

Dyeing with extracts from Amaranth plant – an investigation in respect to mordanting procedures and fluorescence properties

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ABSTRACT

Amaranth is a red colored crop planted as alternative food source. The use of red dye from Amaranth as food colorant and other applications is well described in literature. In contrast, the application of Amaranth for textile dyeing purposes is less described. For this, the actual paper reports an extraction process from Amaranth and the application on textile fabrics. Different mordanting processes are considered, and the fluorescent properties of Amaranth extract are determined but a transfer of fluorescence properties on the dyed fabrics is not observed. In respect to realized color intensity, the use of iron sulphate based mordant is superior compared to alum based mordant or dyeing without mordanting. However, the red coloration of Amaranth turns into brown and greyish coloration after application onto the fabric. Suitable washing and rubbing fastness are reached. Finally, it can be stated that textile dyeing with aqueous extract from Amaranth is possible but due to the change of the attractive red coloration into brownish color shades during dyeing process, the use in textile coloration is obviously limited.

Keywords

Amaranth,
Amaranthus hypochondriacus,
natural dye,
dye extraction,
fluorescence spectroscopy,
mordanting,
dyeing processes

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1 Introduction

Extracts from plants are used for dyeing of textiles in manifold ways and recipes [1,2]. In combination with different types of mordants a broad range of different colorations and color intensity can be reached on natural fibers [3-6]. An overview on the broad range of different natural dyes and product is given recently together with infrared spectroscopic data and chemical structures [7-10]. The combination of

natural fiber materials and natural plant dyes can be part of a concept to realize a fully bio-based and biodegradable textile and clothing materials [11,12]. However, compared to synthetic dyestuffs the application of natural dyes often leads to inferior light fastness [13,14]. In respect to this, the application of natural plant dyes is often part of a handcraft culture with a broad and manifold description of dyeing recipes and results in several handbooks [15-18]. One of those handbooks describes the dyeing with extracts from the plant Amaranth [19]. In this procedure, an aqueous extraction process in presence of alum salt is used [19]. Additionally, several Chinese patents describe the use of Amaranth plant as basis for dyeing processes of textile fabrics [20-22]. Especially, in one patent the use of alum and iron mordant is described [20]. Both the other patents are dedicated to the dyeing of bamboo materials and hair [21,22]. A Korean publication reports on the use of Amaranth plant as natural dye source for the dyeing of wool. In this publication the influence of alum, iron, zinc and titanium mordanting agents in different combinations is discussed [23]. Amaranth is a red colored plant which can act also as food source [24,25]. The leaves and the inflorescence of Amaranth are presented in photographs in Fig. 1. The red coloration in those parts of the plants is clearly visible. Both parts can be also used as food source and are reported to be part of a so-called “trend food” [26]. For comparison, Fig. 2 shows a microscopic image of the inflorescence of Amaranth in higher magnification taken by an electron microscope. The details of plant structure and cells are clearly seen. Remark, the natural dye extracted from Amaranth plant should be not confused with the synthetic food colorant Amaranth (E123; Food Red2). This food colorant is a synthetic anionic dye containing an azo group [27,28].

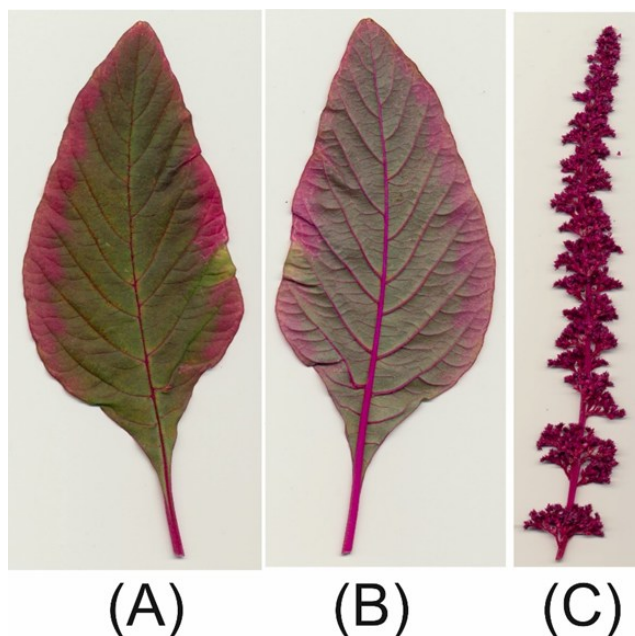


Fig. 1 Photographs of different plant parts of Amaranth. (A) leaf top side; (B) leaf bottom side; (C) inflorescence.

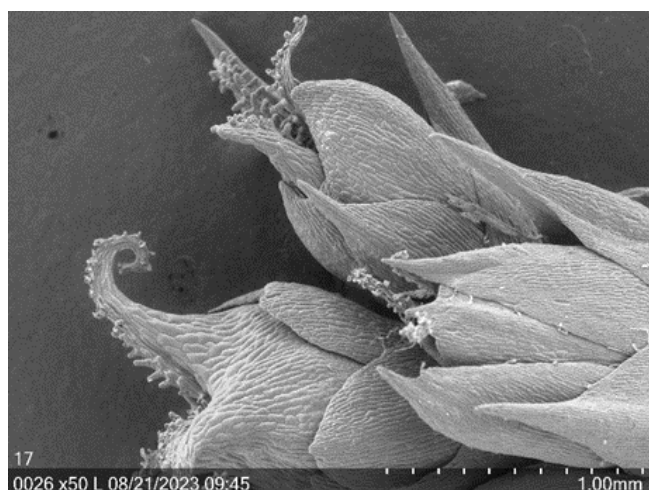


Fig. 2 Scanning electron microscopic (SEM) image of the inflorescence from Amaranth.

The antioxidant properties of Amaranth seed, leaf and flower pieces are reported [29]. Also, the production of natural food colorants from the Amaranth plant is described [30, 31]. A special application of Amaranth based dyes is the use as light-harvesting component in dye-sensitized solar cells [32,33]. Further applications are related to food packaging and antioxidant properties [34,35]. The Amaranth plant is reported to contain the colored components mainly in the leaves and the inflorescence [36]. Two main colored components are reported for Amaranth – Amaranthine and Rutine [36]. The chemical structures of both compounds are presented in Fig. 3 and 4. However, also a broad range of other various types of colored components related to betaines can be extracted from Amaranth plants, e.g. by using a methanolic based extraction procedure [37]. Amaranthine is also named as 2-(O-glucuronosylbetanin) ($C_{30}H_{34}N_2O_{19}$ / 727 g/mol). Additional to the betanin chromophore unit, this molecule contains two attached glucose units (Fig. 3). Altogether this molecule exhibits three units of carboxylic acid. For this, Amaranthine is soluble in water and if it is solved in water it gains a negative net charge by deprotonation of the acid groups. Due to the negative net charge, a good solubility in water and a good affinity to wool fabrics can be expected comparable with acid dyes.

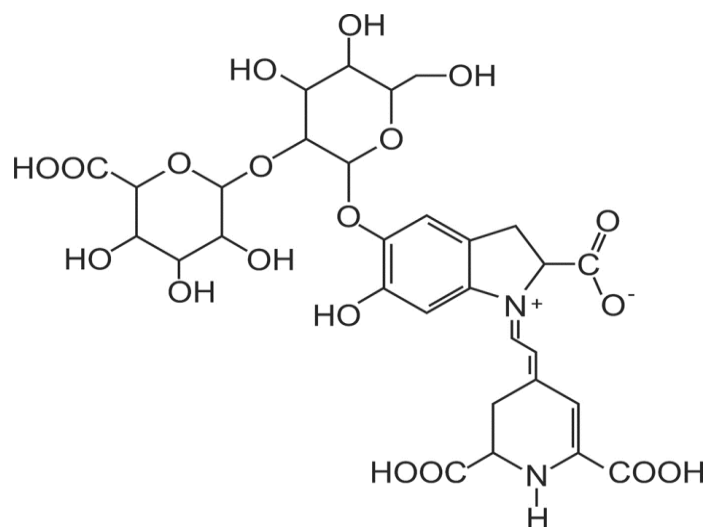


Fig. 3 Chemical structure of Amaranthine as one main colored component in the Amaranth plant.

Rutine is also named as Rutoside or Quercetin rutinoside ($C_{27}H_{30}O_{16}$ / 611 g/mol) [24]. It is indicated as C.I. 75730 and C.I. Natural Yellow 10. Similar to Amaranthine, Rutine exhibits two glucose units attached to a flavonoid chromophore (Fig. 4). Due to the high number of hydroxy groups, a certain solubility in water and good affinity to hydrophilic natural fibers like wool or cotton can be expected.

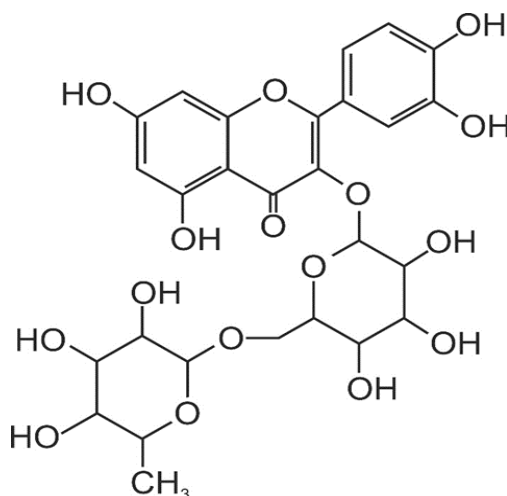


Fig. 4 Chemical structure of Rutine as one main colored component in the Amaranth plant.

2 Experimental section

2.1 Materials

For dyeing experiments, a plain-woven cotton fabric is used. This fabric is bleached and exhibit a weight per area of 200 g/m². Additional for dyeing experiments a multi-fabric test specimen is used containing the following materials: wool – WO, acrylic – PAC, polyester – PES, polyamide – PA, cotton – CO, cellulose acetate – AC. The Amaranth plants used for extraction and dyeing experiments are harvested in September 2023 in Hückelhoven-Baal (Germany). They are from the species *Amaranthus hypochondriacus* and grown from seeds supplied by the company Saatgut Dillmann (Berglen, Germany) [38]. For documentation purposes and quality control, the IR spectra of different part of the Amaranth plant are recorded and presented in Fig. 5. For cleaning of cotton fabrics before dyeing experiments, the detergent Perlavin LMO is used as washing agent. Perlavin LMO is supplied by Dr. Petry Textilchemie GmbH (Reutlingen, Germany). As mordanting agents, iron (II) sulphate and aluminum acetate are used. The iron (II) sulphate – FeSO₄ 7 H₂O – is gained from Merck KGaA (Germany) and the aluminum acetate – Al(CH₃CO₂)₂OH – is gained from Fa. Bernd Kraft GmbH (Germany).

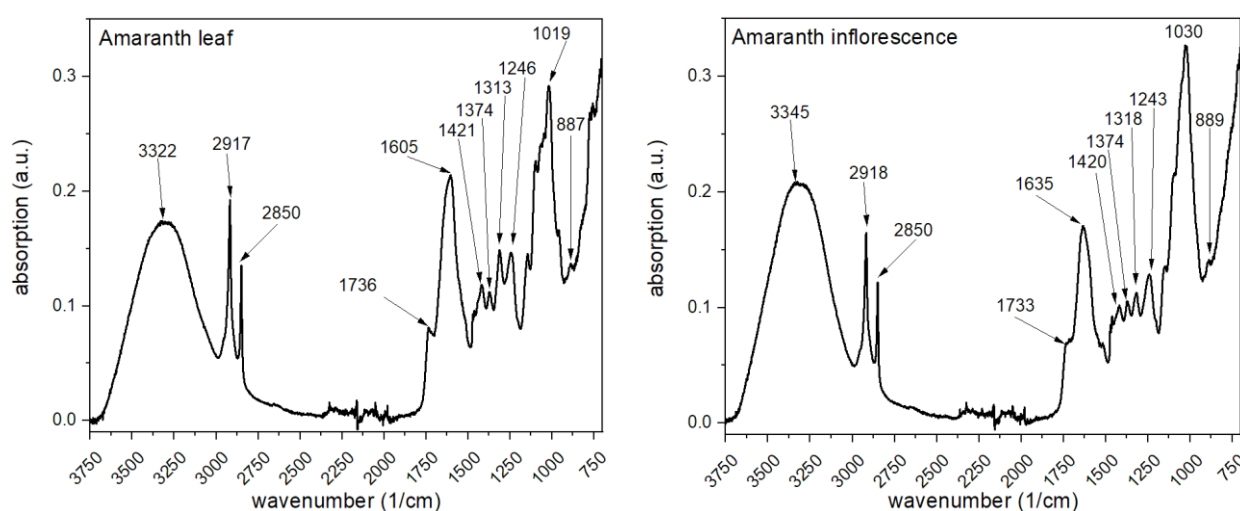


Fig. 5 Infrared spectra recorded from different parts of Amaranth plant – left: IR spectrum recorded from the leaf; right IR spectrum recorded from the inflorescence.

2.2 Extraction

The extraction of dye from the freshly harvested Amaranth plants is done from the inflorescences of the plants. For this, the inflorescences are carefully separated manually. After separation, the selected flowers are finely pureed. Water is added and mixed with a hand blender processed into a homogeneous mass. The mass ratio is adjusted to 10 g plant to 40 g water. This finely pureed mass is then used poured through a sieve. This mixture is kept at 80 °C while stirring regularly. The received red extract is finally used for dyeing experiments. A photograph of the gained red solution is presented in Fig. 6.



Fig. 6 Extract red solution from inflorescence of Amaranth.

The optical spectrum of absorption from the solution of Amaranth extract is presented in Fig. 7. For recording this spectrum, the original extract is diluted by water with a ratio of 1:10 to realize absorption values below 1.0 in the visible range. The spectrum is recorded from UV range till NIR range (200 nm to 1400 nm). To support an enhanced view on the visible spectral range, a part of the recorded spectrum from 400 nm to 800 nm is additionally presented (Figure 7). The absorption of UV light is significantly higher compared to the absorption of visible light of the Amaranth extract solution. Compared to this, NIR light is nearly not absorbed by the liquid extract. The absorption spectrum exhibits a small maximum at 672 nm. Further, two shoulders around 540 nm and around 420 nm are determined. The spectrum for visible light is in good accordance to literature reporting extraction of Amaranth using a water based procedure [32]. Using water for extraction, the red colored components are transferred from plant to extracts. Other extraction processes which are using also organic solvents often also transfer other plant based colored components into the extract and are therefore less selective according to the type of extracted dye [32].

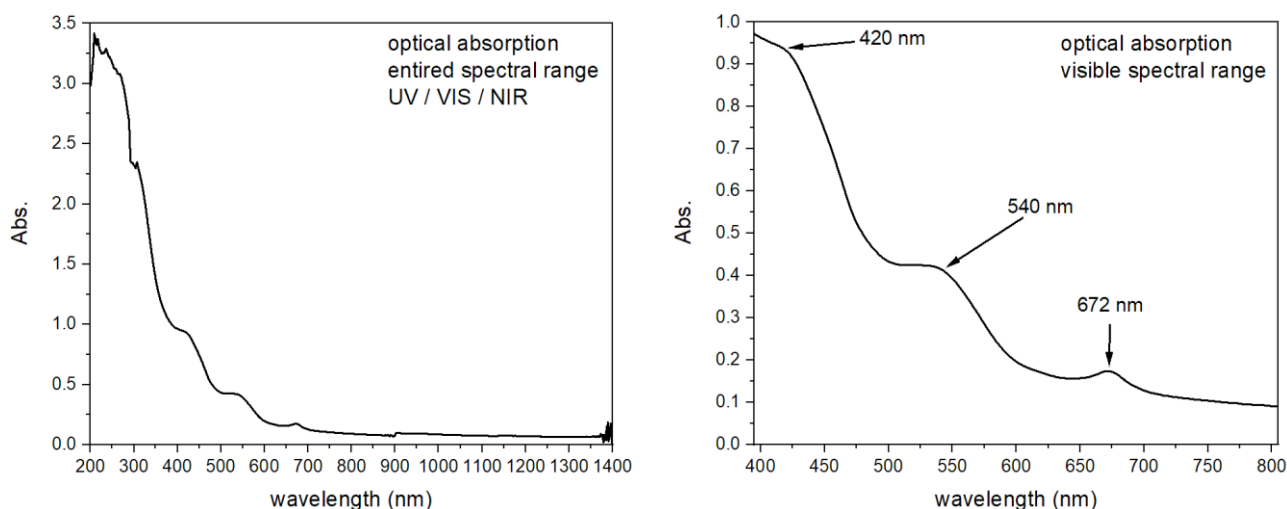


Fig. 7 Optical spectra recorded from extract solution from inflorescence of Amaranth – left: spectrum including UV and NIR range; right: spectrum for visible light. For spectral measurements the original extract solution is 1:10 diluted.

2.3 Dyeing procedures

Before dyeing and mordanting, the used cotton fabrics were washed using the detergent Perlavin LMO (Textilchemie Dr. Petry GmbH, Reutlingen). An amount of 1 g of this detergent is given in one liter of water. The washing is done for 15 minutes at a temperature of 60 °C using an industrial washing machine HC60 (IPSO, Belgium). The washing procedure is followed by line drying. For mordanting and dyeing process, a dyeing machine Datacolor Ahiba IR Pro is used. For mordanting, the cotton fabrics are pretreated before the dye application with the mordants iron (II) sulfate FeSO_4 or aluminum hydroxy acetate $\text{Al}(\text{CH}_3\text{CO}_2)_2\text{OH}$. For mordanting, 100 mL of solutions from these salts are prepared containing 0.1 g, 0.5 g or 1.0 g salt. The cotton fabric sample and a multi-fabric are placed into this 100 mL solution. The heating rate is set to 2 K/min until the process temperature of 80 °C is reached. This process temperature is kept for 60 minutes. After cooling down to 50 °C, the fabric samples are taken out and rinsed with cold soft water to remove excess stain. After rinsing, the tissue samples are line dried at room temperature in the dark. Flow charts illustrating the process temperature as function of process duration are presented in Fig. 8 for both the mordanting and the dyeing procedures.

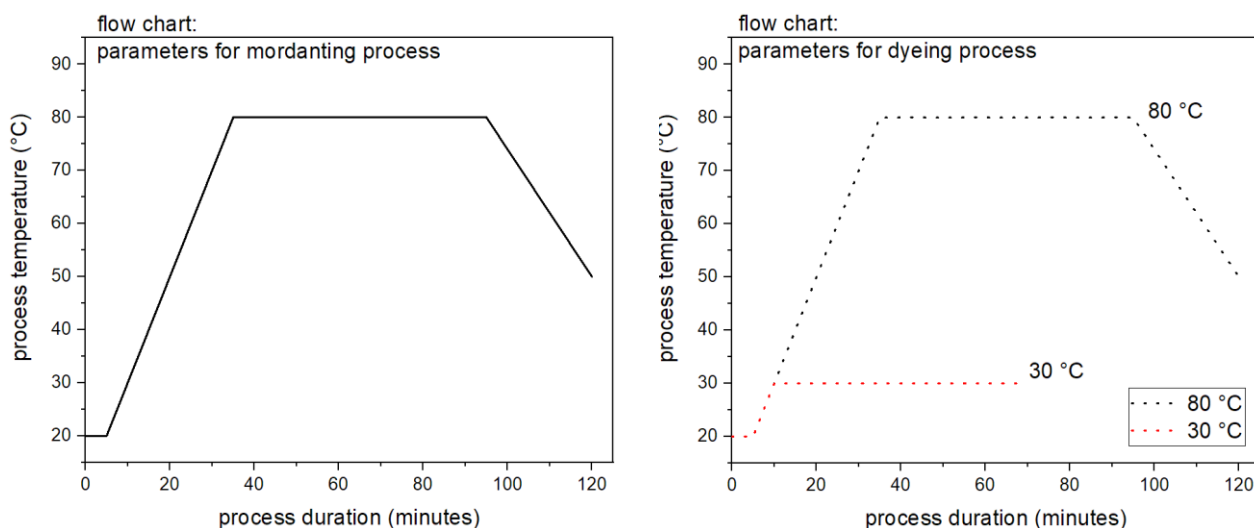


Fig. 8 Flow charts for mordanting and dyeing processes illustrating the process temperatures as function of process duration.

Finally, six differently mordanted fabrics are used for current dyeing experiments. Also, a not mordanted fabric is dyed as reference. The following dyeing experiments are carried out at two different dyeing temperatures at 80 °C and at 30 °C (cf. Fig. 8). As dye bath, the Amaranth extract is used as prepared. The specified weight ratio between the fabrics samples and the dye bath is set to 20% to 80%. The heating rate to reach the dyeing temperatures is set to 2 K/min and the dyeing temperature is kept for 60 minutes. Finally, after cooling down, the dyed fabrics are removed from the vessel and 500 mL of water are used for rinsing to remove excess dye from the fabric. After rinsing, the fabric samples are line dried at room temperature in the dark.

2.4 Analytics

Magnified photographs from the inflorescence of Amaranth are taken with two different digital microscopes. Magnified photographs under illumination with visible light are taken using a Dino-Lite Premier Digital Microscope. A digital microscope PCE-MM200 is used to record photographs with UV light for illumination. Scanning electron microscopy (SEM) is used to realize microscopic images of Amaranth plant with higher magnification. For SEM investigations, a TM-4000 tabletop microscope supplied by Hitachi is used. Fluorescence emission spectra are determined from Amaranth extract using a RF-6000 spectrofluorometer (Shimadzu). With this fluorescence spectrometer, also the record of 2D fluorescence spectra is possible. The optical absorption spectrum of the Amaranth extract solution is determined by using a photospectrometer UV-2600 (Shimadzu). The absorption spectrum is recorded for the spectral range from 200 nm to 1400 nm. By this, the spectral ranges of UV light, visible light and the near infrared light (NIR) area are covered. Infrared spectra (IR spectra) are recorded from freshly harvested Amaranth plants without further drying. For this, an FT-IR spectrometer IRTracer-100 from Shimadzu (Japan) equipped with a Specac Golden Gate ATR unit is used.

2.5 Testing of fastness properties

The wet and dry rubbing fastness of dyed cotton fabric is measured using a crockmeter according to ISO 105-X12. For this rubbing test, specimens of the textile are rubbed with a dry rubbing cotton test fabric and with a wet rubbing cotton test fabric. The change of coloration is determined using a Gray Scale evaluating the staining of the rubbed test fabric. The washing fastness is tested according to ISO-CO6:2010 at washing temperature of 40 °C. The change of coloration after washing is as well determined using a Gray Scale. By Gray Scale, grades of 1 to 5 are given. In this grading system, the grade 5 indicates the best fastness property.

3 Results and Discussion

3.1 Fluorescence properties

The fluorescence properties of Amaranth plant and the produced Amaranth extract solution are investigated by different methods. In fact, the fluorescent properties of plant leaves are intensively reported in the past years [39-41]. The detected fluorescence from plants is caused by the component chlorophyll [39,40]. However, also other plant components are reported to cause fluorescent effects of plants [41]. With this background, it is also of interest to view on possible fluorescence properties of Amaranth. First investigations are done by digital microscopes using different types of light for recording the images (Fig. 9). By using visible light for taking a magnified photograph from the inflorescence of Amaranth, the red coloration of this plant is clearly visible. However, by using UV-light for taking the image, the color of Amaranth appears white to bright pink (Fig. 9). By illumination with UV-light an object only appears in a certain color if it contains fluorescent properties. By this simple image, it is therefore clear that the Amaranth plant exhibits fluorescence properties as other plants also do. Further, a magnified photograph of a leaf from Amaranth is taken under illumination with UV-light (Fig. 10). Here, the pink fluorescent coloration is clearly visible for areas of the leaf containing the red coloration. Green colored areas of the leaf exhibit less fluorescent activity.

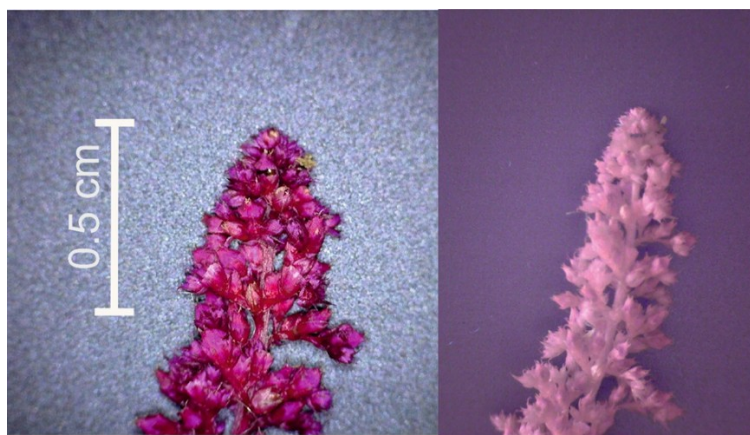


Fig. 9 Magnified photographs from inflorescence of Amaranth – left: with visible light; right: with UV light.

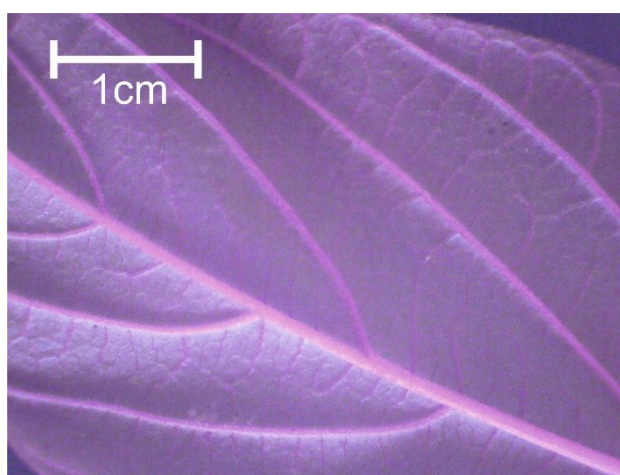


Fig. 10 Magnified photograph from leaf of Amaranth (bottom side) taken under illumination with UV light.

Following the question arises if the extract from Amaranth also exhibits fluorescence properties. This would be the case if the fluorescent component is also extracted and transferred to the liquid extract. The fluorescence spectra recorded from the extract are presented in Fig. 11. For these spectroscopic measurements, the extract is diluted 1:10 with distilled water. The emission spectra are recorded for excitation with light of wavelength 360 nm and 380 nm. Here a broad emission maximum is observed at 482 nm for excitation at 360 nm and at 492 nm for excitation at 380 nm. The strong emission maximum at 679 nm stands for the emission of red light, while the broad maximum at shorter wavelengths is more

likely to a white fluorescence appearance. This color appearance fits quite well to the observation of the plant in microscopy illuminated with UV light (cf. Fig. 9). The excitation spectrum of the extract recorded for emitted light at 480 nm is also presented in Fig. 11. It is recorded for the spectral range from 200 nm to 400 nm. Beside a small maximum at 240 nm, a broad strong maximum is observed at 375 nm. This broad maximum at 375 nm fits well with the emission of the UV lamp of the used microscope, which is in the UV-A range.

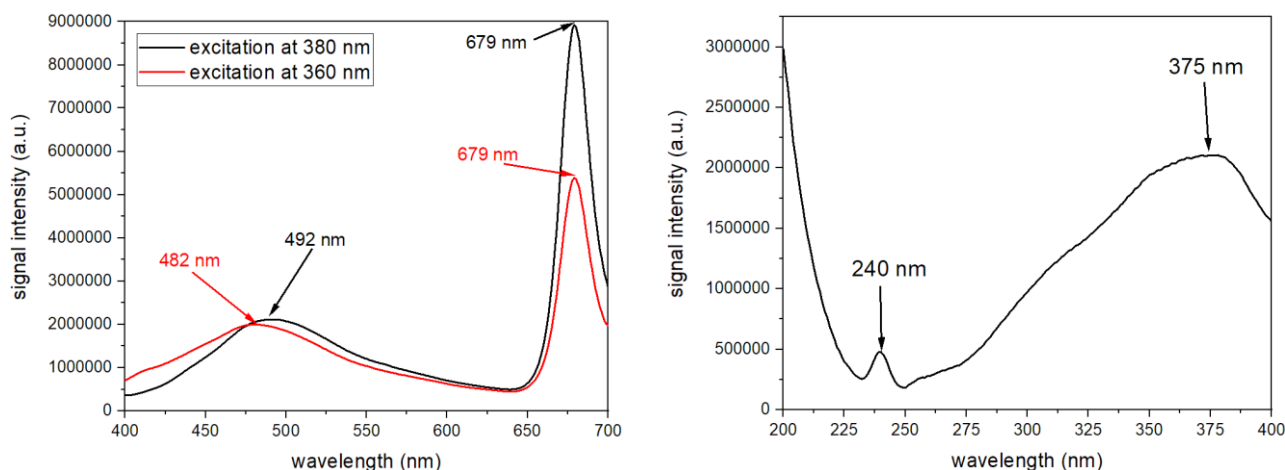


Fig. 11 Fluorescence spectra recorded from extract solution from inflorescence of Amaranth – left: emission spectra for excitation with light at 360 nm or 380 nm; right: excitation spectrum for light emitted at 480 nm.

In addition to the recording of single fluorescence spectra, also a complete 2D fluorescent spectrum is recorded for the Amaranth extract (Fig. 12). In this 2D fluorescence spectrum, a diagonal maximum can be observed, which is related to a feature of the sample holder and the measurement device and not by the measured sample. The most prominent signal from the sample is related to an emission maximum around 680 nm excited by light in the range of 350 nm to 400 nm. This strong signal is in good accordance to the observation made by the single fluorescence spectra (compare Fig. 9 and 11). A second weak maximum is also observed for an emission of blue light around 480 nm excited by UV light around 375 nm. By these fluorescence measurements, the fluorescence properties of the Amaranth extract are clearly determined. However, by fluorescence measurements performed on the dyed fabrics, no fluorescence properties related to Amaranth are determined, so in the actual dyeing process the fluorescent effect cannot be applied by Amaranth application onto textile fabrics.

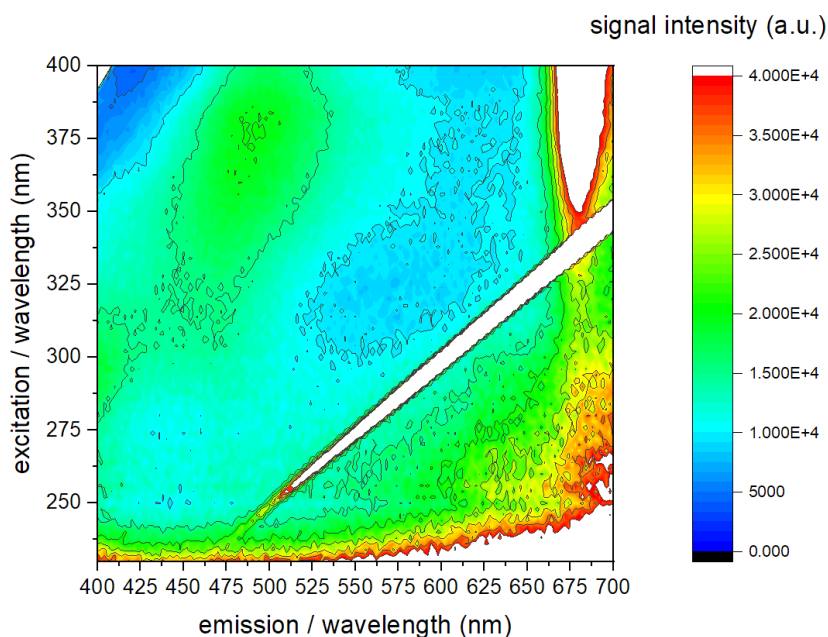


Fig. 12 2D fluorescence spectrum recorded from extract solution from inflorescence of Amaranth.

3.2 Dyeing results

The dyeing results are documented by photographs of samples directly after removing from the dyeing vessel (Fig. 13) and after the complete process including the drying of the textile samples (Fig. 14). The wet samples directly after the dyeing process exhibit different coloration depending on the used temperatures in the dyeing process (Fig. 13). If the dyeing is performed at 30 °C process temperature, the sample exhibits a red coloration which is more intense with mordant compared to the cotton sample dyed without any mordanting. In contrast, with a process temperature at 80 °C, the textile samples appear in yellow to brown coloration. With iron mordanting, the color intensity is stronger (Fig. 13). By this observation it can be stated the colored component from Amaranth extract probable changed its properties at high dyeing temperatures.

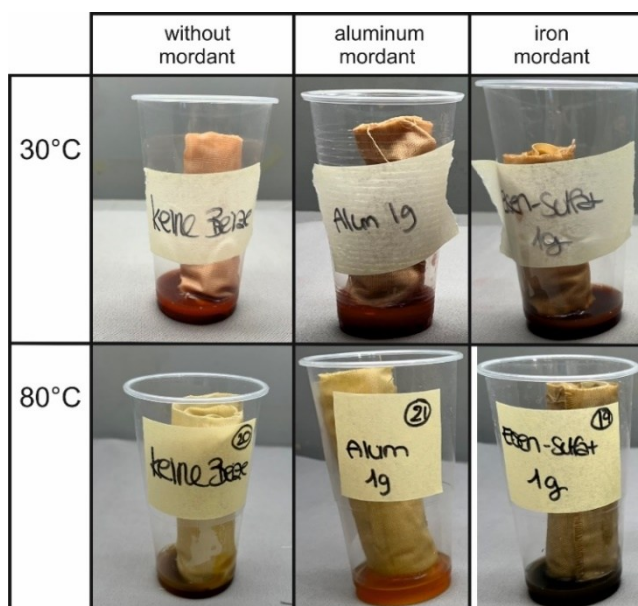


Fig. 13 Photographs of cotton fabrics directly after the dyeing procedure. Shown are samples prepared with different mordanting agents and different dyeing temperatures (30 °C or 80 °C).

After drying the textile samples, the non-mordanted samples exhibit only slight coloration and any remained red coloration disappears and turned into brown. Especially with iron mordanting, a strong color intensity is gained, which is especially intensive for application on wool or cotton from the dyed multi-fabric (Fig. 14).

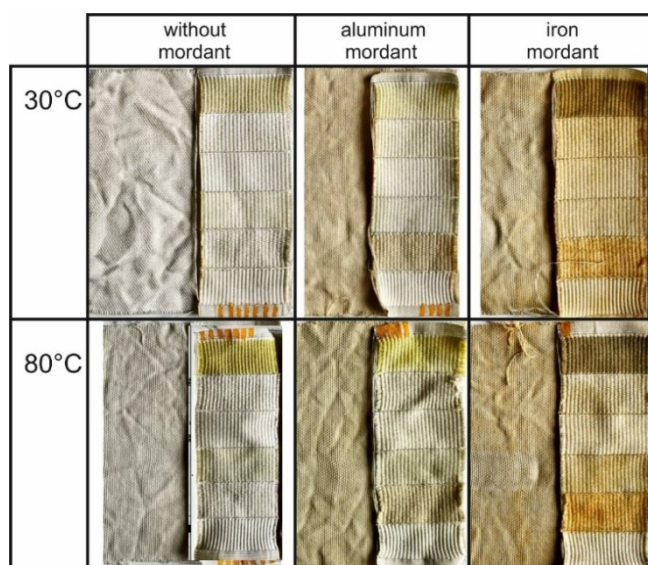


Fig. 14 Photographs of cotton fabrics and multi-fabrics after dyeing with Amaranth extract, using different mordanting agents and different dyeing temperatures (30 °C or 80 °C). The multi-fabric contains following samples (up to down): wool—WO, acrylic—PAC, polyester—PES, polyamide—PA, cotton—CO, cellulose acetate—AC.

Finally, it can be stated that the red coloration of Amaranth is not stable during the drying process and a brown coloration remains. One possible explanation for this color change can be an oxidation of the colored components during drying at air. With the used method, no red colored textile fabrics can be realized for dyeing with Amaranth.

3.3 Fastness properties

The fastness properties are determined for the dyed cotton fabric and shown as function of increasing concentration of applied mordanting agent (Tables 1 and 2). Table 1 summarizes the results with aluminum mordant and Table 2 presents results with iron mordanting. Using the lower dyeing temperature at 30 °C leads to insufficiently low washing fastness, independent from the type of mordant or mordant concentration. In cases the dyeing is done at 80 °C, the washing fastness is medium to good and best values are gained with iron mordanting applied with higher iron concentration. By view on the rubbing fastness, this trend is less clear.

Table 1. Overview on rubbing and washing fastness of realized samples with alum mordanting.

Mordanting agent used amount	Dyeing temperature (°C)	Rubbing fastness dry	Rubbing fastness wet	Washing fastness
None	30	4-5	4-5	2
Alum / 0.1 g	30	4-5	4-5	2
Alum / 0.5 g	30	4-5	4-5	1
Alum / 1.0 g	30	4-5	4	1
None	80	5	5	3-5
Alum / 0.1 g	80	4-5	4-5	3-4
Alum / 0.5 g	80	3-4	3	3
Alum / 1.0 g	80	4	3-4	3

Table 2. Overview on rubbing and washing fastness of realized samples with iron mordanting.

Mordanting agent used amount	Dyeing temperature (°C)	Rubbing fastness dry	Rubbing fastness wet	Washing fastness
None	30	4-5	4-5	2
Iron / 0.1 g	30	4-5	4	2
Iron / 0.5 g	30	4-5	3-4	2-3
Iron / 1.0 g	30	3-4	3-4	2-3
None	80	5	5	3-5
Iron / 0.1 g	80	3-4	3-4	3-4
Iron / 0.5 g	80	2-3	3	4
Iron / 1.0 g	80	1	1-2	4-5

4 Conclusions

The dyeing of textile fabrics with Amaranth extract is possible and with iron mordanting good color intensity is gained. The used dyeing temperatures have less influence on the received color intensity but the dyeing at higher temperatures leads to better washing fastness. However, the original intensive red coloration of the Amaranth plant and extract cannot be transferred to the dyed textile. Finally, a brown coloration appears after dyeing with Amaranth. Also, the fluorescent properties determined for the extract of Amaranth is not determined after application on textile. By this it can be concluded that the colored components in Amaranth are not stable in a textile dyeing process. This low stability limits probably the use of Amaranth for purposes of textile dyeing.

Author Contributions

Hwanhee Lee: extract procedures, dyeing experiments, spectroscopic measurements, textile testing; Boris Mahltig: supervision, writing, review, editing, spectroscopy, microscopic measurements, planting and harvesting the Amaranth plants. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest

The authors declare no conflict of interest.

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