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Tools of the Master Dyer

Dye Materials in Seventeenth and Eighteenth Century South Asian Painted Cotton Textiles at the Metropolitan Museum of Art

Sylvia Houghteling and Nobuko Shibayama

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Abstract | This article reports on an analysis of the dyes used in painted cotton textiles from seventeenth- and eighteenth-century India in the collections of the Metropolitan Museum of Art. The purpose of our study was to identify which red dyes were used to produce the textiles, and to determine whether dyers used cheaper substitutes for indigo as dyestuffs. Our findings preliminarily suggest that *chay* root (*Oldenlandia umbellata* L.) was not only a dye material used along the Coromandel Coast, but also traveled through overland trade networks for textile dyeing in central India. The results of this analysis contribute to understandings of regional specializations in dyestuffs. More broadly, this study may provide evidence for the mobility of dye materials and the agency that South Asian dyers had to choose their dye materials.

FIG. 1
Map of India showing different textile producing centers in the 17th and 18th centuries.

In the period before the development of synthetic dyes, South Asian dyers had perfected some of the most complicated dyeing processes and had become, in Mattiebelle Gittinger's words, "master dyers to the world."[1] Gittinger explored the uniqueness of India's blue dyes, forged from indigo, and the potency of its red dyes, which came from a variety of roots and insects. India's diverse ecology and cultural communities meant that dyers in different regions of the subcontinent specialized in distinctive dye materials, which often yielded textiles of differing hues. This regional specialization seems to have been a particular characteristic of the red dyes used to color cotton cloth, although there has been little scientific evidence to suggest that textile dyers in fact relied exclusively upon local materials, rather than using dyestuffs traded throughout South Asia. After analyzing the dye materials used on a small sample of textiles that have been attributed to different regions of India, this study has found surprising evidence for the pervasiveness of *chay* root dyes (*Oldenlandia umbellata* L.) across our textile samples. This finding may indicate that the use of this dye material, which is associated with specific regions in southeastern India, was more widespread than previously believed.

Historical texts from the seventeenth century praise the *chay* root dye, a colorant that was an essential part of the dyer's palette in the textile producing regions of the Coromandel Coast (fig. 1).[2] A seventeenth-century observer noted that the potency of *chay* root varied greatly depending upon where it had been grown, and also upon factors such as water quality and dye bath components: "*Chay*, a small thin root, a span or a span and a half long, is used for dyeing red, but the quality varies greatly; in one place it may be half as good again as in another…It does not fade, but the more it is washed, the better it becomes."[3] It is believed that the high concentration of decomposing seashells in the riverbeds of the Krishna (also

Notes

1 Gittinger 1992.
2 Cecil 2013, pp. 68–73.
3 Moreland, Methold, and Schorer 1931, p. 77.

known as the Kistna) River and the Kaveri River in the northern Coromandel Coast region increased the calcium content in the soil, improving the red hue of the *chay* root dyes. Gittinger also cites Dutch sources that suggest that *chay* root was cultivated in northern Sri Lanka and the Madurai region of South India, and *chay* root grew wild in the Puri region of Orissa and in Bengal, but was exported and not used as a dye material.[4] The southern Coromandel Coast (the site of production for many of the textiles for European export) was said to grow an inferior form of *chay* root, and dyers in the southern port of Madras (Chennai) imported *chay* root from the northern Coromandel Coast for dyeing.[5] Beyond the specialized *chay* root, the most common and widespread red dyes were madder (*Rubia tinctorum* L.) and its related species, *manjīstha* (*Rubia cordifolia* L.). Both dyes were used throughout India, although madder grew best in the temperate, mountainous regions of northern India.[6] The third well-known and widely-used red dye was *āl* (*Morinda citrifolia* L.), whose root bark produces a red color.

In terms of usage, researchers have assumed that dyers making textiles along the Coromandel Coast utilized *chay* root, while dyers situated in western India in Gujarat or in the central Indian city of Burhanpur deployed *manjīstha* or *āl* due to economics and availability. G.W. Taylor notes that besides the unique *chay* root dyes, there did not seem to be a clear pattern in the selection of madder-type dyes as opposed to *āl*, suggesting that selection was a matter of simple economic calculation. He writes: "both dyes were widely available, give similar shades of colour and presumably whichever was used was based on simple economics—a reasonable conclusion for these mass market textiles."[7] Yet as late as the 1850s, distinct regions were nonetheless known to specialize in the production of different dyestuffs. When South Asian dye materials were displayed at the 1851 Great Exhibition in London, the dye materials for *āl* came from present-day Madhya Pradesh in Central India, the *manjīstha* came from throughout India (Assam, Nepal, and Bombay), while the *chay* root came directly from the town of Masulipatnam, the central historical trading center of the northern Coromandel Coast.[8]

Historians have demonstrated that regional specializations in dyestuffs gave economic and political importance to the entire textile industries of various dye producing regions.[9] Ian Wendt has pointed out, however, that not only indigo, but also *chay* root dyes became "commercial commodities" that were "used in dyeing centers throughout South Asia."[10] It has been difficult to confirm from extant textiles how widely *chay* root actually circulated. Art historians often use stylistic analysis of decorative motifs to identify the region where a cloth was made, and then employ that visual information to narrow down the possibilities for dye materials to the dyestuffs characteristic of that region. A study of the textile dyes used in South Asian cotton textiles contributes to an understanding of the actual dyes used in the dyers' palettes, but can also potentially help in identifying where textiles with lesser-known stylistic patterns may have been made.

Finally, the literature has assumed the central role of indigo (*Indigofera tinctoria* L.) in the production of not only blue dyes, but as a combination color in green dyes as well. However, historical texts suggest that green dyes were produced not only from indigo, but also from ingredients such as dried pomegranate rinds, turmeric, henna, and the tannin produced from *halila*, or myrobalan fruit (*Terminalia chebula,* Retz.).[11] In her analysis of archival sources relating to dyes, Hamida Khatooon Naqvi has hypothesized that dyers substituted these local, more easily obtainable dyes for the more expensive indigo dyes in order to save on costs.[12] Our study has sought to identify whether all of the green and blue dyes on the samples derive from indigo, or whether they might have been made from lesser-known, local dyestuffs.

4 Gittinger 1982, p. 21; Cecil 2013, p. 69.
5 Irwin 1956, p. 33.
6 Taylor 1993, p. 23.
7 Taylor 1993, p. 24.
8 Bhardwaj and Jain 1982, p. 71.
9 Subrahmanyam 1990, p. 92.
10 Wendt 2005, p. 79.
11 Naqvi 1980, pp. 62–3.
12 Naqvi 1980, p. 160.

FIG. 2
Tent hanging, attributed to southeast India, c. 1640–1650. Cotton; mordant-dyed and resist-dyed, 231 × 193 cm (91 × 76 in), Victoria and Albert Museum IS.19-1989, © Victoria and Albert Museum, London.

Literature Review

Historical studies and investigations of current dyeing practices have provided a great deal of insight into the techniques used to produce Indian painted cotton textiles.[13] Technical studies that identify the dye materials responsible for India's brilliantly colored cloths can aid in deepening our knowledge of cloth production techniques.

A comprehensive dye analysis study from 1988 tested seventy samples from thirty-nine South Asian textiles held in the Calico Museum of Ahmedabad.[14] The study was informative for the range of dyes used in India. Although it did not specify the fibers tested, the textile objects listed as samples primarily included woven silks and wool carpets and shawls; there seem to have been only two cotton textiles tested. Important studies also exist for the dyes used in Mughal carpets.[15] Recent years have brought further, comparative studies of the dye materials used for silk cloth.[16]

Yet there are many more extant cotton textiles than there are silk textiles from the pre-1800 period. Ruth Barnes initiated dye analysis of two collections of medieval South Asian cotton textiles produced in Gujarat which had been found in Egypt. The study of the Ashmoleon collection provided unique insight into the variability of dyes used: of thirty samples (dating from the eleventh century through the seventeenth century), twenty were dyed with dyes in the madder family (*Rubia tinctorum/cordifolia* L.), nine with *āl* (*Morinda citrifolia* L.), and only one textile produced results consistent with *chay* root (*Oldenlandia umbellata* L.).[17] G.W. Taylor conducted a study that tested many of the Ashmoleon objects, as well as one seventeenth-century Coromandel Coast textile from the Victoria and Albert Museum. Of the forty-one textiles, only the Victoria and Albert Museum's textile produced results consistent with *chay* root (fig. 2).[18] A recent project at the Metropolitan Museum of Art that tested the dye materials in another sample group of medieval and early modern textiles imported from India to Egypt had findings that were consistent with Barnes's study.[19] Our project has sought to identify the dyes for a small, later group of textiles, most of which were likely made in the eighteenth century.

Samples for Study

Our study examined samples from six textiles from the collections of the Metropolitan Museum of Art. The textiles were all cotton cloths that had been painted and dyed, as opposed to block printed. In seventeenth- and eighteenth-century Europe, these textiles were known as "chintz." In contemporary India, the cloths are called *kalamkāri*, a word that refers to the pen (*kalam*) with which the mordants and resists are painted onto the cotton. *Kalamkāri* means "pen-work."[20]

Of the textiles under examination, three were likely produced for export to Europe (figs. 3, 4, and 5); in their loose arrangement of the floral motifs, and the large scale of the blooms, they resemble the exported "chintz" textiles sent to Europe in large numbers starting in the second half of the seventeenth century.

13 Mohanty, Chandramouli, and Naik 1987; Balfour-Paul 2006.
14 Kharbade and Agrawal 1988, pp. 1–8.
15 Böhmer 1997, pp. 155–9.
16 See Wouters 1998; Shibayama, Wypyski, and Gagliardi-Mangilli 2015.
17 Barnes 1997, p. 58. See also, Barnes 1993.
18 Taylor 1993, p. 24.
19 Rosenfield 2016.
20 Crill 2008, p. 8.

FIG. 3 (top)
Hanging (detail), attributed to India, late 17th century. Cotton; printed, 203 × 155 cm (80 × 61 in). The Metropolitan Museum of Art (28.78.3), gift of Harry Wearne, 1928.

FIG. 4 (left)
Fragment of a veil, attributed to India, 18th century. Cotton; pounced and painted, 38 × 55.80 cm (15 × 22 in). The Metropolitan Museum of Art (26.265.112), purchase, William Sloane Coffin Gift, 1926.

FIG. 5 (right)
Fragment of a veil, attributed to India, 18th century. Cotton; pounced and painted, 44.45 × 66 cm (17.5 × 26 in). The Metropolitan Museum of Art (26.265.114), purchase, William Sloane Coffin Gift, 1926.

FIG. 6
Sash, attributed to India, 17th century. Cotton; printed and painted, 254 × 33 cm (100 × 13 in). The Metropolitan Museum of Art (36.26), Rogers Fund, 1936.

The other three textiles that we studied have a more regularized arrangement of their motifs and were probably produced for consumption within India (figs. 6, 7, and 8). Judging from the style of the decorative motifs on the textiles, the density of the colors, and the history of textile exports from the Coromandel Coast, the three European-style textiles and the most saturated in color of the South Asian–style textiles can be associated with the Coromandel Coast region (figs. 3, 4, 5, and 7), while the other two (figs. 6 and 8) more likely derive from the northern Deccan city of Burhanpur or from central India more broadly. We therefore hypothesized that the four textiles from the Coromandel Coast would contain *chay* root as the red dye, while the other two would display the markers of *manjīstha* or *āl*. We also sought to determine whether any of the samples contained signs of non-indigo substitutes for the production of green and blue colors.

Methods of Analysis

Although it would have been ideal to conduct analysis using non-destructive techniques, it is currently not possible to analyze organic pigments accurately and comprehensively without taking samples. We therefore took small thread samples (of approximately 5 mm–1 cm) of each cloth to conduct this research. After the colorant was extracted from the thread sample, the analysis of these samples was conducted using high-performance liquid chromatography-photo diode array detection (HPLC-PDA). Using the technique of HPLC-PDA, color components in a dye, which have been extracted from a thread sample, are separated and each component is eluted in a specific time (a retention time) according to its chemical properties. Then, the ultraviolet-visible (UV-Vis.) spectra of each separated color component is measured, and the UV-Vis. spectra and the retention time of the compounds are compared with the UV-Vis. spectra and the retention time of standard color compounds, allowing identification of the separated color components in the dye. The result suggests a type of dye that contains the color components identified through HPLC-PDA. The details of this method and the summary of the results are provided in appendix A.

Results

Dyes used for each color were suggested based on the current color and detected colorants from the sample, although there may have been dyes which were not detected because of degradation. A result in which detected color components did not appear relevant was considered to have emerged from cross-contamination. For example, indigotin on a beige sample was not recognized as having resulted from an effort to dye the cloth blue; rather, such a result was regarded as cross-contamination. There were samples in which overlapping colored parts were difficult to separate, and as a result, it was not unequivocal to determine the dye for each color. The difference of colors on the samples on which the same colorants were detected may have also been due to different mordants.

FIG. 7
Floral decorated textile, India, Coromandel Coast, early 18th century. Cotton; mordant and resist dyed, 40.64 × 96.52 cm (16 × 38 in). The Metropolitan Museum of Art 2012 (487.1), gift of Mr. and Mrs. Yukikazu Iwasa, in memory of Seizaburo and Shizuko Iwasa, 2012.

Among the red, pink, and purple samples of the textiles, alizarin was found to be a major colorant. In *chay* root (*Oldenlandia umbellata* L.), the main colorant to the exclusion of other colorants is alizarin.[21] Alizarin also appears in madder (*Rubia tinctorum* L.) as one of the main colorants, along with other colorants such as purpurin, pseudopurpurin, or munjistin.[22] Sometimes purpurin was detected with alizarin in our samples; however, the amount of purpurin was minute. The ratios of alizarin to purpurin in our textile samples, determined through a comparison of their chromatographic peak areas, was 98-99/1-2, while the ratio of alizarin to purpurin from several reference samples dyed with madder was approximately 90-50/10-50, in addition to the presence of other colorants such as pseudopurpurin and munjistin (see table in appendix A).[23] The variation of the ratios seen in the madder samples may come from differences in dyeing methods, the processing method of the plant, or the location where the plant was cultivated. However, the differences in ratio of alizarin to purpurin between *chay* root and madder dyes are much more pronounced.

The main colorants in *manjīstha* (*Rubia cordifolia* L.) are purpurin, munjistin, and pseudopurpurin, and alizarin is not found in a significant amount.[24] Alizarin is not found in *āl* (*Morinda citrifolia* L.) as a major colorant.[25] The main colorants of *āl* are morindone and soranjidiol.[26] The overall absence in our samples of the component purpurin, found in madder and *manjīstha* makes it unlikely, though not impossible, that either of these other dyes were used to create the red color. This finding cannot fully exclude the possibility of madder because color components in a dye are subject to change due to ageing or the dyeing process.[27] With these possibilities in mind, the results strongly suggest that *chay* root was the colorant used for all of the red areas on the textiles.

FIG. 8
Fragment of a floorspread, attributed to India, late 17th century. Cotton; plain weave, mordant-painted and dyed, resist-dyed, 169.54 × 360.68 cm (66 ¾ × 142 in). The Metropolitan Museum of Art 1982 (239a), The Alice and Nasli Heeramaneck Collection, gift of Alice Heeramaneck, 1982.

21 Cardon 2007, p. 139.
22 Mouri and Laursen 2012, pp. 105–13; Cardon 2007, pp. 112–3.
23 These analyses were conducted on reference samples dyed with madder that were made by textile conservators in the Department of Textile Conservation at the Metropolitan Museum of Art over the last thirty years.
24 Mouri and Laursen 2012, p. 179, figs. 1–2 and pp. 109–10.
25 Taylor 1993, pp. 23–6, fig. 25.
26 Cardon 2007, p. 144.
27 Synthetic alizarin dye introduced in 1868 would also produce these findings, meaning that synthetic alizarin also cannot be ruled out entirely. The visual and material features of these textiles, as well as historical factors, suggest that all of the textiles under study were produced prior to the invention of synthetic alizarin. A recent study has suggested that two compounds may serve as markers for certain types of synthetic alizarin; those markers were not found in our analysis. See Liu, et. al. 2016, pp. 182–4.

Among the dark colored samples, ellagic acid was detected. Ellagic acid is produced from ellagitannins under hydrolytic conditions. Ellagitannins represent one of two subdivisions of hydrolysable tannins (the other is gallotannins).[28] Because ellagic acid was detected, a substance containing abundant hydrolysable tannins appears to have been used. Myrobalan (*Terminalia chebula,* Retz.), a plant rich with hydrolysable tannins and often used for South Asian painted and printed textiles, was likely used on our textile samples.[29]

Indigotin was detected in the blue and green-colored thread samples, indicating that an indigotin-containing dye such as indigo (*Indigofera tinctoria* L.), was used. Indirubin, also found in indigo dye along with indigotin, was also detected on some samples.

Thread samples from figure 6 appeared to show a smaller amount of ellagic acid in contrast to alizarin among the six textiles indicating that much less tannin was used on the textile. The red sample of figure 8 also suggested that a more limited amount of tannins was used in the preparation of that textile. One textile (fig. 3) showed a slight difference in the components of tannins as compared to the other textiles.

Interpretation of Results and Preliminary Conclusions

Our analysis confirmed the use of indigo for both blue and green colors, which is not unusual due to the ubiquity and known superiority of indigo dyes for the coloring of cotton cloth. We did not find evidence for unusual dye combinations in the formation of green dyes, nor did we find cheaper substitutes for blue dyes. This suggests that for both export and domestic cotton cloths of this high level, indigo was used consistently.

The omnipresence of ellagic acid from the tannins in myrobalan fruits (*Terminalia chebula*, Retz.) indicates that this substance, derived from a dried local tree fruit, was widely used in seventeenth- and eighteenth-century India. In our samples, myrobalan seems to have been used for a range of colors, from light beige to black. Eighteenth-century dye recipes also list myrobalan (called *halila*) as a component added to achieve purple and deep red dyes.[30] In contemporary practices, the cotton fabric used for *kalamkāri* is also soaked in a mixture of ground myrobalan fruit and buffalo milk before it is painted; after it is dried, the tannins in the myrobalan fruit work as a ground for the application of an iron acetate mordant that reacts with the tannins to create the black outlines seen on *kalamkāri* textiles. According to Mohanty, Chandramouli, and Naik, the buffalo milk in the myrobalan mixture helps to "brighten the colour when printed and prevents its spreading to the other side."[31] It seems possible, then, that the evidence of tannins from myrobalan fruit may have occurred both because of the fruit's use as a dye colorant and because of its role in the preparatory stages of painting.

We found that there were smaller amounts of ellagic acid in the samples whose style suggests central Indian origin (figs. 6 and 8) than in those from the Coromandel Coast, perhaps suggesting different approaches to the use of myrobalan fruit in regional dyeing techniques. This could either indicate alternative ways that dyers prepared their cloths, or more limited use of myrobalan as an addition to the dyebath in central India. The distinction in the amounts of ellagic acid provides evidence for material differences between the textiles whose style indicates production in central India and those produced on the Coromandel Coast, even though their primary dyes appear to be the same.

The most surprising finding of our analysis is the prevalence of *chay* root red dyes, in contrast to the relative absence of *manjīstha* or *āl* among the red dyes used to color the cloths. This is unexpected because the use of *chay* root is typically thought to have been localized and largely confined to the eastern Coromandel Coast of India. The red dyes found on two of the samples whose style most clearly suggests their production in central India, perhaps

28 Halslam 1966.
29 Mohanty, Chandramouli, and Naik 1987, p. 5.
30 Naqvi 1980, p. 61.
31 Mohanty, Chandramouli, and Naik 1987, p. 103.

FIG. 9
Bed cover (palampore), attributed to India, Coromandel Coast, 18th century. Cotton; plain weave, mordant painted and dyed, resist-dyed; 271.8 × 197.5 cm (107 × 77 ¾ in). The Metropolitan Museum of Art (1982.66), purchase, bequest of George Blumenthal and gift of Indjoudjian Freres, by exchange, and the Friends of the Islamic Department Fund, 1982.

Burhanpur (figs. 6 and 8) also seem to have been produced using *chay* root dye, as the thread samples showed strong results only for alizarin, the colorant in *chay* root, and not for purpurin, which is found in madder-type dyes (the ratio for the red and purple threads was 99/1).

Visually, the textiles made in central India are characterized by a more symmetrical and regular arrangement of the floral motifs. They almost resemble textiles woven on a loom in the geometric layout of their patterns. This style is more similar to the floral ornament on carpets and textiles made for the Mughal court.[32] It can be contrasted to the more irregular dispersal of patterns in textiles made along the Coromandel Coast that can be seen on textiles such as figure 9, although the dyes on this textile were not analyzed for this study. The Mughal imperial style was more prevalent in art objects made in Burhanpur and other regions of central India because of the presence of the Mughal emperors and their noblemen in this region in the seventeenth century. Furthermore, as scholars have recently noted, architectural and wall decorations from Burhanpur bear a striking similarity to the symmetrical, poppy-patterned textile that we analyzed (fig. 8), further supporting the Burhanpur attribution of this textile.[33] The appearance of *chay* root dyes on textiles that have been stylistically attributed to the Burhanpur region therefore suggests that the use of *chay* root may not have been limited to the Coromandel Coast, as studies of earlier textiles have suggested, but, by the seventeenth century, *chay* may have traveled through overland trade networks for use by dyers in central India.

The best quality *chay* root was a highly localized product due to the unique ecology of the northern Coromandel Coast described above. Ian Wendt notes that the finest quality dyestuffs were harvested in the wild and grew in sandy soil; *chay* root that was commercially cultivated was of lower quality.[34] Within the British East India Company records, evidence of the trade along the Coromandel Coast in high-quality *chay* root appears in letters written from Madras (Chennai). Dyers in this southern Coromandel Coast city imported the far superior *chay* root dyes from their northern neighbors. The British East India Company officials wrote in 1700 of trying to prevent dyers "from mixing the Southern *Chay* with the Northern, the latter being the best and costs much more."[35] Beyond accounts of importing *chay* root in the regions surrounding Madras (Chennai), there has not been substantial archival evidence that *chay* root also traveled as a commodity to inland sites such as Burhanpur in central India, or to western India.[36] The findings of *chay* root on the textiles associated with Burhanpur in our study, therefore, serve as inspiration to investigate the lesser-known aspects of the history of the *chay* root dye trade.

Finally, the use of *chay* root dyes on the textiles intended for European export provides yet another reason that Europeans were determined to import painted cotton textiles from India on such a grand scale in the seventeenth and eighteenth centuries. Past studies have emphasized the novelty of lightweight and easily washed cotton as one major inspiration; others have demonstrated that European dyers would not learn how to bind red dyes to cotton until Armenian dyers first introduced the techniques of "Turkey red" dyeing in Marseilles in the eighteenth century.[37] The relative ubiquity of madder dyes in Europe has been taken to suggest that European dyers were only missing the proper techniques in order to be able to fully replicate the vibrant colors of South Asian cloth. However, dyers were also missing a crucial dye material. While European colonies in the Americas and West Indies were, by the eighteenth century, producing large quantities of Indian varietals of indigo, *chay* root has only been found very rarely on European-made textiles.[38] Although the French imported quantities of *chay* root dyes in the eighteenth century, and the English experimented with the dye material, *chay* root was never cultivated on a large scale in Europe.[39] It was also never imported from India in great amounts because, as George Watt warned, "chay-root rapidly deteriorates when kept in a dark place, such as the hold of a ship."[40] By the twentieth century, dyeing with *chay* root declined in South Asia, and the plant-based dye had been replaced by synthetic alizarin. These textiles, therefore, are a testament not only to a unique moment in the manufacturing history of South Asia, but also to a moment in botanical history that was never replicated again.

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32 See Walker 1997.
33 Haidar and Sardar 2015, p. 26 and pp. 285–6.
34 Wendt 2005, pp. 79-80.
35 Quoted in Irwin 1956, p. 33.
36 Wendt mentions that *chay* root was traded commercially, but on what scale and to where is unclear. Wendt 2005, p. 78.
37 Styles 2007, p. 114; Raveux 2011, pp. 291–307; Riello 2010, pp. 1–28.
38 Phipps 2013, pp. 128 and 134.
39 Taylor 1990, pp. 32–3; Nesbitt 2013, pp. 100–5.
40 Quoted in Cecil 2013, pp. 68–73.

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Appendix A

Methods:
Small cotton yarn samples were taken from the textile, and each sample (a few threads of approx. 5 mm–1 cm) was extracted using 40μl of a mixture of 0.01 M aqueous oxalic acid, pyridine, and methanol (3/3/4, v/v/v) in a small test tube.[41] The thread sample was left for half an hour at room temperature (RT), subsequently heated at 55–60°C for 20 minutes. The extract was then removed to an insert and 80 μl of the new mixture mentioned above was added to the test tube and heated at 90–100°C for 10 minutes; this extract was then moved to the same insert. The tube was rinsed with 20 μl of methanol twice, and the rinsing solution was also added to the insert. The extract in the insert was dried in a vacuum desiccator using an aspirator. The residue was mixed with 8 μl of methanol and 8 μl of 1 percent aqueous formic acid (v/v). The solution was centrifuged for 10 minutes at 3500 g; the supernatant was injected into the HPLC system.

The analytical system used consisted of a 1525μ binary HPLC pump, 2996 PDA detector, 1500 series column heater, in-line degasser and a Rheodyne 7725i manual injector with 20 μl loop (Waters Corporation, Milford MA), an XBridge BEH Shield RP18 (3.5 μm-particle, 2.1 mm I.D. x 150.0 mm, Waters Corporation, Milford MA) reverse-phase column was used with a guard column (Xterra RP18 3.5 μm-particle, 2.0 mm I.D. x 10.0 mm, Waters Corporation, Milford MA) with a flow rate of 0.2 ml/min. The column pre-filter (Upchurch ultra-low Volume pre-column filter with 0.5 μm stainless steel frit, Sigma-Aldrich, St. Louis MO) was attached in front of the guard column. Column temperature was 40°C. The mobile phase was eluted in a gradient mode of 1 percent formic acid in high purity water (v/v) (A) and a mixture of methanol and acetonitrile (1/1, v/v) (B). The gradient system was 90 percent (A) for 3 minutes → to 60 percent (A) in 7 minutes in a linear slope → to 0 percent (A) in 24 minutes in a linear slope, and then to 90 percent (A) in 1 minute and held at 90 percent (A) for 10 minutes. A summary of the result appears in the table below.

41 Mouri and Laursen 2011, pp. 7325–30.

Summary of the Result

Accession number		Main color or overlapped different colors of a sample	Main dye compounds which were detected	Alizarin Purpurin	Ellagic acid/ Alizarin	Suggested dyes
28.78.3 Figure 3	1	Pink—brown	alizarin, ellagic acid	98/2	84/16	Brown : tannin dye, *chay* root dye (*Oldenlandia umbellata* L.)
	2	Blue 1	indigotin, alizarin, ellagic acid	NP	67/33	Red, pink : *chay* root dye (*Oldenlandia umbellata* L.)
	3	Blue—black (blue 2)	indigotin, alizarin, ellagic acid	NP	34/63	Blue : indigotin-containing dye
	4	Blue—pink	alizarin, ellagic acid	99/1	64/36	Black : tannin dye
	5	Red part of a pink—red sample	alizarin, ellagic acid	NP	72/28	
	6	Pink part of a pink—red sample	alizarin, ellagic acid	NP	35/65	
	7	Brown part of a pink-brown sample	alizarin, ellagic acid	NP	39/61	
	8	Pink part of a pink- brown sample	alizarin, ellagic acid	NP	37/63	
26.265.112 Figure 4	1	Black	alizarin, ellagic acid, indigotin	98/2	64/36	Black : tannin dye
	2	Brown	alizarin, ellagic acid	NP	76/24	Brown : tannin dye
	3	Purple	alizarin, ellagic acid	99/1	31/69	Purple : *chay* root dye (*Oldenlandia umbellata* L.)
	4	Red	alizarin, ellagic acid	98/2	48/52	Red: *chay* root dye (*Oldenlandia umbellata* L.)
	5	Purple part of a blue-black sample	alizarin, ellagic acid, indigotin, indirubin	99/1	63/37	Blue : indigotin-containing dye
26.265. 114-113 Figure 5	1	Blue – black	alizarin, ellagic acid, indigotin, indirubin	98/2	69/31	Blue : indigotin-containing dye
	2	Purple – black	alizarin, ellagic acid	97/3	44/56	Purple : *chay* root dye (*Oldenlandia umbellata* L.)
	3	Beige	ellagic acid, indigotin, alizarin	NP	25/75	Red: *chay* root dye (*Oldenlandia umbellata* L.)
	4	Red	alizarin, ellagic acid	98/2	64/36	Beige: tannin dye Black: tannin dye

Note Alizarin/Purpurin: the ratio of the peak areas of alizarin and purpurin in the chromatogram at 450 nm, Ellagic acid/Alizarin: the ratio of the peak areas of ellagic acid and alizarin in the chromatogram at 370 nm, NP: purpurin was not detectable in the chromatogram at 450 nm.

Accession number		Main color or overlapped different colors of a sample	Main dye compounds which were detected	Alizarin Purpurin	Ellagic acid/ Alizarin	Suggested dyes
36.26 Figure 6	1	Purple	alizarin, indigotin, ellagic acid	99/1	5/95	Purple : *chay* root dye *(Oldenlandia umbellata* L.) + indigotin-containing dye
	2	Pink – the sample was not taken				
	3	Red	alizarin, ellagic acid	NP	7/93	Red : *chay* root dye (*Oldenlandia umbellata* L.)
	4	Yellow (purple and red stains were on the sample)	alizarin, ellagic acid	NP	17/83	Yellow : tannin dye (?)
	5	Green	alizarin, indigotin, indirubin, ellagic acid	NP	17/83	Green : indigotin-containing dye, yellow dye (?) or *chay* root dye (*Oldenlandia umbellata* L.) (?)
	6	Blue	alizarin, ellagic acid, indigotin	NP	20/80	Blue : indigotin-containing dye
2012.487.1 Figure 7	1	Yellow	alizarin, ellagic acid	NP	75/25	Yellow : *chay* root dye (*Oldenlandia umbellata* L.)
	2	Purple – the sample was not taken Red – the sample was not taken				
	3	Blue	indigotin, indirubin, ellagic acid	NP	76/24	Blue : indigotin-containing dye
	4	Green	alizarin, ellagic acid, indigotin	NP	79/31	Green : indigotin containing dye, yellow dye (?) or *chay* root dye (*Oldenlandia umbellata* L.) (?)
	5	Black (deteriorated)	alizarin, ellagic acid, indigotin	99/1	71/29	Black : tannin dye
	6	Red	alizarin, ellagic acid, indigotin	99/1	39/61	Red : *chay* root dye (*Oldenlandia umbellata* L.)
1982.239a Figure 8	1	Medium green (dyed part)	ellagic acid, alizarin, indigotin	NP	100/0	Green (dyed) : tannin dye + indigotin—containing dye
	2	Brown (dyed part)	ellagic acid, alizarin, indigotin	NP	93/7	Brown (dyed) : tannin dye
	3	Red (painted part)	alizarin, ellagic acid	99/1	2/98	Red : *chay* root dye (*Oldenlandia umbellata* L.)
	4	Blue sample on the medium green and the brown outline	alizarin, indigotin, indirubin, ellagic acid	NP	91/9	Blue : indigotin-containing dye
	5	Dark red (the sample was not taken)				

Note Alizarin/Purpurin: the ratio of the peak areas of alizarin and purpurin in the chromatogram at 450 nm, Ellagic acid/Alizarin: the ratio of the peak areas of ellagic acid and alizarin in the chromatogram at 370 nm, NP: purpurin was not detectable in the chromatogram at 450 nm.

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