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# Ultrasound and Microwave Aided Natural Dyeing of Nettle Biofibre (*Urtica dioica* L.) with Madder (*Rubia tinctorum* L.)

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**Abstract**
*In this study, eco-friendly 100% nettle bast bio-fibre fabric was dyed with eco-friendly natural dye, madder (Rubia tinctorum L.), using alternative dyeing methods such as ultrasound and microwave energy. Dyeings were performed with conventional-exhaustion, ultrasound and microwave methods using green tea and alum as mordants. The colorimetric, colour fastness properties and FTIR-ATR spectra of the nettle-biofibres dyed were investigated. Although the colour strengths of the microwave and ultrasound energy-aided dyeings were not as high as in conventional dyeings, the usage of microwave-energy in dyeing is important in terms of shortening the process time. All dyed nettle fabrics exhibited very high and commercially acceptable wash, dry-rub, alkaline-perspiration, acidic-perspiration and water fastness properties. Overall the conventional and ultrasound dyeing methods caused slightly higher light fastness than the microwave assisted dyeing method.*

**Key words:** *nettle biofibre, microwave, natural dyeing, Rubia tinctorum L., ultrasound.*

## Introduction

Nettles belong to the Urticaceae family, which consists of various species (***Figure 1***). They grow widely in tropical or sub-tropical areas [1], and the temperate climate regions of Europe, Asia and America are suitable for growth of the plant [2]. There are one-year and perennial species of the nettle plant [1]. Perennial species are claimed to be available for fibre production for 10-15 years; however, four years cultivation is suitable for maximum yield [2]. Textile usage of the nettle plant is not a fresh idea; in fact, it has been used throughout history for textile purposes [1, 3, 4]. Ancient Egyptians, Greeks and Romans used nettle fibre as a textile raw material [5, 6]. It is known that nettle was cultivated in Scandinavia for textile production [3]. Also fishing net and sail applications of nettle fibre were reported [4]. In recent history, during World War I, German army used military equipment made from nettle fibres [5, 7].

The nettle plant can be grown on unproductive marginal lands, and once the nettle is planted, nettle fibre production can be sustained for the next 10 consecutive years [1]. Nettle production cost is less than that of cotton [10], and the substitution of nettle fibre for cotton could save large amounts of water [10]. Nettle fibre is produced in a similar way to other bast/stem fibres, which are extracted from the stalks of the plant, and the nettle fibre content can reach a maximum of 16% of the stalk dry matter [11]. The nettle stem and its cross-section as well as nettle fibre cells in the cross-section of the nettle stem are shown in ***Figure 2*** [11].

Nettle fibre is one of the important bio-fibres which are purely derived from vegetal sources and fully biodegradable in nature [12, 13]. Nettle biofibre is primarily composed of cellulose. To be more

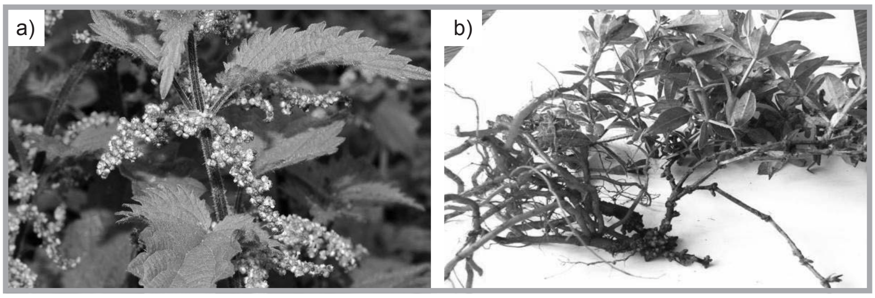


***Figure 1.*** *a) Nettle plant (Urtica dioica) b) and Madder (Rubia tinctorum L.) roots [9].*

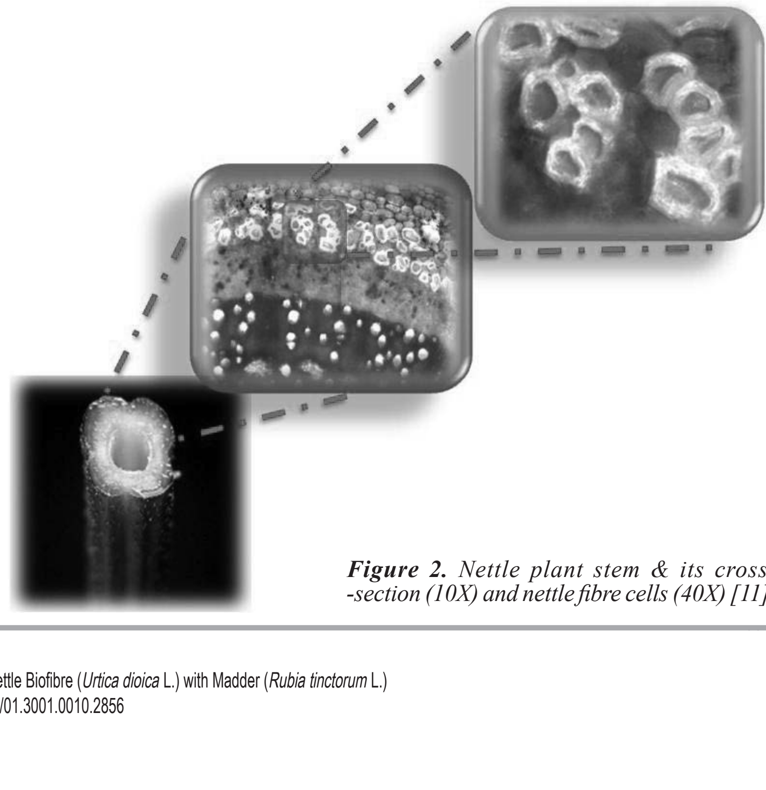

***Figure 2.*** *Nettle plant stem & its cross--section (10X) and nettle fibre cells (40X) [11].*

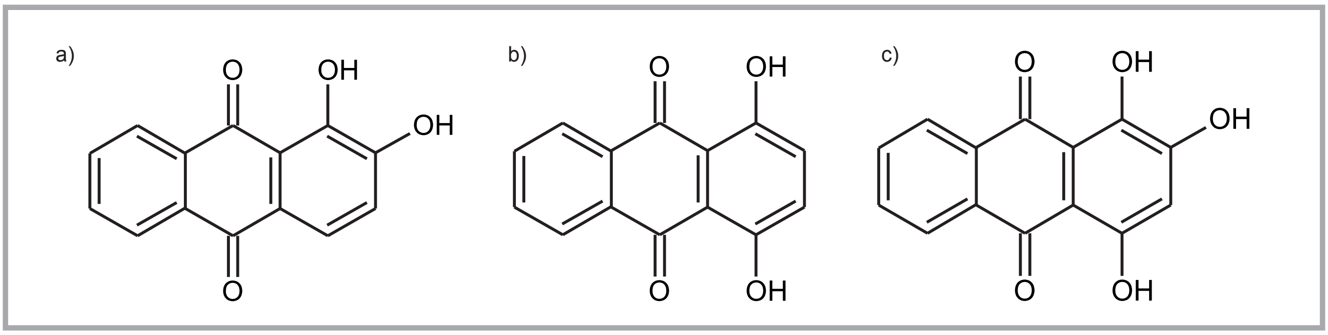


***Figure 3.*** *Molecular structures of: a) alizarin, b) quinizarin and c) purpurin.*

precise, nettle bast biofibre (*Urtica dioica* L.) is a cellulosic fibre that contains approximately, on average, 82% cellulose, 9% hemicellulose and 4% lignin [11, 14], which is the closest to cotton fibre among all other bast fibres [15], and meets the requirements of textile production such as fibre strength, thickness and length, as well as exhibiting a superior moisture absorption property. Moreover nettle fabric has a soft handle [3]. Nettle fibres also have a hollow structure as a characteristic feature (***Figure 2***), and the air captured it provides isolation from the external environment. The degree of the isolation may be controlled by twisting, and the higher the twist, the lower the isolation, since higher twisting leads to a smaller hollow volume [16]. Overall nettle biofibre usage provides bio-degradable, sustainable, renewable and eco-friendly textile production, and requires low energy consumption during production [5]. Apart from textile usage, the nettle plant also is used for many medical purposes due to its several medicinal properties, such as astringent, tonic, anti-asthmatic and diuretic properties, for the treatment of gout, dropsy, rheumatism, weight loss, eczema and hair dandruff illnesses and problems [11].

Nowadays environmental concerns are at a very high level and the textile dyeing industry faces serious criticisms due to the heavy pollutant content in its effluents. Therefore natural dyes have attracted attention for textile coloration due to their compatibility with nature, low toxic effects and non-allergenic features [17, 18]. Since prehistoric times, natural dyes have been used for dyeing natural fibres such as wool, cotton and silk as well as fur and leather [17, 19]. Madder (C.I. Natural Red 8) [6] is a very important dye source for red colour (***Figure 1***). Also different shades of various colors can be obtained from this plant depending on the mordant used [20]. Thirty six anthraquinones have been detected so far in madder roots in different researches [21]. Farizadeh *et al.* [22] determined that alizarin, purpurin and quinizarin are the three most common anthraquinones in madder roots. Alizarin is the most important colorant among other anthraquinones in madder [21]. The molecular structures of alizarin, purpurin and quinizarin are shown in ***Figure 3***.

Ultrasound is sound waves between 18 kHz – 10 MHz, which is beyond human senses [23]. Ultrasound can enhance mass transfer during ultrasound-assisted dyeing processes [24]. The mechanism of action of ultrasound in wet processes mainly depends on the phenomenon known as cavitation, which is the emerging and explosive collapsing of microscopic bubbles [23, 25], which results in local high temperature, high pressure and shock wave occurrence, leading to a chemical bond break – down [25]. Possible improvements due to ultrasound usage during dyeing can be attributed to cavitation phenomena, as mentioned earlier, and to other final physical effects such as strong agitation of the liquid (thickness reduction of fibre-liquid boundary layer), dye dispersion (breaking up of aggregates with high relative molecular mass), degassing (expulsion of dissolved or entrapped air from fibre capillaries), and swelling (enhancement of the dye diffusion rate inside the fibre) [24]. As stated in several researches, ultrasound assistance in textile wet processes reduces the process time as well as chemical and energy consumption [23, 26], accompanied by improved product quality [26]. For instance, Kamel *et al.* [25] investigated the effect of ultrasound energy on the natural dyeing of wool with lac dye and reported that ultrasound assistance resulted in more efficient dye extraction and dye uptake of wool fibres. Merdan *et al.* [27] stated that ultrasonic energy had affected the dye uptake and % exhaustion positively.

It is known that microwave energy can also be used in textile finishing applications [28, 29]. The purpose of high-frequency industrial applications is not only less energy consumption but also time-saving, leading to ensuring adequate quality products which are comparable to those produced via conventional processing methods. In a medium treated with microwave energy, heat occurs due to the activation of polar molecules [30]. When an electrical field at microwave frequencies is applied to a substrate, polar molecules attempt to rearrange their dipole moment and rotate with the changing electrical field. Heat is generated by the intermolecular friction [31]. In a 2.5 GHz microwave electromagnetic field, polarity changes nearly five billion times per second. The whole volume of the object is heated by microwave energy, while only the surface is heated by conventional methods [30]. More rapid, uniform and efficient results are obtained by microwave heating compared with conventional methods [32]. Microwave treatment decreases the processing time [33] and has low energy requirement [34]. For example, Nourmohammadian and Gholami [33] reported that for basic dyeing with acrylic dyes at low concentrations, adsorption using the microwave-based procedure is higher and much faster than by the conventional method.

In this study, the main goal was the colouration of an eco-friendly promising bast biofibre with an eco-friendly natural dye using alternative dyeing methods. Indeed there are very limited studies about nettle biofibre, and there is not any scientific study reporting about the colouration of nettle biofibres. Therefore conventional and ultrasound assisted and microwave energy assisted natural dyeing of 100% nettle biofibre fabric, as an eco-friendly material for textile purposes, with madder (*Rubia tinctorum L.*) were investigated and compared. One natural (green tea) and one chemical (alum) mordanted dyeing were carried out in addition to an un-mordanted process as a control process. Colour properties and wash, rub (dry and wet), perspiration (alkaline and acid), water and light fastness performances were investigated and compared.

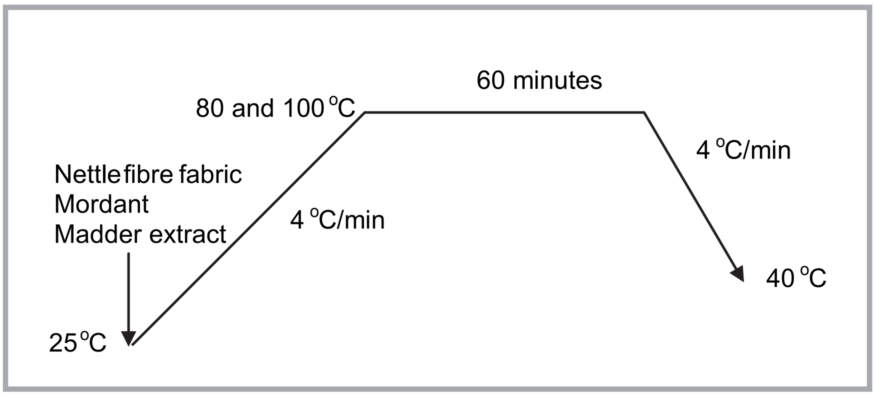


***Figure 4.*** *Madder dyeing procedure used for nettle fibre fabric.*

## Experimental

### Materials

Single jersey knitted fabric constructed from 100% nettle bast biofibre 19.68 tex spun yarns was obtained from Octans Fabrics (South Korea) and used for experiments. Nettle fabric was bleached via the conventional peroxide bleaching process, leading to a whiteness degree of 68.79 Stensby. The powder form of madder, *Rubia tinctorum* L., roots was obtained from the Siirt Region, Turkey. Alum ($KAl(SO_4)_2 \cdot 12H_2O$ – Emboy) was selected as a chemical mordant, being of technical grade. Green tea was chosen as a natural mordant. The green tea leaves used were in a coarse-grained particle form, obtained from the Rize region of Turkey.

### Dye extraction

Powder madder roots were processed with 100 °C water for 1 hour using a 1/40 madder powder/water ratio for dye extraction. Afterwards the solution was cooled at room temperature and subsequently filtered for purification. This extract was used as the liquor for dyeings.

### Dyeing operations

As indicated earlier, natural nettle dyeings with madder were performed with conventional ultrasound and microwave exhaustion methods. All dyeing methods were carried out at a 1:40 liquor ratio with 20% owf green tea mordant or 20% owf alum mordant according to the simultaneous mordanting method. All dyeings were carried out three times. All nettle fibre fabric samples dyed, after each respective dyeing method, were cold rinsed for 5 minutes and then air-dried.

### Conventional dyeing

An ATAC LAB-DYE HT laboratory type dyeing machine was used for exhaustion dyeing operations. Nettle samples were dyed at 80 °C and 100 °C at a 1:40 liquor ratio with 20% owf green tea and 20% owf alum mordants. The dyeing process is shown in ***Figure 4***. The simultaneous mordanting method was chosen due to its simplicity and less processing time in total. Dyeings were carried out for 60 minutes at both 80 °C and 100 °C. The dyeing temperature of 80 °C was chosen for an even comparison with ultrasonic bath dyeing, whose maximum dyeing temperature was also 80 °C.

### Ultrasound dyeing

The assistance of ultrasound technology was explored for natural dyeing to see its effect on the colour and colour fastness properties of nettle fibre fabrics. Therefore an ultrasonic bath (WiseClean WUC-D10H (40 kHz) 200 W HF-power, Korea) was used for this purpose. The maximum dyeing temperature of the ultrasonic bath was 80 °C, and hence ultrasonic bath dyeing was carried out at 80 °C for 60 minutes for even comparison with conventional exhaustion dyeing.

### Microwave dyeing

The microwave energy assisted natural dyeing process was carried out using an Arçelik MD 565 model microwave oven (900 watt) via the exhaustion process. Nettle fabrics were dyed with microwave energy assistance for 5, 10 and 15 minutes. Dyeing liquor was prepared at room temperature and poured into a closed glass container. The dyeing liquor was evaporated after 15 minutes of application. Therefore further application times were not studied.

### Colorimetric measurements

The CIE *L*, a*, b*, C** and *h*° co-ordinates were measured and calculated from the reflectance values at an appropriate wavelength of maximum absorbance ($\lambda_{max}$) for each dyed sample using a DataColor SpectraFlash 600 (Datacolor International, Lawrenceville, NJ, USA) spectrophotometer (D65 day light, 10° standard observer). Each fabric sample was read in four different areas, twice on each side of the fabric for consistency, and the average value was calculated. The colour strength value ($f_k$) was calculated with the equation below using the sum of the weighted K/S values in the visible region of the spectrum. The values of x, y and z are the colour matching functions for the 10° standard observer at each wavelength measured [35, 36].

$$f_k = \sum_{\lambda=400}^{700} (K/S)_\lambda \, (\bar{x}_{10,\lambda} + \bar{y}_{10,\lambda} + \bar{z}_{10,\lambda})$$

### Colour fastness tests

Wash fastness, rub fastness and light fastness properties were investigated. Wash-fastness to domestic laundering (C06) was carried out according to the ISO 105:C06 A2S test (40 °C) in a M228 Rotawash machine (SDL ATLAS, UK). Both dry and wet rub fastness testing was performed following the ISO 105: X12 protocol. Alkaline and acidic perspiration fastness testing were determined according to the test method ISO 105-E04; color fastness to perspiration protocol (ISO 105-E04). Water fastness testing was determined according to the test method ISO 105-E01. The ISO grey scale was used for estimation of the color fastness of the dyed fabrics to washing, dry and wet rubbing, alkaline and acidic perspiration, and to water. Light fastness testing was carried out according to ISO 105: B02: colour fastness to artificial light (Xenon arc lamp). Colour fastness to light was determined using the blue wool scale.

### Infrared spectroscopy

Infrared analysis was performed using a Perkin Elmer Spectrum Two™ Infrared

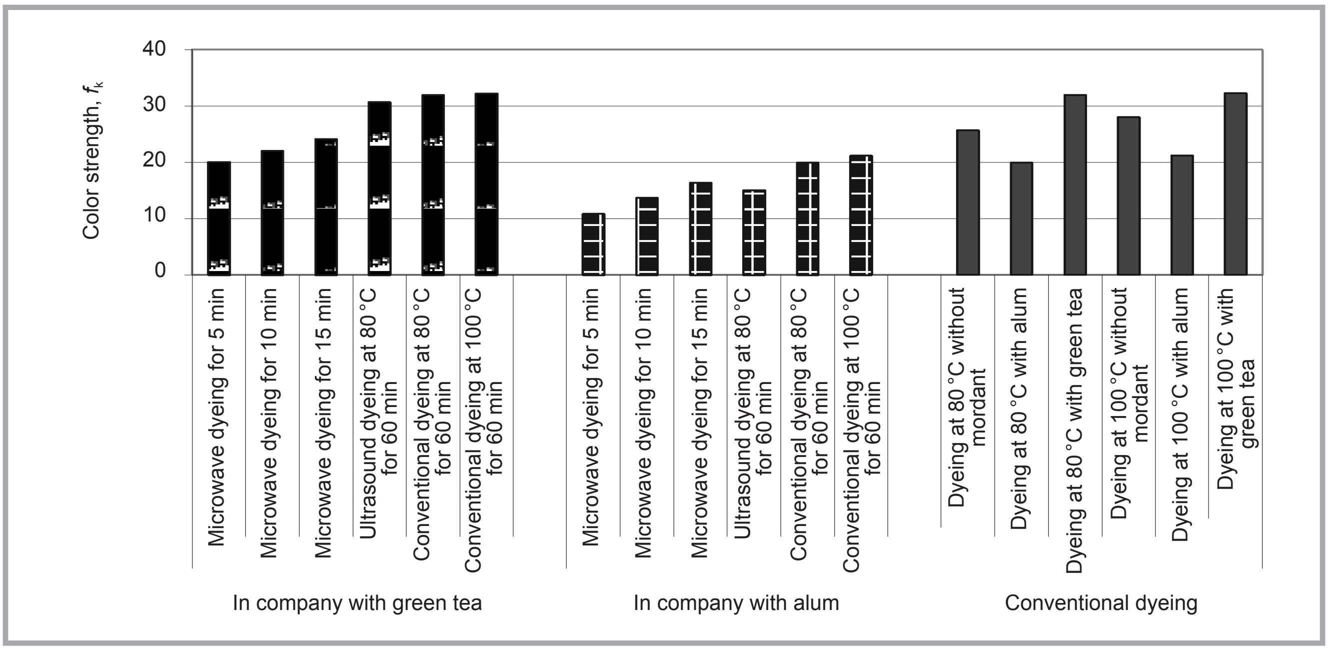


***Figure 5.*** *Effects of different madder dyeing methods, dyeing temperature and mordant type (alum and green tea) on the colour strength of nettle fibre*

Spectrometer (FT-IR) with a diamond universal ATR accessory I then ATR mode, employing a diamond crystal that give an effective depth of penetration of 1 micron, at a resolution of 4 $cm^{-1}$. The spectrum recorded for each sample was the average of 4 scans.

## Results and discussion

Data obtained from the assessments of fabric colorimetric properties appears in ***Table 1*** and ***Figures 5-6***, while the results of the colour fastness properties are in ***Tables 2-7***.

***Table 1.*** *Colorimetric properties and shades of nettle fibres dyed with madder obtained using different dyeing methods with alum and green tea mordants*

| Dyeing process type and mordant type | *L** | *a** | *b** | *C** | *h°* | $f_k$ |
|---|---|---|---|---|---|---|
| Conventional dyeing (80 °C-60 min) without mordant | 55.0 | 25.4 | 8.5 | 26.8 | 18.6 | 25.7 |
| Conventional dyeing (80 °C-60 min) with alum | 59.1 | 25.1 | 10.0 | 27.0 | 21.7 | 20.0 |
| Conventional dyeing (80 °C-60 min) with green tea | 51.7 | 19.3 | 9.5 | 21.5 | 26.3 | 32.0 |
| Conventional dyeing (100 °C-60 min) without mordant | 54.0 | 27.0 | 9.1 | 28.5 | 18.6 | 28.0 |
| Conventional dyeing (100 °C-60 min) with alum | 58.8 | 25.3 | 12.1 | 28.0 | 25.5 | 21.2 |
| Conventional dyeing (100 °C-60 min) with green tea | 51.7 | 22.9 | 9.4 | 24.8 | 22.4 | 32.3 |
| Ultrasound dyeing (80 °C-60 min) with alum | 62.6 | 25.2 | 8.3 | 26.5 | 18.3 | 15.1 |
| Ultrasound dyeing (80 °C-60 min) with green tea | 52.4 | 26.0 | 8.6 | 27.4 | 18.3 | 30.7 |
| Microwave dyeing (5 min) with alum | 67.7 | 23.2 | 10.9 | 25.6 | 25.0 | 10.9 |
| Microwave dyeing (5 min) with green tea | 58.7 | 25.3 | 9.0 | 26.9 | 19.5 | 20.1 |
| Microwave dyeing (10 min) with alum | 64.5 | 24.6 | 10.4 | 26.7 | 23.0 | 13.7 |
| Microwave dyeing (10 min) with green tea | 57.5 | 25.2 | 9.5 | 26.9 | 20.7 | 22.1 |
| Microwave dyeing (15 min) with alum | 62.3 | 24.8 | 11.6 | 27.4 | 25.1 | 16.4 |
| Microwave dyeing (15 min) with green tea | 56.1 | 23.9 | 9.6 | 25.7 | 21.9 | 24.2 |

**Colour properties**

The colorimetric data and colors obtained for conventional, ultrasound and microwave aided natural madder dyeings of nettle biofibre fabrics are shown in ***Table 1***. The highest colour strength ($f_k$) achieved for nettle biofibre was 32.3 for the conventional dyeing process at 100 °C with a green tea mordant (***Table 1*** and ***Figure 5***). The effect of dyeing temperature (80 or 100 °C) and mordant type (without mordant, alum or green tea) for 60 minutes conventional dyeing on the nettle fibre colour strength is clearly shown in ***Figure 5***.

Overall, 100 °C dyeings resulted in slightly higher colour strength than 80 °C dyeings for all mordanted and unmordanted nettle fibre fabrics (***Figure 5***). For instance, conventional dyeing without any mordant at 80 °C and 100 °C resulted in a colour strength ($f_k$) of 25.7 and 28, respectively (***Figure 5*** and ***Table 1***). Alum mordanted nettle samples exhibited even less colour strength than un-mordanted nettle samples for both conventional dyeing temperatures (***Figure 5***). Green tea mordanted nettle fibres exhibited higher colour strength than alum mordanted and un-mordanted ones for both conventional dyeing temperatures (***Figure 5***). Even conventional dyeing at 80 °C with green tea resulted in higher colour strength than conventional dyeing at 100 °C without mordant presence (***Figure 5***).

This performance difference could be due to the tannin and other metal traces present in green tea. Indeed tea contains tannins [37], with green tea containing many different compounds such as polyphenols (different catechins), flavonoids (tannins: kaempferol, quercetin, and myricetin), anthocyanidin, caffeine, theophylline, theobromine, saponins, carotene, some vitamins, flouride, iron, magnesium, calcium, strontium, copper, nickel, zinc, and traces of elements such as molybdenum and phosphorus [38]. Tannins, having a relatively high molecular mass of a typical aromatic ring structure with hydroxyl substituents, are plant phenolics, which is one of the most widespread and complex groups of chemical compounds in plants [39]. Tannins can be used as a mordant material for natural textile dyeing, but also they can be used for textile coloration accompanying a mordant (metal ion) [40]. Indeed tannin or tannin agents can be generally used for colour shifts toward darker shades [39].

The effects of different madder dyeing methods on the colour strength of nettle biofibre mordanted with alum and green tea are shown on ***Figure 5***. It is very clear from both alum and green tea mordanted samples that the conventional dyeing process at both 80 °C and 100 °C dyeing temperatures resulted in higher colour strength than both the ultrasound assisted and microwave assisted dyeing methods (***Figure 5***).

As stated earlier, conventional dyeings at 100 °C caused slightly higher colour strength than their 80 °C counterparts. As in conventional dyeing processes, green tea mordant resulted in higher colour strength values than for alum mordant for both the ultrasound assisted and microwave assisted dyeing methods (***Figure 5***).

In the case of microwave energy assisted madder dyeings, the longer application time resulted in higher colour strength for both mordanted nettle fibres (***Figure 5***). For example, microwave energy assisted madder dyeings of nettle with green tea lasting 5, 10 and 15 minutes led to colour strength values of 20.1, 22.2, and 24.2, respectively (***Figure 5***). Therefore 15 minutes of microwave energy assisted madder dyeing exhibited the highest colour strength values amongst the microwave assisted ones (***Figure 5***). As mentioned earlier, further application times (more than 15 minutes) could not be studied; since after 15 minutes of ap-

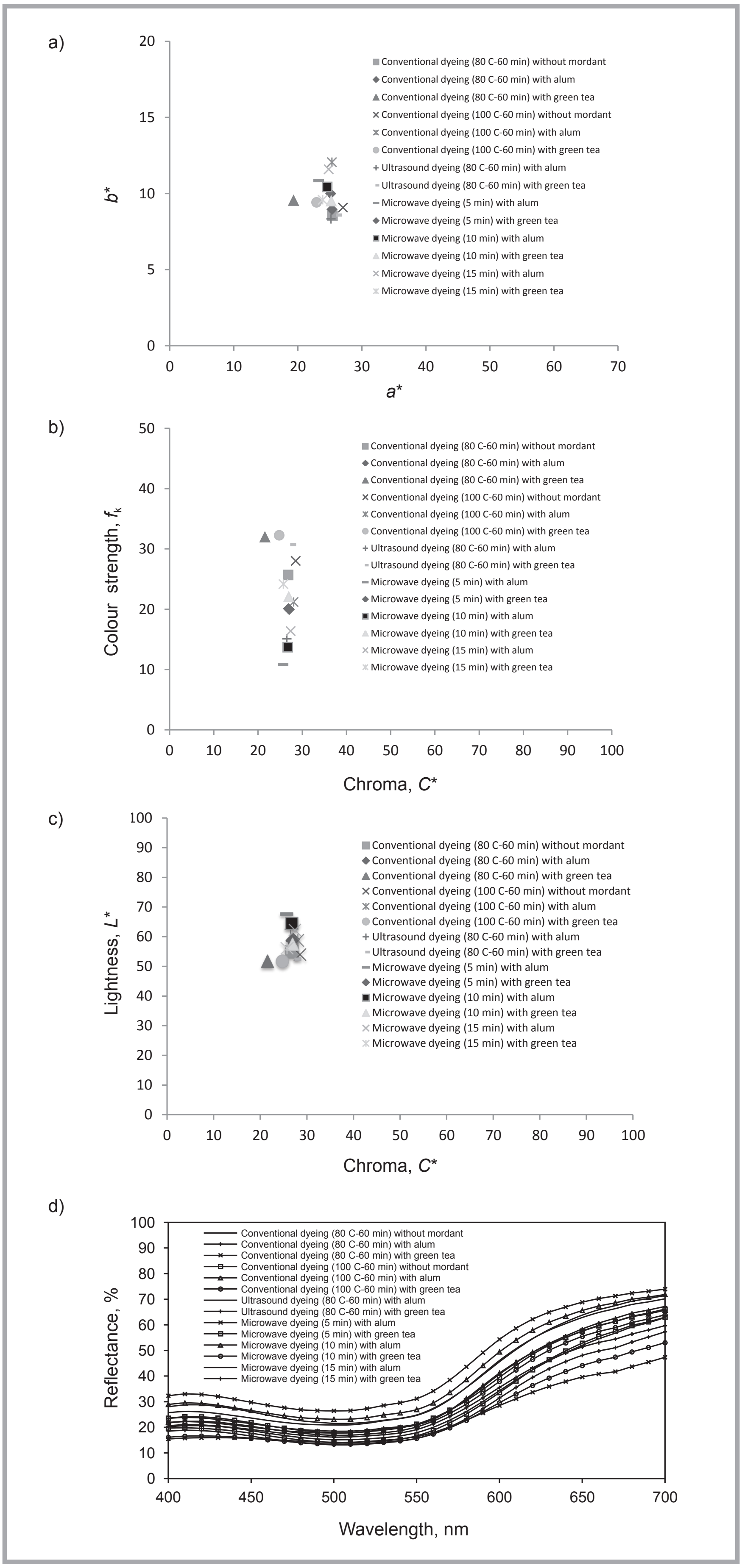


***Figure 6.*** *Colour properties: a) a*-b*, b) $f_k$-C*, c) L*-C* plots, and d) reflectance-wavelength spectra of nettle fibres dyed with madder*

plication, all dyeing process liquor was evaporated.

Ultrasound assisted madder dyeing of nettle biofibre with green tea mordant at 80 °C for 60 minutes exhibited higher colour strength (30.7 versus 24.2) than that dyed with microwave energy assistance for 15 minutes (***Figure 5***). On the other hand, those dyeings led to approximately similar results (15.1 versus 16.4) in the case of alum mordanted nettle fibres (***Figure 5***). Therefore ultrasound assisted dyeing for 60 minutes and microwave assisted dyeings for 15 minutes resulted in comparably close colour strength values. However it should not be forgotten that the dyeing time of microwave energy assisted dyeing was only 15 minutes, whereas the dyeing time of their counterparts, conventional and ultrasound assisted dyeings, were fourfold – 60 minutes.

As can be seen from ***Table 1*** and ***Figure 6***, different shades of red were observed according to different dyeing process types. The colour strength and colorimetric changes imparted to each madder nettle dyeing by the different dyeing methods with different mordants are clearly more visible in the a* vs b*, C* vs ($f_k$), C* vs L* plots and reflectance-wavelength spectra shown in ***Figure 6.a-d.***, respectively. It is clear that the use of mordant materials was effective for the shades and colorimetric properties of the dyed nettle fibre fabrics observed. Different color properties were observed depending on the mordant material applied (***Table 1*** and ***Figure 6***). It is known that the final color properties are not only dependent on the natural dye itself but also on the mordant material used [41]. Indeed mordant material usage can change the colour properties of the dyed textile substrate and also the resultant hue of the material [39]. Although the hue angle values measured (*h*°) were below 90° (yellow-red axis zone), leading to a red shade appearance for all dyed nettle samples, mordant matter as well as the dyeing type and dyeing time changed the shades of colours with various hue angle values due to different *a** and *b** values of the dyed samples (***Table 1*** and ***Figure 6.a***). Chroma values of all dyed samples were close to each other and in between the values of 24.8-28.5, with one exception being the conventionally dyed nettle at 80°C with green tea mordant, 21.5 (***Figure 6.b***). Overall the mordanted and unmordanted nettle fibres displayed close chroma (*C**) values.

Conventionally dyed green tea mordanted nettle samples exhibited the darkest appearance according to *L** values (***Figure 6.c***). Then ultrasound assisted dyeing follows conventional dyeing in respect of darkness according to lightness values. Since, as mentioned earlier, tea has tannins [37], tannin or tannin agents can be generally used for colour shifts toward darker shades [39]. It is also clearly visible from ***Figure 6.c*** that alum mordanted nettle samples exhibited the lightest appearance again according to *L** values. And, in general, alum mordanted samples also showed slightly higher chroma values than, especially, conventionally dyed samples mordanted with green tea (***Figure 6.c***). It is known that the colour becomes brighter when the chroma and colour strength are both increasing [42]. In general, but not as a rule, alum mordanted nettle samples led to a brighter appearance, due to their higher lightness and chroma properties, than green tea mordanted samples. For example, conventional dyeing of nettle fibre mordanted with alum and green tea at 100 °C caused 58.8 (*C**) and 28.0 (*L**) versus 51.7 (*C**) and 24.8 (*L**), respectively, leading to brighter appearance in the case of the alum mordanted one. This is actually in line with Bechtold and Mussak [39], who stated that alum salts as mordants intensify the color brilliance obtained but do not influence the colour shade to the same extent. They determined that alum mordant usage led to brighter colors [43]. Ferreira et al. [20] also stated that brilliant colours were observed by alum mordant usage in the application of particular dyestuffs.

In general, microwave assisted dyeing led to lighter appearance in comparison to conventional and ultrasound assisted dyeings (***Table 1*** and ***Figure 6.c***). A prolonged microwave dyeing time resulted in darker appearance with lower *L** values, which is in parallel with the higher colour strength values ($f_k$) (***Table 1*** and ***Figure 6.c***). The lightness values of ultrasound assisted dyeing were in between those of conventional dyeings and microwave assisted dyeings.

### Colour fastness properties

Wash fastness, rub fastness, perspiration fastness, water fastness and light fastness properties of nettle biofibres dyed with madder using different dyeing methods with alum and green tea mordants are

***Table 2.*** *Wash fastness properties of nettle fibres dyed with madder using different dyeing methods with alum and green tea mordants.* ***Note:*** ** Intermediate rating with underlined numbers indicates that the specimen's staining tended to be towards the underlined end of the range, and an underlined figure indicates that the grading was probably within a 0.25 point of that value.*

| Dyeing process type and mordant type | $f_k$ | Wash fastness staining (C06-A2S)* | | | | | |
|---|---|---|---|---|---|---|---|
| | | Wool | Acrylic | Polyester | Polyamide | Cotton | Acetate |
| Conventional dyeing (80 °C-60 min) without mordant | 25.7 | 5 | 5 | 4-<u>5</u> | 4-5 | 5 | 5 |
| Conventional dyeing (80 °C-60 min) with alum | 20.0 | 5 | 5 | 4-<u>5</u> | 4-5 | 5 | 5 |
| Conventional dyeing (80 °C-60 min) with green tea | 32.0 | 5 | 5 | 4-<u>5</u> | 4-5 | 5 | 5 |
| Conventional dyeing (100 °C-60 min) without mordant | 28.0 | 5 | 5 | 4-<u>5</u> | 4-5 | 5 | 5 |
| Conventional dyeing (100 °C-60 min) with alum | 21.2 | 5 | 5 | 4-<u>5</u> | 4-5 | 5 | 5 |
| Conventional dyeing (100 °C-60 min) with green tea | 32.3 | 5 | 5 | 5 | 4-5 | 5 | 5 |
| Ultrasound dyeing (80 °C-60 min) with alum | 15.1 | 5 | 5 | 5 | 4-5 | 5 | 5 |
| Ultrasound dyeing (80 °C-60 min) with green tea | 30.7 | 5 | 5 | 4-<u>5</u> | 4-5 | 5 | 5 |
| Microwave dyeing (5 min) with alum | 10.9 | 5 | 5 | 5 | 4-<u>5</u> | 5 | 5 |
| Microwave dyeing (5 min) with green tea | 20.1 | 5 | 5 | 4-<u>5</u> | 4-5 | 5 | 5 |
| Microwave dyeing (10 min) with alum | 13.7 | 5 | 5 | 5 | 4-<u>5</u> | 5 | 5 |
| Microwave dyeing (10 min) with green tea | 22.1 | 5 | 5 | 4-<u>5</u> | 4-5 | 5 | 5 |
| Microwave dyeing (15 min) with alum | 16.4 | 5 | 5 | 4-<u>5</u> | 4-5 | 5 | 5 |
| Microwave dyeing (15 min) with green tea | 24.2 | 5 | 5 | 4-5 | 4-5 | 5 | 5 |

shown in ***Tables 2, 3, 4, 5*** and ***6***, respectively.

***Wash fastness***
Wash fastness properties are shown on ***Table 2***.

All nettle biofibre samples dyed with madder with different dyeing techniques exhibited very high, commercially acceptable (equal to grey scale of rating 4 or above) and close wash fastness properties with a grey scale rating of between 4-5 and 5 for staining (***Table 2***). Only polyester and polyamide fibres of the adjacent multifibre fabric were stained by madder during the wash fastness test. Nettle dyeing with madder extract resulted in quite high results for wash fastness.

***Rub fastness***
Rub fastness properties are shown in ***Table 3***.

***Table 3.*** *Rub fastness properties of nettle fibres dyed with madder using different dyeing methods with alum and green tea mordants.* ***Note:*** ** Intermediate rating with underlined numbers indicates the specimen's staining tended to be towards the underlined end of the range and an underlined figure indicates that the grading was probably within a 0.25 point of that value.*

| Dyeing process type and mordant type | $f_k$ | Rub fastness (X12) (cotton staining)* | |
|---|---|---|---|
| | | Dry | Wet |
| Conventional dyeing (80 °C-60 min) without mordant | 25.7 | 4-5 | 3 |
| Conventional dyeing (80 °C-60 min) with alum | 20.0 | 5 | 3-4 |
| Conventional dyeing (80 °C-60 min) with green tea | 32.0 | 4-5 | 3 |
| Conventional dyeing (100 °C-60 min) without mordant | 28.0 | 4-5 | 3-4 |
| Conventional dyeing (100 °C-60 min) with alum | 21.2 | 5 | 4 |
| Conventional dyeing (100 °C-60 min) with green tea | 32.3 | 4-5 | 3-4 |
| Ultrasound dyeing (80 °C-60 min) with alum | 15.1 | 5 | 4 |
| Ultrasound dyeing (80 °C-60 min) with green tea | 30.7 | 5 | 3-4 |
| Microwave dyeing (5 min) with alum | 10.9 | 5 | 4-5 |
| Microwave dyeing (5 min) with green tea | 20.1 | 4-5 | 3-4 |
| Microwave dyeing (10 min) with alum | 13.7 | 5 | 4-5 |
| Microwave dyeing (10 min) with green tea | 22.1 | 4-5 | 3-4 |
| Microwave dyeing (15 min) with alum | 16.4 | 5 | 4 |
| Microwave dyeing (15 min) with green tea | 24.2 | 4-5 | 3-4 |

Most of the dyed nettle biofibre samples exhibited adequate rub fastness properties (***Table 3***). Although dry rub fastness values of all dyed nettle fibre samples were found to be quite high and within a commercially acceptable range, such as the wash fastness and wet rub fastness results being moderate to good, wet rub fastness values were up to 1.5 points lower than the corresponding values of dry rub fastness. Indeed the wet rub fastness values of some dyed nettle fibres were below 4 (***Table 3***). The wet rub fastness of dyed nettle samples were generally in the range of 3 and 4 on the grey scale, with two exceptions: the nettle samples dyed with microwave assisted dyeing for 5 minutes with alum mordant and microwave assisted dyeing for 10 minutes with alum mordant. Nevertheless it should be pointed out that these two nettle samples also exhibited the lowest colour strength properties, 10.9 and 13.7, in this study. It is known that rub fastness performance is associated with unfixed dyes deposited on the fibre surface. Thus the measure of rub fastness performance is related to clearing procedures after dyeing. In this study, samples were only cold-rinsed for 5 minutes subsequent to dyeing. It is believed that a more aggressive washing might resulted in a better wet and dry rub fastness performance. There are no specific trends according to the different dyeing methods. The wet rub fastness of 100 °C conventionally dyed nettle samples were up to a 0.5 point better than that of 80 °C conventionally dyed nettle samples (***Table 3***). A similar observation can be made in the case of dry rub fastness, but with only up to a 0.25 point higher improvement. This may be due to the better penetration and diffusion of dyes into the amorphous regions of the nettle fibre, leading to possibly less unfixed dyes deposited on the nettle fibre surface. It is also interesting that wet rub fastness values of alum mordanted nettle samples were up to a 0.5 point better than the wet rub fastness values of green tea mordanted nettle samples (***Table 3***). This could be due to the lower color strength levels of alum mordanted samples in comparison to green tea mordanted ones (***Table 3***). This is in line with the finding of Ferreira *et al.* [20], who stated that good fastness properties were observed with alum mordant usage in the application of particular dyestuffs.

***Perspiration fastness***
Alkaline and acidic perspiration fastness properties are shown in ***Tables 4*** and ***5***, respectively.

All nettle biofibre samples dyed with madder with different dyeing techniques exhibited very high and commercially acceptable (equal to a grey scale rating of 4 or above) alkaline and acidic perspiration fastness properties with a grey scale rating of between 4 and 4-5 for staining (***Tables 4*** and ***5***). Alkaline perspiration fastness results of dyed nettle fibres were generally slightly better than those of acidic perspiration fastness, especially for polyamide and cotton fibre component stainings in the multifibre adjacent fabric (***Tables 4*** and ***5***). In the case of acidic perspiration fastness, in general, fabrics exhibiting higher colour strength resulted in slightly lower fastness levels especially for polyamide and cotton stainings (***Table 5***).

***Water fastness***
Water fastness properties are shown on ***Table 6.***

Again, as in washing, perspiration fastness and water fastness values of the dyed nettle biofibres exhibited quite high and commercially acceptable water fastness performance (***Table 6***). The water fastness levels were in the range of 4-5 and 5.

***Light fastness***
Light fastness properties are shown on ***Table*** *7*.

Light fastness results of nettle biofibres dyed with madder using different dyeing methods with alum and green tea mordants were in the range of 2 and 3 (***Table 7***). The low light fastness levels observed are in line with earlier findings [44-47]. The authors of [44] reported that natural dyes mostly display poor to moderate light fastness values. It is visible from ***Table 7*** that higher colour strength generally resulted in higher light fastness performance. It is known that higher colour strength leads to a colour which is more resistant to fading caused by light exposure. Therefore conventional and ultrasound dyeing methods led to higher light fastness performance levels by 3 for nettle fibres. For example, microwave as-

***Table 4.** Alkaline perspiration fastness properties of nettle fibres dyed with madder using different dyeing methods with alum and green tea mordants.* ***Note:*** *[*] Intermediate rating with underlined numbers indicates the specimen's staining tended to be towards the underlined end of the range and an underlined figure indicates that the grading was probably within a 0.25 point of that value.*

| Dyeing process type and mordant type | $f_k$ | Alkaline perspiration fastness staining (ISO 105-E04)* | | | | | |
|---|---|---|---|---|---|---|---|
| | | Wool | Acrylic | Polyester | Polyamide | Cotton | Acetate |
| Conventional dyeing (80 °C-60 min) without mordant | 25.7 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Conventional dyeing (80 °C-60 min) with alum | 20.0 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Conventional dyeing (80 °C-60 min) with green tea | 32.0 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Conventional dyeing (100 °C-60 min) without mordant | 28.0 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Conventional dyeing (100 °C-60 min) with alum | 21.2 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Conventional dyeing (100 °C-60 min) with green tea | 32.3 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Ultrasound dyeing (80 °C-60 min) with alum | 15.1 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Ultrasound dyeing (80 °C-60 min) with green tea | 30.7 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Microwave dyeing (5 min) with alum | 10.9 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Microwave dyeing (5 min) with green tea | 20.1 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Microwave dyeing (10 min) with alum | 13.7 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Microwave dyeing (10 min) with green tea | 22.1 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Microwave dyeing (15 min) with alum | 16.4 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Microwave dyeing (15 min) with green tea | 24.2 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |

***Table 5.** Acidic perspiration fastness properties of nettle fibres dyed with madder using different dyeing methods with alum and green tea mordants.* ***Note****: [*] Intermediate rating with underlined numbers indicates the specimen's staining tended to be towards the underlined end of the range and an underlined figure indicates that the grading was probably within a 0.25 point of that value.*

| Dyeing process type and mordant type | $f_k$ | Acidic perspiration fastness staining (ISO 105-E04)* | | | | | |
|---|---|---|---|---|---|---|---|
| | | Wool | Acrylic | Polyester | Polyamide | Cotton | Acetate |
| Conventional dyeing (80 °C-60 min) without mordant | 25.7 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4-5 |
| Conventional dyeing (80 °C-60 min) with alum | 20.0 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4-5 |
| Conventional dyeing (80 °C-60 min) with green tea | 32.0 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4 |
| Conventional dyeing (100 °C-60 min) without mordant | 28.0 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4-5 |
| Conventional dyeing (100 °C-60 min) with alum | 21.2 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4-5 |
| Conventional dyeing (100 °C-60 min) with green tea | 32.3 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4-5 |
| Ultrasound dyeing (80 °C-60 min) with alum | 15.1 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Ultrasound dyeing (80 °C-60 min) with green tea | 30.7 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4-5 |
| Microwave dyeing (5 min) with alum | 10.9 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Microwave dyeing (5 min) with green tea | 20.1 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4-5 |
| Microwave dyeing (10 min) with alum | 13.7 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Microwave dyeing (10 min) with green tea | 22.1 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4-5 |
| Microwave dyeing (15 min) with alum | 16.4 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Microwave dyeing (15 min) with green tea | 24.2 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4-5 |

***Table 6.** Water fastness properties of nettle fibres dyed with madder using different dyeing methods with alum and green tea mordants.* ***Note****: [*] Intermediate rating with underlined numbers indicates the specimen's staining tended to be towards the underlined end of the range and an underlined figure indicates that the grading was probably within a 0.25 point of that value.*

| Dyeing process type and mordant type | $f_k$ | Water fastness staining (ISO 105-E01)* | | | | | |
|---|---|---|---|---|---|---|---|
| | | Wool | Acrylic | Polyester | Polyamide | Cotton | Acetate |
| Conventional dyeing (80 °C-60 min) without mordant | 25.7 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Conventional dyeing (80 °C-60 min) with alum | 20.0 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Conventional dyeing (80 °C-60 min) with green tea | 32.0 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Conventional dyeing (100 °C-60 min) without mordant | 28.0 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Conventional dyeing (100 °C-60 min) with alum | 21.2 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Conventional dyeing (100 °C-60 min) with green tea | 32.3 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Ultrasound dyeing (80 °C-60 min) with alum | 15.1 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Ultrasound dyeing (80 °C-60 min) with green tea | 30.7 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Microwave dyeing (5 min) with alum | 10.9 | 4-5 | 5 | 5 | 4-5 | 4-5 | 5 |
| Microwave dyeing (5 min) with green tea | 20.1 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Microwave dyeing (10 min) with alum | 13.7 | 4-5 | 5 | 5 | 4-5 | 4-5 | 5 |
| Microwave dyeing (10 min) with green tea | 22.1 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Microwave dyeing (15 min) with alum | 16.4 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Microwave dyeing (15 min) with green tea | 24.2 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |

sisted dyed nettle fibre fabrics exhibited up to 1 point lower light fastness levels than their counterparts, most probably due to their lower dye content, which in turn leads to lower colour strength, leading to more distinct fading under a light source. For instance, the light fastness performance of the nettle fibre sample dyed with microwave assistance for 5 minutes with alum mordant, which exhibited the lowest colour strength value of 10.9, was only 2 (***Table** 7*).

**Infrared spectroscopy**

FTIR-ATR spectra of the dyed nettle biofibres were investigated to detect changes occurring in the nettle fibre after different dyeing processes (conventional, ultrasound and microwave dyeing) (***Figure** 7*). Infrared absorption spectroscopy is extensively used to provide both qualitative and quantitative information about the conformational characteristics, chemical composition, crystallinity and orientation of molecules of a wide range of materials [48]. Infrared (IR) absorption spectroscopy measures the intensity of absorption of infrared radiation by a sample and the FTIR spectrum is a "fingerprint" of a material, with absorption peaks which represent the frequencies of vibrations between the bonds of atoms forming the material. There are not any detectable significant changes in the FTIR (ATR) spectra properties of nettle fibre after different dyeing processes (conventional, ultrasound and microwave dyeing) (***Figure** 7*). The constancy of the FTIR (ATR) spectra (***Figure** 7*) shows that the respective different dyeing treatment processing types have no significant effect on the surface morphology of nettle fibre.

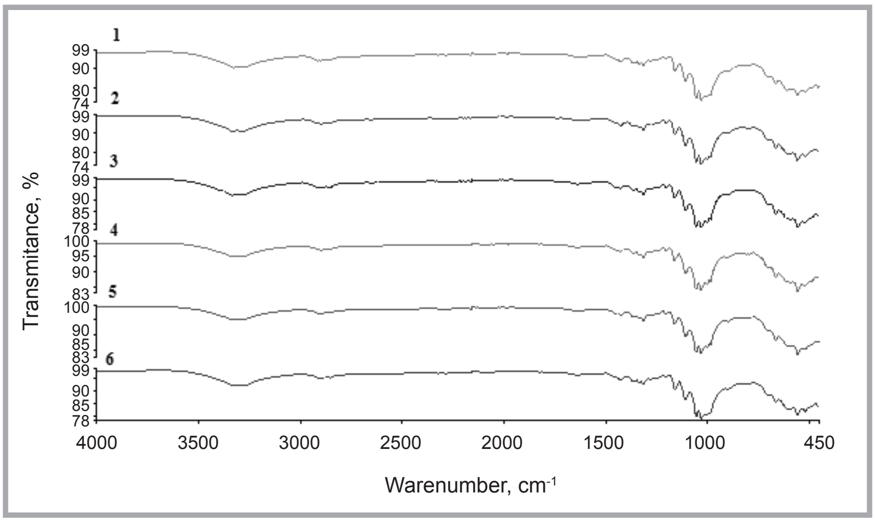


***Figure** 7. FTIR (ATR) spectra of nettle fibres dyed with madder using different dyeing methods with green tea mordant: 1) greige nettle fibre, 2) bleached nettle fibre, 3) nettle fibre after conventional dyeing at 80 °C for 60 minutes, 4) nettle fibre after conventional dyeing at 100 °C for 60 minutes, 5) nettle fibre after ultrasound dyeing at 80 °C for 60 minutes, 6)nettle fibre after microwave dyeing for 15 minutes.*

## Conslusions

The highest colour strength ($f_k$) achieved for nettle biofibre was 32.3 for the conventional dyeing process at 100 °C with a green tea mordant. Conventional dyeings at 100 °C resulted in slightly higher colour strength than for conventional dyeings at 80 °C for all mordanted and unmordanted nettle fibre fabrics. Moreover the conventional dyeing process at both 80 °C and 100 °C led to higher colour strength than for both the ultrasound assisted and microwave assisted dyeing methods. Green tea mordanted nettle biofibres exhibited higher colour strength than alum mordanted ones for all dyeing methods (conventional, microwave assisted and ultrasound assisted). Prolonged microwave energy assisted madder dyeings led to higher colour strength. Ultrasound assisted dyeing for 60 minutes and microwave assisted dyeings for 15 minutes resulted in comparably close colour strength values. However, it is important to mention that the dyeing time of microwave energy assisted dyeing was only 15 minutes, whereas that of their counterparts, conventional and ultrasound assisted dyeings, were fourfold – minutes. Therefore the usage of microwave energy in dyeing processes is important in terms of shortening the process time.

Different shades of red were observed on nettle biofibre fabrics according to different dyeing process types with madder. Even though the hue angle values measured ($h°$) were below 90° (yellow-red axis zone), leading to a red shade appearance for all dyed nettle samples, different dyeing processes and mordants resulted in various shades of colours with different hue angle values due to different $a^*$ and $b^*$ values of the dyed samples. Mordanted and unmordanted nettle fibres exhibited close chroma values. Conventionally dyed green tea mordanted nettle samples showed the darkest appearance according to $L^*$ values. Overall microwave assisted dyeing led to lighter appearance in comparison to conventional and ultrasound assisted dyeings. Prolonged microwave

***Table** 7. Light fastness properties of nettle fibres dyed with madder using different dyeing methods with alum and green tea mordants. **Note:** * Intermediate rating with underlined numbers indicates the specimen's staining tended to be towards the underlined end of the range and an underlined figure indicates that the grading was probably within a 0.25 point of that value.*

| Dyeing process type and mordant type | $f_k$ | Light fastness (Xenon) (1-8)* |
|---|---|---|
| Conventional dyeing (80 °C-60 min) without mordant | 25.7 | 3 |
| Conventional dyeing (80 °C-60 min) with alum | 20.0 | 3 |
| Conventional dyeing (80 °C-60 min) with green tea | 32.0 | 3 |
| Conventional dyeing (100 °C-60 min) without mordant | 28.0 | 3 |
| Conventional dyeing (100 °C-60 min) with alum | 21.2 | 3 |
| Conventional dyeing (100 °C-60 min) with green tea | 32.3 | 3 |
| Ultrasound dyeing (80 °C-60 min) with alum | 15.1 | 3 |
| Ultrasound dyeing (80 °C-60 min) with green tea | 30.7 | 3 |
| Microwave dyeing (5 min) with alum | 10.9 | 2 |
| Microwave dyeing (5 min) with green tea | 20.1 | 2-3 |
| Microwave dyeing (10 min) with alum | 13.7 | 2-3 |
| Microwave dyeing (10 min) with green tea | 22.1 | 2-3 |
| Microwave dyeing (15 min) with alum | 16.4 | 2-3 |
| Microwave dyeing (15 min) with green tea | 24.2 | 2-3 |

dyeing time resulted in a darker appearance with lower $L^*$ values, which is in parallel with the higher colour strength values ($f_k$). The lightness values of ultrasound assisted dyeing were in between those of conventional dyeings and microwave assisted dyeings. In general, but not as a rule, alum mordanted nettle samples resulted in a brighter appearance, because of their higher lightness and chroma properties, than green tea mordanted samples.

All nettle biofibre samples dyed with madder with different dyeing techniques exhibited very high, commercially acceptable and close wash fastness properties with a grey scale rating of between 4-5 and 5. Most of the dyed nettle fibre samples exhibited adequate rub fastness properties. Dry rub fastness values of all dyed nettle fibre samples were found to be quite high and within the commercially acceptable range. Wet rub fastness values were up to 1.5 points lower than the corresponding values of dry rub fastness. The wet rub fastness of the dyed nettle samples were generally in a grey scale rating range of 3 and 4, with few exceptions. Wet rub fastness values of alum mordanted nettle samples were up to a 0.5 point better than those of green tea mordanted nettle samples, most probably due to the lower colour strength levels of alum mordanted samples. All nettle fibre samples dyed with madder with different dyeing techniques exhibited very high and commercially acceptable alkaline perspiration fastness, acidic perspiration fastness and water fastness levels. Light fastness values of nettle fibres dyed with madder using different dyeing methods with alum and green tea mordants were in the range of 2 and 3. Higher colour strength generally led to higher light fastness performance. Conventional and ultrasound dyeing methods resulted in slightly higher light fastness performance than for the microwave assisted dyeing method due to lower dye content in the case of nettle fibres dyed with microwave assistance. There are not any noticeable noteworthy changes in the FTIR (ATR) spectra properties of nettle biofibre after different dyeing processes (conventional, ultrasound and microwave dyeing).

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