

Spectrophotometric color prediction of mineral pigments with relatively large particle size by single- and two-constant Kubelka-Munk theory

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Abstract

The Kubelka-Munk (K-M) theory combined with the mixing theory has been successively applied to the color prediction of colorant such as paints, dyes and printing inks with fine colored particles. Limited by ancient grinding and sieving technology or for intended use, the mineral pigments used in colored relics usually have a relatively larger particle size. This work investigates the spectrophotometric color prediction of mineral pigments with large particle size by applying the single- and two-constant K-M theory. Results indicate that (a) one sample (i.e., a tint with 80% colored and 20% white mineral pigment) and two samples (i.e., a masstone and a tint with 40%-60% colored mineral pigment) are sufficient to characterize the optical constants of the colored mineral pigment for single- and two-constant K-M theory respectively, and (b) that two-constant K-M theory has a significantly higher prediction accuracy due to the specific particle size and film formation mechanism of the mineral pigments.

Introduction

With great historical, artistic and scientific value, colored relics are important carrier of human civilization. Affected by climate change, natural disasters and other factors, many colored relics have lost the painted film and visual compensation is usually required for the integrality and appreciation of the relics [1-3]. Under the prerequisite that the compensated area has the indistinguishable visual effect from the surrounding area, conservators have to cautiously choose the protection materials and rigorously formulate the protection process [4-7]. The selection and color prediction of mineral pigments herein is one of the most important tasks that conservators have to tackle as mineral pigments are attached on the surface of the relics and form the material bases of their color.

Due to the fact that the visually perceived color of a painting is the combined result of the spectral power distribution of the illuminant, the spectral sensitivity of the observer and the spectral reflectance of the painting [8, 9], an indiscriminate color prediction can cause metamerism phenomenon that the color between the treated area and surroundings only matches under specified illuminant and observer while the match fails when illuminant or observer varies [10]. To eliminate the illuminant and observer dependence and achieve the unconditional match, the spectral reflectance of the treated area should be as similar as possible to that of the surroundings. Researches in the field of multispectral imaging have also proved that spectral reflectance can be utilized to characterize the color effect of paintings without metamerism phenomenon [11-13]. In this case it is necessary to study the spectrophotometric color prediction of mineral pigments so that it can guide the restoration of damaged paintings avoiding preservation damage.

The K-M theory combined with the mixing theory is the most widely used color prediction method in the industries dealing with colorant such as paints, dyes and printing inks [14-18]. With the emergence of low-cost portable spectrophotometry, it has been extended to the identification and color prediction of the pigments used in oil paintings in recent years [19-21]. The ensuing accuracy assessment of the theory on the acrylic paint and oil paint has also appeared recently in regarding to the remarkable achievement it has made [22-24]. One of the common features of the colorant that have been tested is that their pigment particle size is usually at nanometer-to-submicron scales. Limited by ancient grinding and sieving technology or for intended use, however, the mineral pigments used in colored relics (e.g. wall paintings) usually have a relatively larger particle size which is usually at the micrometer scale and can reach up to a few hundreds of microns sometimes [25]. In addition, the dried film of acrylic paint and oil paint is formed by the binder with dispersed pigments since their pigment particle size is very small, while the dried film of mineral paint is formed by the stacking of mineral pigment particles. These pigment particles have direct contact with each other and the binder only acts the role to glue the pigment particles. The dried films of oil paint, acrylic paint and mineral paint are shown in Fig. 1 with a magnification of 300 times. The pigment particles of oil paint and acrylic paint are indiscernible, while that of mineral paint are clear visible. There are also potential gaps between pigment particles in the mineral paint film. The difference between acrylic paint, oil paint and mineral paint in pigment particle size and film formation may result in the discrepancy in color prediction accuracy. On the other hand, the reported assessment on acrylic paint and oil paint has found that the optimal sample for the single-constant K-M theory is a sample with the maximum chroma and that two samples (i.e., a masstone and a tint mixed by the colored pigment and the white pigment at the maximum chroma) are the optimal combinations for the two-constant K-M theory. However, the chroma of acrylic paint and oil paint is usually adjusted by the addition of white pigment and the sample with maximum chroma is often a tint mixed by the colored pigment and the white pigment. As for mineral paint used for colored relics, painters can adjust the chroma of the paint by control the particle size of its pigment. But no matter what the pigment particle size is, the pure colored mineral paint always has the maximum chroma. The addition of white pigment can only reduce its chroma. Fig. 2 illustrates the chroma change of a green mineral pigment with the addition of white pigment. The green mineral pigment has three different particle sizes. The identifiers, 5#, 9#, and 11#, in the figure correspond to the particle size ranges, 150-200 μ m, 30-40 μ m and 15-20 μ m, respectively as provided by the supplier. The difference between acrylic paint, oil paint and mineral paint in maximum chroma sample indicates that the optimal samples used for color prediction may differ.

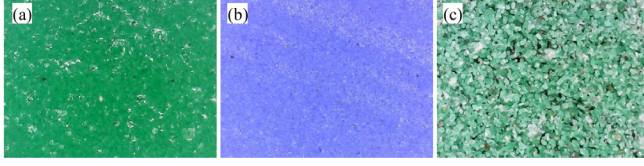


Fig. 1. Dried paint films with magnification of 300 times. (a) Oil paint; (b) acrylic paint; (c) Mineral Paint.

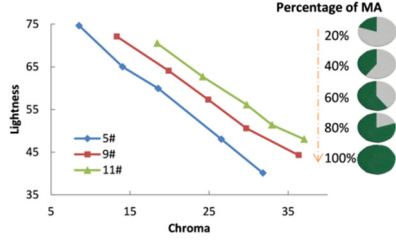


Fig. 2. Chroma change of a green mineral pigment with the addition of white mineral pigment.

Based on this point of view, mineral pigments are selected in this article to investigate their spectrophotometric color prediction by applying the single- and two-constant K-M theory. The background of the color prediction theory is reviewed; the preparation of the samples used for the theory is introduced; the accuracy of the theory is evaluated and the optimal samples for the theory is analyzed. Moreover, the method to determine the optical constants and the potential application of the optical constants to the identification of mineral pigments is also discussed.

Theoretical Background

The aim of spectrophotometric color prediction is to establish a relationship between the spectral reflectance of a mixture and the concentration of its individual component. The K-M theory combined with the mixing theory is the most widely used color prediction method in which the K-M theory relates the spectral reflectance of a film to its absorption and scattering coefficients; the mixing theory describes the relationship of absorption and scattering coefficients between the mixture layer and its individual component considering the concentration of its individual component.

K-M theory

The general problem for characterizing the radiance of light inside an layer is known as the radiative transfer problem [26, 27]. The K-M theory is a special solution to the general radiative transfer problem [28-32]. It aims to predict the spectral reflectance R of a film from its optical constants, absorption K and scattering S coefficients. Assuming the thickness of the film is H and the spectral reflectance of the film substrate is R_g , the general form of K-M theory can be formulated as

$$R = \frac{1 - R_g [a - b \coth(bSH)]}{a - R_g + b \coth(bSH)} \quad (1)$$

here $a = 1 + (K/S)$ and $b = \sqrt{a^2 - 1}$ are two auxiliary variables. When the spectral reflectance of the film no longer changes with

the increase of the film thickness, the film can be regarded as an opaque sample with infinite thickness ($H \rightarrow \infty$). Then Eq. (1) will be simplified as

$$R_\infty = 1 + (K/S) - \sqrt{(K/S)^2 + 2(K/S)} \quad (2)$$

It indicates that the spectral reflectance R_∞ of the opaque film can be determined by the ratio of the absorption coefficient to the scattering coefficient. As well, the ratio can also be calculated by

$$\frac{K}{S} = \frac{(1 - R_\infty)^2}{2R_\infty} \quad (3)$$

Eq. (2) and Eq. (3) are widely used in practice. This study is also limited to the opaque simplification considering the remarkable absorption and scattering properties of mineral pigments.

Mixing theory

After the emerging of K-M theory, it is necessary to establish the relationship between a mixture and its components. In 1949, Duncan regarded the mixture as a linear system and proposed the mixing theory that the absorption and scattering coefficients of a mixture can be linearly combined by those of its components, weighted by their corresponding concentrations [33, 34]. That is

$$K = \sum_{i=1}^n c_i k_i \quad (4)$$

$$S = \sum_{i=1}^n c_i s_i \quad (5)$$

where n is the number of the components mixed; k_i and s_i are the absorption and scattering coefficients of the i th component; c_i is the corresponding concentration. It should satisfy the constraint that $c_i \geq 0$ and $\sum_{i=1}^n c_i = 1$.

The mixing theory transfers the nonlinear relationship between spectral reflectance to the linear relationship between absorption and scattering coefficients which greatly simplifies the problem of pigment mixing. Eq. (4) and Eq. (5) together with K-M theory are known as the two-constant K-M theory since it contains two independent parameters, k and s , for each component. The ratio between absorption and scattering coefficients of a mixture can be calculated by Eq. (4) and Eq. (5) as

$$\frac{K}{S} = \frac{\sum_{i=1}^n c_i k_i}{\sum_{i=1}^n c_i s_i} \quad (6)$$

Under the condition that the pigment particles of different components have the similar scattering ability, Eq. (6) can be further simplified as

$$\frac{K}{S} = \frac{\sum_{i=1}^n c_i k_i}{s} = c_1 \left(\frac{k}{s}\right)_1 + c_2 \left(\frac{k}{s}\right)_2 + \dots + c_n \left(\frac{k}{s}\right)_n \quad (7)$$

It indicates that the K/S of a mixture can be calculated by the linear combination of k/s for each component when the components have the similar scattering ability. Eq. (7) together with the K-M theory

is referred as the single-constant K-M theory since it only includes one independent parameter, k/s , for each component.

Saunderson correction

The discontinuity of refractive index existing in the interface between air and film leads to the multiple internal reflection of light in the interior of the film. The measured spectral reflectance is the result of light multi-reflected. The K-M theory does not take this phenomenon into account, and a correction to the measured spectral reflectance is necessary before inducing the measured spectral reflectance into the theory. The equation used for the correction is well known as the Saunderson correction [35, 36].

Assuming that collimated light from the air strikes the film, a portion r_1 is reflected back to the air. In turn, a portion r_2 is reflected back when the light enters the interface from the film. After infinite internal reflection, the relationship between the measured spectral reflectance R_m and the corrected spectral reflectance R_c can be represented as

$$R_m = \alpha r_1 + \frac{(1-r_1)(1-r_2)R_c}{1-r_2R_c} \quad (8)$$

where α is the adjustable factor that changes with the measuring geometry. For measuring with specular component included, $\alpha=1$. Otherwise $\alpha=0$. $\alpha=0$ is adopted in this study to implement the correction since the samples are measured by $45^\circ/0^\circ$ measuring geometry.

Experimental

The mineral pigments selected are malachite (MA, $\text{Cu}_2\text{CO}_3(\text{OH})_2$), azurite (AZ, $\text{Cu}_3(\text{CO}_3)_2(\text{OH})_2$) and lead white (LW, $2\text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$), which are commonly used in ancient wall paintings. The particle size ranges from 15-20 μm . 10% animal gum is dissolved in 90% pure water as the binder. The mineral pigments is then dispersed in the binder and painted on industrial paperboard. After natural drying of the painted film, the spectral reflectance of each sample is measured by GretagMachbeth SpectroEyeTM, a spectrophotometer with $45^\circ/0^\circ$ ring optic geometry and 4.5mm aperture. The measured spectral data is at 10 nm wavelength intervals between 380 and 730 nm.

Effect of binder content on spectral reflectance

Due to the difference in material density, different mineral pigments having the same weight will have different specific surface areas. Then the binder content required to disperse the mineral pigments differs. To ensure the accuracy of the prediction, the effect of binder content on spectral reflectance is examined. The binder content is controlled over 100 μL to 200 μL in increments of 25 μL with a pipette to disperse 0.35g MA. The spectral reflectance of the films is shown in Fig. 3(a). The spectral reflectance declines slightly as the binder content increases, while it almost remains constant when the binder content is within the range of 125-175 μL . Moreover, the prepared mineral paint within this content range has the most proper consistency. So the initial content is set at 125 μL and slightly optimized within the range of 125-175 μL .

Effect of film thickness on spectral reflectance

The thickness of a film influences its opacity and then determines the application of the color prediction theory. In order to obtain the opaque film, the effect of film thickness on spectral reflectance is investigated. Five hollow patterns with different thickness are used

for the preparation. Fig. 3(b) illustrates the measured spectral reflectance of AZ films. The spectral reflectance decreases slightly with the increase of the patterns, while it almost remains constant when the pattern thickness exceeds 0.20mm. Thus the 0.35mm pattern is used in this study to prepare the opaque film for the reliable application of the theory.

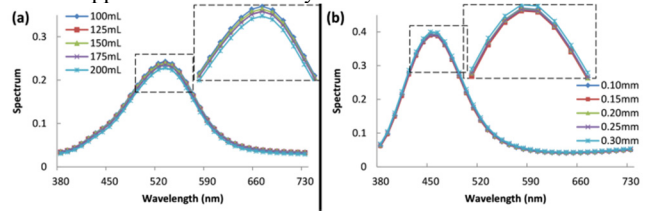


Fig. 3. (a) Effect of binder content on spectral reflectance; (b) Effect of film thickness on spectral reflectance.

Sample preparation

MA and AZ are mixed with LW in concentration increments of 20% respectively to prepare the ladder samples. MA is mixed with AZ in concentration increments of 20% to prepare the testing samples. Other three testing samples are prepared by the random mixture of MA, AZ and LW with concentration ratio 40:40:20. All the samples and their spectral reflectance are shown in Fig. 4.

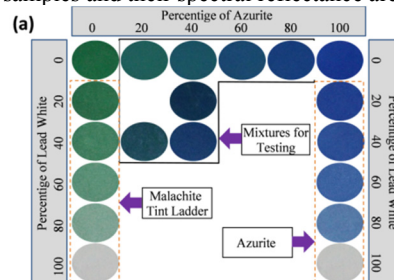


Fig. 4. The prepared samples and their spectral reflectance. (a) Concentrations of all the samples; (b) Spectral reflectance of MA ladder; (c) Spectral reflectance of AZ ladder; (d) Spectral reflectance of MA and AZ mixtures; (e) Spectral reflectance of triple mixtures.

Determination of optical constants

Determining absorption and scattering ratio

The usage of the single-constant K-M theory only involves the absorption and scattering ratio. The ratio of a colored pigment can be calculated by Eq. (3) with the corrected spectral reflectance of its masstone (100% colored pigment). Due to the intense absorption ability of the masstone sample, its spectral reflectance is usually very small. The directly using of the sample, however, will result in a higher color prediction error since the measure noise has a relatively significant influence on the small spectral data. Thus a tint mixed by the colored pigment and the white pigment is usually utilized in practice to reduce the influence of the measure noise and then to improve the accuracy of the calculated ratio. Given the ratio $(k/s)_w$ of the white pigment, according to Eq. (7), the ratio of a tint can be expressed as

$$\left(\frac{K}{S}\right)_{mix} = c \cdot \frac{k}{s} + (1-c) \left(\frac{k}{s}\right)_w \quad (9)$$

where k/s is the absorption and scattering ratio of the colored pigment; c is its concentration in the tint; subscript w and mix represent the white and the tint respectively. The absorption and scattering ratio of the colored pigment can be calculated by

$$\frac{k}{s} = \frac{(K/S)_{mix} - (1-c)(k/s)_w}{c} \quad (10)$$

Eq. (10) indicates that a tint sample is enough to determine the absorption and scattering ratio of a colored pigment for the single-constant K-M theory if the ratio of the white pigment is pre-known. If there are several tints, the absorption and scattering ratio in Eq. (9) can also be solved by applying the least squares technique using all the tints.

The numerator and denominator in Eq. (10) should keep a linear relationship in theory in that the absorption and scattering ratio of the colored pigment is constant. In fact, they are not entirely content with the linear relationship. As shown in Fig. 5(a), the relationship is always a concave curve. Fig. 5(b) is the calculated ratios of MA by different tints. With the increase of the colored pigment, the calculated ratio increases. This is the reason that the absorption ability of the film is enhanced with the addition of the colored pigment that lead to the nonlinear relationship. It can be inferred the tint used to determine the ratio will influence the color prediction accuracy. It is necessary to find the optimal tint for determining the absorption and scattering ratio of the colored pigment. On the other hand, the normalized ratios are shown in Fig. 5(c). The curves have the similar geometry profile, which can be used to identify the colored mineral pigments.

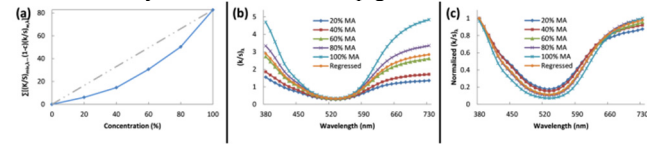


Fig. 5 The absorption and scattering ratio of MA. (a) Relationship between optical property and concentration; (b) The ratios calculated by various tints; (c) Normalized ratios.

Determining absorption coefficient and scattering coefficient

Due to the weak absorption and intense scattering ability, the scattering coefficient of a white pigment is usually assumed as unit at all wavelengths. Then the absorption coefficient of the white pigment can be determined as $(k/s)_w$ by applying Eq. (3). As for the colored pigment, Eq. (6) can be transposed and collected as

$$\frac{K}{S} \cdot s - k = \frac{1-c}{c} \cdot \left[\left(\frac{k}{s} \right)_w - \frac{K}{S} \right] \quad (11)$$

where k and s are the absorption and scattering coefficients of the colored pigment. To determine these two unknowns, at least two samples are required. The masstone is usually chosen as one of the samples because it can be easily prepared. Another sample can only be a tint. It is necessary to examine the optimal tint used for determining the absorption and scattering coefficients of the colored pigment. Besides, these coefficients can also be determined by applying the least squares technique when several tints at various concentrations are prepared.

Fig. 6 is the calculated absorption and scattering coefficients of MA. The curves in Fig. 6 (a-b) have more similar geometry profiles than that in Fig. 5(b). Moreover, as shown in Fig. 6c, the ratio curves calculated by the two coefficients in Fig. 6 (a-b) are completely coincided with each other. All these may indicate that the influence of tint selection on color prediction accuracy of the two-constant K-M theory is relatively slighter than that of the single-constant K-M theory. Besides, the spectral curve of the colored pigment holds an opposite trend to its absorption curve and a similar trend with its scattering curve along wavelengths. In other words, the scattering coefficient represents the original geometry profile of the spectral reflectance, whereas the absorption coefficient emphasizes the geometry profile of the small spectral reflectance. A combination of the absorption and scattering coefficients may improve the identification of the colored mineral pigments used in colored relics.

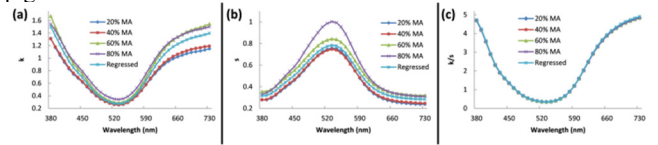


Fig. 6 The absorption and scattering coefficients of MA. (a) Absorption coefficients; (b) Scattering coefficients; (c) The ratios of the absorption coefficients in (a) to the scattering coefficients in (b).

Results and discussion

The optimization objective of spectrophotometric color prediction is to find the concentrations that minimize the difference between the predicted and measured spectral reflectance from optical constants. If root mean squared error (RMSE) is set as the accuracy criteria, the optimization objective can be formulated as

$$\min f(c_1, c_2, \dots, c_n) = \left[\sum_{\lambda} (R_{predicted, \lambda} - R_{m, \lambda})^2 \right]^{1/2} \quad (12)$$

where the concentrations need to satisfy the constraint that $c_i \geq 0$ and $\sum_{i=1}^n c_i = 1$; λ is the wavelength; $R_{predicted}$ is the spectral reflectance predicted by the theory.

All the samples are predicted to verify the accuracy of the single- and two-constant K-M theory for mineral pigments with large particle size. The RMSE between the predicted and measured spectral reflectance is selected as the spectral metric. The CIEDE2000 color difference ΔE_{00} under the CIE standard illuminant D65 and the CIE 1931 standard observer is calculated as the colorimetric metric. Involved by weighting precision, mixture homogeneity and interface smoothness, the actual and predicted concentrations often differ. Thus Euclidean distance between the actual and predicted concentrations is adopted as the concentration error (CE) metric. Statistical results of the single- and two-constant K-M theory are shown in table I and II respectively.

Table I. Statistical results of single-constant K-M theory

c (%)	ΔE_{00}		RMSE (%)		CE (%)	
	Max	Mean	Max	Mean	Max	Mean
20	11.5	4.51	28.69	14.08	46.04	20.20
40	7.44	2.55	23.67	9.77	44.85	12.76
60	3.53	1.67	23.67	7.35	40.98	12.08
80	3.50	1.32	23.67	6.77	43.33	11.12
100	5.14	1.55	23.67	8.04	40.64	12.96
Reg*	2.98	1.53	23.67	7.18	42.70	11.65

* Reg means regressed.

Table II. Statistical results of two-constant K-M theory

c (%)	ΔE_{00}		RMSE (%)		CE (%)	
	Max	Mean	Max	Mean	Max	Mean
20	1.49	0.32	5.30	1.42	40.07	10.39
40	1.31	0.27	3.61	0.82	31.10	7.70
60	1.25	0.30	5.11	1.58	34.47	9.32
80	2.12	0.63	10.51	3.18	38.50	9.91
Reg	0.95	0.24	3.03	1.05	31.35	8.21

Table I shows that the tint used to determine the absorption and scattering ratio has a remarkable influence on the color prediction accuracy of the single-constant K-M theory. For all the tints, the maximum of the mean color difference reaches to 4.51 ΔE_{00} while the minimum is 1.32 ΔE_{00} ; the maximum of the mean spectral error reaches to 14.08% while the minimum is 6.77%; the maximum of the mean CE reaches to 20.20% while the minimum is 11.12%. The remarkable influence leads to the result that the mean accuracy obtained by the least squares technique are worse than that by a specific tint. Moreover, the prediction accuracy are improved at first with the increase of the concentration of the colored pigment, and then decline as the increase of the concentration continues. The best sample for the single-constant K-M theory is the tint with 80% colored mineral pigment. It can be inferred that a tint with 80% colored mineral pigment is enough to characterize the absorption and scattering ratio of the colored pigment. Distinct from acrylic paint and oil paint [24], however, this tint for mineral paint is not the sample with the maximum chroma since the masstone of mineral paint possesses the maximum chroma.

It can be seen from table II that the prediction accuracy of the two-constant K-M theory are also improved at first with the increase of the concentration of the colored pigment, and then decline with the further increase of the concentration. Although least squares technique can reduce the maximum error to some extent, its improvement to the mean error is indistinctive. In addition, the best performance of the two-constant K-M theory occurs when the tint has 40%-60% colored mineral pigment. It indicates that two samples, a masstone and a tint with 40% - 60% colored mineral pigment, are the best choice for the two-constant K-M theory. Distinct from acrylic paint and oil paint [24], however, this tint for mineral paint is also not the sample with the maximum chroma.

On the whole, the color prediction accuracy of the two-constant K-M theory are significantly better than that of the single-constant K-M theory for mineral paint. It is also better than the accuracy of the two-constant K-M theory for acrylic paint and oil paint [22, 24]. While the accuracy of the single-constant K-M theory for mineral paint are worse than that for acrylic paint and oil paint [23]. The results may indicate that the larger particle size and

typical film formation mechanism of mineral paint match the general assumptions that derive the two-constant K-M theory well, but fail to match the specific prerequisite for the derivation of the single-constant K-M theory. The two-constant K-M theory is more suitable to the color prediction of mineral paint.

Conclusion

The single- and two-constant K-M theory were examined to predict the color of the mineral paint with large pigment particle size. It shows that the best sample for the single-constant K-M theory is a tint with 80% colored mineral pigment and that a masstone of the colored mineral pigment and a tint with 40%-60% colored mineral pigment are the best choice for the two-constant K-M theory. It also shows that, due to the larger particle size and typical film formation mechanism of the mineral pigments, the prediction accuracy of the two-constant K-M theory for mineral pigments with particle size range of 15-20 μ m are comparable with that of acrylic paint and oil paint, while the accuracy of the single-constant K-M theory are worse than that of acrylic paint and oil paint. Therefore, the two-constant K-M theory is more suitable to the color prediction of the mineral pigments with larger particle size. Moreover, this article also discussed the potential application of the optical constants to the identification of mineral pigments used in colored relics. In the future, it would be interesting to apply these optical constants to the identification of mineral pigments.

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