P. = United Nations Development Programme.

Chapter one

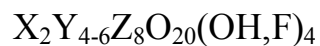
Introduction and literature review

1. Introduction and literature review:

The term ‘mica’ does not relate to a particular mineral, but to a group or family of minerals of similar chemical composition and to some extent similar physical properties. These minerals are predominantly potassium aluminium silicates with varying amounts of magnesium, iron and lithium. They have an internal structures of the layered lattice type in which the silicon atoms are in the centre of a tetrahedral grouping of oxygen atoms. (Komov I.L. et al. 1994)

The mica layers are bounded by oppositely charged surface, with positively charged alkali ions linking the negatively charged silicon-oxygen layers. They are easily identified because of their crystal shape, elasticity, and near-perfect basal cleavage; and, because of their molecular structure, they can be readily spilt into thin films.

The general chemical formula of mica is: (Jessica et al. 2006)



Where:

X= mainly K, Na, Ba, Ca, Cs, $(H_3O)^+$, $(NH_4)^+$.

Y= mainly Al, Mg, Fe^{2+} , Li, Cr, Mn, V, Zn.

Z= mainly Si, Al, Fe^{3+} , Be.

1.1 History:

The Romans were properly responsible for the name, from the Latin word ‘*micare*’ meaning to shine or glitter. (Robert 2009)

Mica is mineral that has rich history of industrial applications. Dating back to early as 2000BC, the ancient Hindus believed mica was a mystical medical cure. (Jessica et al. 2006)

1.2 Geological aspects of mica:

Mica is the natural resource and occurs in nature in a specific type of rock known as pegmatite. Therefore certain characteristics of the geological aspects of mica need to be elucidated. Mica is a mineral which crystallises (potassium and aluminum rich environments) or in the rock as a crude form. (Sanjay 1993)

1.3 Classification:

1.3.1 On the basis of major element different varieties of mica is classified as: (Sanjay 1993)

- i. Muscovite (potash mica or white mica)
- ii. Phlogopite (magnesium or amber mica)
- iii. Biotite
- iv. Lepidolite
- v. fuchsite
- vi. Rascolite
- vii. fluorphlogopi
- viii. paragonite

1.3.2 On the marked forms: (Jessica et al. 2006)

- i. Sheet mica refers to books of mica mined from pegmatite or loosely consolidated clay deposits. The books can be readily split into thin sheets with specific thickness ranging from 0.003 to 0.10 mm.
- ii. Built-up mica was developed in the late 1800s because of the scarcity and cost of sheet mica, and served as ready substitute for electrical parts. Formed by layering pieces of mica splitting and binding them with inorganic or organic binders, the constituted sheets are pressed under high temperature.
- iii. Reconstituted mica is mica paper matted from thin delaminated flakes of scrap mica. The mat is usually impregnated with an organic binder and dried at 110°C.
- iv. Flakes mica is finer mica recovered from scrap or trimmings produced during the processing of sheet mica, or can be beneficiated mica recovered from feldspar, clay and sand operations.
- v. Ground mica is produced by wet or dry grinding and sizing of scrap or flakes mica. It is by far the largest use of mica, both quantitatively and by application.
- vi. Synthetic mica is produced by crystal growth in slowly cooled melts of various chemical oxides. Cooling periods at controlled viscosities dictate the size of crystals; extended cooling times result in larger crystals.

1.3.3 On The structure: Classification mica is based on their octahedral layers: (Lecture Notes)

- i. Trioctahedral layers: in this type every oxygen is bonded to three cations, typically a divalent cation like Mg^{2+} or Fe^{2+} .
- ii. Dioctahedral layers: in this type every oxygen is bonded to two trivalent cations, generally Al^{3+} .

In either case the each oxygen needs additional bonds that provide an additional electrostatic bond strength of one.

1.4 Nature and Properties of mica:

Mica group comprises a number of mica-minerals broadly classified under phyllosilicates of different major elements such as potassium, aluminium, magnesium, iron, etc. The characteristic property of such mineral is that they can be split into thin lamellae without any distortion. This property is due to excellent basal cleavage and a high degree of flexibility, elasticity and toughness of extremely thin cleavable flakes. Most of the members of mica group crystallized in the monoclinic system of crystallography. Mica is virtually un-affected by water, heat, acids, alkalies or electricity. Of many economic minerals occurring in nature, mica is one of the very strategic minerals. On account of its excellent insulating properties and high heat resisting capacity, it is indispensable in electrical industries. On the basis aforesaid major elements, different varieties of mica produced showing different characteristics. (Sanjay 1993)

1.4.1 Chemical composition of mica:

- i. Muscovite (hydrated silicate of potassium aluminium).
- ii. Phlogopite (hydrated silicate of potassium aluminium-magnesium with fluorine).
- iii. Biotite (hydrated silicate of potassium, magnesium, iron and aluminium).
- iv. Lepidolite (hydrated silicate of potassium, lithium and aluminium with fluorine).

- v. Fuchsite (hydrated silicate of potassium chromium and aluminium).
- vi. Rascolite (hydrated silicate of potassium vanadium and aluminium).
- vii. Flurophlogopite (silicate with fluorine, potassium magnesium and aluminium).
- viii. Paragonite (hydrated silicate of sodium and aluminium).

Of these, muscovite (potash/ mica or white mica) and Phlogopite (magnesium or amber mica) are important from economic point of view. Muscovite mica contains consistent and high dielectric properties, high heat resistance and it has a low thermal conductivity. Further, it can be cut easily into thin sheets, which are elastic, flexible, strong and transparent. (Sanjay 1993)

Table (1): End-member Chemical compositions of rock-forming micas: (Mica Mineral 2013)

No	Type	Chemical composition
1	Biotite	$K(Mg, Fe^{2+})_3(Al, Fe^{3+})Si_3O_{10}(OH, F)_2$
2	Phlogopite	$KMg_3Si_3AlO_{10}(F, OH)_2$
3	Lepidolite	$K(Li, Al)_3(Si, Al)_4O_{10}(F, OH)_2$
4	Muscovite	$K Al_2 (Si_3, Al) O_{10} (OH, F)_2$
5	Paragonite	$Na Al_2 (Si_3, Al) O_{10}(OH)_2$
6	Glauconite	$(K, Na)(Fe^{3+}, Al, Mg)_2(Si, Al)_4O_{10}(OH)_2$

The types of mica (1,2,3) are trioctahedral- i.e, they have three atoms in the Y position, and The types of mica (4,5,6) are dioctahedral- i.e, they have two atoms in the Y position.

1.4.2 Important properties of muscovite sheet mica: (General Description of Muscovite Mica)

Table (2): Physical properties of muscovite sheet mica:

Parameter	Unit	Number
Hardness	Mohs	2.8 – 3.2
Density	g/cm ³	2.6 – 3.2
Tensile strength	kg/cm ²	~1750
Shear strength	kg/cm ²	2200 – 2700
Compression strength	kg/cm ²	1900 – 2850
Modulus of Elasticity	g/cm ²	1400 – 2100
Specific Heat	—————	0.21
Expansion Coefficient per °C perpendicular to cleavage plane	—————	9x 10 ⁻⁶ – 36 x 10 ⁻⁶
Maximum operating temperature	°C	500 – 600
Thermal conductivity perpendicular	g.cal/sec/cm ² /°C/cm	0.13x 10 ⁻³
Thermal conductivity parallel	g.cal/sec/cm ² /°C/cm	0.31
Dielectric strength@20 °C	KV/mm	3 – 5
Apparent electric strength	KV/mm	120 – 200
Volume resistivity @ 25 °C	Ohm.cm	4 x10 ⁻¹⁵ – 2x10 ⁻¹⁷
Power factor (loss tangent) @ 15 °C	—————	0.0001 – 0.0004
Optical angle	degree	50 – 75
Refractive index	—————	1.56 – 1.61
Permittivity @15 °C		6 – 7
Calcining temperature	°C	700 – 800

1.4.3 Mineralogical description of some mica:

i. Muscovite:

The name muscovite is derived from *Muscovy glass*, which was the name used for this mineral when first described from the Russian province of Muscovy. (M. E. Fleet 2003)

This mineral crystallizes in monoclinic system. Prismatic class-2/m crystals are usually tabular and possess a rhombic or hexagonal form. Tapering pyramidal habits are also observed. Crystals are often large and tough, measuring at times several feet in diameter.

Muscovite mica has a high perfect basal cleavage. It permits very thin, transparent and elastic leaves to be split. It has hardness of 2 to 3, specific gravity 2.8 to 3.1. It is colorless or yellowish, redish or brownish. It has transparent to translucent and pearly to vitreous lustre properties.

Muscovite frequently contains small amount of calcium, lithium, iron, magnesium and sodium. It occurs in granites, syenites and especially in pegmatites which is the main source rock, especially where pneumatolytic action has been effective. (Sanjay 1993)

ii. Phlogopite:

This mineral crystallises in monoclinic system. It has prismatic class-2/m as studied according to crystallographical methods. The crystals usually resemble those of biotite in form and habit and sometimes large and coarse. They may be hexagonal or rhombic in form, commonly found in disseminated scales, plates or aggregates.

It has a high perfect basal cleavage. Thin laminae are tough and elastic. Its specific gravity 2.8 to 3.2 and it has pearly to submetallic lustre. The indices of refraction vary from 1.540 to 1.606(-). Thin leaves are transparent which often show asterism.

Phlogopite usually contains small amount of iron and sodium. It whitens and fuses on thin edges when heated strongly. It is slightly affected by hydrochloric acid but really decomposed by hot sulphuric acid. It occurs in crystalline magnesium limestones, dolomites, schists, serpentine periodolite and laucite basalt. (Sanjay 1993)

iii. Biotite:

This mineral crystallises in monoclinic system. Prismatic class-2/m crystals are usually tabular with a hexagonal or rhombic habit, sometimes striated horizontally. Crystals are rare. It is generally found in plates, lamellae, masses or disseminated scales.

It has a highly perfect basal cleavage. Hardness according to Moh's scales 2.5 to 3.0 Specific gravity is 2.7 to 3.2. It is usually dark brown or black in colour but at times it is also light brown or greenish. The indices of refraction vary generally from 1.541 to 1.680(-). The streak of the mineral is white or greenish with transparent to opaque. It sometimes shows asterism.

Biotite is important constituent of many intrusive igneous and metamorphic rocks, such as granite, syenite, diorite, gnesis and mica schist. It is often associated with muscovite. Biotite is of little commercial use. (Sanjay 1993)

iv. Lepidolite:

This mineral crystallises in monoclinic system. Prismatic class-2/m crystals are short prismatic but very rare. It is usually available in scaly, granular masses, often resembling granular limestone, and in tabular, cleavable plates.

It has perfect basal cleavage. Hardness 2 to 4, Specific gravity 2.8 to 2.9. It is generally rose red in color, but sometimes it also white, gray, greenish or brown, pearly lustre.

Lepidolite occurs commonly in pegmatites, also in granites and gneiss. It is usually the result of pneumatolytic action. The common associates are tourmaline, spodumene, amblygonite, cassiterite, muscovite, albite and topaz. It is important source of lithium compounds. (Sanjay 1993)

1.5 Uses of mica:

Over the centuries, mica uses have evolved from decorative ornaments, mirrors, stove windows, and shock-resistant material in early military applications to its more high-tech applications to day in construction materials, paper, plastic, rubber, paints, cement, wallpaper, cosmetics, inks, surface coating, Electronics(as a capacitor in condensers or as cables) and others. (eds Jessica et al. 2006)

Mica sheets are used to provide structure for heating wire (such as in Kanthal or Nichrome) in heating elements and can withstand up to 900°C (1,650°F). (Metal uses mica)

Another use of mica is in the production of ultraflat, thin film surfaces (e.g. gold surfaces) using mica as substrate. Although the deposited film surface is still rough due to deposition kinetics, the back side of the film at mica film interface provides ultraflatness, when the film is removed from the substrate. Such ultraflat substrates are common substrates for sample preparation for the atomic force microscopy. Freshly cleaved mica surfaces have been used as clean imaging substrates in atomic force microscopy, enabling for example the imaging of bismuth films, plasma glycoproteins, membrane bilayers, and DNA molecules. (Metal uses mica)

1.6 Occurrence and production:

The United States is the leading supplier of mica, with about 46% of the world's production. The US produces only ground muscovite. The only significant production of phlogopite occur in a crystalline limestone, dolomites and serpentines in Canada, Finland and Madagascar which together account for about 15% of total worldwide mica output. India produces 80% of the world's sheet mica. In the US fully half the mica produced is used in wallboard joint cement. (Peter 1996) (Greg, Edward & Hussein 2004)

Muscovite and biotite occur chiefly in pegmatite dikes associated with feldspar and quartz although they are abundant also in granites and syenites. They are chiefly obtained in India, Brazil and United States, although, for electrical uses, 70% of world supply comes from the first two countries. (Greg, Edward & Hussein 2004)

Table (3): World production of Muscovite: (J.J. SCHOEMAN 1989)

Country	Production t	World		M.E.*	
		%	Rank	%	Rank
USA	125000	57,1	1	74,2	1
Canada	12000 10100	5,5	3	7,1	2
France	8000	4,6	4	6,0	3
India	4000	3,8	5	4,7	4
Norway	2200	1,8	6	2,4	5
RSA	7200	1,0	7	1,3	6
Other M.E. †		3,3		4,3	
Total M.E.	168500	77,1		100,0	
USSR	50000	22,9	2		
Total world	218000	100,0			

Source: BGS World Mineral Statistics, 1981-85.

M.E. * denotes mica-exporting countries.

M.E.† Spain, Yugoslavia, Madagascar, Morocco, Mozambique, Sudan, Zimbabwe, Mexico, Argentina, Brazil, Sri Lanka and Portugal.

1.7 Mica in Sudan:

1.7.1 Rahaba Series:

The rocks of the Rahaba Series are conformably overlying the Kurmut Series and in parts interfingering with this series. The Rahaba Series occupies almost the overall central and western parts of the Bayuda desert. In the basal parts of the series, biotite and hornblende gneisses predominate

with numerous amphibolites intercalations. Acidic muscovite-bearing quartz feldspar gneisses, with amphibolites intercalations in thin layers, and lenses of calc-silicate garnetiferous quartzitic mica schists and graphitic schist, form the typical rocks of the upper parts of the Rahaba Series in the northeastern and central part of the Bayuda desert. The acidic gneisses contain many pegmatoids, and in parts thin out and are replaced by a monotonous sequence of biotite gneisses with rare intercalations of amphibolites. Anatexis and granitisation up to the stage of autochthonous granite formation are widespread and well developed in this series. The rocks of the Rahaba Series are generally well foliated and intensely folded. The fold axes dip west and southwest in the southern, central and western parts of the Bayuda desert, and southeast and northwest in the southern part of the area [Figure (4)&(5)]. (Ahmed AY, Fathi AK & Abdel gadir E. 1984)

1.7.2 Shereik area:

Shereik is a populated place (class P - Populated Place) in River Nile State, Sudan (Africa) with the region font code of Africa/Middle East. It is located at an elevation of 335m above sea level. Shereik is also known as Ash Shireq, Ash Shurayk, Esh Shereik, Shereik and Shereikh. Its coordinates are 18°48'0" N and 33°34'0" E in DMS (Degrees Minutes Seconds) or 18.8 and 33.5667 (in decimal degrees).

Mica is found at Nahral-Neel State west of Shereik area (near Algarreb Village- Souq Alarbeaa). Its reserve at this area is estimated at around

147378 tons. Around 700 tons of this mineral has been produced until 1971.

Muscovite mica occurs west of the Nile near Shereik in the northern Province [Figure (3), (4) & (5)]. It lies within the Rahaba Series of the Basement Complex (granitic gneisses, hornblende and amphibolite gneisses). The deposit was investigated by Kabesh (1959, 1960, 1962b, 1962d), Geological and Mineral Resources Department, U.N.D.P. and Vail (1971a).

Mica is found as books up to 1/2 m in diameter and associated with quartz, feldspar, tourmaline, aplite and beryl.

The mica is extracted for export by the Sudanese Mining Corporation but complementary detailed study of the geological controls of the mineralization will enhance successful exploitation of the deposit. (Ahmed, Fathi, Abdel gadir 1984)(Ahmed A.A.1998)

1.7.3 Demazin area:

Also mica occurrences were reported southeast of Demazin (11°10' N, 34°25' E) and in the southern Region (4°25' N,31°15' E) [Figure (4)& (5)]. It should be noted that some of the mica symbols on the map are misplaced in the Nubian Sandstone Formation. (Ahmed, Fathi, Abdel gadir 1984)

1.8 Previous studies:

One of the previous studies were done by Mohamoud Lofty Abdel Hamid Kabesh and comparative with Indian mica. The results are found in the table (4):(Kabesh M.L. 1960)

Table (4): Analysis of moscovite in Rubatab (Sudanese mica) area & Bihar state (Indian mica):

No	Compound	Rubatab area (Sudanese mica)	Bihar state(Indian mica)
1	Al ₂ O ₃	35.1%	36.72%
2	Fe ₂ O ₃	2.3%	0.95%
3	TiO ₂	0.4%	—————
4	CaO	—————	0.21%
5	BaO	—————	—————
6	SiO ₂	45.1%	45.57%
7	K ₂ O	9.8%	8.81%
8	Na ₂ O	1.0%	0.62%
9	H ₂ O	4.6%	5.05%

1.9 Analytical Methods:

1.9.1 Classical analysis:

1.9.1.1 Gravimetric analysis:

In this case we separate the component to be determined from a solution in the form of an insoluble compound to know the chemical composition, and then determine the weight of the compound; from this we can calculate the amount of the substance present. (Treadwall 1919)

1.9.1.2 Volumetric analysis:

In this case the constituents are not weighed but they are determined by measuring the amount of reagents of known strength which react with them. (Treadwell 1919)

1.9.2 Flame photometry:

Flame transfers solid or liquid into vapour state and decomposes them to simpler molecules or atoms. The vapours of natural metal atoms or molecules containing atoms are excited by thermal energy of the flame. From the excited levels of atoms, the electrons tend to return to the ground state by emission of radiation. A particular element would give a characteristic spectrum of its own. The electron return to the ground state in stages leading to generation of several spectral lines. Though energy level diagrams can be constructed for all atoms, those for mono- and diatomic molecules like Na, K, Ca etc. are relatively simpler. In such case the most outstanding line is due to transition of electrons between the lowest excited state and ground state. For, alkali metal Li, like Na, K and Ca such transitions occur at 671 nm, 589 nm, 76605 nm and 423 nm respectively. It is possible to regulate the energy level transitions by controlling the temperature of the flame. However, the intensities of spectral lines are dependent on the concentration of the element.

The essential parts of the instrument are pressure regulator and flow meter, atomizer, burner, photosensitive detector and recording output of the detector. The pressure regulator and flow meter are used for proper adjustment of pressure and flow of gasses. The atomizer is used to

introduce liquid sample into the flame at a stable rate. The capillary type atomizer is advantageous as it atomizes at slow rate(4 ml per minute). It draws the solution up through the capillary intake tube from a small beaker. When the beaker is withdrawn, the capillary tube empties instantaneously. The outside of the tube can be wiped clean with a piece of tissue paper. The capillary tube is flushed clean of the previous solution as a new solution is fed. (Rabindra 2012)

1.9.3 EDX analysis:

EDX & EDS make use of the X-ray spectrum emitted by a solid sample bombarded with a focused beam of electrons to obtain a localized chemical analysis. All elements from atomic number 4 (Be) to 92 (U) can be detected in principle, though not all instruments are equipped for 'light' elements ($Z < 10$). Qualitative analysis involves the identification of the lines in the spectrum and is fairly straightforward owing to the simplicity of X-ray spectra. Quantitative analysis (determination of the concentrations of the elements present) entails measuring line intensities for each element in the sample and for the same elements in calibration Standards of known composition. By scanning the beam in a television-like raster and displaying the intensity of a selected X-ray line, element distribution images or 'maps' can be produced. Also, images produced by electrons collected from the sample reveal surface topography or mean atomic number differences according to the mode selected. The scanning electron microscope (SEM), which is closely related to the electron probe, is designed primarily for producing electron images, but can also be used for element mapping, and even point analysis, if an X-ray spectrometer is

added. There is thus a considerable overlap in the functions of these instruments. (Introduction to Energy Dispersive X-ray Spectrometry)

1.9.4 SEM analysis:

Scanning electron microscopy is used for inspecting topographies of specimens at very high magnifications using a piece of equipment called the scanning electron microscope. SEM magnifications can go to more than 300,000 X but most semiconductor manufacturing applications require magnifications of less than 3,000 X only. SEM inspection is often used in the analysis of die/package cracks and fracture surfaces, bond failures, and physical defects on the die or package surface. During SEM inspection, a beam of electrons is focused on a spot volume of the specimen, resulting in the transfer of energy to the spot. These bombarding electrons, also referred to as primary electrons, dislodge electrons from the specimen itself. The dislodged electrons, also known as secondary electrons, are attracted and collected by a positively biased grid or detector, and then translated into a signal. To produce the SEM image, the electron beam is swept across the area being inspected, producing many such signals. These signals are then amplified, analyzed, and translated into images of the topography being inspected. Finally, the image is shown at monitor.

A SEM may be equipped with an EDX analysis system to enable it to perform compositional analysis on specimens. EDX analysis is useful in identifying materials and contaminants, as well as estimating their relative concentrations on the surface of the specimen. (Scanning electron microscopy)

1.10 Objectives of the study:

The objectives of this study is:

- To determination the content of mica in Shereik area.
- To do the analysis by using several methods and compare with them.
- Finally to choose the best method of analysis mica.

Chapter two
Materials and Methods

2. Materials and Methods:

2.1 Chemicals:

- Sodium Carbonate (assay 99.5%, India).
- Hydrochloric acid (assay 35-39%, Spain).
- Distilled water (Produce by machine[Bibby Sterilin LTD, England]).
- Oxalic acid (assay 99.5%, India).
- Concentrated ammonia solution (assay 25%, India).
- Saturated disodium hydrogen orthophosphate (assay 99.5%, England).
- Methyl red indicator (England).
- Zinc dust (assay 95%, India).
- Ortho phosphoric acid (assay 85%, England).
- Barium diphenylamine sulfate indicator (Chadwell heath, Essex, England).
- Potassium dichromate (assay 99.9%, England).
- Potassium solution 1000 ppm (w/v of Na in approx. 0.1N HClO₄, The metal content of this solution is within 0.5% of the nominal value).
- Sodium solution 1000 ppm (w/v of K in approx. 0.1N HClO₄, The metal content of this solution is within 0.5% of the nominal value).

2.2 Apparatus:

- Porcelain mortar
- Sensitive balance (Pioneer TM, OHAUS, China).

- Porcelain crucible
- Furnace (CARBOLITE, England).
- Volumetric flask 250ml (Class A BS 1792 in 20°C, England).
- Conical flask 250ml (Pyrex, England).
- Beaker (Pyrex, England).
- Platinum crucible
- Filter paper
- Wash bottle of distilled water (Plastic bottle).
- Moisture instrument
- Oven (BTL, Chadwell heath, Essex, England).
- Flame (Gas burner).
- Pipette (Germany Brand, Silber Brand, ETERNA).
- Burette (Gold line B.S.846).
- Desiccator.
- Funnel (Pyrex, England).
- Spatula.
- Water bath (LabTech, England).
- Fume Hood (OBROMAX).
- Hot plate (Scott Science, UK).
- Flame photometer (Jenway, UK).
- EDX instrument (Shimadzu, Tokyo).
- SEM instrument (Brno – Kohoutovice, Czech Republic).

2.3 Methods:

2.3.1 Sample preparation:

The flakes of mica were crushed in a porcelain mortar until become homogeneous powder.

2.3.2 Determination Loss of ignition :

In a porcelain crucible 1.0013 g of mica was weighted and was heated in a furnace at 512-1000°C. The crucible was put in furnace at 1000°C for 30 minutes, was cooled, dried, and weighted.



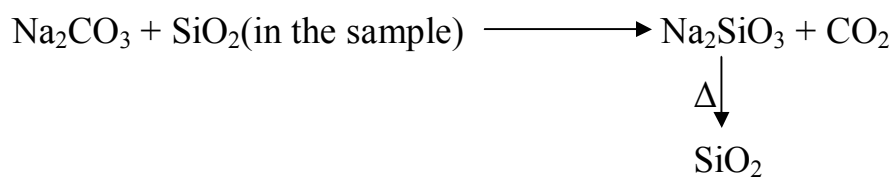
2.3.3 Determination of moisture:

0.129g of mica was weighted in moisture instrument and the result was measured.

2.3.4 Determination of silicate:

In a platinum crucible 0.5126 g of mica was weighted and was added about 7.0 g of sodium Carbonate, the contents in crucible were heated in a flame then in furnace at 1000°C at half an hour.

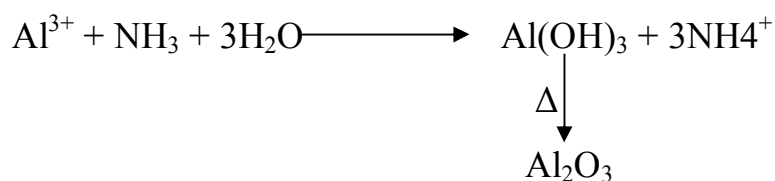
The crucible was placed in beaker contain 50 ml boiled distilled water and HCl (conc.) was added until the entire sample dissolved. The solution in beaker was put in flame until it become solid, and then the content in beaker was heated in oven 110°C at over night. The content in beaker was dissolved in 50 ml of hot distilled water and about 8 ml HCl (conc.). The solution was filtrated in 250ml volumetric flask and was completed to the mark (solution A); the precipitate was washed two wise by hot distilled water, was ignited in furnace at 1000°C and then was weighted.



2.3.5 Determination of oxides compound:

2.3.5.1 Determination of Al_2O_3 :

100 ml of solution (A) was pipetted into a beaker, three drops of methyl red indicator were added (the solution is pink color), the concentrated ammonia was added until the solution was became yellow color. The solution was heated in a flame (not boiled), was filtrated, ignited in furnace at 1100°C and then was weighted.



2.3.5.2 Determination of CaO :

The filtrated in upper method was put in a beaker and completed into 400 ml by distilled water, was heated, 2g of oxalic acid was added (the solution is pink color), the concentrated ammonia was added until the solution was became yellow color. The solution was heated in a flame (not boiled), was filtrated, ignited in furnace at 1100°C (in this sample the solution is clear and there is no CaO precipitate).

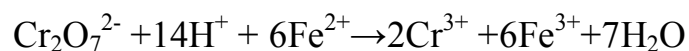
2.3.5.3 Determination of MgO :

The filtrated in upper method was boiled in flame, 10ml of saturated disodium hydrogen orthophosphate (25 g in 100 ml) was added with stirring in the flame, the solution was cooled, 30-40 ml of concentrated ammonia was added with stirring, was put in cold water at 30 minute with stirring, after at least three hours was filtrated, ignited in furnace at 850°C

and then was weighted. (in this sample the solution is clear and there is no MgO precipitate).

2.3.6 Determination of Fe₂O₃:

20 ml of solution (A) was transferred into beaker, zinc dust was added, was boiled in the flame, was filtrated in conical flask, 5 ml of ortho phosphoric acid was added, a drop of Barium diphenylamine sulfate indicator was added and then was titrated with potassium dichromate solution (0.1 M).



2.3.7 Determination of K and Na:

2.3.7.1 The preparation Sample solution:

In a beaker 0.1002 g of the mica sample was weighted and to it about 10 ml of HCl (1:19) , the contents in a beaker were heated until boiling, were filtered in a volumetric flask 100ml, then 10 ml of ortho phosphoric acid (1:19) was added and completed to the mark.

2.3.7.2 Preparing standard solution:

10ml of potassium solution 1000 ppm, 10ml of sodium solution 1000 ppm were pipetted into a 100 ml volumetric flask and the solution were completed to the mark.

The solutions in 2.3.6.1 and 2.3.6.2 were measured by flame photometer.

2.3.8 Determination of the content using EDX:

The sample was put in glass crucible and was measured by EDX instrument [table(7)].

2.3.9 Determination of the content using SEM-EDS:

The sample was put at stage of SEM instrument and was taken picture of the sample [figure (2)], was measured the contents by using EDS [table(8)].

Chapter three
Results and Discussion

3.1 Results:

In this experiments there are four type of analysis were used they are: classical analysis, flame photometric analysis EDX analysis and SEM analysis.

- The results of classical analysis show in the below:

Table (5):

Compound	Classical analysis%
Loss of ignition	2.41
H ₂ O	4.10
SiO ₂	44.57
Al ₂ O ₃	36.21
CaO	Null
MgO	Null
Fe ₂ O ₃	0.7

- The results of flame photometric analysis show in the below:

Table (6):

Analyte	Flame photometric analysis %
Na	Null
K	11.9

- The results of EDX analysis show in the below:

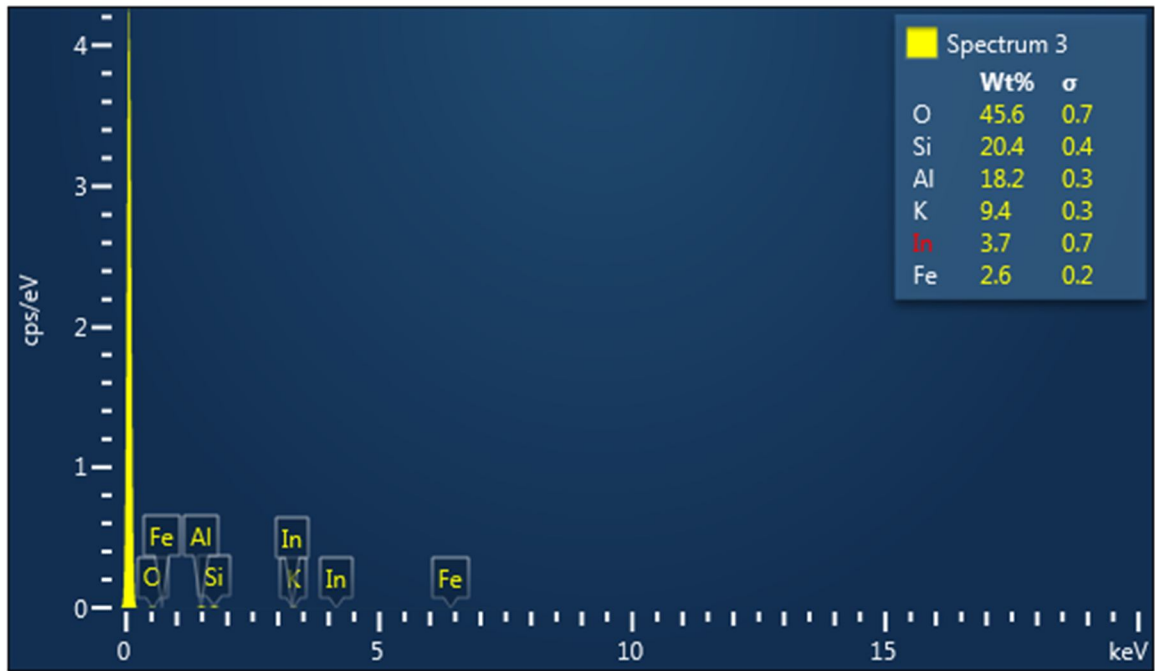
Table (7):

Analyte	Result %
K	35.814
Si	30.489
Al	19.115
Fe	12.543
Ti	0.656
Rb	0.597
Cl	0.324
Zn	0.147
Mn	0.141

- The results of SEM – EDS analysis show in the below:

Table (8)

Analyte	Result (Wt) %
O	45.6
Si	20.4
Al	18.2
K	9.4
In	3.7
Fe	2.6



Figure(1): Diagram of SEM-EDS instrument result

3.2 Discussion:

This work was direct toward the study and analysis of mica in Shereik area by several methods.

The flakes of mica were taken from the west side of Nile river near Algareeb village in Ashereik area, were crushed until become powder and analysis by classical analysis, flame photometer, EDX and SEM) and compare the results between them.

Table (5) and Table (6) presents the results by classical & Flame photometric analysis, it give percentage yield by weight and it shows that there are simple amount of organic cpds, H_2O and Fe_2O_3 , and high amount of SiO_2 , Al_2O_3 and K. O, H & C cannot measured by these methods.

These types of method takes several time and they have high percentage error.

Table (7) presents the results by EDX, the elements K, Si, AL & Fe were detected and a new elements appeared like Ti, Rb, Cl, Zn & Mn these element have very small percentage yield that mean it may be from the sample (the instrument is more accurate) or the sample chamber was contaminated and this the problem of EDX. this instrument is also cannot measured O, H & C.

Table (8) presents the results by SEM-EDS, the elements O, K, Si, AL & Fe wer detected and a new element appeared (In) which have very small percentage yield that mean it may be from the sample (the instrument is more accurate) or may be the instrument cannot decided the exactly element (the result written by red color). SEM uses

nanotechnology, this technique is superior in analysis, it provides fine details of samples, but it is very expensive.

3.3 conclusion :

Based upon the results obtained in this study; use the instruments like EDX & SEM is more accurate and provides very small details than classical & Flame photometric analysis.

3.4 Recommendations:

The results prove that the analyzed mica samples in Ashereik area is high quality & it could be exported or utilized in different industries.

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Appendix

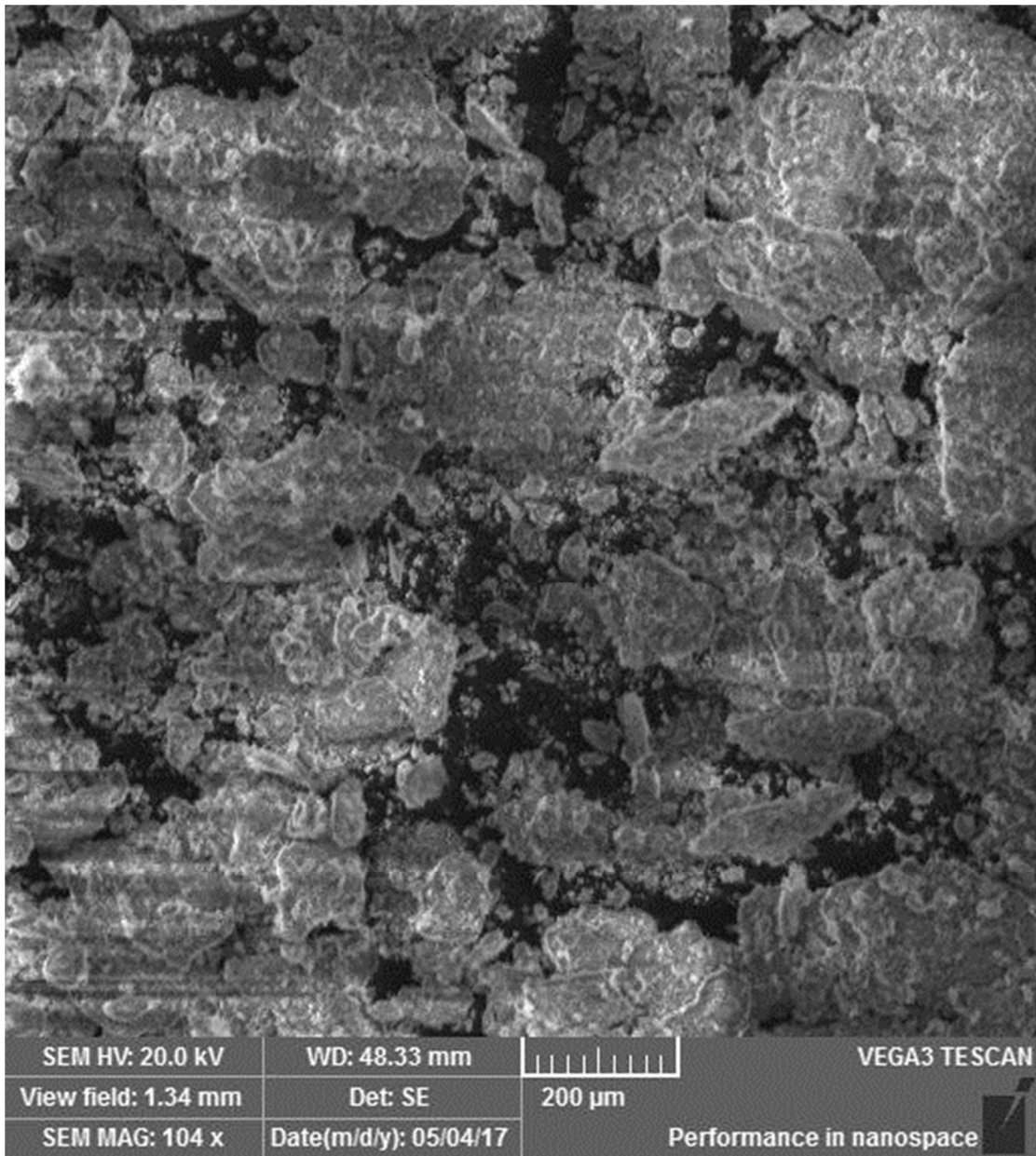


Figure (2): Mica sample under scanning electron microscope



Figure (3): Muscovite mica in Shereik area

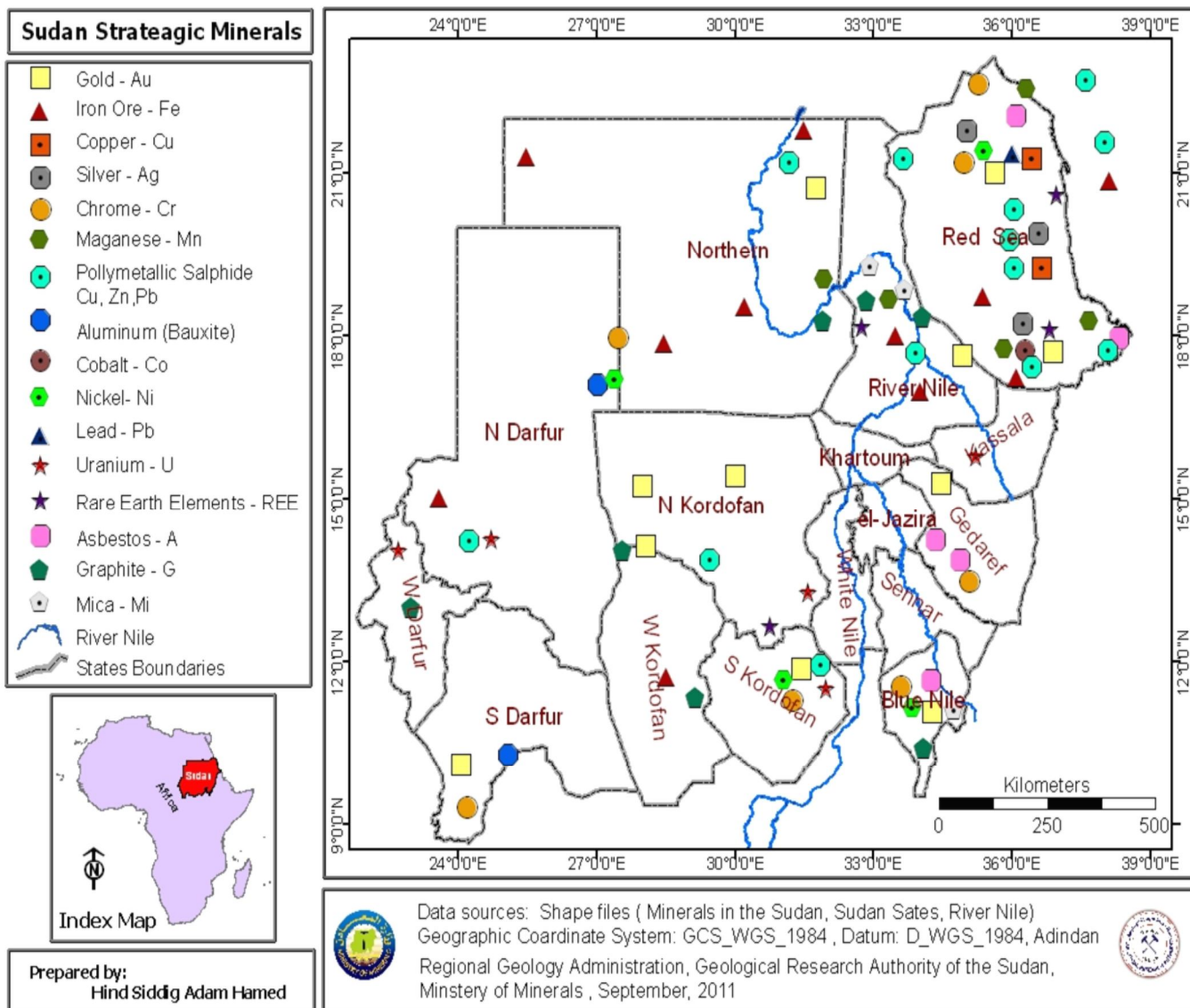


Figure (4): Map of some mineral in Sudan (The Field of Mineral Potential of the Sudan)

Graphite - Mica - Asbestos

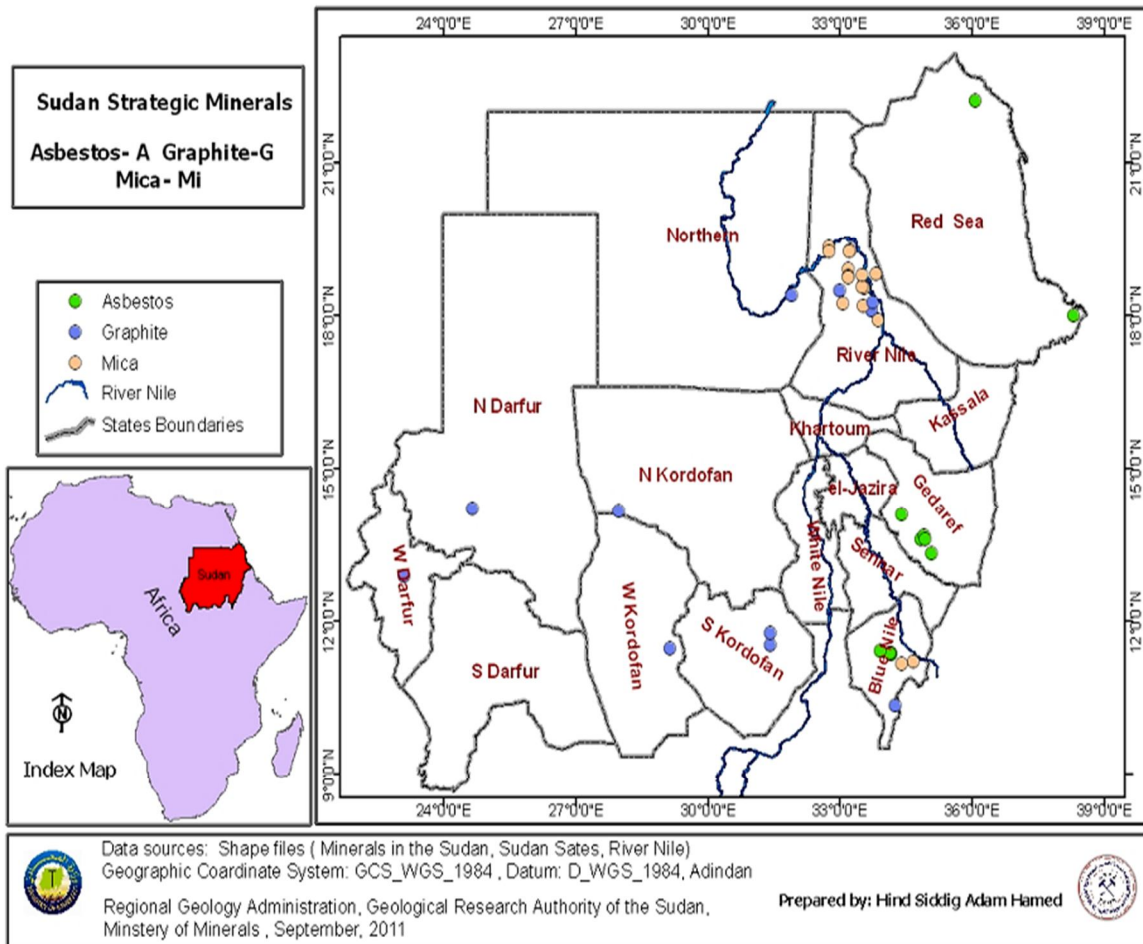


Figure (5): Map of Graphite - Mica – Asbestos in Sudan (The Field of Mineral Potential of the Sudan)