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# The Heart of the Madder: An Important Prehistoric Pigment and Its Botanical and Cultural Roots

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THE HEART OF THE MADDER:  
AN IMPORTANT PREHISTORIC PIGMENT AND ITS BOTANICAL AND CULTURAL  
ROOTS

by  
Michelle L. LaBerge

A Thesis Submitted in  
Partial Fulfillment of the  
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in Anthropology

at  
The University of Wisconsin-Milwaukee

May 2018

## ABSTRACT

### THE HEART OF THE MADDER: AN IMPORTANT PREHISTORIC PIGMENT AND ITS BOTANICAL AND CULTURAL ROOTS

by

Michelle L. LaBerge

The University of Wisconsin-Milwaukee  
Under the Supervision of Professor Bettina Arnold, PhD

In recent years, an interest in natural botanical dye sources has prompted new research into the cultivation and processing of prehistoric dye plants. Advances in chemical analyses of ancient European textiles have provided more information about dye plants such as woad (*Isatis tinctoria*) weld (*Reseda luteola*) and madder (*Rubia tinctorum*), which were important sources of color in early textile production. Evidence of madder dye has been reported in the archaeological record of the European Bronze and Iron Ages in textiles preserved in the Hallstatt salt mines, Scandinavian bog sites and other elite European burials but the picture of madder usage from the Late Bronze Age into the medieval era is still unclear. The use of other indigenous plants related to madder also complicates this picture. This thesis critically reviews the history of research on madder and the evidence for its use in archaeological contexts in Europe. The experimental component of the thesis involved 1) growing madder root plants and analyzing them for growth rate, hardiness, and fecundity and 2) using madder root with and without mordants, at various temperatures and pH levels on a range of archaeologically attested textiles to determine the spectrum of red hues that could have been obtained in prehistory. This research contributes to the literature on growing and dying with madder root, its use in prehistoric textile and dye production and the significance of color in prehistory.

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To my children,

Gwen L. Maurer and Jacob A. Maurer,

who have grown up watching me become an archaeologist. You are both a big reason why this journey was so meaningful.

## TABLE OF CONTENTS

<b>List of Figures</b> .....	vii
<b>List of Tables</b> .....	x
<b>Acknowledgements</b> .....	xi
<b>Chapter 1. Introduction</b> .....	1
A. The Long History of the Color Red.....	1
B. A Brief History of Dyer’s Madder, or <i>Rubia tinctorum</i> .....	2
C. Basic Botanical Aspects of <i>R. tinctorum</i> .....	3
D. Dyer’s Madder in Prehistoric Technological Context.....	6
E. Related Research.....	8
F. Current Interest in and Importance of Dyer’s Madder.....	9
G. Research Questions.....	10
<b>Chapter 2. Literature Review</b> .....	12
Part 1: The Botanical Aspects of Madder.....	12
A. Botanical Characteristics of <i>R. tinctorum</i> .....	12
B. Distribution of <i>R. tinctorum</i> .....	15
Part 2: Chemistry, Alchemy, and Madder.....	18
A. Organic Dye Fundamentals.....	18
B. Dyes and Alchemy .....	20
C. Dyes and Their Chemistries.....	21
D. Other Chemical Components of <i>R. tinctorum</i> .....	25
E. Methods of Dye Analysis in Archaeological Textiles.....	26
F. Textile Conservation and Ancient Dyes.....	28
Part 3: Archaeological Evidence for Madder.....	29
A. An Introduction to Ancient Dyes and Archeological Textiles.....	29
B. Earliest Textiles, Pigments, Organic Dyes, and Ancient Evidence for Red.....	34
C. The Archaeological Evidence for Madder.....	35
D. The European Neolithic (before 2000 BC) .....	37
E. The Bronze Age (2000 BC to 700 BC) .....	38
F. The Early Iron Age (700 BC to 450 BC) .....	40
G. La Tène Period (450-400 BC to the Roman Conquest).....	42
H. The Roman Period (Roman Conquest to AD 410) .....	44
I. Historical Written Evidence for Madder.....	50
J. Evidence for Dyeing as a Profession.....	51
K. Madder Legends and Lore.....	52

L. Modernity and Madder: The Current State of Madder Usage.....	54
<b>Chapter 3. Methods</b> .....	56
Research Questions.....	56
Phase 1: Growing Madder.....	62
A. Cultivation.....	62
B. Harvest.....	68
Phase 2: Dye Experiment Structure.....	73
A. Textiles.....	74
B. Equipment.....	79
C. Color.....	80
D. Madder Sources.....	84
Phase 3: Dye Bath Processes.....	86
A. Sources of Dye Recipes.....	86
B. Mordants.....	87
C. Other Dye Experiment Variables.....	92
<b>Chapter 4 Results</b> .....	96
Phase 1: Madder Cultivation: Results.....	96
A. Cultivation: Results.....	96
B. Harvesting: Results.....	96
Phase 2-3: Dye Experiments: Results.....	99
A. Basic Alum Dyebaths: Results.....	99
B. Dye Bath Temperature Variations: Results.....	101
C. Dye Bath pH Variations: Results.....	106
D. Dye Bath Timed Experiments: Results.....	112
E. Mordant Experiments: Results.....	118
F. Wisconsin Madder Dye Baths: Results.....	122
Overall Summary.....	124
<b>Chapter 5 Summary and Future Research</b> .....	126
<b>References Cited</b> .....	132
<b>Appendix A: Online Sources</b> .....	145
<b>Appendix B: Dye Bath Results Images</b> .....	146
<b>Appendix C: Dye Bath Results Tables</b> .....	178

## LIST OF FIGURES

All images are by the author unless otherwise stated

Fig. 1.1a-d.	<i>R. tinctorum</i> botanical anatomy.....	4
Fig. 1.2a-b.	Kermes and Cochineal.....	6
Fig. 2.1.	A simplified plot of <i>R. tinctorum</i> 's botanical lineage.....	12
Fig. 2.2a-b.	<i>R. tinctorum</i> pollen.....	13
Fig. 2.3.	<i>R. tinctorum</i> , botanical drawing.....	13
Fig. 2.4a-c.	Equatorial views of <i>Rubieae</i> tribe pollens.....	15
Fig. 2.5.	Worldwide distribution of dye plants in the <i>Rubiaceae</i> family.....	15
Fig. 2.6a-d.	Madder and madder-type plants.....	17
Fig. 2.7a.	Anthraquinone group chemical structure.....	24
Fig. 2.7b.	Alizarin chemical structure.....	24
Fig. 2.7c.	Purpurin chemical structure.....	24
Fig. 2.7d.	Pseudopurpurin chemical structure.....	24
Fig. 2.8a.	Kermesic acid chemical structure.....	24
Fig. 2.8b.	Carminic acid chemical structure.....	24
Fig. 2.8c.	Laccaic acid chemical structure.....	24
Fig. 2.9a.	Boletol chemical structure.....	25
Fig. 2.9b.	Dermorubin chemical structure.....	25
Fig. 2.10.	Hallstatt textiles showing the variety of color and patterns.....	40
Fig. 2.11a-b.	Evebø/Eide textiles.....	50
Fig. 3.1.	USDA Plant Hardiness Zones for Wisconsin .....	63
Fig. 3.2.	Global Plant Hardiness Zones .....	64
Fig. 3.3.	Plant Hardiness Zones for Europe.....	64
Fig. 3.4a-b.	Overwintered madder.....	67

Fig. 3.5a-d.	Wisconsin madder plants.....	68
Fig. 3.6a-b.	Harvesting madder.....	70
Fig. 3.7a-b.	Harvested madder.....	70
Fig. 3.8a-b.	Comparison of 1-year and 2-year plants .....	70
Fig.3.9a-b.	Madder root close-ups.....	71
Fig.3.10a-b.	Washing madder roots.....	72
Fig.3.11a-b.	Drying madder.....	72
Fig. 3.12.	Mordants.....	74
Fig. 3.13a-d.	Fibers included in experiments.....	76
Fig.3.14.	The range of reds.....	83
Fig. 3.15a-b.	Soaking Pakistani madder.....	85
Fig. 3.16a-b.	Soaking the dried homegrown madder.....	85
Fig. 3.17a-d.	Equipment used in experimental component of this project.....	86
Fig. 3.18a-b.	Heating the ground oak galls.....	89
Fig. 3.19a-b.	Club moss mordant preparation.....	90
Fig. 3.20a-b.	Development of the iron liquor over several weeks.....	91
Fig. 3.21a-d.	Soapwort mordant.....	92
Fig. 3.22a-b.	Wood ash water examples.....	94
Fig. 4.1.	Dried madders.....	97
Fig. 4.2a-d.	Magnified images of madder plant.....	98
Fig. 4.3a-b.	Untreated examples of the experiment fiber types.....	99
Fig. 4.4	Madder-dyed fiber types with and without an alum mordant.....	100
Fig. 4.5a-b.	Wool fibers at differing temperatures.....	102
Fig. 4.6a-b.	Silk fibers at differing temperatures .....	103
Fig. 4.7a-b.	Linen fibers at differing temperatures.....	103

Fig. 4.8a-b.	Nettle fibers at differing temperatures.....	103
Fig. 4.9a-b.	Wool fibers in dye baths of different pH.....	108
Fig. 4.10.	Silk fibers in dye baths of different pH.....	109
Fig. 4.11.	Linen fibers in dye baths of different pH.....	110
Fig. 4.12.	Nettle fibers in dye baths of different pH.....	111
Fig. 4.13.	All fiber types in a 0.5-hour dye bath.....	112
Fig. 4.14.	All fiber types in a 12-hour dye bath.....	113
Fig. 4.15.	All fiber types in a 24-hour dye bath.....	113
Fig. 4.16.	Mordant baths.....	119
Fig. 4.17.	Prehistoric mordants on all fiber types.....	119
Fig. 4.18.	Wisconsin madder on all fiber types.....	122

## LIST OF TABLES

Table 1.1.	Classical and historical references to madder.....	3
Table 2.1.	Red dyes in prehistory.....	22
Table 3.1.	Monthly weather averages for Oshkosh, Wisconsin.....	65
Table 4.1.	Munsell colors: different fibers used in dye baths.....	99
Table 4.2.	Fiber samples with and without an alum mordant.....	101
Table 4.3.	Temperature experiments with madder dye baths.....	104-105
Table 4.4.	Dye bath pH experiments on wool.....	108
Table 4.5.	Dye bath pH experiments on silk.....	109
Table 4.6.	Dye bath pH experiments on linen.....	110
Table 4.7.	Dye bath pH experiments on nettle.....	111
Table 4.8.	Wool fibers in dye baths of varying duration.....	114
Table 4.9.	Silk fibers in dye baths of varying duration.....	115
Table 4.10.	Linen fibers in dye baths of varying duration.....	116
Table 4.11.	Nettle fibers in dye baths of varying duration.....	117
Table 4.12.	Color and pH of dye mordants.....	119
Table 4.13.	Wool fibers with different mordants.....	120
Table 4.14.	Silk fibers with different mordants.....	120
Table 4.15.	Linen fibers with different mordants.....	121
Table 4.16.	Nettle fibers with different mordants.....	121
Table 4.17.	Comparison of Pakistani and Wisconsin madder.....	123-124

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## **CHAPTER 1: INTRODUCTION**

### ***A. The Long History of the Color Red***

Color has been used on human bodies and material objects for millennia. There is evidence for the use of the iron oxide red ocher in European Neanderthal contexts from 200-250 kya, roughly the same time ocher usage occurs in the African record as well (Roebroeks et al. 2012: 1889). The color red has a long history of significance that is universal to humans; it is perhaps the most symbolic of all the colors (Cardon 2007: 107). It is thought that red is the first primary color that newborn humans can see and human color vision is particularly adept at seeing the color red; recent studies suggest this may be a factor in social signaling, including detecting the flush of anger or embarrassment in someone's face (Hiramatsu et al. 2017: 2). There is also research that suggests it may have been an adaptive response in primates as an enhanced ability to find ripe fruit (Jacobs 2010: 429). The color red strikes at something fundamental in being human, both in the past and in the present. This holds true for the prehistoric past in Europe as well. Perhaps red signified birth or death, perhaps it signified high status or a warning. Perhaps it was symbolic of the onset of menses, or marriage, or warlike aggression. Because modernity was not left with any texts to decode the usage of certain colors by the non-literate cultures of prehistoric Europe, the significance of red is open to interpretation. We do know, however, that it was a color of importance and in high demand as a pigment.

It is likely that red has always been a special color for humans, and the desire to apply this color to ourselves and our material goods reaches far back into prehistory. Some sources for red, like red ocher, are mineral and have great staying power in the archaeological record. Other sources, such as those from animals and plants, are not so long-lived, and researching their past is problematic. One of the most important sources for the color red in the Old World, both

prehistoric and historic, has been the madder plant, *Rubia tinctorum*. Madder red was likely not the most elite source of red textile dye in the prehistoric Europe and the Near East--that was a status reserved for the most luxurious red dye pigment indigenous to Europe, the kermes scale insect. By the early Middle Ages in Europe, however, dyer's madder was by far the most commercially important vegetal source for the color red (Cardon 2007: 107-108). Evidence of its usage in archaeological contexts from the Neolithic through the Roman Era in Europe allows us to trace the development of this important dye crop.

### ***B. A Brief History of Dyer's Madder, or Rubia tinctorum***

*Rubia tinctorum*, the source of madder red, is a well-known dye plant from ancient times that is encountered frequently in archaeological analyses of prehistoric and historic textiles as well as classical texts and alchemical writings. Madder-dyed textiles and pigments have been identified in Tutankhamen's tomb (Crowfoot and Davies 1941:123), on the Shroud of Turin (van der Hoeven 2015: 706), in the Oseberg ship burial (Ingstad 1981: 89) and the paintings of Vermeer (Jansen 2017). Madder dyeing is mentioned in the writings of Pliny (especially his *Naturalis historia*), Dioscurides, and Heraclius (Faber 1938: 287, 294, 310), amongst others (Table 1.1). The philosopher Democritus or pseudo-Democritus is said to have written four texts on dyeing methods, though these are all now lost (Faber 1938: 289). Madder has been a staple dye plant in Europe and Asia for millennia, yet the cultivation, processing, exchange and use of this important color all have a nebulous past. The madder root species known in the Near East and Europe, *R. tinctorum*, most likely originated in the Middle East or Eurasian steppe (Cardon 2007: 108), and was cultivated in Egypt as far back as 1400 BC (Eastwood 1984: 10). The earliest known madder used in human adornment was a dyed cotton thread found in a bead at Mohenjo-daro in the Indus Valley and dates to 3250-2750 BC (Marshall 1931:32-33), although

this was likely a different species than *R. tinctorum*.

**TABLE 1.1. CLASSICAL AND HISTORICAL REFERENCES TO MADDER**

Date	Source/Title	Location	Details of Usage
Ca. 450 BC	Herodotus 4.189	Greek, visiting Egypt	Describes Libyan women in garb dyed with madder
Ca. 400 BC	Hippocrates "On Regimen in Acute Diseases" Appendix No. 31	Greece	Cites a remedy for dysentery involving a mixture of cleaned beans, madder shoots and fat.
Ca. 350-287 BC	Theophrastus <i>Enquiry into Plants (Historia Plantarum)</i> IX, XIII 4,8	Athens	Describes madder's growing habits and root color, and its use for "pains in the loin or hip disease".
Ca. 350 BC	Democritus or pseudo-Democritus	Ptolemaic Egypt	First to collate dye practices. His four-volume work is now lost.
Ca. 7 BC	Strabo <i>Geographica</i> 13.4.14	Amaseia (modern Turkey), part of the Roman Empire	Account of superior color in madder dyeing in Hierapolis, due to the water quality from the hot springs.
50-70 AD	Pedanius Dioscorides <i>De Materia Medica</i> iii 160	Anazarbus, Cilicia (modern Turkey)	Account of madder cultivation in Cyrene and Caria, refers to madder as <i>erythrodanon</i> (ἐρυθρόδανον).
77-79 AD	Pliny the Elder "Historia Naturalis" Book XIX 17	Rome	Refers to madder as <i>Rubia</i> and correctly describes dyeing methods for the root. Describes the excellent quality of madder grown near Rome.
Ca. 200 AD	The Mishna Shev 5:4	Palestine	Collection of Talmudic Laws that allows the cultivation of madder for domestic, but not commercial, use.
Ca. 300 AD	Papyrus Graecus Holmiensis (or the Stockholm Papyrus)	Discovered in Thebes, Egypt	Over 150 dye recipes for textiles and gemstones, including several methods using madder root
Ca. 622	Forged Charter of Dagobert	France	Allowed subjects to sell madder root at the Landit Fair of St. Denis.
Ca. 771-800	Charlemagne "Capitulare de villis"	Carolingian Empire	Madder ( <i>warentia</i> ) listed as a supply necessary to "women's workshops" (43) and recommended to be grown in subjects' gardens (70)

### C. Basic Botanical Aspects of *R. tinctorum*

*R. tinctorum* is an herbaceous low-growing plant with lanceolate leaves and a trailing habit. Mature plants produce pale yellow star-shaped blooms and round black seeds that resemble peppercorns (Figure 1.1a-d). The stems of the plant are not sturdy enough to hold it upright, and are covered with small spines; the leaves of *R. tinctorum* are also edged with these small spines (Figure 1.1d). Madder roots are large, branching and tubular and are bright red in color. Only the root of the plant is used in dyeing. Fifteen dye compounds occur naturally in

mature madder roots (Cardon 2007: 109), but the primary dye compounds are alizarin, purpurin, and pseudopurpurin. Of these three compounds, alizarin is considered the major textile dye compound, which, while not an exceptionally light-fast dye (MacEvoy 2016), is considered far more long-lasting than purpurin or pseudopurpurin, which are more fugitive dyes.



Fig. 1.1a-d. *R. tinctorum* botanical anatomy. 1.1a. Upper left: *R. tinctorum* flowers, 1.1b. Upper right: *R. tinctorum* seed, 1.1c. Lower left: *R. tinctorum* root, 1.1d. Lower right: *R. tinctorum* stem and leaf.

*Rubiaceae* species are found globally, on every continent save Antarctica, although not all have been used as dye plants in the prehistoric past. Prehistoric use of madder dye has been documented in North Africa, southwest Asia and Europe, China, and South America (Cardon 2007: 107). Within Europe, several closely related dye plants were utilized in the prehistoric past, including members of the *Galium* and *Asperula* genera, as well as a wild version of madder, *Rubia peregrina*. It has been suggested that not all of these plant species contain the same levels of dye compounds or produce the same quality dye, although it is also very likely that dying

methods varied greatly between cultures. Even in Europe and the Near East, very different dye methods and mordants were developed to achieve reds, oranges, and purples with madder.

Madder red was one of several prehistoric dyes important to Europe and the Near East, probably introduced to the Greek world by at least the late Neolithic or early Bronze Age. Many other dye sources were available, some mineral, some plant-based, and some derived from insects or fungi. Other historically and prehistorically important sources of red were kermes (Old World) and cochineal (New World), both derived from insects. Kermes was a high prestige and expensive crimson red obtained from the dried bodies of the female scale insects *Allokermes kingii* (Fig. 1.2a) that live on scarlet oak trees in areas around the Mediterranean in Spain, North Africa and Eastern Europe (Cardon 2007: 607). Kermes produced a brilliant bright red (the word *kermes* is the Arabic word for crimson) that was highly prized and very much a prestige dye (Cardon 2007: 608). Pliny states that the Romans often took tribute in kermes dye, and kermesic acid has been identified on textiles from the 6<sup>th</sup> c. BCE Hochdorf grave in southwest Germany (Walton Rogers 1999: 241-43). Cochineal is primarily a New World dye, introduced by the Spanish in the 16<sup>th</sup> century after Cortes encountered its usage among the Aztecs. Polish or Armenian varieties of cochineal did exist in Europe in prehistory (Taylor 1990: 37), but these do not seem to have had the impact on the dyeing world that the Spanish introduction had in historic times. Cochineal is also derived from a scale insect, *Dactylopius coccus* (Fig. 1.2b), that was used expertly as a red dye by Mesoamerican and South American cultures, although their early dye methods involving cochineal are not known (Cardon 2007: 624).



Fig. 1.2a-b. Kermes and cochineal. Fig. 1.2a. Left: *Allokermes kingii*, the source for kermes dye. (Image by Paul Starosta). Fig. 1.2b Right: *Dactylopius coccus* on opuntia cactus, the source for cochineal. (Image by Steven Schwartzman).

#### ***D. Dyer's Madder in Prehistoric Technological Context***

While madder is often discussed in relation to other red dyes, it is also often linked to common vegetable dyes that produced other colors as well. Madder, along with woad (*Isatis tinctoria*), and weld (*Reseda luteola*), are generally considered to be a sort of primary color trinity of dye plants, providing clear reds, blues, and yellows, respectively. These plants were all grown and used extensively through much of Europe and the Near East from prehistory into historic times and are assumed to have been commonly available. Woad has been the subject of several interesting recent studies on ancient dye techniques and is currently cultivated as a sustainable and locally grown source of indigo dye (Hartl et al. 2015). The dye compound indigotin has been identified in many archaeological contexts stretching all the way back to Neolithic Çatalhöyük in modern day Turkey (Zohary 2012:166). It is clear that blue was also a culturally important color. There are many instances of textiles that were dyed with both woad and madder to achieve a purple hue, as true purple dye was generally rare and costly. The leaves, flowers and seed of the weld plant yield a clear yellow dye, although weld is by no means the only source of yellow dye. Unfortunately, the flavonoid compounds in yellow dyes are less stable than other dye compounds and yellow is considered to be the color least likely to preserve in the archaeological record (Gleba et al. 2012: 19).

Other sources of dye were also important but were limited either regionally or socially due to their scarcity. Lichen dyes were important in Britain, Scotland and Scandinavia, where they were used as sources of brown, blue or purple colors (Casselman 2001). Safflower (*Carthamus tinctorius*) was a source of yellow or orange dye, but was more common in the Near East than in Europe (Marinova 2009). The legendary Tyrian purple, which is produced by several species of murex shellfish, was difficult to obtain, in high demand and limited to the social elite (Barber 1991: 228-230; Taylor 1999: 37-49). In prehistoric Europe, several distant relatives of the madder plant were also used as dye plants. One example is “ladies bedstraw” (*Galium verum*), which was a perennial plant native to Europe that was hardy into Scandinavia and was used for various purposes, including as an aphrodisiac and a dye plant. Ladies bedstraw yielded a yellow, orange or red dye, depending on which source one consults.

The picture of dye plants in prehistoric Europe is exceedingly complex. There were actually hundreds of ancient dye sources, plant, animal, and mineral. These were often used in combination with each other and at times very complicated dye methods resulted. Extremely nuanced shades of every color became associated with certain regions or dyers, as texts like the Leyden and Stockholm Papyri attest (Caley 2008). Some dye materials listed in ancient sources have yet to be identified. It is also a matter of debate as to when and where these dye sources were found in Europe and the Near East. The path madder took into Europe is one such debate.

Mordanting techniques are an important component in dye research and will be explored in the experimental component of this thesis as well. Mordanting is the process of fixing dye colors through the addition of some sort of mineral or metallic salt. The type of mordant used can also affect the resulting color produced in the dye bath, and madder root dyeing is a good example of this. Certain colors could be achieved solely as a result of particular mordanting

techniques. Several texts (Casselman 2001; Dean 2014; Goodwin 1982; Grierson 1986) detail these dying methods, as well as varieties and sources for ancient mordants.

### ***E. Related Research***

A significant amount of research has also been conducted on the chemical compounds found in madder root (Jäger 2006; Nakanishi et al 2005) and on madder propagation (Sepaskhah and Beirouti 2009). Modern laboratory methods for working with ancient dyes and textiles (Clementi et al. 2007; Surowiec et al. 2007; Yeonhee et al. 2008) will be reviewed in Chapter 2.

Several previous publications have influenced this research project. The woad research discussed above inspired my own research, while Anna Hartl's methods and research approach influenced the structure of this thesis (Hartl et al. 2015). Hartl and her colleagues at the University of Natural Resources and Life Sciences in Vienna and the Cultural Heritage Agency of the Netherlands adopted several approaches to ancient dye plants that have yielded interesting data on ancient dyeing techniques. Hartl's approach incorporated a significant experimental component, growing woad, spinning textiles, and experimenting with dying techniques for best results. Hartl and her collaborators have also published data on the dye components of Iron Age textiles from Hallstatt and in general take a more academically rigorous approach to prehistoric European textiles than many more readily available sources.

Dominique Cardon's 2007 publication *Natural Dyes* presents extensively researched dye sources on a global scale. Although Cardon's work focuses on presenting botanical and chemical information on various dye plants and emphasizes their historical usage rather than archaeological data her approach to collecting worldwide dye methods as a type of ethnography is interesting and inspiring. Lastly, my thesis has been shaped by the research on dye plants and methods throughout Europe that Margarita Gleba and Ulla Mannering compiled in their 2012

publication *Textiles and Textile Production in Prehistoric Europe: from Prehistory to AD 400*.

This research compendium presents dye methods from the Bronze Age through Roman times in all parts of Europe and western Asia. Taking this type of comprehensive approach to dyeing across a large time frame and across a vast geographic area may produce more visible patterns for the progress of madder dye in a geographical and a developmental sense as well. A combination of these research styles, focused on one dye plant, may further illuminate the prehistory of madder.

#### ***F. Current Interest in and Importance of Dyer's Madder***

Madder is important in more ways than just as an artifact of past technology. As interest in natural, more eco-friendly dyes has increased, there has been a corresponding increase in the production of madder as a crop. There has also been an increased interest in studies of ancient dyes and dyeing methods (Ozen et al. 2014; Yusef et al. 2017). However, ancient European technologies such as dyeing are also generating a great deal of online folklore and a proliferation of misinformation that could be countered by more careful and scholarly examinations of the prehistory of these dye plants. Celtic re-enactors and a general interest in European heritage have produced information that is sometimes useful and interesting but is unfortunately rarely well-documented. For instance, there is a legend that the Celts fed madder to horses and falcons to dye their teeth and beaks red (Roberts 2017 “Madder Dye”). This legend may have some basis in fact, as subsequent studies have shown that madder does indeed dye the long bones of animals (Chamberlain 1944; du Monceau 1739: 391; Richter 1937: 591). However, the source of this Celtic tale is nebulous. Did the Celts have this practice? An analysis of this and other dye-related legends is presented in the literature review in Chapter 2.

The antecedents of madder root in Europe – where it originated and when it arrived in

Europe, how and where it was cultivated and traded – have not received a thorough treatment in archaeology or history. These antecedents may be impossible to track down, but at the very least a collection of information on archaeological evidence for madder, combined with dyeing methods and botanical research may provide a solid foundation for future research on dye plants in prehistoric Europe. There is certainly room for a comprehensive study of the data available from European sources to assess what can be learned about the prehistory of this important plant.

### ***G. Research Questions***

Madder root was undoubtedly an important dye source in prehistory, yet its early history in Europe has so far not been subjected to the same rigorous scholarly investigation as in other parts of the world. Little is known of its origins or cultivation methods, although this may be due in part to the secrecy surrounding dye methods generally, such as that seen with murex purple, or later with cochineal dyes. This research project aims to ground-truth both the body of literature around madder root and what is known about the plant itself. The following questions in particular are addressed in the literature review:

- 1) Can a systematic examination of the literature on madder lead to a clearer understanding of its "roots"?
- 2) How has madder been identified in past archaeological research and how has recent dye analysis affected this?
- 3) Can scientific material analyses of ancient textiles, such as HPLC and mass spectrometry, provide a more complex picture of the chemical signature of madder dyes that might help identify it in archaeological contexts and create a better understanding of the development of madder as an important European dye crop?
- 4) Can an experiment focused on identifying the growing and dying properties of *R.*

*tinctorum* provide insights into the accessibility and/or exclusivity of a good quality red dye source in Iron Age Europe given that very few people would have had access to kermes red dyes?

A combined synthesis of known archaeological sources along with better documented textual references are tested against an experimental component to generate a better understanding of the use and importance of madder and textile dyes in general in Iron Age Europe.

For the experimental component of this research, I grew madder root, tested it for cold hardiness (in a Wisconsin winter), fecundity, and root development over the course of two summers. This was done to test the idea that madder could have been readily grown on a scale large enough to supply the needs of non-elites as well as higher status individuals. Roots were harvested, dried, and processed for dye, using methods and materials appropriate for prehistoric Europe. Other aspects of the plant were also examined—in particular, pollen and seeds were harvested and examined in the second summer, and microscopic images of plant morphology were obtained. The distillation of the scientific literature and firsthand experience with the plants themselves allowed a better idea of the nature of this dye plant to emerge. A synthesis of existing research and data on madder root combined with the experimental data yielded insights into the importance of this material as potentially an exchange item and a status marker in ancient societies. It has also addressed the question of the importance of the color red in ancient cultures by comparing the different intensities produced by combining various mordants and material with the dye, and in identifying other important sources of red in prehistoric Europe. The thesis has been designed to provide insights into the use of these perishable materials and how they were involved in social identity in prehistoric Europe.

## **CHAPTER 2: LITERATURE REVIEW**

### **Part 1: The Botanical Aspects of Madder**

#### ***A. Botanical Characteristics of *R. tinctorum****

Dyer's Madder (*R. tinctorum*) is a member of the *Rubiaceae* family (Fig.2.1), a large botanical family that contains over 450 genera and 6500 species, mostly large woody plants.

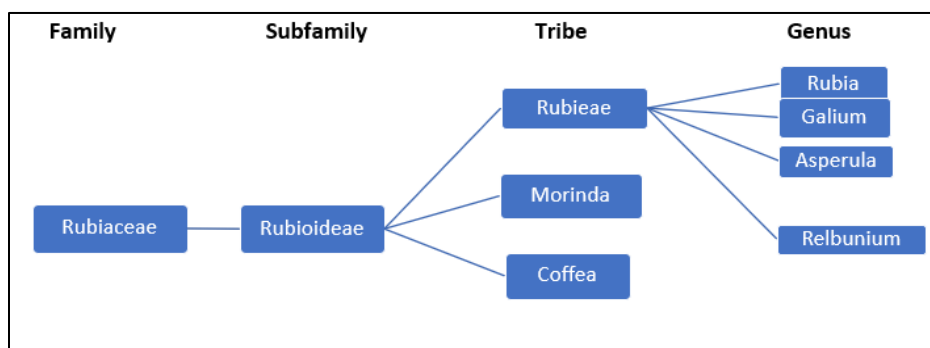


Fig. 2.1. A simplified plot of *R. tinctorum*'s botanical lineage. The genera *Galium* and *Asperula* also occur in Europe, but the genus *Relbunium* is a dye plant only found in the Americas.

Madder's tribe, *Rubieae*, contains 14 genera and over 900 species, which are mostly herbaceous perennials (Natali et al. 1995: 428). The largest of these genera is the *Galium* genus, while the *Asperula* genus is the second largest. The genus *Rubia* contains 78 accepted species and is now considered to be an older clade than most of the other genera (Natali et al. 1995: 436). The earliest fossil pollen evidence for the *Rubia* genus (Fig. 2.2a-b) comes from the Miocene, in Venta del Moro, Spain (Muller 1981: 94), although genetic evidence suggests an earlier origin from the basal species for the *Rubiaceae* family (Natali et al. 1995: 436). Other genera within this tribe appear to have originated in the Pliocene, making this tribe a relatively recent one.

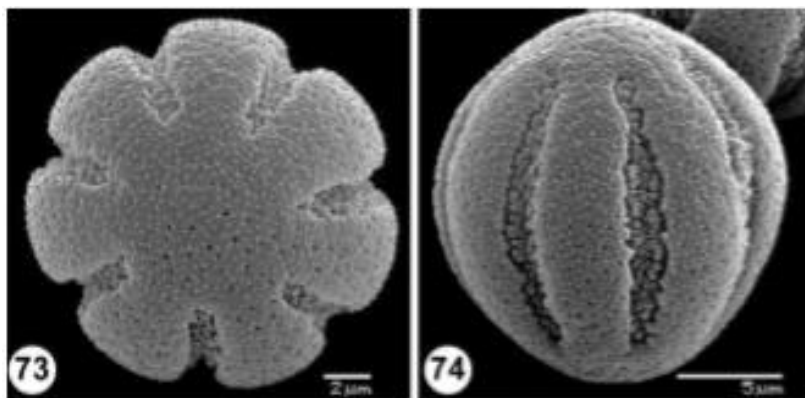


Fig. 2.2a-b. *R. tinctorum* pollen. 2.2a. Left: polar view, 2.2b. Right: equatorial view. (Images: Marcel Verhagen in Huysmans et al. 2013 #73-74).

*R. tinctorum* is an herbaceous perennial with a semi-trailing habit, and its fleshy stems and lanceolate leaves are edged with small retrorse spines that allow the plant to cling to other plants or to passersby. Pale yellow star-shaped flowers form during the second season of growth, and usually occur in pairs at the leaf joints (Fig. 2.3).



Fig. 2.3. *Rubia tinctorum*, botanical drawing. (Image: Textile Research Centre)

Fruits form in late summer and are small and green, eventually ripening to black by late summer or early fall. While the plant dies back to the ground during winter, the roots are perennial. The red branching roots can extend for several feet under the soil. Madder roots contain several important dye compounds that create madder dye, which will be explored more fully in the section on its chemistry below. Several different strains of *R. tinctorum* exist today. For instance, some strains will spread by long rhizomes, but the famous strain developed in the Netherlands during the heyday of madder production does not; this variety remains neatly in rows. This Zeeland madder was famously of high quality up until the crash of the madder market at the end of the 19<sup>th</sup> century (Cardon 2007: 108-9). The quality of madder dye can be noticeably affected by soil quality. Chalky humus-rich soils, such as those in southern France, produce a brighter scarlet red than clay soils, where the dye colors remain in the orange range (Cardon 2007: 110).

The pollen morphology for the *Rubicaee* family is not well studied, but the general view is that the three genera of European dye plants are very similar (Fig. 2.4a-c). It is felt that *Rubia*, *Galium*, and *Asperula* cannot be differentiated based upon pollen morphology alone, and while recent work suggests that there are slight but noticeable morphological differences (Huysmans et al. 2013), how well ancient pollens might be reliably typed to a species in the *Rubieae* tribe remains debatable. Pollen analysis can also be problematic in some of the environments in which textiles are preserved in Europe, especially bogs (Munksgaard 1981: 42). Unfortunately, SEM images for *R. peregrina* were not available.



Fig. 2.4a-c. Equatorial views of *Rubieae* tribe pollens. 2.4a. Left: *Asperula* spp. 2.4b. Middle: *Galium* spp. 2.4c. Right: *Rubia tinctorum*. (Images: Marcel Verhagen in Huysmans et al. 2013 #2,37,74)

### **B. Distribution of *R. tinctorum***

Dye plants belonging to the *Rubiaceae* family have a worldwide distribution (Fig. 2.5), and most were utilized for dyeing by the indigenous cultures in these locations. *R. tinctorum* has a range throughout southern Europe into the British Isles, Northern Africa and the Levant. Much of this range may be due to madder's extensive cultivation in prehistoric and historic times, and the fact that it has naturalized in many areas.

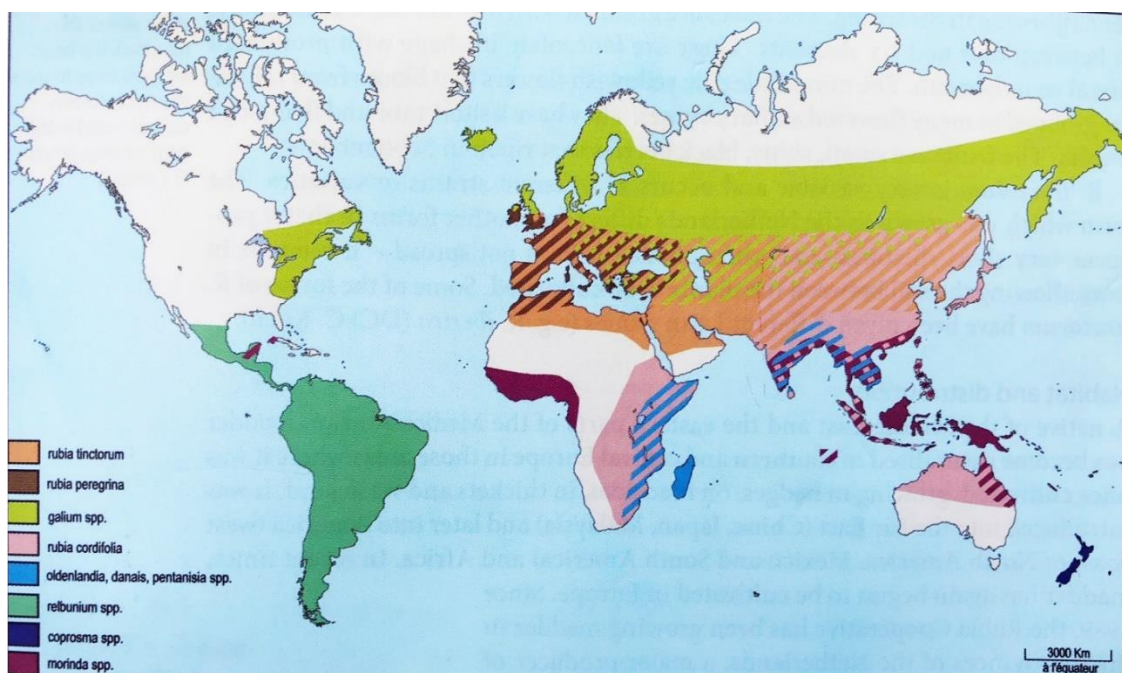


Fig. 2.5. Worldwide distribution of dye plants in the *Rubiaceae* family. As the map shows, Europe has a complex mix of several closely related genera, and *R. tinctorum* now has a very wide distribution. Map created by D. Augerd/D. Cardon, CNRS.

Native species of three of the major genera (*Rubia*, *Galium*, *Asperula*) can be found in Europe (Fig. 2.6a-d) and were possibly in cultivation and use beginning in the Neolithic, perhaps as dyes or as medicinal plants (Cardon 2007: 122). Morphologically, these three genera share some qualities, and interestingly, there are dye plants in each genus. *R. tinctorum* is also not the only species of madder found in Europe. While *R. tinctorum* is not considered indigenous to Europe, especially northern Europe, there was a species of *Rubia* that would have been available to prehistoric European dyers well before the Bronze Age: *R. peregrina*. This “wild madder” is an evergreen perennial that is thought to be native to southern and central Europe (Cardon 2007: 122-123). *R. peregrina* is a common wild plant in south-central Europe and the southern half of the British Isles. Cultivation, harvest and dye preparation are identical to *R. tinctorum* (Cardon 2007: 123). *R. peregrina*, however, contains different dye compounds than other *R. spp.*: pseudopurpurin is the primary dye compound while alizarin plays less of a role (Cardon 2007: 123). There is some written evidence that wild madder was collected in Egypt and in Roman times and was used in combination with other dyes (especially woad) to create purple and black hues (Cardon 2007: 123).

Several other dye plants comprise the “madder-type” dye group (Fig. 2.6), but two of particular interest here are *Asperula tinctoria* and *Galium verum*. *Asperula tinctoria* is a “woodruff” that produces a red dye from its thin roots. While in a separate genus than the *Galium spp.*, it is often included with this other genus in terms of its dye qualities, as it has very similar dye chemical concentrations (Cardon 2007: 127). *Galium verum*, or ladies bedstraw, is another dye source closely related to *R. tinctorum*. *G. verum* is one of several species in this genus that have been used as dye plants in the past (*G. boreale*, *G. molugo*, and *G. sylvaticum* are other minor bedstraw dyes) (Cardon 2007; 123-126). *G. spp.* are very cold-hardy plants, native

throughout Europe and into Scandinavia and Scotland, which also produce a red dye. All these madder-type dye plants contain small concentrations of alizarin, the primary dye compound found in dyer's madder. However, these concentrations are almost undetectable; purpurin is the primary dye in all these plants, along with several other minor dye compounds (Cardon 2007: 127). It is thought that with the right technique, each of these dye plants can produce a variety of colors ranging from reds and oranges into pinks and violets. The high concentration of alizarin in *R. tinctorum*, however, separates it from all these other madder-type plants.



Fig. 2.6a-d. Madder and madder-type plants. Fig. 2.6a. Upper left: *A. tinctoria* or dyer's woodruff. (Image: Tomáš Mrázek). 2.6b. Upper right: *G. verum* or ladies bedstraw. (Image: Per Aasen). 2.6c. Lower left: *R. peregrina* or wild madder. (Image: Leslie Linares). 2.6d. Lower right: *R. tinctorum* or dyer's madder.

## **Part 2: Chemistry, Alchemy, and Madder**

### ***A. Organic Dye Fundamentals***

Dyer's madder is one of several important vegetative sources of color for which there is physical evidence from at least the Late Bronze Age on in Europe. A brief analysis of its chemistry is important, as the unique set of compounds in madder affects the techniques required to create the dye and is the critical element in modern analyses of ancient textiles in efforts to identify those dyes. A summary of the nature of plant dyes, and madder in particular, follows below.

Dyes are organic compounds that are often derived from plants, but can also be sourced from insects or shellfish, as in the case of murex purple. Pigments are inorganic, mineral sources for colorants, and are found mainly in paints or ceramics. Red ocher, copper compounds, cobalt, cinnabar or lapis lazuli for ultramarine blue are examples of pigments. Pigments, as mineral sources, tend to preserve better than dyes. Dye compounds often deteriorate over time, although their degradation products can sometimes leave a chemical signature that is strongly indicative of the presence of that dye, such as the hydro-benzoic acids that remain after the deterioration of flavonoid yellow dyes (Surowiec et al. 2007: 2070). For a plant or insect extract to function as a dye, it must chemically bond to the fibers. Dye compounds should not break down and their colors should remain light-fast and not become muddy. Dyes should not be removed by washing. The mechanism by which dyes adhere to fibers is not well understood, but it is felt that charged parts of the fibers and dye molecules directly bond; hydrogen bonds and hydrophobic interactions also may be involved (Simpson et al. 2001: 372). It is also thought that the fibers'

structural configuration may be altered to create more binding sites, which increases their affinity for dye compounds (Simpson et al. 2001: 372).

There are several different types of dyes that require different methods to achieve a fast bond with fibers. Direct dyes are derived from dye plants that are either boiled or soaked in water to prepare the dye bath and fibers are immersed in the bath. Direct dyes can also be directly applied to fibers, such as being painted onto a textile. Most dyes, however, require a more complex treatment to achieve a colorfast result. Mordant dyes require a pretreatment with a mordant agent to alter the fibers chemically or physically to allow dye molecules to adhere. Mordants, which are generally metal salts, can also allow certain dye compounds that would normally be insoluble in water to go into solution and interact with the fibers. The history of mordants is unclear, as many do not leave chemical traces that are detectable. Those that do leave detectable traces, however, such as aluminum or iron salts, can be difficult to identify in textiles that have been buried, as iron and aluminum are universally common in soil. It is uncertain how prevalent mordants were in the Bronze Age (Barber 1991: 225-6, 236-7), but alum was being mined and synthesized by the Egyptians and Greeks by the Iron Age (Cardon 2007: 24). Certain plants that naturally store aluminum in high concentrations have also probably been used as mordants. These include the club mosses (discussed more fully in Chapter 3), a group of plants native to Europe. Dye plants growing in areas with high concentrations of iron or aluminum in the soil may contain high levels of these metals as well and may be superior dye plants with a sort of built-in mordant that is region-specific (Strand et al. 2015: 56). The majority of dyes, including madder and all other red dyes, are mordant dyes. However, these come from a wide variety of sources, including plants, fungi and insects.

Vat dyes are another variety of dye that requires intermediate steps to achieve a color-fast result. This technique is used with indigo dyes and murex purple. Both these dyes are insoluble in water and require special reducing conditions in alkaline conditions before they can go into solution and adhere to textiles. Once fibers are removed from the dye bath and make contact with oxygen, they will begin to change color to blue or purple as the dye compounds precipitate within the fibers (Strand et al. 2015: 56; Cardon 2007: 4).

### ***B. Dyes and Alchemy***

The early history of the practice of dyeing is documented in archaeological contexts, especially in the form of textiles, and will be covered later in this chapter. However, another fascinating source of information on dyeing techniques is represented by the alchemical writings that have survived from the Classical world. Two papyri in particular, the Papyrus *Graecus Holmiensis* (also known as the Stockholm Papyrus) and the Leyden Papyrus detail dye methods that have been frequently referenced in discussions of dye practices in the past. These two documents were written in Egypt in Demotic Greek around AD 300 and contain descriptions of technical methods for dyeing textiles and coloring or cleaning precious stones. Both are almost certainly from looted graves, probably in the area of Thebes in Egypt, and were in remarkably good condition (Caley 2008: 1-2). These ancient writings were acquired by Johann d'Anastasy, an antiquarian working as the Swedish-Norwegian Vice-Consul at Alexandria in 1828 (Caley 2008: 1). In 1829, D'Anastasy sold a collection of papyri, including the Leyden Papyrus, to the Museum of Antiquities at the University of Leyden. A few years later in 1832, he donated another collection to the Swedish government (Caley 2008: 1). Both these papyri were translated and analyzed by European researchers in the late 19<sup>th</sup> and early 20<sup>th</sup> centuries, as interest in alchemy gained momentum. However, neither of these documents could be classified

as mystical or magical alchemical texts, as both essentially consist of lists of recipes or techniques for dyeing (Caley 2008: 3). It is worth noting, however, that these papyri were part of a collection of writings that did include such texts, and it is speculated that these documents may have been secreted away after the Diocletian decree of 290 AD, which included an order to destroy alchemical writings (Caley 2008: 4).

Both of these documents are likely an amalgam of instructions and techniques from earlier works that have not survived; they may be all that remains of the lost four-volume work on the art of dyeing by Pseudo-Democritus and seem to be part of a tradition of practical recipes that began with Assyrian cuneiform tablets of glass recipes and continued into Medieval times (Caley 2008: 5). While these writings are not magical in nature, they most likely constitute proprietary trade and technical knowledge, and were possibly secret documents in that regard. For the purposes of this research, the Stockholm Papyrus has far more to offer, as it lists several dye recipes involving madder root, as well as information on ancient mordanting techniques. These recipes informed some of the experimental component of this research and will be further explored in Chapter 3. As a final note on dyeing and ancient alchemy, it also seems clear that the process of dyeing, involving mordants, heat and pH, became a sort of alchemical template that was transferred to other materials, although applied to a lesser degree in metallurgical alchemy. The process of coloring textiles was directly applied to coloring gemstones in the Stockholm Papyrus, for example, and this practice extends unchanged into the Middle Ages as a persistent link between dyeing and alchemy (Linden 2003: 146).

### ***C. Dyes and Their Chemistries***

Part of the complexity of both dyeing and analyzing textiles is that there are many sources of red dye and these are derived from different animal or plant sources with very

different chemistries. Table 2.1 lists the most important red dye sources that existed in prehistory, and the main active dye compound found in each. Many of these sources, the plants in particular, contain multiple dye compounds that can vary according to climate and the nature of their preservation. Analysis and identification, therefore, can be complicated to say the least. Many of these dye compounds are chemically closely related (Taylor 1999: 39).

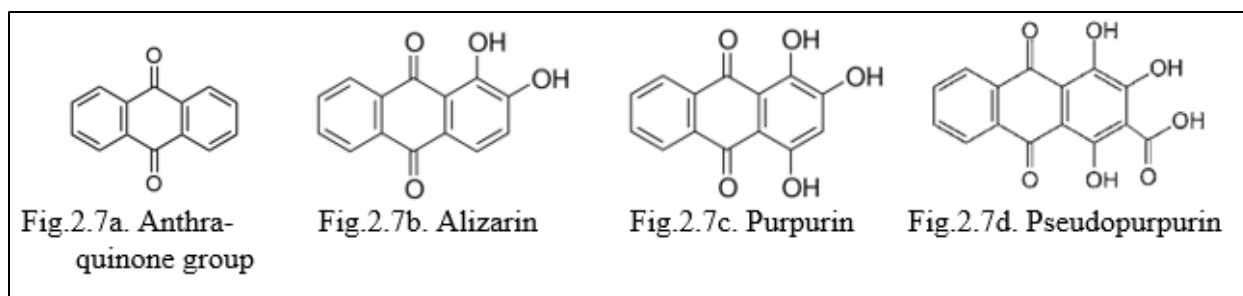
**TABLE 2.1. RED DYES IN PREHISTORY**

Dye Name	Primary Dye Compound	Prehistoric Distribution	Dye Type	Source of Dye
<b>Madder red</b>	Alizarin	Mediterranean, Near East, Eurasian steppe	Mordant	<i>R. tinctorum</i> root
<b>Kermes</b>	Kermesic acid	Mediterranean coast Spain, Italy and N. Africa	Mordant	<i>Kermococcus vermilio</i> , scale insect
<b>Armenian cochineal</b>	Carminic acid	Armenia	Mordant	<i>Porphyrophora hameli</i> , insect
<b>Polish cochineal</b>	Carminic and kermesic acids	Poland	Mordant	<i>Porphyrophora polonica</i> , insect
<b>New World cochineal</b>	Carminic acid	Mexico and Central America	Mordant	<i>Dactylopius coccus</i> , insect
<b>Lac</b>	Laccaic acid	India	Mordant	<i>Laccifer lacca</i> , scale insect
<b>Madder-type reds, bedstraws</b>	Purpurin and pseudo-purpurin	All of Europe and the British Isles	Mordant	<i>Asperula tinctoria</i> , <i>Galium spp</i> , <i>R. peregrina</i> , roots
<b>Fungal red dyes</b>	Boletol and dermorubin	Scandinavia	Mordant	<i>Boletus</i> and <i>Cortinarius</i> , fungi

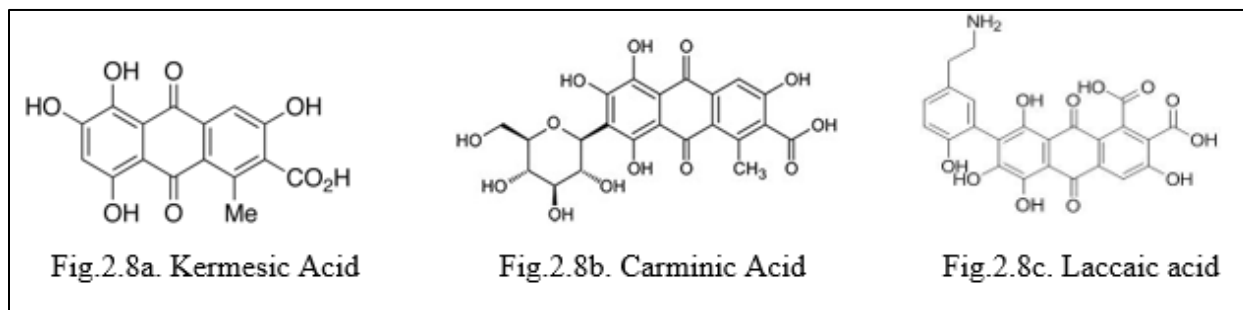
In Europe and the Near East, madder and kermes were by far the most important red dyes available. Polish and Armenian cochineals, while desirable reds, had very small areas of distribution, and there is less evidence of their presence in the archaeological record, although

there are written accounts of their existence and usage (Cardon 2007: 635-652; Taylor 1990: 37; Walton Rogers 2003: 27). Red dyes from the *Boletus* and *Cortinarius* fungi are documented in some textiles especially in Scandinavia and Scotland, but their limited distribution and their status as a wild-gathered plant also limits their presence in the archaeological record. Cochineal from the New World and lac from India both are ancient and enormously important red dyes. However, these were not known in Europe and the Near East in prehistory and therefore do not factor heavily into this research. Kermes red was indigenous to Europe and the Near East, and the earliest evidence of red dye in Europe contains kermesic acid (Barber 1991: 224). Madder-type plants, which would also have been available to the earliest Europeans, were apparently a later development in dye technology.

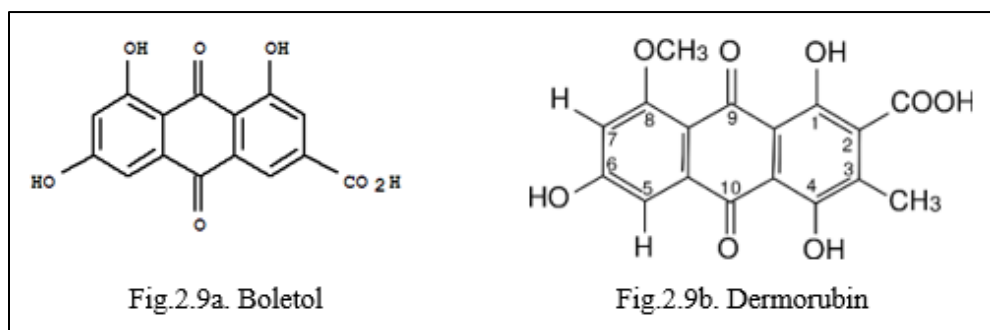
It is worth reviewing the different dye chemical compounds in each of these red dyes here, and as the chemical illustrations below show (Figs. 2.7a-j), there is a great deal of similarity between these compounds on a chemical level. It is speculated that this similarity is shared by dyes that give a red hue (Taylor 1999: 39). All these red dyes are anthraquinone natural dyes, an important dye group, and a promising alternative to the problematic health and environmental concerns surrounding azo dyes (Shahid-Ul Islam 2017: 82). As anthraquinone dyes, these compounds each share the organic anthraquinone group, as seen in Figure 2.7a. The dye components of madder and madder-type plants all contain the anthraquinones alizarin, purpurin and pseudopurpurin in varying amounts. As Figures 2.7a-d below show, these compounds are chemically closely related.



The insect dyes kermesic acid, carminic acid, and laccaic acid also share some chemical similarities, with the same anthraquinone group in their chemical makeup (Figure 2.8 a-c). All of these dye compounds also share an acid, as these chemicals are produced by the insects as a predator deterrent. Carminic acid has a sugar group attached to the anthraquinone and that sugar group is the difference between carminic and kermesic acid (Figure 2.8a-c) (Sequin 2017: 155). Lac dye is more chemically distant to the other insect dyes, having an amino group ( $\text{NH}_2$ ) as a part of a carboxyl group on the anthraquinone compound.



Lastly, the fungal red dyes comprise another subset of anthraquinone dyes (Fig. 2.9a-b). The fungi that produce these dyes are occasionally found in Europe, Scandinavia, the British Isles and North America, and have limited use as a dye crop because of their scarcity. However,



fungus dyes appear to have been a tradition in Scandinavia before the introduction of dyes from other parts of Europe and the Classical world (Taylor 1999: 39).

#### ***D. Other Chemical Components of *R. tinctorum****

While the anthraquinone dye content of *R. tinctorum* is certainly its most economically and historically important chemical component, dyer's madder is a true cocktail of ingredients that have also been studied for centuries. Recent analyses of madder have found that the root actually contains 36 anthraquinone compounds, although only 15 are involved in dyeing (Cardon 2007: 112). However, this combination of colorants is a part of what gives the natural dye its rich and complex color. One of the anthraquinones in madder is lucidin, which has undergone a great deal of recent research as a carcinogen (Nakanishi et al. 2005; Yasui et al. 1983), whether it is consumed as a food colorant in Japan (Inoue et al. 2009: 409) or even used in textiles, as there is concern that lucidin could be absorbed through the skin (Jäger et al. 2006: 28). On the other end of the health spectrum there is research into the potential for alizarin as a cancer treatment in bone cancers (Fotia et al. 2012). As madder dyes have been demonstrated to have an affinity for bone, there is research into madder as a means of creating targeted treatments for specific cancers. Madder has been used in the past as a bone stain as it stains growing or malignant bone

(Schorr et al. 1959: 410); new medicines are exploring ways of taking this characteristic of bone dyeing even further. This trait of madder dye will be explored more fully later in this chapter.

### ***E. Methods of Dye Analysis in Archaeological Textiles***

Because of the variety of dye compounds that could potentially be present in prehistoric red dyes, chemical analysis of prehistoric textile dyeing practices has been complicated. Dye analysis on archaeological textiles is a fairly recent development. Prior to the late 1980s, any reports of specific dyes on ancient textiles can be assumed to be educated guesses. Over the past few decades, there has been a refinement of techniques in dye analysis that have trended toward better, more inclusive results, and less destruction of precious textiles. Dye extraction for archaeological textiles was developed in the early 1980s, when dyes were extracted from textiles found at Baynard's Castle in London, and dye compounds were identified (Walton and Taylor 1991: 5). In the 1980s, dyes were extracted with a series of solvents and analyzed with a UV/visible spectrophotometer; paper and thin-layer chromatography (TLC) were used to confirm results (Walton 1988: 144). One of the difficulties with methods such as this is that different dyes have very different chemistries, and a series of solvent systems must be used to extract all types of dyes (Walton and Taylor 1991: 5). Vat dyes and direct dyes, for instance, require a different solvent system for extraction than the anthraquinone mordant dyes. Therefore, dye compounds can potentially be missed in analysis if they were simply not extracted for spectrophotometric analysis. To a certain extent, one has to know what one is looking for in order to find it or to develop a truly inclusive solvent system. Moreover, the large samples required by the analyses available at the time meant that only very few archaeological contexts could be studied.

The initial steps into dye analysis were taken by Mark Whiting and G.W. Taylor while methods of performing TLC on ancient textiles were described by Schweppe (1979: 20). In the mid-1980s, pioneering research was also conducted by Penelope Walton and Lise Bender Jørgenson on the dyes in ancient textiles from Germany, Norway and Denmark. Bender Jørgenson and Walton concluded that the results tended to underscore the similarities between local textiles and strengthened the possibility that some items had been imported. Dye analysis proved to have the potential to reveal valuable information about ancient textiles. It became clear that, along with more sophisticated and less damaging methods of analysis, a chemical database on dyes, both local and imported, was necessary (Walton 1988: 144). This is slowly being developed, as more textiles are being tested for dyes and the unique fingerprints of dye recipes and even the sources for dyes are being recorded (Nowik et al. 2005: 843). There are also efforts to provenance wool via strontium isotope testing to help trace the sources of some textiles (Frei et al. 2009). By the mid-2000s, high performance liquid chromatography (HPLC) and mass spectrometry (MS), and in some research, gas chromatography (GC), had become improved methods for dye analysis of ancient textiles, with many examples of analyses of dyes and their degradation products carried out via this method (Boldizsár et al. 2006; Clementi et al. 2007; Nowik et al. 2005; Surowiec et al. 2006; Surowiec et al. 2007).

Dye analyses in more recent years have focused heavily on non-destructive approaches to archaeological materials. This is important in the analysis of especially fragile, important or rare textiles. However, it seems that while these methods are non-destructive, they are not as inclusive as HPLC-MS or TLC, and data may be missed in the analysis. These techniques also require costly new equipment that may not be available to many researchers as well as training and possibly new methods of organizing results, which could impact databases already being

built. Methods such as *in-situ* spectrofluorimetry, Raman spectroscopy (MRS) and infrared spectroscopy (Van Elslande et al. 2008; Zaffino et al. 2017) all offer colorant analysis without destructive solvents, and as this field appears to be quickly developing, there may be new methods that can capture more compounds. Capillary electrophoresis (CE) has been tested on historical madder dyes and offers high sensitivity from minute samples (Goltz et al. 2012). X-ray fluorescence (XRF) is another method that may hold promise in dye analyses, as it is also a non-destructive method. However, some dye components can be missed with this method as XRF cannot test for all elements (Nowik et al. 2005: 842). XRF is particularly good at detecting mineral and metal elements, therefore it may have potential in the detection of dye mordants (Nowik et al. 2005: 842).

Another aspect of recent dye analysis appears to be a refinement of extraction techniques. Newer, less damaging solvents are being developed that would allow extraction of the dye compounds without degradation of the textile substrate and would conserve more of the chemicals present in the samples (Ford et al. 2017; Frei et al. 2010). The past decade has seen an incredible proliferation of analytical techniques for dye analysis; clearly more will follow in the future, but the trend toward more sensitive, non-destructive analysis is encouraging. However, finding low-cost options for the multitude of textiles in need of analysis should also be a priority.

#### ***F. Textile Conservation and Ancient Dyes***

Parallel to the story of chemical analyses is the saga of textiles in museums and other collections. The survival story of ancient textiles does not end with their excavation. A great deal of information on ancient textiles is lost after they have been recovered, as these items deteriorate quickly once out of the environment in which they were found. Many textiles

specimens have been lost in museums or other collections because of poor preservation or storage practices (Desrosiers and Lorquin 1998). This is in part because the technology required to save these items did not exist until fairly recent times. But it is also true, unfortunately, that textiles were not always valued by early excavators and museums (Leene et al. 1981: 239) and unless they were truly spectacular often were not described in excavation records. Efforts to preserve textiles should be focused as much on museums and other public collections as on material from excavations, as these all are endangered artifacts. With new developments in dye analysis, it may be possible to test some of the textiles currently in collections, provided they have been treated in a manner that would still make chemical tests viable. Some conservation methods, unfortunately, can have an adverse effect on chemical analysis and dating methods (Munksgaard 1981: 42). As any chemical additives would skew results, it is important that these textiles remain free of any processes or storage practices that would contaminate samples, and that dye analysis and dating is done by qualified experts (Leene et al. 1981: 242-3). A synthesis of laboratory analysis with the reality of museum collections would be ideal.

### **Part 3: Archaeological Evidence for Madder**

#### ***A. An Introduction to Ancient Dyes and Archaeological Textiles***

Archaeological evidence for madder root is primarily associated with ancient textiles. Although other types of evidence for madder exist, textiles are the main source for tracing the history of this plant and its usage. Because organic dyes are linked to some of the most ephemeral of ancient materials, the record for madder is incomplete. The lack of evidence for dyed textiles is in part reflected in the lack of archaeological textiles in general. As textiles do

not preserve well in all areas, the record for dyes is spotty at best, so to speak. Unfortunately, the climate and soil conditions of Europe generally do not promote good preservation of organics such as textiles. However, in certain conditions or microclimates, exceptional preservation can occur. Anaerobic waterlogged bog sites, the Hallstatt salt mines, or frozen conditions such as that in which Ötzi was preserved have produced newsworthy preservation of organics. On an even smaller scale, micro-environments such as soils high in carbonic acid, or close proximity to metals from jewelry or weapons in burials or votive deposits can also promote preservation of textiles and organic dyes. However, it is very hit and miss, and creating a cohesive timeline for textiles or dyes is a challenging undertaking.

Before we begin an exploration of dyed ancient textiles, however, it seems important to address the difference between dyed textiles in modernity and dyed textiles in prehistory. Firstly, fiber choices in prehistory were limited to flax, hemp, nettle, some silk in later times, and especially wool. The evidence for the use of these fibers is covered briefly in Chapter 3. Cotton textiles, which originated in the Indus Valley, were not commonly available in Europe until historic times. With few exceptions, dyed textiles were limited to animal fibers, especially wool. Most vegetable fibers were not dyed, although linen and other plant fibers take indigo dye from woad well, and there is early evidence for this in Egypt and Europe (Cardon 2007: 374). Madder is known as a difficult dye to use on vegetal fibers; linen is notoriously difficult to dye with madder and was dyed poorly in the belt of Tutankhamun, for example (Crowfoot and Davies 1941: 123). With all of these fiber types, dyeing can be done on the fiber, the yarn, or the woven textile, depending on the process used and the effect sought.

The vast majority of dyed textiles found in prehistoric Europe are of wool. There are exceptions to this, but very few. This is in large part because silk was rare, and plant fibers do

not dye readily. Wool, however, takes dyes very well, and a wide spectrum of satisfyingly vivid colors can be made from vegetable dyes and wool yarns. Wool was an important Bronze Age addition to European textiles, but wool was not a constant or homogenous material throughout prehistory. The earliest domesticated sheep were far hairier, with a wiry outer coat of kemps, and a fine undercoat of short wool (Ryder 1981: 224). With time and selective breeding, wool fibers changed and the amount of kemps declined in woolen textiles. Wool evolved as sheep were bred for better fibers, and as methods changed for collecting that wool (Walton 1988: 146). Textiles can be analyzed for this change from hairier wools to the more advanced short wools of later times (Wild 1981: 15). Sheep were not sheared until the Roman or Migration Periods, but molted and were instead “plucked” or “rooed”, and the fiber content of woolen textiles reflects this practice (Walton 1988: 144-6). Wool eventually was available as a white or nearly white fiber and this made it especially conducive to dyeing. Not all wools were white, however, and there are many multi-colored textiles and patterned weaves that owe their colors solely to the sheep of many colors that provided the fibers.

Another important difference between modernity and the past in terms of the textiles is that textiles were also not woven as bolts of cloth prior to the medieval era. Garments were woven on the loom, so to speak, rather than cut from the cloth and sewn together. The lack of waste in this process makes this practice understandable. Only with the advent of the broad horizontal handloom in the Middle Ages did textile technology move to bolts of cloth or meterware (Wild 2013: xv). Therefore, in prehistory we should think of “garments” rather than “cloth” as being produced, traded, and dyed.

Lastly, there is the issue of color and modern textiles. With the advent of modern synthetic dyes toward the end of the 19<sup>th</sup> century, the idea of “prestige” colors, expensive or rare

colors, came to an end. Perhaps the last of these moments in history was the excitement over the color mauve when it was synthesized in 1856 (Chenciner 2000: 260-1). Prior to the advent of synthetic dyes, though, new colors and new dye techniques created textile crazes, as in the case of the introduction of cochineal from the New World into Europe by the Spanish, or the vibrant calico prints imported from India that were banned in Europe until the process could successfully be stolen by European textile manufacturers (Chenciner 2000: 186-189). In prehistoric and Roman times, certain color sources were also in high demand. Kermes red was a tribute paid to the invading Romans during their tenure in Europe; Spain paid half its tribute to Rome in kermes insects (Cardon 2007: 617). Color was legislated in Rome, and red was an important military color for the Romans, as they used it for their crimson red cloaks (Taylor 1999: 41). It appears that the Romans used madder for their cloaks as well (Chenciner 2000: 39; Taylor 1990: 41). Tyrian purple was not just a rare and valued as a dye, but a heavily legislated one. Only high prestige individuals could wear this color; Roman Sumptuary Law dictated that only the Emperor could wear purple, for instance. Madder had a part to play in this history as well, if a somewhat underhanded one. Madder was an integral part of the legendary Turkish Red, a particularly rich, deep red with a recipe so complex (and secret), that Europeans were never able to truly perfect it (Chenciner 2000: 189-190). Madder was also one half of the recipe for Egyptian Purple, a sort of poor man's Tyrian purple (Taylor 1999: 38). Madder root was a relatively inexpensive source of red dye (Barber 1991: 227), not as ancient or as expensive as kermes as a source of red in Europe, but by the late Bronze Age, coming into its own as a widespread red colorant.

Madder is often listed as one of a trinity of dyes that make up the three primary colors of red, yellow and blue: madder, weld and woad. It is possible that this trio of dyes is a modern

grouping to align with our idea of color theory and a more scientific approach to color and the behavior of light. However, it appears that these three dyes were associated with each other long before science delineated the primary colors. A poem from the 14<sup>th</sup>-century poet Geoffrey Chaucer, “The Former Age”, indicates that “mader, welde and wood” had been grouped together since at least the 14<sup>th</sup> century, well before the advent of optics or the nature of light and color were discovered. Perhaps these dye plants were grouped together because of the clear colors they produced, or because of their symbolic importance. Perhaps there was another reason for this grouping, but at this point, that reason is unknown.

The last general theme in European archaeological textiles is that of preservation. As has been noted before, Europe is not an area of the world where preservation is beneficial to organics. Even in the rare instances when textiles survive, dyes may not. Textile analyses have shown that even if a textile has been dyed, that dye may not preserve even if the textile survives, as preservation conditions may remove the dye (Strand and Nosch 2015: 55). Textile quality may also affect dye results, as finer woolens take dye better than coarser weaves (Bender Jørgensen 1981: 51-2). The acts of carding and combing wool act to remove or damage the waterproof outer coating, or epicuticle, and allow dye to penetrate (Bender Jørgensen 1981: 52). Preservation of madder dye in particular depends on several factors: an anaerobic environment is conducive to alizarin and purpurin preservation while exclusion of light aids in dye compound preservation (Clementi et al. 2007: 47). While European peat bogs can provide such conditions, and have been rich sources of archaeological materials they also are not without problems. Peat acids can often either dissolve or conceal dyestuffs (Wild 2003: 79). To add to the problem of preservation, Europe has also been the locus for centuries of avocational archaeologists, two World Wars, and numerous well-meaning individuals and institutions whose actions have

inadvertently contributed to the loss of many important textiles finds (Desrosiers and Lorquin 1998). The story of textile survival does not end once the excavation is complete. Often there is a great deal more that happens (some of it tragic) after these textiles are discovered. Ancient textiles have sometimes suffered greatly both during and after recovery over the last two centuries.

### ***B. Earliest Textiles, Pigments, and Organic Dyes and Ancient Evidence for Red***

The earliest European textiles found are fishing nets from Mesolithic sites in Finland and woven cloth from the submerged Ertebølle settlement site at Tybrind Vig in Denmark ca. 4200 BC (Bender Jørgensen 1990: 2; 2003: 54). The earliest evidence for organic dyes originates in Mesopotamia, with the Sumerians and the Akkadians using sumac and pomegranate on wool by the end of the third millennium BC (Walton Rogers 2003: 25). Woad appears to be the earliest of the important vegetal dyes utilized (Walton Rogers 2003: 26), and evidence of woad cultivation is found at such ancient sites as Çatalhöyük (Barber 1991: 223). Woad would remain the primary source for blue dye in the Near East and especially in Europe until it was ousted from prominence by imported indigo in the 16<sup>th</sup> century (Eastwood 2003: 163). Early evidence for dyes has also been found on textile fragments from Nahal Mishmer in Israel from the 4<sup>th</sup> millennium BC, in Mesopotamia from the 3<sup>rd</sup> millennium BC, and in dyed linen mummy wrappings in the 3<sup>rd</sup> millennium (1<sup>st</sup> dynasty or 3100-2890 BC) (Barber 1991: 224). It is possible that tattoos may have contained organic dyes, although there is little confirmed evidence for this. Tattoos generally involved pigments such as soot or mineral colorants such as red ochre. There is pictorial evidence for tattooing in prehistoric Europe dating back to the Magdalenian, and Greek ceramics depict tattooed Thracian men and women (Zidarov 2017: 138-189). There is some evidence from the kurgan burials on the steppe that organic pigments were

also used (Yablonsky 2017: 228), but at this point there seems to have been no analysis of these materials. Red in tattoos is rare even into historic times, and usually indicates the use of the (toxic) mercuric mineral cinnabar (Angel 2017:117). All evidence suggests that pigments, rather than dyes, were used in ancient tattoos, usually in mineral form.

Red ochre was almost certainly the first red pigment; its iron stains would have made distinctive and permanent color changes to early textiles. A red thread found inside a bead at Çatalhöyük dated to the 7<sup>th</sup> millennium BC has been cited as the first evidence for red dyes (Barber 1991: 223; Mellart 1967: 219). There is no confirmation, however, that this red dye was madder. Red is also mentioned in connection with a particular type of red linen cloth used in Egyptian funeral rites and religious practices (Barber 1991: 230) that was at first a multi-colored cloth, but by the New Kingdom, was exclusively a red cloth. Kermes red made an early appearance in Europe as a dye and was identified as one of the pigments used in fibers found in a Neolithic cave burial at Adaouste, France (Barber 1991: 224). Depictions of red clothing appear in pictorial representations of merchants in Egyptian wall paintings, such as those at Beni Hasan, ca 1890 BC (Kanawati et al. 2010), and in multi-colored costumes from frescoes painted in the mid-2<sup>nd</sup> millennium BC in the Aegean (Barber 1991: 224).

### ***C. The Archaeological Evidence for Madder***

Because ancient cultivated madder has become naturalized in many parts of the Old World, it has been difficult to ascertain its origins. *R. tinctorum* is thought to be native to South or Southeastern Europe and the Mediterranean area (Derkson 2001: 2, Cardon 2007: 108), or possibly southwest and central Asia (Zohary 2012: 167), and it was domesticated along with many other dye plants in those areas in ancient prehistory (Zohary 2012: 167). Its usage in

ancient times in Egypt and the Levant have been noted in archaeological studies and historical writings. Madder's introduction into Europe is also a matter of debate. There are differing ideas on the timeline for *R. tinctorum*'s introduction into Europe; these range from possibly the Late Neolithic to the Middle Ages. There is enough evidence at this point to strongly suggest that madder was in use as a dye plant in Europe by at least the Late Bronze Age, as dye analyses of textiles from the site of Hallstatt attest (Hofmann-De Keijzer et al. 2013: 147). Madder cultivation appears to have appeared at a later date; Roman written sources attest to its cultivation in Gaul (Walton 1988: 154-155; Wild 1970: 81) although there is not much supporting evidence for this.

The earliest evidence for madder dye comes from the Egyptians, Greeks and Romans (Walton Rogers 2003: 26). Madder seems to have been the most common red dye in the classical world; there is also evidence of kermes, Polish cochineal and Armenian cochineal from near Mt. Ararat, but these dyes are less common (Walton Rogers 2003: 27). While there is textual evidence for madder or madder-type plants as medicinal cures (though no evidence of its efficacy), and even in cosmetics (Van Elslande et al. 2008: 1879), the primary source for evidence of madder is in ancient textiles. Textiles are a technological and economic necessity in all cultures, and textile manufacture in prehistoric Europe and the Near East was certainly a household enterprise although some prestige textiles would have been the product of specialists and traded over long distances. These types of elite textiles could include prestige dyes such as Tyrian purple or kermes red. Madder-dyed cloth probably fit under both categories—as a household good, and as a prestige textile. The prestige value of madder may have relied more on the technique employed and the skill of the dyer rather than in the scarcity or value of the dye plant itself, as the experimental section of this thesis will attempt to demonstrate.

#### ***D. The European Neolithic (before 2000 BC)***

Linen was the most prevalent textile of the European Neolithic and worked hides are attested in the British Isles around the same time (Ryder 1981: 224). There is no known evidence of wool at this point, as sheep fibers could not yet be spun into yarn (Ryder 1981: 224). Some early attempts at using wool in textiles may have been identified in Denmark, as sheep kemps have been found in early textiles there (Ryder 1981: 224). Evidence for madder cultivation could potentially be attested by the presence of high concentrations of *Rubiaceae* pollen at certain sites. This has indeed been noted in some instances, such as at the lake dwelling site of Charavines (Cardon 1998: 3). It is quite likely that this accumulation of pollen was the result of a dye crop, but it may have been one of several species of dye plants that are related to *R. tinctorum*, as the pollen morphology of these plants is very similar (Huysman et al. 2003: 219). Textiles evidence for dyes is scanty from this time, although there is the possibility that two well-preserved linen tunics from a Copper Age settlement in Lorca, Spain may have been dyed red (Alfaro 1992: 27).

True woven textiles as clothing begin to appear in the archaeological record in the Neolithic (Bender Jørgensen 2003: 54-55). Textiles from this time period are generally only found in permanently frozen sites such as in the Pazyryk kurgans, or in permanently waterlogged sites, such as at lake dwellings, coastlines or in wells (Gleba et al. 2012: 2; Wild 2003: 9). Woad usage can be traced back to the European Neolithic (Zech-Matterne and LaCoste 2010: 137), probably because it is one of the few vegetal dyes that will dye linen well. Along with the kermes insects mentioned above, blue-dyed fibers were also found at the l'Adaouste Cave at

Bouches-du Rhône in France (Cardon 2003: 288). Dyeing with indigo was apparently accomplished early in European prehistory (Kramell et al. 2014: 215). There are arguments that the elaborately woven brocade cloths of the Late Neolithic cultures in Switzerland and Germany must have been colored for the patterns to be seen. It has been suggested that some “brocade” linen cloths from this time must have once been colored and red colorants were cited for three linen textile finds from Robenhausen (Barber 1991: 224). Significant concentrations of madder pollen (*Rubia sp.*) were found by A. Emery-Barbier at Charavines, Lake of Paladru, in France (Cardon 1998: 5). The first village was occupied for a short time from 2668-2580 BC, and flax seeds and microdenticulate tools were found, but no animal fibers (Cardon 1998: 3). Dwarf elder seeds were also found, suspected to be a source of dark purple dye (Cardon 1998: 5). This settlement at Charavines may also have had a textile workshop, and later settlements were known for their “fine and rare” textiles (Cardon 1998: 18).

#### ***E. The Bronze Age (2000 BC-700 BC)***

Dyeing has been carried out in Europe certainly since the Bronze Age, at first with mineral stains and plant pigments (Gleba 2012: 17). These pigments, however, were not chemically bound to the fibers and therefore not long-lasting (Gleba 201: 18). Dyeing methods, such as the use of vat dyes and mordant dyes, were a later development. There is evidence for the circulation or trade of textile fibers in Bronze Age northern Europe (Bergfjord et al. 2012: 1), but little evidence for dyed textiles until the late Bronze Age with the finds from Hallstatt. Bronze Age wool is less hairy than the coats of wild sheep and is actually identical to that of Soay sheep (Ryder 1981: 225 -6). As far as wool types are concerned, Bronze Age wools in many parts of Northern Europe tend to be browner than later Iron Age wools, when white wool (and more often dyed wool) becomes more common. Color variation in Bronze Age woolen textiles often

is more dependent on natural wool coloration than on dyes (Walton 1988: 146). While dyed textiles almost certainly existed in Greece from the Bronze Age on, and madder was cultivated in the eastern areas of the Classical world by the Middle Bronze Age (Gleba 2012: 19), very few textiles from this region have been preserved (Wild 2003: 70).

While rare, Bronze Age dyed textiles have been found. Dyed textiles were identified at a Bronze Age lake dwelling site in Switzerland, but the dye sources have so far not been determined (Walton Rogers 2003: 25). Several examples of Bronze Age dyed textiles have also been identified in burials and weapons deposits, such as red-dyed cords at Bad Frankenhausen, in Saxony-Anhalt, Germany (Möller-Wiering 2011: 121). There are also reports of two dyed cloths from the Middle Bronze Age: pieces of red wool, a fancy belt and a net, from Roswinkel, Holland (Barber 1991: 224). The textiles from the Hallstatt salt mines, however, provide the largest cache of dyed textiles from this period.

The Hallstatt salt mines provided ideal conditions for the preservation of textiles and other organics. Textile finds from this region span a long period of time, from the Middle Bronze Age (1600-1200BC) into the Early Iron Age (Gleba et al. 2012: 19). Clothing, tools, basketry and even food left behind by Bronze Age and Early Iron Age salt miners were found in exceptionally good states of preservation. The combination of darkness, salt, low humidity and the fact that these areas remained undisturbed for centuries all combined to provide a wealth of archaeological materials. The textiles found still retained their bright colors and patterns (Fig. 2.10). Textiles from the Hallstatt graves are more poorly preserved than those found in the mines and colors and patterns are less clear (Grömer 2011: 15). The main fiber from Hallstatt is wool, with some horsehair as weft for bands (Grömer 2011: 15).



Fig. 2.10. Hallstatt textiles showing the variety of color and patterns. (Image: Natural History Museum Vienna).

Dye analyses were performed on two separate occasions: the first of these detected woad dye, weld and kermes on Hallstatt textiles but madder was not detected, nor were any textiles noted as being red in color (Joosten et al. 2006: 172-173; Hofmann de-Keijzer et al. 2013: 147). A more recent analysis was carried out in 2013 using HPLC-PDA (photo diode array) with 79 samples from 60 textile finds from both the Late Bronze Age and the Early Iron Age (Hofmann de-Keijzer et al. 2013: 137). This new analysis did identify purpurin as a dye compound, probably from a bedstraw or wild madder (*R. peregrina*), in several yellow or dark textiles (Hofmann de-Keijzer et al. 2013: 147). This madder or madder-type dye seems to have been combined with several other dyes to augment or darken other colors, rather than being used alone for a red hue (Hofmann de-Keijzer et al. 2013: 147). This is to date the earliest confirmed known usage of madder-type dyes in European textiles.

#### ***F. The Early Iron Age (700 BC-450 BC)***

It is in the Early Iron Age that dyed textiles begin to appear more frequently in the European archaeological record. The increase in dyed goods may have accompanied an increase

in fleecier, whiter wool as sheep breeding advanced (Ryder 1981: 225). Sheep shearing was also an Iron Age innovation, along with breeds of sheep that exhibited continuous hair growth, rather than those that molted (Ryder 1981: 225). Of course, wools were different in different geographical regions, and the variety of wool types can help suggest a source for the textile in question. At this time in Europe, fashions also began to change, as trousers were introduced into Europe by the Scythians in the 6<sup>th</sup> c. BC (Beck et al. 2014: 225; Munksgaard 1981: 41). The precursors to checked textiles also appear at this time. In Europe, plant and insect dyes were in general use by the Early Iron Age, as the textiles from Hallstatt demonstrate. Another significant textile find from this period is the princely grave at Hochdorf, where checked textiles dyed with kermes and woad were found (Walton 1999: 242-243; Walton Rogers 2003: 25). The Hochdorf textiles were not a promising find visually, as most were a brown color. After analysis of 49 textile samples via HPLC, however, it was discovered that the dyes had preserved quite well and the results “put color back in the prince’s clothes”: dye analysis revealed a scarlet kermes red and woad blue mixed with oak galls or another tannin to appear a bluish-black, but no evidence of madder (Walton 1999: 240-241).

While dyed textiles were certainly present in most of Europe by the Early Iron Age, it wasn’t until the Late Iron Age that dyes came to Scandinavia (Frei et al. 2017: 652). Checkered and striped textiles could be dyed like the Hochdorf example, but in non-elite contexts, checked fabrics were often produced by variations in natural wool colorings, such as those from Kroghs Mølle and Haraldskaer Mose (Walton 1988: 146). Checked fabrics were common in northern European textiles, and it seems these were regarded as a northern fashion (Wild 1981: 16, 2003: 88). Checked fabrics in different colors were most common (Wild 1981: 16), although checked textiles could be produced by variegating different weaves as well. There is even evidence of

red and white patterned textiles found at Gordion in Anatolia dating to ca. 700 BC (Barber 1991: 224). A confirmed presence of alizarin in Early Iron Age textiles is rare. Alizarin, a chemical indication of *R. tinctorum*, shows up in the Early Iron Age Hallstatt textiles, as do garments dyed red (Hofmann de-Keijzer et al. 2013). However, it has been noted that chemical analysis cannot necessarily determine the species of madder or rule out bedstraws as the source of a red dye; the variability of dye methods and of the chemical composition of dye plants from different soils and regions can affect their chemistries (Hofmann de-Keijzer et al. 2013: 148-149).

### ***G. La Tène Period (450-400 BC to Roman Conquest)***

Most grave finds from the La Tène Period provide little evidence for color, patterns or dyes because the fibers are usually mineralized (Grömer 2011: 18). Woad was found as part of a plant assemblage in a storage pit at a site near Roissy north of Paris dated to the late Iron Age (Zech-Matterne and Lacoste 2010: 137). Three charred and mineralized woad seeds were also found at Hochdorf (Zech-Matterne and Lacoste 2010: 138). Both finds seem to give a strong indication of local woad cultivation by this time (Zech-Matterne and Lacoste 2010: 139).

Exceptions to this generally poor preservation have been found, however, and there is evidence of dyes, including red dyes, in La Tène textiles. A La Tène grave at Nove Zamky in Slovakia contained linen cloth with red wool embroidery (Bender Jørgensen 2003: 70). Brightly colored Etruscan garments are also depicted in tomb paintings from this time period. The Dürrenberg, another salt mining site in Austria, also preserved Iron Age textiles embedded in salt deposits. These, again, showed great preservation and retained their bright colors of yellow, red and blue (Grömer 2011: 15). Here, though, the main fiber found was predominantly flax rather than wool (Grömer 2011: 15), and it is unclear whether the dyes on the Dürrenberg textiles have been chemically analyzed.

There has been some recent dye analysis of French archaeological finds from this time. Although many of the Martres-de-Veyre burial textiles were lost due to poor conservation and destruction of the Rheims archaeological warehouse in WWI, some excellently preserved woolens from the 2<sup>nd</sup> c. AD site have survived (Desrosiers and Lorquin 1998: 54-5). Carbonic acid in the soil is credited with the exceptional preservation of organics in this region. Along with several small fragments, a red and black tunic, a fringed sash, and a distaff wound with colored wool were analyzed for dye content (Nowik et al. 2005: 838-841). Red fibers containing both purpurin and alizarin were identified amongst the textiles. The tunic was dyed red with purpurin, and alizarin was the primary dye detected on one fragment of a dark brown tabby (Nowik et al. 2005: 839). The bronze distaff found in the burial of a young girl contained yarns or ribbons dyed with a combination of purpurin, alizarin and indigotin dyes (Nowik et al. 2005: 837, 840). Other textile finds have suggested the presence of dyes, but as there has been no analysis done on these materials, it is presumptuous to assume that madder was present. A Celtic Iron Age Gallic coat, from Rønberj, in Jutland, has been described as red and is presumed to be madder-dyed (Munksgaard 1981: 42). Textile fragments found under a bronze vessel in a grave at Neudorf-Bornstein, dating from the 2<sup>nd</sup> half of 3<sup>rd</sup> c. AD, contained polychrome fibers of red and blue (Möller-Wiering 2011: 123-4).

Textiles have been found in over 350 Danish Iron Age graves and occasionally with bog bodies (Bender Jørgensen 1981: 26). Danish bog bodies date to the late Bronze Age and the Pre-Roman Iron Age, between 650 BC and AD 50 (Bender Jørgensen 1981: 29). Again, dye analyses are scarce, or, if available, reveal that the fibers were undyed or colored with local dyestuffs as in the case of the garments of Huldremose Woman (Frei et al. 2009: 1966). Several weapons deposits have also been recovered from bogs originating from this time period and

stretching into the Roman or Migration Period. These deposit sites were often used for long periods of time, and a wide assortment of weapons and other items have been found in them. One such bog find at Nydam in southern Jutland, contained weapons and four boats (Möller-Wiering 2011: 81). The chemistry of this bog is such that iron and vegetal matter preserves, but wool does not preserve well (Möller-Wiering 2011: 81). Many weapons were found here, along with four boats dating to at least AD 190, one of which had been ritually-destroyed (Möller-Wiering 2011: 81). Textiles were hardly mentioned in the original excavation reports from the 19<sup>th</sup> century, but several woolen fabrics were found wrapping spearheads and shields and rolled up in bundles for sacrifice (Möller-Wiering 2011: 82, 96-97). Several weapons and shields appear to have been ritually killed as well (Möller-Wiering 2011: 89). Woolen textiles were also found in the caulking materials of the boats (Möller-Wiering 2011: 85). There is no mention of dyed textiles in these finds, but later discoveries of a very similar nature would yield dyed textiles.

#### ***H. Roman Period (Roman Conquest to AD 410)***

In Roman times, the cloth culture changed little from the preceding La Tène period (Grömer 2011: 19). Mass production of textiles in regions such as Austria did not start with the Romans, but the Romans found these “structures and this organization already in place and used them” (Grömer 2011: 19). That being said, the expansion of the Roman Empire brought textile techniques from Eastern Europe, the Greek world, and the British Isles into contact with each other as technology and people traveled through this vast new Empire. The introduction of tapestry production into Europe from Greece was an example of this type of change (Wild 1981: 15). Access to new techniques and materials from geographically and culturally distant areas did have an effect on textiles from this time and it appears to be difficult at times to distinguish trade

goods from new local productions. Textile dyes also appear to have undergone a certain amount of standardization at this time. From Roman times through the Anglo-Saxon and Viking eras, it has been noted that while local dye sources remained available, textiles from these later times were most often dyed with a small group of well-known dyes, such as dyer's madder (Bender Jørgensen and Walton 1986: 178).

Part of the new wider world of textiles was the introduction of silks into the Roman Empire. Han silks were brought to the West along the fabled Silk Road, and trade was mediated by the Parthian Empire in what is now northern Iraq (Wild 2003: 108). Silk was highly regarded by the Romans (Wild 2003: 81), and according to Pliny (*Naturalis Historia* XXII.2), became a craze associated with the decadence of Roman women in their almost transparent silk garments. An entry in the *Edict of Diocletus* concerning the price of a loom for silk damask (Chapter XII, line 32A) suggests that silk was eventually produced in the Roman Empire (Wild 1981: 17). Roman damask silk clothing was the "height of luxury" and quite expensive in the 4<sup>th</sup> century AD (Wild 1981: 18-9). A variety of textiles, including silk, from different archaeological contexts have been found dating to this time period. There are textiles from burials, from military settlements, and from weapons caches. Some have been analyzed for dyes, or dyes have been observed on them, and it is these that will be the focus of this section.

During Roman times, it appears that madder was cultivated in Gaul, as Pliny notes that the province "abounds with it" (Wild 1970: 81). Alum and iron mordants were also probably available in Gaul at this time (Wild 1970: 81). A wide variety of indigenous wild plants were used as dyes in northern Europe at this time, some of which have yet to be identified (Wild 1970: 81). Textiles dyed with madder have been found at Vindolanda, a Roman fort near Hadrian's Wall (Taylor 1999: 41). The Vindolanda site contains one of the largest collections of wool

textiles from Roman times (Ryder 1981: 226; Wild 1992: 66). Not only are well-preserved textiles present, but documents that mention textiles have been found as well (Wild 1992: 66). Textiles dyed with bedstraw or other madder-type plants were also identified at Vindolanda, including a piece of wool dyed with orchil purple (Taylor 1999: 41). It is assumed that madder was an imported item in these areas, while the madder-type dyes were locally gathered (Taylor 1990: 41). At Vindonissa, another Roman military camp in Switzerland, scraps of various dyed fibers were found that were probably stuffing for a bolster (Wild 1970: 120-121).

In Roman popular culture, colors had special significance. As has been mentioned previously, purple was legislated and restricted to the emperor until late in Roman times. Analysis of dyed textiles and residue analysis of a Roman dyeworks at Athribis in Egypt suggest that red and blue were the most popular colors in Roman society (Wild 2003: 115-116), although it is worth noting that these can be combined to make purple. The red and blue dyes found at this site were madder and woad respectively (Wild 2003: 116). While madder was a southern dye plants, and its presence is not surprising in Rome, its usage was still sporadic in northern Europe. Some of the most famous textile finds from this time period, while red-colored, were not dyed with madder. One of the best-known is the 1<sup>st</sup>-century AD woman's clothing found in a burial at Lønne Hede in western Jutland in 1969 (Bender Jørgensen 1988: 109). Enough of these textile remains have survived to reconstruct the dress and shawl. Dye analysis revealed that the textiles were comprised of a shawl, skirt, blouse and head scarf. These were primarily blue with a red or orange stripe or a checked design; the blue was from indigo but the red/orange dye remained unidentified (Bender Jørgensen 2003: 94). Vegetal dyes like woad and madder made a late entrance into Scandinavia, roughly around the beginning of the Common Era (Bender Jørgensen 2003: 94). Woad was apparently cultivated and certainly in use as a dye plant. Dyer's

madder would have been imported, but wild madder is native to Scandinavia and could have been employed as a dye (Bender Jørgensen 2003: 96). The Lonne hede textiles, found in western Jutland and dated to the Early Roman Iron Age (1<sup>st</sup>-2<sup>nd</sup> c. AD) are believed to be native products, and the dye analysis supports this (Walton 1988: 146). Other blue textiles from high status graves in the area have also been discovered; these have been confirmed to have been dyed a deep blue and may have had a red and blue tablet weave trim (Krag 1992: 76-77).

Madder-type dye has been identified in weapons caches in some Roman Age Danish and German peat bogs. Bog deposit sites such as Thorsberg in Germany, Vimose and Illerup Ådal in Denmark are all known as weapons deposit sites and are some of the largest known to date. Weapons were consigned to a ritual (votive) disposal in these bogs, after being damaged not in battle but as a part of the ritual (Möller-Wiering 2011: 1-2). At all three of these sites, weapons were wrapped with care in multiple layers of cloth; weapons and shields were often ritually killed, possibly with the textile wrapped around them (Möller-Wiering 2011: 26). At Illerup Ådal, 12-14 textile fragments that appeared red and of high quality were noted, and seemed to be associated with the weapons, as some red textiles were wrapped around several spearheads, and a red tablet weave was attached to a shield boss (Möller-Wiering 2011: 10). No other dyed hues appeared on any other textiles from these sites (Möller-Wiering 2011: 10). Textiles from the Vimose deposit appeared to be undyed (Möller-Wiering 2011: 32).

The bog chemistry at the Thorsberg site was conducive to excellent wool preservation, and the textiles from this site survived well enough to be recognizable items rather than fragments (Möller-Wiering 2011: 40). Very few weapons were preserved at this site, but the few that survived were all ritually damaged. The textiles cannot therefore be associated with weapons, nor can they be precisely dated, although it has been suggested that they are Roman in

origin (Möller-Wiering 2011: 41; Vanden Burghe et al. 2013: 101). A tunic, two fringed cloaks and a pair of pants were found in an exceptional state of preservation and also appear to be ritually damaged; the interior of the tunic was still pigmented a vibrant reddish purple and the garment was slashed in several places (Möller-Wiering 2011: 47-48). Dye analysis revealed that this tunic had been dyed with both wild madder and woad (Möller-Wiering 2011: 47-48; Vanden Burghe and Möller-Wiering 2013: 103). Torn woolen textiles were also found rolled up in a lump and deposited in the bog. These woolens do not appear to be wrapping anything and may have been sacrifices in themselves. However, as iron did not preserve in this bog environment, this is not a certainty (Möller-Wiering 2011: 77). Some chemical dye analyses have now also been carried out on the two fringed cloaks at Thorsberg, confirming the presence of a madder-type dye with purpurin, along with woad, weld and dyer's greenweed (Vanden Burghe and Möller-Wiering 2013: 101).

Late Roman/Migration Period textiles have also been found in rich graves in Europe, with madder identified on the fibers, such as a princely burial at Snartemo, a 5-6<sup>th</sup> c. AD Norwegian site, which contained tablet weaves and textiles of uncertain origins, and what is thought to be locally produced madder dyed plied yarn twill (Walton 1988: 145, 148). Veiem, another rich Norwegian grave from the same period, also contained madder-dyed tablet weaves and a third wealthy Norwegian grave at Evebø/Eide, also from the same period, contained striped textiles in madder red and indigo blue (Nockert 1991: 49-52). Alizarin was present on samples from tablet weaves at all three sites (Walton 1988: 154). It is assumed that this was an imported dyestuff, as the plants would only have been hardy in sheltered areas of Scandinavia and would not have been cultivated on a large scale (Walton 1988: 155). At this point, dyer's madder seems to be found only in rich Scandinavian graves, so perhaps it was an imported dye, or the

textiles were imported already dyed. Another madder-type dye found on non-elite Danish textiles may be a *Galium* or *Asperula spp.*, which is native to the region (Walton 1988: 155).

The textiles at the Evebø/Eide site comprise more than just tablet weaves, and the dye evidence from this late site comprises the last textile example in this research. Evebø/Eide is one of the more spectacular and well-known finds from the Late Roman/Migration Period, known as the “chieftain’s costume” (Bente 1981: 63). Well preserved organics were found in a ca. 5<sup>th</sup>-century AD burial mound erected over a grave lined with birch bark and stone slabs containing the remains of one male of high status, lying on a bear skin and covered with an undyed woolen blanket (Bente 1981: 64-7). Weapons and feasting equipment were present in the burial, as were remnants of several spectacular textiles (Bente 1981: 65-6). As this find was made in 1889, some of the textiles and adornment deteriorated after excavation, and some of the characteristics of the fabrics were lost (Bente 1981: 65). Enough information and material have survived, however, to conclude that the man was dressed in an outer tunic, an inner tunic, trousers, and a mantle, all of wool twill (Bente 1981: 66). The inner tunic was crimson, dyed with kermesic acid (Bender Jørgensen 2003: 135). The outer tunic was blue, with a brocaded tablet weaving of lion figures in a frieze pattern (Fig. 2.11a), in red and black (Bender Jørgensen 2003: 132-134). The mantle (Fig. 2.11b) was fringed and a bright red, dyed with madder, had blue and yellow stripes and may have had checked areas as well (Bender Jørgensen 2003: 135; Bente 1981: 68; Pedersen 1981: 78). The trousers were a red and blue checked woolen (Bender Jørgensen 2003: 132-4; Bente 1981: 69; Pedersen 1981: 80). A mantle from the Snartemo burial also was of a deep red color and had tablet-woven bands (Bente 1981: 68). It is speculated that both the madder and kermes dyestuffs would have been imported to Norway in this period (Bender Jørgensen 2003: 135).



Fig. 2.11a-b. Evebø/Eide textiles. Fig. 2.10a. Left: the animal frieze on a tablet-woven tablet band. Fig. 2.10b. Right: the madder-dyed mantle. (Images: Universitetsmuseet Bergen)

### ***I. Historical Written Evidence for Madder***

Several writers from the Classical world have cited madder in different historical, and botanical contexts. Table 1.1 from Chapter 1 lists several of these early sources for madder usage. The earliest written record for madder is from the Greek historian Herodotus (*Histories* 4, 189), who described the madder-dyed goat hides worn by fashionable Libyan women while traveling in Egypt. Centuries later, Strabo (*Geographica* 13.4.14), another peripatetic historian, would note the especially vivid madder dyes produced at Hierapolis. These reds apparently rivaled murex purple in hue, a color Strabo felt was due to the quality of the water there. Madder also is mentioned as a medicinal plant in the botanical and pharmaceutical works of Hippocrates, Dioscurides, and Theophrastus between the 5<sup>th</sup> and 3<sup>rd</sup> centuries BC (Fuchs and Oltrage 2013: 29). In his *Historia plantarum*, Theophrastus mentions that some plants are used in dyeing and dye plants are also mentioned in relation to paint pigments in a treatise on architecture by Vitruvius from the 1<sup>st</sup> c. BC (Fuchs and Oltrage 2013: 29). Dioscurides is the first source to use the word *erythrodanon* for madder root (Faber 1938: 287). From these early rather general mentions of madder, it is clear that its use as a colorant on a variety of substrates was known, and its variability as a dye had been exploited as well. Written sources that deal with the art of dyeing in a thorough manner are rare, or have been lost, such as the supposed four-volume

treatise on dyes that was written by Democritus that now survives only in fragments in other later texts (Faber 1938: 289). Some of the important and detailed evidence for dye sources and methods comes from the two alchemical papyri, the *Papyrus Holmiensis* and the *Papyrus Leidensis*, described in Section 2 of this chapter.

### ***J. Evidence for Dyeing as a Profession***

Very little evidence of dye production survives in the archaeological record. Most of the evidence for madder dyeing workshops comes from Roman times or later (Tomlinson 1985: 270-272), or is located in the Mediterranean region or in Egypt (Koh et al. 2016: 537; Leix 1938: 423). Given that this equipment was probably very sturdy and often of metal or fired material, it has been noted that there should perhaps be more surviving evidence than there is (Wild 1970: 81). Dyeing tools in ancient workshops would include a source or storage area for water, pots or vats for dyeing, possibly grinders or mortar and pestles for grinding dyes or other compounds. Pollen at these sites may also provide evidence of dyeing (Strand and Nosch 2015: 56). There is some written and pictorial evidence for professional dyers in Rome and the dye workshop at Pompeii has become the type-site for Roman dyeing (Wild 2003: 91). The dyer in the ancient world was an “empirical chemist who was prominent in urban industrial life, either as a free entrepreneur or slave, and was respected for his arcane skills” (Wild 2003: 114). Roman dyeing was done after fulling, but before spinning, as the fibers needed to be scoured of their natural oils before they could be successfully dyed and this was accomplished with fuller’s earth or putrefied urine (Wild 2003: 80, 91). The dyer’s workshop at Pompeii contained several stone-built boilers, stone and lead vats and a small furnace (Wild 2003: 77, 80).

The Roman Era provides us with the most complete picture of textile production. The evidence for textile technology in Europe suggests a long slow trend toward standardization, as the specialized twills of Hallstatt gave way to the tabbies of La Tène Dürrenberg (Wild 2013: xv). Dye practices may have followed this trend, at least during the Roman Empire. In Roman times, wool was probably dyed in the fleece (Wild 1970: 80) and then spun. Dyeing took place in workshops and was performed by professional specialists (Loven 1998: 74). The Latin word *tinctor* is a general term for dyer, an *infector* dyed new fleece, while an *offector* redyed old clothes (Loven 1998: 74). Evidence suggests this was an all-male profession. Roman dyers specialized in certain colors and even different hues of colors (Loven 1998: 75). Plautus refers to red-dyers as *flammarii* but there is no epigraphic evidence to support the use of this term. Only the *purpurarius*, the purple dyer, is a confirmed trade with a higher status that was performed by free citizens. There is one documented instance of a female *purpuraria*, Vicaria Creste, of freed status, whose name and occupation appear on a funerary inscription from Rome (Loven 1998: 75). Women do not seem to have been any other kind of dyer. In other parts of the ancient world, dyeing on a small or household scale was a part of domestic textile production. Textile workshops in Greece were often associated with households, and dyeing could have been done in those contexts (Strand and Nosch 2015: 373). This would indeed have to have been a smaller scale enterprise, as dyeing generally involved large amounts of fairly noxious ingredients, including blood, dung, and copious amounts of putrefied urine.

#### ***K. Madder Legends and Lore***

Several legends about Celtic madder usage have surfaced on the internet, and it seems appropriate to try to trace the origins of these stories back to a concrete source if possible. While most have proved elusive, one of these tales is that the Celts fed madder root to their horses and

falcons to dye their hooves, teeth and beaks red. The origins of this association with the Celts have proven difficult to trace, but numerous studies and observations of the staining power of madder root go back to the 16th century and the French astrologer and physician Mizaldus, who noted that the bones of cows and sheep turned a bright red when the animals were fed with madder root for "some days" (Du Hamel 1739: 392). The bone-staining properties of madder have been taken up at various points in history since the days of Mizaldus. In the mid-18<sup>th</sup> century, a British surgeon named John Belchier received the Copley Medal for his observations that madder dye on bone was especially concentrated at the site of bone growth (Murphy 2005:3). This observation led to new developments in the study of anatomy that were advanced by other contemporary European physicians.

Henri-Louis Du Hamel du Monceau, an 18th century French physician and botanist, also noted the dying effects of madder on bone, and conducted several experiments on madder's bone-dyeing properties. Du Hamel's inspiration for his experiments, however, did not come from Celtic lore, but from the story of a roast pork dinner of a "surgeon and a callicoe-printer" (the surgeon was possibly Belchier), wherein it was discovered that the bone at the center of the roast was a bright red (Du Hamel 1739: 390). The red pig bone was due to the practice of feeding hogs the mash leftover from the madder dye vats, which was known to dye their bones red. Du Hamel did note, however, that the pig's teeth remained undyed (1739: 391). His own experiments were with various birds, and he did successfully dye the birds' bones, but their beaks and talons remained undyed (1739: 395). Interestingly, the only soft tissues that took up the madder dye were in the lining of the digestive tract and the vitreous lining of the eye, so that the bird's eyes appeared red (1739: 397). Du Hamel also noted that none of the birds subjected to his experiments survived more than a week after being fed madder, which he speculated interfered

with their digestive uptake of both food and water and proved highly toxic to them (1739: 392-5).

The total effect of these madder experiments on these birds indicates that the Celts were probably not feeding madder to their falcons or horses. If the Celts were coloring the hooves, teeth and beaks of their war animals, it is possible that these were topically applied with dye instead. While there are horse burials in Europe, these are rare and often contain only a few horse bones (Heckett 1998: 36-37). Dyed horse bone has not been documented in any of these graves. Raptor remains are even rarer and there is likewise no evidence of dyed raptor bone. Purple-stained human bone has been found in medieval graves (680-1100 AD) near Worcester Cathedral that is not completely understood, but is thought to be the result of fungal processes, and has been noted in human remains from medieval leper colonies (Cole and Waldron 2016: 2). Experiments with synthetic alizarin in the 19th and early 20th centuries would attempt bone dyeing on other animals. This synthetic alizarin was again fed to laboratory animals but with disappointing results—it did not dye bone (Richter 1937: 591). It is possible that alizarin is not the dye compound that is responsible for bone pigmentation, as there are many possible dye compounds in the fresh root. It is also possible that it is a combination of chemicals within the root that contribute to the phenomenon of red animal bones. Medical research has also focused on madder's affinity for bone, and it has potential as a stain to identify malignant growth on bone (Schorr et al. 1959: 410).

#### ***L. Modernity and Madder: The Current State of Madder Usage***

In recent decades, questions have been raised about the safety of synthetic dyes in cosmetics, medicines and food (Simpson et al. 2001: 373). Recent interest in madder and other

vegetable dyes has also increased with the green movement. Dyeing with vegetable dyes never really vanished—artisans worldwide continued to use these plants. As has been demonstrated by the experimental component of this thesis, madder can be raised in a backyard garden and textiles can be dyed in an average kitchen to achieve vibrant color without the use of more toxic methods or materials. While this practice remained consistent on a small scale, it did not necessitate the return to madder production that was seen in Europe just prior to the synthesis of alizarin in 1868. Recently, however, interest in madder production on a far larger scale has resurfaced, allied with the movement toward more organic materials and green production, and madder dyeing is seen at both the small scale and the industrial levels now (Hartl 2003: 19-20). Zeeland madder fields are again in evidence (Cardon 2007: 109) and madder cultivation on a large scale has resumed in many parts of Europe and the Near East. Organic vegetable dye production appears to be similar in many ways to the spice industry—yields and quality appear to be dependent on local climates and agricultural methods (Hartl 2003: 34).

The fact that madder is a plant dye and therefore an eminently renewable dye source has counted as a point in its favor as well. The fate of insect dyes such as kermes is a different one. The habitat for kermes has been disappearing in the Mediterranean, as the wild areas where it had existed are destroyed for development. Kermes insects are now somewhat endangered, and samples of the dried insects are very rare and expensive. Lichen and fungal dyes are also treated as endangered, and dyers are cautioned against using these dyestuffs, or gathering these in the wild. If there is a larger move away from synthetic dyes and back to traditional methods, madder will be at the forefront of the commercially available anthraquinone dyes.

### **CHAPTER 3: METHODS**

The review and synthesis of the existing literature on *R. tinctorum* presented in Chapter 2 was the first phase in ground-truthing claims about madder dye that lay at the core of this thesis. Just as fundamental to this ground-truthing effort was the cultivation and harvesting of this plant combined with an examination of madder dye methods. These goals are encompassed in the experimental component of this research. The combination of literature review and experimental results will hopefully present a clearer picture of *R. tinctorum* and its prehistory.

#### ***Research Questions:***

The data available on the historical, archaeological, and botanical qualities and distribution of *R. tinctorum*, or dyer's madder, were subjected to a qualitative analysis. The research questions outlined in Chapter 1 were tested via a series of experiments in growing and dying various materials with madder in order to ground-truth what is known of the history of this plant and its properties. The experimental component of this research was executed in three phases: 1) madder plants were grown over the course of two seasons; 2) roots were harvested and prepared for dyeing; and 3) various materials attested in archaeological contexts were dyed with madder. These experiments were designed to answer the following core questions:

- 1) Can madder (*R. tinctorum*) be successfully grown in a harsher climate than that found in much of south and central Europe, the putative source of the plants in ancient times? The plant must survive at least one winter to produce seed and to truly develop all the dye characteristics of madder dye.
- 2.) Are two growing seasons long enough to produce material for a good dye bath? Most sources recommend three growing seasons.

- 3) Would the home-grown madder be able to compete with commercially available madder in terms of color and intensity?
- 4) How much of an impact do various mordants have on color intensity? Could an almost true red be achieved?

The literature on experimental approaches to archaeological interpretation is extensive (Ferguson 2010; Mathieu 2002; Millson 2011) and there have been several recent analyses of experimental archaeology not as an artifact of past processualist approaches but as a more integrated study, sensitive to the theoretical debates that have raged within anthropology in the recent past (Koerner 2011: 63). Experimental archaeology offers rich learning opportunities, often at low cost, and with no risk to archaeological materials. The real challenge is to make experimental studies relevant to archaeology by generating useful data from the experiments. Experimental archaeology has had an impact on the field of archaeological textiles as well. Technical experiments on perishable materials, such as testing the tensile strength of ancient cordage, or experimenting with weaves of varying difficulty, have provided a great deal of information on these archaeological items (Jolie and McBrinn 2010: 156-9). In general, however, most experiments with textiles have involved the fibers: spinning, weaving or loom construction as well as the reconstruction of prehistoric clothing (Olofsson 2015: 26-7). As several aspects of textile manufacture have survived into modern times, such as loom construction and tablet weaving, experimental archaeology has often been combined with ethnographic sources. This practice has been subjected to some criticism, as most of the source material—even that coming from long-established traditions—is still far removed from the prehistoric past (Strand 2010: 5, 373). It is still open to debate how well a craftsperson's knowledge fits in with experimental archaeology and how much can be learned from this

approach, but the technical expertise of master craftspeople, especially in textiles, has undoubtedly been valuable in archaeological interpretation (for instance, see Cooke and Christiansen 2005: 71). Textile research in general also tends to be somewhat isolated from the rest of the archaeological community and tends to be less integrated into general archaeological research or the discourse on social, cultural and technological aspects of prehistory (Olofsson 2015: 35). One important aspect of experimental textile research is that it provides an opportunity for hands-on experience with a type of archaeological artifact that is generally off-limits to the student or the general public. Because archaeological textiles are rare, fragile, and most analyses are destructive, the opportunity to interact with them is unavailable to most. Only a few researchers are given the opportunity to work with ancient dyed textiles. For the rest of us, experimental methods provide an *entrée* into the field.

While experimental archaeology has been an important part of archaeological textile research, very little of it has focused on dyes. Most experiments with vegetable dyes have tended to be carried out either by chemists, reenactors, or artisans (Dean 2014; Derkson 2001; Goodwin 1982; Gunderson 2015), rather than archaeologists. When experiments with archaeologically significant dyes have been carried out, they have tended to focus almost exclusively on Tyrian purple and the Mediterranean region (Haubrichs 2005; Ruscillo 2006). The pioneering efforts of Penelope Walton (1988, 1994), recent work by Anna Hartl (2003, 2015a, 2015b) and more recent analysis of the Hallstatt textiles (Hofmann-de Keijzer et al. 2013) are exceptions, and have helped forge a new path into the nature of other dye sources, including temperate European contexts.

My own connection to experimental techniques and textiles does stem in part from a desire to learn more about dyed textiles. My graduate advisor, Dr. Bettina Arnold, once told me

that she was inspired to become an archaeologist when she saw a fingerprint impressed in the clay of a Roman vessel. Experimental archaeology works a little like that for me. It is also experiential, connecting the doer with the craftspeople of the past. As Dana Millson (2011: 3) has pointed out, the experiential aspect of experimental archaeology can “put the humanity back into the ancient people we study”. And while this experiential aspect may be subjective and have limited scientific utility, it is not without value. I regard the subjectivity of this process as a good thing: that subjectivity is what drives the professional and personal passions of many in this field. Archaeology is social as well as a science. By practicing these dyeing methods, I feel a little closer to those ancient dyers and gain confidence and a better understanding of the technology employed.

But as Millson and others have also pointed out, I will forever be entrenched in my own modernity, my habitus, in these experiments. In addition, it is more than time that isolates me in these experiments. As an American, working with European plants, I am isolated by geography as well. I would have preferred to be able to gather fresh oak galls and club mosses for these experiments, but unfortunately, these are not plants indigenous to the Upper Midwest where I live. Throughout these experimental processes, I acutely felt that distance “across the pond”, and my limited context. However, experiments can be seen as opportunities to create new inquiries, and to add new complexities to what had been thought to be givens (Koerner 2011: 78); this is where experimental archaeology can offer exciting new information. The connection to the past is moving, but the connection to future studies on ancient textiles and dye plants is more so. Also, in a sense, isolation by time and geography would not have been unknown obstacles in the European past either. There is evidence that technological processes were reinvented in many times and places in prehistory.

While there have been important recent theoretical contributions to the idea of experimental archaeology, the fundamentals as set forth by Coles (1979) remain an excellent strategy for structuring experiments. Based on recent textile experiments and with several caveats regarding both time and space, I chose to use the basic investigative structure provided by Coles to design the following procedural approach and set of experimental guidelines (1979: 38-42). Coles recommended that an experimental design include the following principles:

- the materials used should be appropriate; that is, they should have been indigenous or readily available to the ancient peoples being researched.
- the methods used to reproduce results should also be appropriate and be within the means of the peoples or time periods being studied.
- modern technology should be avoided (with the exception of modern instrumentation for analysis) and if it is necessary, should not interfere with results.
- the researcher should establish the scope of the project in advance.
- the experiment should be repeatable, and the repeated results compiled.
- researchers will be uncertain whether their methods will succeed and therefore should be ready to improvise with diverse procedures and materials.
- when the experiment is complete and the results suggest particular conclusions, the researchers should not claim to have absolute proof that a prehistoric or historic process occurred a certain way. Corroborative evidence should be employed to increase the degree of probability, but proof should never be assumed.
- the experiment should be assessed: errors should be openly stated, the procedure and materials should be considered in terms of their reliability and plausibility, and the questions asked should be reevaluated.

Building upon these experiment design principles, the three phases of this project were planned using the following deductive approach to test the assumptions below:

1) Madder was a plant that was difficult to grow or could only be grown in certain limited areas of Europe, essentially southern Europe. Because of the nature of this plant, it had to survive at least one winter to grow to maturity as a dye plant.

2) Madder was a plant that could not have been produced on a scale large enough to have been generally available in prehistoric Europe and was probably restricted to certain social groups or regional areas.

3) Madder dye has qualities that would have made it useful mainly in dyeing certain types of textiles (wool rather than plant textiles) and these limitations may have affected its use.

4) Madder requires several growing seasons to produce a fully chemically developed dye. Three seasons is generally thought to be the minimum.

5) Madder requires mordants, which are thought to vary in their efficacy based on the material used.

In all three phases of this experiment, I worked to incorporate Cole's principles along with techniques for raising madder and dyeing with madder that I gathered from a wide variety of sources, both ancient and modern. While the experiment was tailored to a particular set of variables in each phase, a set of back-up strategies was also planned in the event that initial experiments failed. One last word on variables: as has been pointed out by other archaeological researchers, the biggest variable in many experimental archaeological projects is the researchers themselves (Ferguson 2010: 7). A beginning weaver will have very different results from a weaver with years of experience, for example. Archaeologists are also asking different questions of certain processes that might lead to results that differ from what ancient practitioners would

have been aiming at. While I have decades of experience dyeing fabrics, that experience was solely with synthetic dyes until I carried out a biochemistry experiment with woad in 2010 at the University of Wisconsin-Oshkosh. That year I grew a small stand of woad, processed it, and created an indigo dye in laboratory conditions. From that point on, dye plants have held a permanent place in my garden. I do not consider myself to be an expert on vegetable dyes at this point, however, and that is an important qualification to bring to these experiments. However, that woad experiment definitely informed the structure of this component of my research.

## **Phase 1: Growing *R. tinctorum***

### ***A. Cultivation:***

The first phase of the experiment was the successful cultivation of madder in an attempt to discern its cold hardiness as well as its growth habits, fecundity, and ease of cultivation. This was done to test the hypothesis that madder was a plant that would not have been readily available to ancient peoples in certain areas of Europe due to its sensitivity to climatic conditions or to certain social groups due to propagation challenges that might have made it relatively rare. Sources vary on the hardiness of this plant, but the most encouraging suggested that madder could be successfully grown at Plant Hardiness Zone 4, a geographic climatic region where plants can be expected to survive in areas where winter temperatures can reach -30°F (Buchanan 1995: 52-53). See Figures 3.1-3.3 for the geographic distribution of Plant Hardiness Zones globally, as well as Europe and Wisconsin. Temperatures did not reach the Zone 4 lows during the winter in which madder was grown. Madder plants were successfully overwintered in two different growing conditions. Most plants were grown in a raised bed in a somewhat sheltered garden; the climate in this area of Wisconsin is designated as USDA Hardiness Zone 5a and as I live within four city blocks of a large lake, my property may even be a somewhat milder

microclimate in this neighborhood. Oshkosh temperatures for the months of this experiment are presented in Table 4.1 and give a good indication of a typical growing season in this area.

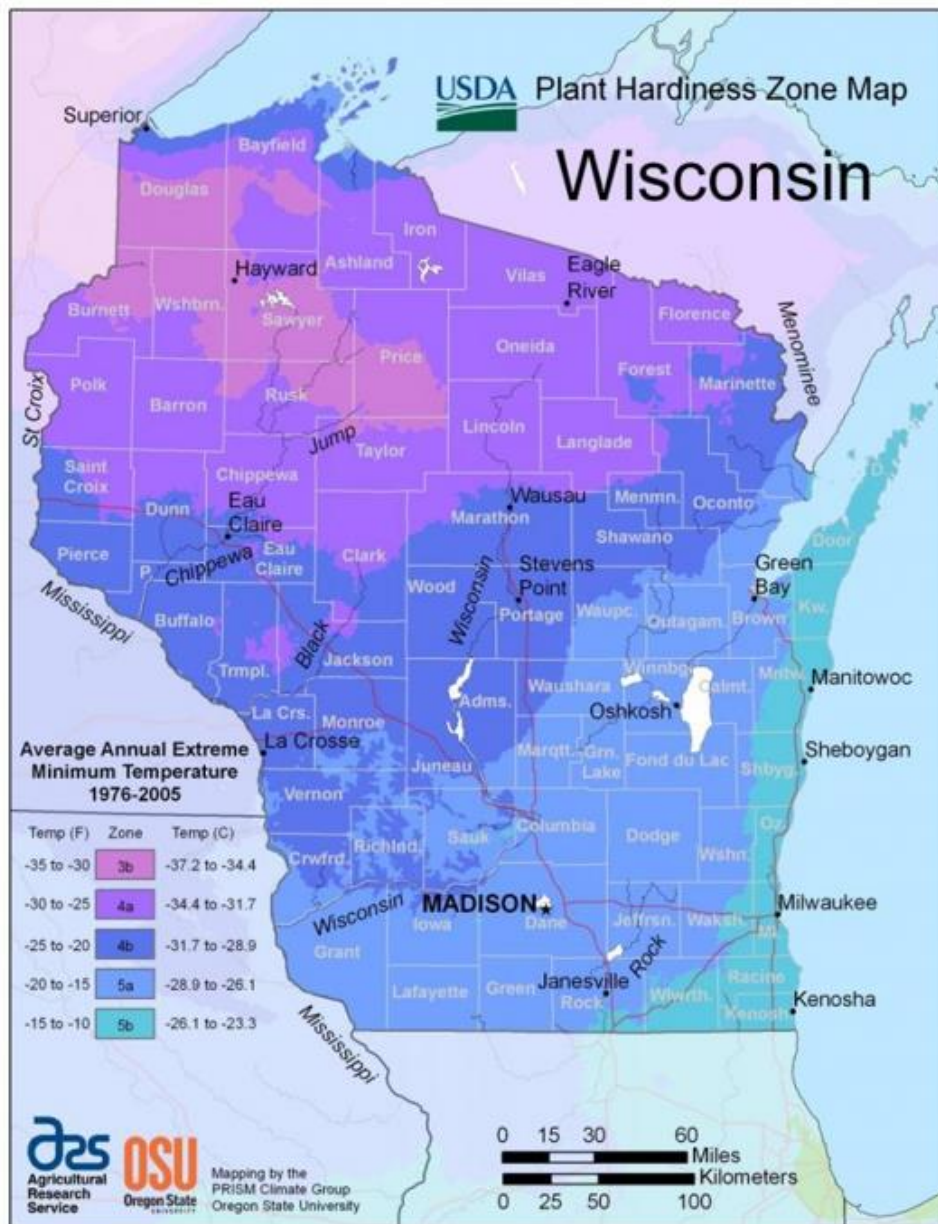


Fig. 3.1. USDA Plant Hardiness Zones for Wisconsin. Image: UW-Extension

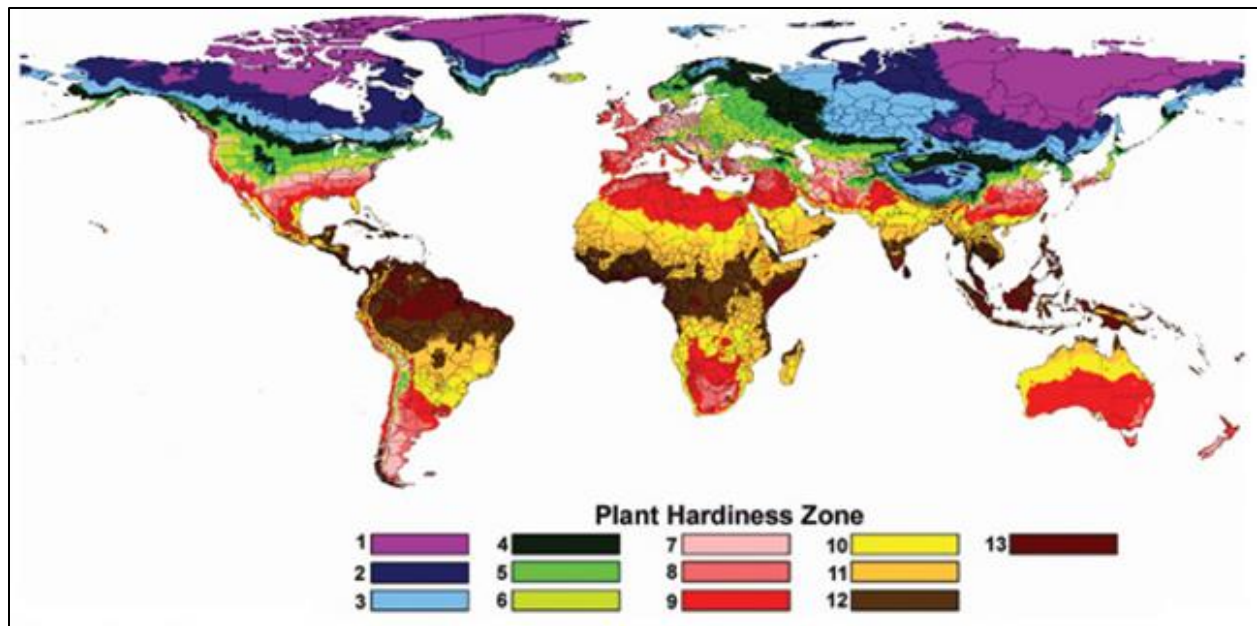


Fig.3.2. Global Plant Hardiness Zones. Image: Scientia Agricola

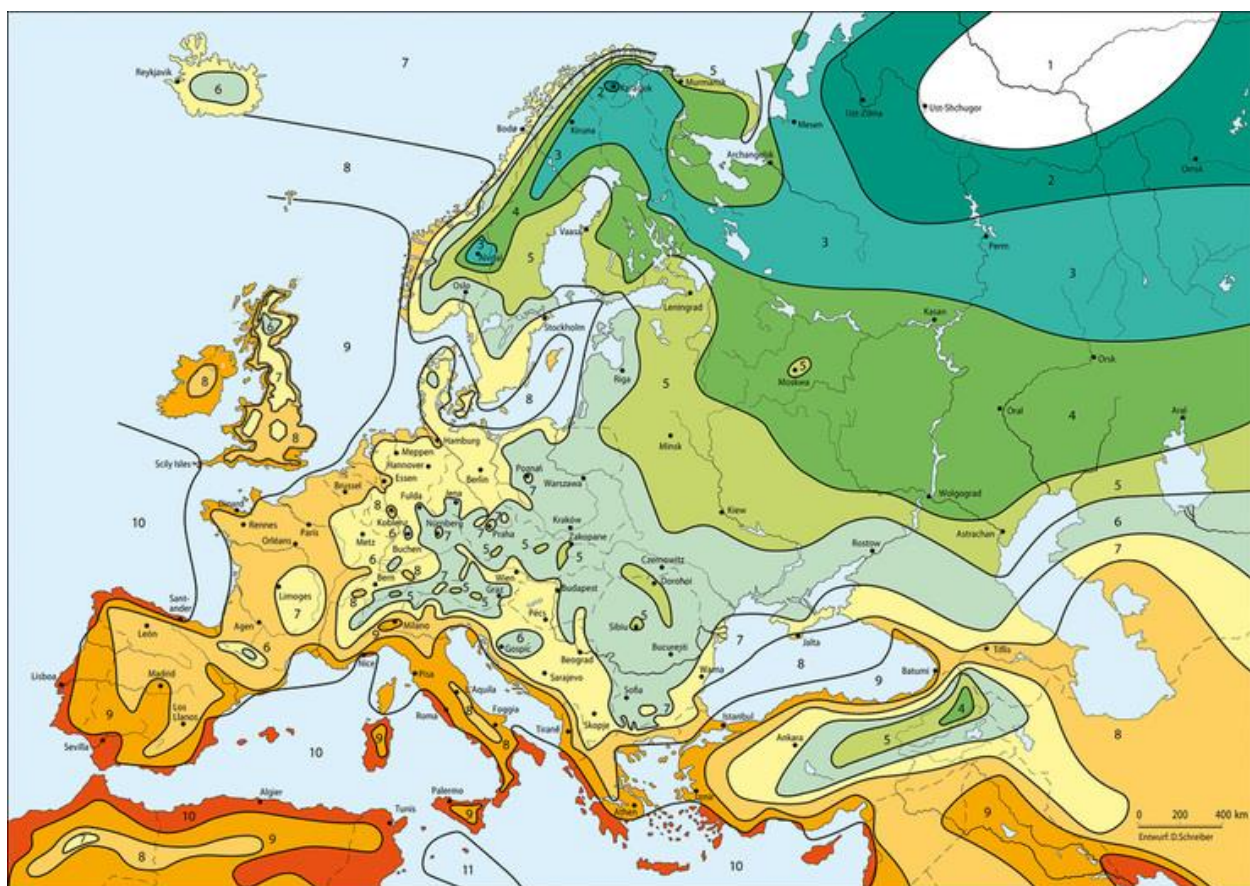


Fig.3.3. Plant Hardiness Zones for Europe. Image: Andreas Bärtels, Enzyklopädie der Gartengehölze

**TABLE 3.1 Monthly Weather Averages for Oshkosh, Wisconsin\***

Month/Year	High Temp (°F)	Ave. Temp (°F)	Low Temp (°F)	Ave. Precip. (inches)	Sea Level Pressure (inches)	Day Length (hours:min)	Days Below Freezing
Apr. 2016	79	43	18	0.95	30.03	12:50	8
May 2016	86	58	30	3.58	29.97	14:15	1
June 2016	90	68	46	4.18	29.93	15:17	0
July 2016	90	72	53	2.82	29.93	15:24	0
Aug. 2016	89	72	53	2.59	29.98	14:31	0
Sept. 2016	88	65	48	5.25	30.03	13:08	0
Oct. 2016	73	53	33	1.46	30.06	11:38	0
Nov. 2016	69	43	21	1.54	30.04	10:09	7
Dec. 2016	43	23	-13	1.49	30.07	9:06	27
Jan. 2017	41	22	-7	2.24	29.99	8:59	22
Feb. 2017	64	30	1	0.96	29.93	9:56	20
Mar. 2017	61	32	6	1.75	30.17	11:15	19
Apr. 2017	75	49	30	4.57	29.95	12:49	1
May 2017	84	55	33	2.29	29.85	14:14	0
June 2017	89	68	44	7.40	29.84	15:17	0
July 2017	87	71	52	2.98	29.73	15:24	0
Aug. 2017	82	66	46	4.13	29.99	14:32	0
Sept. 2017	90	64	42	3.19	30.03	13:08	0
Oct. 2017	79	53	28	2.99	29.94	11:39	2
Nov. 2017	63	35	10	1.03	30.06	10:10	20

\* Weather data provided by Wittman Field Airport, Oshkosh WI online at Weather Underground (wunderground.com).

The sources available indicated that ideally, madder should have at least three growing seasons to reach a large enough size for a sufficient harvest of roots (Dean 2014: 118; Goodwin 1982: 65) and according to older accounts, seven years is optimal for best color (Cardon 2007: 109). However, time constraints forced me to limit the project to just two growing seasons. There is evidence that this is the same amount of growing time that the Dutch gave their madder crops in the 18th and 19th centuries (Goodwin 1982: 65), so there is a precedent for a two-year growth span. Accessibility of plants and seeds was also a challenge. Seed for *R. tinctorum* is not commonly sold by most large seed vendors, and in 2016, I had only located one online source from the United Kingdom for madder seed (Appendix A.1). Since that time, I have located domestic seed sources (Appendix A.2) and several sellers online who sell both madder seed and

dried root (Appendix A.3). Since there are several different species of madder, it was important to acquire the madder variety that was grown in Europe. *R. tinctorum* seed is the most common species of madder available as seed. The only other madder species seed occasionally available for sale is *R. cordifolia*, or Indian madder. Wild madder, *R. peregrina*, which may be the only madder indigenous to Europe, was unavailable as seeds or as dried root.

As a growing environment, I sacrificed a section of the lawn and constructed a raised garden bed specifically for dye plants. The raised bed measured 4 ft. by 6 ft. and was filled about 12 inches deep with topsoil. The soil in East Central Wisconsin is characterized by heavy, iron-rich red clays and is relatively acidic, but as I had created a raised bed with amended soil, the soil used for this project was not typical of the area. The soil characteristics were black loamy soil with some sand and clay and an annual spring addition of organics in the form of composted manure. Plant matter is generally left to cover the soil in the fall and winter, and its breakdown also adds nutrients to the soil. Soil pH was neutral to slightly alkaline (between 7.0 and 7.5), and was ascertained with a soil testing kit (Environmental Concepts Soil Test Kit). The bed was located in a sunny sheltered location. I grew a variety of dye plants in the bed, including madder, woad, weld, ladies' bedstraw and soapwort. As the madder plants thrived and grew larger during the first year, plants had to be moved or harvested to create more growing space for the madder.

Several other madder plants, planted in a commercial potting mix in large ceramic pots, were moved to a sheltered southwest corner outside my house for the winter. After a killing frost in late October 2016, all madder plants died back to the ground and were covered in marsh hay in November as a protective mulch. The marsh hay cover was removed in early May 2017, when all danger of a hard frost had passed and signs of growth were observed. All plants

survived and began to sprout shoots in mid-May (Fig. 3.4a). The potted plants were removed from their pots and replanted in the raised beds at this time, as they appeared to be getting root bound (Fig. 3.4b). (All images of madder plants provided by the author unless otherwise noted.)



Fig. 3.4 a-b. Overwintered Madder. Fig. 3.4a. Left: madder shoots appear at base of plant. Fig.3.4b. Right: rootbound madder plants being transplanted to the raised beds.

The established and newly transplanted madder plants were watered in the early spring as needed, but once plant growth had essentially covered the bed, regular watering was suspended. The soil was also amended with compost in the early spring when new plants were transplanted, but this was the only fertilizer added to the bed that season. Weeding was done regularly, although madder produces such a mat of drooping stems that it functions almost like a ground cover (Fig. 3.5a). Woad and soapwort plants had to be moved or removed, as the madder mat began to creep over its boundaries and cover neighboring rows. During the summer of 2017, the second-year plants flowered and produced seed (Fig. 3.5b-d). Samples of these were collected.



Fig. 3.5a-d. Wisconsin madder plants. Fig.3.5a. Upper left: madder growth sprawls over the raised bed. Fig.3.5b. Upper right: madder flowers and immature seeds in midsummer. Fig.3.5c. Lower left: close-up of madder flowers. Fig.3.5d. Lower right: maturing madder seed darkens by late summer.

### ***B. Harvest:***

The second phase of the experiment was carried out in the summer and early fall of 2017, when the madder plants were harvested. Flowers were collected for SEM pollen images, and seed collected for future plantings. The roots of nine madder plants were collected and dried for dye experiments. There was the potential to use fresh root in dyeing, but as the dyeing portion of the experiment grew more complex, I decided to use dried root for all the dyeing processes. While there are modern dyers who prefer to use fresh madder in dyeing (Roberts 2017), there are also several sources that state the madder color is enhanced by drying, and the desirable chemicals in the plants develop and intensify as a result (Cardon 2007:109). The SEM pollen analysis that was planned did not occur, both because access to an SEM could not be arranged

and because it was questionable how useful madder pollen might be in archaeological analysis (Huysman et al. 2003: 219). There are very few good digital images of madder pollen available, however, and this is definitely an avenue for further research. Madder flowers were saved, therefore, to make them available for later analysis if this project is developed further.

Madder plants were harvested at the end of September, as foliage began to die back and seed began to drop. I harvested some mature seed at this time and allowed some to remain to self-seed. The madder plants at this point were a tangled mass and had to be lifted back to get at the root area (see Fig. 3.6a-b). Nine plants were harvested, which was all the raised bed would allow for, and the roots of each had spread surprisingly far throughout the bed, traveling over into other rows of plants. The soil at this point was very dry because Wisconsin was experiencing a drought summer in 2017, and as a result many of the small, filamentous roots were torn off as the roots were dug out. A dark brown epidermal covering on some of the larger roots was also noted (Fig. 3.7a-b). Most of this covering came off as the roots were washed. One madder plant was selected at random and measured for root spread, at 26 cm. This was compared to a randomly selected plant which was dug up after the first year of growth and later replanted, which was 16.5 cm (Fig. 3.8a-b). The row of 2-year-old madder plants had grown up against one side of the raised bed. It is therefore possible that the root spread could have been even greater if the plants had been allowed to spread in all directions. One madder plant (not used as a representative sample) had sent out a long root that had crossed the raised bed and measured just over 50 cm in length and 1.5 cm in diameter (Fig. 3.9).



Fig. 3.6a-b. Harvesting madder. Fig.3.6a. Left: moving the mass of madder stems to dig the roots out. Soapwort blossoms can be seen in the background. Fig. 3.6b. Right: a close-up of the biomass of madder plants at harvest.



Fig. 3.7a-b. Harvested madder. Fig.3.7a. Left: madder roots in situ. Note the red tuberous root and bright orange pith. Fig.3.7b. Right: the total harvested madder crop of 2-year-old plants, prior to washing.



Fig. 3.8a-b. Comparison of 1-year and 2-year plants. Fig. 3.8a. Left: a washed and cleaned 2-year-old madder specimen. Fig. 3.8b. Right: a washed 1-year-old specimen.



Fig.3.9a-b. Madder root close-ups. Fig.3.9a. Left: a cross-section shot of a larger madder root, showing the bright orange pith inside. Fig.3.9b. Right: the same root, an example of robust root growth.

The 2017 madder crop harvest was washed by a method provided/inspired by WildColours.com, using two 5-gallon plastic buckets. Madder roots were first rinsed with the garden hose, then allowed to soak in rain water in one bucket for 20 minutes, then transferred to the second bucket and soaked again in rainwater, and the process was repeated until the roots were clean (Fig. 3.10a-b). This approach allowed the stubborn mud clumps to sink to the bottom of the buckets, to be dumped out and removed after the madder was transferred. This was also minimally destructive to the madder roots themselves. Once cleaned, the roots were placed in a fiberglass tray to be cut and dried. The samples were cut into longer sections than is generally the case, again using recommendations from WildColours.com (the sections were about 6 cm long), but as all samples were later liquified in a blender, this did not seem to pose too great a problem. Leaving a little length to the roots also allowed some of the root morphology to remain apparent

after drying. The washed and cut madder harvest was weighed and left to dry for the next four months (Fig. 3.11a-b).



Fig. 3.10a-b. Washing madder roots. Fig. 3.10a. Left: madder roots soaking to remove mud. Fig. 3.10b. Right: cleaned madder.



Fig.3.11a-b. Drying madder. Fig.3.11a. Left: the fresh madder harvest from late September 2017. Fig.3.11b. Right: the dried harvest, four months later in January 2018.

The madder harvest was dried in a shaded area in my garage for the first month, then moved indoors as the weather turned colder and allowed to finish drying under a cotton cloth in a

fiberglass tray for another three months. Although there is evidence that heating madder root can improve the quality of madder dye (Cardon 2007: 110) this crop of madder was dried at room temperature (about 23°C).

## **Phase 2: Dye Experiment Structure**

The third phase focused on madder dyeing methods. Because madder is a mordant dye, and there were potentially several mordants available in prehistoric Europe, dyeing methods for this plant could have been quite complex. Aside from madder root sources and mordant type, there were also the variables of dyeing time, temperature, pH and the type of textile fibers used. Experiments were designed to test all of these factors. I selected four textile types that would have been available in prehistoric or Roman Europe: linen, nettle, silk and wool (Barber 1991: 11-31). Along with modern alum, I also selected four mordant types, using materials that also would have been available at various times in prehistoric Europe: club moss, an aluminum mordant; oak galls, a tannic acid mordant; iron liquor, an iron mordant; and soapwort, a natural plant-based detergent (Fig. 3.12) (Dean 2014: 29-30; Faber 1938: 287; Goodwin 1982: 32). Experiments were carried out in my kitchen using equipment that was not specific to prehistoric Europe but based on Coles' specifications would not affect the results and would approximate prehistoric methods. The materials used and the variables of time, temperature and pH were addressed and manipulated in the experiments are described in detail in the sections below.



Fig. 3.12. Mordants. Upper left: club moss (*Lycopodium clavatum*) (Image: Spiridonov-D.) Upper right: stem oak galls (Image: Ian the Green). Lower left: soapwort (*Saponaria officinalis*) in bloom. (Image: Garden Herbs). Lower right: potash alum crystals. (Image: canacopehdl.com.)

### A. Textiles

Several archaeological analyses of ancient textiles mention the fact that very few of them were dyed, and detectable dyes are almost non-existent before the Late Bronze Age (Hoffmann de Keiser 2013a: 23; Walton 1988: 146). In general, textiles made with vegetable fibers were left in a natural state while animal fibers, such as wool or silk, were dyed far more frequently. It has been suggested that this was in part because vegetable fibers do not take dye very well or are at least problematic to dye (Barber 1991: 21; Crowfoot and Davies 1941: 123; Grierson 1986: 40). To test this assumption, a selection of the vegetal fibers available to prehistoric peoples were included in the dye experiments. Fibers such as nettle, linen and silk (which was available in Europe in Roman times) were only available to me as woven fabric, so testing for color was used on these woven fabrics. Wool roving, which was far less expensive than undyed woolen fabric, was used to test dye effectiveness on that material. The wool roving took dye far better

than the other fiber samples, and proved to be superior in illustrating the differences in hue, saturation and the color spectrum that could result from different combinations of variables more effectively than plant textiles.

The mix of raw fibers and woven fabrics was in part a budget decision, as these were the least expensive or most readily available fiber forms. This mix of fiber forms also would have been available to ancient dye baths, depending on the culture. Raw unspun fibers were most likely dyed in Roman times (Wild 1970: 80), but spun fibers may also have been dyed, and the weft of some woven fabrics appears to have been dyed a red color while the warp was a different hue (Nowik et al. 2005: 837). In some instances, dyes were painted onto woven fabrics as well (Barber 1991: 226), and there is evidence for resist-dyeing and printed fabrics (Wild 1970:80), so a dye treatment of fully woven cloth would not have been unknown in prehistoric times either. Barber (1991: 236) has also pointed out that color-fastness may not always have been the goal of textile dyeing, as finished clothing could have been periodically re-dyed in various shades to suit certain rituals or seasons, rather than being permanently dyed one color, as is typical today. Therefore, textiles could have been at various stages of production when dyes were added. All fiber samples in this experiment were dyed with purchased Pakistani madder and tested for color differences in pH, temperature, and duration. These samples were also subjected to several different mordant dye baths to ascertain whether different textile fibers would have responded differently to different chemical treatments. Listed below are the fiber types, and how they were used (Fig. 3.2a-d).



Figure 3.13a-d. Fibers included in experiments. Fig.3.13a. Upper left: wool roving. Fig. 3.13b. Upper right: raw silk fibers. Fig. 3.13c. Lower left: woven nettle fabric. Fig. 3.13d. Lower right: woven linen.

*Linen:* Linen, from the flax plant *Linum usitatissimum*, is a textile that is found very early in the Neolithic lake dwelling sites of the Alpine region in Europe (Maier et al. 2011: 567; Barber 1991: 10), where some of the finest linen textiles ever found were produced (Barber 1991: 15). This fiber has continued to be grown and spun into thread for clothing to the present day. Linen has always been difficult to dye, even in present times: it does not take most dyes well, as the fibers are so hard and impenetrable (Barber 1991: 15). However, there is apparently archaeological evidence for dyed linen, including the red linens of New Kingdom Egypt (Barber 1991: 230). Samples of undyed and untreated linen fabric were obtained from an Etsy source in Hohenstein, Germany (Appendix A.4) that provided linen fabric from the Orsha linen mill in Belarus. The linen fabric was a pale tan color (Munsell 10YR 7/1) (Fig. 3.13d).

*Nettle:* Nettle (*Urtica dioica*, or stinging nettle) was an important textile fiber that was certainly in cultivation by the Late Bronze Age in Europe, and was possibly in use in the Neolithic as well although it is unclear if nettle was cultivated for fibers or food (Barber 1991: 19; Hillbrand 2014: 31, 36). It is likely that nettle was locally exploited as a wild plant for textile production across much of northern Europe, as it is indigenous to that area. Nettle is also known to grow well in disturbed ground, especially ground fertilized by urine, so it was possibly found very near early European settlements (McIntosh 2006: 197). Because it is difficult to differentiate between nettle, flax, and other plant fibers in degraded vegetal textile samples, nettle is often misidentified as flax or other plant fibers, and it is possible that many examples of nettle textiles have been overlooked. As an example of this, several “linens” from the Oseberg ship burial near Tønsberg Norway, dating from 834 AD, were later identified as nettle textiles (Barber 1991: 20). Other types of evidence, however, have suggested that nettles may have been in use as a textile fiber at a very early date. Some microdenticulate stone tools from Late Mesolithic and Neolithic sites in the Netherlands and Denmark show wear patterns that suggest they were scraping or stripping tools for nettle, perhaps functioning almost like combs to produce a fine fiber (Hurcombe 2010: 137). Bronze Age textiles found wrapping cremated human remains in the Lusehøj burial in Denmark have been identified as imported nettle most likely from a location in southwest Austria (Bernfjord et al. 2012: 664). Very few modern sources exist for nettle fabric. The only source I found that could ship to the U.S. was an Etsy seller, WoolFinch Studios in County Clare, Ireland (Appendix A.5). The raw undyed nettle fabric was very coarse and heavy, and a dark brown (Munsell 2.5Y 6/3) (Fig. 3.13c). While I postulate that nettle fabrics of the past were probably of a much finer quality and a lighter color than my modern example, I felt it was still of value to include nettle in these dye experiments.

*Wool:* The Bronze Age introduction of wool as a textile fiber was probably a major factor in the development of dyeing techniques (Strand and Nosch 2015: 55). The plant fibers available in Europe at that time are all difficult to dye and do not produce the vibrant hues of wool and later, silk. The earliest known textile made of wool fibers was found in a grave at Lützendorf in Germany, dated to the 3<sup>rd</sup> millennium BC. This item was apparently a corded skirt made of wool, linen and bast that unfortunately did not survive World War II (Bender Jørgensen 2003: 55). Wool ushered in a Bronze Age textile revolution. A few “textile people” have offered the opinion that the Bronze Age really should be renamed the Wool Age for this fiber’s impact on textiles and dye development (Strand and Nosch 2015: 375). Wool examples abound in the archaeological record from the late Bronze Age onward. For example, it is the most common fiber type found in the archaeologically important textile finds in prehistoric Europe, the textiles of the Hallstatt salt mines (Grömer 2011: 15). Not all prehistoric wool textiles were dyed, however, as there are many natural variations in raw wool coloring, including a reddish brown, that were utilized in textiles to create woven patterns without dyes. Rather than using wool fabric for these dye experiments, I chose to work only with white wool roving that was obtained from an online source, Shepswool.com (Appendix A.6). The wool roving was a very pale yellow (Munsell 2.5Y 8/2) (Fig. 3.13a).

*Silk:* The possible presence of silk in prehistoric temperate European burials seems to have been the source of lively archaeological debate, although most of these specimens could not conclusively be identified as silk. Silk arrived in Europe much later than the other fibers included in this experiment. Silk fibers were originally reported in 5<sup>th</sup> century Greek burials at Kerameikos and in the Iron Age European burial mounds at Hochdorf and Hohmichele (Good 1995: 960; Hundt 1969: 66). Unfortunately, the evidence for all of these finds has been called

into question (Banck 1994: 51; Banck-Burgess 1999: 234-240; Bender Jørgensen 1992: 105), and it is currently unclear when silk made its entrance into the Classical world and into Europe (Bender Jørgensen 2013: 586-7). At this point, it is safe to say that silk was in evidence in Roman times, as Pliny the Elder (*Naturalis Historia* VI, xx) noted that this revealing imported fabric, which he believed came from fleecy trees, had become a craze in Rome. The Silk Road trade continued, with interruptions, through the end of the Roman Era and the Migration Period. Silk was not domestically produced in Europe until the Byzantine Era (Barber 1991: 32).

Silk fibers were chosen for this experiment in part because I wanted another non-vegetal fiber for these dye experiments, and because of its presence in Europe in Roman times. Silk is an insect fiber, but like wool and other true animal fibers, it is made up of proteins rather than the cellulose of vegetal fibers (Turner 1949: 972). Untreated and undyed silk was also difficult to find, so I opted for the most minimally-processed silk that I could find. I purchased undyed silk handkerchiefs from yet another Etsy seller, Luthvarian Fiber Arts in Missouri (Appendix A.7). These proved to be somewhat “webby” and difficult to work with, but took dye well. The silk had a tendency to felt while being processed, but this did not interfere with the results. The raw silk fabric was a very clean white color (Fig. 3.13b).

### ***B. Equipment:***

I did not try to replicate ancient dying equipment, although there have been interesting experiments involving dye production rates with such equipment (Hopkins 2013: 119-133). Instead, I opted to use modern materials that are as non-reactive as possible so that modern chemicals and metals did not skew the results. Mordants and dried madder were prepared in glass jars, rain water was used for the dye baths, and some mordants and the madder dye baths were heated in a non-stick crock pot. A blender was used to break up the madder for the dye

bath, primarily because the purchased root was available in very large pieces, and needed to be broken up. A salad spinner was used to dry the dyed fibers, as many of the textile samples used were wool roving that can felt very easily if handled too much or wrung out. Samples of mordants and dried roots were weighed on a Salter scale and these amounts were standardized for all experiments. Temperature was monitored with a digital thermometer and Munsell charts were used to type color. Aside from the experiments on different mordants, a modern alum mordant was used on the fibers and an unmordanted sample was also dyed with it to serve as a control.

### ***C. Color:***

As color is such an integral component of this research, the aspects of color that were analyzed--hue, value, and saturation--need a brief explanation. *Hue* is essentially the dominant wavelength of light in a color, such as “red” or “blue” (Agoston 2013: 12). Other wavelengths may be present, and these can become important in defining hue as well, such as a “blue-green”. *Value*, or brightness, refers to the lightness or darkness of the hue, or how much white or black is present (Agoston 2013: 14). A shade is a darker version, and a tint is a lighter version. Value essentially adds the dimension of lightness and darkness to the color palette. Finally, *saturation* (also known as *chroma*) refers to the intensity of the color (Agoston 2013: 16). A true color with little gray in it is a very saturated color, while a less saturated color has more gray in it and the hue is less dominant (Agoston 2013: 13).

All three of these components are contained in the Munsell color system (Appendix A.8). Archaeology regularly uses Munsell systems, and there are special color books for soils and beads that I have worked with in the past as a means of organizing and standardizing a color language that can be easily understood by others. The Munsell system divides hue into five

primary hue categories: red (R), yellow (Y), green (G), blue (B), and purple (P). The hues make up the five letter categories that begin each Munsell number, such as R for red. There are also five intermediate categories for hue along a continuum, for instance as red (R) moves to yellow-red (YR) and on to yellow (Y). Each of these ten categories is then subdivided again into four different numerical categories in increments of 2.5, with the truest hue at 5. The range of subdivisions, in the Munsell system used here, are 2.5, 5, 7.5, and 10. Therefore, a full hue designation would read, for example, 2.5R or 7.5YR. The next digit in a full Munsell number designates color value. Color value varies vertically along a Munsell color page, with the lowest or darkest value at the bottom of the page, and the highest value at the top. Color value in this Munsell system began at 2 (darkest) and moved in increments of 1, up to 9 (lightest). The last digit in the Munsell color number designates color saturation. Saturation or chroma is measured horizontally from left to right, with the left (2) being the least saturated, and the right being the brightest or the most saturated. This horizontal measurement is in increments of even numbers (2,4,6,8). In this system, the most saturated position was at /16. A full Munsell color number that incorporates all three facets of color would then read, for example, 7.5R 4/12.

There are several Munsell color books of varying sizes and color palettes, but the Munsell system I chose to use was the Munsell Book of Color. After some experimentation with the Munsell Soils and Bead color books, I found that a more extensive color system was necessary, especially in the red range. I was fortunate to have access to this system through the Anthropology Department at the University of Wisconsin-Oshkosh. The data provided by this system revealed shifts in hue between dye baths in a standardized manner and proved to be an invaluable resource in determining changes in the dye results for each variable tested. The

Munsell designations will provide a foundation for comparative analyses with other dye experiments of this nature.

In addition, an IFRAO (International Federation of Rock Art Organization) color scale was used in all the images (Appendix A.9). All images were taken with my iPhone and exposure was often affected by the samples and the backgrounds in sometimes unexpected ways. There were efforts to control and correct this, but there was still variation in the lighting of some of the images that remained. Also, due to the number of images needed for the experiment results, these were not all done on the same date or at the same time of day. Therefore, there was some inconsistency of color in the images, as the lighting may have been different at different times. Color is also always distorted to some extent both in the electronic or photographic images taken, but also in subsequent printing or other transference of the images. The IFRAO designed this color scale as a means of standardizing not only size but color as well. When photographing items such as rock art, which must remain *in situ*, lighting cannot always be adjusted to give true hue, value and chroma. The IFRAO scale used in situations such as these serves as a means of adjusting for these factors, enabling a truer record of the color of these objects to be presented. As color is a crucial factor in this experiment, an effort to record it as accurately as possible was critical. In a sense, the IFRAO scale also provide a more objective record of color than the Munsell charts, which although an excellent method of standardization, are still affected by the subjective eye of the researcher assigning their numbers. The inclusion of both the Munsell Book of Color and the IFRAO scale are efforts to standardize the documentation of color in archaeological research, as these methods are both in regular use in this community.

As a final note on the issue of color, the range of reds possible from the prehistoric dye sources used must also be mentioned. While no experiments were done with kermes red, lac, or

cochineal, these reds are historic, and have classic red hues. Kermes is known to be crimson, while cochineal produces carmine or scarlet (both slightly bluer reds than crimson). Vermilion red is from the mineral cinnabar, while lac is lake, red lac or Indian lake. Madder red is considered a brick red, but as the experimental component of this research demonstrated, it can be manipulated with different dye methods to approximate a wide range of colors (including Turkey Red). These specific terms for red, however, are often used interchangeably, and therefore are not as reliable as a visual comparison between the reds. See Figure 3.14 for a representation of the range of visible differences in the red color spectrum.



Figure 3.14. The range of reds. (Image: [drawingblog.mycolouringland.com](http://drawingblog.mycolouringland.com))

#### ***D. Madder Sources:***

When it became clear that I would not have enough homegrown madder for all the experiments planned for this project, I looked for other sources for dried roots. I had hoped to find European *R. tinctorum*, but could not find any sources at the time of this experiment. I found several sources for whole root madder, all from Pakistan, but for consistency purchased my supply solely from The Woolery, a fiber arts supplier in Kentucky (Appendix A.10). Several potential issues with these purchased roots need to be noted here. Firstly, it is assumed that these roots are from *R. tinctorum* rather than *R. cordifolia*, which is the species of madder indigenous to Asia. The roots were sold as *R. tinctorum*, and are almost certainly that species, but this could not be confirmed. Also, it proved impossible to ascertain how old these roots were when harvested; it is assumed they were older than two and were likely three years old.

Madder roots were prepared for the dye bath by allowing them to soak in rain water in a glass fermenting jar for three days, as three to seven days were recommended (Goodwin 1982: 48) (Fig. 3.15a-b and 3.16a-b). I chose to use rain water for the presoaks and the dye baths, as I wanted to avoid any possible metals or ions (iron or chlorine) that might be in my city water and could affect color results. The roots were then chopped up in the electric blender, and except where time or temperature were the variables being tested, warmed for two hours in the crock pot before the bath was strained and the fibers were added. Fibers were left in the warm dye bath for another two hours before being removed, rinsed and spun dry in a salad spinner (Figure 3.17a-d).



Fig. 3.15a-b. Soaking Pakistani madder. Fig. 3.15a. Left: ten minutes after water was added. Fig. 3.15b. Right: the same batch after 12 hours.



Fig. 3.16a-b. Soaking the dried homegrown madder. Fig. 3.16a. Left: the homegrown madder, after 20 minutes in rainwater. Fig. 3.16b. Right: the same madder bath, after a 12-hour soak.



Fig. 3.17a-d. Equipment used in the experimental component. Fig. 3.17a. Upper left: modern yet non-reactive equipment used in dye bath preparation. Fig. 3.17b. Upper right: madder root pieces were chopped in a blender. Figure 3.17c. Lower left: wool roving in a madder dye bath, heated in a nonstick crock pot. Figure 3.17d. Lower right: mordanted wool roving spun dry in a salad spinner.

### Phase 3: Dye Bath Processes

#### *A. Sources of Dye Recipes:*

Fundamental modern techniques for madder dyeing were used to establish a baseline for madder color. These techniques were adapted from several sources, mainly online blogs or modern publications on dyeing with vegetable dyes (Dean 2007: 118-122; Goodwin 1982: 48,66; Roberts 2017). To generate a set of consistent practices based on these sources, I developed a strategy that was an amalgam of several recommended approaches to madder dyebaths. Ancient sources for madder dye methods and mordants were also considered. An alchemical document

from about 300 AD Greece known as the *Papyrus Graecus Holmiensis*, or the Stockholm Papyrus, contains over 150 dye recipes, including several techniques for utilizing madder (Caley 2008: 74, 79, 84-5), in a process known as “bluing” with woad to obtain a better red (Caley 2008: 77, 84-5), and recipes for dozens of mordants, including the use of oak galls, an iron mordant (Caley 2008: 81), and soapwort (Caley 2008: 83). The mordant recipes in the latter source in particular were the basis for choosing the mordants used in these experiments.

### ***B. Mordants***

Because madder is a mordant dye, i.e. it requires a separate treatment to fix the color in the material being dyed, an extra step to chemically prepare the fibers was necessary. For each of these experiments, fabric or fibers were chemically treated in a mordant bath, then rinsed and sometimes dried before the dyeing process. All of these mordants either chemically strip the fiber surfaces or break them down slightly so that the dye may be absorbed better. Mordants chosen for this experiment were selected to represent a range of chemical qualities, as some are acidic, some are natural soaps, and some are metal salts. Some mordants exhibited more than one of these traits. Mordants for these experiments were also chosen based on their availability in Bronze and/or Iron Age Europe. Oak galls, soapwort and club mosses are all indigenous to much of Europe and Britain. Iron oxides and tannic mordants would also have been fairly easy to obtain in Europe in prehistory. Copper mordants would also have been a possibility, but these are not used with red or purple dyes, as their effect is generally to enhance yellows, greens or blues. While mineral alum mordants were mined in Egypt and in use in the prehistoric Mediterranean region, especially in Greece and Egypt, it is generally thought that alum did not make its way into European chemical processes until historic times (Barber 1991: 238-9; Dean 2014: 10; Duff and Sinclair 1988: 25; Hall et al. 1984: 58). However, because alum is the most

common and basic mordant used in modern dyeing, it was used in these experiments strictly to establish baseline color when other variables in the dye process were being tested and to determine how an alum mordant combined with madder might affect color. Alum was obtained from an online source (Appendix A.11). The details of the specific prehistoric mordants utilized are provided below.

*Oak Galls:* European oak galls are known to create a dark brown ink, and can also be used to create a tannic mordant. Whole oak galls proved to be a prohibitively expensive item, perhaps because these are sometimes considered items of prosperity in the Wiccan tradition. I was not prosperous enough to afford more than a small number of them (Appendix A.12). However, ground oak galls are much more affordable and are available at many herbalist sites online (Appendix A.13) so this is what was used for the oak gall mordant. The recipe I decided on was a combination of two modern methods (Dean 2014: 29; Goodwin 1982: 32), and the modern process used for mordanting with alum. The oak gall mordant was prepared by mixing the powdered oak galls in rainwater and simmering the liquid for about one hour. Powdered oak galls were added to the mordant bath in the amount of about 10% of the dry weight of the fibers. The mordant bath was colorless, but had a wonderful aroma of fresh cut wood while simmering. The resulting liquid was cooled for one hour--during which time it did darken (Fig. 3.18a-b)—then pre-soaked fibers were added to the mordant bath, and the temperature was slowly raised to around 60-70° C, and maintained for one hour. Fibers were then removed, and spun to remove excess liquid. The mordant was not rinsed until the fibers were ready for the dye bath itself.



Fig. 3.18a-b. Heating the ground oak galls. Fig. 3.18a. Left: powdered oak galls minutes after dissolving in simmering water. Fig. 3.18b. Right: the same mordant, cooled for 30 minutes. The oak galls have darkened the bath with a deep brown tint.

*Club Moss:* Aluminum mordants, in particular alum, have historically been considered some of the most important and best mordants to use in dyeing (Barber 1991: 238; Cardon 2007: 20). However, it is debatable whether alum as a mordant was in general use in Europe in prehistoric times. Alum, the most commonly used mordant today, occurs in mineral form and was mined in Egypt. Alum was also possibly chemically produced in Classical Greece and Rome, and may have been traded in Europe from Phocaea (Cardon 2007: 24). The word *alum* itself may even be of Gaulish origin (Cardon 2007: 21). It is uncertain when alum arrived in Europe, but it may only have been used by the Romans and apparently fell out of use after the decline of the Roman Empire (Dean 2014: 148). However, several plant species that have a naturally high aluminum content were used as dye mordants in prehistoric Europe, including the club mosses (*Lycopodium spp.*) Club mosses are indigenous to much of northern Europe, Britain and Scandinavia, and are associated with madder dyeing (Hall et al. 1984: 58-60; Duff et al. 1988: 25). There is some archaeological evidence that club mosses were used in Viking times at York (Tomlinson 1985: 275). Preparing club moss as a mordant, however, is a rather lengthy and labor-intensive process. The harvested and dried moss must be gently warmed for several

hours (preferably overnight), taking care that the mordant bath does not boil. Using purchased dried clubmoss (Appendix A.14), I employed the crockpot to keep the club moss mordant solution at a fairly constant low temperature (around 60° C). The resulting liquid was then strained and allowed to cool. Wetted fibers were added and the bath was warmed to about 60° C again and heated for one hour. The bath was allowed to cool until the fibers could be safely handled, when they were strained out (Fig. 3.19a-b).



Fig. 3.19a-b. Club moss mordant preparation. Fig. 3.19a. Left: dried club moss mordant warming in the crock pot overnight. Fig. 3.19b. Right: finished club moss mordant, a greenish yellow, with a pleasant herbal aroma.

*Iron Liquor:* As was mentioned above, the Stockholm papyrus cites a recipe for an iron mordant, also known as iron vitriol. A true iron liquor was made with rusted iron and pyrolignous acid, or wood acid, a combination of acetic acid, acetone and wood alcohol. The active and dominant ingredient, however, is the acetic acid. The ingredients in this mordant, then, are seductively simple: rust and vinegar. This was by far the least costly mordant of those tested, as all it required was a rusted object, water, vinegar and time. This is also a very popular modern mordant for those interested in traditional dyeing techniques, and there are several online sources touting the virtues of mordanting with what is known as iron liquor. Inspired by the

Stockholm papyrus, and aided by the advice of modern dyers, I opted to create an iron liquor from a rusted railroad spike and white vinegar. This is not an instant mordant, however, as it takes weeks to develop (Fig. 3.20a-b). The Stockholm Papyrus recommended that this mordant bath be cold, but modern dyers tend to make a warm iron mordant bath. Both methods were tested, in one-hour time periods, with all the fiber types.



Fig. 3.20a-b. Development of the iron liquor over several weeks. Fig. 3.20a. Left: the iron liquor solution just after creation. Fig. 3.20b. Right: iron liquor after six weeks.

*Soapwort:* Soapwort, or soapweed, is derived from both the leaves and the roots of the *Saponaria officinalis* plant (Fig. 3.9a). The plant contains saponins, and has been used historically as a natural detergent. The Stockholm Papyrus mentions soapweed several times as a means of cleaning and preparing fibers for dyeing. For this experiment, ground and dried root was used. I grew soapwort as a part of the dye plant bed experiment, and it proved so vigorous that most of it had to be removed as it was crowding out the other plants. I did harvest some roots from the remaining plants (Fig. 3.9b), but I supplemented this with some inexpensive dried soapwort from an online seller, Gift from Nature, a Bulgarian herbal source (Appendix A.15).

The homegrown root and the purchased roots were all in small pieces (about 1 cm), which precluded using a blender to liquify them. I allowed the dried root to soak in rain water for one day in a glass fermenting jar, then warmed the roots in a stainless steel pot for one hour. I gently whisked the mixture, which created a soapy foam (Fig. 3.21c-d). This liquid was then strained and the fibers were added and allowed to soak at a constant temperature (roughly 65°C) for one hour. The mordant was allowed to cool, and fibers were removed and spun dry.



Fig. 3.21a-d. Soapwort mordant. Fig. 3.21a. Upper left: young soapwort plants. Fig. 3.21b. Upper right: freshly harvest soapwort root. Fig.3.21c. Lower left: heating the dried and fresh soapwort—note suds. Fig. 3.21d. Lower right: fibers warming in a sudsy soapwort mordant.

### ***C. Other Dye Experiment Variables***

Aside from the mordant tests, other variables were determined to have an effect on the color qualities of madder dyebaths. These were time, temperature, and pH. Each of these variables was tested with the wool roving and with the four textile types. The details of these tests are listed below. The set up for each of the experiments was generally the same. Madder

roots were soaked in a glass fermenting jar in rain water for at least three days. The madder was then blended in an electric blender and poured into the non-stick crock pot to warm for two hours. It was then strained to remove the roots and rinsed and wetted fibers were added to the pot. Fibers were dyed for a period of about 90 minutes in the dye bath, then removed and rinsed.

Time: To test whether time played a significant factor in the hue and saturation of color, three samples were tested for different lengths of time in the dye bath. The four fiber types and wool roving samples (both mordanted and unmordanted with alum) were dyed for the standard 90 minutes in the dye bath, but other sets of fiber samples were dyed for only 30 minutes, and one was placed in the dye bath for a 12-hour period. All samples were then rinsed and spun dry. Results were photographed and color recorded using the Munsell chart.

Temperature: Several sources stated that madder dye baths should ideally be “warm” or “well below boiling” (Goodwin 1982: 48), that is, above room temperature (over about 27° C) but under 70° C. I aimed for a “warm” dye bath at around 60-70° C as the optimal temperature, with the aid of a non-stick coated crock pot. This was the temperature range applied to all other experiments with pH and different mordants. I also tested dye baths that had been boiled for five minutes, and unheated madder dye baths to see if the resulting samples would differ in hue and saturation. Results were photographed and Munsell color was recorded.

pH: I constructed three separate tests for the effects of differing pH on the dye results, using three separate acidic or alkaline sources that could have been used in the prehistoric dyeing process. White vinegar (acetic acid), wood ash water (lye or potassium hydroxide), and dilute ammonia (as a substitute for stale urine), were chosen for these tests. Ammonia and white vinegar were both purchased at a local hardware store. Wood ash water was made by adding around two cups of coarsely sifted wood ash from my burn pit to a glass fermenting jar and

covering the ash with rainwater (Fig 3.22a-b). This was allowed to sit for two weeks and the pH was then tested. These pH tests could have been included in the mordant section as many modern sources and the Stockholm Papyrus list these chemicals as mordants. However, I wanted to test the effects of pH on color without the interference of metals such as iron in the dyeing process, and decided to test these solutions as something other than mordants. Therefore, while these acids and bases had the effect of mordanting fibers, the samples of the fibers were also tested with an alum mordant and with no mordant. The ground madder dye bath was heated in the crock pot to between 60 and 70°C for two hours with the chosen acid or base and the pH was ascertained using Lab Rat brand pH test strips. The dye bath was then strained, the fibers added, and the bath heated for 90 minutes. The dye bath was then allowed to cool slightly, and the fiber samples removed, rinsed and spun dry. Results were photographed and Munsell color recorded.



Fig. 3.22a-b. Wood ash water examples. Fig. 3.22a. Left: wood ash water when first mixed. Fig. 3.22b. Right: wood ash water after 14 days. The aged wood ash water is almost clear. Care was taken not to mix the ash sediment into the dye bath.

The results from all of these experiments are compiled in the following chapter. Each of these experiments was compared against the standards of unmordanted and alum-mordanted fibers, and against each other for all aspects of color. The result of these experiments could potentially be compared against other dye experiments as well, as an effort was made to quantify the results, especially the color data, to be capable of comparison with other research in other times and places. Hopefully this can add to the growing body of data on vegetal dyes.

## **CHAPTER 4: RESULTS**

### **Phase 1. Madder Cultivation Results**

#### ***A. Cultivation Results***

While the section that follows is quite short, the success of this portion of the research was critical to the success of the rest of the experimental component of this thesis. All the specimens of *Rubia tinctorum* successfully survived two growing seasons in Wisconsin's climate. These plants did not seem to have any trouble growing in this region. They weathered several increasingly severe frosts with no damage until temperatures really fell below freezing for sustained periods of time. In other words, they are not tender perennials in this climate. Shoots also emerged in spring with most of the local perennials. All nine plants bloomed and set seed during the second summer of growth. Dyer's madder seems to be fairly well suited to this area, and can easily survive a "typical" Wisconsin winter. That said, my backyard is a fairly sheltered space, and while this experiment demonstrates that this particular species of madder can withstand this climate, that might not hold true in a large open field, if grown on a commercial scale, or if a wild European madder were to be grown here.

#### ***B. Harvest Results***

The harvest of nine two-year-old madder plants produced 92 grams of dried madder root. As this was a first effort in growing this plant, it is impossible to determine if this can be considered a respectable harvest or not; it has become the baseline for future madder harvests. I will say that this amount of dried madder would supply several smaller dye baths or one large one. Thus, two years of growing a plant would yield one large dye bath, which isn't a great deal of dye for such a long period of time. It would take a large yard, or a large dye garden, to grow

the amount of madder plants necessary for multiple dye processes. It would also necessitate some proficiency with this plant, as there is little room for error. The color of these two-year-old plants was also a slightly different hue than the purchased Pakistani madder root, which I assume to be from plants that are older than two years (although this could not be confirmed). My Wisconsin madder was more orange-red in hue than the purchased madder. Again, it is impossible to determine whether this is due to its young age, or because of the climate, or because the particular plant strains are different. Future harvests of older plants will help answer that question. The homegrown madder crop was visually compared to the purchased Pakistani madder, and looked, in my humble opinion, just a bit redder (Fig. 4.1a-b).



Figure 4.1. Dried madders. On the left, a sample of the dried homegrown madder. On the right, the purchased Pakistani madder.

As a part of this harvest, flowers and seed were also collected and dried and saved for the future. Seed was also sown in the bed at the end of September 2017. Sections of root, stem, leaf and seed were collected and photographed under magnification (Fig. 4.2a-d). The retrorse spines on the leaves and stem are clearly visible. A cross section of the root also shows the bright orange center pith of the root. Most of the dye compounds are located in the fleshy outer layer of deep orange.

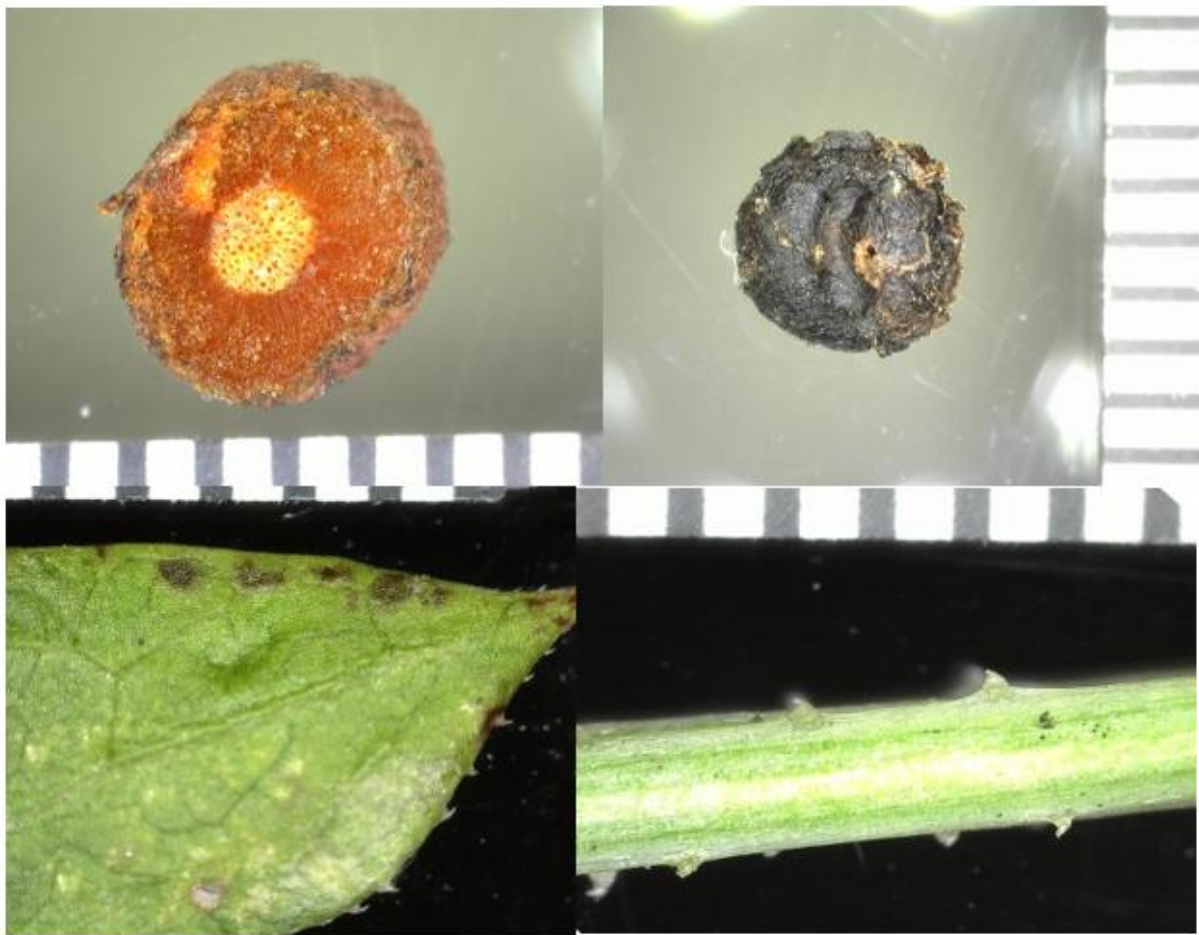


Fig. 4.2a-d. Magnified images of madder plant. Fig.4.2a. Upper left: a cross-section of madder root. Fig.4.2b. Upper right: a semi-desiccated madder seed. Fig.4.2c. Lower left: a section of madder leaf, with spines visible along edge. Fig.4.2d. Lower right: madder stem section with spines. (Images: Jennifer Haas. Scale: 1 mm)




### Phases 2-3. Dye Experiment Results

With the luxury of 400 grams of purchased madder root, I was able to test many variables in madder dye baths. However, with each experiment, it was clear that there were so many other options that could be tested as well. Mordants could be combined with different pH, and pH agents could be applied in different ways (such as a dip in an acid or base at the end of the dye bath). Time and temperature could be combined for new effects as well. Again, these results point to future possibilities in new dyeing techniques. Each of the dye bath experiments that were performed will be reviewed separately below. All dye bath experiments were tested on the four fiber types. Untreated examples of these fibers (Fig. 4.3, Appendix B/Fig. B1), and their Munsell numbers (Table 4.1, Appendix C/Table C1) are shown below.



Fig. 4.3. Untreated examples of the experiment fiber types.

TABLE 4.1. MUNSELL COLORS OF DYE BATH FIBERS

Fiber Type	Thumbnail	Munsell
Wool		5Y 8.5
Silk		White
Linen		10YR 6/2
Nettle		10YR 5/2

#### A. Basic Alum Dye Baths: Dyed Fiber Samples with and without an alum mordant.

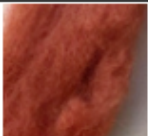


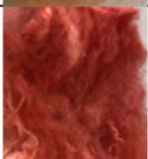

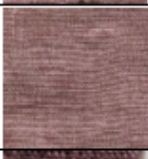


Dye experiments with fiber samples began by establishing a set of standards for each fiber that included the fibers without a mordant and with a modern alum mordant. It was apparent that the alum mordant produced much brighter, more vibrant colors on the animal fibers (Fig. 4.4). Both the wool and the silk exhibited redder hues when combined with an alum

mordant (the silk exceptionally so). The alum mordant also appeared to have a slight bleaching effect on the plant fibers, as the mordanted samples tended to be more red or brown and lighter than their unmordanted counterparts. I decided to continue to dye an unmordanted version in other experiments as this often showed some color variations that were masked by the alum mordant in other samples. The unmordanted linen and nettle were more of a purple color than expected, although this may be due in part to the darker color of the original fibers. See Table 4.2 (also Appendix C/Table C2) for complete results. All images for this experiment in Appendix B/Figs. B3-B6).



Figure 4.4. Madder-dyed fiber types with and without an alum mordant. Clockwise from upper left: wool, silk, nettle, and linen.

**TABLE 4.2. FIBERS SAMPLE WITH AND WITHOUT AN ALUM MORDANT**

Fiber type	Thumbnail	pH	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Wool		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/8
Wool		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/8
Silk		7	62°/144°	1.5	None	Purchased Pakistani	10R 6/6
Silk		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 5/8
Linen		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/4
Linen		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 6/4
Nettle		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/4
Nettle		6	62°/144°	1.5	Alum	Purchased Pakistani	10R 5/6

***B. Dye Bath Temperature Variations: Results***

I expected great variation in color with temperature changes, but this only held true for the cold dye bath. The results from the madder dye bath at room temperature were more pink than orange, but did not have the depth of color that the heated dye baths produced. The boiled dye bath produced wool and silk fibers with a more orange-tinged brick red hue than the redder

warmed bath, but this color change was not pronounced (Figs. 4.5-4.6). The warmed dye bath was kept at about 160° F, but perhaps this was too hot to preserve the more pinkish reds that were observed from the dye bath at 70° F. A temperature in the low 100°'s might produce a redder or pinker color. The response of the other fiber types made me realize how “wool-centric” this set of experiments actually was. All the other fibers types took up dye much better at boiling temperatures. The mordanted plant fibers were a much stronger red than at other temperatures (Figs. 4.7-4.8). The silk in particular always had a streaky dye result in the warm dye baths, but not with the boiled samples (Fig. 4.6b). There, the color was vibrant, saturated and even throughout the fiber sample. The boiled silk sample with the alum mordant was one of the most saturated and beautiful color samples in the entirety of the experiment. Refer to Table 4.3 (Appendix C/Table C3) for results. The complete set of imaged related to temperature variation can be found in Appendix B/Figs. B7-B26.



Fig. 4.5a-b. Wool fibers at differing temperatures. Fig. 4.5a. Left: unmordanted wool. Fig. 4.5b. Right: mordanted wool.



Fig. 4.6a-b. Silk fibers at differing temperatures. Fig.4.6a. Left: unmordanted silk. Fig. 4.6b. Right: mordanted silk.

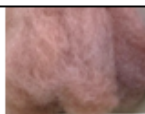

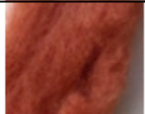






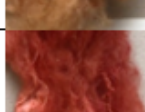

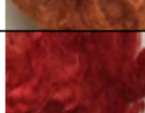


Fig. 4.7a-b. Linen fibers at differing temperatures. Fig.4.7a. Left: unmordanted linen. Fig. 4.7b. Right: mordanted linen.



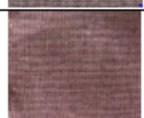
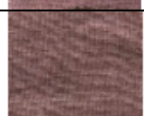




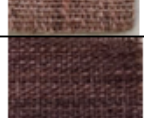





Fig. 4.8a-b. Nettle fibers at differing temperatures. Fig. 4.8a. Left: unmordanted nettle. Fig. 4.8b. Right: mordanted nettle.

**TABLE 4.3. TEMPERATURE EXPERIMENTS WITH MADDER DYE BATHS**

Fiber type	Thumbnail	pH	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Wool		7	21°/70°	1.5	None	Purchased Pakistani	7.5R 6/4
Wool		6	21°/70°	1.5	Alum	Purchased Pakistani	7.5R 6/8
Wool		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/8
Wool		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/8
Wool		7	100°/212°	1.5	None	Purchased Pakistani	10R 4/8
Wool		6	100°/212°	1.5	Alum	Purchased Pakistani	10R 4/10
Silk		7	21°/70°	1.5	None	Purchased Pakistani	10R 6/4
Silk		6	21°/70°	1.5	Alum	Purchased Pakistani	7.5R 6/8
Silk		7	62°/144°	1.5	None	Purchased Pakistani	2.5YR 5/6
Silk		6	62°/144°	1.5	Alum	Purchased Pakistani	5R 5/8
Silk		7	100°/212°	1.5	None	Purchased Pakistani	2.5YR 5/8
Silk		6	100°/212°	1.5	Alum	Purchased Pakistani	7.5R 3/8

**TABLE 4.3. TEMPERATURE EXPERIMENTS WITH MADDER DYE BATHS (continued)**

<b>Fiber Type</b>	<b>Thumbnail</b>	<b>pH</b>	<b>Temp. C°/F°</b>	<b>Time (hours)</b>	<b>Mordant</b>	<b>Dye Source</b>	<b>Munsell</b>
Linen		7	21°/70°	1.5	None	Purchased Pakistani	7.5R 7/2
Linen		6	21°/70°	1.5	Alum	Purchased Pakistani	7.5R 6/2
Linen		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/4
Linen		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 6/4
Linen		7	100°/212°	1.5	None	Purchased Pakistani	7.5R 5/4
Linen		6	100°/212°	1.5	Alum	Purchased Pakistani	7.5R 5/6
Nettle		7	21°/70°	1.5	None	Purchased Pakistani	2.5YR 5/2
Nettle		6	21°/70°	1.5	Alum	Purchased Pakistani	2.5YR 5/4
Nettle		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/4
Nettle		6	62°/144°	1.5	Alum	Purchased Pakistani	10R 5/6
Nettle		7	100°/212°	1.5	None	Purchased Pakistani	7.5R5/4
Nettle		6	100°/212°	1.5	Alum	Purchased Pakistani	7.5R 4/6

### ***C. Dye Bath pH Variations: Results***

Some of the most dramatic color changes occurred in the experiments on pH. It took some adjusting to get the right concentration of acids and bases into these dyebaths. In all cases, the bath started out too strong and had to be diluted. Weak concentrations produced better colors in all pH experiments.

The acidic dye bath, using dilute acetic acid ( $\text{CH}_3\text{COOH}$ ), was assessed at 4 pH. The acetic acid was probably too concentrated in the initial attempts and the dye colors looked very similar to unmordanted fibers. More diluted dye baths produced much more distinctive colors. The results from these dye baths were very vibrant orange-red on the animal fibers and had a subtler but noticeable effect on the plant fibers as well. The acidic dye bath may have functioned as a mordant in itself, as the unmordanted wool sample, for example, was nearly as vibrant as the sample treated with alum.

The alkaline dye baths also produced dramatic results, after some experimentation with levels of dilution. To approximate stale urine, I opted to dilute ammonia to about a pH of 9 or 10. A preliminary dye bath of a more concentrated ammonia solution ( $\text{NH}_3$  with a pH of 11-12) resulted in very pale grayish pink animal fibers and was probably the result of the highly alkaline solution denaturing the dye compounds or possibly even the fibers. A more dilute solution produced much more vibrant results. Ammonia was one of two alkaline sources used in this set of experiments. Wood ash water or lye ( $\text{KOH}$ ), was the other choice for alkaline dye baths. I assumed the results would be the same for both substances, but they were not. The mordanted animal fibers did turn out the same, with a deep reddish-purple color. The unmordanted samples,

however, were quite different. While the ammonia gave unmordanted wool a light pinkish hue, the unmordanted wool sample in the lye dye bath was almost identical to the mordanted sample. The lye dye bath was slightly higher in pH (at 9 rather than 10), but it is unclear if that change made the difference, or if the different chemical makeup of the solutions caused the difference in the unmordanted samples.

I had assumed that all these acids and bases would function almost as mordants themselves, but the mixed results of the unmordanted samples did not confirm this. The deep purplish reds of the mordanted alkaline samples were a wonderful rich color, and it seemed that I had achieved one of the desired colors of madder red with these samples.

The pH experiments are among the few cases in which the protein-based fibers (wool and silk) did not respond in the same way to the dye process. The wool samples (Fig. 4.9) in the alkaline dye baths produced a red-violet color that was not seen in the silk (Fig. 4.10) which remained in the pure red range. The plant-based fibers also remained in the red range and did not achieve that red-violet color (Figs. 4.11-4.12). See Tables 4.4-4.7 (Appendix C/Tables C4-C7) for results. (All images from this set of experiments are in Appendix B/Figs. B27-B46).



Fig. 4.9a-b. Wool fibers in dye baths of different pH. Fig.4.9a. (left) unmordanted wool. Fig.4.9b. (right) mordanted wool.

**TABLE 4.4. DYE BATH pH EXPERIMENTS ON WOOL**

Fiber type	Thumbnail	pH/ source	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Wool		4 Acetic Acid	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/10
Wool		4 Acetic Acid	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/12
Wool		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/8
Wool		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/8
Wool		9 <u>WoodAsh</u> Water	62°/144°	1.5	None	Purchased Pakistani	5R 3/6
Wool		9 Wood Ash Water	62°/144°	1.5	Alum	Purchased Pakistani	5R 3/8
Wool		10 Dilute Ammonia	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/8
Wool		10 Dilute Ammonia	62°/144°	1.5	Alum	Purchased Pakistani	5R 3/8



Fig. 4.10a-b. Silk fibers in dye baths of different pH. Fig. 4.10a. Left: unmordanted silk. Fig. 4.10b. Right: mordanted silk.

**TABLE 4.5. DYE BATH pH EXPERIMENTS ON SILK**

Fiber Type	Thumbnail	pH/source	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Silk		4 Acetic Acid	62°/144°	1.5	None	Purchased Pakistani	10R 5/8
Silk		4 Acetic Acid	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 3/10
Silk		7	62°/144°	1.5	None	Purchased Pakistani	2.5YR 5/6
Silk		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 5/8
Silk		9 Wood Ash Water	62°/144°	1.5	None	Purchased Pakistani	7.5R 6/6
Silk		9 Wood Ash Water	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/10
Silk		10 Dilute Ammonia	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/8
Silk		10 Dilute Ammonia	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 5/10



Fig. 4.11. Linen fibers in dye baths of different pH. Fig. 4.11a. Left: unmordanted linen. Fig. 4.11b. Right: mordanted linen.

**TABLE 4.6. DYE BATH pH EXPERIMENTS ON LINEN**

Fiber Type	Thumbnail	pH/source	Temp. C°/F°	Time (hours.)	Mordant	Dye Source	Munsell
Linen		4 Acetic Acid	62°/144°	1.5	None	Purchased Pakistani	7.5 6/4
Linen		4 Acetic Acid	62°/144°	1.5	Alum	Purchased Pakistani	7.5 5/4
Linen		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/4
Linen		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 6/4
Linen		9 Wood Ash Water	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/4
Linen		9 Wood Ash Water	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 5/6
Linen		10 Dilute Ammonia	62°/144°	1.5	None	Purchased Pakistani	7.5 5/4
Linen		10 Dilute Ammonia	62°/144°	1.5	Alum	Purchased Pakistani	7.5 5/4

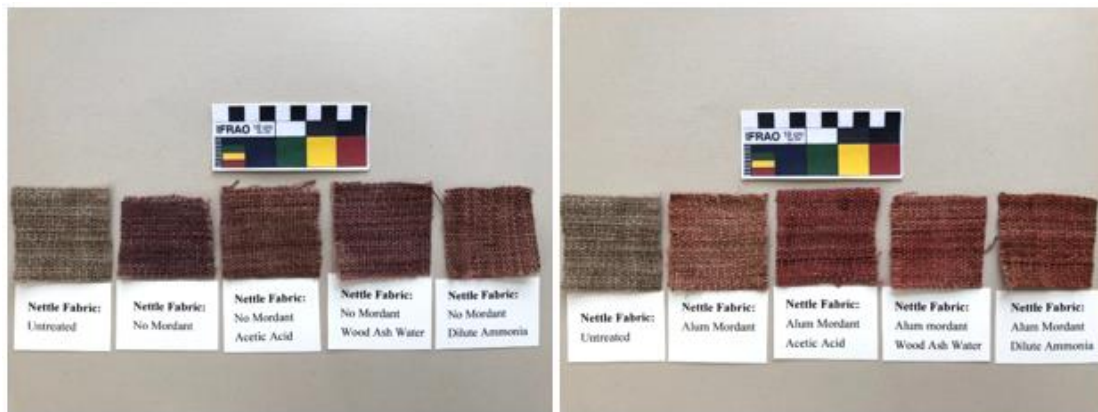


Fig. 4.12. Nettle fibers in dye baths of different pH. Fig. 4.12a. Left: unmordanted nettle. Fig. 4.12b. Right: mordanted nettle.

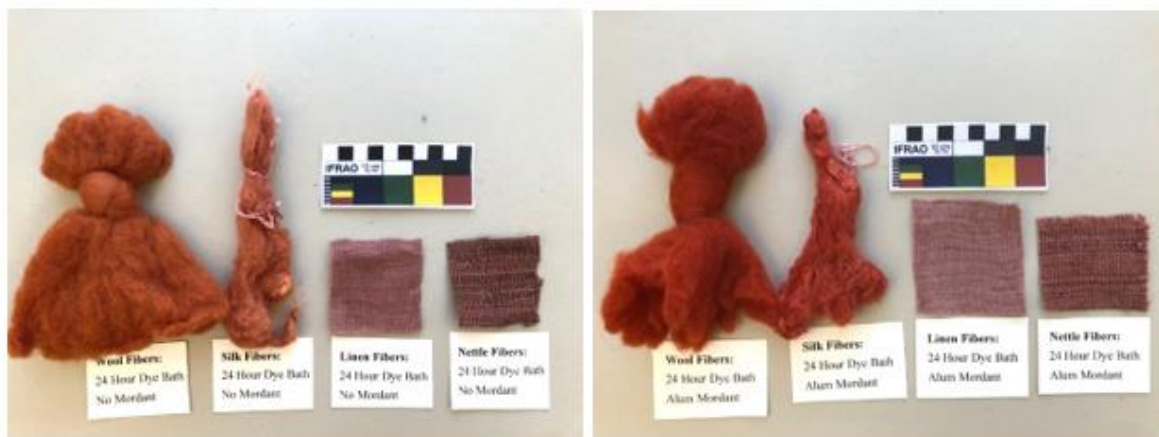
**TABLE 4.7. DYE BATH pH EXPERIMENTS ON NETTLE**

Fiber Type	Thumbnail	pH/source	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Nettle		4 Acetic Acid	62°/144°	1.5	None	Purchased Pakistani	10R 4/4
Nettle		4 Acetic Acid	62°/144°	1.5	Alum	Purchased Pakistani	7.5 4/6
Nettle		7 Wood Ash Water	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/4
Nettle		6 Wood Ash Water	62°/144°	1.5	Alum	Purchased Pakistani	10R 5/6
Nettle		9 Wood Ash Water	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/4
Nettle		9 Wood Ash Water	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/6
Nettle		10 Dilute Ammonia	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/4
Nettle		10 Dilute Ammonia	62°/144°	1.5	Alum	Purchased Pakistani	7.5 4/6


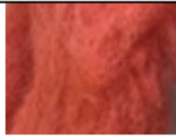


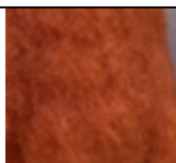


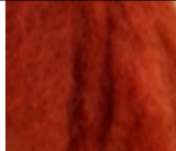
#### ***D. Dye Bath Time Experiments: Results***

Timed dye bath results confirmed my expectations in some regards. I assumed the colors would deepen the longer the fiber samples remained in the dye bath. The 0.5-hour samples were much lighter than all samples dyed for longer periods of time (Fig. 4.13a-b). The 1.5-hour, examples that had already been dyed with the alum mordant samples (Fig. 4.4) exhibited a deep saturation of color that was comparable to later samples. There was little to no difference in the color saturation between the 12-hour and the 24-hour samples (Fig. 4.14a-b and 4.15.a-b), either because the dye bath was exhausted, or because the fibers could not take up more dye.





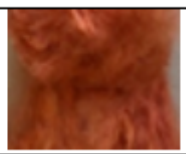



The change in hue, however, was unexpected. The 0.5-hour and 1.5-hour samples maintained a pinkish or purplish hue that was lost or replaced by an orange-red by the 12-hour sample. Perhaps more alizarin was taken up by the fibers as time passed, or some of the more ephemeral dye compounds that had contributed the pink/purple hues broke down in the warm dye bath over time. The change in hue was noticeable, however, as both the images and Munsell numbers indicate, with a general shift out of the pinker 7.5R range to the more orange 10R range in hue (Tables 4.8-4.11). (Appendix C/Tables C8-C11 contains all timed dye bath results. All images from this set of experiments can be found in Appendix B/Figs. B47-B60).



**TABLE 4.8. WOOL FIBERS IN DYE BATHS OF VARYING DURATION**

Fiber Type	Thumbnail	pH/ source	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Wool		7	62°/144°	0.5	None	Purchased Pakistani	10R 6/8
Wool		6	62°/144°	0.5	Alum	Purchased Pakistani	7.5R 6/12
Wool		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/8
Wool		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/8
Wool		7	62°/144°	12.0	None	Purchased Pakistani	10R 5/10
Wool		6	62°/144°	12.0	Alum	Purchased Pakistani	10R 4/12
Wool		7	62°/144°	24.0	None	Purchased Pakistani	10R 4/10
Wool			62°/144°	24.0	Alum	Purchased Pakistani	10R 4/12



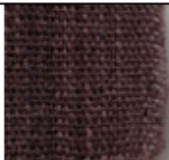





**TABLE 4.9. SILK FIBERS IN DYE BATHS OF VARYING DURATION**

<b>Fiber Type</b>	<b>Thumbnail</b>	<b>pH/ source</b>	<b>Temp. C°/F°</b>	<b>Time (hours)</b>	<b>Mordant</b>	<b>Dye Source</b>	<b>Munsell</b>
Silk		7	62°/144°	0.5	None	Purchased Pakistani	10R 6/8
Silk		6	62°/144°	0.5	Alum	Purchased Pakistani	7.5R 5/10
Silk		7	62°/144°	1.5	None	Purchased Pakistani	2.5YR 5/6
Silk		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 5/8
Silk		7	62°/144°	12.0	None	Purchased Pakistani	10R 5/8
Silk		6	62°/144°	12.0	Alum	Purchased Pakistani	7.5R 4/10
Silk		7	62°/144°	24.0	None	Purchased Pakistani	10R 5/8
Silk		6	62°/144°	24.0	Alum	Purchased Pakistani	7.5R 4/10

**TABLE 4.10. LINEN FIBERS IN DYE BATHS OF VARYING DURATION**

<b>Fiber Type</b>	<b>Thumbnail</b>	<b>pH/ source</b>	<b>Temp. C°/F°</b>	<b>Time (hours)</b>	<b>Mordant</b>	<b>Dye Source</b>	<b>Munsell</b>
Linen		7	62°/144°	0.5	None	Purchased Pakistani	7.5R 5/4
Linen		6	62°/144°	0.5	Alum	Purchased Pakistani	7.5R 6/6
Linen		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/4
Linen		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 6/4
Linen		7	62°/144°	12.0	None	Purchased Pakistani	7.5R 5/6
Linen		6	62°/144°	12.0	Alum	Purchased Pakistani	7.5R 5/6
Linen		7	62°/144°	24.0	None	Purchased Pakistani	7.5R 5/6
Linen		6	62°/144°	24.0	Alum	Purchased Pakistani	7.5R 5/6

**TABLE 4.11. NETTLE FIBERS IN DYE BATHS OF VARYING DURATION**

<b>Fiber Type</b>	<b>Thumbnail</b>	<b>pH/ source</b>	<b>Temp. C°/F°</b>	<b>Time (hours)</b>	<b>Mordant</b>	<b>Dye Source</b>	<b>Munsell</b>
Nettle		7	62°/144°	0.5	None	Purchased Pakistani	7.5R 4/4
Nettle		6	62°/144°	0.5	Alum	Purchased Pakistani	7.5 5/8
Nettle		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/4
Nettle		6	62°/144°	1.5	Alum	Purchased Pakistani	10R 5/6
Nettle		7	62°/144°	12.0	None	Purchased Pakistani	7.5R 4/6
Nettle		6	62°/144°	12.0	Alum	Purchased Pakistani	7.5R 4/8
Nettle		7	62°/144°	24.0	None	Purchased Pakistani	7.5R 4/6
Nettle		6	62°/144°	24.0	Alum	Purchased Pakistani	7.5R 5/8

### ***E. Mordant Experiments: Results***

The various mordants each provided interesting results. The soapwort and oak gall mordants, both of which had a brown hue when prepared, also produced vibrant red-brown dyes on the animal fibers, but had little effect on the plant fibers. The oak gall mordant produced a markedly acidic dye bath with the tannic content and acidity seems to have contributed to the vibrancy of the dye on the animal fibers. The color, though, was a rich warm brown rather than an orange-red as with the acetic acid dye bath. The soapwort mordant contributed a brown color to the fibers as well, but this was a duller brown, and the plant fibers did not show much color change from their untreated state. The club moss mordant, when viewed in the context of other non-alum mordants, did provide a pinkish tone to the dyed animal fibers, although this was not nearly as vivid as the fibers with a modern alum mordant. The iron mordant produced a dramatic deep purple hue on the animal fibers. There was also a noticeable brown rust on some of the animal fibers and especially on the plant fibers, which turned a brown color rather than purple. The color change in the plant fibers appeared to be mostly due to this rusty brown, which seemed to be more of a discoloration than a dye. The grainy rust content in the iron liquor contributed to this effect, and perhaps filtering the iron liquor before using it as a mordant, or using a more dilute concentration, would be advisable in future experiments.

Mordants were all prepared according to the methods outlined in Chapter 3 and samples of each mordant bath were photographed, assigned a Munsell color, and tested for pH (Fig. 4.16 and Appendix B2). Fiber samples from each of the four fiber types were treated with these mordants and dyed with Pakistani madder. Refer to Tables 4.13-4.16 (Appendix C/Tables C13-C16) for mordant experiment results. All images from this set of experiments can be found in Appendix B/Figs. B61-B84.

TABLE 4.12. COLOR AND pH OF DYE MORDANTS



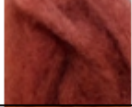



Mordant	pH	Color (Munsell)
Alum	5	Clear
Club Moss	6	5Y 8.5/4
Soapwort	6	10YR 7/6
Oak Galls	4	2.5Y 4/4
Iron Mordant	5	2.5YR 5/8

Fig. 4.16. Mordant baths. From left to right: Alum, club moss, soapwort, oak galls, iron liquor.


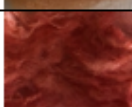



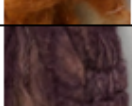


Fig. 4.17a-d. Prehistoric mordants on all fiber types. Fig. 4.17a. Upper left: club moss mordant on all fibers. Fig. 4.17b. Upper right: soapwort mordant on all fiber types. Fig. 4.17c. Lower left: oak gall mordant on all fiber types. Fig. 4.17d. Lower right: iron liquor mordant on all fiber types.


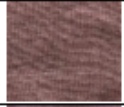




**TABLE 4.13. WOOL FIBERS WITH DIFFERENT MORDANTS**

Fiber Type	Thumbnail	pH/source	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Wool		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/8
Wool		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/8
Wool		7	62°/144°	1.5	Club Moss	Purchased Pakistani	7.5R 5/8
Wool		7	62°/144°	1.5	Soapwort	Purchased Pakistani	10R 5/10
Wool		4	62°/144°	1.5	Oak Galls	Purchased Pakistani	10R 4/8
Wool		5	62°/144°	1.5	Iron Liquor	Purchased Pakistani	7.5 4/2







**TABLE 4.14. SILK FIBERS WITH DIFFERENT MORDANTS**

Fiber Type	Thumbnail	pH/ source	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Silk		7	62°/144°	1.5	None	Purchased Pakistani	2.5YR 5/6
Silk		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 5/8
Silk		7	62°/144°	1.5	Club Moss	Purchased Pakistani	10R 6/6
Silk		7	62°/144°	1.5	Soapwort	Purchased Pakistani	5YR 5/6
Silk		4	62°/144°	1.5	Oak Galls	Purchased Pakistani	5YR 5/8
Silk		5	62°/144°	1.5	Iron Liquor	Purchased Pakistani	5R 3/2

**TABLE 4.15. LINEN FIBERS WITH DIFFERENT MORDANTS**

Fiber Type	Thumbnail	pH/ source	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Linen		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/4
Linen		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 6/4
Linen		7	62°/144°	1.5	Club Moss	Purchased Pakistani	10R 5/4
Linen		7	62°/144°	1.5	Soapwort	Purchased Pakistani	10R 6/4
Linen		4	62°/144°	1.5	Oak Galls	Purchased Pakistani	2.5YR 6/4
Linen		5	62°/144°	1.5	Iron Liquor	Purchased Pakistani	10R 5/2

**TABLE 4.16. NETTLE FIBERS WITH DIFFERENT MORDANTS**

Fiber Type	Thumbnail	pH/ source	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Nettle		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/4
Nettle		6	62°/144°	1.5	Alum	Purchased Pakistani	10R 5/6
Nettle		7	62°/144°	1.5	Club Moss	Purchased Pakistani	10R 4/4
Nettle		7	62°/144°	1.5	Soapwort	Purchased Pakistani	7.5R 4/4
Nettle		4	62°/144°	1.5	Oak Galls	Purchased Pakistani	10R 4/4
Nettle		5	62°/144°	1.5	Iron Liquor	Purchased Pakistani	10YR 4/2









## ***F. Wisconsin Madder Dye Baths: Results***

The harvest from two seasons of growth in the Wisconsin backyard plot provided very satisfying results. The dyed animal fibers were more vibrant and the colors more saturated than those dyed with the Pakistani madder. The mordanted plant fibers took the madder dye better and produced some of the reddest results obtained for either linen or nettle. The Wisconsin madder hues were more orange-red than the Pakistani madder, possibly because juvenile plants do not develop the full complement of dye compounds required for the really wonderful madder red. A colder climate may also inhibit the production of the madder red compounds. Further experiments and chemical analysis could test these ideas. Refer to Table 4.17 (Appendix C/Table C17) for complete homegrown madder dye results. All images from this experiment can be found in Appendix B (Figs. B85-B94).



Fig. 4.18a-b. Wisconsin madder on all fiber types. Fig. 4.18a. Left: unmordanted fibers. Fig. 4.18b. Right: mordanted fibers.

**TABLE 4.17. COMPARISON OF PAKISTANI AND WISCONSIN MADDER**

<b>Fiber Type</b>	<b>Thumbnail</b>	<b>pH/ source</b>	<b>Temp. C°/F°</b>	<b>Time (hours)</b>	<b>Mordant</b>	<b>Dye Source</b>	<b>Munsell</b>
Wool		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/8
Wool		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/8
Wool		7	62°/144°	1.5	None	Homegrown Wisconsin	10R 4/8
Wool		6	62°/144°	1.5	Alum	Homegrown Wisconsin	7.5R 4/12
Silk		7	62°/144°	1.5	None	Purchased Pakistani	2.5YR 5/6
Silk		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 5/8
Silk		7	62°/144°	1.5	None	Homegrown Wisconsin	2.5YR 6/8
Silk		6	62°/144°	1.5	Alum	Homegrown Wisconsin	7.5R 4/12

**TABLE 4.17. COMPARISON OF PAKISTANI AND WISCONSIN MADDER (continued)**

<b>Fiber Type</b>	<b>Thumbnail</b>	<b>pH/ Source</b>	<b>Temp (C°/F°)</b>	<b>Time (hours)</b>	<b>Mordant</b>	<b>Dye Source</b>	<b>Munsell</b>
Linen		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/4
Linen		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 6/4
Linen		7	62°/144°	1.5	None	Homegrown Wisconsin	7.5R 6/6
Linen		6	62°/144°	1.5	Alum	Homegrown Wisconsin	7.5R 5/6
Nettle		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/4
Nettle		6	62°/144°	1.5	Alum	Purchased Pakistani	10R 5/6
Nettle		7	62°/144°	1.5	None	Homegrown Wisconsin	7.5R 4/4
Nettle		6	62°/144°	1.5	Alum	Homegrown Wisconsin	7.5R 4/8

***Overall Summary:***

The overarching theme throughout these experiments was the incredible variability produced by the various madder dye baths. This idea has been expressed by almost everyone who has worked with this dye plant (Cardon 2007: 108; Dean 2014: 118; Goodwin 1982: 65;

Grierson 1986: 195), and my experiments further confirmed this. Obtaining consistent results is a challenge with this dye plant, as every variable can change the hue and saturation of the final product in spite of the fact that a controlled environment was maintained for all experiments in terms of consistent temperatures, known chemical and pH factors in the mordants, water and additives. I also either grew my own madder or purchased it from a reliable source, although the age of the purchased roots remained unknown. Madder trade was apparently not so reliable in the past, indicated by the fact that madder quality was regulated in historic times; methods of testing the quality of madder dye to avoid being cheated by inferior product are mentioned in the Papyrus *Graecus Holmiensis* (Caley 2008: 78). Ultimately, I wish I had found a modern madder source from Europe (or even the U.S.), or that I could expand this project to cultivate my own madder on a larger scale. Using dyestuffs that I had grown myself was immensely satisfying.

On a final note, I have to add that along with the satisfaction of producing my own dyestuffs is the constant impulse to experiment when dyeing fibers. Now that I have become an organic dyer, I notice this impulse in other publications by experienced dyers as well. Once I started using certain techniques, I immediately began thinking of ways to change things up and try new ideas. I have already begun planning other methods or combinations of variables to manipulate to see new results. The exciting thing about madder is also its most challenging aspect—it is always different with every dye bath, and this root lends itself extraordinarily well to experimentation. The variety of results that can be obtained from this one plant is immensely satisfying.

## **CHAPTER 5: SUMMARY AND FUTURE RESEARCH**

This research project was structured to test the viability of madder cultivation in central and northern Europe, and look for solid evidence of its existence in prehistoric Europe. The primary effort here, in both the experimental component and the review of literature on madder, was to ground-truth this plant: its botanical aspects, dye methods, and concrete archaeological evidence for its presence and use in prehistoric Europe. The success of the experimental component of this research suggests that *R. tinctorum* could certainly have been grown at least on a small scale in any region of Europe into southern Scandinavia. The growing body of research into ancient dyes has also helped plot the spread of madder usage into Europe. These two components of this project have contributed to the existing data base for the use of this important dye plant. Further work on the data generated by this project may allow the use of madder in Europe to be mapped in more detail as additional concrete evidence becomes available from systematically excavated prehistoric sites with textile materials.

Ultimately this research project focused on testing the variability of madder as a dye source, a variability that manifested itself on several levels. At the most fundamental level, madder root dye creates a wide variety of hues when its chemistry is manipulated by dye bath variables, as the experimental component of this project has demonstrated. As the dye baths using *R. tinctorum* clearly showed, a significant range of color changes resulted from manipulating different aspects of the dyeing process, including temperature, time, pH and the choice of mordant. As the dye experiments progressed, and the different techniques associated with madder became more familiar, it became clear that this set of experiments has provided the starting point for further investigations of a wide variety of madder-dyeing techniques that were employed historically and likely prehistorically. It would be interesting to take on the specific

recipes of “Egyptian Purple” and “Turkey Red”, for example, or a fermented pomegranate mordant, or some of the more esoteric recipes listed in the Stockholm Papyrus. There appear to be abundant possibilities for future dye experiments: further experimentation with mordants, exploration of other ancient recipes, and the use of other wild plants and insect dyes as dye sources. A deeper exploration of the color red, its sources and cultural importance is also warranted.

Another aspect of the use of madder that surfaced in the course of this research is more semantic in nature. In one sense, this variability involves color, and the problematic descriptions of color. Taylor (1999) has pointed out the subjectivity of color terms in defining “red” and “purple” and the wide variety of hues that these two words can represent. Munsell himself also was concerned with the subjectivity of color definition, and the standardization of color through the development of Munsell charts was an effort to resolve this problem. But the problem of the many shades (or words) for red remains. What is a true red? Is it the same for everyone? Is a true red really a purple for some? It seems that the spectrum of colors between red and purple exhibits an innate variability. This makes it very problematic to take historic descriptions of color as givens. As every good anthropologist knows, based on their recollections of Sapir-Whorf, color perception can be affected by culture and language. Prehistoric Europe was far from being a culturally or linguistically homogenous area. Could dye usage and color choice have been affected by this phenomenon? Even in Classical and Egyptian written accounts, which are relied upon heavily as source material here, there are wide gaps in terms of time, culture and linguistics. My own modern idea of these hues may—pardon the expression—color my own view of what “red” really is.

Another semantic variability issue has emerged in this thesis as an obstacle to seeing the trajectory of the presence and use of the madder plant in prehistoric Europe. Descriptions of dyes on textiles have been documented since at least the turn of the last century. Madder is frequently identified as the source of a red colorant on some textiles with no clear evidence to support those claims. A claim for “madder” implies *R. tinctorum*, yet this is not supplemented by supporting recent dye analysis except in a few exceptional cases. While this determination may have been correct for textiles in Egypt, the Classical World and the other parts of the Near East, it has created problems for early European textiles. As the dye analyses of the Hallstatt textiles, the Hochdorf textiles and others have shown, “madder” (*R. tinctorum*) was not present. The presence of kermes dye at Hochdorf or Hallstatt is not a surprise, but the absence of alizarin is significant. The presence of purpurin in these same dye analyses possibly indicates that wild madder or bedstraws rather than *R. tinctorum* were used as red dyes well into Late Roman or Migration Period Europe. Cardon and Hofenk de Graaff have raised questions about certain dye processes, and whether these could result in changes to the chemical signature of some dye plants, removing evidence of alizarin in textiles dyed with *R. tinctorum* (Cardon 2007: 123; Hofenk de Graaff and van Bommel 2004: 109-110). Chemical analysis of fibers subjected to a variety of madder dye methods might prove useful in answering that question. The dye experiments also posed another question regarding ancient madder dyeing: how similar is the *R. tinctorum* grown now to the dyer’s madder of prehistoric Europe? A millennium of madder cultivation separates the Celts from modern large-scale madder cultivation. Given changes in agricultural and horticultural practices, there would almost certainly have been some refinements of desirable traits in all those years, including changes in the cold hardiness of the plants. Can we know what those changes might have been? Would they have affected the chemical makeup

of the dye elements in the plant over time? Would the growth habits of the plant have been affected in other ways, including root quantity and a change from an upright to a creeping habit?

Based on the chemical data available at this time, however, it appears that *R. tinctorum* was not in use as a dyestuff in northern and central Europe until historic times. Wild madder, *R. peregrina*, or possibly the bedstraw plant group, seem to have been the primary sources of red for domestically dyed textiles into historic times. This hypothesis is supported by Scottish legislation passed in 1695 to prevent over-harvesting of wild bedstraws by local dyers, a practice which caused significant erosion to precious topsoil along the Scottish coast (Grierson 1986: 76). There seems to have been a preference for the bedstraw reds at that time, which also may have held true for earlier European dyers. At this point, the evidence suggests that kermes was known as a source of red from at least the Neolithic in parts of Europe and was an elite dye. If madder was not generally available as a substitute for the exclusive and expensive kermes, this has implications for the possible existence of sumptuary restrictions on the use of true red cloth and clothing in pre-Roman Iron Age Europe and affects the interpretation of elite contexts in which kermes red is found. Further experiments comparing kermes red and madder red could determine whether madder red comes closest to the true red of kermes dye. If so, madder would certainly have been sought after if it had been available. There is evidence for what has been called competitive emulation among secondary elites in Iron Age Europe (Banck-Burgess 2012) and the area of textiles and dress is still very under-theorized in this discussion. Future investigations into dye analyses of other sources for red are planned.

Madder-type plants, such as wild madder and bedstraws, seem to have been in use at an early stage in the development of European textiles. Dyer's madder appears to have been introduced at a much later time. The wild madders and bedstraws may never have really fallen

out of favor as dye plants, but as *R. tinctorum* cultivation grew into a large-scale enterprise from the Middle Ages on, it is likely that cultivated madder was easier to obtain than wild-gathered plants. As the experimental component demonstrated, it took two years and nine mature dyer's madder plants, to generate enough root material for one large dye bath. Historic accounts of wild-gathered bedstraws suggest that one or two sacks full of roots made one dye bath, and better colors were obtained from older plants (Grierson 1986: 76). This suggests that while true madder red would not have been as exclusive as kermes red in prehistoric Europe, it would certainly have ranked as an uncommon color and a valuable dye source. It is also possible that an increased availability of alum may have played a part in the growth of madder usage, and an investigation into the development of an alum trade in Europe could yield information on dye usage as well. The development of the madder or alum trade, and the increased availability of red dye may have affected the status of red, and the status of madder as it became less restricted. A more in-depth analysis of the development of madder in early historic times to address these ideas is also planned.

Research into archaeological textile dyes also needs to be expanded. As more dyes are analyzed, particularly from non-elite contexts, the use of cultivated madder versus wild madder may be revealed. The research in this thesis concerning archaeological textiles stopped at the end of the Roman era, but it appears that this was just when dyer's madder began to make an appearance in central and northern Europe. It is likely that an expansion of the research scope into Anglo-Saxon and Viking Age textiles will give a more complete picture of the development of *R. tinctorum* as an important dye plant in Europe. Perhaps *R. tinctorum* never became a source for the choicest red in early Europe, as there is evidence that both kermes and *R. peregrina* and the bedstraws were preferred dyestuffs, although for different reasons, and likely

used by different social strata. This is speculative, however; it is clearly too early in the investigation of prehistoric reds to determine the status of dyer's madder in prehistoric Europe. This thesis serves as a starting point for a more in-depth investigation into the prehistory of this plant that could include chemical analysis as well as a comparison of the use and distribution of madder with the other important dye plant, woad. As both the research and the dye experiments unfolded, new ideas and avenues of exploration were revealed. Future research is already being planned to further investigate the use and spread of *R. tinctorum* in Europe, and the potential effects that an easily obtainable source of a red textile dye would have had on European societies in the transition into the historic era.

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## **APPENDIX A: ONLINE SOURCES**

From Chapter 3: Methods

- A.1. Wild Colours Natural Dyes website at <http://www.wildcolours.co.uk/index.html>
- A.2. Strictly Medicinal Seed website at <https://strictlymedicalseeds.com/>
- A.3. The Woolery website at <https://woolery.com/>
- A.4. Vilenda Craft Etsy site at <https://www.etsy.com/shop/VILENDACraft>
- A.5. Woolfinch Studios Etsy site at <https://www.etsy.com/shop/WoolFinchStudio>
- A.6. Shepswool website at <https://sheps-wool.myshopify.com/>
- A.7. Luthvarian website at <https://www.etsy.com/shop/luthvarian>
- A.8. The Woolery website at <https://woolery.com/>
- A.9. The Woolery website at <https://woolery.com/>
- A.10. Natures Curios Etsy shop at <https://www.etsy.com/shop/Naturescurios>
- A.11. Cupid Falls Etsy shop at <https://www.etsy.com/shop/CupidFalls>
- A.12. Raven Moon Emporium Etsy shop at <https://www.etsy.com/shop/RavenMoonEmporium>
- A.13. Gifts from Nature Etsy shop at <https://www.etsy.com/shop/GIFTfromNATURE>

## **APPENDIX B: DYE BATH RESULTS IMAGES**



Fig. B1. Untreated examples of the experiment fiber types.

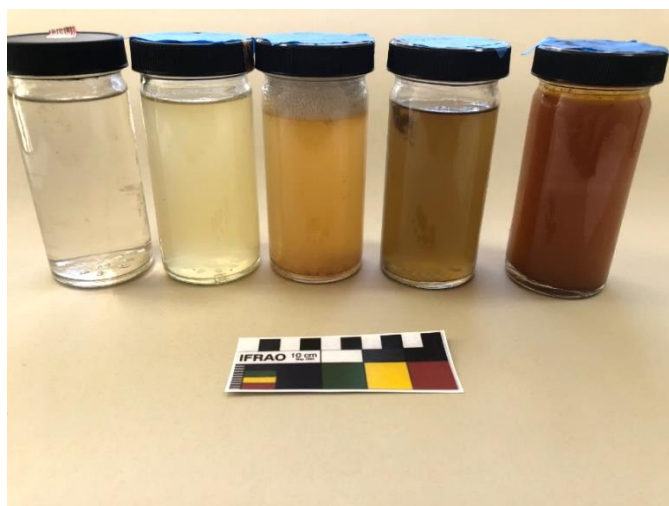


Fig. B2. Mordants, from left to right: Alum, Club Moss, Soapwort, Oak Galls, Iron Liquor.

### *Alum Mordant Experiments:*



Fig. B3. Wool samples, dyed with and without an alum mordant.



Fig. B4. Silk samples, dyed with and without an alum mordant.



Fig. B5. Linen samples, dyed with and without an alum mordant.



Fig. B6. Nettle samples, dyed with and without an alum mordant.

### *Wool fibers: Temperature Experiments*



Fig. B7. Unmordanted wool samples at different temperatures



Fig. B8. Mordanted wool samples at varying temperatures.



Fig. B9. Cold dye bath wool samples, with and without a mordant.



Fig. B10. Warm dye bath wool samples, with and without a mordant.



Fig. B11. Boiled dye bath wool samples, with and without a mordant.

### *Silk fibers: Temperature Experiments*



Fig. B12. Silk fibers with no mordant, in dyes baths at different temperatures.

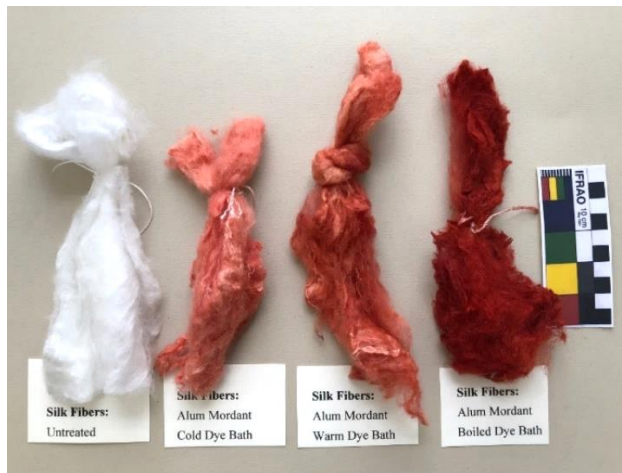


Fig. B13. Silk fibers with an alum mordant, at different temperatures.

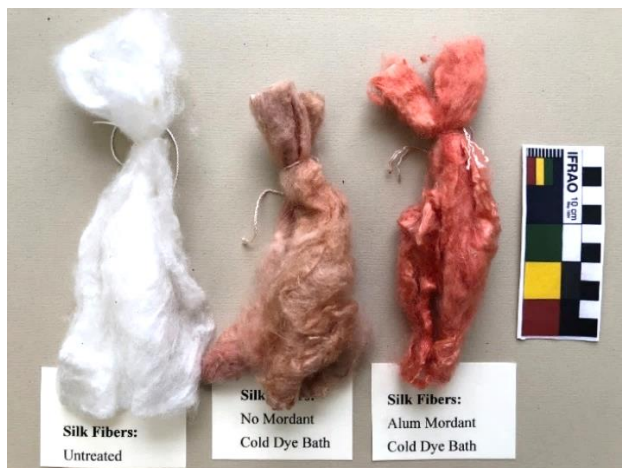


Fig. B14. Silk fibers in the cold dye bath, with and without a mordant.



Fig. B15. Silk fibers in the warm dye bath, with and without a mordant.



Fig. B16. Silk fibers in the boiled dye bath, with and without a mordant.

### ***Linen Fibers: Temperature Experiments***



Fig. B17. Linen fabric with no mordant, from dye baths at different temperatures.



Fig. B18. Linen fabric with an alum mordant, from dye baths at different temperatures.



Fig. B19. Linen fibers from a cold dye bath, with and without a mordant.



Fig. B20. Linen fabric from a warm dye bath, with and without a mordant.



Fig. B21. Line fabric from a boiled dye bath, with and without a mordant.

### *Nettle Fibers: Temperature Experiments*



Fig. B22. Nettle fabric with no mordant, from dye baths at different temperatures.



Fig. B23. Nettle fibers with alum mordant, from dye baths at different temperatures.



Fig. B24. Nettle fibers from a cold dye bath, with and without a mordant.



Fig. B25. Nettle fabric from the warm dye bath, with and without a mordant.



Fig. B26. Nettle fabrics from a boiled dye bath, with and without a mordant.

## Wool: pH Experiment Results



Fig. B27. Wool fibers with no mordant from dye baths of different pH.



Fig. B28. Wool fiber samples with alum mordants from dye baths of different pH.



Fig. B29. Wool fibers with acetic acid, with and without a mordant.



Fig. B30. Wool fibers with wood ash water, with and without a mordant.



Fig. B31. Wool fibers with dilute ammonia, with and without a mordant.

### ***Silk Fibers: pH Experiment Results***

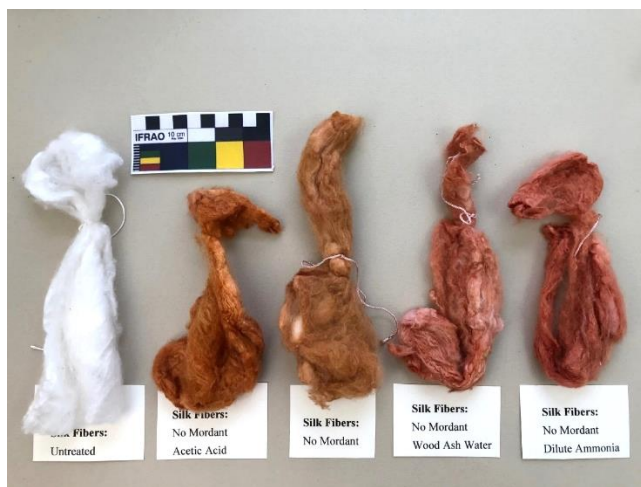


Fig. B32. Silk fibers with no mordant from dye baths of different pH



Fig. B33. Silk fibers with an alum mordant from dye baths of different pH.



Fig. B34. Silk fibers dyed in acetic acid dye bath, with and without a mordant.



Fig. B35. Silk fibers in wood ash water dye bath, with and without a mordant.



Fig. B36. Silk fibers in dilute ammonia dye bath, with and without ammonia.

### *Linen Fibers: pH Experiment Results*



Fig. B37. Linen fibers with no mordant from dye baths of different pH.

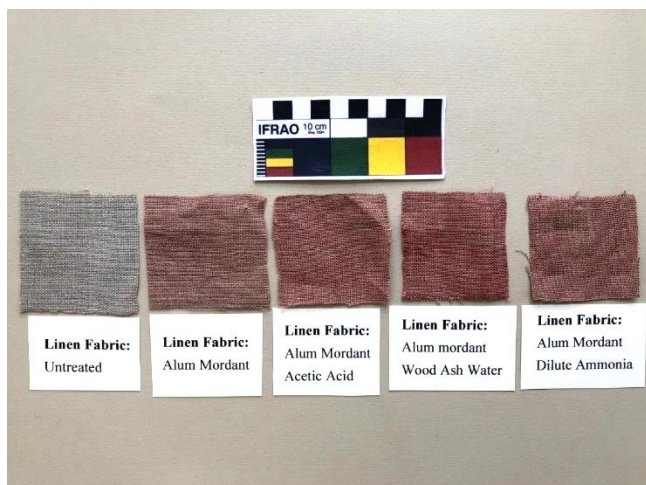


Fig. B38. Linen fibers with an alum mordant from dye baths at different pH.



Fig. B39. Linen fibers in acetic acid bath, with and without a mordant.



Fig. B40. Linen fibers in wood ash water bath, with and without a mordant.



Fig. B41. Linen fibers in a dilute ammonia bath, with and without a mordant.

### *Nettle Fibers: pH Experiment Results*



Fig. B42. Nettle fibers with no mordant from dye baths of different pH

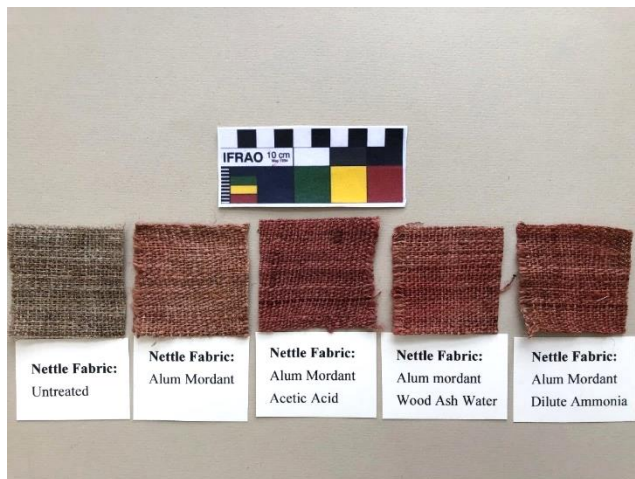


Fig. B43. Nettle fibers with an alum mordant from dye baths of different pH.



Fig. B44. Nettle fibers in an acetic acid dye bath, with and without a mordant.



Fig. B45. Nettle fibers in a wood ash water dye bath, with and without a mordant.



Fig. B46. Nettle fibers in a dilute ammonia dye bath, with and without a mordant.

### ***Timed Dye Bath Results:***



Fig. B47. All fiber types with no mordant, from the 0.5-hour timed dye bath.



Fig. B48. All fiber types with an alum mordant, from the 0.5-hour timed dye bath.



Fig. B49. All fiber types with no mordant, from the 12-hour timed dye bath.



Fig. B50. All fiber types with an alum mordant, from the 12-hour timed dye bath.

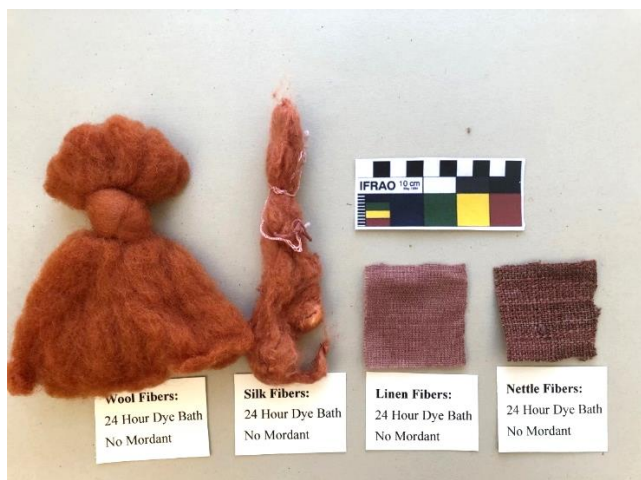


Fig. B51. All fiber types with no mordant, from the 24-hour timed dye bath.



Fig. B52. All fiber types with an alum mordant, from the 12-hour timed dye bath.

### ***Wool Fibers in Timed Dye Baths:***



Fig. B53. Wool fibers with no mordant, from dye baths of different time length.



Fig. B54. Wool fibers with alum mordant, from dye baths of different time length.

***Silk Fibers in Timed Dye Baths:***



Fig. B55. Silk fibers with no mordant, from dye baths of different time length.

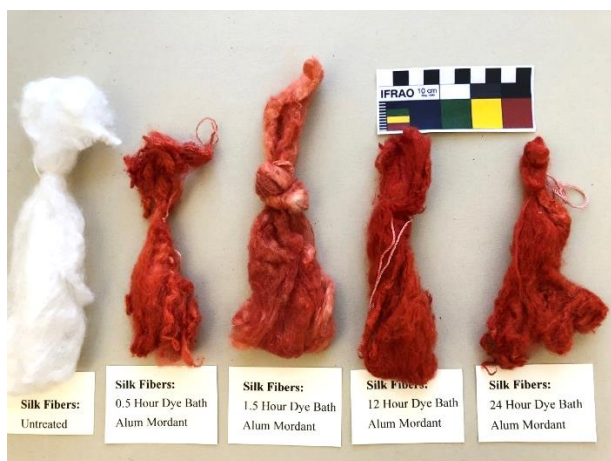


Fig. B56. Silk fibers with alum mordant, from dye baths of different time length.

### *Linen Fibers in Timed Dye Baths:*

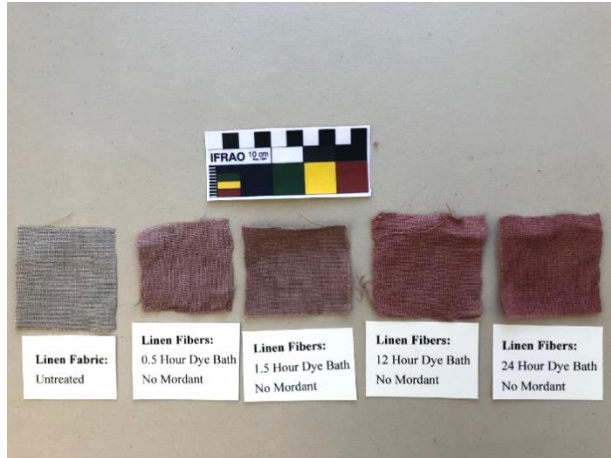


Fig. B57. Linen fibers with no mordant, from dye baths of different time length.

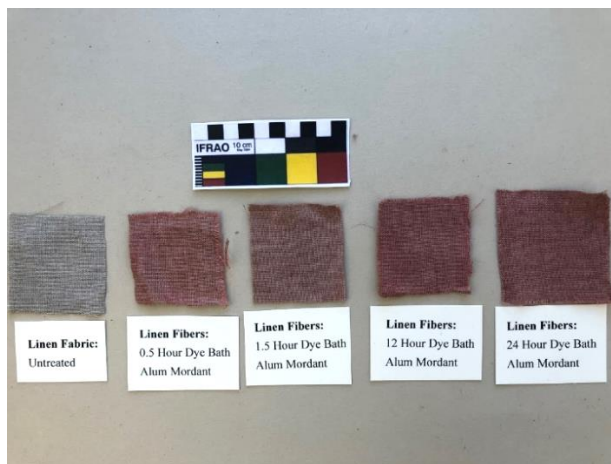


Fig. B58. Linen fibers with alum mordant, from dye baths of different time length.

### *Nettle Fibers in Timed Dye Baths:*



Fig. B59. Nettle fibers with no mordant, from dye baths of different time length.



Fig. B60. Nettle fibers with alum mordant, from dye baths of different time length.

***Mordants on All Fiber Types:***



Fig. B61. Alum mordant on all four fiber types.



Fig. B62. Club moss mordant on all four fiber types.



Fig. B63. Soapwort mordant on all four fiber types.



Fig. B64. Oak gall mordant on all four fiber types.



Fig. B65. Iron Liquor mordant on all four fiber types.

***Wool Fibers with Different Mordants:***



Fig. B66. Wool fibers with no mordant and a club moss mordant.



Fig. B67. Wool fibers with no mordant and a soapwort mordant.

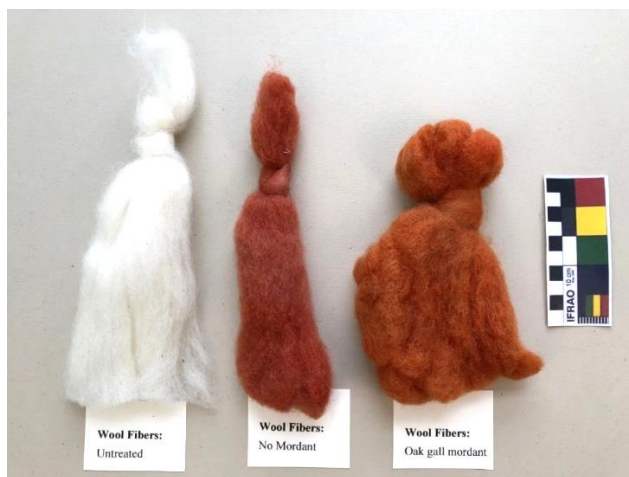


Fig. B68. Wool fibers with no mordant and an oak gall mordant.



Fig. B69. Wool fibers with no mordant and an iron liquor mordant.

***Silk Fibers with Different Mordants:***



Fig. B70. Silk fibers with an alum mordant.



Fig. B71. Silk fibers with a club moss mordant.



Fig. B72. Silk fibers with a soapwort mordant.



Fig. B73. Silk fibers with an oak gall mordant.



Fig. B74. Silk fibers with an iron liquor mordant.

*Linen Fibers with Different Mordants:*



Fig. B75. Linen fibers with no mordant and an alum mordant.

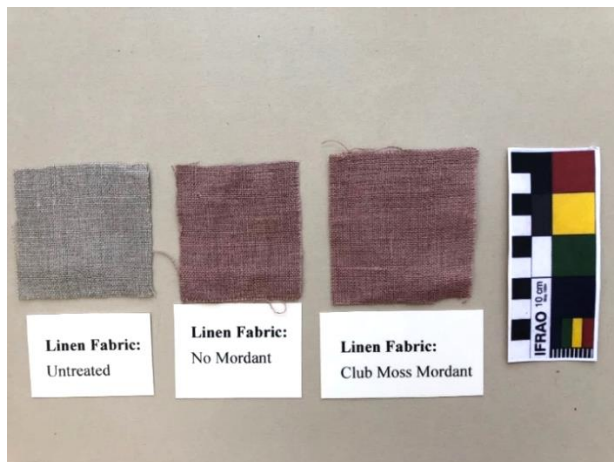


Fig. B76. Linen fibers with no mordant and a club moss mordant.



Fig. B77. Linen fibers with no mordant and a soapwort mordant.



Fig. B78. Linen fibers with no mordant and an oak gall mordant.



Fig. B79. Linen fibers with no mordant and an iron liquor mordant.

### ***Nettle Fibers with Different Mordants:***



Fig. B80. Nettle fibers with no mordant and an alum mordant.



Fig. B81. Nettle fibers with no mordant and a club moss mordant.



Fig. B82. Nettle fibers with no mordant and a soapwort mordant.



Fig. B83. Nettle fibers with no mordant and an oak gall mordant.



Fig. B84. Nettle fibers with no mordant and an iron liquor mordant.

### ***Wisconsin Madder Dye Experiments:***



Fig. B85. All fiber types with Wisconsin madder dye and no mordant.



Fig. B86. All fiber types with Wisconsin madder dye and an alum mordant.



Fig. B87. Wisconsin madder on unmordanted wool.



Fig. B88. Wisconsin madder on mordanted wool.



Fig. B89. Wisconsin madder on unmordanted silk.



Fig. B90. Wisconsin madder on mordanted silk.



Fig. B91. Wisconsin madder on unmordanted linen.



Fig. B92. Wisconsin madder on mordanted linen.




Fig. B93. Wisconsin madder on unmordanted nettle.



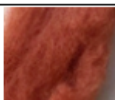

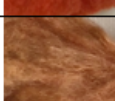





Fig. B94. Wisconsin madder on mordanted nettle.

## **APPENDIX C: DYE BATH TABLES**

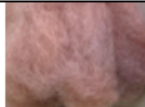

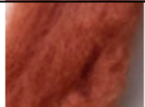






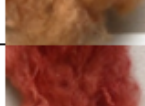

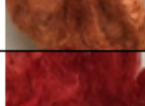
**TABLE C1. UNTREATED FIBERS**

Fiber Type	Thumbnail	Munsell
Wool		5Y 8.5
Silk		White
Linen		10YR 6/2
Nettle		10YR 5/2



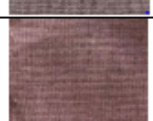
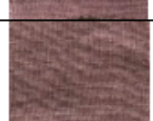








**TABLE C2. FIBERS SAMPLE WITH AND WITHOUT AN ALUM MORDANT**

Fiber type	Thumbnail	pH	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Wool		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/8
Wool		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/8
Silk		7	62°/144°	1.5	None	Purchased Pakistani	10R 6/6
Silk		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 5/8
Linen		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/4
Linen		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 6/4
Nettle		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/4
Nettle		6	62°/144°	1.5	Alum	Purchased Pakistani	10R 5/6

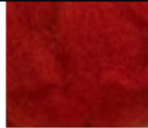


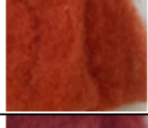


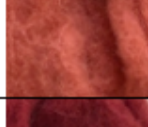

**TABLE C3. TEMPERATURE EXPERIMENTS WITH MADDER DYE BATHS**

Fiber type	Thumbnail	pH	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Wool		7	21°/70°	1.5	None	Purchased Pakistani	7.5R 6/4
Wool		6	21°/70°	1.5	Alum	Purchased Pakistani	7.5R 6/8
Wool		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/8
Wool		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/8
Wool		7	100°/212°	1.5	None	Purchased Pakistani	10R 4/8
Wool		6	100°/212°	1.5	Alum	Purchased Pakistani	10R 4/10
Silk		7	21°/70°	1.5	None	Purchased Pakistani	10R 6/4
Silk		6	21°/70°	1.5	Alum	Purchased Pakistani	7.5R 6/8
Silk		7	62°/144°	1.5	None	Purchased Pakistani	2.5YR 5/6
Silk		6	62°/144°	1.5	Alum	Purchased Pakistani	5R 5/8
Silk		7	100°/212°	1.5	None	Purchased Pakistani	2.5YR 5/8
Silk		6	100°/212°	1.5	Alum	Purchased Pakistani	7.5R 3/8




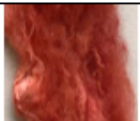




**TABLE C3. TEMPERATURE EXPERIMENTS WITH MADDER DYE BATHS (continued)**

<b>Fiber Type</b>	<b>Thumbnail</b>	<b>pH</b>	<b>Temp. C°/F°</b>	<b>Time (hours)</b>	<b>Mordant</b>	<b>Dye Source</b>	<b>Munsell</b>
Linen		7	21°/70°	1.5	None	Purchased Pakistani	7.5R 7/2
Linen		6	21°/70°	1.5	Alum	Purchased Pakistani	7.5R 6/2
Linen		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/4
Linen		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 6/4
Linen		7	100°/212°	1.5	None	Purchased Pakistani	7.5R 5/4
Linen		6	100°/212°	1.5	Alum	Purchased Pakistani	7.5R 5/6
Nettle		7	21°/70°	1.5	None	Purchased Pakistani	2.5YR 5/2
Nettle		6	21°/70°	1.5	Alum	Purchased Pakistani	2.5YR 5/4
Nettle		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/4
Nettle		6	62°/144°	1.5	Alum	Purchased Pakistani	10R 5/6
Nettle		7	100°/212°	1.5	None	Purchased Pakistani	7.5R5/4
Nettle		6	100°/212°	1.5	Alum	Purchased Pakistani	7.5R 4/6

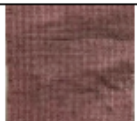




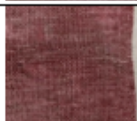
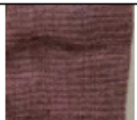

**TABLE C4. DYE BATH pH EXPERIMENTS ON WOOL**

Fiber type	Thumbnail	pH/ source	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Wool		4 Acetic Acid	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/10
Wool		4 Acetic Acid	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/12
Wool		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/8
Wool		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/8
Wool		9 <u>WoodAsh</u> Water	62°/144°	1.5	None	Purchased Pakistani	5R 3/6
Wool		9 Wood Ash Water	62°/144°	1.5	Alum	Purchased Pakistani	5R 3/8
Wool		10 Dilute Ammonia	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/8
Wool		10 Dilute Ammonia	62°/144°	1.5	Alum	Purchased Pakistani	5R 3/8




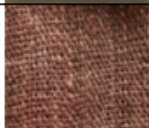



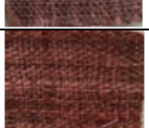
**TABLE C5. DYE BATH pH EXPERIMENTS ON SILK**

Fiber Type	Thumbnail	pH/source	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Silk		4 Acetic Acid	62°/144°	1.5	None	Purchased Pakistani	10R 5/8
Silk		4 Acetic Acid	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 3/10
Silk		7	62°/144°	1.5	None	Purchased Pakistani	2.5YR 5/6
Silk		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 5/8
Silk		9 Wood Ash Water	62°/144°	1.5	None	Purchased Pakistani	7.5R 6/6
Silk		9 Wood Ash Water	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/10
Silk		10 Dilute Ammonia	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/8
Silk		10 Dilute Ammonia	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 5/10


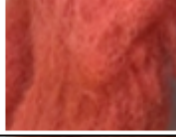






**TABLE C6. DYE BATH pH EXPERIMENTS ON LINEN**

Fiber Type	Thumbnail	pH/source	Temp. C°/F°	Time (hours.)	Mordant	Dye Source	Munsell
Linen		4 Acetic Acid	62°/144°	1.5	None	Purchased Pakistani	7.5 6/4
Linen		4 Acetic Acid	62°/144°	1.5	Alum	Purchased Pakistani	7.5 5/4
Linen		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/4
Linen		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 6/4
Linen		9 Wood Ash Water	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/4
Linen		9 Wood Ash Water	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 5/6
Linen		10 Dilute Ammonia	62°/144°	1.5	None	Purchased Pakistani	7.5 5/4
Linen		10 Dilute Ammonia	62°/144°	1.5	Alum	Purchased Pakistani	7.5 5/4

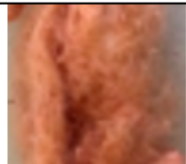
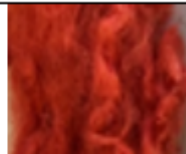


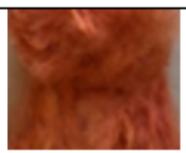
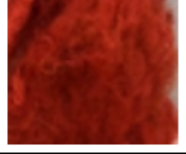


**TABLE C7. DYE BATH pH EXPERIMENTS ON NETTLE**

Fiber Type	Thumbnail	pH/source	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Nettle		4 Acetic Acid	62°/144°	1.5	None	Purchased Pakistani	10R 4/4
Nettle		4 Acetic Acid	62°/144°	1.5	Alum	Purchased Pakistani	7.5 4/6
Nettle		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/4
Nettle		6	62°/144°	1.5	Alum	Purchased Pakistani	10R 5/6
Nettle		9 Wood Ash Water	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/4
Nettle		9 Wood Ash Water	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/6
Nettle		10 Dilute Ammonia	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/4
Nettle		10 Dilute Ammonia	62°/144°	1.5	Alum	Purchased Pakistani	7.5 4/6


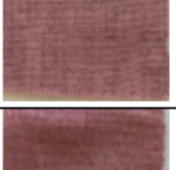
**TABLE C8. WOOL FIBERS IN DYE BATHS OF VARYING DURATION**

Fiber Type	Thumbnail	pH/ source	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Wool		7	62°/144°	0.5	None	Purchased Pakistani	10R 6/8
Wool		6	62°/144°	0.5	Alum	Purchased Pakistani	7.5R 6/12
Wool		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/8
Wool		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/8
Wool		7	62°/144°	12.0	None	Purchased Pakistani	10R 5/10
Wool		6	62°/144°	12.0	Alum	Purchased Pakistani	10R 4/12
Wool		7	62°/144°	24.0	None	Purchased Pakistani	10R 4/10
Wool			62°/144°	24.0	Alum	Purchased Pakistani	10R 4/12





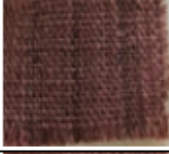


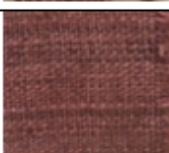
**TABLE C9. SILK FIBERS IN DYE BATHS OF VARYING DURATION**

<b>Fiber Type</b>	<b>Thumbnail</b>	<b>pH/ source</b>	<b>Temp. C°/F°</b>	<b>Time (hours)</b>	<b>Mordant</b>	<b>Dye Source</b>	<b>Munsell</b>
Silk		7	62°/144°	0.5	None	Purchased Pakistani	10R 6/8
Silk		6	62°/144°	0.5	Alum	Purchased Pakistani	7.5R 5/10
Silk		7	62°/144°	1.5	None	Purchased Pakistani	2.5YR 5/6
Silk		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 5/8
Silk		7	62°/144°	12.0	None	Purchased Pakistani	10R 5/8
Silk		6	62°/144°	12.0	Alum	Purchased Pakistani	7.5R 4/10
Silk		7	62°/144°	24.0	None	Purchased Pakistani	10R 5/8
Silk		6	62°/144°	24.0	Alum	Purchased Pakistani	7.5R 4/10

**TABLE C10. LINEN FIBERS IN DYE BATHS OF VARYING DURATION**

<b>Fiber Type</b>	<b>Thumbnail</b>	<b>pH/ source</b>	<b>Temp. C°/F°</b>	<b>Time (hours)</b>	<b>Mordant</b>	<b>Dye Source</b>	<b>Munsell</b>
Linen		7	62°/144°	0.5	None	Purchased Pakistani	7.5R 5/4
Linen		6	62°/144°	0.5	Alum	Purchased Pakistani	7.5R 6/6
Linen		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/4
Linen		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 6/4
Linen		7	62°/144°	12.0	None	Purchased Pakistani	7.5R 5/6
Linen		6	62°/144°	12.0	Alum	Purchased Pakistani	7.5R 5/6
Linen		7	62°/144°	24.0	None	Purchased Pakistani	7.5R 5/6
Linen		6	62°/144°	24.0	Alum	Purchased Pakistani	7.5R 5/6







**TABLE C11. NETTLE FIBERS IN DYE BATHS OF VARYING DURATION**

<b>Fiber Type</b>	<b>Thumbnail</b>	<b>pH/ source</b>	<b>Temp. C°/F°</b>	<b>Time (hours)</b>	<b>Mordant</b>	<b>Dye Source</b>	<b>Munsell</b>
Nettle		7	62°/144°	0.5	None	Purchased Pakistani	7.5R 4/4
Nettle		6	62°/144°	0.5	Alum	Purchased Pakistani	7.5 5/8
Nettle		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/4
Nettle		6	62°/144°	1.5	Alum	Purchased Pakistani	10R 5/6
Nettle		7	62°/144°	12.0	None	Purchased Pakistani	7.5R 4/6
Nettle		6	62°/144°	12.0	Alum	Purchased Pakistani	7.5R 4/8
Nettle		7	62°/144°	24.0	None	Purchased Pakistani	7.5R 4/6
Nettle		6	62°/144°	24.0	Alum	Purchased Pakistani	7.5R 5/8







**TABLE C12: PH AND COLOR OF DYE MORDANTS**

Mordant	pH	Color (Munsell)
Alum	5	Clear
Club Moss	6	5Y 8.5/4
Soapwort	6	10YR 7/6
Oak Galls	4	2.5Y 4/4
Iron Mordant	5	2.5YR 5/8



**TABLE C13. WOOL FIBERS WITH DIFFERENT MORDANTS**

Fiber Type	Thumbnail	pH/source	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Wool		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/8
Wool		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/8
Wool		7	62°/144°	1.5	Club Moss	Purchased Pakistani	7.5R 5/8
Wool		7	62°/144°	1.5	Soapwort	Purchased Pakistani	10R 5/10
Wool		4	62°/144°	1.5	Oak Galls	Purchased Pakistani	10R 4/8
Wool		5	62°/144°	1.5	Iron Liquor	Purchased Pakistani	7.5 4/2







**TABLE C.14. SILK FIBERS WITH DIFFERENT MORDANTS**

Fiber Type	Thumbnail	pH/ source	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Silk		7	62°/144°	1.5	None	Purchased Pakistani	2.5YR 5/6
Silk		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 5/8
Silk		7	62°/144°	1.5	Club Moss	Purchased Pakistani	10R 6/6
Silk		7	62°/144°	1.5	Soapwort	Purchased Pakistani	5YR 5/6
Silk		4	62°/144°	1.5	Oak Galls	Purchased Pakistani	5YR 5/8
Silk		5	62°/144°	1.5	Iron Liquor	Purchased Pakistani	5R 3/2

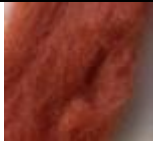







**TABLE C.15. LINEN FIBERS WITH DIFFERENT MORDANTS**

Fiber Type	Thumbnail	pH/ source	Temp. C°/F°	Time (hours)	Mordant	Dye Source	Munsell
Linen		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/4
Linen		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 6/4
Linen		7	62°/144°	1.5	Club Moss	Purchased Pakistani	10R 5/4
Linen		7	62°/144°	1.5	Soapwort	Purchased Pakistani	10R 6/4
Linen		4	62°/144°	1.5	Oak Galls	Purchased Pakistani	2.5YR 6/4
Linen		5	62°/144°	1.5	Iron Liquor	Purchased Pakistani	10R 5/2





**TABLE C16. NETTLE FIBERS WITH DIFFERENT MORDANTS**

<b>Fiber Type</b>	<b>Thumbnail</b>	<b>pH/ source</b>	<b>Temp. C°/F°</b>	<b>Time (hours)</b>	<b>Mordant</b>	<b>Dye Source</b>	<b>Munsell</b>
Nettle		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/4
Nettle		6	62°/144°	1.5	Alum	Purchased Pakistani	10R 5/6
Nettle		7	62°/144°	1.5	Club Moss	Purchased Pakistani	10R 4/4
Nettle		7	62°/144°	1.5	Soapwort	Purchased Pakistani	7.5R 4/4
Nettle		4	62°/144°	1.5	Oak Galls	Purchased Pakistani	10R 4/4
Nettle		5	62°/144°	1.5	Iron Liquor	Purchased Pakistani	10YR 4/2

**TABLE C18. COMPARISON OF PAKISTANI AND WISCONSIN MADDER**

<b>Fiber Type</b>	<b>Thumbnail</b>	<b>pH/ source</b>	<b>Temp. C°/F°</b>	<b>Time (hours)</b>	<b>Mordant</b>	<b>Dye Source</b>	<b>Munsell</b>
Wool		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/8
Wool		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 4/8
Wool		7	62°/144°	1.5	None	Homegrown Wisconsin	10R 4/8
Wool		6	62°/144°	1.5	Alum	Homegrown Wisconsin	7.5R 4/12
Silk		7	62°/144°	1.5	None	Purchased Pakistani	2.5YR 5/6
Silk		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 5/8
Silk		7	62°/144°	1.5	None	Homegrown Wisconsin	2.5YR 6/8
Silk		6	62°/144°	1.5	Alum	Homegrown Wisconsin	7.5R 4/12

**TABLE C18. COMPARISON OF PAKISTANI AND WISCONSIN MADDER (continued)**

<b>Fiber Type</b>	<b>Thumbnail</b>	<b>pH/ Source</b>	<b>Temp (C°/F°)</b>	<b>Time (hours)</b>	<b>Mordant</b>	<b>Dye Source</b>	<b>Munsell</b>
Linen		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 5/4
Linen		6	62°/144°	1.5	Alum	Purchased Pakistani	7.5R 6/4
Linen		7	62°/144°	1.5	None	Homegrown Wisconsin	7.5R 6/6
Linen		6	62°/144°	1.5	Alum	Homegrown Wisconsin	7.5R 5/6
Nettle		7	62°/144°	1.5	None	Purchased Pakistani	7.5R 4/4
Nettle		6	62°/144°	1.5	Alum	Purchased Pakistani	10R 5/6
Nettle		7	62°/144°	1.5	None	Homegrown Wisconsin	7.5R 4/4
Nettle		6	62°/144°	1.5	Alum	Homegrown Wisconsin	7.5R 4/8