


Case Report

# Raman Spectroscopy Analysis of the Mural Pigments in Lam Rim Hall of Wudang Lamasery, Baotou Area, Inner Mongolia, China

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**Abstract:** This paper presents scientific analyses of the wall paintings in Wudang Lamasery, which is located in the Baotou area of Inner Mongolia, China. Raman spectroscopy was used to analyze the pigments of the mural of the Lam rim Hall. The results show that vermilion, red lead, chrome yellow, emerald green and synthetic ultramarine were used. The existence of synthetic pigments provides a clue for the date the mural was painted.

**Keywords:** Wudang Lamasery; mural pigments; chrome yellow; emerald green; synthetic ultramarine; Raman spectroscopy



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## 1. Introduction

The first appearance of the Gelug sect lamasery in the Mongolia region can be traced back to the 16th century AD when Tibetan Buddhism was introduced into this area. After the 17th century, the Qing Dynasty supported the Gelug sect, consolidating its religious domination among the Mongol tribes and having a profound impact on Mongolian society, politics and culture. Among the many lamaseries in today's Inner Mongolia, Wudang Lamasery, which is located in the Baotou area, was one of the most important preaching locations during the late Qing period [1].

The Wudang Lamasery was first established in 1749 and quickly grew to become the largest Gelugpa lamasery in the Mongolia region [2]. Nowadays, the Wudang Lamasery, situated on a hillside of the Yin Mountains, consists of dozens of traditional Tibetan architectural structures. Six main halls and three residences of the Living Buddha were decorated with ancient murals over 1000 m<sup>2</sup>. The Lam rim Hall, the most recent main building at Wudang Lamasery, was dedicated to Master Tsong Khapa, the founder of the Gelug Sect, and was completed in 1892. The name of the hall, adopted from his book <Lam rim chen mo>, means the specific way of becoming Buddha [3]. The mural, drawn on paper and preserved on the south wall of the hall, depicts Tibetan Buddhist heavenly kings and guardians. The depiction of the figures had short and thick contours, which was a typical characteristic of Tibetan Buddhism wall paintings in the late Qing Dynasty. The frame was diversified in color, including red, yellow, green, blue, gold and other hues. The mural has suffered significant deterioration, such as craquelure, flaking, and paint loss, as a result of environmental aging or human influences.

Integrated multi-analytical approaches to investigate wall paintings could provide a full characterization of the materials. In recent years, several non-invasive techniques, such as fiber-optic reflectance spectroscopy (FORS) and hyper spectral imaging, have been adopted to investigate the ancient pigments of cultural heritage, but Raman spectroscopy remains the most frequently used analytical techniques [4,5].

The following examples show how analytical equipment complements each other, with Raman spectroscopy playing a key part in each. In 2009, Franquelo et al. combined Raman, FTIR and SEM/EDX to investigate the pigments in polychromed sculptures, canvas and wall paintings from southern Spain's cultural heritage [6]. The results showed that the vibrational information obtainable by Raman and FTIR spectroscopy is complementary rather than identical. A UV Raman study in conjunction with an FTIR absorbance investigation by using synchrotron radiation techniques was reported in 2018. The UV excitation leads to less thermal degradation and a better signal-to-noise ratio. Additionally, the tunability of the synchrotron radiation source permits us to work in resonant conditions between the excitation wavelength and the wavelength of UV absorption for the samples [5].

As a vibrational spectroscopy method, Raman spectroscopy provides information about characteristic vibrational levels, representing the most versatile and reliable technique to understand the molecular composition for the identification of pigments [7]. In this study, Raman spectroscopy was used to identify the pigments in the mural of Lam rim Hall in Wudang Lamasery, and features of the spectra are useful in conjecturing pigment processing techniques and sources.

## 2. Materials and Methods

Five representative colors, namely red, orange, yellow, green and blue, were sampled from the mural of Lam rim Hall. Figure 1c,d show the location of the samples.



**Figure 1.** Images of Wudang Lamasery; (a) overview; (b) the Lam rim Hall; (c) mural in the Lam rim Hall and sample positions; (d) detail of the mural and sample positions.

Samples were analyzed using a Horiba XploRA confocal Raman microspectrometer equipped with an Olympus microscope. The excitation wavelengths were 532, 638 and 785 nm. The spectra spanned from 100 to 3000  $\text{cm}^{-1}$  using a grating with 1200  $\text{gr}/\text{mm}$  and the spatial resolution was not more than 1  $\text{cm}^{-1}$ . The spectrometer is calibrated by monocrystalline silicon. Red pigments were analyzed with the excitation at 785 nm, the yellow pigment

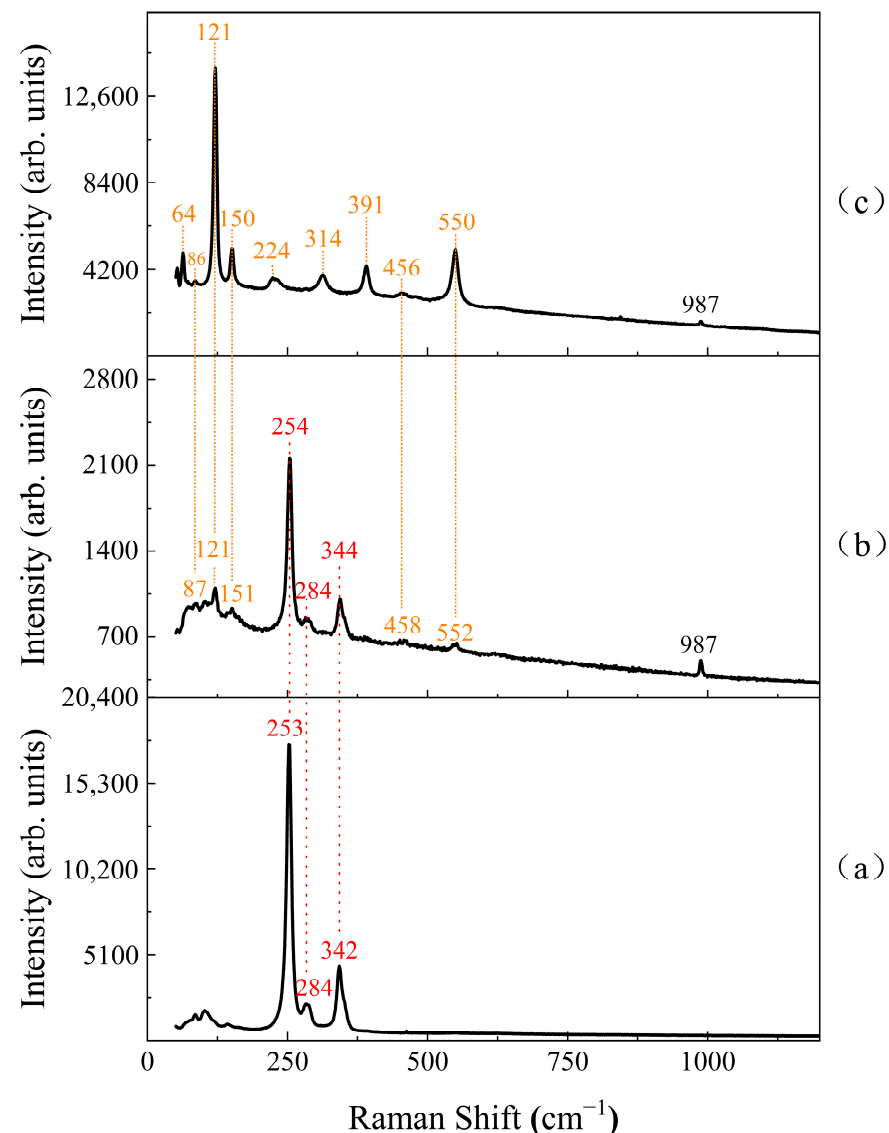
was tested with the 638 nm laser, and the green and blue pigments were at 532 nm. With an acquisition time of 20–30 s and five acquisitions per spectrum, the power on samples was kept to no more than 5 mW.

Most samples were analyzed directly, while sample W-r-3 was embedded in epoxy resin and polished before examination. The microscopic observation was carried out using Keyence VHX-6000 microscopy under a magnification of 1000.

### 3. Results

#### 3.1. Reds Pigments

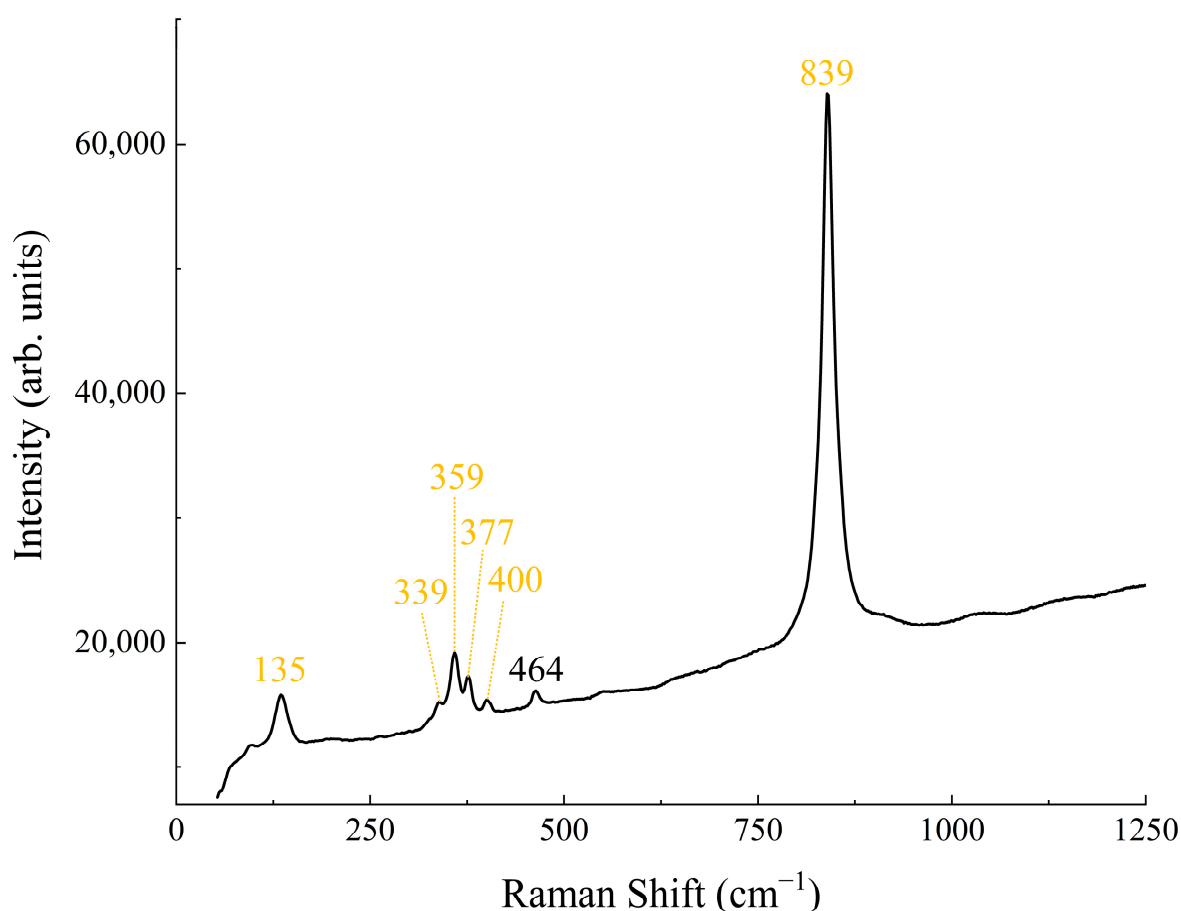
Figure 2 shows the spectra of three red samples. The characteristic peaks near 252, 282 and 343  $\text{cm}^{-1}$ , marked with red numbers, as well as their relative intensity are consistent with the Raman spectrum of vermilion [8]. The peaks marked with orange numbers at 64, 86, 121, 150, 224, 314, 391, 456 and 550  $\text{cm}^{-1}$  match the red lead perfectly [4]. Therefore, it can be determined that vermilion and red lead were used as red pigments in the mural of Lam rim Hall. Moreover, a weak peak near 988  $\text{cm}^{-1}$  could be observed in both the (b) and (c) spectra of Figure 2, which can be attributed to barite ( $\text{BaSO}_4$ ) [4].



**Figure 2.** Raman spectra of red samples in Lam rim Hall. (a) W-r-1, (b) W-r-3, (c) W-r-2. The peaks of vermilion are marked in red, those in red lead are marked in orange, and the peak in black is for barite.

### 3.2. Yellow Pigment

The spectrum of the yellow pigment(W-y) is reported in Figure 3, and the bands at 839, 400, 377, 359 and 135  $\text{cm}^{-1}$  identify the lead chrome ( $\text{PbCrO}_4$ ). The strong peak at 839  $\text{cm}^{-1}$  is due to the symmetric Cr-O stretch of the tetrahedral  $\text{CrO}_4^{2-}$  anion, and several peaks near 359  $\text{cm}^{-1}$  are due to bending modes of the  $\text{CrO}_4^{2-}$  anion [9,10]. The weak peak at 464  $\text{cm}^{-1}$  marked in black in Figure 3 is the vibration of Si-O, which may be caused by trace quartz particles in the sample [11].



**Figure 3.** Raman spectrum of the yellow sample W-y in Lam rim Hall.

### 3.3. Green Pigment

The Raman spectrum of the green pigment in the Lam rim Hall is shown in Figure 4, and the characteristic peaks marked in green are consistent with emerald green [12–14]. The Raman spectrum of emerald green, namely cooper acetoarsenite ( $3\text{Cu}(\text{AsO}_2)_2 \cdot \text{Cu}(\text{CH}_3\text{COO})_2$ ), contains a series of typical bands. The peaks below 600  $\text{cm}^{-1}$  are attributed to  $\text{AsO}_2^-$ , and the peak at 949  $\text{cm}^{-1}$  belongs to the stretching vibration of C-C in acetate [15,16].

As a synthesized pigment, emerald green was first produced by Wilhelm Sattler in Schweinfurt, Germany in 1814, and it was quickly used in painting, interior design and textile [17]. However, it is not a stable pigment. Emerald green would release acetic acid, arsenite and  $\text{Cu}^{2+}$  under an acidic environment. This would result in the formation of a new green mineral, namely lammerite ( $\text{Cu}_3(\text{AsO}_4)_2$ ) [18].

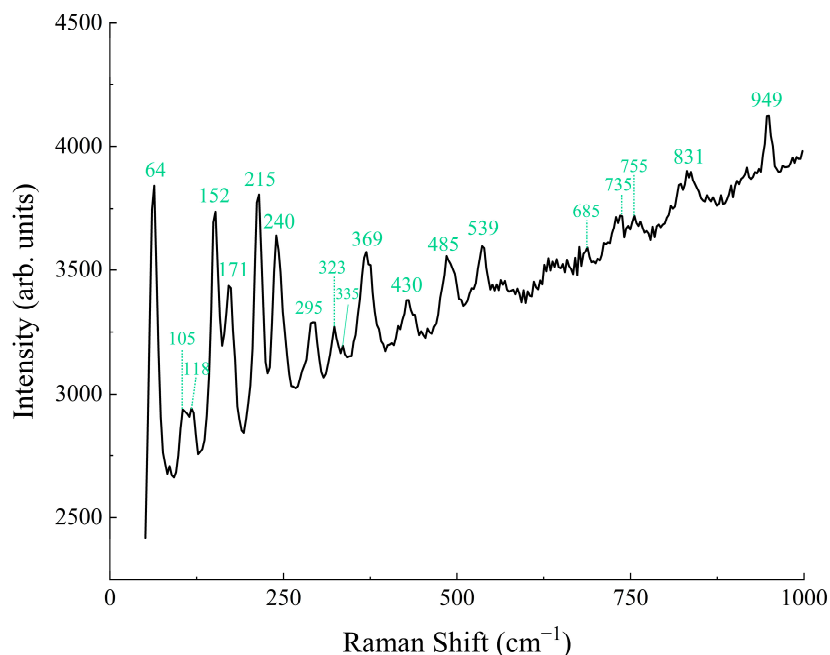


Figure 4. Raman spectrum of the green pigment in Lam rim Hall.

### 3.4. Blue Pigment

The spectrum of the blue pigment is shown in Figure 5. Raman bands at 254, 544, 580, 802, 1093, 1355, 1645 and 2183  $\text{cm}^{-1}$ , are consistent with ultramarine blue precisely, and all the bands are related to trisulfur ( $\text{S}_3^-$ ) radical or disulfur ( $\text{S}_2^-$ ) [19,20]. Bending vibration of  $\text{S}_3^-$  occurs at 254  $\text{cm}^{-1}$ , symmetrical stretching vibration of  $\text{S}_3^-$  at 544  $\text{cm}^{-1}$ , and the shoulder that appears at 580  $\text{cm}^{-1}$  is commonly regarded as antisymmetric stretching of  $\text{S}_3^-$  or overlapping with the stretching bands of  $\text{S}_2^-$  [21,22]. The overtones at 1093, 1645 and 2183  $\text{cm}^{-1}$  are due to the  $\text{S}_3^-$  stretching mode [23].

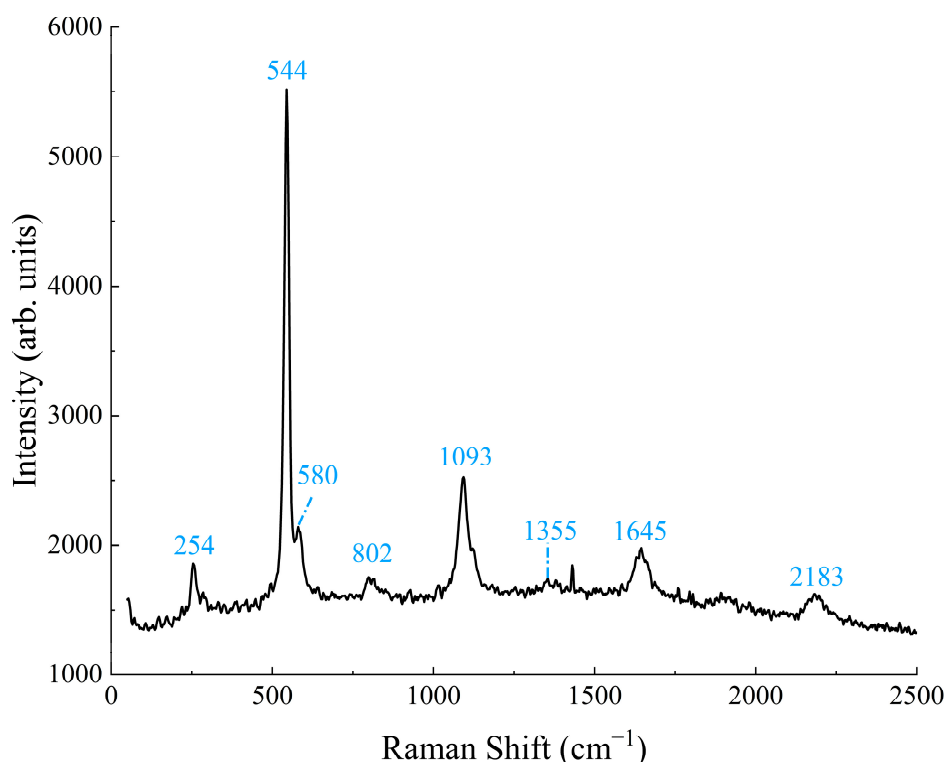


Figure 5. Raman spectrum of the blue pigment in Lam rim Hall.

## 4. Discussion

### 4.1. Analysis of Pigment Sources Based on Spectral Characteristics

#### 4.1.1. Chrome Yellow

Lead chrome ( $\text{PbCrO}_4$ ) occurs naturally as crocoite, and it appears red in bulk and orange-red in powder. Crocoite has been used as a yellow pigment for thousands of years. It was used on cuneiform tablets dating from the third millennium BC in ancient Mesopotamia, as well as in thirteenth century wall paintings of the Siena Cathedral (Italy) [24,25]. It was also found in a mediaeval mural in Northern Bohemia in the second half of the 13th century [26]. The rarity of the mineral in nature determines its rare application in painting, though this evidence renews the knowledge about the  $\text{PbCrO}_4$ -based yellow pigments.

Chrome yellow, the common name of the  $\text{PbCrO}_4$ -based yellow pigments, was synthesized at the end of the 18th century and refers to a group of pigments ranging from medium yellow to primrose [27]. It mainly includes three types of yellows, which are chrome yellow, chrome deep and chrome lemon. Their industrial production steps are essentially the same, while the process modifications would influence the colors, with different hues resulting from different pH conditions [28]. Chrome yellow ( $\text{PbCrO}_4$ ), an orange yellow based on pure monoclinic lead chromate, results in neutral conditions; chrome deep ( $\text{Pb}_2\text{CrO}_5$  or  $\text{PbCrO}_4 \cdot \text{PbO}$ ) is a yellowish red resulting in alkaline conditions, contains the latter admixed with basic lead chromate; chrome lemon ( $\text{Pb}(\text{Cr,S})\text{O}_4$ ) is a deep lemon yellow based on mixed crystals of lead chromate and lead sulphate in acidic solutions [29].

Raman spectroscopy can be used to distinguish the three chrome yellows based on their differing chemical structures.  $\text{Pb}_2\text{CrO}_5$  would show three very strong Raman bands at 825, 837 and 846  $\text{cm}^{-1}$ , distinct from those of  $\text{PbCrO}_4$ . Chrome lemon also shows different spectral features at 437, 448 and 976  $\text{cm}^{-1}$ , due to the  $\text{SO}_4^{2-}$  bands [29]. In this case, the observed bands just show the presence of pure  $\text{PbCrO}_4$ , and the weak signal of quartz at 464  $\text{cm}^{-1}$  may have been caused by the mixing with the plaster layer of the mural during sample storage. Considering the rarity of crocoite in nature and the extensive use of chrome yellow after it was synthesized, it is likely that the synthetic form of  $\text{PbCrO}_4$  was used in the mural of Lam rim Hall, rather than crocoite.

#### 4.1.2. Ultramarine

Ultramarine was one of the most desirable and expensive blue pigments in the ancient world. It was obtained by grounding lapis lazuli, which is an aggregate of minerals, among which lazurite is the most valuable 'blue' mineral component [30]. The formula of lazurite can be expressed as  $\text{Na}_8\text{Al}_6\text{Si}_6\text{O}_{24}\text{S}_n$ . The blue color is attributed to sulfur polyanion radicals trapped in the  $\beta$ -cage structure.  $\text{S}_3^-$  is mainly responsible for the blue color, but contributions from  $\text{S}_2^-$  and tetrasulfur ( $\text{S}_4^-$ ) radicals can shift the color towards yellow or red, respectively [31]. The first known use of blue pigment dates back to the 13th century B.C. in wall paintings from the site of Gla, Greek [32]. Since the third to the fifth centuries, ultramarine has been widely used in Chinese wall paintings, such as Kizil Grottoes, Dunhuang Grottoes, and Yungang Grottoes [33,34].

In 1828, a synthetic version of the ultramarine blue pigment was invented.  $\text{Na}_{6-10}\text{Al}_6\text{Si}_6\text{O}_{24}\text{S}_{2-4}$ , which has a similar composition to lazurite, quickly spread across Europe during the 19th century because it was cheaper and easier to produce while retaining the appreciated bright blue aspect [21]. After being imported to China, the synthetic ultramarine replaced the natural lazurite in many of its applications, including wall paintings, architecture decorative paintings, and painted sculpture [35].

Different combinations of techniques, including LIBS, SEM/EDX, Raman, and FORS, have been found to be useful to distinguish natural and synthetic ultramarine pigments. Nevertheless, the use of a single analytical technique, such as Raman, to achieve this goal is still an analytical challenge [21]. In this case, no minerals commonly associated with lapis lazuli were detected by Raman spectroscopy. Based on times and compari-

son with the literature, the blue pigment from the analyzed murals is more likely to be synthetic ultramarine.

#### 4.2. Implications for Dating

The pigments used on the mural in Lam rim Hall are mainly synthetic pigments, including chrome yellow, emerald green and synthetic ultramarine. They may provide a reference to surmise when the mural was painted.

These three synthetic pigments were synthesized in Europe in the early 19th century and were imported to China during the late Qing Dynasty (1840–1912) [36]. According to the trade archives of pigment imports and exports in the late Qing dynasty (from 1859 to 1902), synthetic ultramarine was first brought to China in 1860, followed by emerald green in 1894 and chrome yellow in 1902 [37]. On the other hand, the mural of the Que-Yi-La Hall, another main building in Wudang Lamasery, was repainted in the 1940s [3]. The pigments used in this mural are mainly traditional mineral pigments, such as vermilion, red lead, atacamite and orpiment, except for synthetic ultramarine [38]. Differences in yellow and green pigments usage indicates the different date of these two murals painted, and there is no record of mural painting or repainting in Wudang Lamasery after 1949.

In summary, the mural of the Lam rim Hall was not painted during the construction of the Hall, but between the early 20th century and the 1940s.

## 5. Conclusions

Raman spectroscopy was used to analyze pigments of the mural in the Lam rim Hall, Wudang Lamasery. The results show that vermilion, red lead, chrome yellow, emerald green and synthetic ultramarine were used. Based on related literature and the features of Raman spectra, yellow and blue pigments are probably the synthetic version. The use of these synthetic pigments further implied that the mural of Lam rim Hall was painted between 1902 and the 1940s.

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