

Article

Transformation of Goethite to Hematite: Dependence of Ochre Color on Temperature and Time of Heating

India F. Buff ¹, Lina N. Hassini ¹, Joan E. Reichert ¹, Karen Wu ¹ , Giovanni Cavallo ²  and Walter J. Bowyer ^{1,*} 

¹ Department of Chemistry, Hobart and William Smith Colleges, 300 Pulteney St., Geneva, NY 14456, USA; india.buff@hws.edu (I.F.B.); lina.hassini@hws.edu (L.N.H.); joan.reichert@hws.edu (J.E.R.); kw969@scarletmail.rutgers.edu (K.W.)

² Department of Construction Environment Design, University of Applied Sciences and Arts of Southern Switzerland, Via Flora Ruchat-Roncati, 15, 6850 Mendrisio, Switzerland; giovanni.cavallo@supsi.ch

* Correspondence: bowyer@hws.edu; Tel.: +1-315-781-3608

Abstract

Ochres, natural pigments based on iron oxide and (oxy)hydroxide minerals, range in color from yellow to red to violet to brown. Ochres have been used and manipulated by humans for more than 100,000 years, and they continue to be valued because they are intensely colored, stable to light and heat, and cheap. Ochres offer critical evidence for the evolution of human cognition and symbolic behavior. Interpretation of the archeological evidence requires an understanding of ancient processing of ochre. Of particular importance in the Paleolithic is the heating of yellowish-brown goethite to convert it to red hematite. We heat natural ochres containing goethite in a furnace and characterize the colors of the resulting hematites. Within the temperatures achievable by early humans in simple wood fires (300 to 800 °C), the color is almost entirely independent of temperature and time of heating with $\lambda_m \geq 580$ nm and reflectance at 780 nm varying by less than 5%. At higher temperatures (800 to 1100 °C) natural ochres may become a brighter red (increasing reflectance by 15%), purplish red, or various shades of brown depending on the accessory minerals in the ochre.

Keywords: pigments; ochre; goethite; hematite; color; diffuse reflectance spectroscopy

1. Introduction

Ancient use of ochre offers some of the best evidence for cognitive development in early humans [1–5]. These bold pigments, natural earth materials colored by iron oxides, may be brown, orange, yellow, red, and even purple. They are durable, so they offer exceptional preservation in archeological contexts, and they have high thermal and photostability as well as excellent opacity and tinting strength. Humans used ochre in the earliest known cave paintings (for example, 40,000 years ago in Indonesia [1,6]), and hematite has been found in a 100,000 year old “tool kit” [7]. Humans have deliberately collected and used ochres for more than 300,000 years [8–11]. The wide occurrence of ochres in archeological contexts and the diversity of its uses prove its importance to early humans [6,12–19].

Thus, ochres are vital artifacts for modern archeologists. The variety and complexity of human selection, mining, transport, processing, use, and trade of ochres make them some of the earliest and most important artifacts for understanding the development of human cognition and symbolic thinking. Much recent research and many review articles



Academic Editor: Jae-Hong Choi

Received: 29 May 2026

Revised: 30 June 2026

Accepted: 3 July 2026

Published: 13 July 2026

Copyright: © 2026 by the authors.

Licensee MDPI, Basel, Switzerland.

This article is an open access article distributed under the terms and conditions of the [Creative Commons Attribution \(CC BY\)](https://creativecommons.org/licenses/by/4.0/) license.

emphasize the importance of ochre in studies of the development of hominid cognition and symbolic thought [1–5].

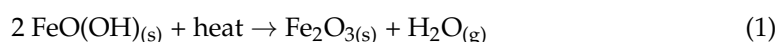
Humans have long demonstrated a preference for certain colors. Red was often preferred [2], and MacDonald et al. demonstrated that bright red was preferred over a more easily accessible duller red [16]. Purple ochre was selected in Germany during the Paleolithic [11]. Color choice and control is a powerful source of evidence for human cognitive development and symbolic thinking [2,4,5,10,20–25]. For example, ochres offer critical evidence for the comparison of the cognition of Neandertals and anatomically modern humans [13]. Langley and Litster ([2], p. 2) write that “color selection and use is an important aspect of burgeoning symbolic cognition”, but that the study of ochre has “been underexplored”.

Goethite ($\text{FeO}(\text{OH})$), a yellow-brown mineral, is the most abundant iron oxide on earth [18,26,27]. Hematite (Fe_2O_3) is not as abundant as goethite, but was often preferred for its intense red color [18,26]. These are the two most important minerals in ochres, but other minerals and organic matter may affect ochre properties, including color. (Manganese oxides offer another class of ochres that are dark brown to black, but are not treated here.)

At least 100,000 years ago and probably much earlier, humans learned that heating yellowish-brown goethite converts it into red hematite [13,28–30]. This intentional firing of ochre to change its color is a very early example of complex material processing, much older than, for example, pottery making. Its *chaîne opératoire* requires selection and transport of yellowish-brown ochre combined with the technological control of its heating in open wood fires. The preplanning and intent required in the process offer important evidence for the symbolic and ritual importance of color. Hematite resulting from heating goethite is the earliest known example of synthetic pigment, preceding Egyptian blue, Han blue, and lead white by tens of thousands of years.

Interest in how Paleolithic people prepared red pigments by heating goethite has motivated a multitude of studies [28–35]. The color of heated ochres in more recent cultures are also intensely studied, for example, in Roman wall paintings [36–38], Chinese ceramic glazing [39], and the synthesis of Mars yellow and red in eighteenth century Europe [40]. Recently, Ilmi and colleagues have studied hematite color in many rock art sites throughout the Indonesian archipelago [1,6,23,25].

Modern laboratory experiments demonstrate that goethite undergoes a topotactic conversion in the solid state at temperatures above 250 °C by dehydration to form hematite (Equation (1)). The mechanism has been studied by a wide variety of techniques including X-ray diffractometry (XRD), Raman and Mossbauer spectrometry, thermogravimetric analysis, differential scanning calorimetry, and more [23,28,31–34,41,42].



Hematite absorbs blue and green light by a charge transfer between the iron and oxygen; and it reflects red. Goethite also absorbs blue, but it reflects some of the green and a lot of yellow, orange, and red, giving it a yellowish-brown appearance. Of course, natural ochres are complex mixtures, so the colors of ochres are highly varied.

Given the complexity of the chemistry of ochres and their importance to archeology and art history, the effect of heating on the colors of ochre continues to generate important questions. Of particular interest is the effect of temperature on the color of the resulting pigment. In early studies, archeologists attributed the variable colors of ochres to the temperature of heating with little empirical evidence. For example, ref. [43] found “blocks of ochre in different states of calcination” and thereby concluded that Paleolithic people could control the color of the hematite by controlling the temperature. Some recent studies also have suggested that heating goethite over a range of temperatures and/or time can

generate a wide range of colors [44,45]. In contrast, other researchers find little dependence of color on temperature [40,46,47].

In this paper, we investigate the effect of temperature and time of heating on 12 different goethites, and we characterize their colors using photography, Munsell colors, and diffuse reflectance spectroscopy. We studied five highly variable, natural, yellow ochres of differing geologic origins, textures, accessory minerals, and percent goethite. We also studied six commercial pigments by Rublev Colours™ (Willits, California, USA) [48] prepared from natural goethite. Finally, we include synthetic goethite for comparison.

2. Materials and Methods

2.1. Ochres

Synthetic goethite (SYN) was made from iron (III) nitrate and potassium hydroxide (Thermo Fisher Scientific, Waltham, MA USA) using the standard procedure developed by Schwertmann and Cornell [27,49]. Four natural ochres (PVG, PVA, SABO, TP3) were collected in northern Italy and are described in detail in [50]. One natural goethite (JPSS) from Hibbing, MN, USA, was purchased from Wards Scientific (Rochester, NY, USA) and is well characterized [34,46]. The solid samples were ground in a mortar and pestle and sieved to <75 microns. (While grinding is a fundamental step to standardize grain size, it is worth noting that mechanical milling can significantly influence the mineral microstructure by inducing lattice strain or structural defects, which can subsequently alter their thermal reactivity and long-term properties [51].) Six natural yellow ochres, again of differing geological origins, textures, accessory minerals, and percent goethite, (BRY, YOL, LO, IYE, NYO, JCL) were purchased as powders from Rublev Colours™ [48]. See Table 1 for details of the twelve ochres.

Table 1. Descriptions of ochres with abbreviations.

Country	Locale	Abbreviation	Iron Oxide	Other Minerals	Contains Carbonate
Laboratory	Synthetic	SYN	goethite	none	No
Italy	Ponte di Veja	PVG	goethite	quartz	No
Italy	Ponte di Veja	PVA	goethite	calcite, quartz	Yes
Italy	Sant'Andrea	SABO	goethite	ankerite	Yes
Italy	Ponte di Veja	TP3	goethite	ankerite, quartz, muscovite, montmorillonite	Yes
USA	Hibbing, MN USA	JPSS	goethite	quartz	No
USA	Blue Ridge Yellow Ochre	BRY	goethite	clay, gypsum	No
France	Yellow Ochre Light	YOL	goethite	clay, quartz	No
Italy	Lemon Ochre	LO	goethite	clay	Yes
Italy	Italian Yellow Earth	IYE	goethite	clay	No
Cyprus	Nicosia Yellow Ochre	NYO	goethite	clay	No
France	Yellow Ochre JCL	JCL	goethite	clay	No

2.2. Heating of Ochres

Goethites were heated in a Fisher (Waltham, MA, USA) Isotemp Furnace at controlled temperatures in air from 300 to 1100 °C for 1, 3, 8, and 16 h in unlidded ceramic crucibles.

2.3. Diffuse Reflectance Spectroscopy (DRS)

An Ocean Optics USB 200 spectrometer with a PX-2 pulsed xenon light source (Ocean Insight, Orlando, FL, USA) was used to record diffuse reflectance spectra from 380 to 780 nm (% reflectance vs. wavelength). For recording the spectra, powders were placed in a well on a glass microscope slide, and the well was filled to a level slightly higher than the flat

portion of the slide. To reproducibly place the probe of the spectrometer precisely 1.0 mm above the sample at an angle of 60°, the powder was covered with a second microscope slide, and the fiber optic holder was placed on top. For each sample, independent replicate spectra were recorded to test reproducibility by filling the cell, recording a spectrum, emptying the well of the slide, and repeating two to seven times.

2.4. Recording Munsell Codes and Colors

Munsell codes (e.g., 10R 4/6) and colors (e.g., “Red”) were recorded using the same sample holder for recording reflectance spectra. In some cases, the color of an ochre falls between two shades in the Munsell color book. Because we independently determine each color, the same color may be identified as one shade or its adjacent shade. Thus, we consider two Munsell colors as the “same” if they are adjacent to each other and virtually indistinguishable. Photographs included an International Federation of Rock Art Organizations color card (Caulfield South, Victoria, Australia).

2.5. Powder X-Ray Diffraction (XRD)

XRD patterns were recorded on randomly oriented samples deposited in the hollow of a Si monocrystal zero-background plate (Assing Spa, Monterotondo, Italy) with a Bruker D8 Advance XRD in the θ : θ mode. The X-rays were generated with a Cu anode (Cu K α = 1.5418 Å) at 40 kV and 40 mA over a 2θ range of 5° to 80° with a step size of 0.02° and a scan speed 0.5 s/step. A Lynxeye PSD detector reduced fluorescence and improved the S/N ratio.

3. Results

3.1. Appearance

Figure 1 illustrates synthetic goethite powder and five natural goethites (lumps and powder) before heating. All six show the characteristic brownish yellow color of goethite, and the variability of textures is illustrated in the lumps.

The palette of colors after heating the synthetic goethite and the four natural goethites sourced from the Lessini Mountains in northern Italy is illustrated in Figure 2.

The synthetic goethite, SYN, heated to 300, 600, and 800 °C shows the typical color of hematite. This range is especially important to understand the colors of Paleolithic ochres because it is difficult to achieve and sustain temperatures over 800 °C with simple wood fires [52–55].

Heating the PVG goethite at 300, 600, and 800 °C (Figure 2, second row) generates a color similar to, but lighter than, SYN. The lighter red of PVG relative to SYN is because PVG contains 80% colorless quartz [50]. Again the color is independent of temperature up to 800 °C.

Heating JPSS from 300 to 800 °C is again very similar to SYN and PVG. JPSS is slightly darker than PVG, consistent with JPSS having a higher content of iron [46].

At the highest temperatures (which would have been achievable only after development of kilns), heating SYN at 1000 and 1100 °C produces a darker purplish color (Figure 2, first row). In contrast to SYN, PVG becomes brighter at 1000 and 1100 °C (Figure 2, second row). More similar to SYN, JPSS becomes darker above 800 °C, but not as dramatically.

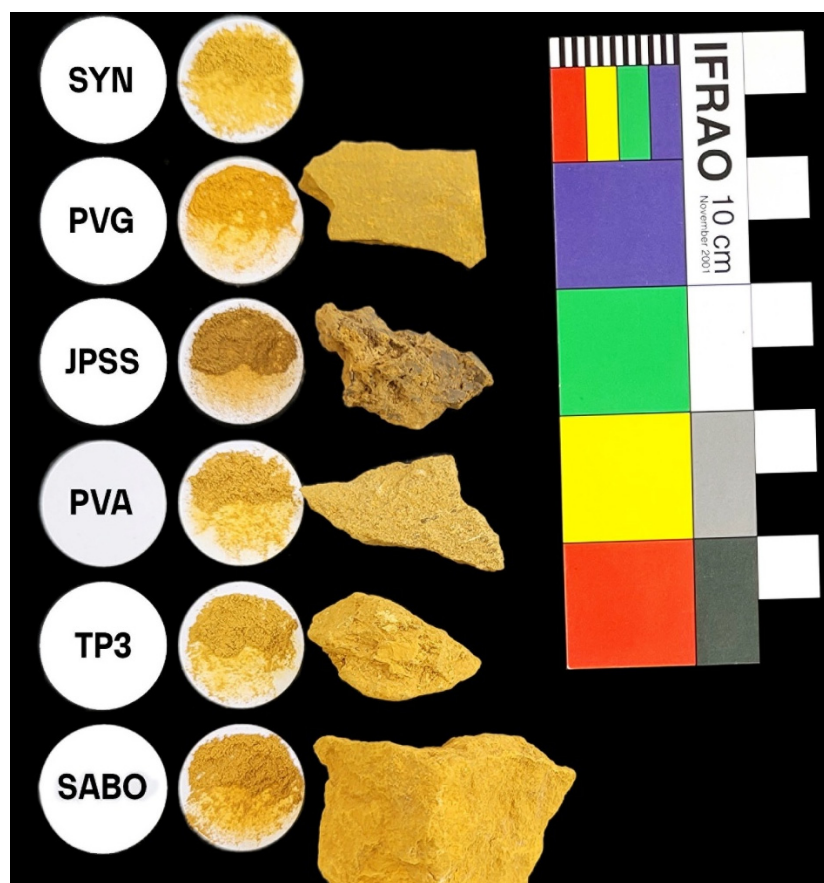


Figure 1. Photograph of synthetic goethite (powder) and five natural ochres (as lumps and powders). See Table 1 for abbreviations. In the upper half of each circle is a thick layer of pigment while in the lower half the pigment is rubbed into the white paper.

The darker, slightly purplish color can be attributed to recrystallization and fusion of the hematite crystallites at the highest temperatures. In a thorough investigation, Castagnotto et al. [36] demonstrated convincingly that at and above 900 °C, relatively pure hematite recrystallizes, and smaller crystals agglomerate into larger crystals. The resulting larger crystals of hematite (greater than two micrometers) appear dark purple [1,23,25,36,37,56]. This explains the dark color of SYN at 1000 and 1100 °C. Unlike SYN, PVG contains 80% quartz, and the iron is scattered as very small particles on the surface of the much larger quartz crystals [50]. Thus, the hematite particles in heated PVG are separated from each other by the dominant mineral, quartz, so they cannot agglomerate and become darker. In fact, PVG becomes slightly brighter in color at the highest temperatures. Although agglomeration of the hematite crystals is not possible in PVG, recrystallization makes a more ordered crystal [57], resulting in brighter red at 1000 and 1100 °C.

The goethites PVA, TP3, and SABO all have the characteristic color of hematite after heating to 300, 600, and 800 °C (Figure 2, rows 3 to 5). However, above 800 °C, they become brown. These three goethites (PVA, TP3, and SABO), unlike SYN, PVG, and JPSS, contain significant quantities of metal carbonates [50]. Calcite and dolomite thermally decompose at high temperatures, producing white metal oxides, CaO and MgO, as per ref. [58], that reflect all wavelengths including blue and green. X-ray powder diffraction patterns for two ochres (PVA, SABO) confirm that metal oxides are in fact generated in these ochres at 1000 °C (see Supplemental Table S1). Thus, the change to brown can be attributed to decomposition of the calcite and dolomite in those ochres.

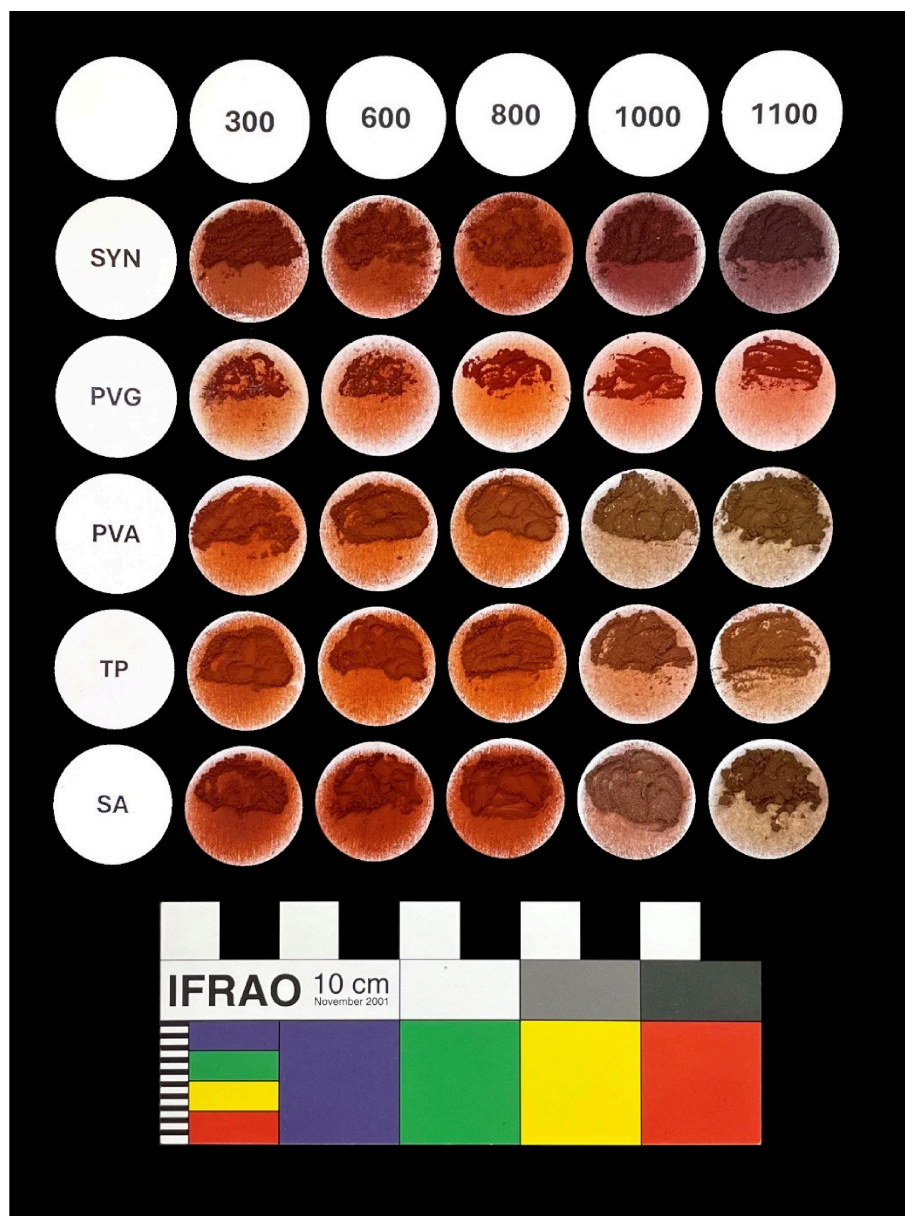


Figure 2. Photograph of five goethites after heating for 3 h at 300, 600, 800, 1000, and 1100 °C. In the upper half of each circle is a thick layer of pigment while in the lower half the pigment is rubbed into the white paper.

3.2. Munsell Codes and Colors

Table 2 contains the Munsell codes and colors for the heated goethites, and they are consistent with the colors illustrated in Figure 2. For all these ochres, the Munsell code and color are independent of temperature from 300 to 800 °C.

The Munsell code and color of PVG is constant from 300 to 1000 °C despite the slightly brighter appearance at the highest temperatures. The Munsell hue of SYN is independent of temperature up to 800 °C, but above 800 °C, the Munsell codes have a lower value (meaning they are darker) and lower chroma (less vivid color). JPSS, similar to SYN, has a Munsell color independent of temperature up to 800 °C, but becomes darker at 1100 °C. The Munsell colors of PVA, TP3, and SABO are independent of temperature up to 800 °C, but heating to 1000 °C results in Munsell colors and codes that are consistent with the brownish appearance of these ochres at the highest temperatures.

Table 2. Munsell codes and colors for SYN, PVG, JPSS PVA, SABO, and TP3 at 300 °C, 600 °C, 800 °C, 1000 °C, and 1100 °C after 3 h [59].

Pigment	Temperature/°C	Munsell Code	Munsell Color
SYN	300	2.5YR 4/6	Red
SYN	600	10R 4/6	Red
SYN	800	2.5YR 4/6	Red
SYN	1000	10R 3/3	Dusky Red
SYN	1100	5R 2.5/2	Very Dusky Red
PVG	300	10R 4/6	Red
PVG	600	10R 4/6	Red
PVG	800	10R 4/6	Red
PVG	1000	10R 4/6	Red
PVG	1100	10R 4/8	Red
JPSS	300	2.5YR 3/4	Dark Reddish Brown
JPSS	600	2.5YR 3/4	Dark Reddish Brown
JPSS	800	2.5YR 3/4	Dark Reddish Brown
JPSS	1000	2.5YR 3/4	Dark Reddish Brown
JPSS	1100	10R 3/2	Dusky Red
PVA	300	10R 4/6	Red
PVA	600	2.5YR 4/6	Red
PVA	800	2.5YR 5/4	Reddish Brown
PVA	1000	7.5YR 4/4	Brown
PVA	1100	10YR 4/4	Dark Yellow Brown
SABO	300	2.5YR 4/6	Red
SABO	600	10R 4/6	Red
SABO	800	2.5YR 4/6	Red
SABO	1000	2.5YR 5/3	Reddish Brown
SABO	1100	7.5YR 3/4	Dark Brown
TP3	300	2.5YR 4/8	Red
TP3	600	2.5YR 4/6	Red
TP3	800	2.5YR 4/6	Red
TP3	1000	10R 4/4	Weak Red
TP3	1100	5YR 4/6	Yellowish Red

3.3. Diffuse Reflectance Spectroscopy (DRS)

While Munsell colors offer a relatively objective characterization of color, diffuse reflectance spectroscopy (DRS), in which the percent reflectance of light is measured over the span of the visible spectrum, is more sensitive and quantitative than Munsell colors [40]. Using DRS, Elias et al. demonstrated that the “extremely wide range of [colors] from pale yellow to dark red” of ochres could be quantified, including in mixtures of pigments [60].

To achieve excellent precision when making DRS measurements, it is necessary to place the fiber optic of the spectrometer consistently at the same angle and distance from the sample. We found that to achieve optimal precision, the sample holder must be slightly overfilled. Then, covering the ochre with a high-quality microscope slide allows the fiber optic of the spectrometer to be placed precisely 1.0 mm, the thickness of a microscope glass slide, from the sample. Because careful filling is critical for precise quantification of

reflectance, we determine the precision by filling the sample holder, recording a spectrum, emptying the sample holder, and repeating. We recorded replicate spectra of all our samples, and the variability of peak reflectance is $\pm 2\%$ (standard deviation).

To illustrate this, in Figure 3 seven replicate spectra of synthetic goethite heated at 300 °C for 1 h are depicted. As expected [60], the reflectance of blue and green light (from 380 to 560 nm) from hematite is very low. Reflectance increases through the orange and red, peaking at 740 nm. The percent reflectance, %R, is a quantitative measure of brightness at each wavelength. The standard deviation of %R at 740 nm in these seven replicate spectra is $s = 2\%$.

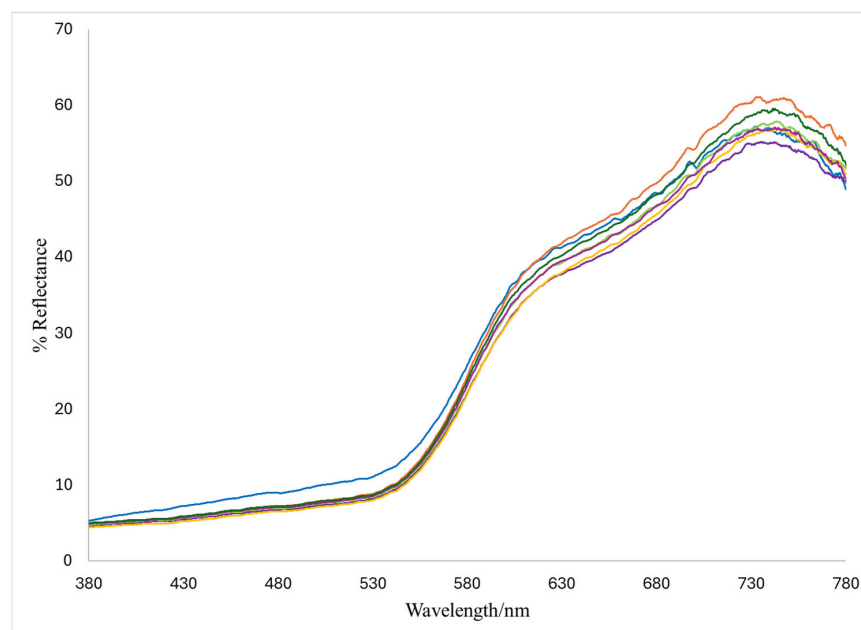


Figure 3. Seven replicate spectra of synthetic goethite, SYN, heated at 300 °C for 1 h to illustrate reproducibility of the spectrometry.

Consistent with their appearance and Munsell colors, the reflectance spectra of SYN, PVG, and JPSS are all independent of temperature from 300 to 800 °C (see Supplemental Figure S1). At higher temperatures the spectra become more interesting. Figure 4 demonstrates the differing effect of the highest temperatures on SYN and PVG.

For PVG, the reflectance of red light increases from 800 to 1000 to 1100 °C (Figure 4a–c) resulting in its brighter red color. In contrast, for SYN the reflectance of red light decreases, while reflectance of the blue wavelengths *increases* slightly (Figure 4d–f). The decrease in red and the increase in blue results in its dark purplish appearance (Figure 1). The spectra of SYN are consistent with the colorimetric measurements in which a^* and b^* decrease above 900 °C [36].

JPSS is 65% goethite, so it is intermediate between SYN (100%) and PVG (20%). The crystallites of hematite in heated JPSS are abundant enough that some can agglomerate into larger, darker crystals. The darkening is not as dramatic in JPSS compared to SYN because its intermediate purity allows a portion of the hematite crystals, but not all, to fuse.

To quantitatively graph the effect of temperature on reflectance for these three ochres, Figure 5 plots the %R at 740 nm vs. the temperature of heating. The %R of heated PVG is illustrated in the magenta bars, and it is constant from 300 to 800 °C (45.3 and 45.4%). After heating above 800 °C, the increase in brightness of PVG, barely apparent by visual inspection (Figure 1), is easily quantified by DRS, and the %R increases to 60.5% at 1100 °C. Note the increase in the height of the magenta bars with temperature (Figure 5a). Thus, when carefully performed, DRS can easily detect differences barely detected by eye.

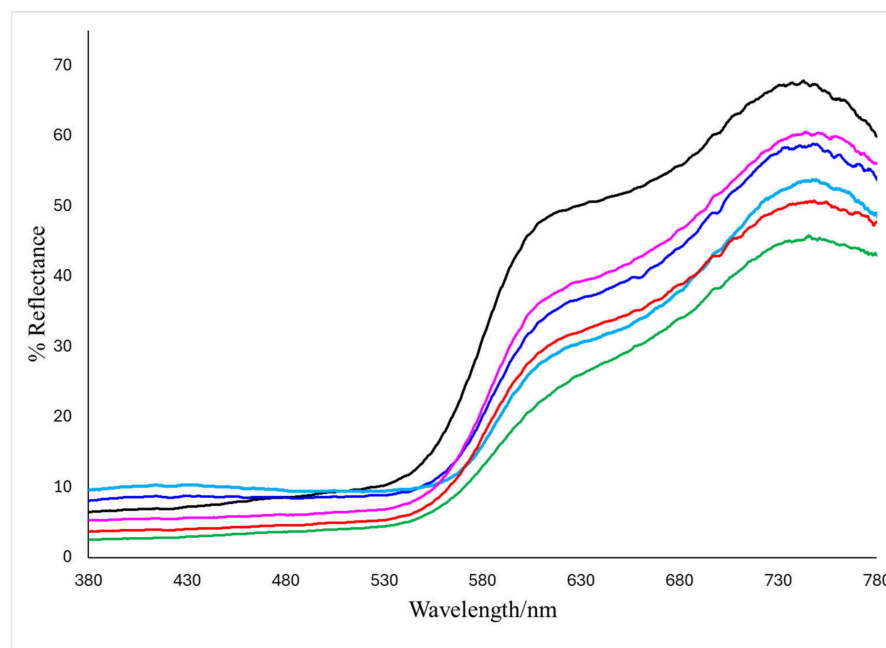


Figure 4. Diffuse reflectance spectra (DRSs) of two ochres heated for three hours at high temperatures. PVG: a. 800 °C (green); b. 1000 °C (red); c. 1100 °C (magenta). SYN: d. 800 °C (black); e. 1000 °C (blue); f. 1100 °C (light blue).

Unlike PVG, the %R of SYN at the highest temperatures *decreases* from 68% to 45%, again easily quantified by DRS. This is illustrated by the decrease in the height of the light green bars from 800 to 1000 to 1100 °C (Figure 5b). For JPSS, the %R is relatively constant from 300 to 1000 °C (41.8, 40.3, and 41.0%), but at 1100 °C the darkness (barely apparent by visual inspection) is easily quantified via the decrease of %R to 35.7% (dark green bars in Figure 5).

The colors of PVA, TP3, and SABO are independent of temperature from 300 to 800 °C, but they exhibit important color changes above 800 °C (Figure 6). Again, DRS allows for quantification of this effect. For example, Figure 6 shows the spectra of PVA after heating for 3 h at 300, 800, 1000, and 1100 °C. At higher temperatures, the reflectance of red light decreases while reflectance increases in the blue, green and yellow range. By 1100 °C, the reflectance of blue has almost doubled and the reflectance of red has decreased by one-third compared to the spectrum at 300 °C (Figure 6a,d). These measurements quantify the observation made by Munsell color, and they are consistent with the color change from red to light brown. DRSs of SABO are very similar to those of PVA (The commercial pigment LO similarly contains carbonates and shows the same effect on color, as discussed below in Section 3.5).

In contrast, the reflectance of all wavelengths of heated TP3 increases steadily as the temperature increases from 300 to 1100 °C (Figure 7), but the effect is small—only 5 to 10%. Although this increase in reflectance is reproducible and easily detected by DRS, no change in color is apparent by visible inspection up to 800 °C, and its appearance changes only slightly up to 1100 °C (Figure 2).

In DRSs, yellow goethites typically have an inflection point, λ_m , of about 550 nm [60]. With conversion to hematite, the band shifts to longer, redder wavelengths, and the inflection point for pure hematite is typically equal to or greater than 580 nm. In this study, we determined the λ_m of many of the goethites at various temperatures and times, and the values ranged from 580 to 615 nm (Supplemental Tables S2–S4). This is consistent with complete conversion to hematite, at least for the samples that were measured.

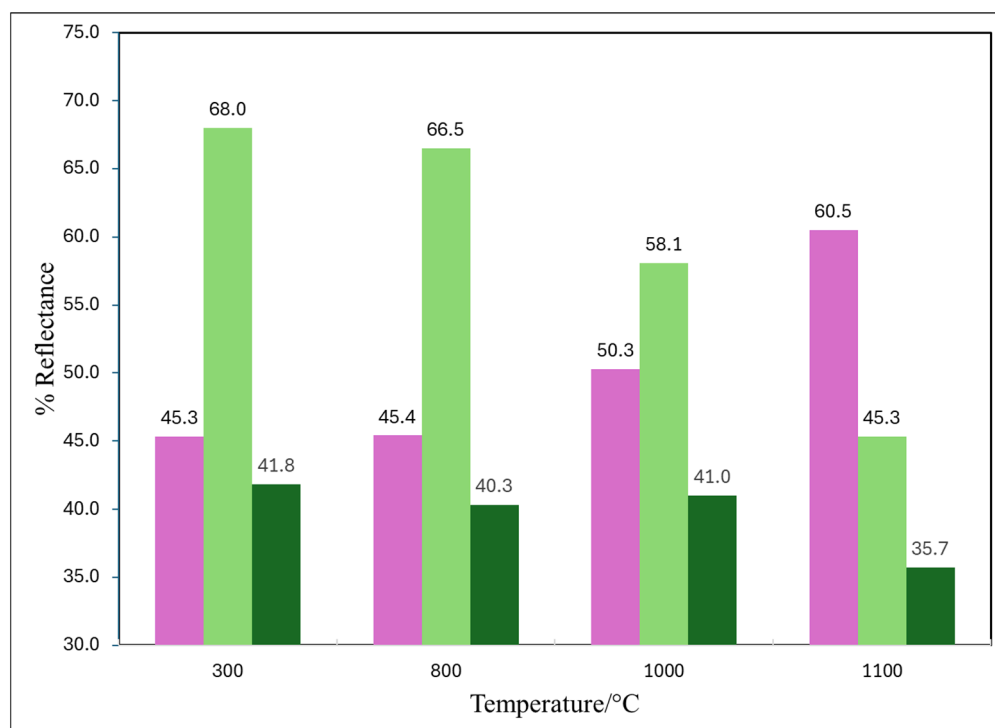


Figure 5. Bar chart of the % reflectance at 740 nm of a. PVG (magenta), b. SYN (light green) and c. JPSS (dark green) heated for 3 h at 300, 800, 1000, and 1100 °C. (Results at 600 °C are similar to 300 and 800 °C, so are not illustrated here to avoid complicating the figure.)

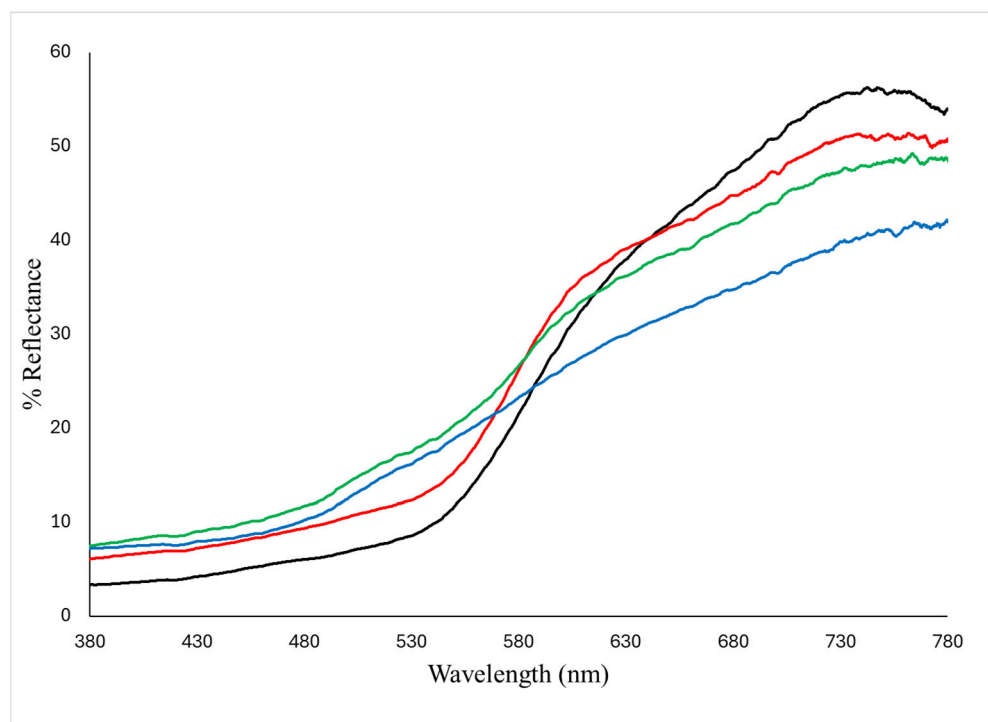


Figure 6. Reflectance spectra of PVA heated for 3 h at a. 300 °C (black), b. 800 °C (red), c. 1000 °C (green), and d. 1100 °C (blue).

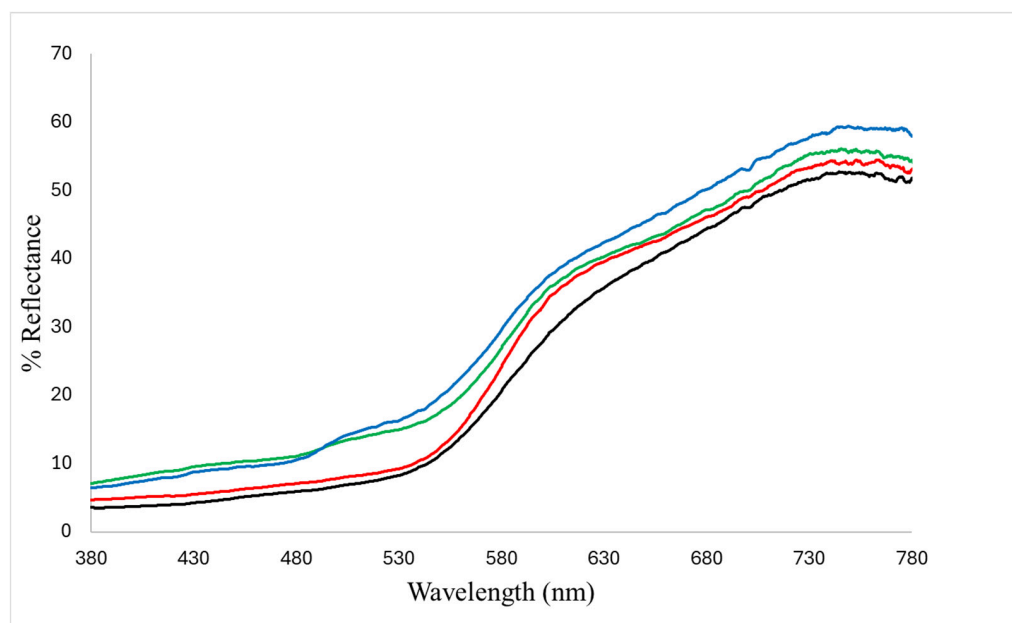


Figure 7. Reflectance spectra of TP3 heated for 3 h at 300 °C (black), 800 °C (red), 1000 °C (green), and 1100 °C (blue).

3.4. Effect of Time on Color

In addition to the effect of temperature, we have also studied the effect of time. Thus, we heated SYN, PVG, PVA, SABO, and TP3 for 1, 3, 8, and 16 h. Time of heating has a minimal effect on any of the pigments at either 300 or 800 °C. For example, the appearance and Munsell colors are the same for PVG at 300 °C from 1 to 16 (10R 4/6). To quantify this, Figure 8 illustrates the reflectance spectra for PVG heated at 300 °C for 1, 3, 8, and 16 h, and they overlap nicely. The variation in reflectance (45 to 47%) is well within the precision of the technique. The same is true when PVG is heated at 800 °C at various times except that %R at 740 nm increases very slightly (<5%) after heating 16 h (Supplemental Figure S2).

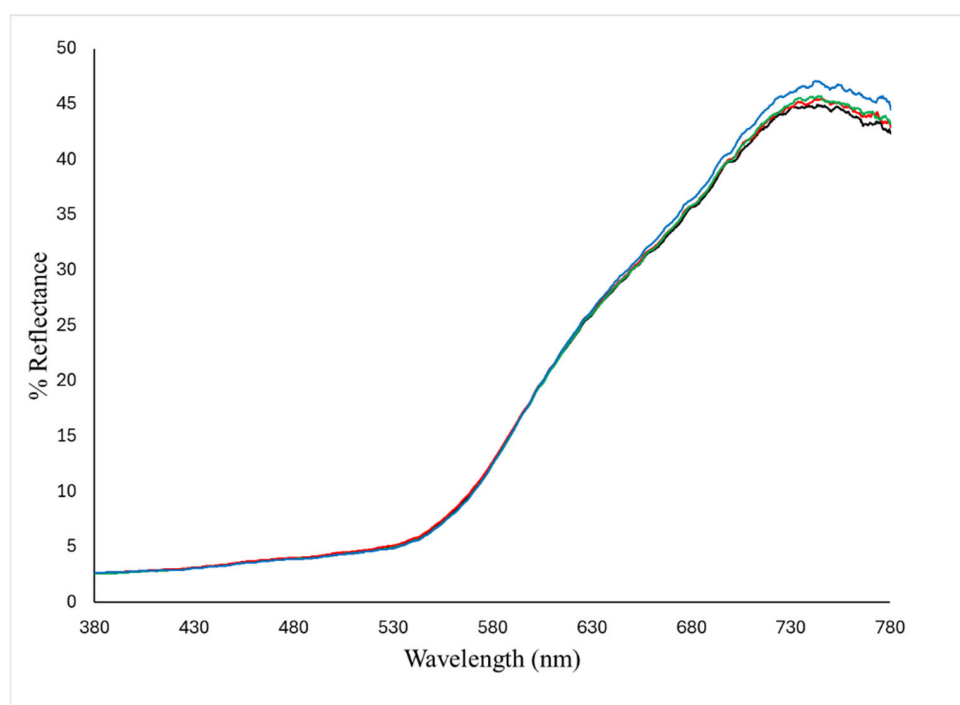


Figure 8. Reflectance spectra of PVG heated at 300 °C for 1 (black), 3 (red), 8 (green), and 16 h (blue).

In fact, there is a minimal effect of time on color for any of the pigments at 300 or 800 °C (see Supplemental Figures S3–S6). The kinetics of the conversion of goethite to hematite is typically rapid at 300 °C, so the conversion is complete after one hour [3,56,61]. Thus, the longer heating time is not expected to change the color or reflectance spectra for most goethites, and the spectra in Supplemental Figures S3–S6 exhibit only slight variation.

3.5. Temperature Dependence for Heating Six Commercial Goethites

Commercial pigments undergo considerable processing including grinding, blending, and proprietary additives [36,40,47]. For example, mechanical stress can induce structural defects or partial amorphization in mineral phases, thereby altering their thermal behavior and the durability of the resulting materials [51]. In spite of these potential complications, we include in this study six natural yellow ochres containing goethite that were purchased from Rublev Colours™. Figure 9 illustrates the palette and Table 3 lists the Munsell colors before and after heating the six Rublev yellow ochres at 300, 400, 600, 900, 1000, and 1100 °C. (See Table 1 for complete names and abbreviations of the pigments.) As can be seen in Figure 9, the colors of four (BRY, YOL, IYE, and JCL) show no obvious change in color with the temperature from 300 to 1100 °C.

LO, which is the only one of the Rublev pigments reported to contain significant amounts of carbonates, ref. [48] (<https://www.naturalpigments.com> (accessed on 5 May 2026)), becomes light brown at 1000 °C. This is the same effect demonstrated in PVA, TP3, and SABO with carbonates decomposing to metal oxides [58] that reflect green and blue light, thereby producing a brownish color as discussed in more detail above in Section 3.1.

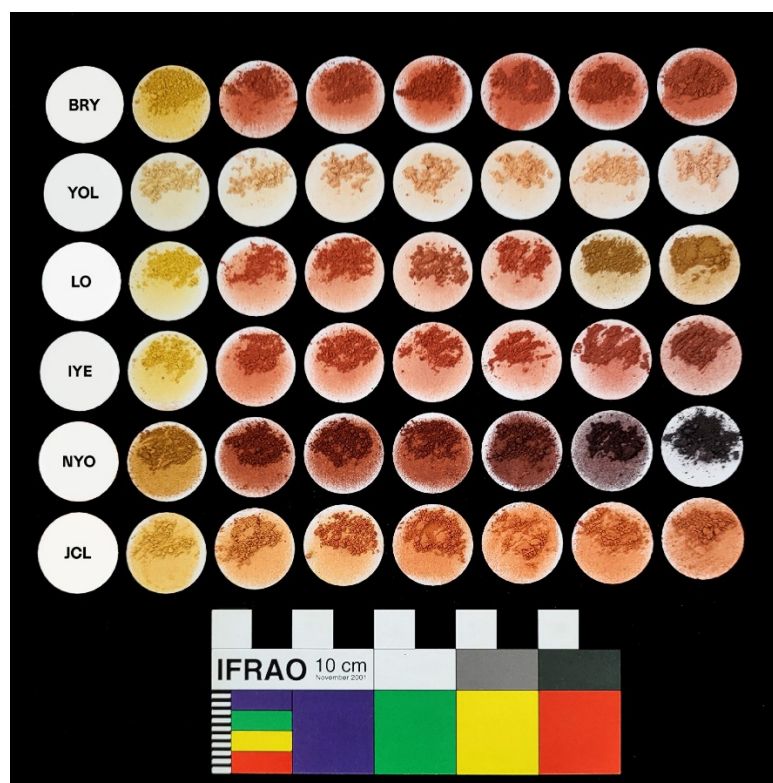


Figure 9. Photograph of six Rublev Colours™ yellow ochres (natural goethite). From left to right, unheated, 300, 400, 600, 900, 1000 and 1100 °C.

NYO demonstrates the most dramatic change in color. At 900 °C it becomes brown (2.5YR 2.5/3) and at 1000 °C almost black (5R 2.5/1). Powder X-ray diffractograms of

heated NYO did not offer evidence for what causes the dark color, but it may be due to accessory minerals or proprietary additives.

Table 3. Munsell codes and colors for Rublev Colors TM pigments: unheated, 300, 400, 600, 900, 1000, and 1100 °C.

Pigment	Temperature/°C	Munsell Code	Munsell Color
BRY	Unheated	10YR 7/6	Yellow
BRY	300	10R 4/6	Red
BRY	400	10R 4/6	Red
BRY	600	10R 4/6	Red
BRY	900	10R 3/6	Dark Red
BRY	1000	10R 3/6	Dark Red
BRY	1100	10R 4/6	Red
YOL	Unheated	2.5Y 8/4	Pale Brown
YOL	300	7.5YR 8/6	Reddish Yellow
YOL	400	5YR 7/6	Reddish Yellow
YOL	600	5YR 7/6	Reddish Yellow
YOL	900	5YR 7/6	Reddish Yellow
YOL	1000	5YR 7/6	Reddish Yellow
YOL	1100	2.5YR 7/4	Light Reddish Brown
LO	Unheated	2.5Y 8/8	Yellow
LO	300	10R 4/6	Red
LO	400	10R 4/6	Red
LO	600	10R 4/4	Weak Red
LO	900	10R 4/6	Red
LO	1000	2.5Y 4/4	Olive Brown
LO	1100	10YR 4/6	Dark Yellowish Brown
IYE	Unheated	2.5Y 7/6	Yellow
IYE	300	10R 4/8	Red
IYE	400	10R 4/6	Red
IYE	600	10R 3/6	Dark Red
IYE	900	10R 3/6	Dark Red
IYE	1000	7.5R 3/5	Dusky Red
IYE	1100	10R 4/4	Weak Red
NYO	Unheated	10YR 5/6	Yellowish Brown
NYO	300	2.5YR 3/6	Dark Red
NYO	400	2.5YR 3/6	Dark Red
NYO	600	2.5YR 3/6	Dark Red
NYO	900	2.5YR 2.5/3	Dark Reddish Brown
NYO	1000	5R 2.5/1	Reddish Black
NYO	1100	GLE Y1 2.5/N	Black
JCL	Unheated	10YR 6/6	Brownish Yellow

Table 3. Cont.

Pigment	Temperature/°C	Munsell Code	Munsell Color
JCL	300	5YR 5/6	Yellowish Red
JCL	400	2.5YR 4/6	Red
JCL	600	2.5YR 4/8	Red
JCL	900	2.5YR 4/6	Red
JCL	1000	2.5 YR 4/6	Red
JCL	1100	2.5 YR 5/6	Red

Diffuse reflectance spectra are all consistent with the colors in Figure 9 and the Munsell colors in Table 3. In Figure 10 the reflectance spectra for the six Rublev pigments after heating at 1000 °C for 3 h are presented. The spectra of BRY, IYE, and JCL (Figure 10a,d,f) are typical of hematite. YOL, the lightest of the pigments, shows significant reflectance across the visible spectrum, but most intensely in the red wavelengths (Figure 10b), consistent with its pale pink color and high content of white clay [48]. LO reflects mostly red, but the reflectance of the green portion of the spectrum (450–550 nm) is high (Figure 10c) resulting in its light brown color. Most dramatic, the spectrum of NYO reflects little light at any wavelengths (less than 25% in the red and about 6% through the blue, green, and yellow) consistent with reddish black.

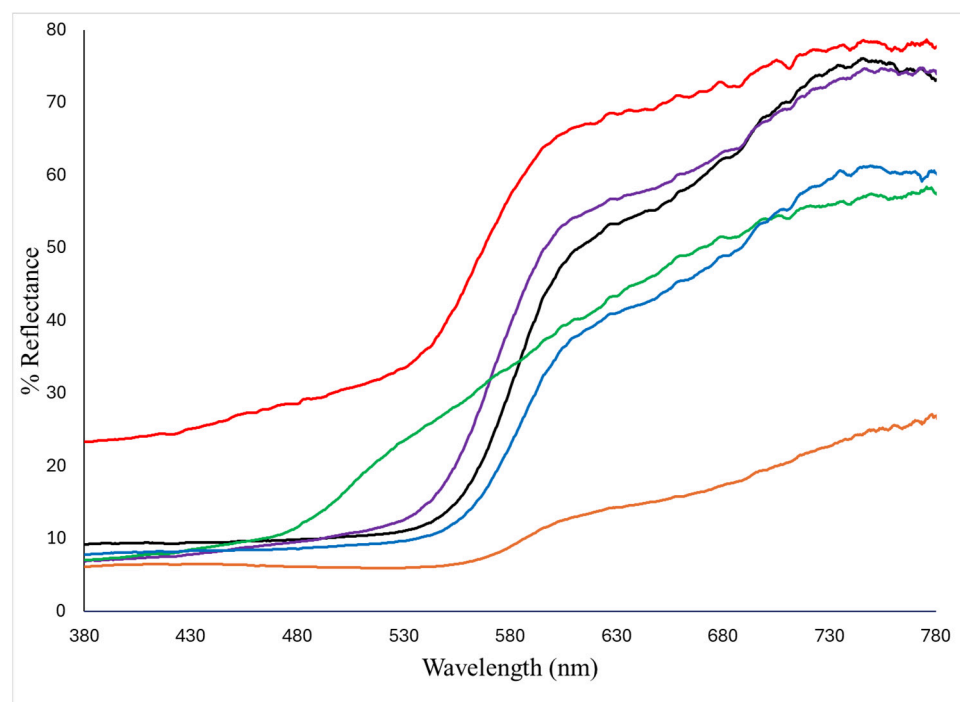


Figure 10. Reflectance spectra of six Rublev Colors™ goethites after heating to 1000 °C for 3 h. a. BRY (black), b. YOL (red), c. LO (green), d. IYE (blue), e. NYO (orange), f. JCL (purple).

4. Discussion

The ochres studied here vary widely in source, geological origin, texture, color, goethite concentration, and accessory minerals. Thus it is remarkable that in every case, when the goethite is converted to hematite, the temperature of conversion from 300 to 800 °C has virtually no influence on the color of the resulting ochre. This temperature range is of particular importance to the understanding of early human behavior because with simple wood fires it is difficult to achieve and sustain temperatures over 800 °C [46,52–54].

In the late Neolithic, humans devised kilns and furnaces that can achieve higher temperatures. Thus we also studied the effect of higher temperatures on colors of the hematites produced. Above 800 °C, there can be three effects. a. If the goethite is almost pure, very small hematite crystals can agglomerate into larger crystals, giving the ochre a dark purplish color. In that case, the %R of red is lower, and the %R of blue is higher. This was the case in pure synthetic goethite (SYN) as well as in the ochre from Minnesota, USA (JPSS), which is almost 70% goethite. b. In the presence of accessory minerals, agglomeration is prevented by mechanical separation of crystallites, and the darker, purplish color does not form. If the accessory minerals are inert (e.g., quartz), recrystallization at highest temperatures may result in brighter red pigments and a higher percent reflectance (for example in PVG). c. In the presence of carbonates (e.g., PVA, SABO, TP3, LO), heating above 800 °C results in light brown pigments.

The combination of photography, Munsell colors, and DRS is a powerful strategy to study the colors of ochres because it offers objective descriptions of color, both qualitative and quantitative. This study contradicts some earlier suggestions that the color of ochres could have been controlled by temperature or time of heating [43,44], and it complements and extends important work in this field [36,40,47].

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/colorants5030024/s1>, Figure S1. Reflectance spectra of PVG heated at 300 °C (1 h: black; 3 h: red) and 800 °C (1 h: green; 3 h: blue). Figure S2. Reflectance spectra of PVG heated at 800 °C for 1 (black), 3 (red), 8 (green), and 16 h (blue). Figure S3. Reflectance spectra of SABO heated at 300 °C for 1 (black), 3 (red), 8 (green), and 16 h (blue). Figure S4. Reflectance spectra of SABO heated at 800 °C for 1 (black), 3 (red), 8 (green), and 16 h (blue). Figure S5. Reflectance spectra of PVA heated at 300 °C for 1 (black), 3 (red), 8 (green), and 16 h (blue). Figure S6. Reflectance spectra of PVA heated at 800 °C for 1 (black), 3 (red), 8 (green), and 16 h (blue). Table S1. Minerals identified by XRD in five natural ochres before and after heating. Table S2. Inflection points, λ_m , of unheated and heated (one hour) goethites in nanometers. Table S3. Inflection points, λ_m , of heated (three hours) goethites in nanometers. Table S4. Inflection points, λ_m , of heated (sixteen hours) goethites in nanometers.

Author Contributions: Conceptualization, W.J.B. and G.C.; methodology, I.F.B., L.N.H., J.E.R., K.W., G.C. and W.J.B.; software, I.F.B., L.N.H., J.E.R., K.W., G.C. and W.J.B.; validation, I.F.B., L.N.H., J.E.R., K.W., G.C. and W.J.B.; formal analysis, I.F.B., L.N.H., J.E.R., K.W., G.C. and W.J.B.; investigation, I.F.B., L.N.H., J.E.R., K.W., G.C. and W.J.B.; resources, W.J.B.; data curation, W.J.B.; writing—original draft preparation, W.J.B.; writing—review and editing, I.F.B., L.N.H., J.E.R., K.W., G.C. and W.J.B.; visualization, I.F.B., L.N.H., J.E.R., K.W., G.C. and W.J.B.; supervision, W.J.B.; project administration, W.J.B.; funding acquisition, W.J.B. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by Hobart and William Smith Colleges, grant number 5-21-24.

Institutional Review Board Statement: Not applicable.

Data Availability Statement: All data is provided in the article.

Acknowledgments: We thank Jessica Finnerty, Angelina Jordan, Zhenyu Lin, Ameer Muse, Jules Picuri, and Shruthi Shyam for technical assistance heating some samples.

Conflicts of Interest: The authors declare no conflicts of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

Abbreviations

The following abbreviations are used in this manuscript:

XRD	X-ray Diffraction
DRS	Diffuse Reflectance Spectroscopy

References

- Ilmi, M.M.; Maryanti, E.; Tjan, I.P.; Oktaviana, A.A.; Mas'ud, Z.; Saiyasombat, C.; Noerwidi, S. The First Insight to Materiality of Rock Art Pigments from Western Papua Region (Berau Gulf, Fakfak). *Archaeol. Anthropol. Sci.* **2025**, *17*, 51. [\[CrossRef\]](#)
- Langley, M.C.; Litster, M. The Paleolithic in Color: Color and Other Visual Qualities in Archaeological Discussions of Early Symbolic Behavior. *Top. Cogn. Sci.* **2025**, *18*, e70033. [\[CrossRef\]](#) [\[PubMed\]](#)
- Mastrotheodoros, G.P.; Beltsios, K.G. Pigments—Iron-Based Red, Yellow, and Brown Ochres. *Archaeol. Anthropol. Sci.* **2022**, *14*, 35. [\[CrossRef\]](#)
- Popelka-Filcoff, R.S.; Zipkin, A.M. The Archaeometry of Ochre Senu Lato: A Review. *J. Archaeol. Sci.* **2022**, *137*, 105530. [\[CrossRef\]](#)
- Rosso, D.E.; Regert, M.; d'Errico, F. First Identification of an Evolving Middle Stone Age Ochre Culture at Porc-Epic Cave, Ethiopia. *Sci. Rep.* **2023**, *13*, 13261. [\[CrossRef\]](#) [\[PubMed\]](#)
- Ilmi, M.M.; Maryanti, E.; Nurdini, N.; Setiawan, P.; Kadja, G.T.M. Ismunandar A Review of Radiometric Dating and Pigment Characterizations of Rock Art in Indonesia. *Archaeol. Anthropol. Sci.* **2021**, *13*, 120. [\[CrossRef\]](#)
- Henshilwood, C.S.; d'Errico, F.; van Niekerk, K.L.; Coquinot, Y.; Jacobs, Z.; Lauritzen, S.-E.; Menu, M.; García-Moreno, R. A 100,000-Year-Old Ochre-Processing Workshop at Blombos Cave, South Africa. *Sci. New Ser.* **2011**, *334*, 219–222. [\[CrossRef\]](#)
- Dapschaskas, R.; Göden, M.B.; Sommer, C.; Kandel, A.W. The Emergence of Habitual Ochre Use in Africa and Its Significance for The Development of Ritual Behavior During The Middle Stone Age. *J. World Prehist.* **2022**, *35*, 233–319. [\[CrossRef\]](#)
- Helwig, K. Iron Oxide Pigments: Natural and Synthetic. In *Artists' Pigments: A Handbook of their History and Characteristics*; National Gallery of Art: Washington, DC, USA, 2007; Volume 4, pp. 39–109.
- Velliky, E.C. Unveiling the Multifunctional Use of Ochre in the Middle Stone Age: Specialized Ochre Retouchers from Blombos Cave. *Sci. Adv.* **2025**, *11*, eads2797. [\[CrossRef\]](#) [\[PubMed\]](#)
- Velliky, E.C.; MacDonald, B.L.; Porr, M.; Conard, N.J. First Large-scale Provenance Study of Pigments Reveals New Complex Behavioural Patterns during the Upper Palaeolithic of South-western Germany. *Archaeometry* **2021**, *63*, 173–193. [\[CrossRef\]](#)
- Dayet, L. Invasive and Non-Invasive Analyses of Ochre and Iron-Based Pigment Raw Materials: A Methodological Perspective. *Minerals* **2021**, *11*, 210. [\[CrossRef\]](#)
- Dayet, L.; d'Errico, F.; Garcia-Moreno, R. Searching for Consistencies in Châtelperronian Pigment Use. *J. Archaeol. Sci.* **2014**, *44*, 180–193. [\[CrossRef\]](#)
- Hunt, A.; Thomas, P.; James, D.; David, B.; Geneste, J.-M.; Delannoy, J.-J.; Stuart, B. The Characterisation of Pigments Used in X-Ray Rock Art at Dalakngalarr 1, Central-Western Arnhem Land. *Microchem. J.* **2016**, *126*, 524–529. [\[CrossRef\]](#)
- Kleijne, J.P.; Bērziņš, V.; Huisman, D.J.; Kalniņš, M.; Krause-Kyora, B.; Meadows, J.; van Os, B.J.H.; Schmölcke, U.; Steinhagen, F.; Lübke, H. No Time to Waste. Evidence for Communal Waste Management among Hunter-Gatherer-Fishers at Rīņņukalns, Latvia (5400–3200 BC). *Quat. Environ. Hum.* **2024**, *2*, 100003. [\[CrossRef\]](#)
- MacDonald, B.L.; Chatters, J.C.; Reinhardt, E.G.; Devos, F.; Meacham, S.; Rissolo, D.; Rock, B.; Le Maillot, C.; Stalla, D.; Marino, M.D.; et al. Paleindian Ochre Mines in the Submerged Caves of the Yucatán Peninsula, Quintana Roo, Mexico. *Sci. Adv.* **2020**, *6*, eaba1219. [\[CrossRef\]](#) [\[PubMed\]](#)
- Román, R.S.; Bañón, C.B.; Landete Ruiz, M.D. Analysis of the Red Ochre of the El Mirón Burial (Ramales de La Victoria, Cantabria, Spain). *J. Archaeol. Sci.* **2015**, *60*, 84–98. [\[CrossRef\]](#)
- Seva Román, R.; Landete Ruiz, M.D.; Juan-Juan, J.; Biete Bañón, C.; Straus, L.G.; González Morales, M.R. Sources of the Ochres Associated with the Lower Magdalenian “Red Lady” Human Burial and Rock Art in El Mirón Cave (Cantabria, Spain). *J. Archaeol. Sci. Rep.* **2019**, *23*, 265–280. [\[CrossRef\]](#)
- Shixia, Y.; Jingwen, X.U.; Faxiang, H. Significance of Modern Human Exploration of Ochre in Its Evolution. *Acta Anthropol. Sin.* **2022**, *41*, 649–658. [\[CrossRef\]](#)
- Botticelli, M.; Altamura, F.; Catelli, E.; Pronti, L.; Medeghini, L.; Mussi, M.; Maras, A. Colorimetric Characterization of Ochres in a Palaeolithic Flint Pebble from Maschio Dell'Artemisio, Latium, Italy. *J. Archaeol. Sci. Rep.* **2022**, *43*, 103420. [\[CrossRef\]](#)
- Garilli, V.; Vita, G.; La Parola, V.; Pinto Vraca, M.; Giarrusso, R.; Rosina, P.; Bonfiglio, L.; Sineo, L. First Evidence of Pleistocene Ochre Production from Bacteriogenic Iron Oxides. A Case Study of the Upper Palaeolithic Site at the San Teodoro Cave (Sicily, Italy). *J. Archaeol. Sci.* **2020**, *123*, 105221. [\[CrossRef\]](#)
- Garilli, V.; Galletti, L. Vestiges of the Sedimentary Archive of Late Paleolithic Inhumations from San Teodoro Cave: Insights into ST3 Burial and Site Stratigraphy. *Heritage* **2025**, *8*, 285. [\[CrossRef\]](#)

23. Ismunandar, I.; Nurdini, N.; Ilmi, M.M.; Maryanti, E.; Kadja, G.T.M. Investigation on the Crystal Structures of Hematite Pigments at Different Sintering Temperatures. *KEM* **2021**, *874*, 20–27. [\[CrossRef\]](#)
24. MacDonald, B.L.; Velliky, E.C.; Forrester, B.; Riedesel, S.; Linstädter, J.; Kuo, A.L.; Woodborne, S.; Mabuza, A.; Bader, G.D. Ochre Communities of Practice in Stone Age Eswatini. *Nat. Commun.* **2024**, *15*, 9201. [\[CrossRef\]](#) [\[PubMed\]](#)
25. Nurdini, N.; Maryanti, E.; Ilmi, M.M.; Setiawan, P.; Saiyasombat, C.; Kadja, G.T.M. Ismunandar Physicochemical Investigation of Prehistoric Rock Art Pigments in Tewet Cave, Sangkulirang-Mangkalihat Site, East Kalimantan-Indonesia. *J. Archaeol. Sci. Rep.* **2020**, *31*, 102345. [\[CrossRef\]](#)
26. Gurin, C.; Mazzuca, M.; Gómez Otero, J.; Maier, M.S. Chemical Characterization of Red Pigments Used in Funerary Practices in Northeastern Patagonia (Chubut, Argentina) During the Late Holocene. *Minerals* **2025**, *15*, 1055. [\[CrossRef\]](#)
27. Schwertmann, U.; Cornell, R.M. *Iron Oxides in the Laboratory: Preparation and Characterization*; John Wiley & Sons: Hoboken, NJ, USA, 2008.
28. Cavallo, G.; Ischia, G.; Zorzin, R.; Gialanella, S. Experimental Analysis on Natural Heated Goethite from Ponte Di Veja (Mt Lessini, NE Italy). *J. Archaeol. Sci. Rep.* **2021**, *36*, 102871. [\[CrossRef\]](#)
29. Salomon, H.; Vignaud, C.; Lahilil, S.; Menguy, N. Solutrean and Magdalenian Ferruginous Rocks Heat-Treatment: Accidental and/or Deliberate Action? *J. Archaeol. Sci.* **2015**, *55*, 100–112. [\[CrossRef\]](#)
30. Salomon, H.; Vignaud, C.; Coquinot, Y.; Beck, L.; Stringer, C.; Strivay, D.; D'Errico, F. SELECTION AND HEATING OF COLOURING MATERIALS IN THE MOUSTERIAN LEVEL OF ES-SKHUL (c. 100 000 YEARS BP, MOUNT CARMEL, ISRAEL). *Archaeometry* **2012**, *54*, 698–722. [\[CrossRef\]](#)
31. Cavallo, G.; Fontana, F.; Gialanella, S.; Gonzato, F.; Riccardi, M.P.; Zorzin, R.; Peresani, M. Heat Treatment of Mineral Pigment During the Upper Palaeolithic in North-East Italy. *Archaeometry* **2018**, *60*, 1045–1061. [\[CrossRef\]](#)
32. De Faria, D.L.A.; Lopes, F.N. Heated Goethite and Natural Hematite: Can Raman Spectroscopy Be Used to Differentiate Them? *Vib. Spectrosc.* **2007**, *45*, 117–121. [\[CrossRef\]](#)
33. Périnet, G.; Onoratini, G. A propos des colorants rouges préhistoriques. *ArchéoSciences* **1987**, *11*, 49–51. [\[CrossRef\]](#)
34. Picuri, J.C.; Natoli, J.M.; Shaw, S.E.; Shyam, S.P.; VanHoesen, S.R.; Lin, Z.; Bowyer, W.J. Can X-Ray Diffraction Distinguish Natural from Anthropogenic Hematite? Replication of the Conversion of Natural Goethite in Both Furnace and Campfire. *Colorants* **2022**, *1*, 363–375. [\[CrossRef\]](#)
35. Pomiés, M.-P.; Menu, M.; Vignaud, C. Red Palaeolithic Pigments: Natural Hematite or Heated Goethite? *Archaeometry* **1999**, *41*, 275–285. [\[CrossRef\]](#)
36. Castagnotto, E.; Locardi, F.; Slimani, S.; Peddis, D.; Gaggero, L.; Ferretti, M. Characterization of the Caput Mortuum Purple Hematite Pigment and Synthesis of a Modern Analogue. *Dye. Pigment.* **2021**, *185*, 108881. [\[CrossRef\]](#)
37. De Oliveira, L.F.C.; Edwards, H.G.M.; Frost, R.L.; Klopogge, J.T.; Middleton, P.S. Caput Mortuum: Spectroscopic and Structural Studies of an Ancient Pigment. *Analyst* **2002**, *127*, 536–541. [\[CrossRef\]](#) [\[PubMed\]](#)
38. Garofano, I.; Perez-Rodriguez, J.L.; Robador, M.D.; Duran, A. An Innovative Combination of Non-Invasive UV-Visible-FORS, XRD and XRF Techniques to Study Roman Wall Paintings from Seville, Spain. *J. Cult. Herit.* **2016**, *22*, 1028–1039. [\[CrossRef\]](#)
39. Li, Q.; Wu, A.; Zhang, M.; Li, J.; Cao, J.; Li, H.; Jiang, Y. Study on the Influence of Calcination Temperature of Iron Vitriol on the Coloration of Ancient Chinese Traditional Iron Red Overglaze Color. *Materials* **2024**, *17*, 2800. [\[CrossRef\]](#) [\[PubMed\]](#)
40. Botticelli, M.; Appolloni, G.; Candeias, A.; Mirão, J.; Maras, A. Synthesis of Yellow and Red Ochre: A Preliminary Multi-Analytical, Micro-Invasive Investigation on Recipes and Recognition Criteria. *Eur. Phys. J. Plus* **2021**, *136*, 322. [\[CrossRef\]](#)
41. Goss, C.J. The Kinetics and Reaction Mechanism of the Goethite to Hematite Transformation. *Mineral. Mag.* **1987**, *51*, 437–451. [\[CrossRef\]](#)
42. Pomiés, M.P.; Morin, G.; Vignaud, C. XRD Study of the Goethite-Hematite Transformation: Application to the Identification of Heated Prehistoric Pigments. *Eur. J. Solid State Inorg. Chem.* **1998**, *35*, 9–25. [\[CrossRef\]](#)
43. Leroi-Gourhan, A. Les fouilles d'Arcy-sur-Cure (Yonne). *Galip* **1961**, *4*, 3–16. [\[CrossRef\]](#)
44. Barnett, J.R.; Miller, S.; Pearce, E. Colour and Art: A Brief History of Pigments. *Opt. Laser Technol.* **2006**, *38*, 445–453. [\[CrossRef\]](#)
45. Legodi, M.A.; De Waal, D. The Preparation of Magnetite, Goethite, Hematite and Maghemite of Pigment Quality from Mill Scale Iron Waste. *Dye. Pigment.* **2007**, *74*, 161–168. [\[CrossRef\]](#)
46. Lin, Z.; Natoli, J.M.; Picuri, J.C.; Shaw, S.E.; Bowyer, W.J. Replication of the Conversion of Goethite to Hematite to Make Pigments in Both Furnace and Campfire. *J. Archaeol. Sci. Rep.* **2021**, *39*, 103134. [\[CrossRef\]](#)
47. Mastrotheodoros, G.; Beltsios, K.G.; Zacharias, N. Assessment of the production of antiquity pigments through experimental treatment of ochres and other iron based precursors. *Mediterr. Archaeol. Archaeom.* **2010**, *10*, 37.
48. Earth Pigments for Artists—Natural Ochres, Siennas & Umbers. Available online: https://www.naturalpigments.com/pigments/pigments-by-color/earth-pigments.html?_gl=1*ksvuhy*_up*MQ..*_gs*MQ..&gclid=CjwKCAiAtq_NBhA_EiwA78nNWHYId2sshoNdpt-DoZ2OCPwqKEN_aHcDLav1WF75BdA5rGTKPPnTgxoCDf4QAvD_BwE&gbruid=0AAAAAD_X-acYWxkRuadNDwYS6IKg6V39h (accessed on 7 March 2026).
49. Schwertmann, U.; Taylor, R.M. The Influence of Silicate on the Transformation of Lepidocrocite to Goethite. *Clays Clay Min.* **1972**, *30*, 159–164. [\[CrossRef\]](#)

50. Cavallo, G.; Riccardi, M.P.; Zorzin, R. Powder Diffraction of Yellow and Red Natural Earths from Lessini Mountains in NE Italy. *Powder Diffr.* **2015**, *30*, 122–129. [[CrossRef](#)]
51. Miriello, D.; Bloise, A.; Crisci, G.M.; Barrese, E.; Apollaro, C. EFFECTS OF MILLING: A POSSIBLE FACTOR INFLUENCING THE DURABILITY OF HISTORICAL MORTARS. *Archaeometry* **2010**, *52*, 668–679. [[CrossRef](#)]
52. Aldeias, V.; Dibble, H.L.; Sandgathe, D.; Goldberg, P.; McPherron, S.J.P. How Heat Alters Underlying Deposits and Implications for Archaeological Fire Features: A Controlled Experiment. *J. Archaeol. Sci.* **2016**, *67*, 64–79. [[CrossRef](#)]
53. Bentsen, S.E. Controlling the Heat: An Experimental Approach to Middle Stone Age Pyrotechnology. *S. Afr. Archaeol. Bull.* **2013**, *68*, 137–145.
54. March, R.J.; Lucquin, A.; Joly, D.; Ferreri, J.C.; Muhieddine, M. Processes of Formation and Alteration of Archaeological Fire Structures: Complexity Viewed in the Light of Experimental Approaches. *J. Archaeol. Method Theory* **2014**, *21*, 1–45. [[CrossRef](#)]
55. Murphree, W.C.; Aldeias, V. The Evolution of Pyrotechnology in the Upper Palaeolithic of Europe. *Archaeol. Anthropol. Sci.* **2022**, *14*, 202. [[CrossRef](#)]
56. Gialanella, S.; Girardi, F.; Ischia, G.; Lonardelli, I.; Mattarelli, M.; Montagna, M. On the Goethite to Hematite Phase Transformation. *J. Therm. Anal. Calorim.* **2010**, *102*, 867–873. [[CrossRef](#)]
57. Gialanella, S.; Belli, R.; Dalmeri, G.; Lonardelli, I.; Mattarelli, M.; Montagna, M.; Toniutti, L. ARTIFICIAL OR NATURAL ORIGIN OF HEMATITE-BASED RED PIGMENTS IN ARCHAEOLOGICAL CONTEXTS: THE CASE OF RIPARO DALMERI (TRENTO, ITALY). *Archaeometry* **2011**, *53*, 950–962. [[CrossRef](#)]
58. Dean, W.E. Determination of Carbonate and Organic Matter in Calcareous Sediments and Sedimentary Rocks by Loss on Ignition: Comparison With Other Methods. *J. Sediment. Res.* **1974**, *44*, 242–248. [[CrossRef](#)]
59. Munsell Color. *Munsell Soil Color Book: With Genuine Munsell Color Chips*; Munsell Color: Grand Rapids, MI, USA, 2019.
60. Elias, M.; Chartier, C.; Prévot, G.; Garay, H.; Vignaud, C. The Colour of Ochres Explained by Their Composition. *Mater. Sci. Eng. B* **2006**, *127*, 70–80. [[CrossRef](#)]
61. Löffler, L.; Mader, W. Anisotropic X-Ray Peak Broadening and Twin Formation in Hematite Derived from Natural and Synthetic Goethite. *J. Eur. Ceram. Soc.* **2006**, *26*, 131–139. [[CrossRef](#)]

Disclaimer/Publisher’s Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.