

Research Article

Spectral Characterization of Pigment from the No. 1 Cave, Kizil Cave-Temple Complex

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The Kizil Cave-Temple Complex has been registered as a World Heritage site and was formerly a part of Kucha—one of the most powerful and prosperous regions of ancient China. The No. 1 Cave is of great significance due to its three surviving clay sculptures. The mural paintings inside the cave are experiencing severe degradation. Scientific methods such as optical microscopy (OM), scanning electron microscopy combined with energy dispersive X-ray analysis (SEM-EDS), Raman spectroscopy, Fourier transform infrared spectroscopy (FTIR), and X-ray diffraction (XRD) were applied to analyze the pigments and organic coating used in the No. 1 Cave. The results show that paratacamite, gypsum, and lapis lazuli were used as the green, white, and blue pigments, respectively. Poly-*n*-butyl methacrylate (PBMA) was used as an organic coating of the blue pigment and has accelerated the aging of the mural paintings. This study shares insights into the materials and techniques employed and assesses the preservation status of the mural paintings, providing scientific support for protection and restoration schemes.

1. Introduction

The Kizil Cave-Temple Complex (Kizil Grottoes) is located in Baicheng County, Xinjiang Province, China, which was formerly a part of Kucha—one of the most powerful and prosperous regions of ancient China (Figure 1). Because it was a fortress that connected China and other countries along the Silk Road, Kucha was the center of politics, economy, and culture. After Buddhism was introduced in Kucha, it developed rapidly and made Kucha the Buddhism center of the western regions. Many mural cave paintings have been found in the Kucha region (Buddha logo in Figure 1), and they are considered to be the earliest Buddhist mural cave paintings in China. The content of such paintings includes a combination of the local society with Buddhist stories, providing evidence for the investigation of Kucha's history [1].

The Kizil Grottoes were built during a period between the eastern Han Dynasty (25–220 CE) and the Song Dynasty (960–1279 CE) and have been registered as part of a World

Heritage site, the “Silk Roads: the Routes Network of Chang'an-Tianshan Corridor,” in 2014 [2]. The name “Kizil,” which means red in Uighur, is derived from the color of the sand and stone in the sunshine. A multitude of cultural factors from central China and India were included in the mural paintings, making the caves unique due to the distinct national characteristics.

The Kizil Grottoes has 236 numbered caves to date and is divided into 4 regions according to location. The No. 1 Cave is located in the western part of the valley and was found in 1973. Because most clay sculptures in the Kizil Grottoes are now preserved outside China, the three surviving clay sculptures in the No. 1 Cave are of great value, even though they are incomplete. The loss of the record of the construction year makes it impossible to directly determine the development sequence of the caves. Methods of archaeological typology combined with C14 detection are currently being used to understand the periodization of the caves. Based on an observation of the artistic style and configuration, the No. 1 Cave is considered to have been built in the

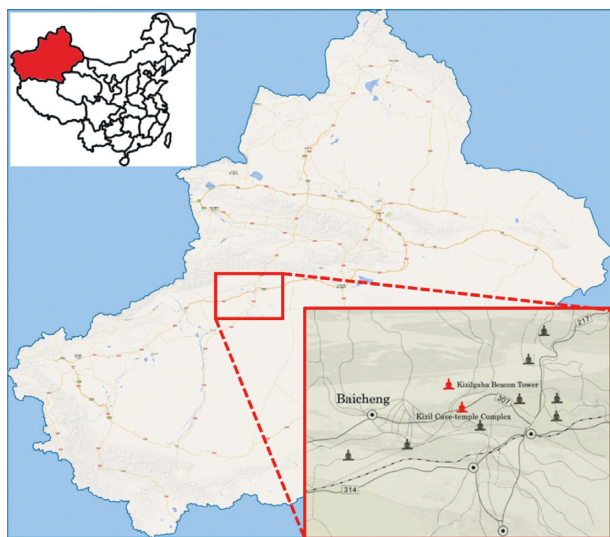


FIGURE 1: Distribution map of Kucha Caves.

period between the Northern and Southern Dynasties (5th–6th century CE) [3–5]. Degradation has occurred in both the mural paintings and the cave, resulting in the delamination and detachment of pigment from the related painting layers (Figure 2).

A technical study was conducted to obtain a better understanding of the content and craft of the mural paintings. This study will share insights into the materials and techniques employed and assess the preservation status of the mural paintings. Many mural painting studies have been reported. Nondestructive methods, such as stimulated infrared thermography based on the spectroscopic study of different paint layers, have been improved and have been used in the study of mural painting [6]. Optical microscopy is used to study cross sections to obtain the layer information of the paintings. Technical methods such as Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy combined with energy dispersive X-ray analysis (SEM-EDS), Raman spectroscopy, portable X-ray fluorescence (XRF), and X-ray diffraction (XRD) are used in the identification of the pigments in mural paintings [7–10]. Studies on paintings from Kucha have identified the use of lapis lazuli, cinnabar, lead oxide, red arsenic sulfides, copper green, bone black, white lead, gypsum, and calcite [11]. Pigment analysis of the Kizil Grottoes has revealed that minerals such as CaSO_4 and CaCO_3 were used for white pigment, atacamite and paratacamite were used for green pigment, and Pb_3O_4 , HgS , and Fe_2O_3 were used for red pigment [5]. Apart from the previous results, a study of pigment from the No. 40 cave has found malachite ($\text{CuCO}_3\text{-Cu(OH)}_2$) in the green pigment used there [12]. Gypsum, minimum, atacamite, and burnt bone (white, red, green, and black pigments) were discovered in Kizil paintings from Guimet Museum in Paris [13]. An analysis of the green pigment used in the No. 1 Cave has not yet been reported.

It should be noted that the No. 1 Cave contains the only mural painting without a ground layer in the Kizil Grottoes. Combined with the cave's existing unique clay sculptures,

this makes the No. 1 Cave of great significance within the Kucha area. Unlike other famous Buddhist grottoes in China (such as the Mogao Grottoes) that have been thoroughly researched, scientific studies of the Kizil Grottoes have rarely been reported. For the first time, an analysis of the pigment will reveal the craft of the mural paintings in the No. 1 Cave, and an assessment of the preservation condition of such paintings will also be performed. Our study will provide support for research in literature and art history, which may help solve currently unsolved puzzles.

2. Materials and Methods

2.1. Materials. Due to the great value of the No. 1 Cave, sample collection directly from the mural painting is forbidden. Three samples were obtained from the fallen part of the No. 1 Cave (one of the most important caves in the Kizil Grottoes) and were studied using several complementary techniques (Table 1). Green pigment and white pigment are present in the left-top part of the rear of the cave, while blue pigment is present in the right-top part. An organic coating was found on the surface of the blue pigment. Due to the length of time that the pigment has survived, degradation has occurred. Pigment loss can be observed in the green sample, which might be caused by degradation of the binding materials. According to a researcher from Xinjiang Kucha Academy, the green and white pigments were collected in areas that do not have ground layers, making them unique among other samples.

2.2. Methods

2.2.1. Microscopic Analysis. Microscopic studies were performed using a KEYENCE VHX-2000C microscope. Samples were observed under different magnifications. Samples with full layer structures were embedded in an epoxy resin, ground, and polished after curing. Unfortunately, due to the limited amount of the sample and its fragility, the layer information could not be clearly observed under a microscope.

2.2.2. SEM. A Zeiss-Gemini SEM 500 was used for SEM-EDS analysis. Samples were analyzed with a 20 kV acceleration voltage and a 5 mm working distance. Blue pigment was analyzed with a Zeiss EVO-MA 25 with a 20 kV acceleration voltage and a 9 mm working distance. Samples were coated with gold prior to imaging due to their poor conductivity. The measurements were repeated at least two times for EDS detection (both spot scanning and surface scanning) to ensure that the result was repeatable.

2.2.3. Micro-Raman Spectroscopy. Micro-Raman analysis of the pigment was performed using a LabRam spectrometer (purchased from Horiba Jobin Yvon, France) coupled with a microscope. Point measurements were performed using an argon gas laser at 514.5 nm and a 50x working distance objective. The spectral resolution was 0.6 cm^{-1} . The laser power was approximately 0.5 mW, which ensured that good-

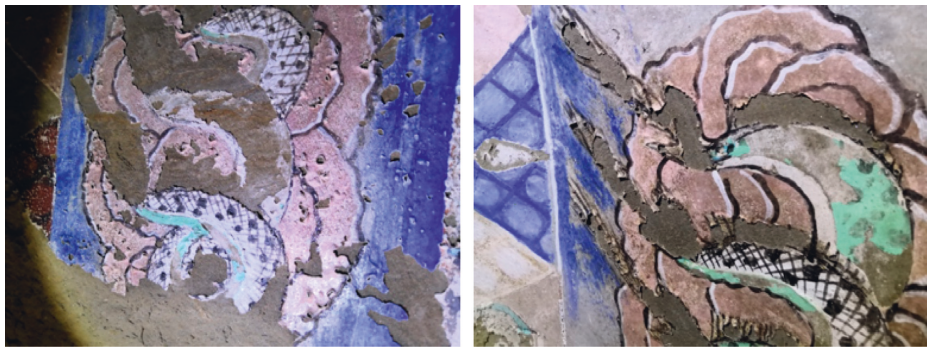


FIGURE 2: Delamination and detachment of pigment.

TABLE 1: Sample collection.

Sample description	Photo	General description
Green (left) and white (right) pigment		Green pigment falls off from the surface in some parts. Soil from cave adheres on the white pigment.
Blue pigment		Organic coating is found on the surface of blue pigment.

quality spectra were recorded. The system used a thermoelectrically cooled CCD detector, which operates at -65°C and an 1800 groove/mm dispersive grating. Since the blue pigment was covered with an organic coating, strong fluorescence was found in the spectra. Toluene was applied with a swab and used to remove the organic coating before testing. Additionally, a laser with a wavelength of 785 nm

was also used to determine whether the fluorescence could be reduced.

2.2.4. *FTIR*. Both micro-FTIR and attenuated total reflection ATR-FTIR were used in the study. Micro-FTIR was used for analysis of the pigment and was performed

using a Nicolet iN10 MX with an MCT/A detector using 32 scans. The wavenumber ranged from 680 to 4000 cm^{-1} with a resolution of 4 cm^{-1} . Pigment particles were selected under a microscope and placed on a sample platform. Three points were detected in each sample, and the spectra with the best signal information were selected for analysis. ATR-FTIR was used to characterize the organic coatings of the blue pigments. ATR-FTIR was performed using a Nicolet-6700 instrument with 64 scans. The wavenumber ranged from 400 to 4000 cm^{-1} with a resolution of 4 cm^{-1} .

2.2.5. XRD. White and green pigment fragments were collected under a microscope. Samples were ground into powder and mixed to meet the necessary test amount. Blue pigment was ground into powder for analysis. Measurements were performed in transmission mode using a STADIP STOE diffractometer with a position sensitive detector and Cu-K α radiation with a wavelength (λ) of 1.5406 \AA ; spectra were captured over a diffraction range of $5\text{--}95^\circ$. XRD was performed on a sample of soil from the cave using a Rigaku D/Max-3C X-ray diffractometer equipped with a Cu-K α radiation source ($I = 0.154056\text{ nm}$) in the range of 10° to 70° , with a tube voltage of 40 kV and a current of 40 mA .

3. Results and Discussion

3.1. Microscopic Analysis. Microscopic observation showed the preservation status of the paint layer. Cracks were present on the surface of the green pigment, and some parts had even fallen off of the surface. Additionally, the soil from the cave had adhered to the white pigment. Some black particles on the surface were observed (Figures 3 and 4). In some places, blue pigment remained and was covered with an organic coating that could reflect light (Figure 5). Due to the loss of pigment in the Kizil Grottoes, restoration was performed during the 1980s. By brushing an organic polymer on the surface of the pigment, conservators could save the surviving pigment [1]. However, degradation gradually occurred in the organic coatings. The deformation and curling of the organic coating caused detachment and delamination of the pigment and destroyed the paint layer.

3.2. SEM-EDS Analysis. SEM-EDS analysis of the samples provided information on the elements (Table 2). The analysis results of the green pigment showed that the mineral contains Cu and Cl, clearly indicating that the pigment is made from a mineral containing copper. The presence of Ca and S in the white pigment indicates that the pigment might contain gypsum. Many elements, such as Al, Si, S, Cl, and Ca, were found in the blue pigment, indicating that the pigment is lapis lazuli, which is a rare and expensive pigment extracted from the blue mineral lazurite $[(\text{Na}, \text{Ca})_{4-8}(\text{AlSiO}_4)_6(\text{SO}_4, \text{S}, \text{Cl})_{1-2}]$ [8, 14]. Azurite and lapis lazuli are the most widely used natural blue pigments in Chinese ancient mural paintings. Lapis lazuli is widely used in the Mogao Grottoes, which is near the Kizil Grottoes. As two important Buddhist grottoes along the Silk Road, they were built during nearly the same period.

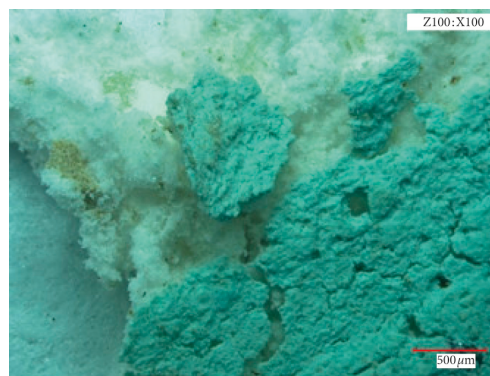


FIGURE 3: Surface of green sample ($\times 100$).

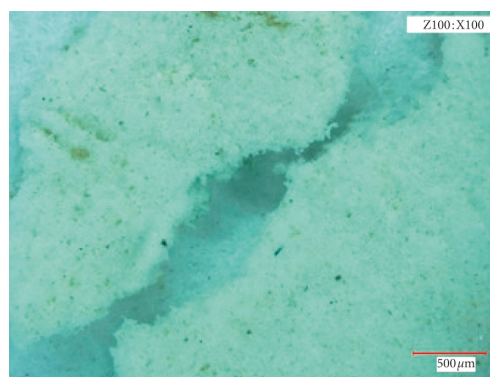


FIGURE 4: Surface of white sample ($\times 100$).

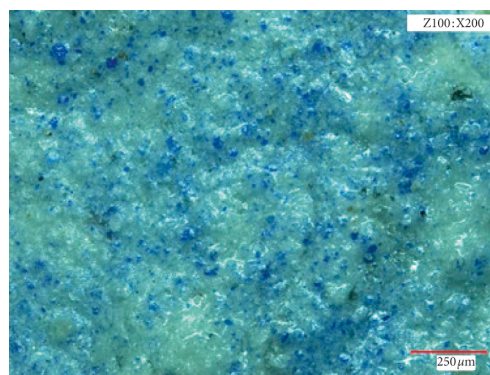
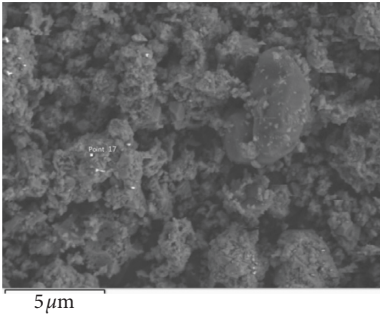
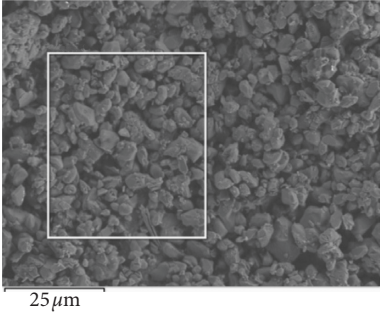
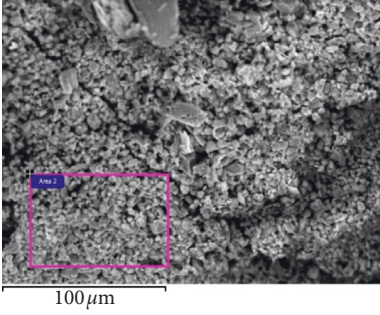
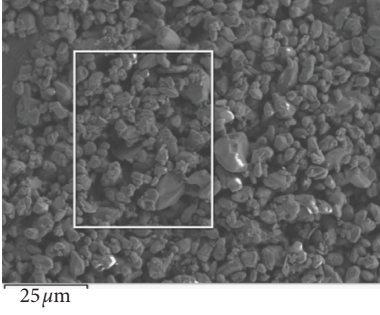


FIGURE 5: Surface of blue sample ($\times 200$).

Azurite has been widely used since the Tang Dynasty (618–907 CE), at which point the construction of Kizil Grottoes was coming to an end [15, 16]. By comparing the lapis lazuli from Kizil with the standard sample from Afghanistan, scientists believe that the mineral may have come from Afghanistan via trading along the Silk Road [5]. Analysis of soil from the caves shows that the cave might contain minerals such as kaolin and quartz.

3.3. Raman Analysis. The peaks at 121 cm^{-1} and 148 cm^{-1} observed for the green pigment can be attributed to O-Cu-O bending, while the peaks at 363 cm^{-1} and 414 cm^{-1} are due to the vibration of Cu-Cl stretching. The peaks at 513 cm^{-1} are

TABLE 2: SEM-EDS analysis.

Sample	SEM	Element by EDS
Green pigment		Cu, Cl, O
White pigment		Ca, Al, S, Si, C, O
Blue pigment		Al, Si, S, Cl, Ca, C, O
Soil from cave		Ca, Al, S, Si, Mg, C, O

due to Cu-O stretching [17]. All of the vibrations indicate that the pigment is paratacamite. In previous work, atacamite, paratacamite, and malachite were found to have been used as green pigments in different caves of the Kizil Grottoes. Considering the special significance of the No. 1

Cave, it remains unclear whether the choice of green mineral corresponded to the chromatic value of the mural painting or to common mineral usage during the period that the mural was created. The spectra of white pigments are in good agreement with the gypsum reference sample (the

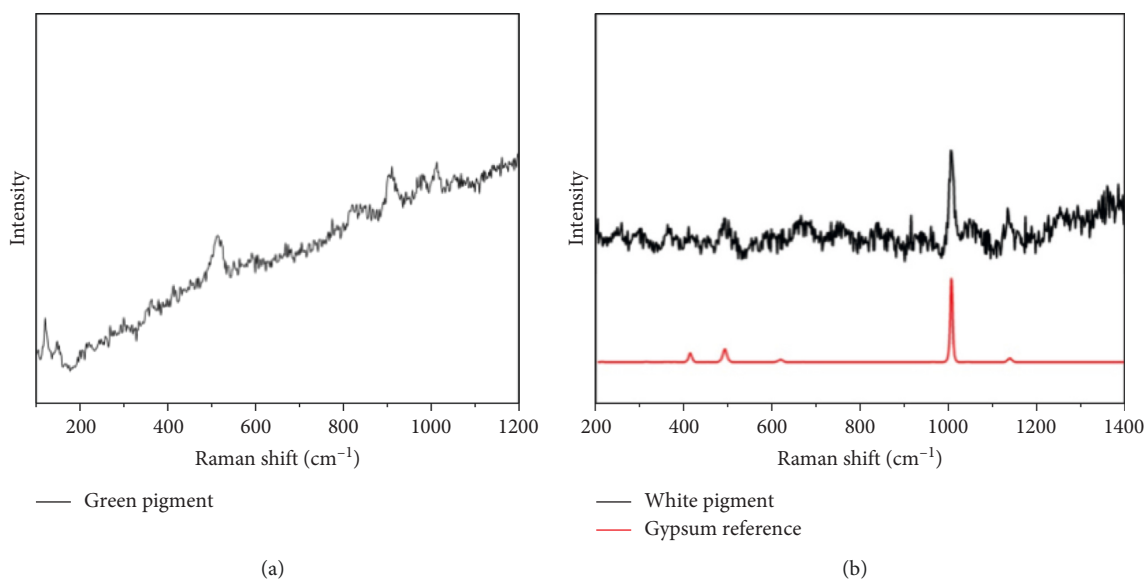


FIGURE 6: Raman spectroscopy of green pigment (a) and white pigment (b).

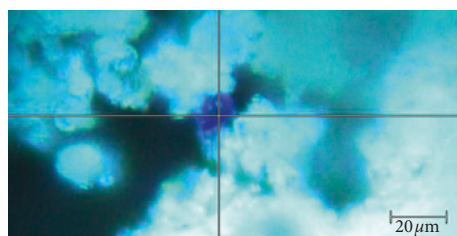


FIGURE 7: Blue pigment (×50).

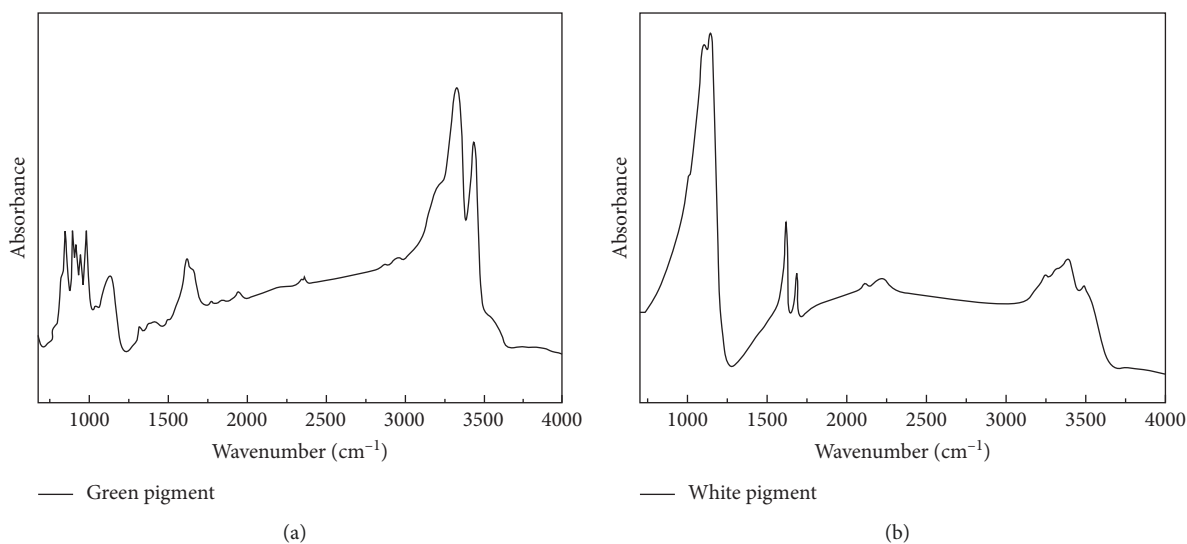


FIGURE 8: FTIR spectroscopy of green pigment (a) and white pigment (b).

spectra were obtained from RRUFF ID R040029). The presence of a band at 1006 cm⁻¹ corresponds to the symmetric stretching of SO₄²⁻ in gypsum (CaSO₄) that was detected [18] (Figure 6).

Due to the strong fluorescence caused by the organic coating, the blue pigment did not directly show a clear

signal on Raman spectroscopy. To reduce the fluorescence, toluene was applied with a swab and was carefully used to remove the organic coating before testing [19, 20]. Lasers with wavelengths of both 514.5 nm and 785 nm were used. Using a limited amount of blue pigment (Figure 7), no signal was found after several methods were applied.

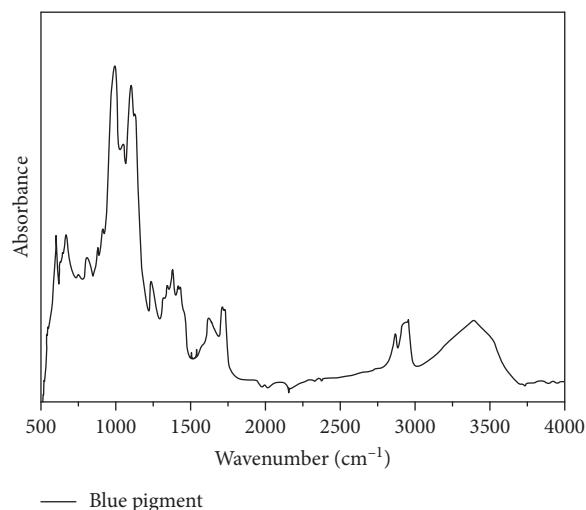


FIGURE 9: FTIR spectroscopy of blue pigment (organic coating).

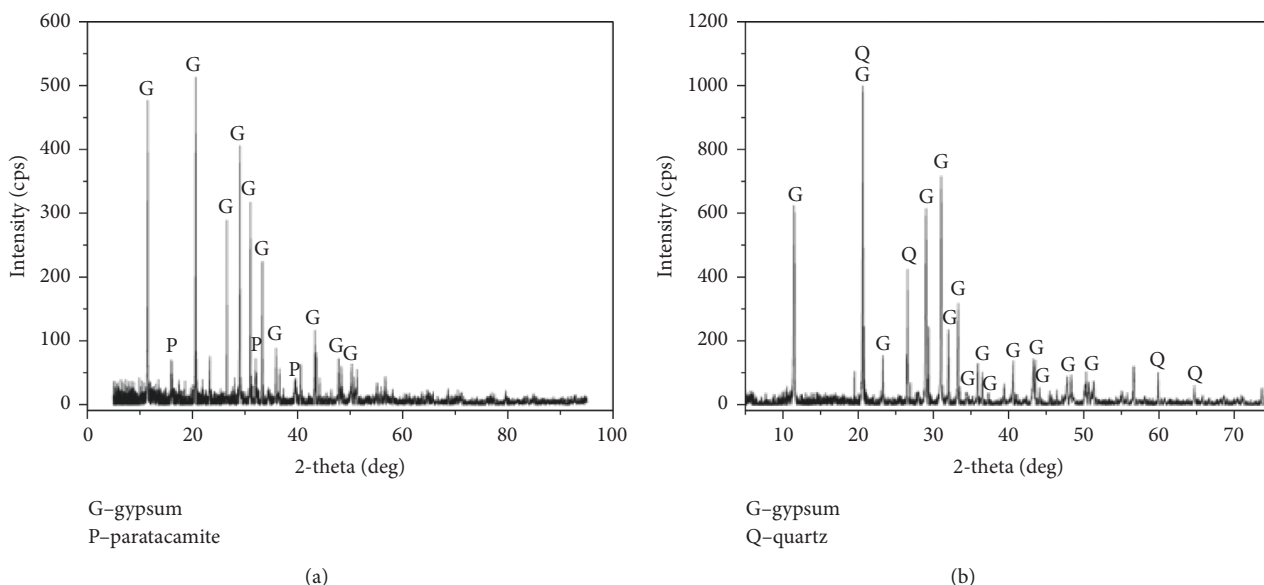


FIGURE 10: XRD analysis of green/white pigment (a) and blue pigment (b).

3.4. FTIR Analysis. FTIR is a sensitive method used to identify pigments with the same chemical position and different crystal systems; this method was used to characterize the content of the white and green pigments (Figure 8). The five peaks found from 800 cm^{-1} to 1000 cm^{-1} were due to the hydroxyl deformation of paratacamite, while the peaks at 3280 cm^{-1} and 3420 cm^{-1} were due to the hydroxyl stretching of individual OH units, which is different from such vibrations in atacamite [21, 22]. The use of gypsum is indicated by the appearance of stretching bands $\nu(\text{OH})$ at 3540 cm^{-1} and 3402 cm^{-1} . The bending vibrations at 1682 cm^{-1} and 1620 cm^{-1} and the two bands at 1110 cm^{-1} and 672 cm^{-1} were attributable to the stretching and bending modes of sulfate anions, respectively [23]. Unfortunately, the spectra did not reveal any information about the binding materials.

Analysis of the blue pigment revealed information about the organic coating (Figure 9). The bands between 2800 cm^{-1} and 3000 cm^{-1} were due to the stretching vibrations of CH_3 , CH_2 , and CH , while the peak at 1730 cm^{-1} was due to the vibration of $\text{C}=\text{O}$, and those at 1370 cm^{-1} – 1450 cm^{-1} were due to the deformation vibrations of CH_3 and CH_2 . C-C vibration bands appeared between 990 cm^{-1} and 1150 cm^{-1} . All of the vibrations indicated the existence of poly-n-butyl methacrylate (PBMA) [24], which has been widely used as a picture varnish on other organic-based museum objects since the 1930s. The sensitivity of PBMA to polar solvents and UV light will easily cause degradation (such as crosslinking of the polymer chain), which will change the properties of the material [19, 25]. According to the record of the Xinjiang Kucha Academy, the organic coating above the blue

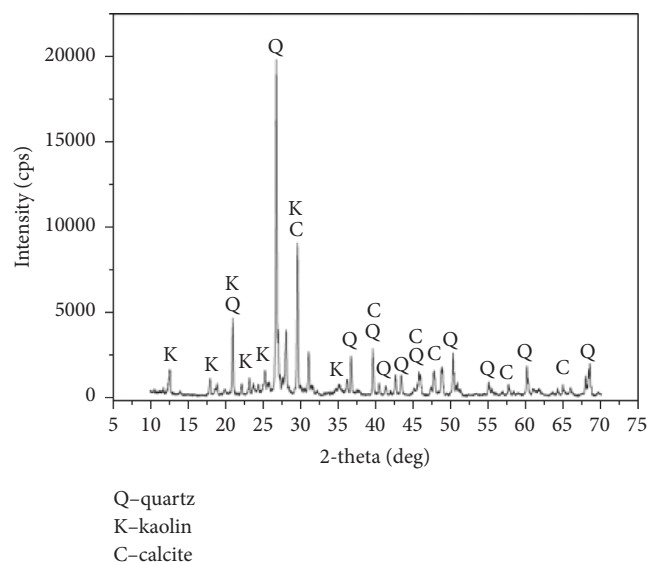


FIGURE 11: XRD analysis of soil from cave.

pigment was introduced as a restoration treatment that was performed during the 1980s, and it has caused the detachment of the pigments. The identification of PBMA will benefit scheduling of the removal of the organic coating and the restoration of the mural paintings.

3.5. XRD Analysis. The XRD results (Figures 10 and 11) were consistent with the Raman results for the green and white pigments. Quartz, kaolin and calcite were found in the soil from the cave, which was also found in previous research [1, 5]. Due to the limited amount of blue pigment particles remaining in the sample, XRD analysis did not indicate the content of the blue pigment.

4. Conclusion

Compared to other Buddhist grottoes along the Silk Road, studies of the Kizil Grottoes have not been well reported to date. Three samples, taken from the fallen part of the No. 1 Cave (one of the most important caves in the Kizil Grottoes), were studied by several complementary techniques to characterize the materials used and assess the preservation status.

Pigment analysis using several scientific methods indicated that paratacamite, gypsum, and lapis lazuli were used as green, white, and blue pigments, respectively, in the No. 1 Cave of the Kizil Grottoes. The green pigment in the No. 1 Cave was analyzed for the first time. In future work, increased attention should be paid to the identification of blue pigments in the Kizil Grottoes to help determine the periodization. Extraction of the binding materials and identification by proteomics methods should be performed to determine whether the selection of the binding material changed over time.

The organic coating applied on top of the blue pigment that was used as a restoration material was identified as PBMA. The PBMA is suffering from severe degradation itself

and has endangered the mural paintings. Studies of the degree of degradation of the PBMA and mural paintings will be scheduled, as will be the removal of the organic coating and the restoration of the paintings.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Acknowledgments

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