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Mineralogical and Gemmological Profiling of Corundum: Distinguishing Karur Natural Rubies from Synthetic Counterparts

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Abstract: We present a comparative study of natural gem-quality ruby from the Karur region, Tamil Nadu, and synthetic ruby powders prepared via sol-gel thermolysis. Using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and gemmological analysis, we characterized the structural, chemical, and physical properties of these materials. XRD analysis confirmed orthorhombic crystal structure in all samples, with natural ruby showing significantly larger crystallite sizes (372.5-425.2 nm) compared to synthetic samples (29.9-47.2 nm). FTIR spectroscopy revealed characteristic absorption bands at 3600-3400 cm^{-1} (OH stretching), 1650 cm^{-1} (OH bending), and 430-635 cm^{-1} (metal-oxygen bonds), confirming the corundum framework with chromium incorporation. SEM imaging showed dense microstructures with distinct grain morphologies between natural and synthetic samples. Gemmological properties of natural ruby included Mohs hardness of 9, refractive index of 1.762-1.778, density of 3.97-4.05 g/cm^3, and strong carmine red fluorescence. Our findings demonstrate that high-purity synthetic ruby can be produced cost-effectively at relatively low temperatures, offering insights for both gemstone authentication and industrial applications.

Keywords: *Ruby, Corundum, X-Ray Diffraction, FTIR Spectroscopy, Sol-Gel Synthesis, Gemmological Properties, Karur Region.*

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I. INTRODUCTION

Ruby has captivated humanity for millennia. In Sanskrit, it's called "ratnaraj"—the king of precious stones—and ancient texts from India to Burma speak of its mystical powers [1]. Beyond its cultural significance, ruby is scientifically fascinating: it's corundum (Al_2O_3) colored by chromium substitution, ranking 9 on the Mohs hardness scale and second only to diamond in durability [2]. The finest rubies, displaying the coveted "pigeon's blood" red, can command prices exceeding those of equivalent diamonds [3].

The Karur region in Tamil Nadu has a rich history of gemstone deposits, and interestingly, the word "corundum" itself derives from the Tamil "kurundam" [4]. This linguistic connection underscores the region's long-standing relationship with these precious minerals. Today, understanding the differences between natural and synthetic rubies is crucial not only for gemmological authentication but also for expanding industrial applications in lasers, medical devices, and abrasives [5], [6].

While natural ruby forms under extreme geological conditions over millions of years, synthetic ruby can be produced through various methods including flame fusion (Verneuil process), flux growth, hydrothermal synthesis, and sol-gel techniques [7], [8]. The sol-gel method, in particular, offers advantages of low processing temperatures, high purity, and excellent compositional control [9]. However, comprehensive comparative studies using multiple analytical techniques on rubies from the Karur region remain limited.

This study addresses that gap by characterizing natural gem-quality ruby from Karur alongside synthetic ruby powders prepared via sol-gel thermolysis. We employed XRD to determine crystal structure and crystallite size, FTIR to identify chemical bonds and functional groups, SEM to examine microstructure, and standard gemmological tests to measure physical properties. Our goal was to establish a robust analytical framework for distinguishing natural from synthetic ruby while evaluating the quality of sol-gel-derived materials for potential applications.

II. MATERIALS AND METHODS

A. Materials

Natural ruby samples were obtained from local miners in the Karur region, Tamil Nadu. For synthetic ruby preparation, we used aluminium nitrate nonahydrate ($Al(NO_3)_3 \cdot 9H_2O$, 98% purity), chromium sulphate ($Cr_2(SO_4)_3$, 99% purity), sucrose ($C_{12}H_{22}O_{11}$, analytical grade), polyvinyl alcohol (PVA, molecular weight ~125,000), and nitric acid (HNO_3, 65% concentration). All chemicals were procured from standard suppliers and used without further purification.

B. Synthesis of Ruby Powders

We synthesized ruby powders using the sol-gel thermolysis method, which involves polycondensation reactions between PVA and saccharic acid (formed by nitric acid oxidation of sucrose) [10]. The process began by dissolving aluminium nitrate and chromium sulphate in deionized water at calculated stoichiometric ratios. We prepared samples with varying chromium concentrations (0%, 2%, 4%, 6%, 8%, and 10% Cr) to study the effect of dopant concentration.

PVA solution (10 wt%) was added to the metal salt solution under continuous stirring. Sucrose was then introduced, followed by dropwise addition of nitric acid to initiate oxidation. The mixture was heated to 80°C and maintained for 6 hours to form a viscous gel. This gel was dried at 150°C for 2 hours, yielding a solid precursor. The dried material was calcined at 550°C for 5 hours in air to decompose organic components and form the oxide phase.

C. Characterization Techniques

- *X-Ray Diffraction (XRD):*

We performed XRD analysis using a Bruker AXS D8 Advance diffractometer with Ni-filtered Cu Kα radiation ($\lambda = 1.5406$ Å). Phase identification was accomplished by comparing experimental d-spacing values with JCPDS standards (04-0880 for natural ruby, 04-0878 for Al_2O_3, and 77-2188 for chromium-doped alumina). We calculated average crystallite size using the Scherrer equation:

$$\mathbf{L = (0.9\lambda \times 180) / (\pi \times FWHM \times \cos\theta)}$$

where L is the crystallite size, λ is the X-ray wavelength, FWHM is the full width at half maximum in degrees, and θ is the Bragg angle [11].

- *Fourier Transform Infrared Spectroscopy (FTIR):*

FTIR spectra were recorded on a JASCO FTIR-4000 series spectrometer in diffuse reflectance mode (DRIFTS) over the range 4000-400 cm^{-1}. We identified characteristic absorption bands corresponding to hydroxyl groups and metal-oxygen bonds [12].

- *Scanning Electron Microscopy (SEM):*

Surface morphology and microstructure were examined using a scanning electron microscope. Samples were mounted on aluminium stubs and sputter-coated with gold to prevent charging. We captured images at various magnifications to assess grain size, porosity, and overall microstructural features.

- *Gemmological Analysis:*

Natural ruby samples underwent standard gemmological testing including hardness measurement (Mohs scale), refractive index determination (using a refractometer with sodium D-line illumination), density measurement (hydrostatic weighing method), and fluorescence observation under long-wave UV light [13].

III. RESULTS AND DISCUSSION

A. X-Ray Diffraction Analysis

- *Phase Identification and Crystal Structure*

XRD patterns of all samples revealed sharp, well-defined peaks characteristic of crystalline corundum structure. Natural ruby from Karur exhibited prominent peaks at $2\theta = 35.0°$ and 66.4°, corresponding to the (104) and (116) crystallographic planes, respectively. These values matched closely with JCPDS card 04-0880, confirming the sample as genuine corundum with chromium substitution.

Pure synthetic Al_2O_3 (0% Cr) showed major peaks at $2\theta = 37.4°$, 45.5°, and 66.9°, indexed to the (311), (321), and (441) planes of the orthorhombic structure (JCPDS 04-0878). As chromium concentration increased from 2% to 10%, we observed systematic peak shifts and intensity variations. The 8% Cr-doped sample displayed peaks closely resembling those of natural ruby, suggesting successful incorporation of chromium into the alumina lattice.

The experimental d-spacing values calculated from Bragg's law ($n\lambda = 2d \sin\theta$) agreed well with standard values, with deviations less than 0.02 Å in most cases (Table 1). This close agreement confirms the high crystallinity and phase purity of both natural and synthetic samples. Interestingly, we noticed that chromium doping caused slight lattice contraction, evidenced by small shifts in peak positions toward higher 2θ values. This is consistent with the smaller ionic radius of Cr^{3+} (0.615 Å) compared to Al^{3+} (0.535 Å) when considering octahedral coordination [14].

Table 1: XRD Data for Selected Samples

Sample	**2θ (JCPDS)**	**2θ (Exp.)**	**d-spacing (JCPDS)**	**d-spacing (Exp.)**	**hkl**
Natural Ruby	35.15	35.0	2.552	2.562	104

Sample	2θ (JCPDS)	2θ (Exp.)	d-spacing (JCPDS)	d-spacing (Exp.)	hkl
Natural Ruby	66.52	66.4	1.405	1.408	116
Pure Al_2O_3	37.77	37.4	2.380	2.402	311
Pure Al_2O_3	45.86	45.5	1.977	1.992	321
Al_2O_3 + 8% Cr	35.20	35.1	2.548	2.555	104
Al_2O_3 + 8% Cr	66.48	66.5	1.406	1.405	116

- *Crystallite Size Analysis*

Crystallite size is a critical parameter that influences optical transparency, mechanical strength, and overall material quality. Using the Scherrer equation, we calculated average crystallite sizes from the most intense diffraction peaks (Table 2). Natural ruby exhibited remarkably large crystallites, ranging from 372.5 to 425.2 nm, reflecting the slow geological processes that formed these crystals over millions of years.

In contrast, synthetic samples showed much smaller crystallite sizes. Pure Al_2O_3 had an average size of 47.2 nm, while chromium-doped samples ranged from 29.9 to 42.8 nm. The 8% Cr-doped sample, which most closely resembled natural ruby in color and XRD pattern, had a crystallite size of 38.5 nm—more than ten times smaller than natural ruby.

Table 2: Crystallite Size Data

Sample	2θ (°)	FWHM (°)	Crystallite Size (nm)
Natural Ruby	35.0	0.021	425.2
Natural Ruby	66.4	0.024	372.5
Pure Al_2O_3	37.4	0.189	47.2
Al_2O_3 + 2% Cr	35.2	0.236	37.8
Al_2O_3 + 4% Cr	35.3	0.251	35.5
Al_2O_3 + 6% Cr	35.2	0.268	33.3
Al_2O_3 + 8% Cr	35.1	0.232	38.5
Al_2O_3 + 10% Cr	35.3	0.298	29.9

This size difference has important implications. Larger crystallites in natural ruby contribute to superior optical clarity and gem quality, as light scattering at grain boundaries is minimized. The smaller crystallites in synthetic samples, while potentially limiting transparency, offer advantages for certain industrial applications where high surface area and reactivity are desirable [15].

We also observed that increasing chromium concentration generally decreased crystallite size, likely due to chromium ions disrupting the growth of alumina crystallites during synthesis. This effect has been reported in other doped oxide systems and reflects the complex interplay between dopant concentration and crystal growth kinetics [16].

B. FTIR Spectroscopy

FTIR spectroscopy provided complementary information about chemical bonding and functional groups present in the samples. All spectra displayed three main regions of absorption: 3600-3400 cm⁻¹, around 1650 cm⁻¹, and 430-635 cm⁻¹.

The broad absorption band at 3600-3400 cm⁻¹ corresponds to O-H stretching vibrations from hydroxyl groups. These groups can be present as adsorbed water molecules or as structural hydroxyl defects within the corundum lattice [17]. Natural ruby showed a relatively weak band in this region, consistent with its high crystallinity and low defect concentration. Synthetic samples exhibited stronger OH bands, particularly those calcined at lower temperatures, indicating residual water or incomplete condensation of the oxide network.

The peak at approximately 1650 cm⁻¹ is attributed to the bending vibration of water molecules (H-O-H). Its intensity correlated with the strength of the OH stretching band, confirming the presence of molecular water rather than isolated hydroxyl groups in some samples [18].

The most diagnostic region for corundum is 430-635 cm⁻¹, where metal-oxygen (M-O) bond vibrations occur. Natural ruby displayed characteristic peaks at 630 and 550 cm⁻¹, assigned to Al-O stretching vibrations in octahedral coordination [19]. Pure synthetic Al_2O_3 showed similar peaks at 620 and 550 cm⁻¹, with slight shifts due to differences in crystallite size and lattice strain.

Chromium-doped samples exhibited additional complexity in this region. The 8% Cr-doped sample showed peaks at 635 and 550 cm⁻¹, with the higher-frequency peak shifted slightly compared to pure alumina. This shift reflects the presence of Cr-O bonds, which have different force constants than Al-O bonds due to the different mass and electronic structure of chromium [20]. The aluminum chromite sample ($AlCrO_3$) displayed distinct peaks at 545 and 430 cm⁻¹, indicating a different crystal structure with mixed Al-Cr octahedral sites.

Table 3: FTIR Band Assignments

Sample	OH Stretching (cm⁻¹)	OH Bending (cm⁻¹)	M-O Bonds (cm⁻¹)
Natural Ruby	3600-3400 (weak)	1650 (weak)	630, 550
Pure Al_2O_3	3600-3400 (medium)	1650 (medium)	620, 550
Al_2O_3 + 2% Cr	3600-3400 (medium)	1650 (medium)	625, 550
Al_2O_3 + 4% Cr	3600-3400 (medium)	1650 (medium)	628, 550
Al_2O_3 + 8% Cr	3600-3400 (medium)	1650 (medium)	635, 550
$AlCrO_3$	3600-3400 (weak)	1650 (weak)	545, 430

The FTIR results confirm successful chromium incorporation into the alumina lattice and provide a rapid, non-destructive method for distinguishing between pure and doped corundum samples. The technique is particularly useful for quality control in synthetic ruby production.

C. Scanning Electron Microscopy

SEM imaging revealed striking differences in microstructure between natural and synthetic samples. Natural ruby displayed well-developed crystal faces with smooth surfaces and minimal porosity. The grains were large and tightly packed, consistent with the large crystallite sizes determined by XRD. This dense microstructure contributes to the excellent transparency and optical properties of gem-quality ruby.

Pure synthetic Al_2O_3 showed a fine-grained microstructure with relatively uniform particle distribution. Some porosity was evident, particularly in samples calcined at 550°C without subsequent sintering. The grain size appeared to be in the range of 50-100 nm, slightly larger than the crystallite size from XRD, suggesting that individual grains may consist of a few crystallites.

Chromium-doped samples exhibited microstructures similar to pure alumina, with homogeneous distribution of chromium throughout the material. We did not observe any secondary phases or chromium-rich regions, indicating good solid solution formation. The 8% Cr-doped sample showed slightly larger grains than samples with higher chromium content, consistent with the crystallite size trend observed in XRD.

The aluminium chromite sample displayed a dense microstructure with well-defined grain boundaries. The grains were larger and more angular than those in the doped alumina samples, reflecting the different crystal structure and formation mechanism of this mixed oxide phase.

These microstructural observations help explain the optical properties of the materials. Natural ruby's large, defect-free grains allow light to pass through with minimal scattering, producing the brilliant transparency prized in gemstones. Synthetic samples, with their smaller grains and residual porosity, tend to be more opaque or translucent, though this can be improved through high-temperature sintering and hot pressing [21].

D. Gemological Properties

Standard gemological testing of natural ruby from Karur confirmed its authenticity and gem quality (Table 4). The hardness of 9 on the Mohs scale places ruby among the hardest natural materials, making it highly resistant to scratching and ideal for jewelry applications [22]. This exceptional hardness arises from the strong ionic and covalent bonding in the corundum structure.

Table 4: Gemological Properties of Natural Ruby

Property	Value	Method
Hardness	9 (Mohs scale)	Scratch test
Refractive Index	1.762 - 1.778	Refractometer
Density	3.97 - 4.05 g/cm³	Hydrostatic weighing
Cleavage	None	Visual inspection

Property	Value	Method
Transparency	Transparent	Visual inspection
Birefringence	-0.008	Polariscope
Luster	Vitreous	Visual inspection
Fluorescence	Strong carmine red	UV lamp (365 nm)

The refractive index (RI) of 1.762-1.778 is characteristic of corundum and allows for positive identification using a standard gemmological refractometer. The birefringence of -0.008 indicates that ruby is uniaxial negative, a property that can be observed using a polariscope [23]. These optical properties result from the anisotropic crystal structure of corundum.

Density measurements yielded values of 3.97-4.05 g/cm³, consistent with published data for natural ruby. This relatively high density reflects the close packing of aluminium and oxygen atoms in the corundum structure. Synthetic rubies typically have similar densities, making this property less useful for distinguishing natural from synthetic stones, though glass-filled or composite rubies will show lower densities [24].

One of the most distinctive features of our Karur ruby samples was their strong carmine red fluorescence under long-wave UV light (365 nm). This fluorescence arises from chromium ions in the crystal lattice, which absorb UV radiation and re-emit it as visible red light. The intensity and color of fluorescence can provide clues about chromium concentration and the presence of other trace elements [25]. Strong red fluorescence is generally considered a positive indicator of natural ruby, though some synthetic rubies also fluoresce strongly.

The absence of cleavage in ruby is another important property. Unlike minerals that break along specific crystallographic planes, ruby fractures conchoidally (like glass), which contributes to its durability. The vitreous (glass-like) lustre results from the high refractive index and smooth crystal faces.

E. Comparative Analysis: Natural vs. Synthetic Ruby

Our multi-technique characterization reveals both similarities and differences between natural and synthetic ruby. Both materials share the same fundamental crystal structure (orthorhombic corundum) and chemical composition (chromium-doped aluminium oxide). XRD patterns show matching peak positions, confirming identical crystal symmetry and lattice parameters.

However, several key differences emerge. The most obvious is crystallite size: natural ruby has crystallites more than ten times larger than synthetic samples. This difference reflects the vastly different formation conditions—millions of years of slow geological processes versus hours of laboratory synthesis. The larger crystallites in natural ruby contribute to superior optical quality and transparency.

FTIR spectroscopy reveals that synthetic samples contain more hydroxyl groups and adsorbed water than natural ruby, particularly when calcined at lower temperatures. This is expected, as the sol-gel process involves aqueous solutions and organic precursors that can leave residual OH groups. High-temperature sintering can reduce these defects, but complete elimination is challenging.

Microstructurally, natural ruby shows well-developed crystal faces and minimal porosity, while synthetic samples have finer grains and some residual porosity. This affects not only optical properties but also mechanical properties and chemical stability. For gemstone applications, natural ruby's microstructure is clearly superior. However, for certain industrial applications—such as abrasives or catalysts—the high surface area of fine-grained synthetic ruby may be advantageous [26].

From a practical viewpoint, our results demonstrate that sol-gel thermolysis can produce high-purity chromium-doped alumina at relatively low temperatures (550°C calcination, compared to 1200-1300°C for conventional solid-state synthesis). This offers potential cost savings and energy efficiency. The ability to precisely control chromium concentration allows tuning of colour and optical properties for specific applications.

IV. CONCLUSIONS

This study provides a comprehensive characterization of natural gem-quality ruby from the Karur region, Tamil Nadu, and synthetic ruby powders prepared via sol-gel thermolysis. Our key findings include:

- *Crystal Structure:*

  Both natural and synthetic samples crystallize in orthorhombic corundum structure, with XRD patterns matching JCPDS standards. Chromium successfully substitutes for aluminium in the lattice without forming secondary phases at concentrations up to 8%.

- *Crystallite Size:*

  Natural ruby exhibits significantly larger crystallites (372-425 nm) compared to synthetic samples (30-47 nm), reflecting different formation mechanisms and timescales. This size difference strongly influences optical transparency and gem quality.

- *Chemical Bonding:*

  FTIR spectroscopy confirms the presence of Al-O and Cr-O bonds characteristic of corundum. Synthetic samples contain more hydroxyl groups and adsorbed water than natural ruby, particularly at lower calcination temperatures.

- *Microstructure:*

  SEM imaging reveals dense, well-crystallized microstructure in natural ruby versus finer-grained, slightly porous microstructure in synthetic samples. Chromium distribution is homogeneous in doped samples.

- *Gemmological Properties:*

  Natural Karur ruby displays classic corundum properties: Mohs hardness 9, refractive index 1.762-1.778, density 3.97-4.05 g/cm³, and strong carmine red fluorescence under UV light.

- *Synthesis Optimization:*

  Sol-gel thermolysis enables production of high-purity chromium-doped alumina at relatively low temperatures (550°C), offering advantages in cost and energy efficiency compared to conventional methods.

The Karur region continues to be a significant source of gem-quality corundum, and understanding the characteristics of these natural rubies helps establish benchmarks for quality assessment. Meanwhile, advances in synthetic ruby production open new possibilities for industrial applications in optics, electronics, and mechanical systems where the unique properties of corundum are valuable.

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