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Multi Spectroscopic Techniques to Analyze Natural Minerals to Determine Corundum Type of Gemstones from Darfur, Sudan.

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Abstract

The main objectives of this study were to know natural minerals contents to classify their types and determine corundum type. Energy Dispersive X-ray Fluorescence spectrometry, X-ray diffraction and Energy Dispersive X-ray Spectrometry coupling with Scanning Electron Microscope have been utilized to determine the mineralogical and elemental composition of semi gemstone samples collected from two different mine locations in Darfur region western Sudan. Results obtained from spectroscopic analyses indicated that Aluminum Oxide (Al_2O_3) was predominated in FDY sample(A) and Silica (SiO_2) was main contain in ODS sample(B) as well as different percentages of some metal oxides including trace and rare earth elements. According to the outcome of the Energy Dispersive X-ray Fluorescence results the FDY sample is characterized as Corundum (Ruby) and while the ODS sample is classified as Quartz (Carnelian). These characterizations are depended on physical appearance and color that when compared to the previous data of gemstones in addition to the analyzing results .The analysis showed that ruby major revealed composition were Al_2O_3 (79.02 %); SiO_2 (10.88%); SO_3(4.96%) Fe_2O_3 (2.27%); CaO (1.06%); TiO_2(1.08%) and quartz majority compositions were SiO_2 (94.23%) ; Al_2O_3 (1.96%); SO_3 (0.66%) Fe_2O_3 (2.10%); CaO (1.05%); TiO_2 (0.03%). X-ray Diffraction scanning tests of the samples showed that a vast majority represented by corundum (93.3%)and quartz (99.1%) in FDY and ODS samples respectively as dominant compound ,similarly for more investigation and delicacy Energy Dispersive X-ray Spectrometry coupling with Scanning Electron Microscope scan analyzing indicated that Al compounds(Al_2O_3) were outnumbered Si compound(SiO_2) in FDY sample while in ODS sample there was a significant presence of Si contents represented by standard level (SiO_2) on the bases of available facts of the study FDY sample considered to be corundum (ruby) and ODS sample as quartz origin.

Keywords: Ruby, Quartz, Composition, Oxide, Gemstone.

المستخلص

الأهداف الرئيسية لهذه الدراسة هي معرفة محتويات المعادن الطبيعية لتصنيف أنواعها وتحديد نوع كرندوم . تم استخدام مطياف طاقة الأشعة السينية المتفلورة ، حيود الأشعة السينية و مطياف طاقة الأشعة السينية المتشتتة مزدوج مع مجهر المسح الإلكتروني ، و ذلك لتحديد التركيب المعدني والعناصر لعينات من الأحجار شبه الكريمة التي تم جمعها من موقعين مختلفين للمناجم في إقليم دارفور بغرب السودان.و أظهرت نتائج التحليل أن أكسيد الألومنيوم (Al_2O_3) هو الغالب في عينة FDY (A) و كما أن السيليكا (SiO_2) هي المكون الرئيس في عينة ODS (B) وكذلك نسب مختلفة من أكاسيد المعادن الاخري بما في ذلك العناصر التي تتبع للعناصر الأرضية والنادرة. وفقًا لنتائج التحليل ، فإن عينة FDY توصف بانها كرندوم (روبي)والعينة ODY تصنف بانها سليكا (كوارتز) تم هذه التوصيفات اعتماداً على المظهر المادي الخارجي و اللون و بالمقارنة مع بيانات الدراسات السابقة للاحجار الكريمة وباللاضافة الي النتائج التي تم التوصل اليها عبر التحليلات الطيفية.و بينت نتاتج التحليل لمطياف طاقه التشتت الاشعه السينية المتفلورة مكونات العينة FDY بنسب مختلفة من اكسيد المونيوم (79.02%) ,كسيد السيلكون (10.88%), ثلاثي اكسيدالكبريت (4.96%), اكسيد الحديد الثلاثي (2.27%) ,اكسيد كالسوم (1.06%) واكسيد تايتانيوم (1.08%) , كما ان مكونات العينة ODS تتكون من اكسيد السيلكون (94.23%)اكسيد المونيوم (1.96%),اكسيد الكبريت (0.66%),اكسيد الحديد الثلاثي (2.10%),اكسيد الكالسيوم (1.5%)واكسيد تايتانيوم (0.03%) .كما اظهرت نتاتج حيود الاشعة السينية لاكسيد الالمونيوم (93.3%) و الاكسيد السليكون (99.1%) للعينات FDY و ODS على التوالى. وللمذيد من التحقيق والدقة تم التحليل بواسطة مطياف الطاقة المتشتتة مزدوج مع مجهر المسح اللاكتروني توصلت بناءاً على الحقائق المتاحة للدراسة ان العينة FDY تمثل رؤبي (كرنروم) والعينة ODS من اصل كوارتز (سليكا).

Introduction

A gem stone is a material that has a special value due to its fundamental quality, beauty durability and rarity. Corundum is chemically an aluminum oxide (Al_2O_3) has a hexagonal structure it is a relatively rare found igneous metamorphic and sedimentary mineral that requires unusual geochemical conditions of combined with high aluminum oxide contents of the host rock and deficit silica activity formation occurs deep extremely high pressures and temperatures. Corundum has two familiar forms ruby and sapphire its pure form is colorless, entrance of trace element usually Cr, Fe or V impurities into their structures, corundum eventually become the colored gem stone so ruby (red) or sapphire (blue), although other colors are also possible as result of localized of those impurities (e.g., Simonet et al., 2008). Ruby corundum that is colored red by trivalent chromium (Cr^{3+}) ions be present in the corundum in various valences and configurations as single or multiple ions combination that mixed valences occur, great to produced variety of colors Al_2O_3– Cr composites are also characterized by their high temperature resistance to oxidation, even up to 1400°C Minerals can be identified by their color, shininess (luster), hardness, shape and various other traits similarly producing of the blue color in sapphire high hardness, high mechanical strength, and because of their strong resistance to abrupt temperature changes within the 25-1300°C range, they can work at temperatures exceeding 1000°C. Historically the first known discovery of corundum from its quantity sufficient to make it of value as an abrasive was that of the emery fields of the Grecian Archipelago, and up to 1847 all the corundum used as an abrasive was obtained from these islands,

principally from the island of Naxos .Natural and synthetic corundum are used in a wide variety of industrial applications because of their toughness, hardness, and chemical stability. Quartz is widely distributed mineral consisting of silicon dioxide (SiO2) or silica when in its purest form (Finkelstein JN, et al. 2000). There are many fine-grained varieties of quartz; as Chalcedony, Agate, Carnelian, Carnelian, Basanite and Jasper. the percentage purity of the variety, mostly quartz contains 99-100% .EDXRF technique is less expensive and more available in gemological laboratories it is considered as a non-destructive method, the color of a gemstone can be affected during the measurement and the method is still quite expensive . EDXRF uses an X-ray beam to irradiate the sample after which the emitted X-ray fluorescence is measured minerals compositions (Min Yao 2015).This research intended to study natural minerals through different spectroscopic techniques to provide some data about natural minerals.

`Fig(1) :Sudan map indicates to the studied samples locations [A](FDY)and[B](ODS) FDY and ODS codes of represented samples (i.e two different natural mine locations in Darfur region)

Materials and Methods:

Samples Collection:

These samples were collected from natural minerals from two regions of western Sudan due to their physical appearance which resembles precious metals as precious stones their presence is very rare only found in certain countries like Burma. To find out the components of these stones and their proportions composition and impurities by spectroscopic techniques.

Sample Preparation:

The studied samples were prepared after collecting them by local traditional miners later some special inspections were carried out, revised and sorted, and some samples were identified as more representative. Symbolized sample in terms of color or transparency and luminosity. Finally, the first sample representative named as (FDY) and the second sample representative named as (ODS) were chosen, cleaned, dried and taken conditions The laboratory's own testing procedure has been taken then each sample were crushed into several parts, and analyzes have been carried out using spectroscopic techniques such as EDXRF, XRD and EDS-TEM coupled. Taking advantage from previous studies that allowed discriminating minerals

through chemical physical analyzing. Each of the these samples have taken as representatives of different rock specimens due to their appearance resembling gemstone that in previous studies given some specifications . This study concerns to investigate the reality of these minerals and their contents in order to determine their origin. However, represented samples have given codes to differentiate between them FDY and ODS as codes for samples [A] and [B] represented of studied minerals independently. The aims of this research paper were to identify natural minerals by multi analyzing techniques as primary target and determine corundum type as well as their contents difference and impurities.

Results and Discussion:

Energy Dispersive X-ray Fluorescence (EDXRF) spectrometry:

EDXRF spectra of natural minerals exhibit strong X-ray peaks of aluminum (Al)and oxygen(O) as basic Al_2O_3(79.02%) composition besides some metal oxides in these details of FDY sample $A1_2O_3$(79.02%)SiO_2(10.28%),SO_3(4.96%),Fe_2O_3(2.27%),TiO_2(1.08%),andCaO(1.06%) while in ODS sample silicon and oxygen were detected in high peaks ,showed that the chemical composition SiO_2 (94.23%), has a great majority this indicated that silicon contents as basic in this samples as well as other contents in details besides of SiO_2 (94.23%), Al_2O_3(1.96%) ,SO_3(0.66%), Fe_2O_3 (2.10%), TiO_2 (0.03%)and CaO(0.59%).In order to determine whether these minerals can be categorized as gemstone or not , we need to compare the mineral contains in the sample .Clearly both samples have similar contains but their percentages were different $A1_2O_3$(79.02%),SiO_2(10.28%) and $A1_2O_3$(1.96%), SiO_2(94.23%) of two sample respectively the majority dominant content which can be determine the rock type as well as both samples have nearly same percentage Fe_2O_3.The total difference between oxide compositions contained in the FDY sample is 166.4% with average 27.73% . Along with that there were a small fraction of same metal oxide such as K_2O, MnO and Cr_2O_3 in both samples, furthermore there have been shown some oxides including Ca_2O and MnO sample ODS did not appeared in other sample this can be clear difference between two samples in this contents. Minor ,major and trace elements both have high level of specific elements like Al, Si, S,Fe, Ca and Ti at percentages (70.91, 12.33, 5.33, 5.22, 2.77and 1,96 %) respectively of sample majority elements but sample ODS Has 1.77% oF Al , 89.78 % of Si ,0.63% of S , 5.35 of Fe and 1.38% of Ca .these analyses showed that the similarity in of Fe element percent . The total difference between oxide compositions contained in the ODS sample was 154.68% with average 14.06% .Table (1) gives details of composition percepts of the samples FDY and ODS obtained from EDXRF analyses (I) shows Comparison of oxide compound compositions of samples and (II)-Shows Major and Minor Elements With Spectral Analysis Data of The Study Samples from EDXRF results.

Tables (1) : Samples compositions and their differences in Oxides , major and minor Elements from

Comparison oxide compound compositions in the samples						
Sample%\composition	Al2O3	SiO2	SO3	CaO	Fe2O3	TiO2
FDY	79.02675	10.88964	4.964388	1.064896	2.272726	1.089837
ODS	1.96264	94.23852	0.66831	0.59283	2.10446	0.033531
Difference	77.06435	83.34888	4.296078	0.472066	0.168266	1.056306
Total difference	166.4					
Average	17.734					

Major and Minor Elements with Spectral analysis data of the study-											
Sample%\Element	Al	Si	S	Cl	K	Ca	Ti	Cr	Mn	Fe	Zr
FDY	70.91	12.60	5.33	0.33	0.53	2.17	1.96	0.29	0.06	5.22	0.11
ODS	1.77	89.78	0.63	0.04	0.03	1.38	0.07	0.07	0.29	5.35	0.00
difference	69.14	77.18	4.7	0.29	0.5	0.79	1.89	0.22	0.23	0.13	0.11
Total difference	154.68										
Average	14.06										

3.2 X-ray Diffraction (XRD)

XRD with Cu-KĮ radiation (Bruker D4 Endeavor). The detector position is recorded as the angle 2theta (2θ) . XRD was performed to detect and identify the phases in the powders to investigate and characterize the structure of the prepared samples (powders). XRD analysis detected aluminum oxides Al_2O_3 and Silica SiO_2 as predominant mineralogical phases can be seen in figure (2) the pattern indicated in corundum Al_2O_3 (93.2%) which has hexagonal structure trigonal crystal system and cell parameters[a = 4.75 Å, c = 12.982 Å; Z = 6], Quartz high SiO_2 (2.8%) also named beta quartz which has cell parameters[a = 4.9133 Å, c = 5.4053 Å; Z=3] these two have high peaks beside of minor fraction of Rutile has chemical formula TiO_2 (0.7%) ,Muscovite2M which has sophisticated chemical formula of $KAl_2(Si_3Al)O_{10}(OH,F)_2$ (2.1%),Dolomite which has chemical formula of CaMg (CO_3) and ,Biotite is phyllosilicate belongs to the mica group has chemical formula of $K(Mg,Fe)_3$ $(AlSi_3O_{10})$ $(F,OH)_2$ (0.4%) the figure (2) showing XDS pattern of FDY sample indicated that the corundum (Al2O3) as main content the crystal , but ODS sample was showed a high majority of Quartz which is a mineral composed of silicon and oxygen has chemical formula SiO2(97.2%) and unit cell parameters [a = 4.9133 Å, c = 5.4053 Å; Z=3] with contrast of FDY sample here corundum is disappeared but shown fractions of some trace elements compound represented by some metal oxide and others such as Rutile TiO2(1.0%) , muscovite KAl_2 $(Si_3Al)O_{10}$ $(OH,F)_2$(1.6%)$CaCO_3$ (0.2%) .Figure (3) displays XRD Patten of ODS sample indicated that obviously quartz as the principal component the crystal .

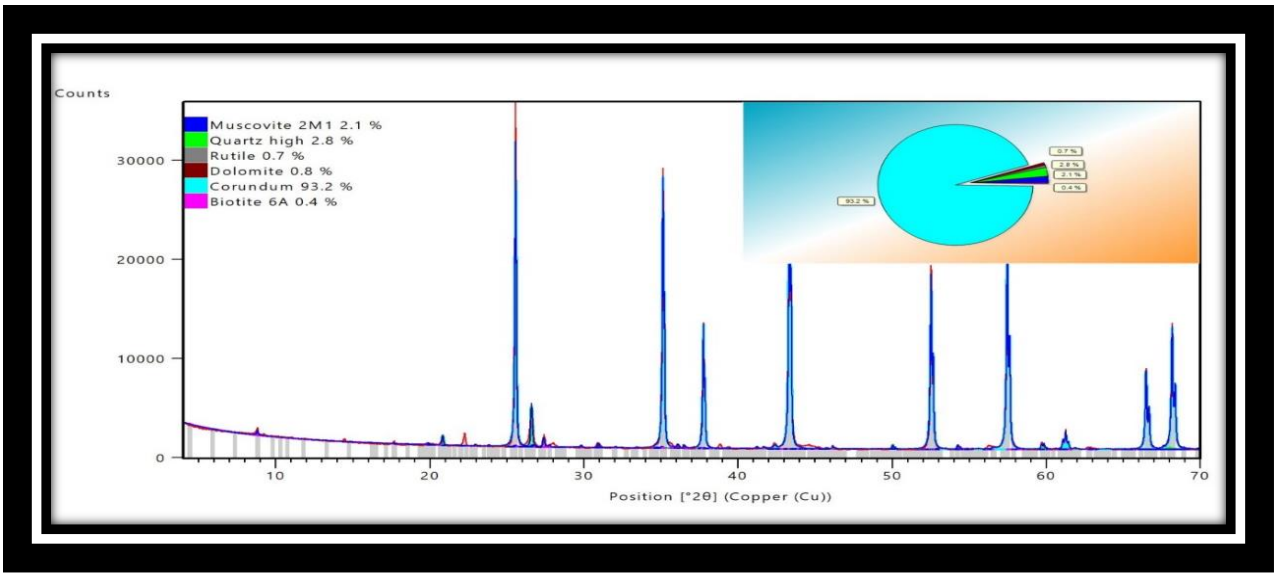


Figure (2). XRD Pattern of Natural Corundum of FDY Sample

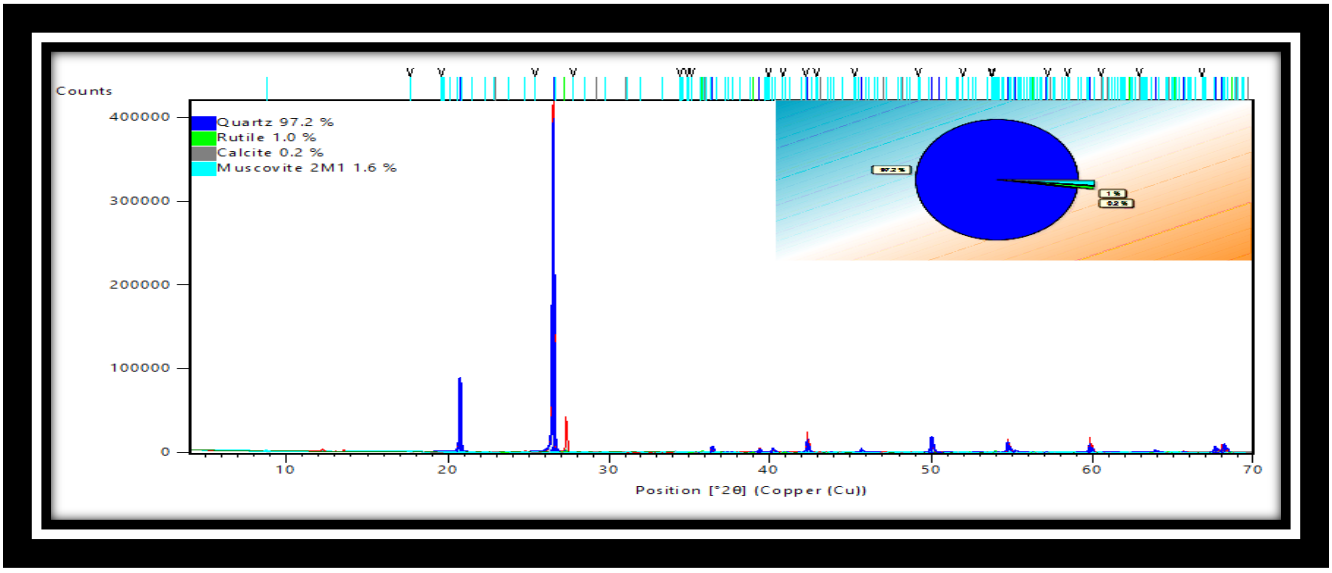


Figure (3) XRD oF Natural Quartz of ODS Sample

Energy Dispersive Spectroscopy (EDS) coupled with scanning electron microscope (SEM):

EDS systems include a sensitive x-ray detector, a liquid nitrogen for cooling, and software to collect and analyze energy spectra. study was to evaluate the potential of scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM/EDS) for determination of mineral phases according to their stoichiometry and assessment of mineral composition. EDS /SEM analyses of sample FDY sample in four division scans for more accuracy and better analyses results: First division scan showed peaks of (O,Al,Si,K,Ca) their weight percent(wt.%)(57.01,16.99,18.09,1.89,6.06) as well as their appropriate stander level (SiO2,Al2O3,SiO2,KBr,Wollastonite) respectively in these results SiO2 has majority of weigh comparing with Al2O3

this can be clear that association between silicon and oxygen elements is greater than between Aluminum and oxygen also the existence of Wollastonite (CaSiO3) is higher than KBr . Second division scan showed (O ,Al, Si, K, Ca ,Fe) their weight percent (wt.%)(52.81,15.66,15.65,1.01,11.88,2.99) as well as their appropriate stander level (SiO2,Al2O3,SiO2,KBr,Wollastonite ,Fe) respectively these results were nearly similar to the first division scan except appearance of Fe element hence , Al and Si quantity were nearly the same. Third division scan showed (O ,Al, Si, K,,Fe) element with their weight percent (wt.%)(50.60,18.70,19.12,8.05,3.53) as well as their appropriate stander level (SiO2,Al2O3,SiO2,KBr, Fe) respectively in these data showed that SiO2 dominated the compound but Al2O3 contents has a small fraction also disappeared of wollastonite as difference with two scans above . Fourth scan showed only two peaks (O ,Al) represented by weight percent (wt%)(44.55 ,55.45) as well as their appropriate stander level (SiO2,Al2O3) respectively this can be clearly Aluminum contents overcame Silicon contents so its represented main contents of the sample . in addition to silicon contents appear with oxygen standard level and silicon as single amount was disappeared . Table(2) displays EDS analyses of sample FDY in four divisions scans and figures (3 , 4 ,5, 6) give EDS /SEM coupled analyses in four divisions given character sample SEM (lift figure)and EDS (right figure)for each division .

Table (2):EDS of FDY sample division scans result

First Division Scan		Second Division Scan		Third Division Scan		Forth Division Scan		Standard Label
Element	**Wt.%**	**Element**	**Wt.%**	**Element**	**Wt.%**	**Element**	**Wt.%**	
O	**57.01**	**O**	**52.81**	**O**	**50.60**	**O**	**44.55**	**SiO2**
Al	**16.99**	**Al**	**15.66**	**Al**	**18.70**	**Al**	**55.45**	**Al2O3**
Si	**18.09**	**Si**	**15.65**	**Si**	**19.12**	**-**	**-**	**SiO2**
K	**1.89**	**K**	**1.01**	**K**	**8.05**	**-**	**-**	**KBr**
Ca	**6.02**	**Ca**	**11.88**	**-**	**-**	**-**	**-**	**Wollastonite**
-	**-**	**Fe**	**2.99**	**Fe**	**3.53**	**-**	**-**	**Fe**
Total:	**100.00**	**Total:**	**100.00**	**Total:**	**100.00**	**Total:**	**100.00**	**-**

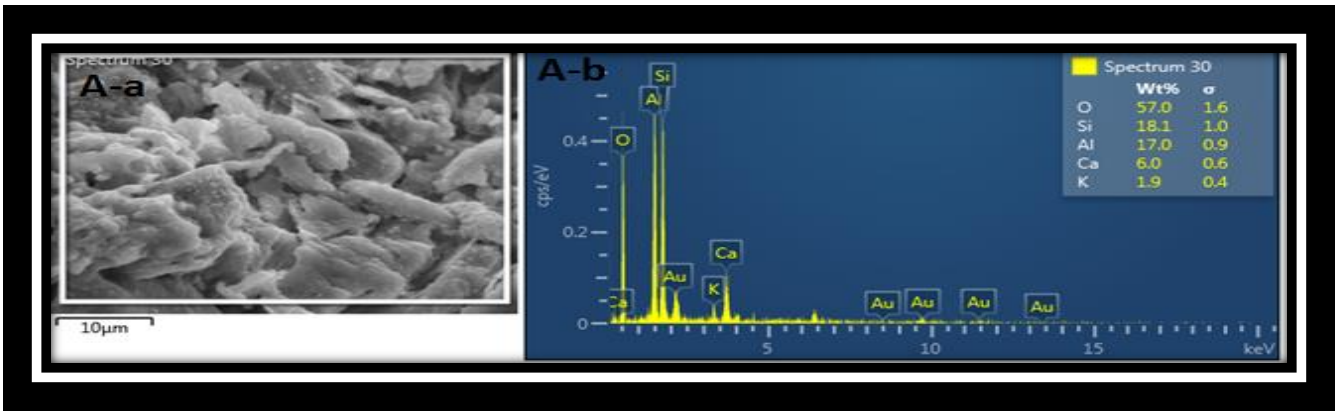


Figure (3);A-a SEM image and A-b EDS analyses of first division scan of FDY sample

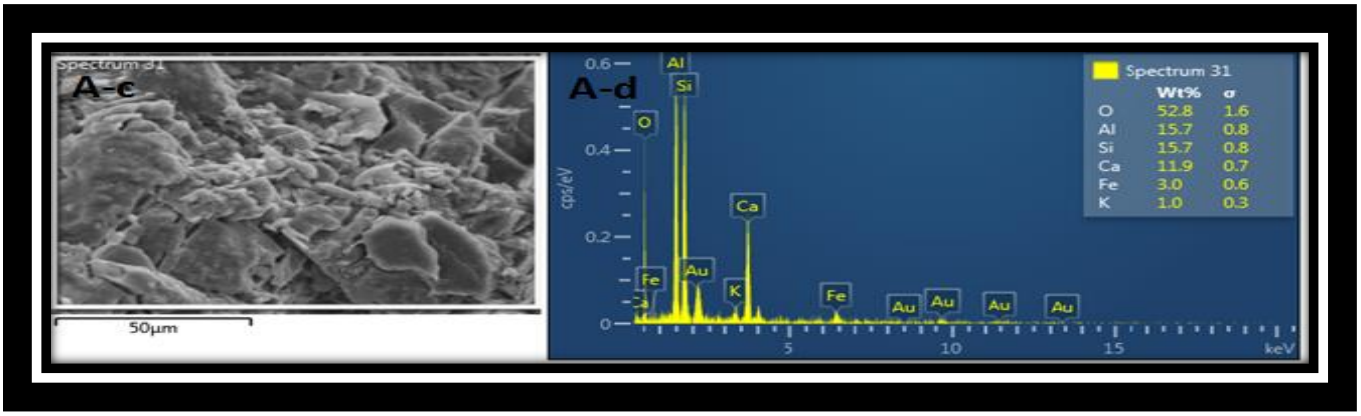


Figure (4);A-a SEM image and A-b EDS analyses of second division scan of FDY sample

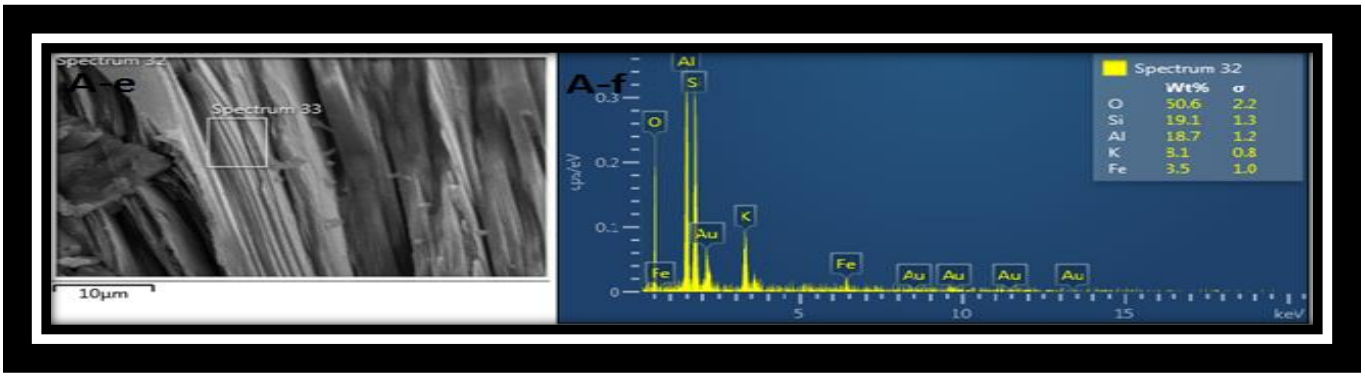


Figure (5);A-a SEM image and A-b EDS analyses of third division scan of FDY sample

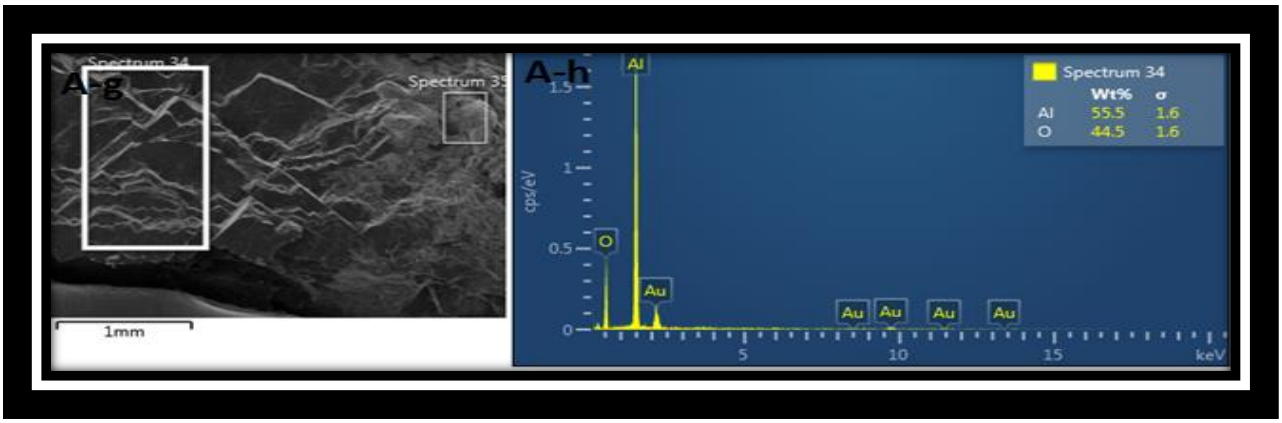


Figure (6);A-a SEM image and A-b EDS analyses of forth division scan of FDY sample

ODS sample the first division scan showed peaks (O ,Al, Si) their weight percent (wt.%)(49.14,1.42, 49,44) as well as their appropriate stander level (SiO2,Al2O3,SiO2) respectively these results showed clearly Aluminum contains(1.42%) is less than Silicon contents(49.44%) .The second division scan showed peaks of (O ,Mg, Al, Si K, Au) in proportion weight percent (wt.%)(47.52,0.50,15.40,21.90,0.61,14.08) as well as their appropriate standard level (SiO2 ,MgO,Al2O3,SiO2,KBr ,Au) respectively as the results have given SiO2 is major content. Third division scan displayed (O, Mg ,Al, Si, K, Ca ,Fe ,Au) peaks in weight percent (wt.%)(46.68, 0.56, 13.89, 22.25, 0.65, 0.69, 1.04, 14.22) as well as their appropriate slandered level (SiO2,MgO,Al2O3,SiO2,KBr,Wollastonite, Fe,Au) respectively in these results indicated that Si contents were greater than Al contents also appearance of Wollastonite as difference .Fourth scan exhibits (O ,Al, Si ,Au) peaks in weight percent (wt.%)(36.60, 0.94, 44.33, 18.12) as well as their appropriate stander level (Sio2,Al2O3,SiO2) respectively . Table(3) demonstrates EDS analyses of sample ODS in four divisions scans and figures (7 , 8 ,9, 10) illustrate EDS /SEM coupled analyses for four divisions given character sample SEM (lift figure)and EDS (right figure) in each division .

Table (3):EDS of ODS sample division scans result

First Division Scan		second Division Scan		Third Division Scan		Forth Division Scan		Standard Label
Element	Wt.%	Element	Wt.%	Element	Wt.%	Element	Wt.%	
O	49.14	O	47.52	O	46.68	O	36.60	SiO2
Al	1.42	AL	15.40	AL	13.89	Al	0.94	Al2O3
Si	49.44	Si	21.90	Si	22.25	Si	44.33	SiO2
-	-	Mg	0.50	Mg	0.56	-	-	MgO
-	-	K	0.61	K	0.65	-	-	KBr
-	-	Au	14.08	Ca	0.69	-	-	Wollastonite
-	-	-	-	Fe	1.04	-	-	Fe
-	-	-	-	Au	14.22	Au -	18.12	Au
Total:	100.00	Total:	100.00	Total:	100.00	Total:	100.00	-

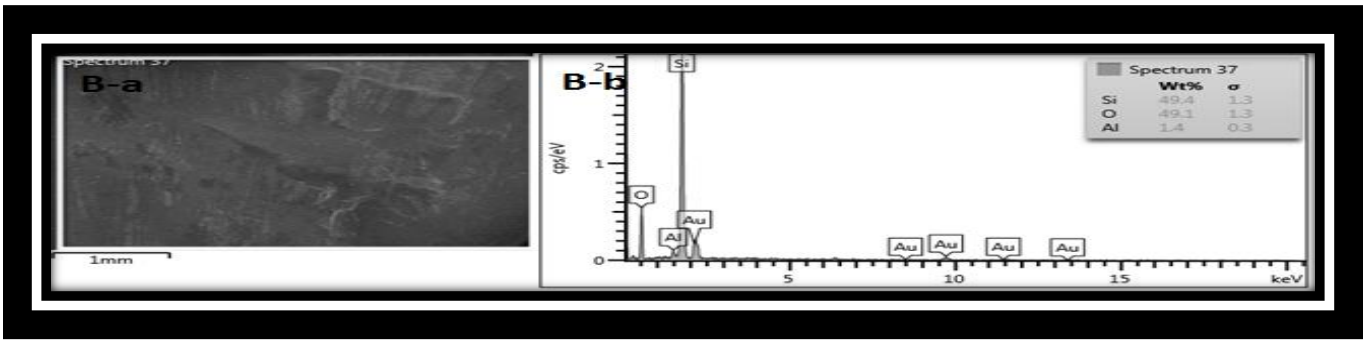


Figure (7) :B-a SEM image and B-b EDS analyses of first division scan of ODS sample

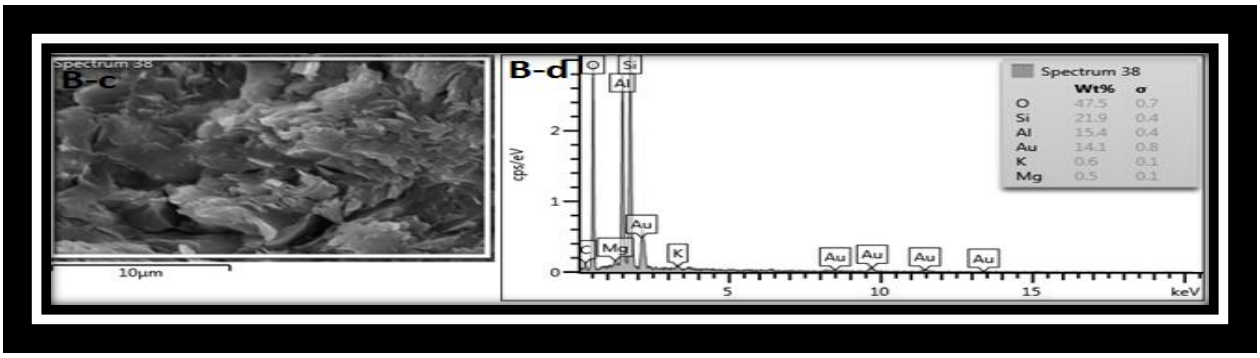
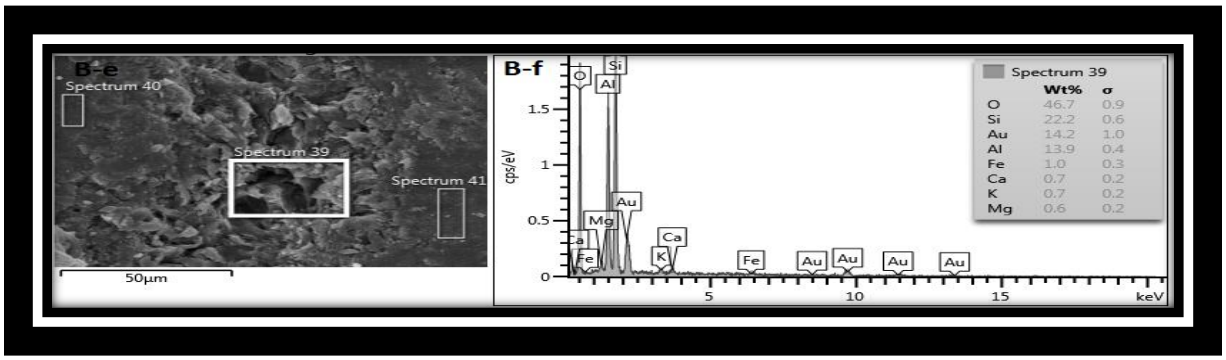


Figure (8) :B-c SEM image and B-dEDS analyses of second division scan of ODS sample



Figure(9) :B-e SEM image and B-f EDS analyses of third division scan of ODS sample

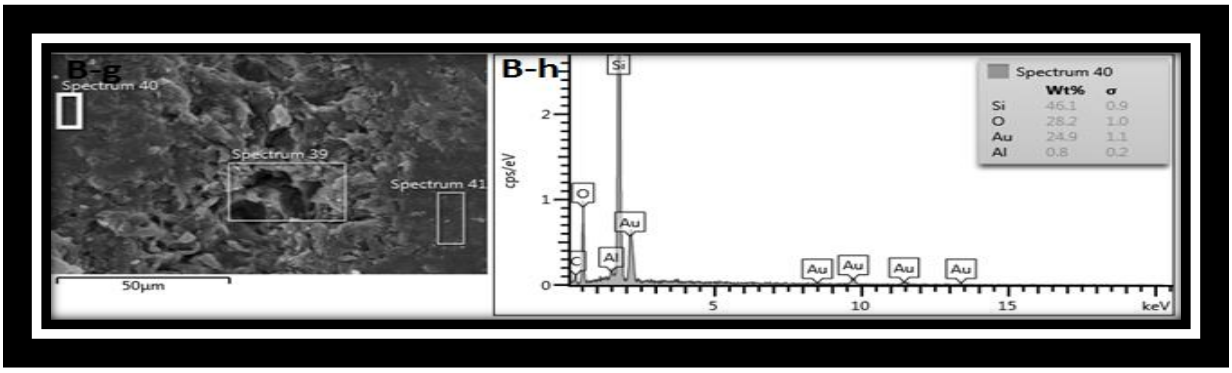


Figure (10) :B-e SEM image and B-f EDS analyses of forth division scan of ODS sample

Conclusion

Analyses and studies for the selected samples were carried out to identify mineral type through physical and chemical characteristics and discrimination of basic contents as well as impurities the possibility to determine chemical and concentration of specific mineral depends on the backbone of the basic body composition, available of chemical data always important to observe behavior of possible gemstone or any precious stone. previous studies have been given details of how to know an specific gemstone due to its color ,specific gravity or other physical properties but in this study we intended to identify gemstone sort via

chemical composition and color with considering in physical appearance Common denominator as a major component of the metal as in Corundum have different types and is the basis of the composition of aluminum oxide and the presence of impurities gain different types and forms .In this study showed most of the results of the sample Al_2O_3 is the dominant component and the rest as impurities and factors associated with the environment and composition .In this study also has investigated the fact that corundum can be found in deficit silica activity environment. Based on the results obtained by (EDRXRF ,XRD and SEM- EDS) the chemical analysis in addition to the previous studies we found that the first study sample represents a ruby due to contains of a large amount of aluminum oxide in different techniques showed about (EDXRF 80%, XRD 93%). On the other hand , the second sample represents as quartz because it contains silicon dioxide as the main component different techniques show about (EDXRF 90%, XRD99 %) of the mineral. Therefore, we conclude that the gemstone of sample A is really can be categorized as corundum specially ruby due to its red color and presence of chromium impurity which gives red color to the corundum and differentiate it from sapphire which has similar composition .

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