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# Characterization of Commercial Corundum and Beryl: A Raman Spectroscopic Insight

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ABSTRACT

In order to enhance the beauty of commercially available corundum (of different colours) and beryl (mainly emerald), these are often subjected to treatments, both permanent (heat treatment, irradiation etc.) as well as temporary (glass filling and filling with temporary dyes). The objective of this paper is to use the spectrum obtained from 785 nm wavelength lasers of Raman spectroscopy to study these temporary treatments and enhancements (glass filling and filling with temporary dyes) in commercial gemstones in contrast to their natural untreated counterparts. The results of these studies show that the two groups of stones, though apparently identical in appearance, have distinctive signature features when observed under microscope and also gives definitive Raman signatures that separates the enhanced ones from the natural untreated ones.

## I. Introduction:

Laser Raman Spectroscopy was utilized to study the two most commonly prevailing gemstone treatments: glass filling and dyeing, from a more scientific point of view. The spectrum obtained from 785 nm wavelength laser was utilized to study 381 number of commercial treated gemstones in contrast to 475 number of natural untreated ones, which are as follows: 127 number of Natural Blue Sapphires against 99 number of blue glass-filled Natural Sapphires; 172 number of Natural Ruby against 180 number Red/Pink/Orange glass filled Natural Ruby; 72 number of Natural Yellow Sapphire against 45 number of yellow glass filled Natural Sapphire and 105 number of Natural Emerald against 57 number of natural beryl that have been dyed with green coloured oils. The comparison of the number of samples studied of each type is shown in the Fig.1.

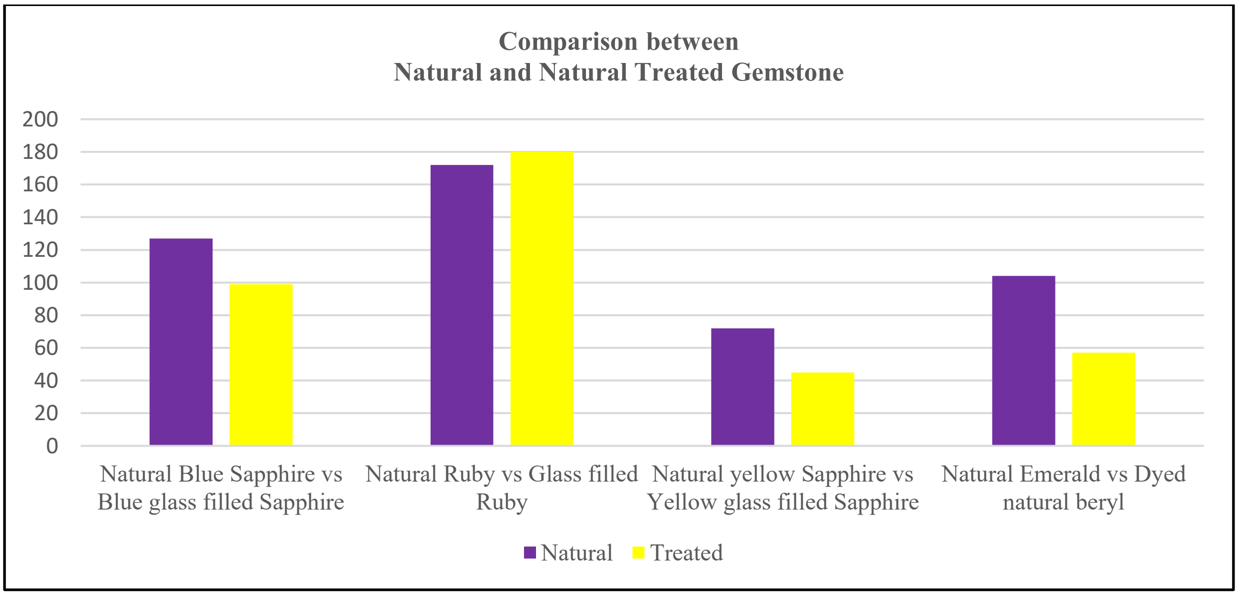

Fig.1: Comparison between Natural and Natural Treated Gemstone

The gemstones examined in this study, including both natural and treated specimens, are cut, polished, and faceted, with variable and undocumented geographic and geological origins. These two categories dominate the Indian gem market and were therefore selected to provide scientific insight into the most prevalent gem treatment practices.

**Methodology:**

Laser Raman Spectroscopy was employed to analyze the vibrational and structural properties of gemstone samples using a Renishaw InVia Reflex Laser Raman Spectrometer housed at the Central Petro logical Laboratory (CPL), Geological Survey of India, Kolkata. This non-destructive analytical technique is based on the Raman effect, in which monochromatic laser light interacts with a sample and a small fraction of the scattered radiation undergoes energy shifts corresponding to molecular vibrations. These shifts produce a characteristic spectral fingerprint that enables identification of mineral phases and the detection of inclusions, zoning, and crystal-structure variations in gemstones.

The Spectrometer consists two laser sources: a 514.5 nm Argon-ion laser (grating: 2400 lines/mm) and a 785 nm diode laser (grating: 1200 lines/mm). Depending on the optical properties and fluorescence sensitivity of the sample, the 785 nm diode laser was utilized for the studies. The laser beam was focused on the gemstone surface using a 50× long working distance objective lens, ensuring high spatial resolution and minimal sample damage. The laser power was adjusted between 2–10 mW, depending on the sensitivity of the gemstone to laser-induced heating.

Spectral acquisition involved three accumulations of 10 seconds each; with a spectral range covering 100 to 3500 $cm^{-1}$, for corundum and 100 to 2000 $cm^{-1}$, for beryl, ensuring the detection of both low and high frequency vibrational modes. The spot size of the laser beam (~1.2 µm) allowed precise targeting of mineral zones and inclusions within the gemstones. The room temperature during measurements was maintained at 22 ± 1°C to ensure thermal stability of the system.

All Raman spectra were processed using Renishaw WiRE 3.4 software, which included baseline correction, cosmic ray removal, and peak fitting using Gaussian-Lorentzian deconvolution methods to resolve overlapping bands. Instrument calibration was verified using a standard silicon wafer, with the characteristic Raman peak at 520 $cm^{-1}$, maintaining an accuracy of $\pm 1$ $cm^{-1}$.

To account for internal heterogeneity and potential crystal-orientation effects, multiple measurements were obtained from different locations on each gemstone. This approach yielded a representative spectral dataset, enabling reliable mineral identification and evaluation of crystallinity, symmetry, and lattice distortion. This analytical protocol enabled high-resolution, non-destructive characterization of commercially available corundum of various colors and beryl, primarily emerald, demonstrating its suitability for gemological and mineralogical investigations.

Prior to Raman spectroscopic analysis, the samples were examined microscopically, where their distinguishing features were readily observed. However, the Raman spectra provided more robust and scientifically substantiated vibrational information than that obtained from qualitative photo micrographic observations.

## Results:

Raman spectroscopic analysis of various gemstone varieties revealed distinct vibrational modes characteristic of their crystal structures and, treatment histories, wherever applicable. The spectra of natural untreated corundum group gemstones—including ruby, blue sapphire, and yellow sapphire—exhibited consistent and well-defined peaks associated with the trigonal crystal structure of α-$Al_2O_3$ (corundum), whereas in case of beryl, the peaks are in consistency to its hexagonal crystal system.

The Raman spectra and photomicrographs presented in this paper are representative of the gemstone variety groups examined, as each group exhibits major spectral peaks common to all samples within that category and are consistent in almost all the samples present in that particular group.

The photomicrographs of natural corundum and beryl, together with their treated counterparts, are representative images selected from individual samples within each group of the numerous pristine natural and treated specimens examined. Consequently, the features illustrated may commonly occur in combination. The images highlight the most prominent and diagnostically significant characteristics observed in each group.

### Natural Untreated Ruby

When observed under microscope, one sample of natural ruby shows the occurrence of mineral grains as the most prominent feature (Fig 2a).

The Raman spectrum of natural, untreated ruby displays prominent peaks at 378 $cm^{-1}$, 416 $cm^{-1}$, 643 $cm^{-1}$, and 752 $cm^{-1}$ (Fig. 2b), corresponding to the vibrational modes of the Al–O lattice in the corundum structure, with minor shifts potentially influenced by trace $Cr^{3+}$ substitution, which imparts the red coloration in ruby.

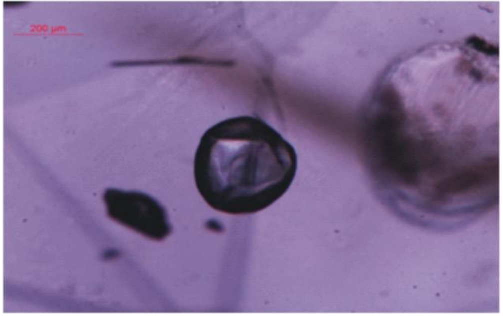

Figure 2a : Mineral grain in an untreated Natural Ruby

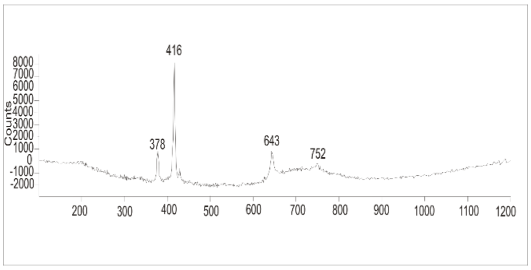

Fig.2b: Raman Spectrum of a Natural Untreated Ruby

## Natural Untreated Blue Sapphire

Just like natural untreated ruby, one of the natural untreated blue sapphires also showed the presence of a trail of mineral grains as inclusions (Fig 3a).

The Raman spectral features of untreated blue sapphire are very similar to that of untreated ruby, with intense peaks at 373 $cm^{-1}$, 416 $cm^{-1}$, 645 $cm^{-1}$, and 751 $cm^{-1}$ (Fig. 3b). The minor shift in peak positions, as compared to the natural untreated ruby is attributed to $Fe^{2+}$ and $Ti^{4+}$ trace element substitutions typical in blue sapphires.

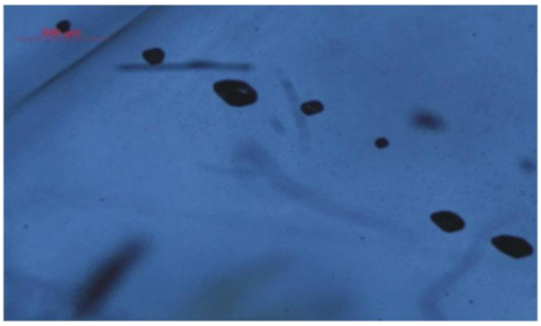

Figure 3a: Mineral grains in an untreated natural blue sapphire

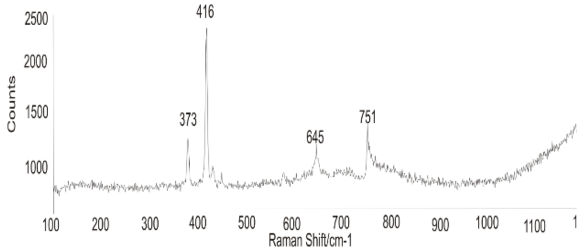

Fig.3b: Raman Spectrum of a Natural Untreated Blue sapphire

## Natural Untreated Yellow Sapphire

One natural yellow sapphire shows the presence of feathers and straight growth lines under microscope (Fig. 4a), both being significant natural signatures besides mineral grains, as seen in natural ruby and blue sapphire.

Yellow sapphire also exhibits typical corundum Raman bands at 378 $cm^{-1}$, 416 $cm^{-1}$, 648 $cm^{-1}$ and 750 $cm^{-1}$ (Fig. 4b), consistent with the $Al_2O_3$ lattice.

Fig. 4a: Feathers and straight growth lines in untreated natural yellow sapphire

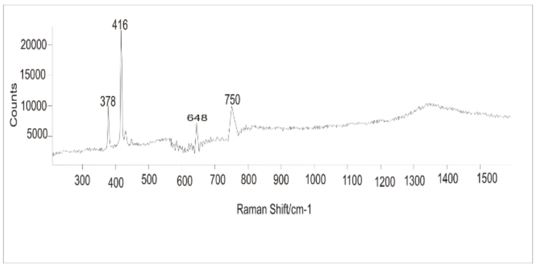

Fig.4b: Raman Spectrum of a Natural Untreated Yellow Sapphire

## Glass-Filled Corundum (Treated Stones)

In contrast, Raman Spectrum from the glass-filled fractures of corundum specimens show all the characteristic peaks of natural corundum, along with additional broad hump-like peaks, indicative of the presence of glassy material:

The pink-red to orange glass-filled ruby shows orange-blue flash effects along the filled fractures as a result of light being reflected from the contact surface between the corundum and the glass, because of the difference in their refractive indices. In Raman spectroscopy, along with the characteristic peaks for corundum, a broad peak is observed at 1560 $cm^{-1}$ (Fig. 5b).

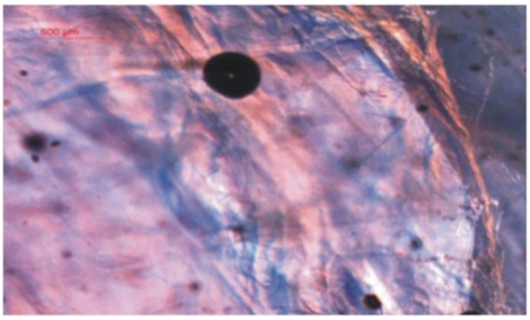

Fig. 5a: Glass filled ruby showing one contraction bubble and blue-orange flash effects

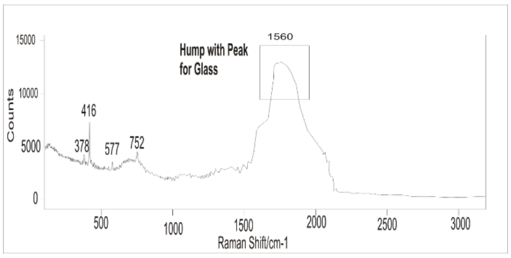

Fig.5b: Raman Spectrum showing an additional hump at 1560 $cm^{-1}$ in Natural Glass Filled Ruby

For blue glass-filled sapphire, orange coloured flash effect is shown due to the difference between the refractive indices of corundum and the filler glass. In Raman Spectrum, a hump like feature occurs at 544 $cm^{-1}$ (Fig. 6b).

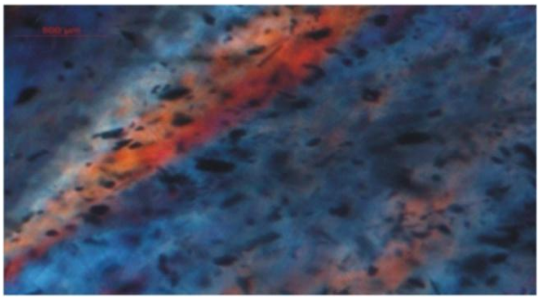

Fig. 6a: Glass filled sapphire showing orange flash effects

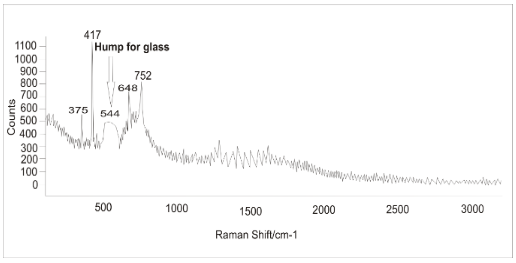

Fig.6b : Raman Spectrum study showing a hump at 544 $cm^{-1}$in Natural Glass filled sapphires (blue)

In the yellow glass-filled sapphire, a clear boundary was observed which had the yellow coloured glass filling on one side, thus making that part of the stone yellow, whereas the other part was devoid of any filling, resulting in no coloration in that part (Fig. 7a). The yellow glass filled part shows an elevated and broader signal at 1570 $cm^{-1}$ (Fig. 7b).

These humps are absent in natural corundum samples and likely result from the glass filling material, confirming treatment.

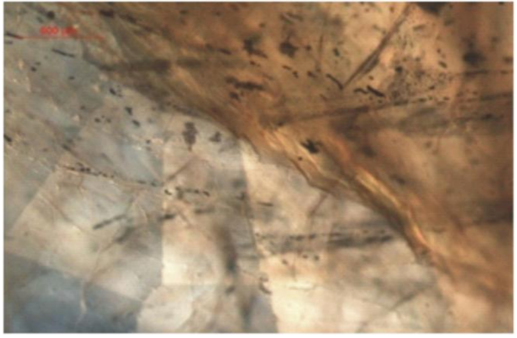

Figure7a: Lead-glass filled sapphire showing yellow-orange flash effect

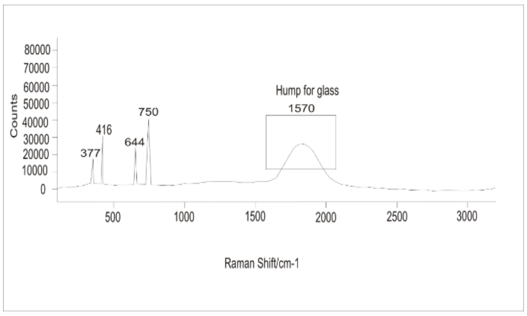

Fig.7b : Raman Spectrum showing a hump at 1570 $cm^{-1}$in Glass filled sapphire (yellow)

## Natural Untreated Emerald

Under microscope, one of the natural emerald sample shows the presence of several fluid inclusions and mineral grains (Fig. 8a). Raman analysis of natural, untreated emerald reveals prominent peaks at 321$cm^{-1}$, 393 $cm^{-1}$, 670$cm^{-1}$, 683 $cm^{-1}$ and 1067 $cm^{-1}$ with two less intense peaks at 518 $cm^{-1}$ and 1021 $cm^{-1}$(Fig. 8b), consistent with the vibrational modes of the beryl structure ($Be_3Al_2Si_6O_{18}$).

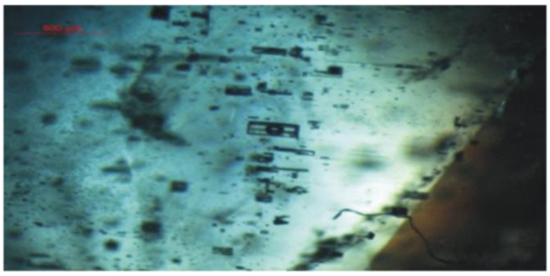

Figure 8a: Mineral grains and fluid inclusions in Natural untreated Emerald.

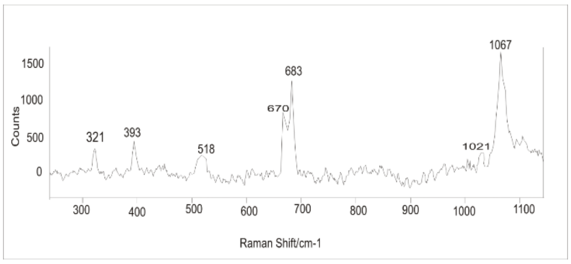

Fig.8b : Raman Spectrum of a Natural Untreated Emerald

## Dyed Beryl

In case of beryl (originally colourless) that have been dyed with a green coloured filler, is observed under microscope, colour concentration is seen only along the fractures whereas the rest of the gemstone remains colourless (Fig.9a).

In Raman Spectroscopy, the spectrum obtained from the filled fractures shows that in addition to the standard beryl peaks as observed in untreated natural beryl (natural emerald), dyed beryl show several broad and weak intensity bands in the higher wavenumber region between 1200 $cm^{-1}$ and 1650 $cm^{-1}$ (Fig. 9b). These features are not intrinsic to natural beryl and are likely related to the organic dye compounds or treatment residues used for colour enhancement.

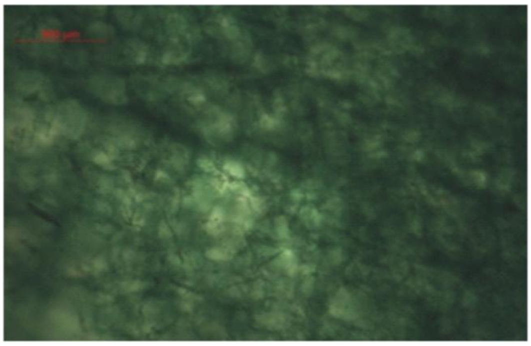

Figure 9a: Dyed Natural Beryl

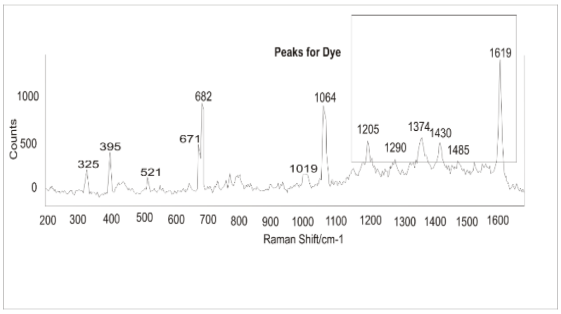

Fig.9b : Raman Spectrum showing additional peaks for Dyed Beryl

## Discussions:

## Glass filling in Corundum:

To enhance the marketability of non-gem-quality corundum, silica-glass filling treatments were introduced in the 1980s. (McClure et.al., 2006[1]). However, because the refractive index of silica glass is significantly lower than that of corundum, fractures often remained visible even after filling. During the 90s, the idea of lead glass filling (red, pink, orange and yellow in colour) was introduced (McClure et.al. 2006[1]), followed by blue coloured cobalt glass in 2007 (Lalous, 2013[2]). Because lead glass and cobalt glass have specific gravity values similar to that of corundum and more closely matched refractive indices, fractures filled with these materials became less visible. Consequently, such treatments were increasingly adopted as more effective methods for enhancing the appearance of corundum. The low quality stones are heated up to a temperature of 900°C to 1400°C in order to remove the impurities (mostly the matrix materials). The stones are subsequently mixed with oxide powders, primarily silica and lead oxides, although sodium, potassium, calcium, and metal oxides such as vanadium and bismuth may also be present. Next the stones are subsequently heated in a controlled atmosphere using electric furnaces. This process fuses the powders into coloured molten glass, which then penetrates the fractures within the stone, imparting colour to a colourless variety of mineral (Pardieu et al., 2010[3]).

In case of ruby and yellow sapphire (Choudhary, 2014[4]), the glass is lead glass where lead acts as a coloring agent (McClure et.al. 2006[1]), whereas in case of blue sapphire the coloring agent used is cobalt glass (Smith, 2013[5]).

During Raman Spectroscopic Analysis, it is found that the samples of red and/or orange and/or yellow glass-filled corundum produce an extra hump between 1560 to 1570 $cm^{-1}$, while the blue glass filled ones produces hump around 544 $cm^{-1}$, in addition to the pre-existing original peaks of corundum. In contrast to the sharp, well-defined peaks as produced by corundum due to their crystallinity, the "hump" type of broad Raman bands produced by the "fillers" indicates their amorphous and non-crystalline nature.

The broad hump like band obtained for red/orange/yellow glass filled ruby or yellow coloured corundum are in concurrence to the study done by Fan et. al. in 2009[6] and Bersani & Lottici, 2010[7], where it was shown that the lead glass filling of ruby shows a broad band at 1563 $cm^{-1}$, thus confirming the fillers studied by the authors to be of "lead glass".

In case of the blue glass filled corundum, the broad band was obtained at 544$cm^{-1}$. In the works done by Pevenage et. al. in October, 2013[8] and Dahiana et. al. in 2023[9], it was found that glasses coloured by cobalt for the blue colour, develops a broad band in Raman Spectrum around 520 cm-1, thus confirming that the blue glass fillers observed by the authors in corundum to be of cobalt glass.

**Dyeing of beryl:**

Dyeing is another age-old technique used to enhance the color of gemstones. Emeralds from most natural sources contain numerous fractures. The practice of "oiling" green gemstones—where emeralds are immersed in organic oils to reduce the visibility of fractures—was first described by Pliny the Younger in 55AD and became common by 14th century (Johnson et. al., 1999[10]). Natural emerald has a specific gravity of 2.72. Emerald mines are often associated with the production of colorless beryl, which has the same specific gravity of 2.72 and is commonly used in gemstone treatment processes. Colorless or poorly colored beryl is treated with fillers—typically colorless oils supplemented with green dyes, producing a "green-colored oil"—to enhance color and reduce the visibility of fractures. In this treatment, the stones are first subjected to an acid bath to remove impurities from the fractures. The gemstones are then placed in a heated hydraulic chamber containing cedar oil and tightly sealed. Heat liquefies the cedar oil, and hydraulic pressure drives it into the fractures, reducing their visibility. In many cases, dyes are added to the cedar oil, producing a green-colored oil that penetrates the fractures of the colorless beryl, thereby imparting an overall green hue to the stone while simultaneously masking fracture visibility (Ringsrud, 1983[11]).

In Raman analysis, along with the common peaks for beryl, dyed beryl produces numerous peaks from 1200 $cm^{-1}$ to 1650 $cm^{-1}$ (Fig. 8), in response to the presence of dye (Hanni et. al, September, 1996[12], Picture two and Hanni et. al., 1997[13], Fig. 4a & 4b). The peaks are also matching with the study done by Keifert & Hanni in 2001[15] (Figure 14), where green artificial coloration was identified using Raman Spectroscopic analysis within quartzite.

The findings of this paper has been summarised in Table 1.

**Table 1: Summary of the studies done**

| **Gemstone type** | **Treatment** | **Key Diagnostic Raman Features** |
|---|---|---|
| Natural Glass filled Ruby | Fractures filled by pink-red to orange lead glass | Broad band observed at 1560 cm$^{-1}$ for the lead glass filling, along with the characteristic peaks for corundum at 378 cm$^{-1}$, 416 cm$^{-1}$, 643 cm$^{-1}$, and 752 cm$^{-1}$. |
| Natural (blue) Glass filled sapphire | Fractures filled by blue coloured cobalt glass | Broad band observed at 544 cm$^{-1}$ for the cobalt doped blue glass along with the characteristic peaks for corundum at 373 cm$^{-1}$, 416 cm$^{-1}$, 645 cm$^{-1}$, and 751 cm$^{-1}$. |
| Natural (yellow) Glass filled sapphire | Fractures filled by yellow coloured lead glass | Broad band observed at 1570 cm$^{-1}$ for the yellow coloured lead glass along with the characteristic peaks for corundum at 378 cm$^{-1}$, 416 cm$^{-1}$, 648 cm$^{-1}$ and 750 cm$^{-1}$. |
| Natural Dyed Beryl | Fractures filled by green coloured oil | Numerous peaks from 1200 cm$^{-1}$ to 1650 cm$^{-1}$ in addition to the characteristic peaks for beryl at 321cm$^{-1}$, 393 cm$^{-1}$, 670cm$^{-1}$, 683 cm$^{-1}$ and 1067 cm$^{-1}$ and two less intense peaks at 518 cm$^{-1}$ and 1021 cm$^{-1}$ |

## Conclusion:

All the treatments discussed in this study produce only temporary improvements in gemstone quality. For example, the glass fillings introduced within corundum are prone to get damaged due to regular wear and tear where the stones are exposed to regular household chemicals (aerosol oven cleaner, ammonia, bleach, and even concentrated lemon juice) and even get removed, thus bringing back all the fissures and fractures that were originally present in that low quality gemstone (Pardieu et.al., 2010[3], McClure et.al., 2006[1]). Similarly, the oil and associated dye fillings of emeralds are also susceptible to damage caused by heat or sunlight.

The study clearly differentiates untreated corundum group gemstones, from their glass-filled counterparts, by an additional broad hump-like Raman bands due to the presence of foreign glassy material. Similarly, natural emeralds are characterized by sharp peaks of beryl while dyed beryl displays additional peaks associated with organic dyes or treatment residues.

Therefore, laser Raman spectroscopy not only facilitates the identification of natural gemstones from their treated counterparts but also plays a critical role in detecting enhancements, thereby supporting transparency and integrity in gemological evaluation and the gemstone trade.

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