NOTES & NEW TECHNIQUES

A NEW TYPE OF COMPOSITE TURQUOISE

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Several conspicuously colored specimens marketed as dyed and/or stabilized turquoise were recently examined. They were purple to purple-pink, yellow-green, and blue, in veined and unveined varieties. Testing revealed that all the specimens were composites of turquoise pieces bonded together; the purple to purple-pink and yellow-green samples were bonded with a colored polymer, and the blue ones had a colorless polymer. This article presents the gemological properties and EDXRF and FTIR analyses of this material, along with its reaction to the solvents acetone and methylene chloride.

Turquoise, best known for its splendid blue color, has been used for adornment since antiquity. Chemically a hydrous copper aluminium phosphate, it has a cryptocrystalline structure composed of fine, randomly oriented groups of triclinic crystals (Webster, 1994). This cryptocrystalline structure gives rise to the gem's porosity, making it susceptible to body oils, ordinary solvents, or even dirt, which can alter its color. For decades, turquoise has been impregnated with wax, plastics, or polymers—a process known as stabilization, which enhances not only the material's durability but also its color and surface luster (Nassau, 1994). Another established treatment is the dyeing of pale-colored turquoise to give it a rich blue color (see, e.g., Kammerling, 1994). In recent years, a few other proprietary treatments have been developed for turquoise. The most prominent is Zachery treatment, which decreases the porosity of the original material, so it takes a better polish; the blue color may also be enhanced through an additional step in the process (Fritsch et al., 1999). Yet another form of treatment seen on the market, developed by Eljen Stones, involves polymer impregnation (Owens and Eaton-Magaña, 2009).

Recently, this author had the opportunity to study a group of distinctively colored purple and yellow-green turquoise samples, as well as blue turquoise (figure 1). When first seen at the Jaipur Jewellery Show in December 2008, their market availability was limited. Since the beginning of 2010, however, the Gem Testing Laboratory of Jaipur has received an increasing number of these treated turquoises for identification. While previous studies have reported on dyed and impregnated blue turquoise (Kammerling, 1994) and green turquoise produced by immersion in mineral oil (Koivula et al., 1992), these specimens were quite different. Upon inquiry, the supplier said they were pieces of natural turquoise that had been dyed and bonded together, though he knew little about the process involved. He did divulge that this material is being manufactured in the United States. We subsequently received a variety of rough and cut samples for study, and this article reports on the properties of these samples, some of which appear similar to those seen by McClure and Owens (2010) in the GIA Laboratory.

MATERIALS AND METHODS

The 21 samples were purple to purple-pink, yellow-green, and blue. Fifteen were fashioned as cabochons weighing 8.10–21.83 ct (again, see figure 1), and the other six were rough pieces with a total weight of 47.90 g (figure 2). The samples were divided into seven groups of three each on the basis of bodycolor, the presence/absence of veining, and whether they were rough or fashioned. The groups were: unveined purple to purple-pink rough, unveined yellow-green rough, unveined and veined purple to purple-pink cabochons (two groups), unveined and veined yellow-green cabochons (two groups), and veined blue cabochons.

Standard gemological tests were performed on all the cabochons to determine their RI, hydrostatic SG, and UV fluorescence. Absorption spectra were observed with a desk-model GIA Prism 1000 spectroscope. A binocular gemological microscope with fiber-optic lighting in addi-

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GEMS & GEMOLOGY, Vol. 46, No. 2, pp. 106–113.

Figure 1. The 15 treated turquoise cabochons examined for this study are divided here into veined and unveined (or minimally veined) varieties that are purple to purple-pink (11.82–21.83 ct), yellow-green (8.10–18.12 ct), and blue (9.78–20.10 ct). Photo by G. Choudhary.

tion to the overhead white light was used to examine the structure and surface features. A needle was used to probe various parts of both the rough and cut samples.

We also noted the reaction of two samples to solvents such as acetone and methylene chloride. Acetone is commonly used to check for the presence of dye in gem materials, and methylene chloride is an excellent solvent for polymers. We brushed an acetone-dipped cotton swab on an unveined yellow-green cabochon and soaked a piece from the same sample in acetone for 48 hours to observe the changes; the rest of this sample was retained for comparison. We also soaked one veined yellow-green cabochon in methylene chloride for 48 hours and noted the reaction at occasional intervals. For comparison, a natural untreated turquoise was immersed in methylene chloride for the same duration.

Figure 2. These rough samples of treated turquoise (5.75–10.11 g) show unusual colors and a polymer-like luster. Concentrations of colored polymer are visible as the yellow-brown area on the right corner of the upper yellow-green sample, and the red areas on the bottom purple to purple-pink samples. Photo by G. Choudhary.

We performed qualitative chemical analyses on all 15 cabochons with energy-dispersive X-ray fluorescence (EDXRF) spectroscopy, using a PANalytical Minipal 2 instrument operated with a voltage of 15 kV and current of 0.016 mA.

Infrared spectra were recorded on all cabochons in the 6000–400 cm^{-1} range with a Shimadzu IR Prestige 21 Fourier-transform infrared (FTIR) spectrometer operating at room temperature in diffuse reflectance mode, using a standard resolution of 4 cm^{-1} and 50 scans per sample. The results were then converted to absorbance using the spectrometer software.

RESULTS AND DISCUSSION

Visual Characteristics. As mentioned earlier, the turquoise samples were purple to purple-pink, yellow-green, and blue (again, see figures 1 and 2). The purple to purple-pink range is not associated with turquoise and indicates artificial coloration. The veins present contained metallic gold-colored "matrix" that generally appeared much brighter than that seen in natural turquoise. Except for the yellow-green unveined variety, the specimens displayed an uneven body color. The purple to purple-pink and veined yellow-green specimens displayed some distinct blue patches representing the original turquoise before treatment. These features were indicative of dyeing. The blue samples did not exhibit any obvious signs of dyeing and had a more natural appearance, though the thick-

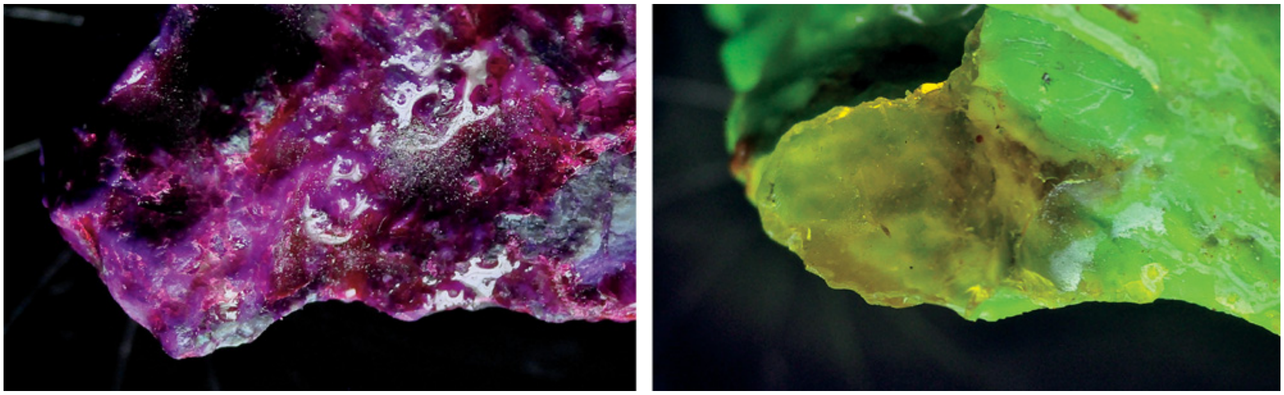


Figure 3. Concentrations of color appear on the surface of these rough samples of purple to purple-pink and yellow-green turquoise. The IR spectrum of the transparent yellow-brown area in the image on the right matched that of a polymer and some of the peaks recorded in the polished specimens. Photomicrographs by G. Choudhary; magnified 30×.

ness of the veins raised suspicions about their origin.

The rough purple to purple-pink specimens displayed obvious reddish concentrations on their surface (figure 3, left) and were easily identified as artificially colored; they also displayed a dull to waxy and oily luster, which is associated with polymers and resins. The color of the yellow-green rough was more even, with only one specimen displaying an obvious concentration of a transparent yellow-brown substance (figure 3, right). Similar concentrations of a transparent colored material were visible on the purple to purple-pink samples, too. The other two yellow-green pieces were fairly evenly colored, though they also displayed the luster associated with polymers and resins.

TABLE 1. Properties of natural turquoise and dyed composite turquoise.

Property	Natural turquoise (Webster [1994] unless noted otherwise)	Dyed composite turquoise (this study)		
Color	"Sky" blue to greenish blue (veined and unveined)	Purple to purple-pink (veined and unveined)	Yellow-green (veined and unveined)	Blue (veined)
Luster	Waxy	Dull to waxy		
RI	1.62 (mean)	1.61 (spot)	1.61 (spot) 1.54 for two samples, probably due to polymers	1.61 (spot)
SG	2.60–2.90	2.23–2.60	2.03–2.55	2.44–2.49
UV fluorescence				
Long-wave	Greenish yellow to bright blue	Strong patchy orange-red	Weak patchy blue; mostly inert	Strong patchy blue
Short-wave	Inert	Moderate patchy orange-red	Inert	Moderate patchy blue
Spectroscope spectrum	Vague band at 460 nm, fine lines at 420 and 432 nm	Bands in the green and yellow region at ~510, 540, and 580 nm	Diffused absorption in the blue region at 450–490 nm	No absorption features
Microscopic features	White clouds, crystals of various mineral inclusions (Fritz et al., 2007; Koivula and McClure, 2009)	Concentrations of red and yellow, patches of polymer, veins of "golden" material with or without brassy yellow fragments.		
EDXRF analysis	$CuAl_6(PO_4)_4(OH)_8{\bullet}4H_2O$ + Fe	Al, P, Fe, and Cu (and Zn in veined samples)		
FTIR analysis	Peaks at 1125, 1050, and 1000 cm^{-1}; band at 1746 cm^{-1} (e.g., Moe et al., 2005)	Peaks related to polymers in the 3200–2800 and 2000–1000 cm^{-1} regions		
Reaction to metal probe	None	Indentations due to lower hardness of polymer		
Reaction to solvents	Blue color turns green after contact with body oils and the like	Loss of the impregnated color when soaked in acetone; veined sample completely disintegrated into fragments when soaked in methylene chloride		

Gemological Properties. The gemological properties of the studied turquoise are described below and summarized in table 1.

Refractive Index. Except for two yellow-green specimens, all samples (whether veined or unveined) yielded a spot RI of approximately 1.61 with a moderate birefringence blink. This value is consistent with those reported for natural turquoise. Two anomalous yellow-green samples—one veined and one unveined—displayed spot RIs of ~1.54, and no birefringence was visible. This lower RI value could have been due to a thicker layer of polymer on the surface or to a larger percentage of polymer in the structure of the turquoise.

NEED TO KNOW

- Purple-pink, yellow-green, and blue composite turquoise has been marketed since late 2008.
- The pieces of turquoise are bonded together using a polymer (colored to produce the purple-pink and yellow-green varieties).
- Some of the turquoise has veins or "matrix" material composed of a gold-colored polymer with fragments resembling pyrite/marcasite.
- The composite turquoise can be identified by its microscopic appearance, UV fluorescence, spectroscope spectrum, and FTIR features.

Specific Gravity. The samples displayed a wide range of SG values, from 2.03 to 2.60. Webster (1994) and O'Donoghue (2006) reported SGs of 2.60–2.91 for untreated turquoise, depending on its porosity. Although porosity can cause fluctuations in SG readings, we did not observe such fluctuations in our samples, as expected for polymer-treated material. Only one of the 15 cut samples had an SG of 2.60, while the rest were below that. These lower values are consistent with the presence of a polymer. No consistent variations in SG were seen across colors or between veined and unveined samples.

UV Fluorescence. The samples showed distinct fluorescence reactions to long-wave UV radiation—which varied according to their bodycolor (figure 4)—and had weaker reactions or were inert to short-wave UV. The purple to purple-pink turquoise fluoresced a bright patchy orange-red to long-wave UV. The blue turquoise displayed a strong patchy blue fluorescence, while the yellow-green samples were mainly inert, with only the residual blue areas fluorescing blue. In all the samples, the veined areas were inert. Webster (1994) also reported greenish yellow to bright blue fluorescence to long-wave UV, and the patchy blue in these specimens likely corresponded to residual areas of natural blue turquoise.

Spectroscope Spectrum. The purple to purple-pink specimens displayed a series of three bands in the green and yellow regions at ~510, 540, and 580 nm; the intensity of the latter two bands was stronger than that of the 510 nm band. In the yellow-green samples, a broad diffused absorption was seen in the blue region between 450 and 490 nm. No absorption features were seen in the blue turquoise.

Magnification. Microscopic examination of the samples revealed the following features.

- *Luster:* Polymer-filled areas and cavities displayed a distinctly dull to waxy and oily luster compared to the turquoise areas (see figures 3 and 5). The areas of accumulated polymer in the cabochons also appeared to be indented.
- *Color Concentrations:* Most of the samples showed distinct color concentrations that varied according to bodycolor. The yellow-green turquoise displayed some yellow concentrations that formed swirly patches or followed veins (figure 6, left). The yellow appeared to be superimposed on the blue base of natural turquoise pieces, resulting in an overall green coloration. A few cavities in the cabochons were filled with the same yellow material. Similarly, the purple to purple-pink samples also displayed color concentrations (figure 6, right),

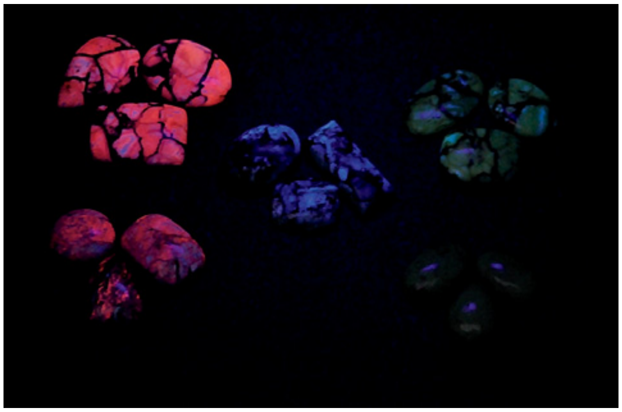


Figure 4. Here, the samples are shown in the same orientation as figure 1 during exposure to long-wave UV radiation. Their reactions varied with color. Note the striking orange-red fluorescence of the purple to purple-pink samples. The blue turquoise displayed a patchy blue fluorescence, while the yellow-green samples were inert with residual blue areas fluorescing blue. Photo by G. Choudhary.

Figure 5. Polymer-filled areas and cavities in the turquoise appear to be indented and display a dull-to-waxy luster. Photomicrograph by G. Choudhary; magnified 45×.

with areas of red present as patches and in veins and cavities. The color concentrations indicated that the yellow-green and purple turquoise varieties had been dyed, and the presence of deep ridges suggested that these samples—veined and unveined—were composites, consisting of individual pieces of turquoise held together by a colored polymer matrix. Also, the yellow-green sample that was broken prior to soaking in acetone displayed uniform color in the broken areas, indicating that the dye penetrated completely through the stone. The blue samples did not display any color concentrations, which indicated that they were not dyed; the polymer seen on the surface and in the ridges of those samples was colorless.

- *Veins:* Natural turquoise usually contains matrix formed by brown-to-black veins of limonite (O'Donoghue, 2006). Grains of pyrite/marcasite may also be present, often along the veins or in the matrix (Fritz et al., 2007). In the study samples, the veins consisted predominantly of a brighter "golden" yellow substance that was colored by fine flakes (figure 7). Commonly present in this substance were fragments with a duller brassy appearance. It appeared that pieces of pyrite/marcasite were embedded in a gold-colored polymer during the production of the artificial veins.
- *Reaction to a Metal Probe:* When probed with a needle to check for the presence of a polymer, both rough and cut samples readily indented. This test has been performed for many years to identify impregnated turquoise (see, e.g., Kammerling, 1994). The needle made indentations in the "golden" yellow veins or "matrix" as well (figure 7, right), revealing the softness expected for a polymer; the duller brassy fragments were not affected by the metal probe.

Reaction to Solvents. A cotton swab dipped in acetone and rubbed on the surface of one unveined yellow-green sample did not result in any loss of color. A piece of an unveined yellow-green sample that was soaked in acetone for 48 hours displayed an apparent loss of color and became patchy (figure 8, right sample), while the acetone became pale yellow-green. The soaked sample also had a significantly duller luster and showed a pimply surface and numerous cracks (figure 9).

A veined yellow-green sample that was soaked in methylene chloride showed significant changes after ~3 hours (figure 10). The metallic-appearing substance that formed the veins started to leave the specimen and showed a flaky appearance. After ~18 hours, individual pieces of the specimen were distinctly visible in the liquid, and the liquid turned slightly greenish yellow. After ~48 hours, the specimen had completely disintegrated into small grains and fragments, similar to the effect described by Rockwell (2008). The surface of the beaker in which the specimen was immersed also showed distinct color concentrations released from the sample after the methylene chloride evaporated. In contrast, a piece of natural untreated turquoise that was immersed in methylene chloride for comparison was unaffected.

The reaction in the methylene chloride bath and the similar structural features (e.g., the presence of deep ridges) seen in all samples confirmed they are composites and not merely dyed and/or impregnated. At this stage, we do not know what impact common household cleaning products and other solvents might have on the polymer(s) used in these composites.

EDXRF Analysis. Qualitative EDXRF analysis of all cabochons (veined and unveined) revealed the presence of Al, P,

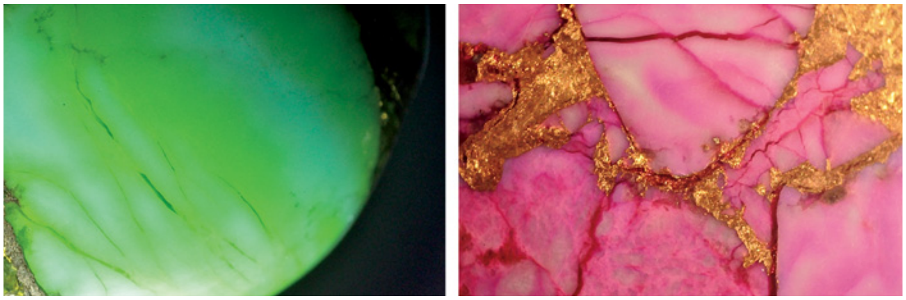


Figure 6. Concentrations of yellow (left, magnified 30×) and red (right, 45×) reveal the presence of an artificial dye. On the left, the yellow color superimposed on the blue base gives rise to a yellowish green bodycolor. Also note the deep ridges filled with color. Photomicrographs by G. Choudhary.

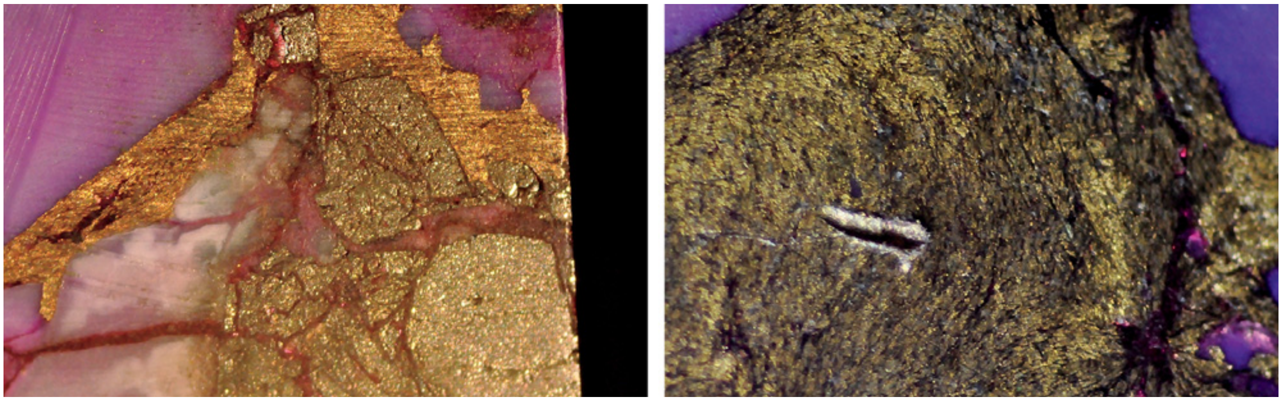


Figure 7. The veins or "matrix" in the turquoise appear to consist of a bright "golden" yellow material containing fragments with a brassy color resembling pyrite/marcasite (left). The photo on the right shows the tiny gold-colored flakes that color the matrix material; the gash made by the needle probe indicates its softness. Photomicrographs by G. Choudhary; magnified 30× (left) and 45× (right).

Figure 8. An 8.90 ct unveined yellow-green turquoise was broken and the portion on the right immersed in acetone for 48 hours, resulting in a duller luster and patchy loss of color. Photo by G. Choudhary.

Fe, and Cu, consistent with the chemical composition of turquoise. No potassium peak was present, which ruled out the possibility of Zachery treatment (Fritsch et al., 1999). Yet the veined samples displayed an additional Zn peak. EDXRF analysis of the "golden" metallic-looking material that flaked off after soaking a veined sample in methylene chloride (discussed above) confirmed the presence of Zn as well as Cu.

FTIR Analysis. FTIR spectroscopy has long been the most powerful tool in the nondestructive identification of polymer-treated stones (e.g., Fritsch et al., 1992). In this study, FTIR analysis of all cabochons (veined as well as unveined) displayed fairly consistent absorption features in the 3200–2800 cm^{-1} and 2000–1000 cm^{-1} ranges.

In the 3200–2800 cm^{-1} region, distinct absorption bands were visible at ~3125 (consisting of twin humps), 2970, and 2875 cm^{-1} (figure 11, left); the latter two are associated with polymers (Fritsch et al., 1992; Moe et al., 2005). However, no peak was detected near 3035 cm^{-1}, which suggests the absence of Opticon or other resins (Johnson et al., 1999).

The 2000–1000 cm^{-1} region (figure 11, right) displayed major absorption bands at ~1750, 1595, 1487, 1270, and 1060 cm^{-1}, in addition to some fine absorption features at ~1896, 1857, 1825, 1380, and 1193 cm^{-1}. Moe et al. (2005) stated that peaks at ~1125, 1050, and 1000 cm^{-1} are associated with natural (not synthetic) turquoise. In the

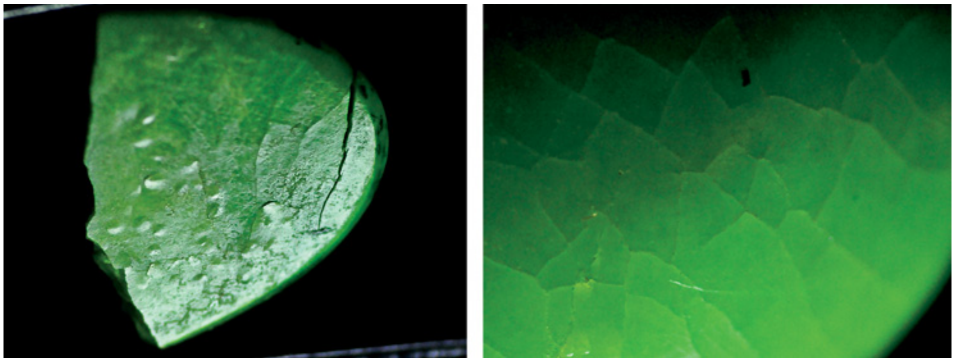


Figure 9. The immersed portion of the sample in figure 8 displayed a pimply surface (left, magnified 30×) and numerous cracks (right, 45×). Photomicrographs by G. Choudhary.

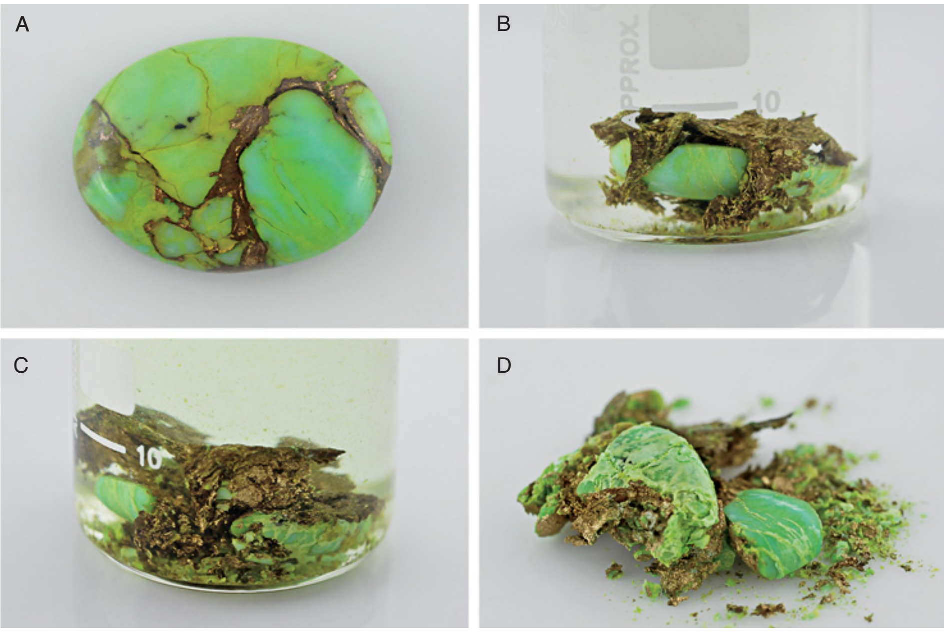


Figure 10. A veined 11.29 ct yellow-green sample (A) was soaked in methylene chloride. After ~3 hours, the metallic-appearing substance in the veins started to leave the specimen (B). After ~18 hours, separate pieces of the specimen were distinctly visible (C). After ~48 hours, the specimen completely disintegrated (D). Also note in C the color of the liquid, which turned slightly greenish yellow. Photos by G. Choudhary.

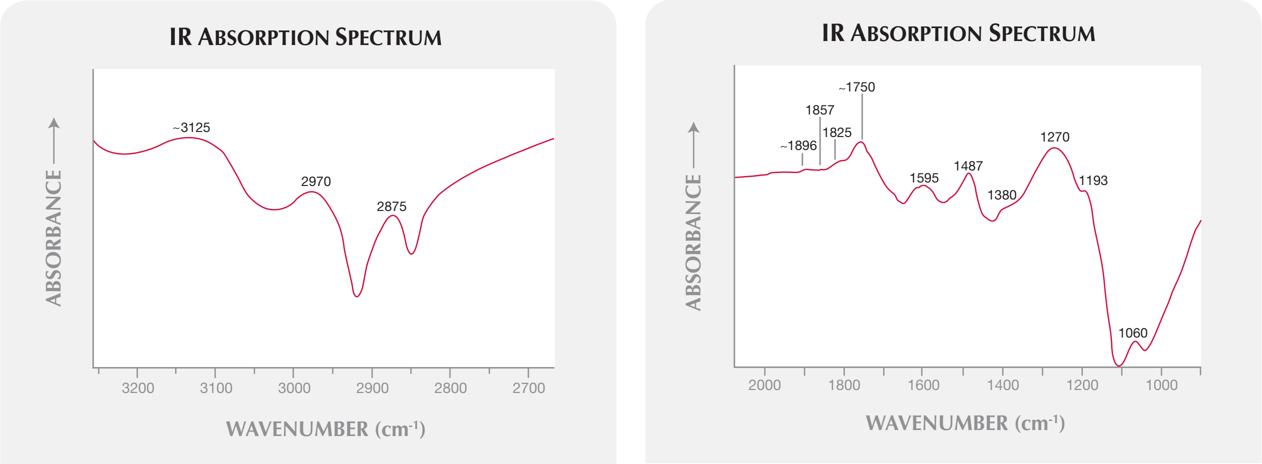
present samples, however, the peak at ~1050 cm^{-1} varied from 1075 to 1045 cm^{-1}, there was no 1125 cm^{-1} peak, and the peak at 1000 cm^{-1} was present at around 1015 cm^{-1} (not shown in figure 11). The bands at ~1750 and 1595 cm^{-1} are related to polymers, in which the former is assigned to C=O stretching (~1744) and the latter to CH$_3$ bending (~1600 cm^{-1}; Moe et al., 2007). The peak at ~1000 cm^{-1} is associated with the styrene phenyl ring, but it does not play an important role in the identification of a polymer, because it is also present in natural turquoise (Moe et al., 2005). The cause of the ~1487 and ~1270 cm^{-1} peaks is not known; the position of the latter varied from 1270 to 1240 cm^{-1}.

The FTIR spectrum of a yellow chip taken from the surface of a yellow-green sample in figure 3 displayed all the polymer-related peaks mentioned above. This confirms the presence of a polymer-like material within or on the surface of these turquoises. The spectrum of this chip displayed an additional peak at ~1270–1240 cm^{-1}, and the cause of this peak in the turquoise could have been the polymer.

CONCLUSION

These composites, typically marketed as "stabilized" turquoise, consist of small fragments of turquoise bond-

Figure 11. The infrared spectrum recorded for all cabochons in the 3200–2800 cm^{-1} region (left) displays distinct absorption bands at ~3125, 2970, and 2875 cm^{-1}; the latter two are associated with polymers. The spectrum in the 2000–1000 cm^{-1} region (right) also displays some polymer-related peaks, at ~1750, 1595, and 1270 cm^{-1}, as well as the 1060 cm^{-1} peak associated with natural (not synthetic) turquoise.



ed together with a colored polymer (in the case of the purple to purple-pink and yellow-green samples) or a colorless polymer (blue samples). Furthermore, veined samples contain interstitial areas formed of a gold-colored polymer containing fragments that resemble pyrite/marcasite.

These materials provide a wider range of turquoise colors for the consumer. Identifying them should not pose any problem. Careful microscopic examination along with UV fluorescence, spectroscope spectrum, and FTIR analysis should easily establish their dyed/composite nature.

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ACKNOWLEDGMENTS

The author thanks Dolphin Gems of Jaipur for providing the study samples, and Tomas Burke for arranging to get the stones from this collection.

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