Journal of Radiation and Nuclear Applications
An International Journal

http://dx.doi.org/10.18576/jrna/100209

Multi-element Analysis of Beryl Mineral from some Selected Sites in the Egyptian Eastern Desert for Color Enhancement by Neutron Irradiation

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Received: 02 Jan. 2025, Revised: 09 Mar. 2025, Accepted: 11 Mar. 2025.
Published online: 1 May 2025.

Abstract: Beryl is a beryllium aluminum silicate, whose chemical symbol is (Be3Al2Si6O18). It is considered the primary raw material for beryllium and is one of the stones that have been processed to become one of the best gemstones in the market. In this research, the raw beryl stones are first examined and selected so that they do not contain many trace elements by subjecting the samples to an analysis using Energy-Dispersive X-ray Spectroscopy (EDS) as well as RAMAN Spectroscopy, The samples are subjected to irradiation by neutrons from the second Egyptian research reactor (which operates at a power of 22 megawatts and with a neutron flux of$1.2\ x10^{14}$ n.cm-2. s^{-1}.) of $6.8\ x10^{11}$ n/(cm2.s)-apart from the core- for short time irradiation samples and irradiation by neutrons flux of 0.37x1014 n/(cm-2.s)-nearby the core-for long time irradiation samples. After that, the samples are transferred for cooling and neutron activation analysis (NAA) to investigate the isotopes resulting from the irradiation and calculate the concentrations of these isotopes. It is noted that the color change occurs after exposure to radiation as a valuable gemstone. Some spectral analyses, especially Raman spectroscopy showed that there is a relationship between the color changes and the changes in the crystal structure components of beryl ore due to the irradiation effect. Finally, theoretical calculations are made to predict the time required for the activity to reach the safe level for transportation.

Keywords: Beryl, Irradiation, Gemstones, RAMAN Spectroscopy.

1 Introduction

Gemstones have been of interest to humans since ancient times because they possess desirable qualities of beauty, durability, and rarity, which have contributed to their popularity and high economic value. Throughout history, different types of gemstones have been considered a symbol of social status, such as diamonds, rubies, sapphires, emeralds, tourmalines, aquamarines, topazes, and others [1]. The intrinsic value of these stones lies in their natural form, and some can be made valuable by enhancing the color using artificial means. These gemstones are called "treated stones". In general, color, clarity, cut, and carat are the most important characteristics that distinguish gemstones. Color is the first and foremost, which can be controlled by several means, including thermal heating, diffusion, coating, bleaching, and radiation methods which include X-rays, gamma rays, electrons, and neutrons. Except for neutrons, the colors created are usually temporary [2]. Topaz is the most common treated stone, as the color can be enhanced or even changed by exposure to radiation, which is often done with neutron rays in a nuclear reactor or electron rays using accelerators, and it can also be done with gamma rays [3-7]. The Egyptian Research Reactor II could serve this purpose, as it is a large source of neutrons [8]. Beryl from Egypt (extracted from the Eastern Desert of Egypt) was chosen for this study because of the interest of gemstone traders and exporters. This is mainly due to the large variation in prices of natural and treated gemstones [9].

Aim of the work:

In this research, raw samples of beryl were exposed to neutron rays from the second Egyptian research reactor to produce them as gemstones. Changes in the color of the stones were observed. This study was conducted through the following steps:

1-The energy dispersive X-ray spectrometer (EDS) is used to determine the raw sample elements and trace at different

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concentrations, Also the inductively coupled plasma mass spectrometer (ICP-MS) is used for elemental analysis. That is, inductively coupled plasma mass spectrometry (ICP-MS) and EDS are used to study and analyze the raw samples. 2- Raman spectroscopy is used for samples before and after treatment because this spectroscopic technique can provide important information about the molecular structure. It is a useful tool in the molecular characterization of inorganic species of materials.

In addition, by using this technique, a relationship is found between the changes in the color of the stone versus the change in the intensity of the scattered peaks resulting from the vibration of the molecular effective group (due to the irradiation). 3- Then the radioactivity resulting from the irradiated samples is measured –after cooling to reach the safe limit for use. This is done by conducting neutron activation analysis (NAA) in cases of short- and long-term radiation. 4- Finally, using radiation protection devices, where doses and exposures to radioactive beryl products are measured.

1.1 Treatments:

The treatment of gemstones is done to maintain their appearance in terms of color and transparency, which results in increasing their commercial value [10]. Radiation treatment is considered the cheapest method. Since color is the most important property of gemstones, treatment is done to change the color for the better. Changing the color is done through different techniques such as dyeing and heating in addition to radiation treatment, which is considered the most important. Radiation treatment is done by different means such as gamma radiation using cobalt-60 or electron rays using cyclotrons or neutron irradiation inside the reactor. Radiation treatment technology has recently increased [11-21].

2 Irradiation Methods

A. Gamma Irradiation.

There are several reports on gamma irradiation, including one published on irradiated topaz using gamma irradiation facilities, by using high doses of ^{60}Co gamma rays on a sample of colorless topaz. The result was that the topaz acquired its color, but it was not uniform in color due to excessive heat produced. Therefore, it is recommended to use lower dose rates to avoid excessive heat build-up within the topaz samples during gamma irradiation, and this often depends on the size of the topaz. Gamma irradiation does not produce radionuclides. In addition, some gemstones receive heat treatment after irradiation to stabilize their color and maintain their appearance [22].

B. Irradiation by neutrons:

Neutron irradiation has many advantages over gamma or electron irradiation, as it gives us a deeper and more stable color. The reason for this is that it gives a greater pigment and a stronger stimulus to change the crystal structure, as it can penetrate the entire crystal structure, unlike the electron beam and gamma rays. A good example of this is the case of irradiating topaz with neutrons, where a strong blue color is produced. It is noted that the number of neutrons required depends on the amount of topaz to be irradiated in addition to the amount of color depth. The neutron flounce required is usually and especially for the case of topaz- as published - in the range of (10^{17}-10^{18}) cm-2. Since the irradiation results in a build-up in temperature, effective cooling methods must be used for irradiated topaz [23]. In general, irradiating any stones using neutrons leads to an increase in temperature, which results in a change in color and damage, and sometimes leads to peeling of the stone. Therefore, it is necessary to cool the batch of stones throughout the irradiation process and control this temperature. One of the major problems related to irradiating stones with neutrons in general is that irradiation leads to the production of radioactive isotopes and thus radioactivity appears in the treated stones. Therefore, stones must be stored for a specific time known as the cooling period, which is very important to reduce this activity to the level of safe handling and thus transportation. This minimum activity is determined by national regulations. For example, European countries have set 74 Bq/g as the minimum for transportation. Note that we mention again that gamma irradiation does not produce radionuclides otherwise the neutron.

This paper aims to recommend beryl to be an Egyptian-treated gemstone. Elemental and concentration analysis of natural beryl samples from Eastern Desert-Egypt have been investigated by EDX, in addition to a high-resolution Inductively Coupled Plasma Mass Spectrometer (ICP-MS).

The samples were irradiated with neutrons,(short and long irradiation). After that, the samples were transported, Elemental and concentrations analysis of irradiated beryl is done by Raman spectroscopy analysis which is an excellent method for examining gemstone samples by producing the absorption spectrum of molecular bonds, which in turn is considered a fingerprint of the molecule. Therefore, it can characterize the molecular structure of samples without any destruction of the sample, in addition to other advantages, including quick ease of preparation and short analysis time.

The radioactivity of irradiated samples was measured and analyzed by (NAA).

Materials and Methods:

Experimental:

1- Sample preparation:

Beryl samples are prepared for irradiation as follows:

The samples are prepared using an ultrasonic cleaner with trichloroacetone, followed by cleaning with ultrapure water, then dried with nitrogen gas, weighed with a sensitive scale, covered with aluminum foil, and placed in a sealed aluminum container. The sample is then ready for irradiation in the reactor, specifically in the radiation positions around the reactor core.

2- Irradiation facilities in the ETRR-2 reactor (neutron irradiation facility) are through the reactor core and irradiation positions around the core. The ETRR-2 reactor is a multi-purpose research reactor MPR with a power of 22 megawatts and a thermal neutron flux of 1.2 x10^{14} n.cm^{-2}.

s^{-1}. cooled by light water. It is used to produce isotopes

and conduct research related to neutron physics as well as to supervise the irradiation of gemstones and others. In this study, beryl samples are irradiated through the irradiation positions arranged around the core, where the first row of irradiation positions is designated for the production of isotopes and the next one is used for our purposes. When selecting irradiation sites for beryl, the coming conditions are taken into account:

(a) The value of fast neutrons required to irradiate beryl.

(b)The effect of the container filled with beryl on the reactivity of the core to ensure the safe operation of the reactor.

(c) The cooling must be sufficient for the heat generated (as a result of irradiation) so that boiling does not occur.

2-Beryl stone irradiation:

The following must be taken into account when irradiating:

(1) Samples are selected and prepared appropriately to minimize contamination. (2) The storage time after irradiation depends on the activity of the radioisotopes. (3) Using (NAA) which is an analytical technique that relies on the measurement of gamma rays emitted from an irradiated sample, where the emitted gamma rays from the sample isotopes are proportional to the concentration of these radioisotopes. (3) The process of irradiating beryl in our experiment is subject to two methods of irradiation.

A- In the case of short irradiation, (after washing, drying, and weighting the samples). By using the rabbit system, samples are sent to radiation positions for some time of 30 seconds; with a fast neutron flux of 6.8 $x10^{11}$ n/ (cm^{-2}. s^{-1}) in the radiation positions concerned. This procedure may be done once or several times to subject the samples to a specific amount of irradiation. The samples are cooled for 1 minute before counting, after that the rabbit is extracted from the irradiation position and transferred the samples to the laboratory to conduct the analytical process for the irradiated stones. The gamma radioactivity of the entire batch of stones is measured, and then the gamma radioactivity of each sample is measured individually, it should be noted that in the case of the short irradiation, It was found systematic to produce the resulting nuclides. Al, Cl, Mg, Na, Ti, V, and K. as shown in table(2).

B- On the other hand for long irradiation. After washing and drying the samples, they are weighed accurately and placed inside aluminum cans, then placed in the positions of fast neutron flux of 0.37x 10^{14} n/(cm^{-2}. s^{-1}), for (2, 4, 8, and 12) hours taking into account continuous adequate cooling. After that, they are removed from the irradiation positions to measure the radiation dose rate by contact. These radioactive aluminum cans are stored for (21-25) days in special places. After this period, the samples are removed from the cans to measure the dose rate and surface contamination for each sample individually. Then the samples are returned to the cans for 3 weeks to open the cans later and measure the samples individually by HPGe detector based on reference recommendations. The radioactivity resulting from irradiation treatment is measured for each stone individually using an ORTEC Axial (HPGe) detector of type N with 100% relative efficiency and 2.1 kV resolutions at 1.333 MeV Co-60 line.

(This detector is operated by Gamma Vision software. In addition, gamma rays are received by the Multi-Channel Analyzer MCA card coupled to the detector. The standard sources used were produced by Isotope Products Laboratories and include point sources Ba-133, Co-60, Cs-137, and Eu-152.).

Counting is done several times in this way to determine all the isotopes resulting from the irradiation. It is often observed that long-term radiation leads to the production of the following radionuclides: Sb, Mn, Ta, As, Ba, Cs, Co, Eu, Fe, Rb, Sc, and Zn. as shown in table(3) [23-26].

3-Radiation Detection Equipment:

Assessment of the irradiated beryl risk to consumers is required to identify the qualitative and quantifies of the resultant radionuclides and hence, the attendant exposure.

Irradiated beryl stones are appreciated while dealing with it. For gamma-ray and beta particle emissions, using a germanium spectrometer and Geiger-Muller counter. Where "Thermo FH 40 G" radiometer model "DB-033-961017 E" exposure rate measuring in addition to "FH 40 TG tele-probe" are used for measurements.

In this study, we need radiation measuring devices, which are divided according to:

A- Radiation worker.

B- A general public member.

The most important of these devices

1. A Portable Radiometer:

This device is used to measure the dose rate as well as the total dose which can be displayed and also to measure the average rate in addition to the possibility of storing the values that were measured or transferring them to computers. This device is shown in the figure (1).

Fig.1. portable radiometer.

2. The Portable Telescopic Probe.

This device allows the measuring of gamma dose rate and beta contamination of external radiation areas. It is connected to a portable measuring device, thus allowing measurement at a variable distance, which reduces direct

exposure. This device is equipped with an acoustic alarm. The acoustic alarm is activated upon reaching either alarm level. These levels can be adjusted and activated when a certain radiation level is reached. This device is shown in the figure (2).

Fig.2. portable telescopic probes.

2. The Large Area Surface Contamination Meter (LACSM) is a device used to measure surface contamination with alpha, and beta, particles. In addition to gamma, It is a battery-operated-measuring unit that is quick, easy to use, and has a large memory for reading isotopes while measuring contamination. This device is shown in the figure (3)

Fig.3. Large Area Surface Contamination Meter (LACSM).

Results and Discussion

1-analyzed by (EDS & LA-ICP-MS) Displaying pre-irradiation results:

The elemental composition of natural beryl is observed through analysis by the Energy-dispersive X-ray Spectroscopy (EDS). The elements presented in the different samples are observed as shown in Table (1), where the table shows the average concentrations of these elements in three selected samples. This is to select the best group that contains elements and traces with the shortest half-life. The main components of the beryl stone are the elements (O, Si, Mg, Al, S, Cl, Cr, Ti, Fe, and Zn. and the half-life of these elements ranges between a few seconds to a few hours., This means that the half-life of the beryl sample elements is short, so it is easy to use these raw beryl stones as gemstones for irradiation since their components do not need a long time to be stored (after treatment with neutron irradiation) to get rid of the remaining radioactive.

Table (1) Elemental analysis % of Beryl samples by EDS.

Element	Wadi Sket	Homret Akarem	Homret Mekbid
O	50.62	52.11	48.97
Na	1.22	0.31	0.37
Mg	1.39	--	--
Al	8.96	12.77	11.95
Si	36.11	33.83	32.87
S	0.04	0.08	--
Cl	0.11	0.10	0.08
K	0.13	0.07	0.07
Cr	0.23	--	--
Ti	--	--	0.15
Fe	1.19	0.51	5.39
Zn	--	0.22	0.15
Total	100	100	100

2-Analysis of Beryl samples after irradiation: Results of neutron activation analysis.

When studying the gamma spectrum of beryl samples in the cases of short and long irradiation based on the NAA technique qualitatively and quantitatively, as shown in Figures (4), and (5) of the gamma spectrum, we notice the gamma spectrum of short-irradiated beryl that it consists of nuclides of isotopes with short half-lives and limited activities such as (Rh-106M, Sb-126, Cl-38, Cs-138, Na-24,) and accordingly these activities disappear within days.

This is also noted in Table (2) which shows the half-lives and concentrations of these radioactive isotopes resulting from irradiation with neutrons. We conclude from this that beryl is one of the preferred stones in the gemstone industry through treatment in the reactor. On the other hand, for long-irradiation as shown in figure (5) of the gamma radiation spectrum using the NAA technique of long irradiated beryl samples other nuclides or in other words radioactive isotopes (Ag-110m, Ta-182, Rh-106M, Br-82, Ac-228, Rb-89, Sr-91, Sb-122, Sb-126, Co-56, La-140, Au-198, Bi-214) are resulting from irradiation with different concentrations and activity (in Becquerels) which are shown in detail in Table (3) besides other isotopes with negligible concentrations, where the isotopes with short half-lives have been disappeared and the isotopes with long half-lives such as Ta-182 and Ag-110m remained to disappear, Which require a longer time for cooling, or in other words, longer period is required for the radiation level to drop to the permissible level for handling

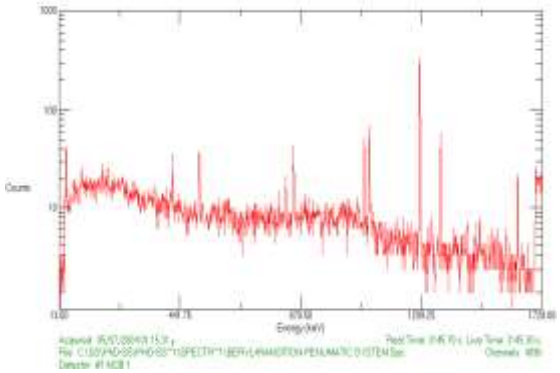

Fig.4. Beryl irradiated short radiation.

Table (2) Elemental analysis of beryl irradiated short radiation by using the NAA technique.

Isotope	Element Name	T ½ (Half-Life)	Energy (KeV)	Intensity /100 Decay (Branching Ratio)
Rh-106M	Rhodium	2.2 hr.	511.80	86.40
Sb-126	Antimony	12.5 d	695.10	99.70
Cl-38	Chlorine	37.24 min.	1642.40	32.80
Cs-138	Cesium	33.41 min.	1435.86	76.30
Na-24	Sodium	14.96 hr.	1368.55	100.00

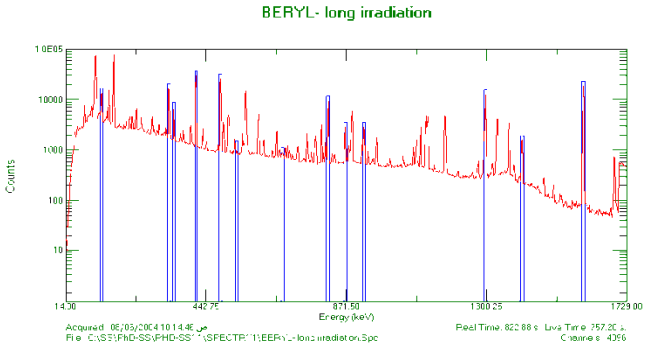

Fig.5. Beryl irradiated long radiation.

Table (3) Isotopes in beryl irradiated long radiation.

Element	Product Nuclide	Energy(keV)	T 1/2 (Half-Life)	Activity (Bq)
Silver	Ag-110m	818.02	249.850d	21323.92
Ta-182	Tantalum	114.740d	1121.28	195663.04
Terbium	Rh-106M	511.80	72.10d	144661.03
Bromine	Br-82	1317-43	35.300hr.	1799.67
Actinium	Ac-228	835.60	6.130 hr.	364586.64
Rubidium	Rb-89	2196.00	18.7d	13333.99
Strontium	Sr-91	555.57	9.500hr.	694.26
Antimony	Sb-122	564..08	2.700 d	3923.41
Antimony	Sb-126	605.00	2.400 d1	76691.99
Cobalt	Co-56	1175.13	78.760 d	107928.47
Lanthanum	La-140	1596.20	40.220 hr.	6695.73
Gold	Au-198	411.80	2.696 d	9923.85
Bismuth	Bi-214	1729.60	19.90m	85047.73

This analysis was performed after two days of cooling.

3-Radiation Protection Measures

After irradiating the beryl stones with neutrons (the sample was exposed to neutron radiation for 8 hs for long irradiation), which in turn led to the activation of trace elements in the stones and an increase in radioactivity. Therefore, the risks to the consumer must be assessed by identifying and estimating the number of radionuclides resulting from the radiation and the amount of accompanying exposure. Indeed, the samples were analyzed and the emissions of gamma and beta radiation in addition to the dose rate to which the user is exposed were estimated. As mentioned in figure (6) which shows the relationship between dose rate and time, it is noted that the radioactivity, or exposure to gamma, decreases until it reaches 3.5 µsv/h after 8 days from the end of the irradiation period. (Which was measured using Tele-probe).

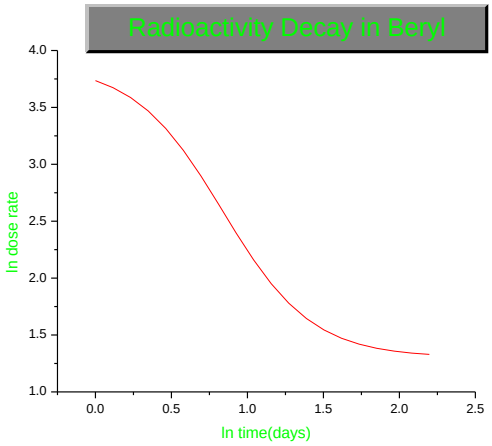

Fig.6. Variation of ln (dose rate in µsv/h) with ln time in days by using Tele-probe.

Also, concerning measuring the surface contamination of the beryl samples after treatment with neutrons, The contamination of the surfaces of the samples was measured using monitor "CONTAMAT" FHT 111 M, and as shown in the figure (7), the level of surface contamination reaches its lowest value after 8 days from the end of the irradiation period.

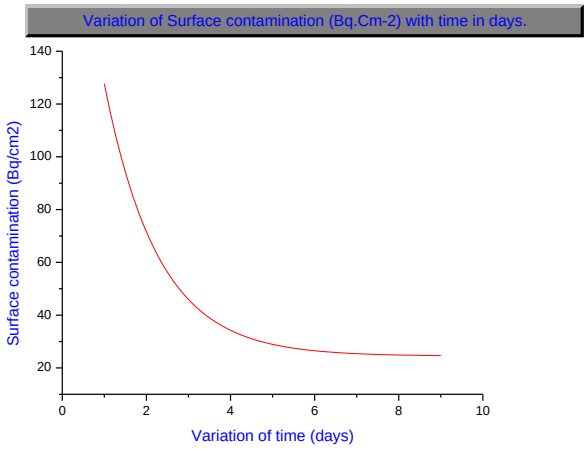

Fig.7. Variation of surface contamination (Bq/cm2) with a variation of time in days by using contamat.

The alpha content of raw samples was determined during a long time of counting in the hours range, using a CONTAMAT. No alpha activity above the background was detected. The CONTAMAT FHT 111 M was used for these measurements.

As shown in Figure (8), the color of beryl was created and enhanced after long neutron irradiation to take on a green color.

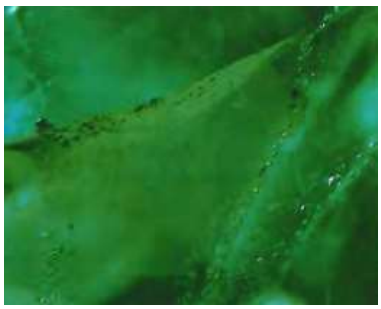

Fig.8. The treated beryl by neutron to induce a green color.

Cooling time calculations

Calculations were done using the FORTRAN Program depending on the Newton-Raphson method. The program was designed on "Microsoft Developer Studio, featuring Fortran Power Station" software. In this regard, the total activity of the stone is (A) which is the sum of the activities of its components, and all that is required is the time of cooling which verifies the transportation limit of activity which is 74(Bq/g) represents the safe level transportation on air according to IATA regulations. Where

$$f(\chi) = \sum_{i=1}^{n} A_i e^{-\lambda i x i} \quad \rightarrow (1)$$

$$f = 74 \text{ Bq / g} \quad \rightarrow (2)$$

$$f \setminus (\chi) = -\sum_{i=1}^{n} \lambda_i A_i e^{-\lambda i \chi i} \quad \rightarrow (3)$$

From Newton Raphson algorithm a better approximation x_1 is:

$$\chi_1 = \chi_0 - \{[f(\chi_0)]/[f/(\chi_0)]\} \quad \rightarrow (4)$$

So;

$$\chi_1 = \chi_0 - [A(\chi_0) / A\setminus(\chi_0)] \quad \rightarrow (5)$$

$$\chi_2 = \chi_1 - [A(\chi_1) / A\setminus(\chi_1)] \quad \rightarrow (6)$$

And so on till:

$$\chi_n = \chi_{n-1} - [A(\chi_{n-1}) / A\setminus(\chi_{n-1})] \quad \rightarrow (7)$$

Apply the condition that the error must be achieving the following;

$$x_n - \chi_{n-1} \leq \sum 10^{-4} \quad \rightarrow (8)$$

Then the value of χ_n is obtained when A = 74 Bq / g

t_{n+1} is a better approximation than t_n for the root x of the function f.

Where t is the time (in yr) required for activity to reach the safety level for transportation by air according to IATA regulations (74 Bq/g)

Since Beryl was irradiated in a nuclear reactor contains many types and amounts of radionuclides as reported before and according to Table (3). It takes to be releasable, (based on this previous program) after radioactive decay 1.38 years, while it takes a little more than two years after irradiation in some cases.

Time in years = 1. 38 yr.

Raman Spectroscopy Measurements:

Spectroscopic techniques such as Raman Spectroscopy are used to give useful information about the structure of minerals and their water content; and to identify color centers. Aiming to understand color processes in treated beryl samples. Beryl stones were subjected to extensive Raman spectroscopy in raw form and then after neutron irradiation. Spectra of all samples were recorded in the range 460 cm^{-1} -1160 cm^{-1} (as a wavelength) and the effect of irradiation time on changes in the spectral structure was studied. As shown in the figures, we find that the Raman spectrum for raw beryl shows bands at different wave numbers as in Figure (9). It was found that these bands are changed and shifted to the right after irradiation as is clear in Figures (10),

In light of the crystal structure, the impact of neutron flux in the range of ($0.1x10^{14}$ n /cm^2/s) on beryl can lead to point defects due to the movement of the SiO bonds in the crystal structure of beryl and by increasing the period of irradiation, this irradiation leads to increasing and accumulating of these point defects with their increased movements of the SiO bonds and which in turn leads to optical absorption in the visible region. As shown in Figure (10), scattered peaks were recorded at wavelengths between 127.6 and 465.1, which correspond to the modes of stretching and vibration of the SiO chemical bond.

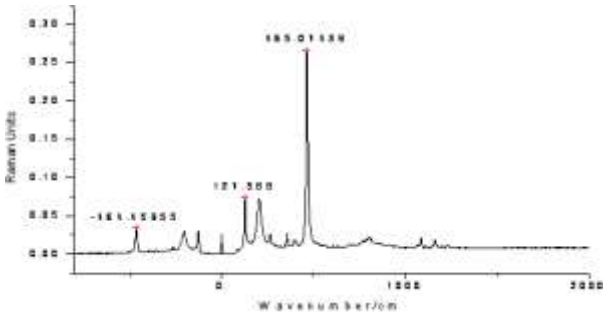

Fig.9. Raman spectrum of raw beryl.

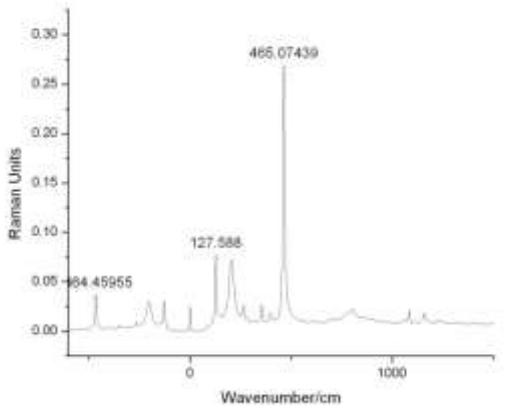

Fig.10. Raman spectrum of beryl irradiated for (8) hours.

It is clear from figures (9),(10), and in general that the intensity of the bands in beryl irradiated spectra with neutrons is higher than the intensity of the bands in raw beryl due to the strong effect of neutrons, as neutrons are more capable of ionization and can penetrate the atoms and the entire crystal structure and change the crystal structure. Also, neutrons interact with the SiO groups, which leads to distortions and thus to a change in the absorption of colors.

4 Conclusion

Beryl is one of those stones whose properties can be enhanced by radiation and become more attractive like other gemstones. Therefore, it becomes highly sought after in commercial markets and its monetary value increases. Since irradiating gemstones requires a high-value neutron flux, the ideal way for this purpose is through research reactors. Therefore, in this research work, the second Egyptian research reactor was used for this purpose, and thus, beryl samples were exposed to a high-value neutron flux in the irradiation positions around the reactor core.

In this research, the experimental work was conducted as follows. First: the best beryl samples were selected for the experiment by analyzing their components using the methods previously mentioned in the research to ensure ease of analysis and study of the irradiation components and subsequent protection measurements after irradiation.

Second: the samples were irradiated in appropriate irradiation positions around the reactor cores, according to the required neutron flux, and for the specified time periods. Third: after the irradiation was completed, the samples were extracted and left for the specified cooling periods of time to be subsequently analyzed using the various analytical methods mentioned in the research. After taking internationally agreed-upon radiation protection measurements for safe handling of the product, the beryl was marketed as a new gemstone product. Finally, a mathematical model was developed to help evaluate the required cooling time periods for the irradiated samples, as one of the important issues in the assigned experimental work.

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