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Overview on natural dyes and their IR-spectra – part V: Plant based dyes

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ABSTRACT

This paper offers a broad overview on several natural dyes from plant origin. By this it is the fifth part in a series of review papers on natural dyes, their properties and infrared spectra. Basic information on origin of natural materials and dyes and their fields of application – especially in the field of textiles – is supported. The chemical structures of dyes are presented together with the measured infrared spectra (IR spectra) of 28 different dye products from various origin. A structure related discussion of the spectra is performed. Main aim of this overview is to offer a broad overview for people interesting in natural plant dyes. Further, the support of IR spectra is a helpful tool for material identification and quality control.

1 Introduction

Natural dyes are fascinating chemical compounds which are connecting the property of coloration with the complexity of the chemical structures of dye molecules composed by chromophores and auxochromic groups. The use of natural dyes goes back to ancient times [1-3]. Due to the popularity of certain colors accompanied by limitation in production capacity, the production and trade of certain natural dyes were the reason for wealth and prosperity. Prominent examples are found in the prominent dyes Indigo and Tyrian Purple [2,3]. Nevertheless, by invention of synthetic dyestuffs from upcoming chemical industry, the importance of natural dyes decreased drastically. Several reasons can be mentioned, as the broad availability of synthetic dyes at considerable low cost. Also, the independence from natural resources is important. Further, the chemical synthesis allows the realization of dye stuff with extraordinary light and wash fastness [4,5]. Examples in field of strong light fastness are phthalocyanine dyes [6]. With the invention of reactive dyes, excellent washing fastness on cotton was achieved [7-9]. For dyeing of the most used synthetic fiber PET, the application of synthetic disperse dye is dedicated [10-12]. With this background, nowadays natural dyes are rarely used in the field of textile dyeing and coloration, even if there is a broad range of popular literature from the handicraft sector reporting various types of recipes and application techniques with natural dyes [13,14]. Still important are natural dyes in the in the area of food and beverage coloration [15,16]. A prominent example in this field is the application of natural dyes in sausages [17,18]. Commercial use is also reported for cosmetics, as e.g. lipsticks [19]. In the textile sector a revival for the use of natural dyes is part of a discussion on the demand for sustainable and fully biodegradable products [20,21]. For a fully biobased textile product not only the fiber material should by naturally based and by this biodegradable. Also, the dyes are demanded to be degradable after the use of the dyed textile material is terminated [22]. This demand is usually not fulfilled by synthetic dyes, so for this aspect the natural dyes are superior. Due to this, in recent years some excellent papers are published comparing different natural dyes and their application onto different types of natural fibers [23,24]. A broad range of achievable colorations are realized [25, 26]. However, it should be clear a natural compound is not automatically non-toxic or even healthy. In fact, some of the most effective poisons are bio-based natural compounds which are even more toxic as synthetically produced poison gas [27]. For consideration of possible toxicity and sustainable issues, currently new standards and certificates are developed evaluating natural dyed textile products and natural organic dyes [28]. To the relevant topic of natural dyes and their application on textiles, a series of overview papers related on natural dyes commercially available on the German market is launched in the beginning of year 2024 with reporting an anthraquinone and naphthoquinone based dyes [29]. Following parts of the overview series are dedicated to indigo related dyes, wood based dyes and insect based natural dyestuffs [30-32]. The actually presented overview paper is dedicated to plant based natural dyes and cover a very broad range dyes differing in chemical structure but also in type of plant origin. For each discussed dye a short background on origin and application is given. The chemical structures of main colored components are shown and discussed. The IR spectra of different available dye products gained from different suppliers are presented. Here a structural discussion of IR spectra and chemical structure is supported. By this also the comparability and identification of dye products are shown and be part of further discussion. However, not only the structural understanding of IR spectra is supported, but also the identification of natural dye products can be enabled.

The IR spectroscopy records the absorption capability of compounds for infrared light as function of wavelength. Usually the absorption maxima of a chemical compound are assigned to different types of vibration in the related chemical molecules [33,34]. By this, the identification of a compound is possible and the presence of certain organic functional groups can be verified [33,34]. A prominent example is the identification of proteins in deposited polymer layers [35]. Similarly, the IR spectroscopy is used for identification of synthetic and natural fibers [36-38]. Also, various fiber based materials as artificial leather or composites can be investigated by use of IR spectroscopy [39]. Advantageous is especially the IR spectroscopy applied in attenuated total reflectance mode (ATR-mode) which allows the sample analysis without sample destruction [40].

The IR spectroscopy is used for identification of natural dyes in historical textiles [41,42]. In recent years few reviews were published reporting on natural dyes and their IR spectra [43]. Unfortunately, these reviews exhibit several disadvantages as limited selection of natural dyes, limited spectra quality and lack of structural discussion of the IR spectra. With this background, the aim of the actual overview is to support a broad view on natural dyes commonly available on the German market. If available different natural dye products from the same dye gained from different sources are compared. Finally, a broad compendium of dye related IR spectra are presented and discussed, which is a helpful tool for scientists and technician working in the dyeing sector and quality control.

2 Materials and Methods

2.1 Dyestuffs

Several plant based dyes are investigated. Together with reference materials, altogether 28 dye products are reported with their IR spectra. These dyes are supplied by six different companies – Kremer Pigmente GmbH (Aichstetten, Germany), Pflanzenfärbershop (Hückelhoven-Baal, Germany), Das

Wollschaf (Zweibrücken, Germany), Narayana Verlag GmbH (Kandern, Germany), AMA Herbal (India) and Carl Roth GmbH (Karlsruhe, Germany). A Curcuma root and saffron fibers are purchased as reference materials from supermarkets in Germany. These materials are given as powder, seed or complete piece. For some dyes additionally the related synthetic chemicals are measured as reference materials. These reference materials are supplied by the companies Carl Roth GmbH (Karlsruhe, Germany) and Merck (Darmstadt, Germany). Detailed information on the reported materials are given in Table 1 together with the supplier and the product webpage of the supplier supporting further information on application, recipes and safety information.

Table 1. Reported plant based natural dye stuffs and reference components.

No.	Content	Form	Supplier	References
P1	Curcumin	Powder, chemical	Roth	[44]
P2	Curcuma root	Powder	Pflanzenfärbershop	[45]
P3	Curcuma / Indian	Powder	Kremer	[46]
P4	Curcuma	Cut pieces, dried root	Kremer	[47]
P5	Curcuma	Complete root	German supermarket	---
P6	Beta-Carotene	Powder, pure chemical	Merck	[48]
P7	Annatto	Seeds	Kremer	[49]
P8	Litmus / C.I.1242	Powder	Roth	[50]
P9	Litmus	Powder	Kremer	[51]
P10	Gallic acid	Powder, synthetic chemical	Merck	[52]
P11	Gallnut	Powder	Pflanzenfärbershop	[53]
P12	Gallnut	Powder	Wollschaf	[54]
P13	Gallnut	Piece	Wollschaf	[55]
P14	Gall Nut of *Querus infectoria*	Powder	Amaherbal	[56]
P15	Kamala	Powder	Kremer	[57]
P16	Reseda, extract	Powder	Pflanzenfärbershop	[58]
P17	Reseda	Cut pieces	Kremer	[59]
P18	Reseda	Cut pieces	Pflanzenfärbershop	[60]
P19	Catechu	Powder	Kremer	[61]
P20	Acecia catechu	Powder	Amaherbal	[62]
P21	Saffron	Red threads	German Supermarket	---
P22	Saffron	Red threads	Kremer	[63]
P23	Safflower	Dried flower	Pflanzenfärbershop	[64]
P24	Ratanhia root	Cut pieces	Pflanzenfärbershop	[65]
P25	Beetroot	Powder	Narayana	[66]
P26	Mulberry / Morus Alba	Powder	Amaherbal	[67]
P27	Pomegranate	Powder	Amaherbal	[68]
P28	Pomegranate	Dried piece cut	Pflanzenfärbershop	[69]

2.2 Analytical methods

All infrared spectra (IR spectra) are measured with an FT-IR spectrometer IRTracer-100 from Shimadzu (Japan) using a Specac Golden Gate ATR unit. Photographic images with enhanced magnification are taken by using a Dino-Lite Premier Digital Microscope. Scanning electron microscopy (SEM) is performed with a TM4000Plus Tabletop microscope from Hitachi (Japan). The SEM measurements are performed on the samples as received and no special treatment is done on the samples before microscopic investigations.

3 Discussion of Dyestuffs

3.1 Curcumin

Curcumin is the dye component gained from the root of the turmeric plant (*Curcuma longa* L.) [41,70]. It is named formally Natural Yellow 3 (color index CI 75300) and as food dye with the number E100 [1,71]. The chemical structure of Curcumin – also named as Diferuloylmethane – is shown in Figure 1. The sum formula is $C_{21}H_{20}O_6$. Here, two isomers related to a keto enol tautomer can be distinguished [72]. The Curcuma plant is well known for its positive effects on human health [73].

Curcuma extract can be applied on textile with and without mordant. The pre-mordanting with alum, iron sulphate and copper sulphate is reported and leads to improved light and wash fastness [74]. The dyeing of banana fiber fabrics with curcumin is reported to be done with pre-mordanting with alum or copper sulphate. Additional to color application for this combination also antibacterial properties are determined [75]. An interesting approach for dyeing with curcumin is the combination with a non-metal based natural mordant using for this purpose Aloe vera [76]. For dyeing application of Curcumin on synthetic fibers as polyester and polyamide, it is reported that by pretreatment of the synthetic fibers using UV light and ozone, the dyeability with Curcumin can be significantly improved [77]. An often-mentioned feature of Curcumin is the antimicrobial property gained for its application on textile substrates [74,78,79]. However, reports of antimicrobial properties of natural dye Curcumin should be evaluated carefully and a possible antimicrobial effect of the used metal mordants might be considered. As further functional property gained from Curcumin application, reports on UV protective properties should be mentioned [79]. In the field of UV protection, the yellow natural dye Curcumin can be used to optimize the performance of the inorganic UV absorber titanium dioxide TiO_2. In several patents the application of Curcumin as dye solved in different solvents is described. Here, mainly wool and silk fabrics are focused. Two Chinese patents describe the use of mixtures from organic solvents for applications of Curcumin [80, 81]. However, the use of organic solvents for dyeing purposes is for economic and safety reasons not applicable for most industrial textile dyeing processes. More advantageous is in comparison the use of supercritical carbon dioxide as solvent for Curcumin in textile dyeing processes [82, 83].

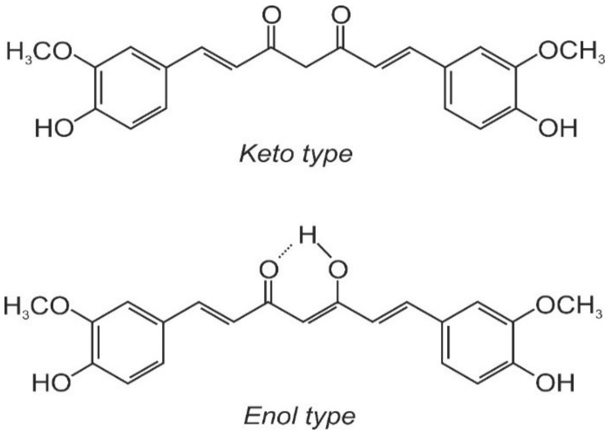


Fig. 1 Chemical structure of Curcumin. Shown are the structures of the keto and the enol type. The hydrogen bridge bond in the enol unit is shown as dotted line.

The magnified photographs of a root from Curcuma in cross section and from the surface is presented in Figure 2. The yellow/orange coloration of Curcuma is clearly visible in the cross section of the root.

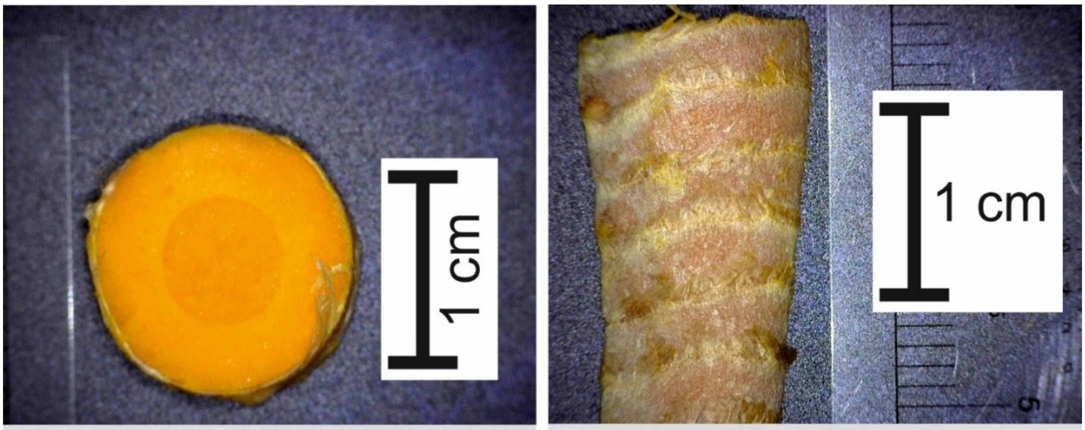


Fig. 2 Curcuma root (product P5). Photographic images with higher magnification. Cross section (left) – surface (right).

The infrared spectrum of pure Curcumin gained from chemical supplier Carl Roth GmbH is depicted in Figure 3. This determined IR spectrum is in good accordance to the IR spectrum reported in literature [84]. The broad signal with the maximum at 3325 cm^{-1} is related to O-H stretching vibrations typically for hydroxy groups -OH [33, 84]. Remarkable is the appearance of the single peak at 3505 cm^{-1}. A peak at this spectral range is more likely for a stretching vibration of an O-H bond from a carboxylic acid -COOH [33]. However, the curcumin molecule does not exhibit a carboxylic acid group. Alternatively, this peak can be explained by the O-H stretching vibration from the central hydroxy group of the enol type. Similarly to the carboxylic acid, here also due to the neighboring of the keto group a certain acidic property of the -OH unit can be estimated. The weak signal at 3017 cm^{-1} is assigned to C-H stretching vibration from hydrogen atoms bonded to an aromatic ring system or to units of conjugated C=C double bonds. The four peaks from 2849 cm^{-1} to 2974 cm^{-1} are related to different C-H stretching vibrations from aliphatic units, which is in case of curcumin the methyl group in the methoxy unit -O-CH_3 [33]. The signals at 1626 cm^{-1} and 1506 cm^{-1} are related to C=O stretching vibrations [84,85]. The peak at 1601 cm^{-1} is assigned to a C=C symmetric stretching vibration of the aromatic ring system. The signals at 1271 cm^{-1} and 1024 cm^{-1} are related to different C-O stretching vibrations [84,85].

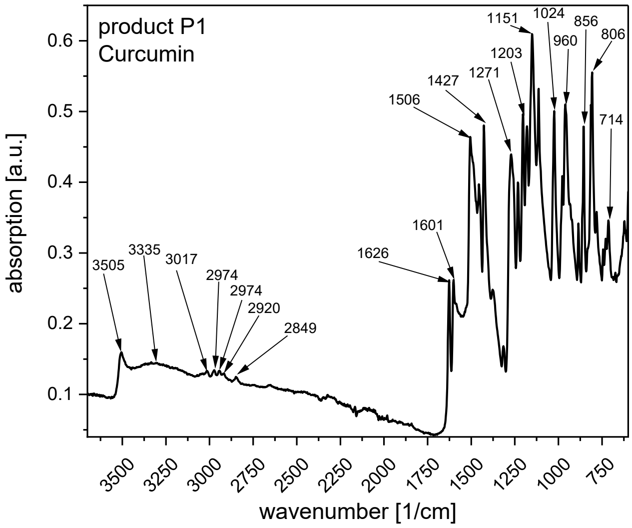


Fig. 3 Infrared spectrum of Curcumin (product P1).

Infrared spectra of Curcuma products from four different sources are presented in the Figures 4 and 5. All spectra exhibit a broad signal around 3290 cm^{-1} related to O-H stretching vibration from the hydroxy groups. For product P3 also a small shoulder signal at 3512 cm^{-1} is observed, which is in similarity to the clear peak at 3505 cm^{-1} of the pure curcumin product P1. Further, the signal at around 2925 cm^{-1} can be clearly assigned to C-H stretching vibration of aliphatic -CH_3 units. In the fingerprint area of all four spectra, typical signals at 1626 cm^{-1}, 1603 cm^{-1}, 1512 cm^{-1}, 1430 cm^{-1}, 1375 cm^{-1}, 1282 cm^{-1} and 1153 cm^{-1} can be observed and used for the identification of curcuma products. However, it should be mentioned that the IR spectra for these products differ in several features from the infrared spectrum reported by Priyadharsini et al. in a review from 2014 [43]. Nevertheless, the spectra quality and image quality in this earlier review is moderate to low, so a comparison to actually made infrared spectroscopic measurements is difficult [43]. Even if the IR spectra of the four Curcuma products are quite similar to each other, they exhibit significant differences to the IR spectra of pure Curcumin (compare Figures 3, 4 and 5). The Curcuma products contain additionally to Curcumin many other components like carbohydrates or proteins, so the occurrence of additional signals in the IR-spectra can be expected. The signal at around 1626 cm^{-1} can be assigned to a C=O stretching vibration either from the keto group in Curcumin or an amide group of also present proteins [33].

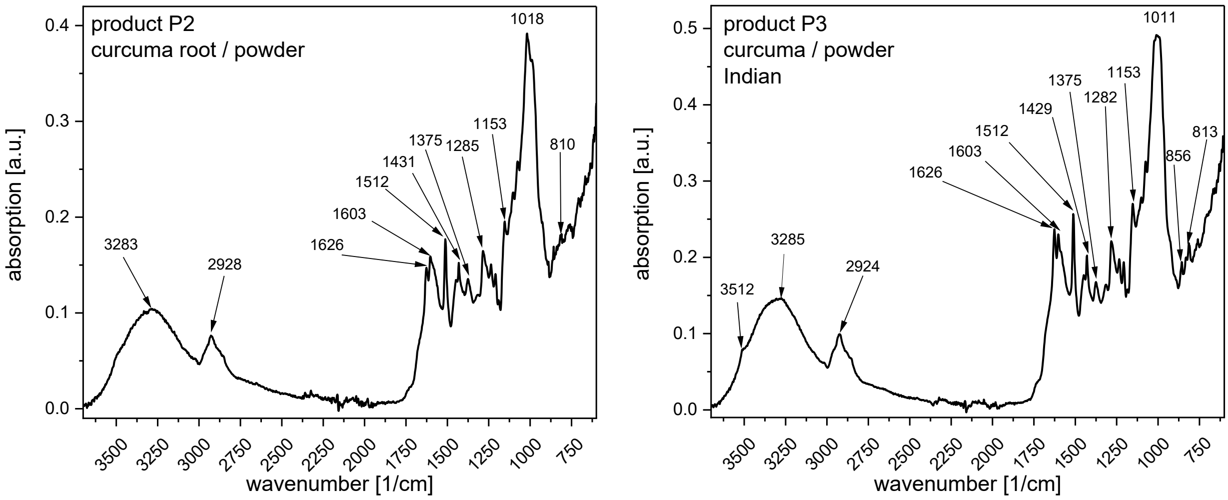


Fig. 4 Comparison of IR spectra of several Curcuma products. Shown are the products P2 and P3 (compare Table 1).

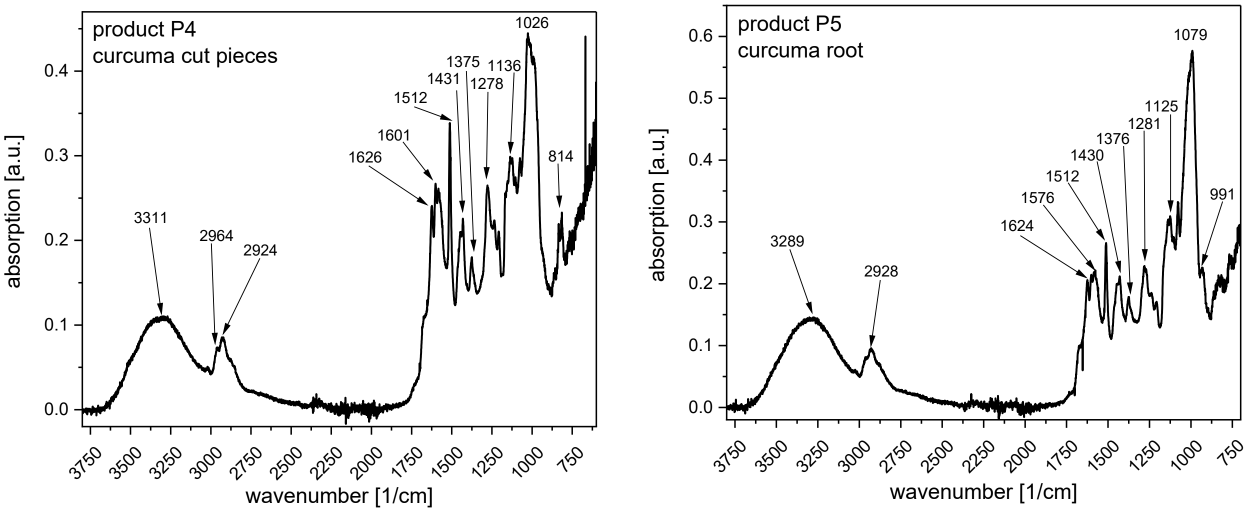


Fig. 5 Comparison of IR spectra of several Curcuma products – cut pieces. Shown are the products P4 and P5 (compare Table 1).

3.2 Carotene related dyes

From chemical point of view, Carotene related dyes are outstanding. First, they are built up only by the two chemical elements carbon and hydrogen. They do not contain any oxygen or nitrogen atoms, whose presence is typical for many types of dyes. Second, the chromophore does not contain any aromatic unit, instead it is a polyisoprenoid. The coloration is simple reached by the conjugation of linear arranged C=C double bonds. It is stated that more than 700 different types of carotenoids are found and characterized from different natural resources [1,86]. The chemical structure of beta-Carotene $C_{40}H_{56}$ is presented in Figure 6. Beta-Carotene (C.I.75130) occurs in different plant sources and plays a role as pro-vitamin A and as antioxidant. By this, there is a certain sensitivity against oxidation which limits the use for textile applications. However, in food coloration Carotenes exhibit high importance [86]. As food colorant carotenes are mentioned under the number E160a [71].

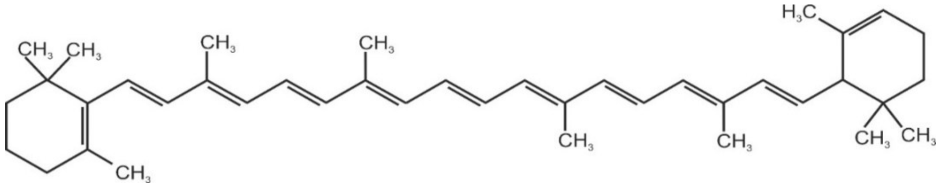


Fig. 6 Chemical structure of beta-Carotene.

An IR spectrum recorded from pure beta-Carotene is presented in Figure 7. The peak at 3028 cm^{-1} can be assigned to the C-H stretching vibration of hydrogen atoms placed at the C=C double bonds. The four signals from 2949 cm^{-1} to 2819 cm^{-1} are assigned to C-H stretching vibrations of aliphatic units, which are the $-CH_3$ methyl groups and the $-CH_2-$ units at the ring systems [33]. The signal at 1368 cm^{-1} is assigned to symmetric deformation of a C-H unit. The most prominent peak at 964 cm^{-1} can be assigned to an out-of-plane deformation vibration of a conjugated alkene -CH=CH- unit. The signal at 723 cm^{-1} is related to a rocking vibration of a $-CH_2-$ unit [87]. By view on the chemical structure of beta-Carotene it is obvious that many different types of isomers are possible depending on the cis/trans arrangement at the C=C double bonds. Depending on this structure arrangement on the C=C double bonds different IR spectra result. An overview on Raman and IR spectra of different isomers of beta-carotene is supported by Koyama et al. [88].

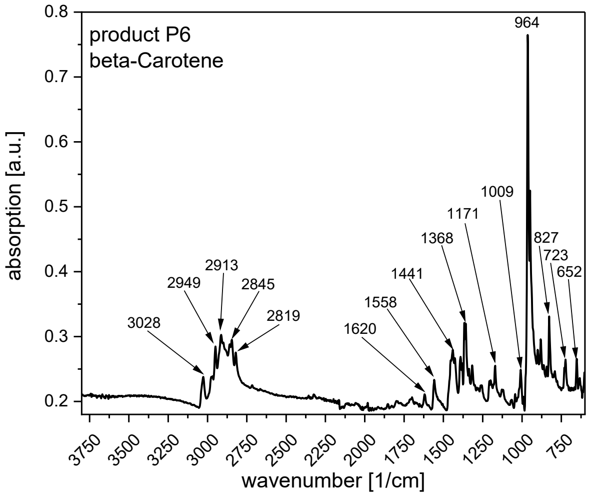


Fig. 7 Infrared spectrum of Carotene (product P6).

A natural dye based on an oxygen containing carotene structure is gained from the Annatto seed (C.I. Natural Orange 4) (*Bixa Orellana* L.) [70]. Annatto is a tropical small tree native in tropical America and nowadays planted worldwide in tropical regions [89,90]. Typically, Annatto dye is used in food industry, e.g. for cheese or cereals [91-93]. As food colorant Annatto is mentioned under the number E160b [71]. One main colored component in Annatto seed is Bixin (C.I. 75120) (compare chemical structure in

Figure 8) [1]. The chromophore of bixin is built up by a conjugated system of nine connected C=C double bonds, which is connected to a methylester group and a carboxylic acid attached at the oppositely end of the chromophore. Compared to Carotene, bixin does not contain aliphatic ring systems.

By deprotonation of the carboxylic acid group the bixin molecular gains a negative charged group, so bixin can be counted to the group of anionic dyes. However, due to the long alkyl chain it is a compound with also hydrophobic properties. It can be used as direct dye for dyeing of cotton, wool and silk [70,94,95]. Good results are achieved in combination with different mordanting agents as alum, ferrous sulphate or stannous chloride [94,95]. For dyeing of jute fabrics, the application of Annatto dye is reported in combination of alum and ferrous sulphate as mordants. Further several organic compounds are evaluated as mordanting agents [96].

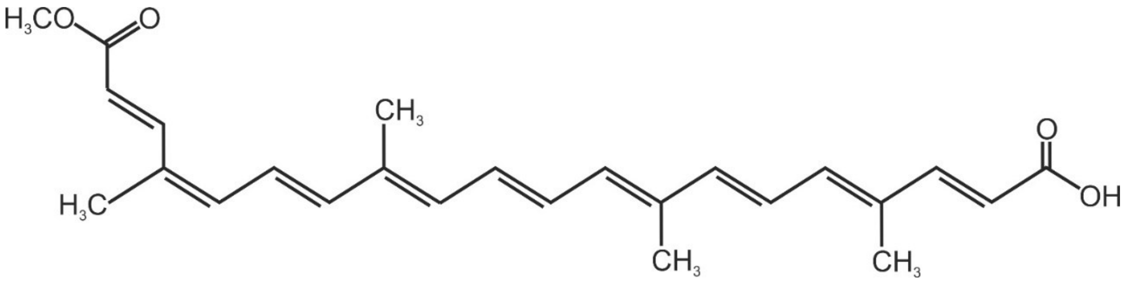


Fig. 8 Chemical structure of Bixin as one main colored compound in Annatto seeds.

Successful investigations are also reported for the application of Annatto dye for coloration of PET fiber materials. The application is done in HT-processes and the reached color fastness are comparable to the application of synthetic disperse dyes applied in the same regime [97,98]. Following this, also the dyeing of recycled PET fibers with Annatto dye is successfully investigated [99]. By this dyeing of recycled PET fibers with a naturally sourced dye, a further step in the development of sustainable textile products is done. A magnified photograph of Annatto seeds is presented in Figure 9. The red coloration of these seeds is clearly visible. The size of the seeds is in the range of two to three millimeters. A highly magnified view on the surface of an Annatto seed is possible by scanning electron microscopy – please compare Figure 10.

Fig. 9 Annatto seeds (product P7). Photographic image with higher magnification.

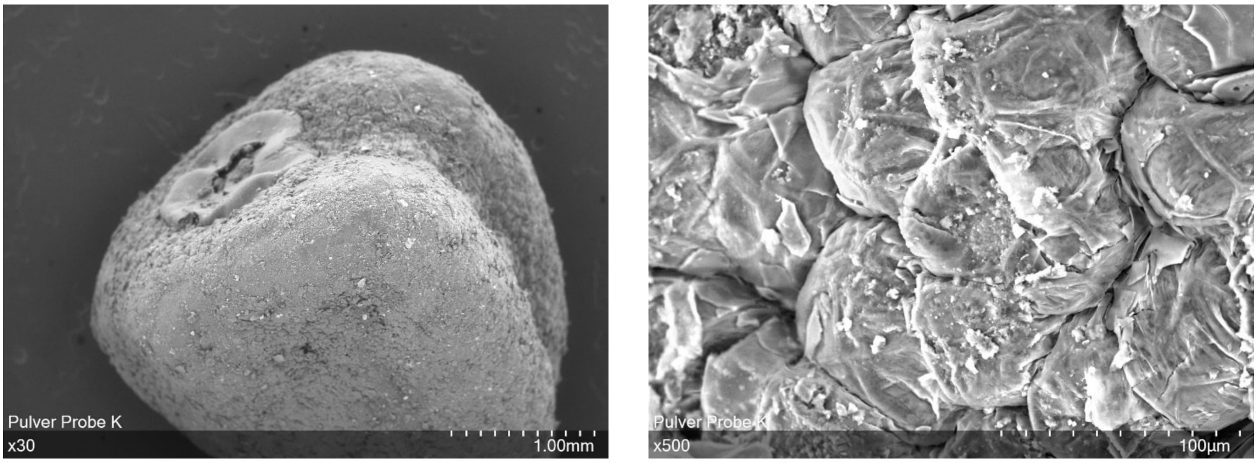


Fig. 10 Annatto seed (product P7). SEM images recorded with low magnification (left) and higher magnification (right).

An infrared spectrum recorded from a complete Annatto seed (product P7) is presented in Figure 11. The recorded IR spectrum is quite different compared to an IR spectrum for natural Annatto dye reported in literature [98]. However, another reported IR spectrum recorded from Annatto extracts from solid dye seeds is in good accordance to the IR spectrum recorded actually from Annatto seed product P7 [100]. Of course, the Annatto seed contains additionally to the red dye bixin also other components such as carbohydrates, proteins and fatty acids [93]. Nevertheless, a discussion of the IR spectrum according to chemical composition is possible. The broad signal at 3282 cm^{-1} is related to O-H stretching vibrations of hydroxy groups. The weak signal at 3034 cm^{-1} is probably assigned to C-H stretching vibration from hydrogen atoms bond to C=C double bonds. However, also a relation to an overtone vibration from amide groups in proteins might cause a signal in this spectral area [34].

The both signals at 2922 cm^{-1} and 2856 cm^{-1} are assigned to C-H stretching vibrations from aliphatic units, as $-CH_3$ and $-CH_2-$. The clear peak at 1717 cm^{-1} is assigned to a C=O stretching vibration of an ester group, as it occurs in Bixin. However, this signal may be related also to ester groups from fatty acids. The signal at 1609 cm^{-1} can be assigned to a C=C stretching vibration as part of a conjugated system [33].

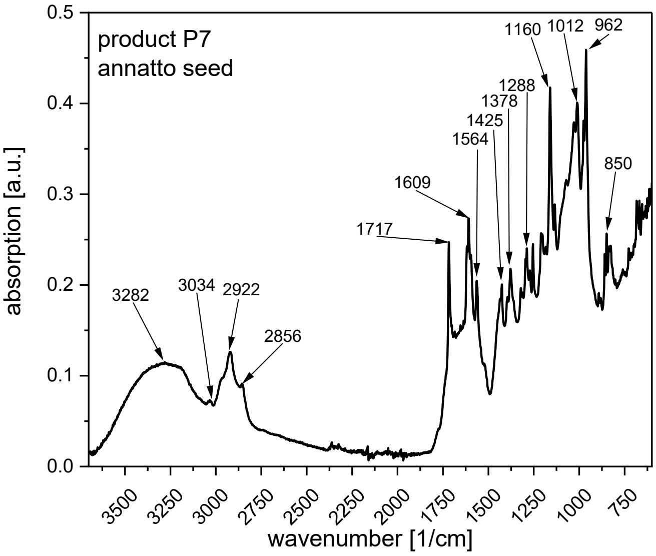


Fig. 11 Infrared spectrum recorded from an Annatto seed (product P7).

3.3 Litmus

Litmus dye (C.I.1242) is gained since centuries from different types of lichen, as e.g. *Rocella fuciformis* [101]. For production of Litmus dye from lichen different extraction processes are used [89]. One of the conventional uses for Litmus dye is the preparation of Litmus paper as indicator for acid/base reaction. Such Litmus indicator papers are used since more than one century [102]. The chemical structure of Litmus is presented in Figure 12 and it can be best described as an oligomer built up by aromatic units made from Orcein [89]. One basic unit in this oligomer is the 7-Hydroxyphenoxazone chromophore [103, 104]. Commonly Litmus is not used for textile dyeing, because of its color change around neutral pH 7 [87]. However, especially because of this pH dependent color change, the modification of textile fabrics with Litmus can be used to prepare textiles for monitoring pH changes, e.g. for monitoring of sweat [104]. In a current study a sol-gel process is used for covalent fixation of Litmus dye molecule onto textile surface. As anchoring component an epoxysilane compound is described [104]. The sol-gel technology is an excellent method to deposit and anchor dye molecules onto textile surfaces [105-107].

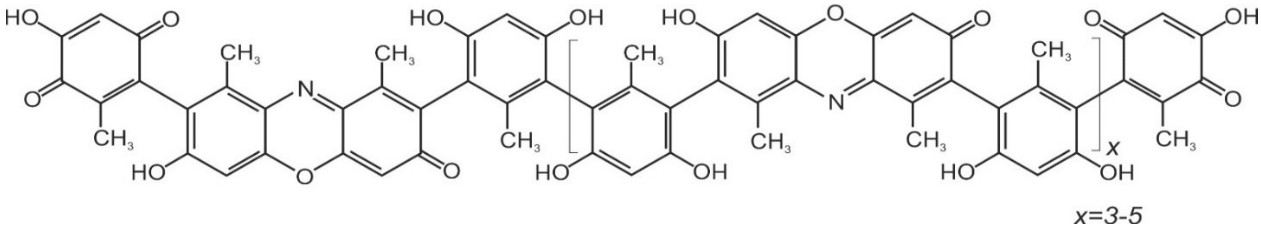


Fig. 12 Chemical structure of Litmus.

The infrared spectra of two different Litmus products gained from two different suppliers are compared in Figure 13. Both Litmus products are supplied as powders. The product P8 is dedicated for the use as pH-indicator compound. In contrast, the purpose of product P9 is related to coloration and painting. The measured two IR spectra are clearly different from each other, so an identification of Litmus by using infrared spectroscopy is difficult. However, the IR spectrum of product P8 is quite similar to an IR spectrum from Litmus reported in literature [104]. In this literature reference the four signals 3275 cm^{-1}, 1730 cm^{-1}, 1634 cm^{-1} and 1454 cm^{-1} are assigned to vibrations of following bonds, O-H, C=O, C=C and C=N and C-C [104]. Further the two signals at 2922 cm^{-1} and 2853 cm^{-1} are related to C-H stretching vibrations from aliphatic units, mostly likely the -CH_3 groups [33]. For product P9, the strong signal at 1620 cm^{-1} can be related to C=O stretching vibration from amide group or from C=C vibration of conjugated aromatic systems [33].

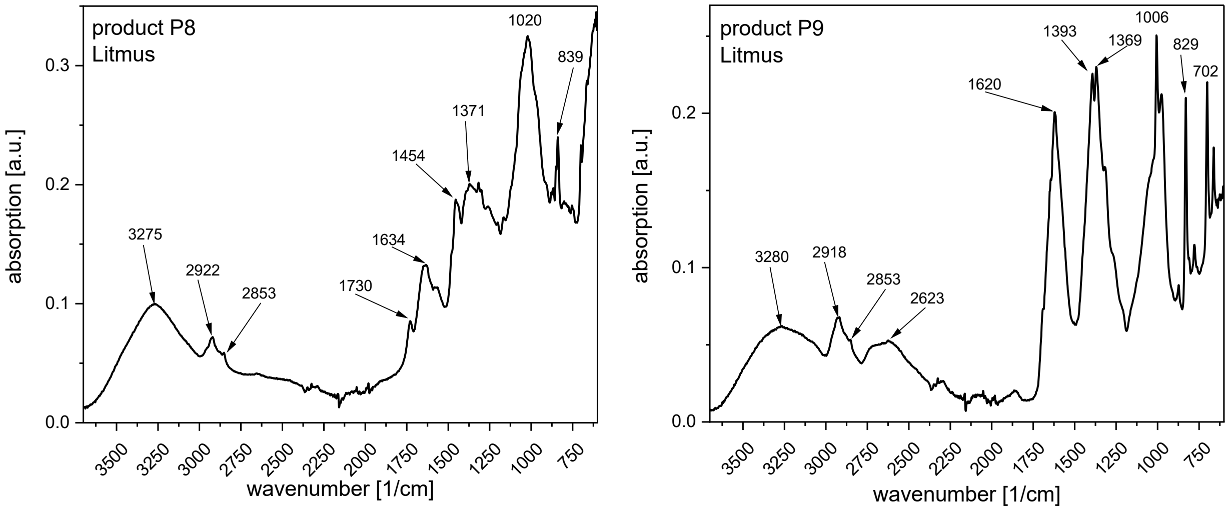


Fig. 13 Comparison of IR spectra of two Litmus products. Shown are the products P8 and P9 (compare Table 1).

3.4 Gallic acid

Gallic acid is part of natural dyeing materials and can act as natural dye to gain brown and black colorations [70,108]. However, in combination with other dyes gallic acid can be applied as kind of mordanting agent [109]. In fact, it is simple trihydroxy benzoic acid, $C_7H_6O_5$. The chemical structure of gallic acid is presented in Figure 14. The molecular structure is planar and between the hydroxy group intramolecular hydrogen bonds are formed [110, 111].

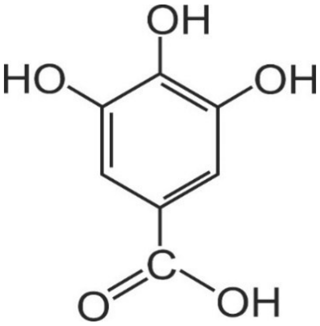


Fig. 14 Chemical structure of Gallic acid (Trihydroxy benzoic acid).

Gallic acid as natural component can be gained from gallnut. Photographs of gallnuts and a magnified photograph of a cross section of a gallnut are shown in Figure 15. The average diameter of a gallnut is around 12 mm. The origin of gallnut is the infection of the plant *Quercus infectoria* L. by eggs of an insect (*Cynips spp.*) [70,111].

Fig. 15 Photograph of gallnuts (product P13) (left) and magnified photograph of a cut gallnut (right).

An infrared spectrum of the synthetic chemical gallic acid is shown in Figure 16. The clear peak at 3491 cm^{-1} is assigned to the O-H stretching vibration of the carboxylic acid [33]. The signal at 3265 cm^{-1} can be assigned O-H stretching vibration of the hydroxy groups [33, 110]. Clear signals related to C-H stretching vibration cannot be identified, probably because this molecule does not contain aliphatic units as $-CH_3$ or $-CH_2-$. The broad absorption signal in the area from 2500 cm^{-1} to 3200 cm^{-1} without clear peaks is also reported in literature for the IR spectrum of a gallic acid standard [112]. The peak at 1663 cm^{-1} is related to C=O stretching vibrations from carboxylic acid. While the peaks at 1605 cm^{-1} and 1423 cm^{-1} are assigned to C-C stretching vibrations from the aromatic ring system. The signal at 1308 cm^{-1} is related to the C-O stretching vibrations from the hydroxy groups attached to the aromatic ring [110]. Remarkable is the assignment for the signal at 731 cm^{-1} made by Masoud et al., which dedicate this signal to the hydrogen bridge bond of the OH unit in the carboxylic acid [110].

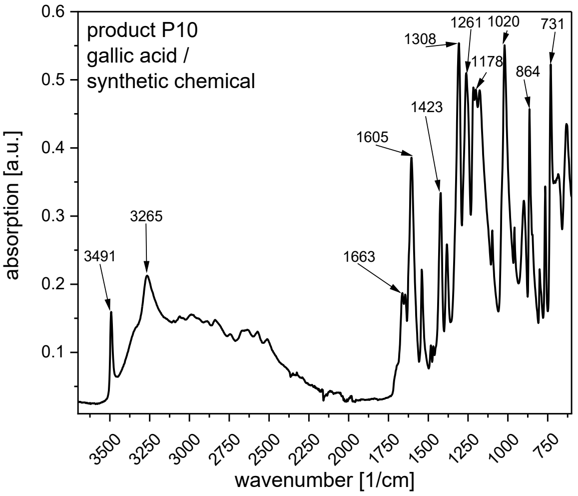


Fig. 16 Infrared spectrum of gallic acid – synthetic chemical; product P10.

Infrared spectra of two different types of gallic apple products from two different suppliers are compared in Figure 17. These products are supplied as powder, probably got by grinding of the complete gallnut. For this, these products contain only a certain amount of gallic acid and also a broad range of different compounds as fatty acids, proteins and carbohydrates. Because of the different composition, it is obvious that the IR spectra of those products differ significantly from the IR spectrum of pure synthetic gallic acid (compare Figures 16 and 17). The IR spectra of two other products from gallnut are shown in Figure 18 and 19. One infrared spectrum is taken from a complete gallnut without further treatment. The other IR spectrum is recorded from an Indian product commercialized for dyeing purposes. All IR spectra (Figures 17 to 19) are mainly indicated with eight typical signals in the fingerprint area – at around 1700 cm^{-1}, 1607 cm^{-1}, 1445 cm^{-1}, 1313 cm^{-1}, 1180 cm^{-1}, 1020 cm^{-1}, 870 cm^{-1} and 755 cm^{-1}. By these signals, the identification of gallnut products is clearly possible. Compared to pure gallic acid, especially the clear peak at around 1700 cm^{-1} is prominent for the gallnut products. This spectral signal might be caused by esters of fatty acids and their related C=O stretching vibration. The signals at around 1609 cm^{-1} and 1020 cm^{-1} occur for the synthetic gallic acid as well as for the investigated gall nut products. The signal at 1609 cm^{-1} is probably caused by C=O stretching vibration from the gallic acid itself or by amide groups of adjacent proteins.

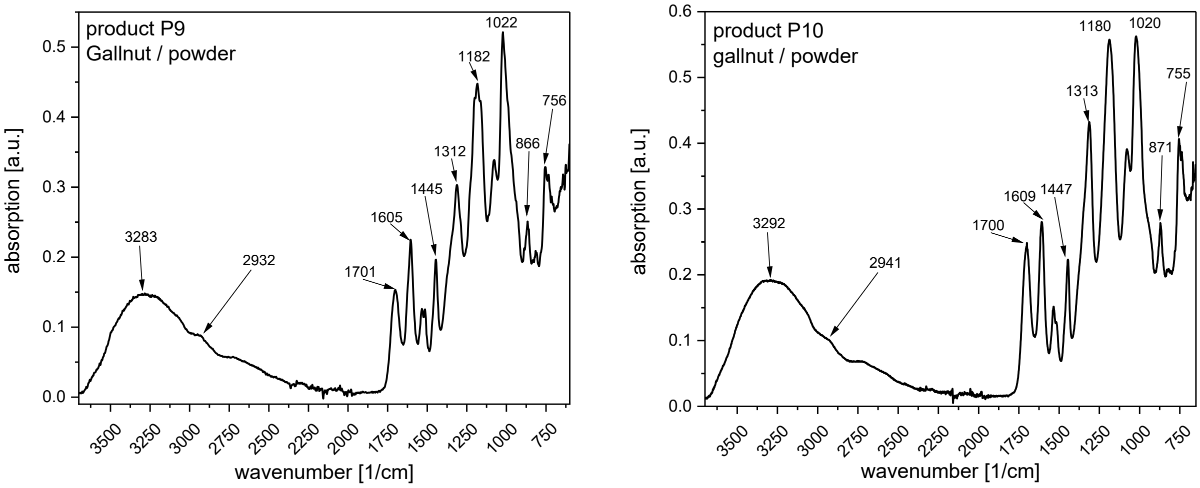


Fig. 17 Comparison of IR spectra of several powders from gallnut. Shown are the products P11 and P12 (compare Table 1).

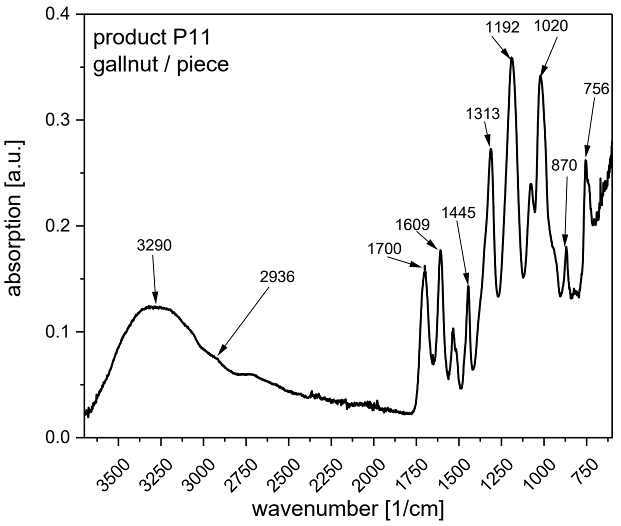


Fig. 18 Infrared spectrum recorded from a piece of a gallnut.

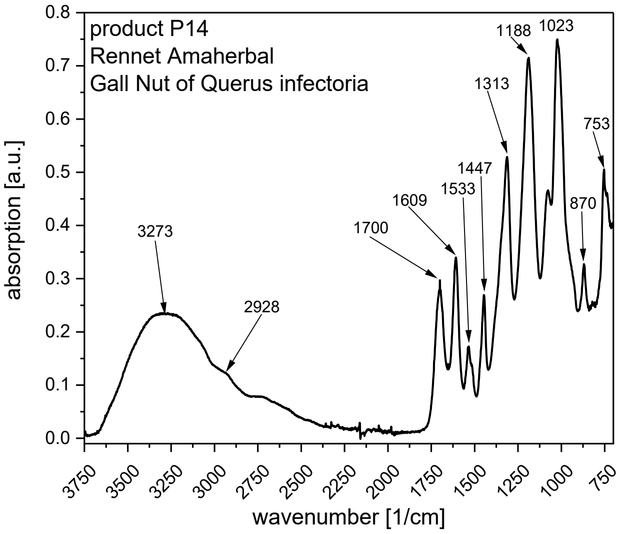


Fig. 19 Infrared spectrum recorded from commercial dye product based on gallnut.

3.5 Kamala

Kamala is a medium-sized tree widespread in South Asia, Philippines and Australia [89]. It is also named as Monkey Face Tree or *Mallotus phillippinensis* [113,114]. The fruit of the tree contain around 11% of Kamala dye (C.I. Natural Orange 2). Also, the extracts of the flowers of the tree are used as dye (C.I. Natural Yellow 25) [1]. Main color components in Kamala are different derivatives of Rotterlin, as Rotterlin, 4-hydyroxy rottlerin and 3,4-dihydroxy rotterlin [115]. The chemical structures of Rotterlin and the two hydroxy derivatives are presented in Figure 20. By combination of Kamala with mordant containing iron and tin a broad range of different color shades can be realized on wool fabrics showing also good light fastness [115].

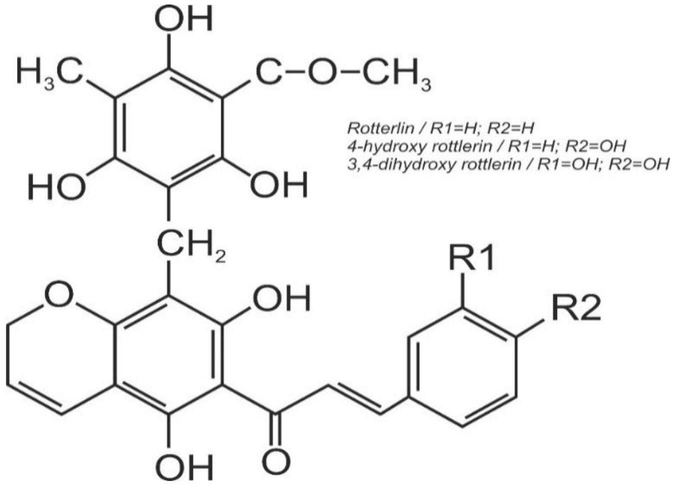


Fig. 20 Chemical structure of some dye components of Kamala. Presented is the basic structure of Rotterlin with variations of its 4-hydroxy and 3,4-dihydroxy derivative.

The infrared spectrum recorded from a Kamala product (product P15) is presented in Figure 21. The product is offered for dyeing purposes and it is supposed that this powder is gained by grinding of plant pieces and no extraction process was done for its production. For this, the color components are accompanied by various other components building up the plant. The broad signal at 3274 cm^{-1} is related to O-H stretching vibration, while the two peaks at 2920 cm^{-1} and 2851 cm^{-1} are assigned to C-H stretching vibrations at aliphatic units. These signals can be related to Rotterlin but also to carbohydrates from the plant. The signal at 1729 cm^{-1} is not related to the structure of Rotterlin but can be assigned to C=O stretching vibration of an ester unit – probably present in some by-products. The strong signal at 1604 cm^{-1} can be assigned to C=O stretching vibration from a keto group, as it is present also in Rotterlin. The strongest signal at 1101 cm^{-1} can be assigned to C-O stretching vibrations [33]. The determined infrared spectrum for the Kamala product P15 is comparable to an IR spectrum reported for Kamala in literature [116]. However, while the peak positions are comparable to the earlier reported IR spectrum, the signal intensity is different – especially for the signals between around 1000 cm^{-1} to 1100 cm^{-1} [116].

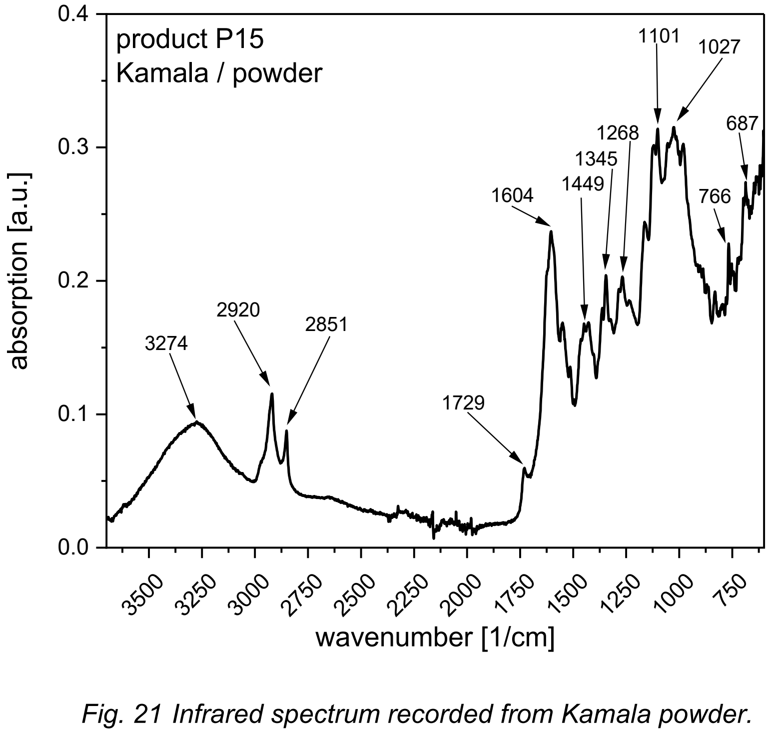


Fig. 21 Infrared spectrum recorded from Kamala powder.

3.6 Reseda

The main color component of Reseda plant is the dye Luteolin (3,4,5,7-Tetrahydroxyflavon) (C.I. 75590; Natural Yellow 2) [1]. It is gained by extraction of leaves, stems and flowers of Reseda [117]. The chemical structure of Luteolin is presented in Figure 22. It belongs to the category of flavonoids [118]. In many traditional European dye recipes weld (*Reseda luteol* L.) is used [119]. With alum as mordant strong yellow colorations can be realized with Reseda on textile fabrics [70,120]. In combination with Indigo, Reseda is used to produce a green pigment distributed under the name “Indigo Green” [30]. By using other types of mordants as copper sulphate or iron sulphate different coloration from olive green to dark brown can be reached by application of Reseda [117]. By application of Reseda with eight different metal mordants (Al, Fe, Cu, Sn, Co, Ni, Cr and Zn) in different combinations a broad range of different coloration on wool fabrics can be realized (yellow, orange, olive, brown, dark brown) [121].

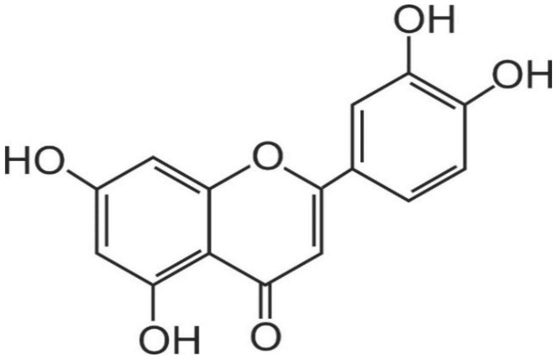


Fig. 22 Chemical structure of Luteolin as one main color component in Reseda.

Surface topographies of dried Reseda plants are presented with different magnifications in Figure 23. In lower magnification a linear ordered structure can be observed. With high magnification also pores on the surface can be detected.

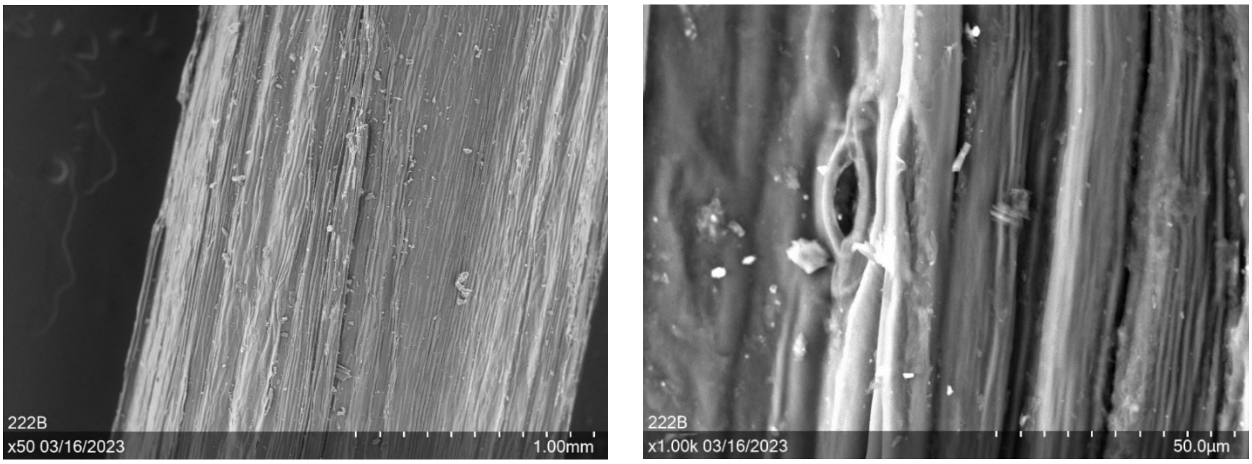


Fig. 23 The surface of a Reseda piece. SEM images recorded with low magnification (left) and higher magnification (right).

The infrared spectrum recorded from a color extract from Reseda (product P16) is shown in Figure 24. This IR spectrum is different from IR spectra of Luteolin reported in different references with different image quality [122,123]. Also, there are clear differences to a reported IR spectrum recorded from *Reseda luteola* L. [124]. However, as starting point the IR spectrum from product P16 can be discussed in related to the chemical structure of Luteolin (Figure 22), keeping in mind that the Reseda extract should contain additionally to Luteolin also other natural products. The broad signal at 3269 cm^{-1} can be assigned to O-H stretching vibration of the hydroxy group. The weak signal at 2930 cm^{-1} is related to C-H stretching vibration. The medium peak at 1593 cm^{-1} is assigned to C=O stretching vibration of the keto group of Luteolin. Usually the peak for C=O stretching vibration of keto groups would be expected for a spectral area > 1600 cm^{-1}. However, in Luteolin the keto group is probably weakened by the attachment to the aromatic chromophore and by a hydrogen bridge to the neighboring hydroxy group. However, this peak can be also assigned to the C=C stretching vibration from the aromatic ring system.

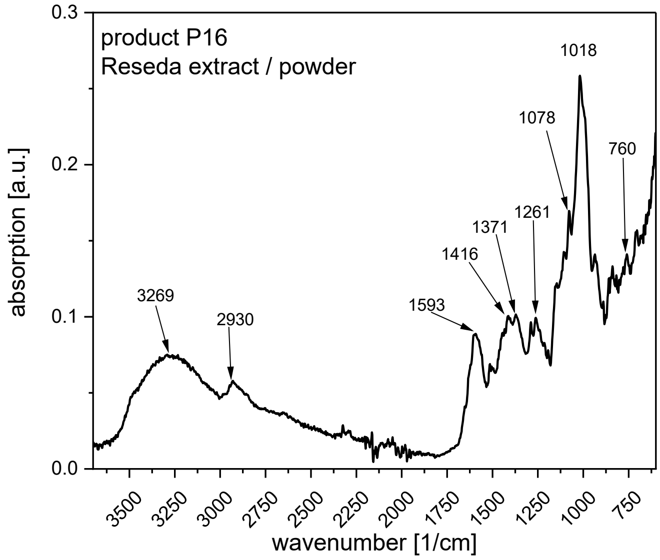
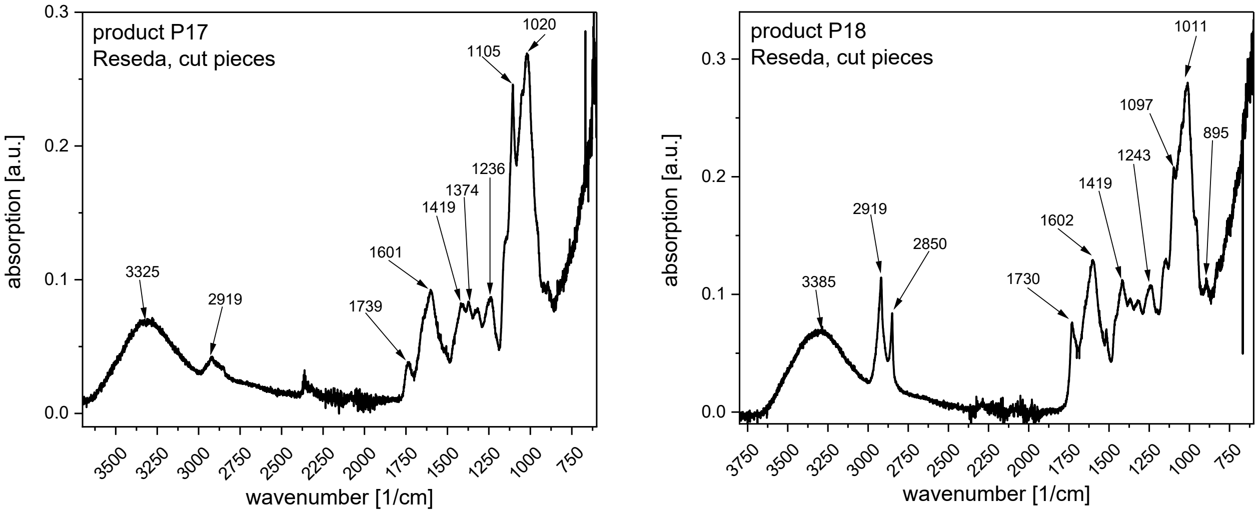


Fig. 24 Infrared spectrum recorded from Reseda extract / powder.

Infrared spectra of two different dried Reseda products are shown in Figure 25. One main difference of those spectra compared to the spectrum from Reseda extract is the appearance of the weak to medium peak around 1735 cm^{-1}. This signal is probably related to a C=O stretching vibration in an ester compound. Due to the fact that Luteolin does not contain an ester group, this signal is probably caused by other components in the plant product, as e.g. ester compounds from fatty acids. The infrared spectra of the two different Reseda products have some similar features but also differing in intensity and shape of several signals, probably because of different content of by-products. For this, it might be difficult to identify clearly Reseda products by IR-spectroscopy.



(a) (b)

Fig. 25 Infrared spectra recorded from different Reseda products – cut pieces; (a) product P17 supplied by Kremer Pigmente; (b) product P18 supplied by Pflanzenfärbershop.

3.7 Catechu

Catechu is a palm plant (Betel palm) (*Areca catechu* L.) originated in the tropical and subtropical areas of southeast Asia [89, 125]. The dye component is gained from the nuts of the palm [89]. Catechu is a brown colorant (C.I. Natural Brown 3). However, in literature different plant sources and procedures are reported to produce Catechu dye [1]. One main colored component of Catechu is Epicatechin, which chemical structure is presented in Figure 26 [126]. The structure of Epicatechin is nearly similar to Luteolin, which is the main color component in Reseda. The main difference is the absence of the keto group in epicatechin. Additional to the application as dye, Catechu can be also used as bio-mordant to improve color and fastness properties of other natural dyes [127]. The application of Catechu on silk in combination with different mordanting agents (alum, copper sulphate, ferrous sulphate and tin chloride) is reported to lead to light brown, red brown and dark brown colorations [128]. The processing of powdered Catechu for dyeing purposes is described in several patent. Here, especially different mordanting procedures and refining processes for the powder are focused [129, 130]. Catechu dye extracts are also used to implement antimicrobial properties on different textile fabrics [131-134]. Also, the implementation of UV protective properties is reported [134].

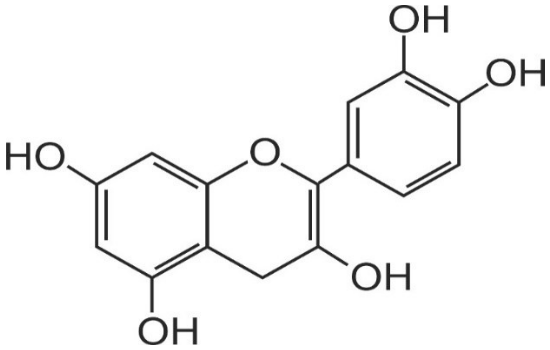


Fig. 26 Chemical structure of epicatechin as main colored component in catechu.

Infrared spectra of two different Catechu products from two different suppliers are presented in Figures 27 and 28. The IR spectra of both products are mainly similar to each other. In literature a medium quality IR spectrum recorded from the seed of Acacia catechu is reported, which exhibit certain similarity to the both actually recorded IR spectra of both Catechu products [135].

The broad signal at around 3250 cm^{-1} is related to O-H stretching vibrations. The small signal around 2940 cm^{-1} is assigned to C-H stretching vibration of aliphatic units [33]. The main signals in the fingerprint area of both spectra are nearly similar, so by use of IR spectroscopy Catechu components can be identified. For both Catechu products a strong signal at 1605 cm^{-1} is recorded, which is probably related to C=O stretching vibrations. However, the color component Epictechin does not contain any kind of C=O bond, so this signal is probably caused by additional components, as e.g. proteins. Additionally, the IR spectrum of product P20 exhibits a weak signal shoulder signal at 1748 cm^{-1}, which can be related to a C=O stretching vibration of an ester group. Xiao et al. presents a summary of compounds found in Catechu fruits, which covering a broad range of different fatty acids and amino acids from proteins [125]. The larges signal around 1034 cm^{-1} can be assigned to C-O stretching vibrations.

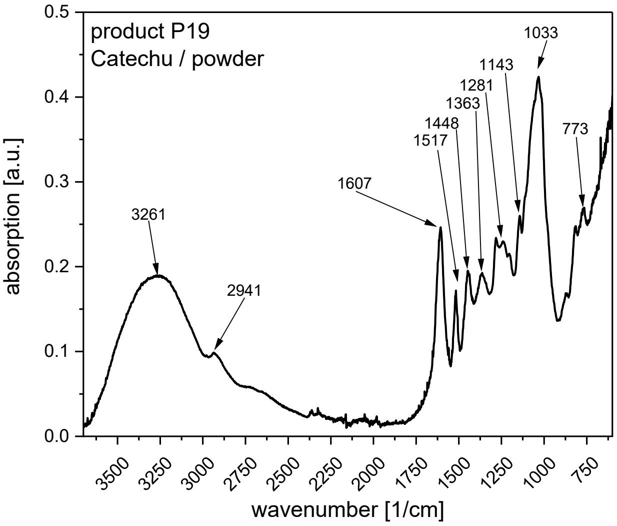


Fig. 27 Infrared spectrum recorded from Catechu powder.

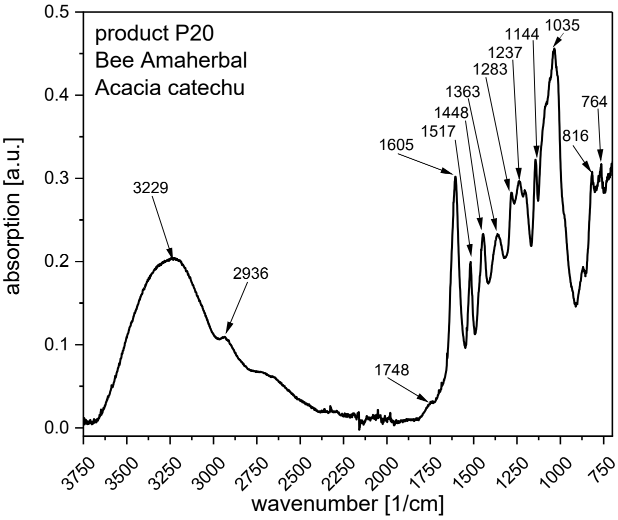


Fig. 28 Infrared spectrum recorded from commercial dye product based on Acacia catechu.

3.8 Saffron

Saffron is gained from the flowers of the plant *Crocus sativus* L. [1,70]. Saffron dye is listed as C.I. Natural Yellow 6 and traditionally applied as direct dye on wool [1]. Saffron can be used to implement yellow coloration either on natural fibers (as wool or cotton) and on synthetic fibers (as nylon or polyester) [136,137]. The main colored component of Saffron is Crocin [70,138]. Its chemical structure is presented in Figure 29. In contrast to many other dye molecules, Crocin does not contain aromatic units. For that, it is in a certain way similar to the chemical structures of carotene or bixin. The chromophore of Crocin is built up by seven C=C double bonds and two attached ester groups. Further, four glucose units are attached to the chromophore.

The application of saffron dye onto cotton is reported in combination with bio-based mordant (tannic acid) and different metal based mordants (copper, aluminum, iron and tin). In an extensive study, these mordants are applied in pre-, meta- and post-mordanting processes, leading to different color intensities and fastness properties. A reported ultrasonic procedure is especially advantageous to improve the light fastness in the pre-mordanting procedures [139]. For dyeing application of Saffron on synthetic fibers as polyester and polyamide, it is reported that by pretreatment of the synthetic fibers using UV light and ozone, the dyeability can be significantly improved [77]. The application of saffron dye to textiles is reported to lead to antimicrobial activity on textile fabrics, especially with combination of chitosan [140, 141]. However, the disadvantage of such studies on antimicrobial properties of natural substances is that often the presented reference measurements are limited, so it is difficult to judge the clear antimicrobial effect of the investigated natural substance. In fact, chitosan is probably one of the most overestimated bio-based products for textile functionalization [142].

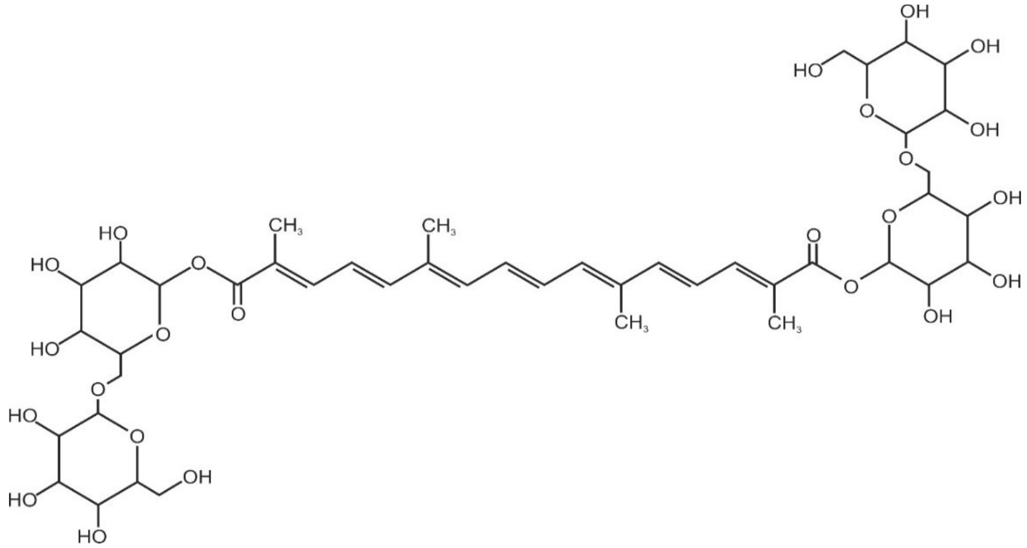


Fig. 29 Chemical structure of crocin as main colored component in saffron.

A magnified photograph to two saffron fiber is shown in Figure 30. Their deep red coloration is clearly visible. Further the surface of saffron fiber is presented in higher magnification recorded by scanning electron microscopy (Figure 31). The plant cell structure is clearly visible in the microscopic image of higher magnification.

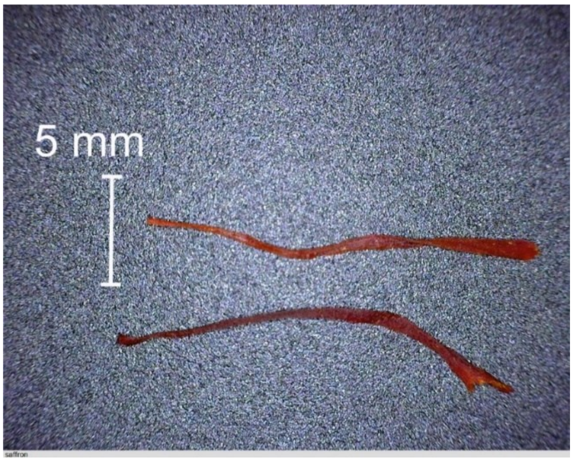


Fig. 30 Saffron fibers. Photographic image with higher magnification.

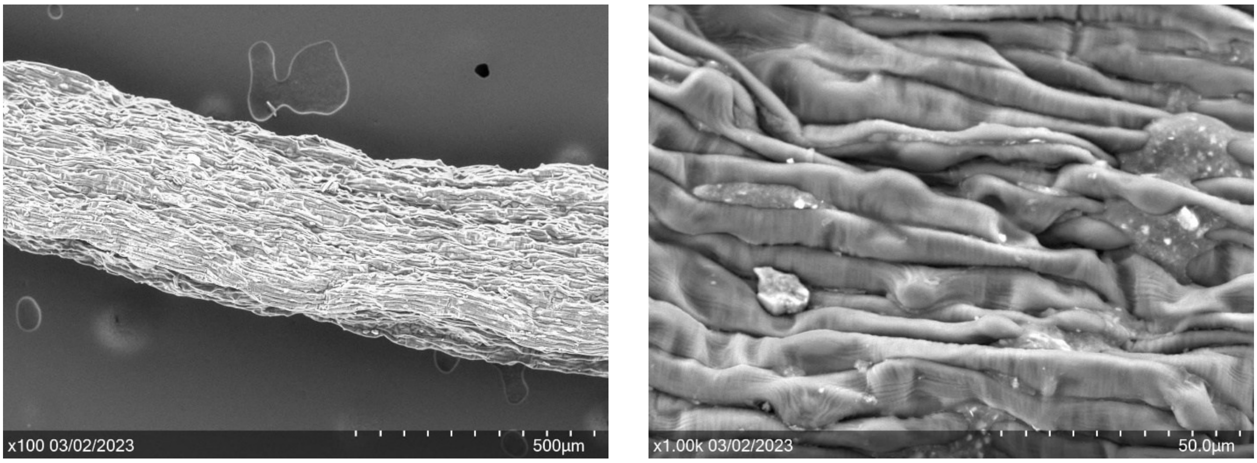
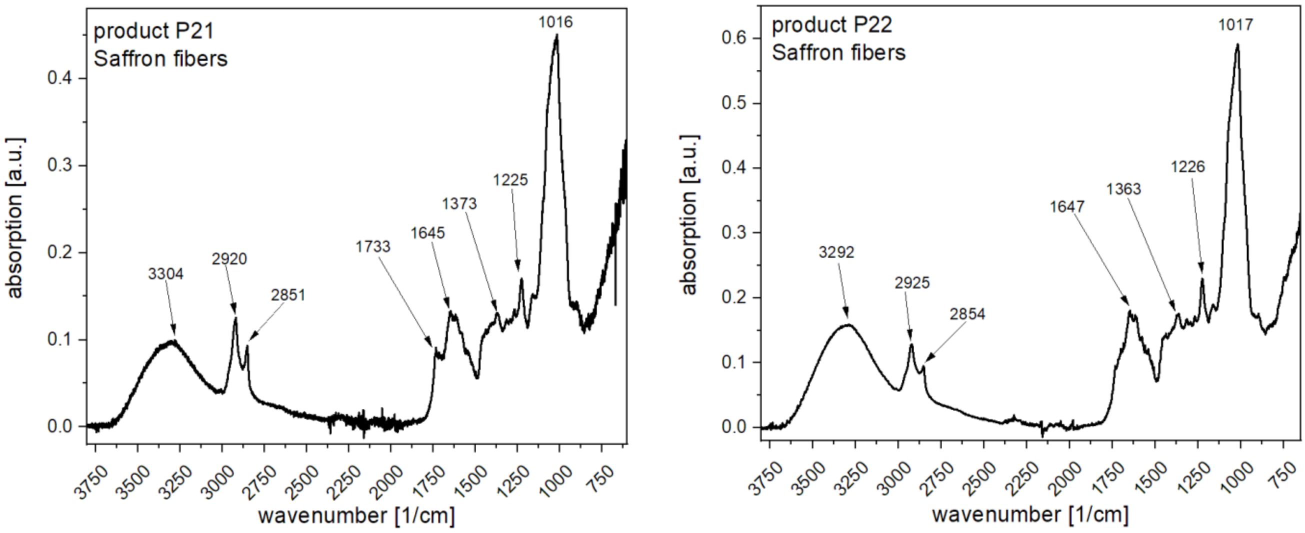


Fig. 31 Saffron fiber. SEM images recorded with low magnification (left) and higher magnification (right).

Two infrared spectra recorded from different saffron fiber samples are presented in Figure 32. Even if the samples of saffron are from different suppliers and probably also from different production countries, mainly all features of both spectra are similar. However, these IR spectra are significantly different from spectra reported for dye extracts from Saffron in literature [136,137]. Of course, the saffron fiber contains more components than only Crocin, so the structural discussion of the infrared spectrum related to the chemical structure is limited. Nevertheless, few statements can be made. The broad signal at around 3300 cm^{-1} is related to O-H stretching vibration from hydroxy groups. The both signals at around 2920 cm^{-1} and 2850 cm^{-1} are assigned to C-H stretching from aliphatic $-CH_2-$ and $-CH_3$ units. The IR spectrum of product P21 exhibits a weak signal at 1733 cm^{-1}, which is for product P22 only visible as weak shoulder. This signal can be assigned to C=O stretching vibration of ester groups as they are also part of Crocin. The signal at around 1645 cm^{-1} can be assigned to C=O stretching vibration from an amide group as part of an eventually protein content in the saffron fiber.



(a) (b)

Fig. 32 Infrared spectra recorded from two different Saffron fibers from different suppliers – (a) product P21 received from a German supermarket; (b) product P22 supplied by Kremer Pigmente.

3.9 Safflower

The blossoms of the plant safflower (*Carthamus tinctorius* L.) are used for dyeing in red, orange and pink coloration [70]. The use of Safflower plant for oil production for food or as bio-fuel is reported [143]. One main colored component of Safflower is Carthamin which is also named as safflor red or carthamic acid (CI Natural Red 26). However, there are different components present, due to processes of oxidation, hydrolysis and isomerization [1]. The dye can be put to the class of benzoquinones [144]. The content of

Carthamin in the blossoms is with values of 0.1% or less low [70]. The chemical structure of Carthamin is presented in Figure 33. Attached to the huge chromophore are two glucose units. Safflower can be applied as direct dye. The dyeing of silk with Safflower can be used to implement different yellow color shades [145,146]. An interesting approach is the printing on natural and synthetic fiber materials by using safflower dye nanoparticles [144]. A Chinese patent described the application of safflower red from mixtures of organic solvents for dyeing of polyester [147]. However, the use of organic solvents for dyeing purposes is for economic and safety reasons not applicable for industrial textile dyeing.

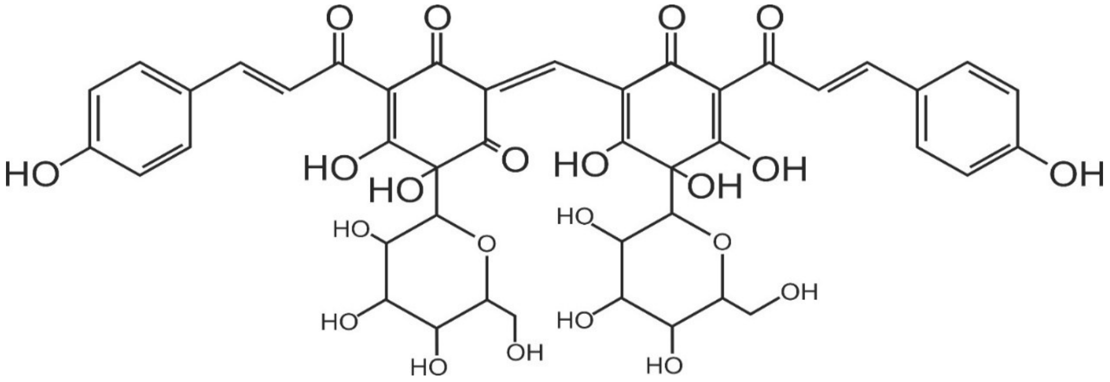


Fig. 33 Chemical structure of carthamin as main colored component in safflower.

A photograph of safflower pieces in high magnification is shown in Figure 34. The strong orange coloration is clearly visible. Compared to saffron fibers, the safflower pieces exhibit larger diameters.

Fig.34 Safflower. Photographic image with higher magnification.

The surface of safflower pieces is recorded in higher magnification with scanning electron microscopy and shown in Figure 35. Additional to the main body of the fiber pieces also structured pieces of pollen are detected. The plant cell structure of safflower is clearly visible in the SEM image recorded in higher magnification.

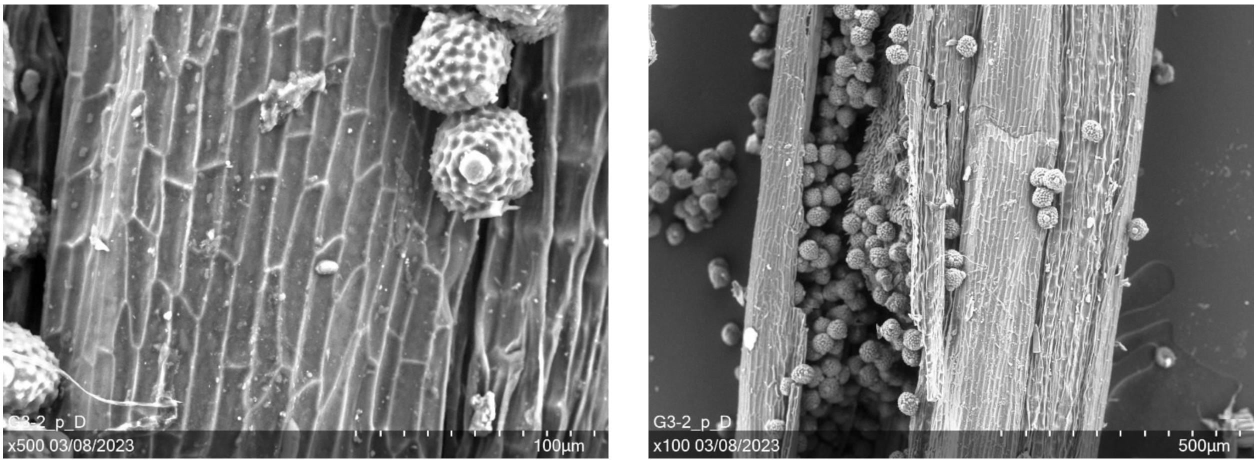


Fig. 35 Safflower. SEM images recorded with low magnification (left) and higher magnification (right).

An infrared spectrum recorded from the safflower pieces is presented in Figure 36. This recorded IR spectrum has a similarity to an IR spectrum from safflower dye reported in literature [144]. However, this reported IR spectrum is only of medium quality and it is not clear if the reported IR spectrum is measured on the complete plant pieces or taken from dye extracts. It is remarkable that the actually recorded IR spectra of saffron fiber and safflower are of good similarity (please compare the figures 32 and 36), even if the chemical structure of the main color component is totally different. One possible reason for this similarity may be the composition of the same type biopolymers, because both products are gained from the same part of the plant. In any case it is not possible by use of IR spectroscopy to distinguish the different color plants saffron and safflower from each other.

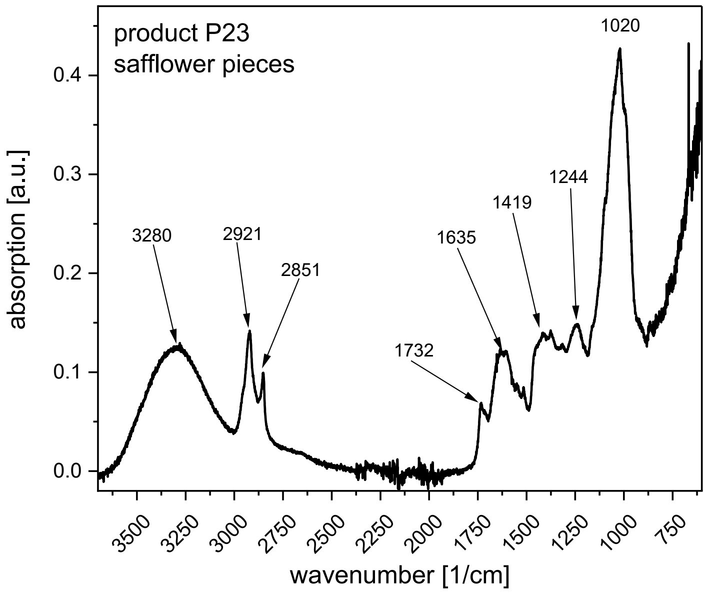


Fig. 36 Infrared spectrum recorded from Safflower.

3.10 Ratanhia root

Ratanhia root is gained from the plant *Krameria triandra*. This plant is originated in South America in high altitudes between 1000 and 2500 meter above sea level [1]. The information to the colored components of Ratanhia and related chemical structures are less reported in literature. The Ratanhia root contains around 8 to 18 % of tannic acid, which is transferred by oxidation into red colored components based on phlobaphene [1]. However, additional to colored components the presence of organic UV absorbers is reported for extracts from Ratanhia root. [148]. One determined UV absorber for this natural product is octylmethoxycinnamate (compare chemical structure in Figure 37). This UV absorber absorbs especially UV light in the spectral range from 340 to 380 nm. A broad range of different synthetic UV absorbers based on cinnamate structure is reported in literature and patents [149-152].

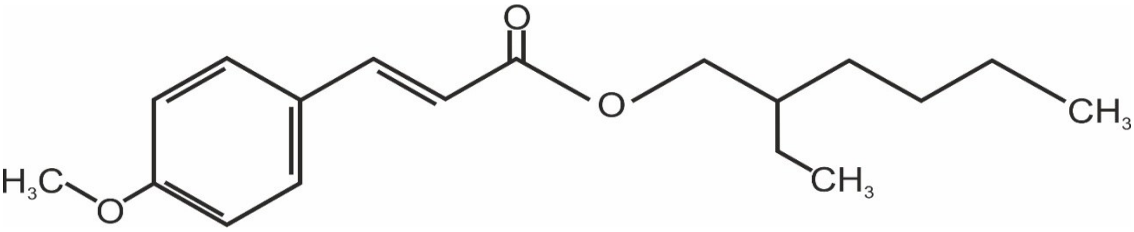


Fig. 37 Chemical structure of Octyl methoxycinnamate as UV absorber occurring in Ratanhia root.

A microscopic image taken from the surface of the Ratanhia root sample is shown in Figure 38. A hairy structure and deeply rough surface can be observed.

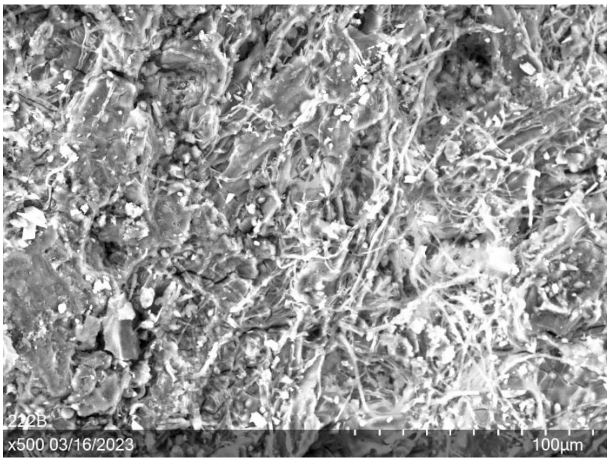


Fig. 38 SEM image of the surface of a Ratanhia root sample.

An infrared spectrum recorded from a piece of Ratanhia root is shown in Figure 39. This IR spectrum exhibits good similarity to the IR spectra of several wooden materials, which are reported recently [31]. Probably the Ratanhia root is mainly built up by the same biopolymers as other wooden samples, so a clear identification of Ratanhia root by IR spectroscopy is not possible. It is also not possible to distinguish it for other types of colored wood, which exhibit also quite similar IR-spectra [31].

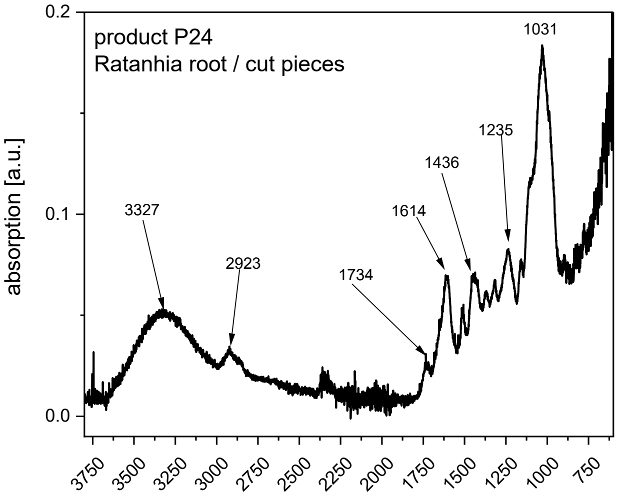


Fig. 39 Infrared spectrum recorded from Ratanhia root pieces.

3.11 Beetroot

Red Beetroot is a worldwide consumed food plant (*Beta vulgaris* L.) [153,154]. As food colorant Beetroot dye is recorded with the number E162 [71]. The main color component of beetroot is the dye Betanin also named as Beetroot Red (compare chemical structure in Figure 40) [1,154]. This dye exhibits three functional groups of carboxylic acid and several hydroxy groups. For this, it is well soluble in water. Solved in water, this dye molecule exhibits a negative net charge, due to deprotonation of the acid groups. It can be seen as anionic dye which has probably good affinity to protein based natural fibers like silk or wool. Further, a glucose unit is attached to the chromophore of the dye molecule.

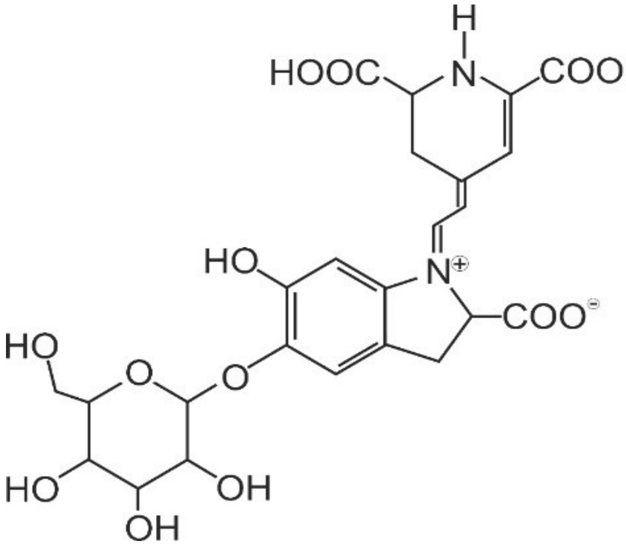


Fig. 40 Chemical structure of Betanin as main color component in Red Beetroot.

Additional to the use as food colorant, Beetroot dye is also investigated for its application on natural textile materials, as e.g. wool, silk or cotton [155,156]. The combination of Beetroot dye with various types of metal based mordant (e.g. alum and iron sulphate) and bio-based acidic mordants is broadly investigated [157,158]. The use of beetroot dye for the dyeing of leather is reported [159].

The IR spectrum of dried beetroot powder (product P25) is presented in Figure 41. For beetroot dye, different IR spectra are reported in literature with medium quality, which are not similar to each other and significantly different to the measured IR spectrum of product P25 [155,157]. In contrast, the IR spectrum of product P25 is quite similar to the IR spectrum of Ratanhia root (compare Fig. 39) and of IR spectra recorded earlier from different types of wood [31]. In fact, the main component in dried Beetroot are carbohydrates, so in fact carbohydrates dominate the IR spectrum and not the color component with much lower content [154].

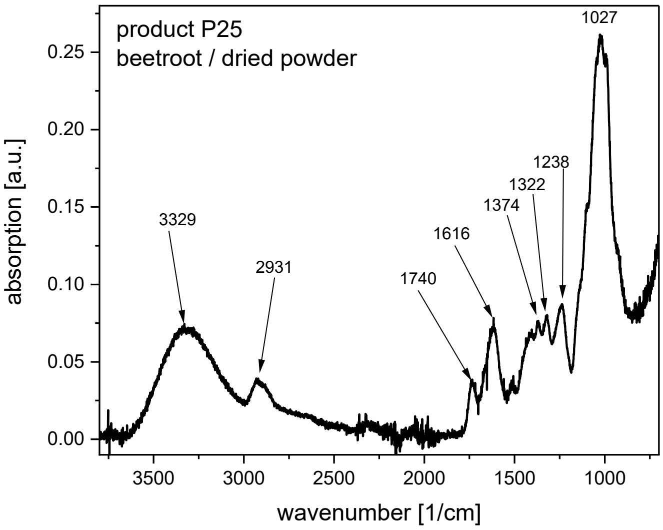


Fig. 41 Infrared spectrum recorded from Beetroot powder.

3.12 Mulberry

The Mulberry tree is a bush or small tree of around ten meters height [1]. It can be distinguished between white Mulberry (*Morus alba* L.), red Mulberry (*Morus rubra* L.) and black Mulberry (*Morus nigra* L.) [160]. From Mulberry tree different types of plant pieces can be used for dyeing purposes, as the leaves, the wood and the bark [1,161]. Also reported is to use the berries of black and red Mulberry for dyeing purposes leading to violet, red and dark coloration [89]. The main color component in the leaves of Mulberry is Rutin (C.I. Natural Yellow 10; C.I. 75730) with a quite high concentration in the range of 2% to 6% [1]. Compare the chemical structure of Rutin in Figure 42. It is Quercetin-3-O-rutinosid and contains two glucose units. Beside the glucose units, Rutin is similar to Luteolin the main color component of Reseda.

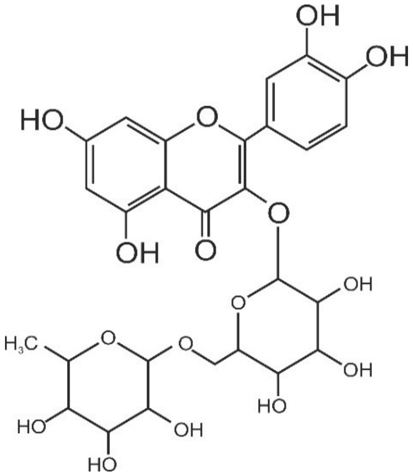


Fig. 42 Chemical structure of Rutin the main color component in the leaves of Mulberry tree.

The infrared spectrum of a dye product form Mulberry plant (product P26) is presented in Figure 43. This dye product is gained as extract from Mulberry leaves and dedicated for color application in green. The infrared spectrum of Mulberry product P26 exhibits a broad signal at 3333 cm^{-1} related to O-H stretching vibration. A clear three peak signal (2957 cm^{-1}, 2923 cm^{-1} and 2854 cm^{-1}) is determined related to C-H stretching vibrations. This signal can be assigned to aliphatic units -CH$_2$- and -CH$_3$. At 1631 cm^{-1} only a small shoulder appears related to C=O stretching vibration from amide or keto group.

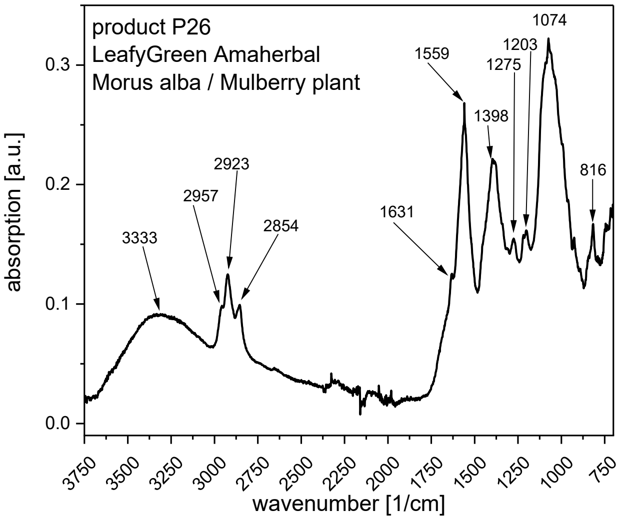


Fig. 43 Infrared spectrum recorded from commercial dye product based on Morus Alba.

3.13 Pomegranate

Pomegranate (*Punica granatum* L.) is a Mediterranean tree of small height till around five meters [89]. The skin of the fruits of Pomegranate contains several natural colorants as gallic acid or Flavogallol [1]. By extraction of dye from the skin of the fruits, the waste accelerated from food production is turn into valuable dye products [162]. The chemical structure of Flavogallol is presented in Figure 44. The chromophore of Flavogallol is built up by a large aromatic system containing six hexa-rings. It contains three cyclic esters, which are part of the chromophore. Attached are further four hydroxy groups. Aqueous extracts from Pomegranate are reported for dyeing of natural fiber cotton and regenerated cellulosic fibers (Tencel) [163,164].

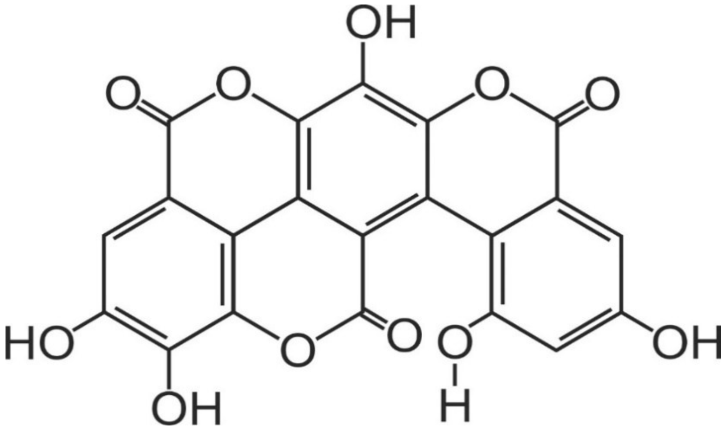
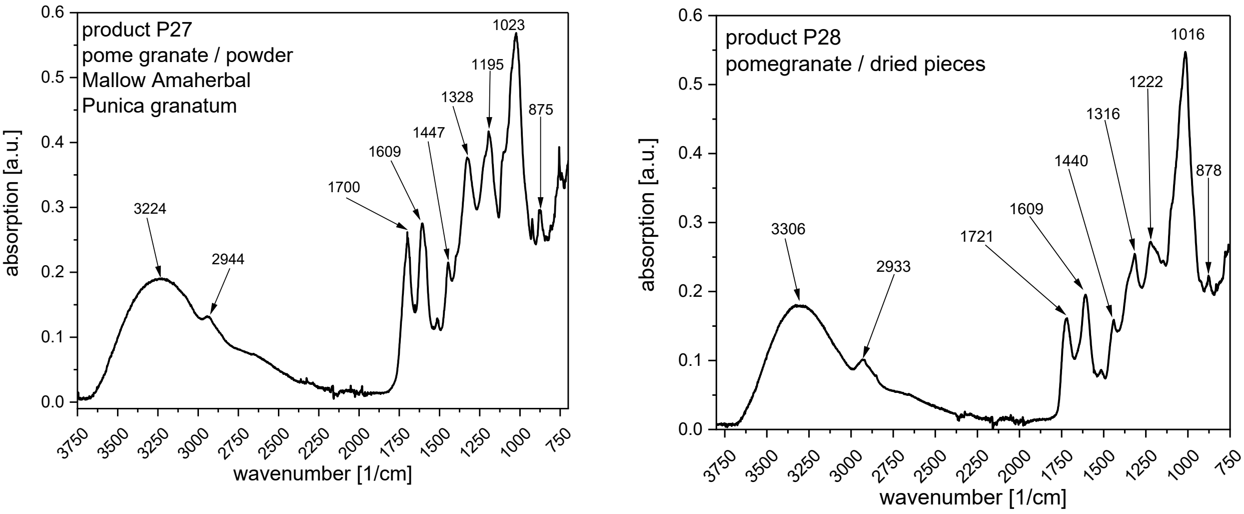


Fig. 44 Chemical structure of Flavogallol as one main colored component in Pomegranate.

Infrared spectrum of two dye products from Pomegranate are presented in Figure 45. Product P27 is an extract from Pomegranate pieces received as residues from food production and is offered for textile dyeing applications. In comparison, product P28 is from dried plant piece, also offered for dyeing purposes. The features of these IR spectra can be discussed in respect to the chemical structure of Flavogallol. However, main features of the infrared spectra are also similar the spectra recorded from gall nut products (compare Figure 18), because gallic acid is also one of the main color components in Pomegranate. Starting the discussion of IR-spectra with the extract product P27. The broad signal at 3224 cm^{-1} can be assigned to O-H stretching vibration of hydroxy groups. The weak signal at 2944 cm^{-1} is related to C-H stretching vibrations. Both peaks at 1700 cm^{-1} and 1609 cm^{-1} can be assigned to C=O stretching vibration, probably related to the cycled ester groups in the dye molecule. One cycled ester group is neighbored in the structure to a hydroxy group, so an intramolecular hydrogen bridge formed here might weaken the strength of the C=O bond. By this, the related signal in the IR-spectrum is shifted to lower wavenumbers. The IR spectrum of product P28 exhibits a nearly similar shape but the positions of the signals are slightly shifted. This shift might be explained with by-products present in the dried plant pieces, which are obviously removed during extraction process used to prepare product P27.



(a) (b)

Fig. 45 Infrared spectrum recorded from commercial dye products based on Pomegranate - (a) product P27 supplied by Amaherbal and (b) product P28 supplied by Pflanzenfärbershop.

4 Conclusions

Altogether 28 different plant based products offered for dyeing purposes are discussed and presented together with their infrared spectra, recorded exclusively for this overview. For all IR spectra a structural discussion was performed in respect to chemical structure of main colored components and content purity of the dye products. However, for natural products which are often also accompanied by different by-products the structural related discussion is limited. Often main IR signals can be only explained by presence of by-products. Nevertheless, the documentation of IR spectra of dye products from different suppliers given in different purity can be a helpful tool for product identification. Finally, this overview paper can be useful for people working in quality control or dyeing sector.

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All product and company names mentioned in this article may be trademarks of their respected owners, even without labeling. There is no conflict of interest and no funding for the presented research.

Conflicts of Interest

The author declares no conflict of interest.

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