

# Natural dyes and their IR-spectra – supplementary data

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## ABSTRACT

*Natural dyes are an essential keystone for realization of fully bio-based textiles and fully biodegradable textile products. Actually, a broad range of different natural dyes are available on the market. To support a comprehensive summary on natural dyes and color components, recently six overview papers were published on a broad range of different natural dyes. These overview papers put a special focus on the application to textile materials and IR spectroscopic data. Nevertheless, several natural color components were not considered leading to the demand for publication of supplementary data on natural dyes and their IR spectra. Following this demand, the current paper finally supports supplementary data of 27 natural color components of different origin. Data of five different maya pigments are presented. Further, seven products of natural mordanting agents based on different tannin and tara products are considered. A structural and systematic discussion of IR spectra is supported. These reported supplementary IR spectroscopic data is a helpful tool for people working with natural dyes, material scientists and for quality control. This overview is also a valuable tool for educational purposes guiding interested persons into the field of natural dyes and infrared spectroscopy.*

## Keywords

Natural dye,  
Infrared spectroscopy,  
Wood originated dyes,  
Insect originated dyes,  
Asphalt,  
Maya pigments,  
Mordanting agents,  
Tannin,  
Tara products

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## 1 Introduction

Natural dyes are originated from a broad range of different sources from nature as plant, animals, algae or mushrooms [1-4]. As living organism also mushrooms can be an effective source for dyes useable for

textile coloration [5-7]. However, also mineral sources are used for some natural dyes [1, 8, 9]. Usually natural dyes are not gained as pure substances from the natural source. They have to be extracted and gained from the natural source, so a clear effort of energy, water and also chemicals is necessary for production of natural dyes [10-12]. Before the invention of synthetic dyes, over centuries the use of natural dyes was the only technology to bring coloration to products made by humans. However, after invention and further development of synthetic dyes the use of natural dyes drastically decreased due to several reasons. Synthetic dyes are available in large amounts independently from natural sources and are quite cost effective [13]. They offer a broad range of different brilliant colors and can reach excellent fastness properties against washing, rubbing and illumination [14, 15]. Excellent washing stability is reached e.g. with reactive dyes on cotton or disperse dyes on polyester fabrics [16-19]. Excellent light fastness is e.g. reached with metal complex dyes as copper phthalocyanine [8, 20]. With this background it is surprising that nowadays there is again an increasing interest in the use of natural dyes for coloration of textiles and clothing [21]. This interest results from different areas. First, there is still the handicraft area with a huge number of persons like to dye natural fabrics by themselves. The importance of this handicraft section is demonstrated by the broad range of literature and books in this field [22-26]. Second, there are scientists from different expertise searching for new natural materials and their application [27, 28]. Third, there is a certain public demand for bio-based and sustainable products, especially due to the discussion on the high environmental impact of the textile industry [29-31]. For textile products from synthetic fibers in recent years the discussion came up on related microplastic pollution originated from synthetic fiber materials [32, 33]. To avoid microplastic pollution and aggregation, the use of bio-based and biodegradable materials is considered which would be decomposed in reasonable short time after their release to natural environment [34, 35]. To realize a fully bio-based and colored textile product, the use of natural dyes is an opportunity [36]. By this, natural dyes can be part of a new approach for developing sustainable and eco-friendly clothes [37, 38]. With this background recently six overview articles on commercially available natural coloration products and their IR spectra were published [39-44]. The composition of those dyes, their origin, application and further properties are described. The IR spectra are reported to guide the reader through a structural discussion on the chemical composition of natural dyes and the chemical structure of main color components. By this also valuable spectroscopic reference data is supported [39-44]. The IR spectroscopy is an established spectroscopic method in organic chemistry and used as well for the analytic of fiber-based materials [45-48]. It is as well used for the identification of natural colorants in ancient and historical textile materials [49, 50]. The IR spectroscopy is as analytical method especially advantageous, because it is a non-destructive method with a fast measurement speed. It is used for investigation of textile and clothing products to analyze and determine fiber compositions and applied additives [51-54]. Additionally to the investigation of clothing, also textile containing materials as artificial leather, composite materials and leather are investigated with IR spectroscopy [55-57].

The recently published six overview articles on natural dyes support a broad view on natural color components from plants, insects and algae and support together IR spectra of more than one hundred substances [39-44]. Nevertheless, even by view on the broad range of provided IR spectroscopic data, there are still many interesting natural color products worth to be described and discussed. To close this gap is the aim of the current paper on supplementary data on natural color materials and their IR spectra. Here different focusses are set. First, earlier not presented materials are considered. Second, recently considered natural dyes are discussed which are delivered by different suppliers or offered in different application form, e.g. as color extract or as part of a ready-made recipe. Third, so-called maya pigments are reported which are combinations of indigoid dyes and layered silicates. Further, some commercially available bio-based mordanting agents are reported. In fact, by supporting these broad range of supplementary data a broad data gap is closed and a nearly complete view on manifold different natural coloration products is presented. These data are useful for persons working in several field as new bio-based products, material analytics and quality control. The current paper can be also used as valuable tool for educational purposes guiding interested persons into the field of natural dyes and infrared spectroscopy.

## 2 Materials and spectroscopic method

### 2.1 Natural color materials

Altogether, 26 different natural color materials are considered for this overview on natural dyes and their IR spectra. These color materials are supplied by six different companies; Das Wollschaf (Zweibrücken, Germany), AMA Herbal (India), Jeromin-Shop (Mannheim, Germany), LIVOS Pflanzenchemie (Wrestedt, Germany), Hammerl GmbH (Baiersdorf, Germany), Pflanzenfärbeshop (Hückelhoven-Baal, Germany). The purpose of these color materials is mainly their application on textile and fiber materials. However, the products from the supplier Hammerl GmbH are dedicated to the treatment of wooden music instruments. The products of the company LIVOS Pflanzenchemie are supplied together with washing agents and mordanting agents as ready-made recipes for home-dyeing purposes and handicraft applications. All considered color materials are listed in Table 1 together with proposed color shades and a reference link leading to further information as application notes and safety data sheets.

Table 1. Overview on discussed dye products. The mentioned color shades are mentioned in the description of the suppliers.

No.	Color material	Supplier	Proposed color shades	References
SP1	Madder extract / Rubia™	AMA Herbal	Red, pink, orange	[58]
SP2	Madder extract	Wollschaf	Red, violet, brown	[59]
SP3	Madder extract	Jeromin	Red, violet	[60]
SP4	Madder red / resin	LIVOS	Red	[61]
SP5	Aloe	Hammerl	Yellow to yellow/brown	[62]
SP6	Yellow wood extract	Wollschaf	Yellow, yellow/brown	[63]
SP7	Red wood extract	Wollschaf	Red, red/violet	[64]
SP8	Log wood extract	Jeromin	Blue, black, brown	[65]
SP9	Quebracho wood extract	Wollschaf	Beige/brown	[66]
SP10	Chestnut wood extract	Wollschaf	Beige/brown	[67]
SP11	Oak bark, cut pieces	Wollschaf	Light beige	[68]
SP12	Barberry bark, cut pieces	Färbeshop	Yellow, green	[69]
SP13	Akaroid	Hammerl	red	[70]
SP14	Reseda extract	Wollschaf	Yellow/olive	[71]
SP15	Reseda extract	Jeromin	yellow	[72]
SP16	Reseda yellow	LIVOS	yellow	[73]
SP17	Catechu extract	Wollschaf	Beige/brown	[74]
SP18	Catechu extract	Hammerl	Beige/brown	[75]
SP19	Catechu	LIVOS	Red/brown	[76]
SP20	Tegetas extract, Yeliona™	AMA Herbal	Bright yellow	[77]
SP21	Terminalia chebula extract, Karrel™	AMA Herbal	Greenish-yellow	[78]
SP22	Green extract mixture	Wollschaf	Green, petrol to olive	[79]
SP23	Extract from sea buckthorn leaves	Wollschaf	yellow, grey to black	[80]
SP24	Yellow berry extract	Wollschaf	Yellow/olive	[81]
SP25	Cochineal extract	Jeromin	red	[82]
SP26	LacRed	LIVOS	red	[83]
SP27	Asphalt	Hammerl	Black/grey	[84]

## 2.2 Maya pigments

Maya pigments are built up by the combination of the natural dye indigo and magnesium aluminum layered silicates [85, 86]. These pigments are also categorized as organic/inorganic complex pigments or clay-organic pigments [87, 88]. In frame of the actual overview five different maya pigments are considered. Four of those pigments contain indigo or indigo-derivatives and organic dye. All pigments are supplied by the company Kremer Pigmente GmbH (Aichstetten, Germany). An overview on discussed Maya pigments is given in Table 2 together with a reference link to additional information given by the supplier, as material composition, recommendation on recipes, applications and safety data.

Table 2. Overview on discussed Maya pigments.

No.	Maya Pigment	Supplier	References
MA1	Maya Blue	Kremer	[89]
MA2	Maya Blue, light	Kremer	[90]
MA3	Maya Blue, grey-blue B	Kremer	[91]
MA4	Maya Blue, deep blue M	Kremer	[92]
MA5	Maya Red	Kremer	[93]

## 2.3 Natural mordanting agents

For most natural dyes the application in combination with a mordanting agents is necessary to achieve intensive colorations with sufficient washing and light fastness. Also, the color shade is strongly influenced by type and amount of used mordanting agent [94-96]. Mostly water soluble metal salts are used for mordanting purposes. Common examples for metal based mordant agents are aluminum acetate, iron (II) sulphate, tin (II) chloride, copper sulphate [94, 96]. However, also organic mordanting agents from natural sources are offered and used [97, 98]. Due to this, in this overview also six commercially available natural mordanting agents are considered. These natural mordanting agents are supplied by five different companies – Carl Roth GmbH (Karlsruhe, Germany), Kremer Pigmente GmbH (Aichstetten, Germany), Das Wollschaf (Zweibrücken, Germany), Pflanzenfärbeshop (Hückelhoven-Baal, Germany) and Jeromin-Shop (Mannheim, Germany). The mordanting agents are listed in Table 3 together with a reference link to additional information as application procedures, recipes and safety instructions.

Table 3. Overview on discussed natural mordanting agents.

No.	Mordanting agent	Supplier	References
M1	Tannic acid	Carl Roth	[99]
M2	Tannin	Kremer	[100]
M3	Tannin	Wollschaf	[101]
M4	Tannin, light	Wollschaf	[102]
M5	Tara powder	Färbeshop	[103]
M6	Tara powder	Wollschaf	[104]
M7	Myrobalane powder	Jeromin	[105]

## 2.4 Analytical methods

All presented infrared spectroscopic data are determined with a FT-IR spectrometer IRTracer-100 from Shimadzu (Japan) equipped with a Specac Golden Gate ATR unit. From selected samples, microscopic images are recorded using scanning electron microscopy. For SEM measurements a Tabletop microscope TM4000 supplied by Hitachi (Japan) is used.

### 3 Dyeing materials and their IR spectra

In this section several natural dye and color components are reported together with their IR spectra. A presentation in different sub-sections is done to support an adequate structure for the reader. Plant dyes with chemical unit of anthraquinone are described, following by the sub-section related to dyes originated from wooden materials. Finally, a third sub-section generally dedicated to plant dyes follows. Other sub-sections are dedicated to color components from insects or asphalt.

#### 3.1 Anthraquinone based dye

Anthraquinone based plant dyes are earlier presented in the first overview article dedicated to "Plant based dyes with naphthoquinone and anthraquinone structure [Overview1]. For comparison the chemical structure of anthraquinone is given in Figure 1. In fact, this structure is the basic chromophore for several types of natural color components containing different functional groups bonded to the chromophore as auxochromic groups [1, 8, 106]. Probable most known is the anthraquinone based dye alizarin which contains two attached hydroxy groups (compare Figure 2) [106]. Red madder (C.I.Natural Red 8) from madder root (*Rubia tinctorum* L) is probable the most know natural dye based on anthraquinone [1, 107, 108]. Additional to alizarin also other color components are part of dye extract gained from madder, as purpurin, munjistin or pseudopurpurin (for chemical structures compare Figure 2) [1, 107, 108].

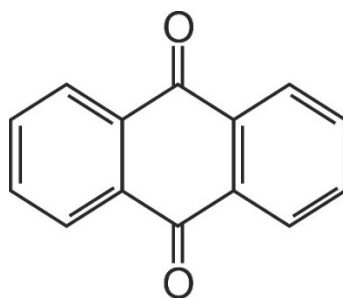


Fig. 1 Chemical structure of anthraquinone.

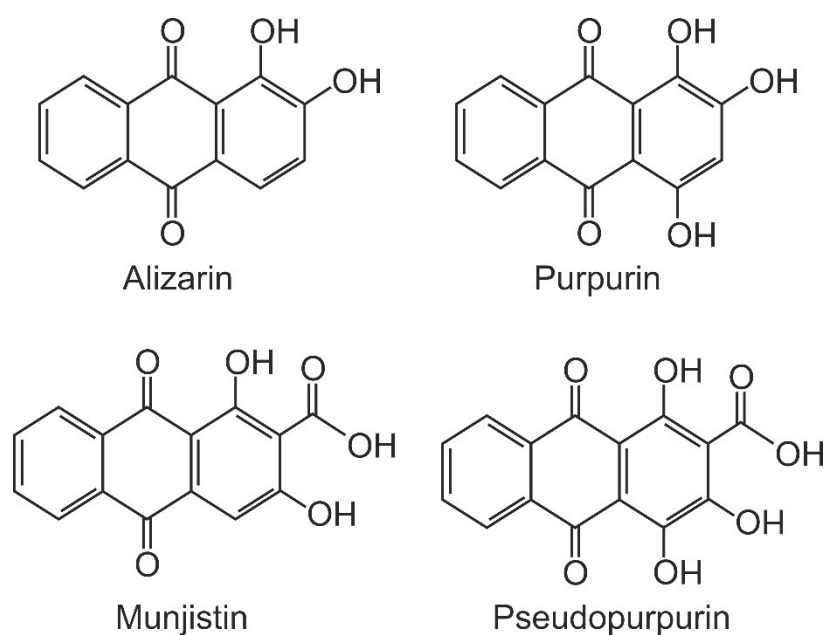


Fig. 2 Chemical structures of different dye components present in madder root extract.

Following, four different products originated from madder are discussed together with their IR spectra (Figures 3 to 5). The first product SP1 is supplied under the name Rubia by the company AmaHerbal. According to company information, this product is gained as extract from *Rubia cordifolia* (named also as Indian madder or Common Madder). Other names of this plant are Manjistha or Majith [58]. This extract from Indian madder is also classified as C.I. Natural Red 16 [108]. According to the supplier, this madder extract contains purpurin, munjistin, alizarin and pseudopurpurin. A color range from red, pink and orange is proposed. The supplier claims for this product also functional properties like antibacterial, antimicrobial and astringent properties. Such antimicrobial properties of madder root products are also reported in literature [109, 110]. However, compared to conventional antimicrobial agents based e.g. on silver, copper compound or ammonium compounds the antimicrobial activity of natural dye components are expected to be minor [111]. The IR spectrum of this product SP1 is presented in Figure 3 and it exhibits nearly no similarity to the IR spectra of the other products (compare Figures 4 and 5) or to the IR spectra for madder products reported earlier [39]. However, the IR spectrum of product SP1 has a better comparability to the IR spectrum of synthetic alizarin [39]. One main difference compared to the IR spectrum of synthetic alizarin is the appearance of the signal at  $1700\text{ cm}^{-1}$  for product SP1. This signal can be related to a C=O stretching vibration, e.g. originated from the carboxylic acid groups in the compounds munjistin and pseudopurpurin, which are claimed to be part of this natural product.

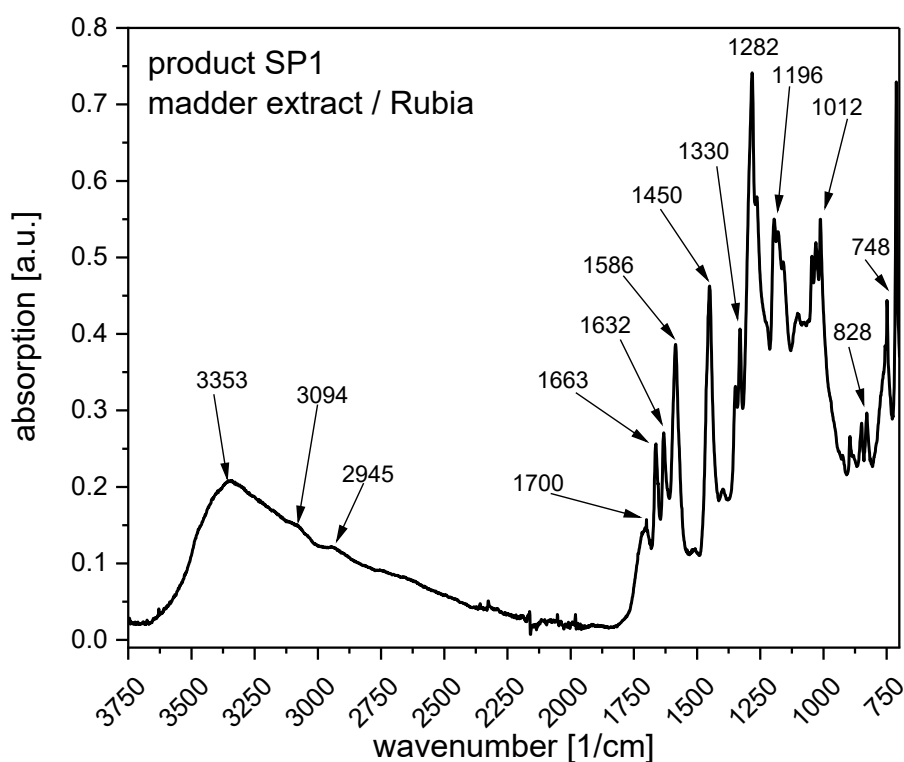


Fig. 3 IR-spectrum of the product Rubia - extract from Indian madder.

The both products SP2 and SP3 are supplied as extract from madder root (*Rubia tinctorum* L) by two different German suppliers. The proposed color range for dyeing on textiles is given with red, violet to brown. The IR spectra of both products show a good similarity to each other and also to earlier reported IR spectra from madder extracts [39]. For this, a clear identification of this product by IR spectroscopy can be stated.

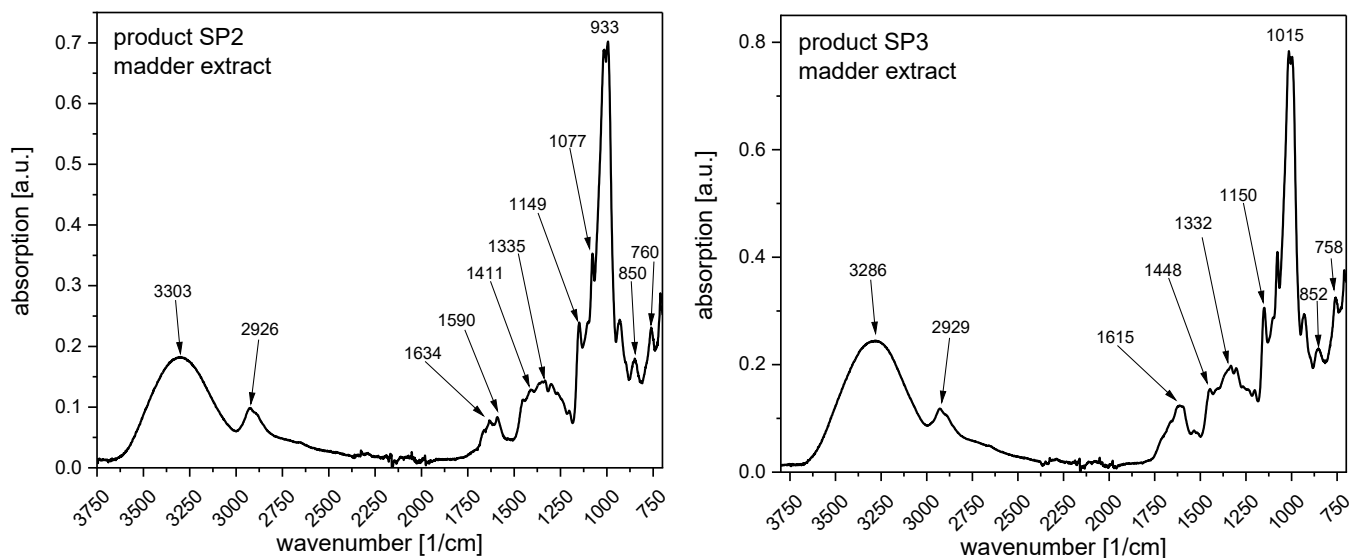


Fig. 4 IR-spectra of two different dye extracts from madder.

The product SP4 supplied by LIVOS is in contrast to the other here described madder powder products a dark red resin mass. However, despite of the different appearance the IR spectrum of product SP4 is nearly similar to the both IR spectra from madder extracts shown in Figure 4. From this it can be stated that even if the optical appearance of these madder products is different, they are from chemical point of view quite similar.

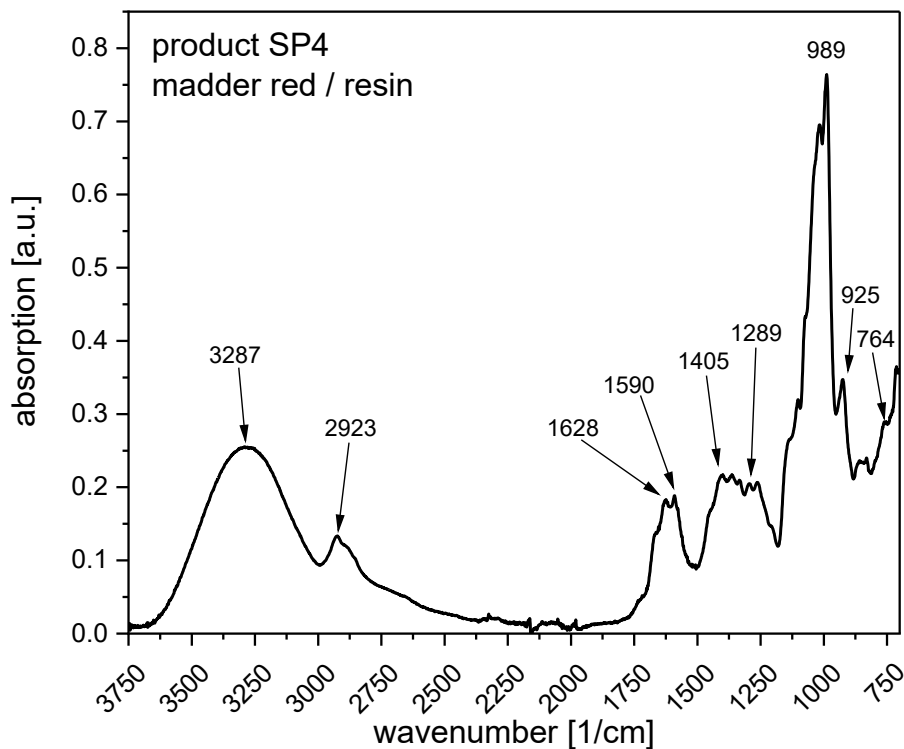


Fig. 5 IR-spectrum of the product KrappRot (madder red) - red resin.

As second anthraquinone based product, here an extract from Aloe is presented. This Aloe extract is supplied by a German company and originally dedicated for the treatment of wooden materials – especially wooden music instruments. Aloe plants belong to the group of *Liliaceae*. The leaves of this plant are protected against UV light from sun by a content of anthraquinone dyes [112]. One main color component in Aloe is Aloe-Emodin (1,8-dihydroxy-3-(hydroxymethyl) 9,10-anthraquinone). Its chemical structure is shown in Figure 6. Here as auxochromic groups, two hydroxy groups are directly bonded to the anthraquinone chromophore. Additionally, a methylene group with a hydroxy group  $-\text{CH}_2\text{-OH}$  is also attached which is not acting as auxochromic group [113].

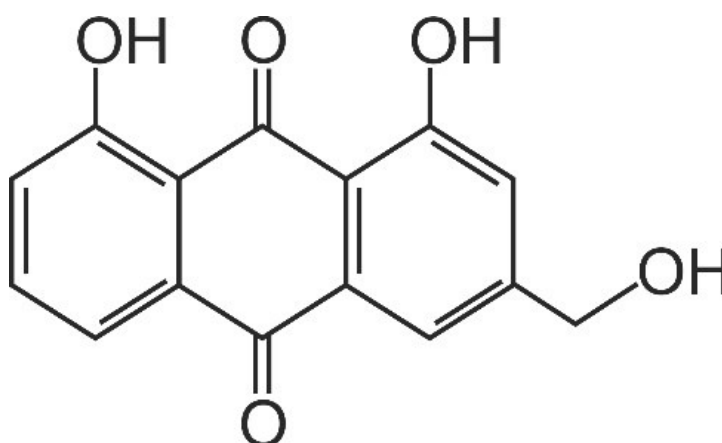


Fig. 6 Chemical structure of Aloe-emodin.

The IR spectrum of the Aloe extract SP5 is shown in Figure 7. This IR spectrum is significantly different to the IR spectra of the other anthraquinone based products presented above. The broad signal at around  $3295\text{ cm}^{-1}$  is related to the O-H stretching vibrations of the hydroxy groups. The signal at  $2925\text{ cm}^{-1}$  is assigned to C-H stretching vibration of aliphatic units as  $-\text{CH}_2-$ . The three signals at  $1712\text{ cm}^{-1}$ ,  $1652\text{ cm}^{-1}$  and  $1601\text{ cm}^{-1}$  are related to C=O stretching vibrations. These signals can be either originated from the aloe-emodin molecule but also from by-components in the natural extract product. In fact, the molecule of aloe-emodin exhibits two different types of keto groups. One keto group is neighbored by two hydroxy groups, here intramolecular hydrogen bridge bonds between the keto and the hydroxy groups are possible. The other keto group cannot build up such intramolecular hydrogen bridges. For this, IR signals at different positions are expected for these both different keto groups. Probable their C=O stretching vibrations are related to the signals at  $1652\text{ cm}^{-1}$  and  $1601\text{ cm}^{-1}$ . In contrast, the signal at  $1712\text{ cm}^{-1}$  is probable related to a C=O stretching vibration from an ester group, which is not part of the molecule of aloe-emodin. Probable this is related to a by-component containing an ester group. The both signals at  $1077\text{ cm}^{-1}$  and  $1032\text{ cm}^{-1}$  are related to C-O stretching vibrations caused by hydroxy units bonded to aliphatic or aromatic carbon. Additionally, these signals can be related to C-O stretching vibrations from ester units of the above mentioned by-component. The peak at  $831\text{ cm}^{-1}$  can be assigned to deformation vibrations of aliphatic  $\text{CH}_2$  units.

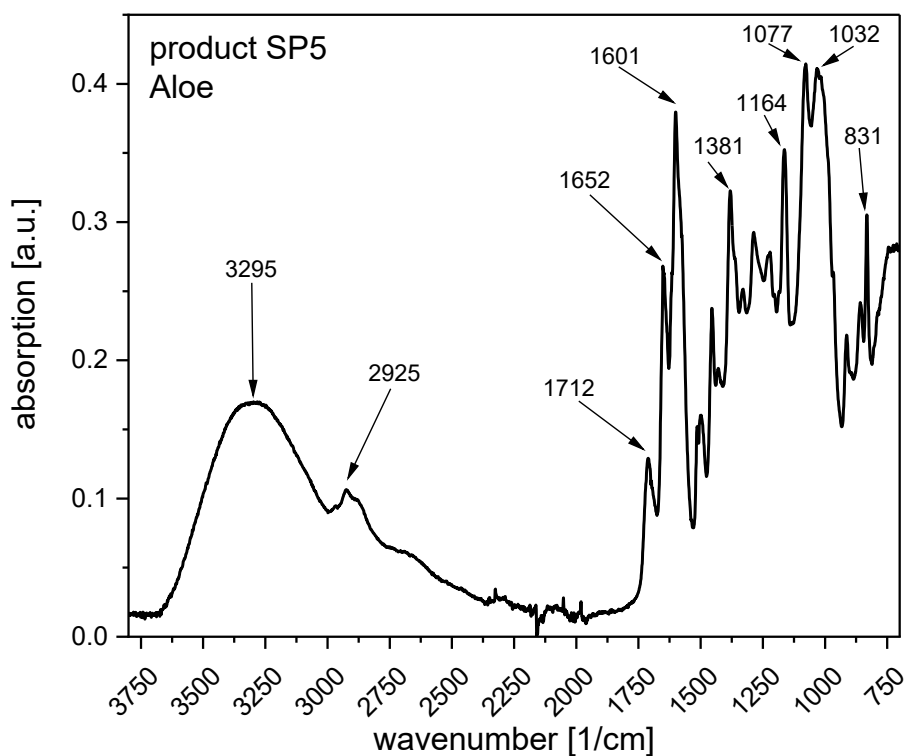


Fig. 7 IR-spectrum of Aloe extract (technical).

### 3.2 Dye materials from wood

In this section different materials sourced from wood and trees used for coloration purposes are presented, these are cut pieces from bark and extracts from colored wood. The first presented product is an extract from yellow wood (*Maclura tinctorial*) supplied by a German supplier [63]. This type of wood is also named as old fustic (*Chlorophora tinctorial*) [1]. The proposed coloration for application on textiles is according to the supplier in the range of yellow to yellow/brown [63]. One main color component in old fustic is Morin (C.I.75660; C.I. Natural Yellow 8, 11), compare its chemical structure in Figure 8. Morin is of flavone basic structure with attached five hydroxy groups acting as auxochromic units [114].

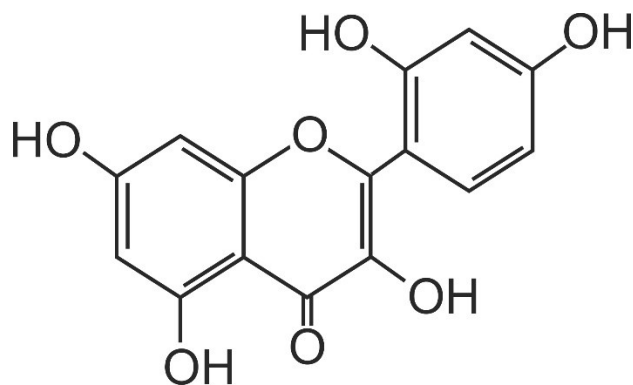


Fig. 8 Chemical structure of Morin, one main color component in yellow wood.

In the earlier overview, only the IR spectrum from cut yellow wood pieces are reported, so the now reported IR spectrum of yellow wood extract closes the gap in the description of yellow wood materials [41]. The actual reported IR spectrum recorded from yellow wood extract exhibit no similarity to the IR spectra of the cut yellow wood pieces (Figure 9) [41]. The chemical composition of extract and wood pieces is mainly different. Some signals in the IR spectrum of yellow wood extract can be set in good correlation to the chemical structure of Morin, as the broad signal around  $3220\text{ cm}^{-1}$  which is assigned to the O-H stretching vibrations of hydroxy groups. Also, the signal at  $1607\text{ cm}^{-1}$  can be related to C=O stretching vibrations of the keto group. However, the weak shoulder signal at  $2925\text{ cm}^{-1}$  is related to C-H stretching vibrations of aliphatic units, which are not present in the Morin molecular structure. Probable these signals and other are caused by by-components which are present in this natural product.

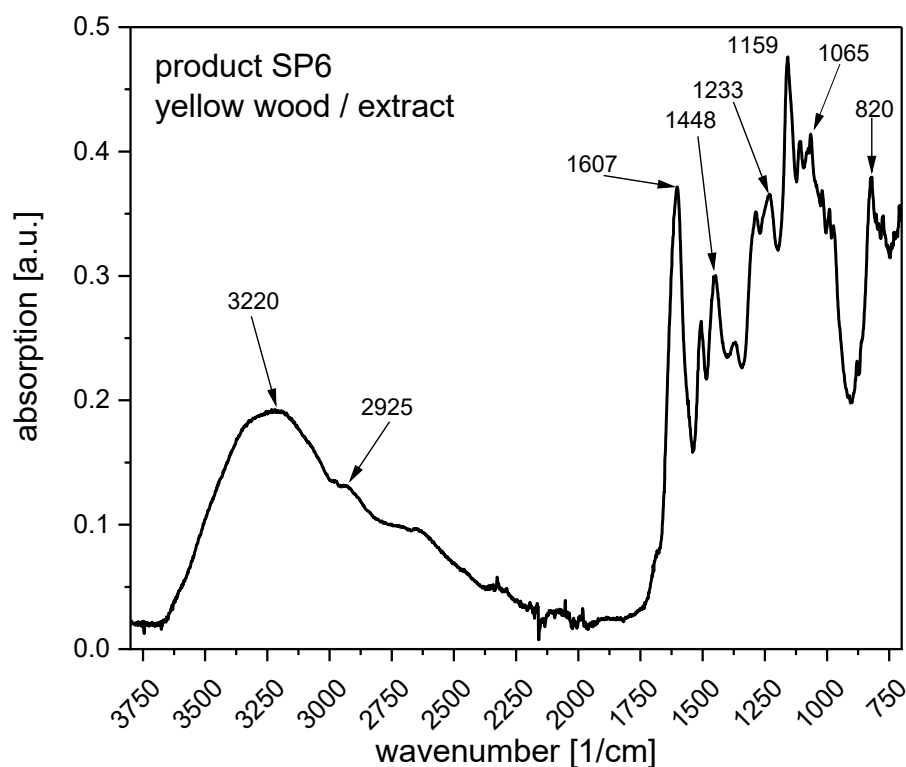


Fig. 9 IR-spectrum from powder of yellow wood extract.

The other three reported dye extracts are from logwood, red wood and quebracho wood and are supplied by different German suppliers. In the earlier overview, extracts from these wood types are already presented [41]. For this, the now supported data are broadening the IR spectroscopic data base on color wood extracts. The product SP7 is an extract from red wood / brazil wood (*Heamatoxylum braziletto*) supplied by a German company. The supplier proposed a range of coloration from red to violet. The IR spectrum of product SP7 is presented in Figure 10. This IR spectrum is nearly similar to earlier reported IR spectra from brazil wood extract [41]. For this, the identification of these type of products is clearly possible by IR spectroscopic methods.

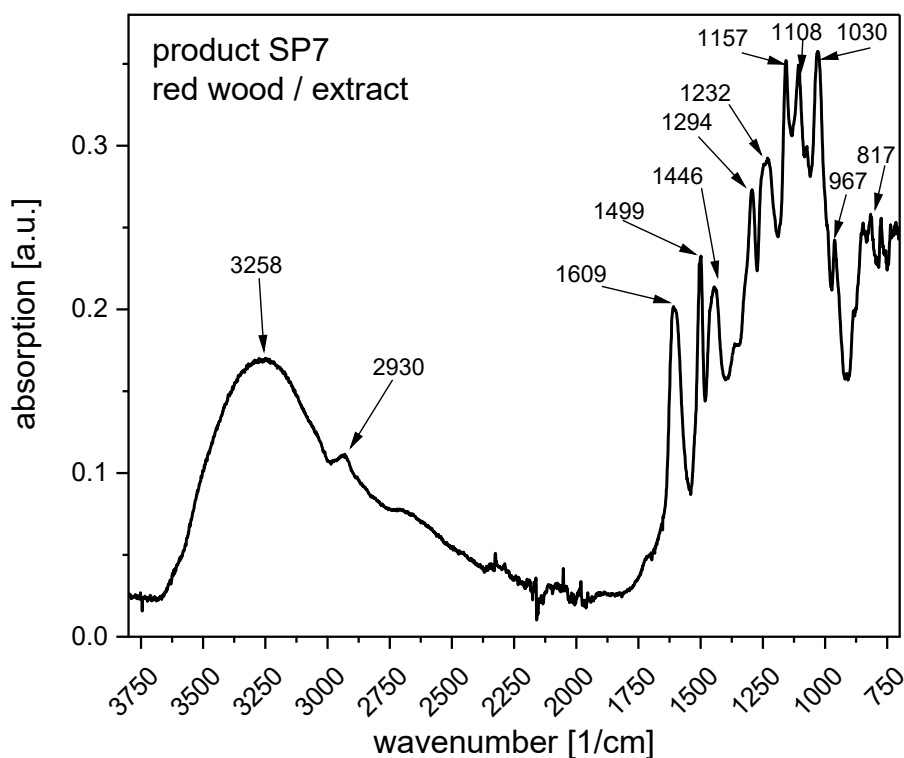


Fig. 10 IR-spectrum from powder of red wood extract.

The product SP8 is an extract from logwood supplied by a German company. The supplier proposed a range of coloration from blue, black to brown. This color shade is strongly determined on the type of used metal mordanting agent and the applied mordant concentration [96]. Logwood extracts are also used for the preparation of black colored monofilament nylon suture and black silk suture yarns for medical applications [115-118].

The IR spectrum of product SP8 is presented in Figure 11. The IR spectrum of product SP8 exhibits significant differences to earlier reported IR spectra of logwood extract powders [41]. Especially remarkable is the clear signal at  $1699\text{ cm}^{-1}$ , which is not determined for other logwood extracts and which cannot be related to the chemical structure of hematoxylin as main color component in logwood extract.

This signal can be related to a C=O stretching vibration of an ester unit which might be part of a by-component present in this logwood product. Finally, for the different commercially available logwood extracts additionally to the main color components a different composition of by-components can be stated, probable because of differently performed extraction procedures or different origin of the logwood used for the extraction.

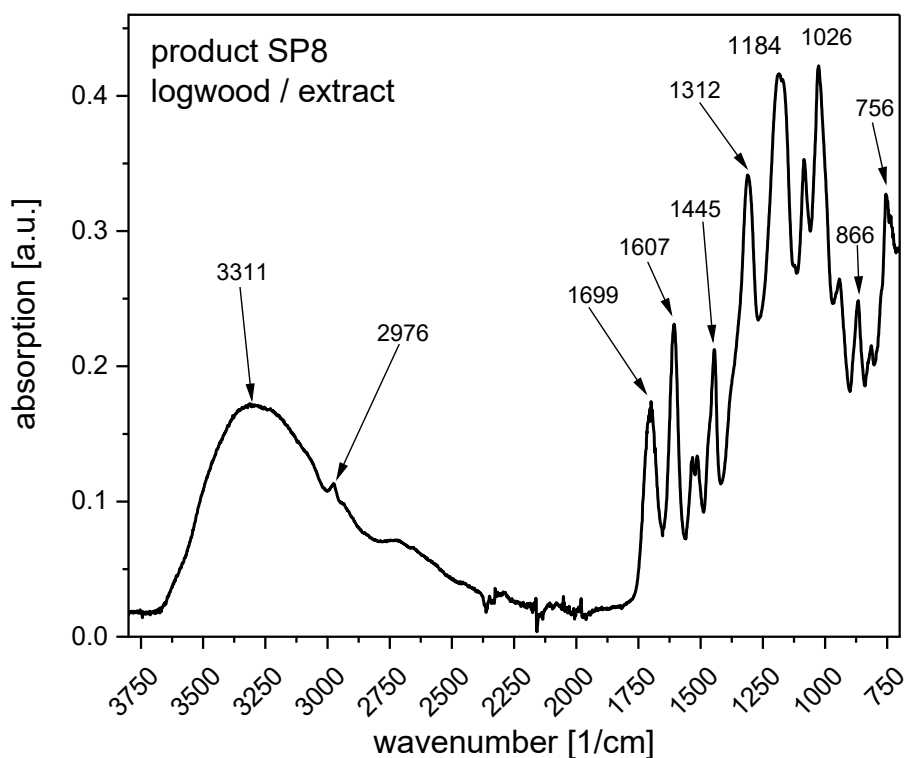


Fig. 11 IR-spectrum from powder of logwood extract.

The product SP9 is an extract from quebracho wood (*Schinopsis lorentzii*) supplied by a German company. The supplier proposed a range of coloration from beige to brown. It is also recommended for the use as bio-based mordanting agent [119]. Quebracho is described as cost-effective tanning material for leather several decades ago [120]. Nowadays, it is presented as vegetable tanning agents for leather goods alternatively to chromate based tanning procedures [121, 122]. The application of quebracho on textiles during dyeing or as pre-treatment can be also used as tool for functionalization of textile products. Improved UV protective properties are reached by quebracho pre-treatments in dyeing processes [123]. For quebracho wood extract also antimicrobial and antiviral activity is described [124]. Additional to the described antimicrobial effect for quebracho wood extract, also a larvicidal activity is reported acting against wood-damaging insects [125]. Based on this property, quebracho extract are widely investigated for wood preservation applications. In this field, the use of quebracho extract is especially attractive as alternative for metal based or chlorine containing biocides used often conventionally for wood treatment [126, 127].

The IR spectrum of product SP9 is presented in Figure 12. This IR spectrum is nearly similar to earlier reported IR spectra from quebracho wood extract [41]. For this, the identification of these type of products is clearly possible by IR spectroscopic methods. The broad signal at around 3257  $\text{cm}^{-1}$  is related to O-H stretching vibration of hydroxy groups. The shoulder signal at 2932  $\text{cm}^{-1}$  is related to C-H stretching vibrations of aliphatic units. The peak at 1607  $\text{cm}^{-1}$  is related to C=O stretching vibrations of carbonyl units. The strongest peak at 1034  $\text{cm}^{-1}$  is assigned to C-O stretching vibrations.

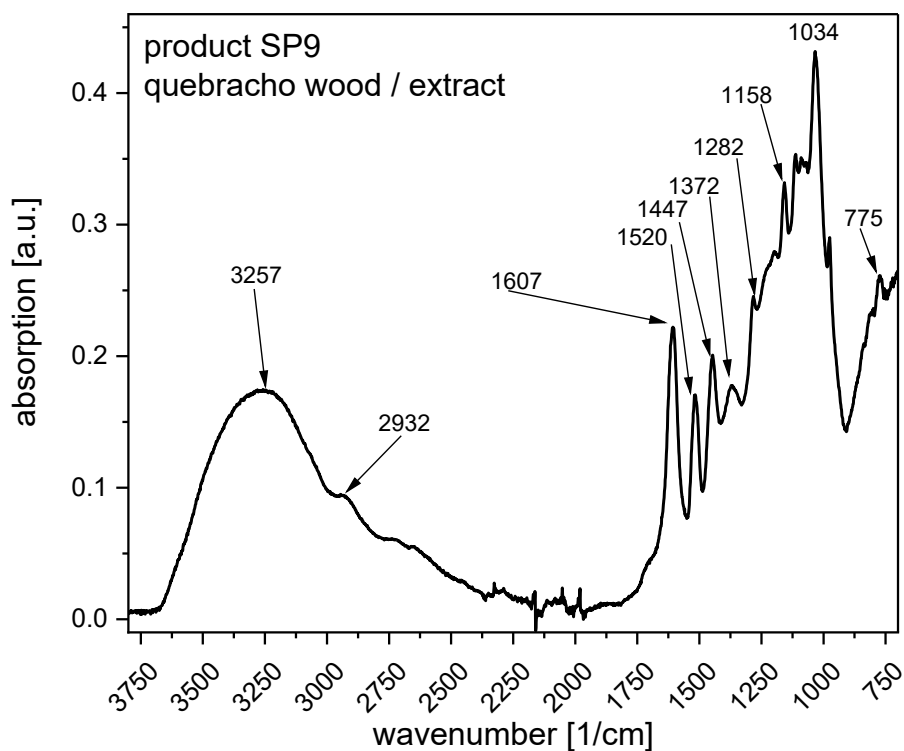


Fig. 12 IR-spectrum from powder of quebracho wood extract.

The product SP10 is a color product from Chestnut wood extract. Chestnut originated materials are not presented and discussed in the earlier overview papers. According to the supplier, the proposed coloration is in the range from brown to beige. The wood of Chestnut contains different related to ellagic acid C.I.75270 [1, 128]. Examples for those tannins from Chestnut are Castalin, Castalagin, Vescalin and Vesalagin [1]. The chemical structure of ellagic acid (2,3,7,8-Tetrahydroxychromeno[5,4,3-cde]chromen-5,10-dion -  $C_{14}H_6O_8$ ) is shown in Figure 13. The chromophore is built up by four aromatic ring systems with four attached hydroxy groups. It contains two intramolecular ester groups and ellagic acid can be seen also as a dimer of gallic acid.

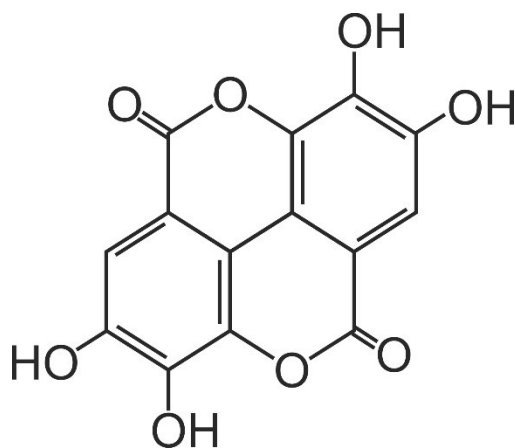


Fig. 13 Chemical structure of ellagic acid.

The IR spectrum of the Chestnut extract is presented in Figure 14. Compared to IR spectra recorded from other colored wood extract, the IR spectrum from Chestnut extract is clearly different. One main difference is the appearance of the clear signal at  $1718\text{ cm}^{-1}$ . This signal is related to the C=O stretching vibration from an ester group. By view on the chemical structure of ellagic acid, two C=O units can be identified which are part of an intramolecular ester group (Figure 13).

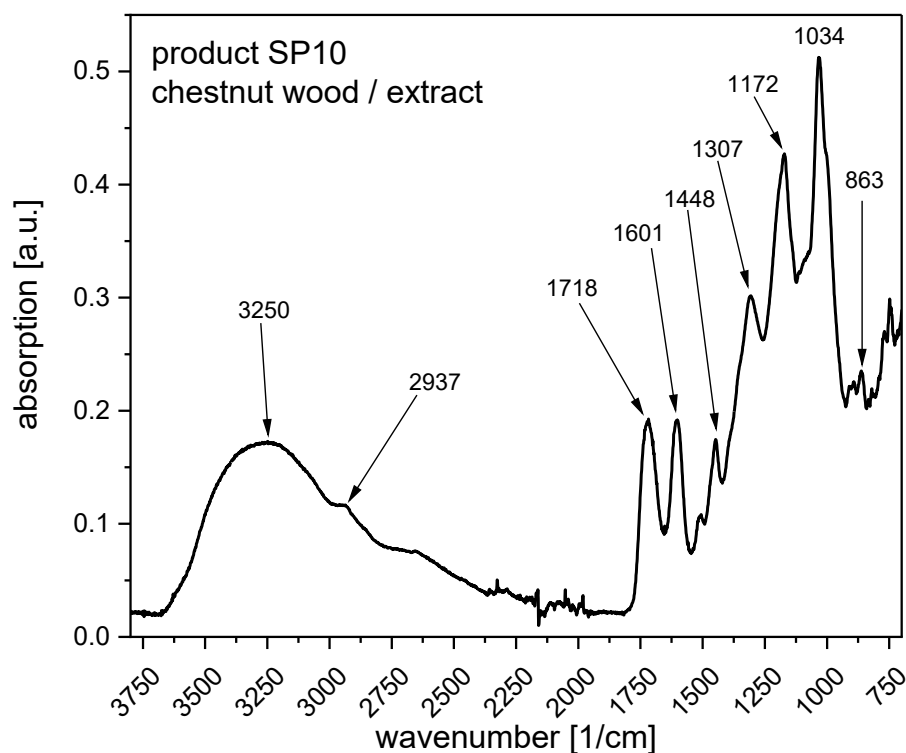


Fig. 14 IR-spectrum from powder of Chestnut wood extract.

Alternatively, to color wood and extracts from wood, often also wood bark is offered for dyeing purposes. In the earlier overview five different bark based products from four different trees are presented [41]. Now following additionally cut pieces from oak bark (*Quercus Cort.*) and barberry bark (*Berberidis*) are considered. Both bark products are supplied by German companies. The proposed coloration for oak bark is described with light beige and for barberry bark with yellow to green. Oak bark is also described for the preparation of sizing paste agents [129]. A fine investigation of dyeing with barberry bark in combination with different mordant is given by Pruthi et al. [130]. The IR spectra of the bark products SP11 and SP12 are presented in Figure 15. The IR spectrum recorded from oak bark exhibits a certain similarity to the IR spectra of linden bark and purple willow bark, which are reported earlier [41]. For this, IR spectroscopic method cannot be used to identify oak bark in comparison to other color bark products. In contrast, the IR spectrum recorded from barberry bark product SP12 exhibit a similarity to earlier reported IR spectra recorded from different wood samples (fresh cut) [41]. In this case, by IR spectroscopic method a general identification as wood material is possible. However, the specification as a product based on barberry wood or bark is not possible.

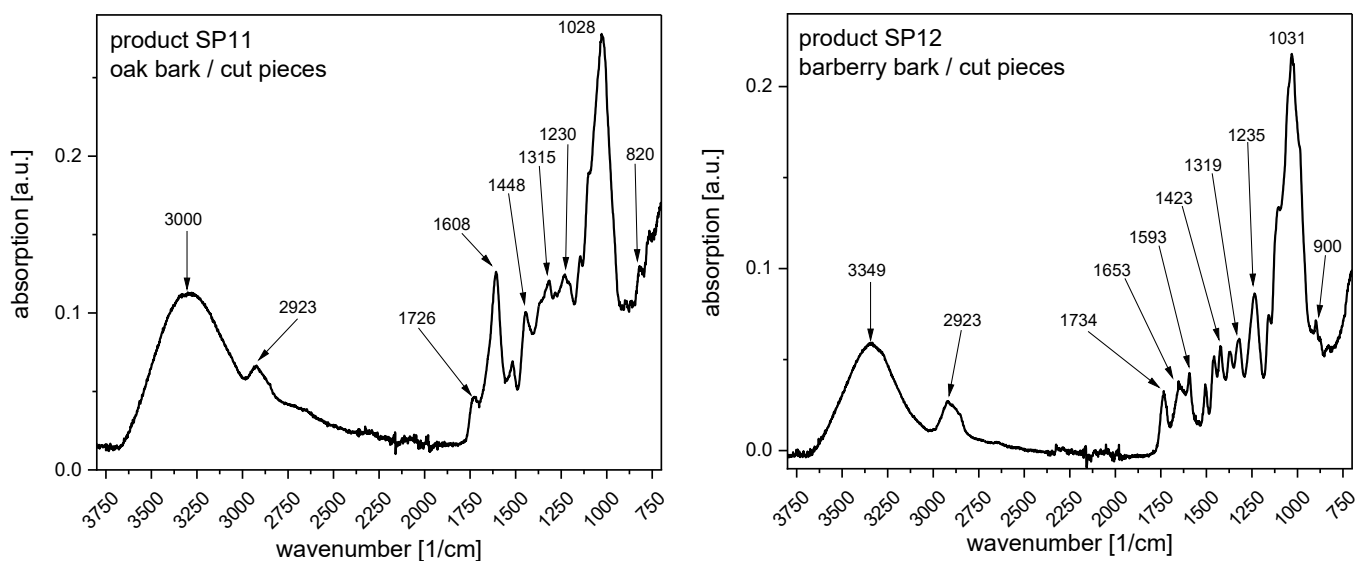


Fig. 15 IR-spectra recorded from oak bark and barberry bark (cut pieces).

Resins or gums gained from trees can be also used for dyeing purposes. A prominent example is the product red dragon blood, which is gained as resin from the dragon tree [41, 131]. In the actual overview, the acaroid resin is presented. This product SP13 is supplied by a German supplier and offered especially to be applied as wood treatment [70]. According to supplier information the proposed coloration is red. Acaroid is a resin gained from the Australian grass-tree (*Xanthorrhoea australis*) [132]. An excellent overview on Australian plant extracts including the Australian grass-tree is given by Georgiou et al. [133]. In this reference a composition of cumaric acid, chalcone, flavanone and polyflavanoid is mentioned [133].

The IR spectrum of the acaroid resin product is shown in Figure 16. This IR-spectrum of product SP13 is in a certain way similar to an IR-spectrum earlier reported for an Australian plant extract from *Xanthorrhoea Australia* [134]. The broad signal at  $3337\text{ cm}^{-1}$  is related to O-H stretching vibration of hydroxy groups. The weak signal at  $3021\text{ cm}^{-1}$  is assigned to C-H stretching vibration of C-H bonds attached to aromatic ring systems. Both weak peaks at  $2937\text{ cm}^{-1}$  and  $2838\text{ cm}^{-1}$  are assigned to C-H stretching vibrations of aliphatic units. The most prominent peak at  $1597\text{ cm}^{-1}$  can be assigned to C=O stretching vibrations of keto groups [45]. By view on this IR spectrum no components containing ester or amide group can be identified. The both peaks at  $1512\text{ cm}^{-1}$  and  $1441\text{ cm}^{-1}$  are probable related to deformation vibrations of  $\text{CH}_2$  or  $\text{CH}_3$  units. The three strongest signals at  $1201\text{ cm}^{-1}$ ,  $1156\text{ cm}^{-1}$  and  $1102\text{ cm}^{-1}$  can be assigned to C-O stretching vibrations according to different functional groups.

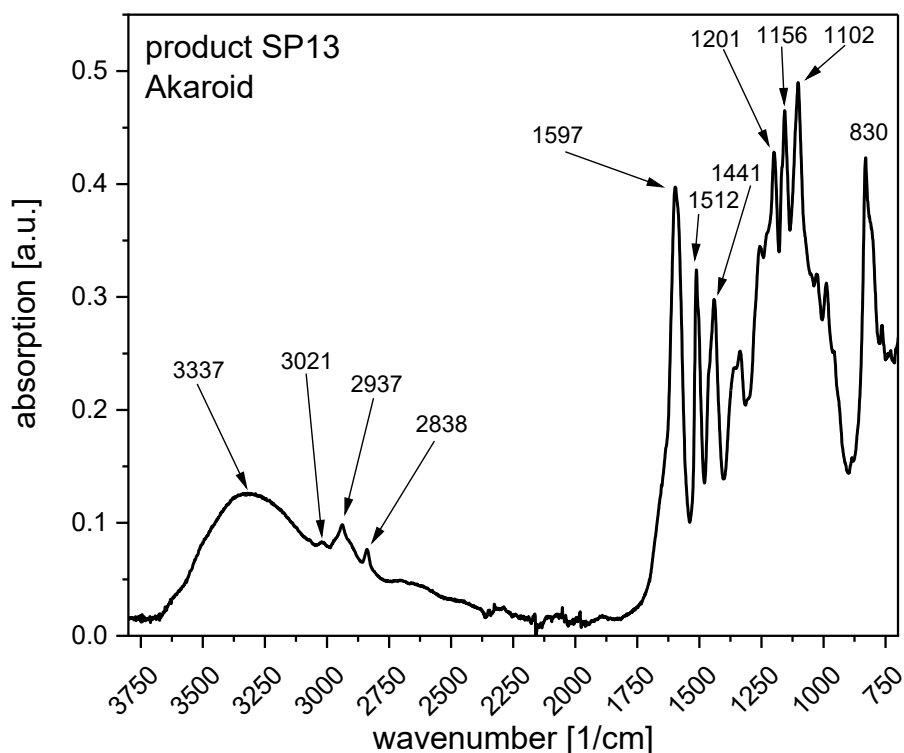


Fig. 16 IR-spectrum recorded from powder of Akaroid resin.

### 3.3 Plant dye materials

In this sub-section several different plant based color materials are reported and compared. The starting point for this report, are reseda based products. Earlier on different reseda based products (plant pieces and extracts) were reported and reported IR spectra were intensively discussed [43]. In comparison to these earlier reported data, actually three further reseda color products are here presented. The reseda products SP14 and SP15 are extracts and offered by two different suppliers for purposes of textile dyeing. The products SP16 is supplied by the company Livos as part of a ready made mixture for home dyeing application. The IR spectra of the three reseda extracts are quite similar to each other (compare Figures 17 and 18). However, they are in a certain way different to an earlier reported IR spectrum for reseda extract [43]. All the IR spectra exhibit a small peak or a shoulder at around  $1660\text{ cm}^{-1}$  which is not part of the earlier reported IR spectrum. This signal is probable caused by C=O stretching vibrations from amide or keto groups, which might be part of present by-products in the reseda extracts.

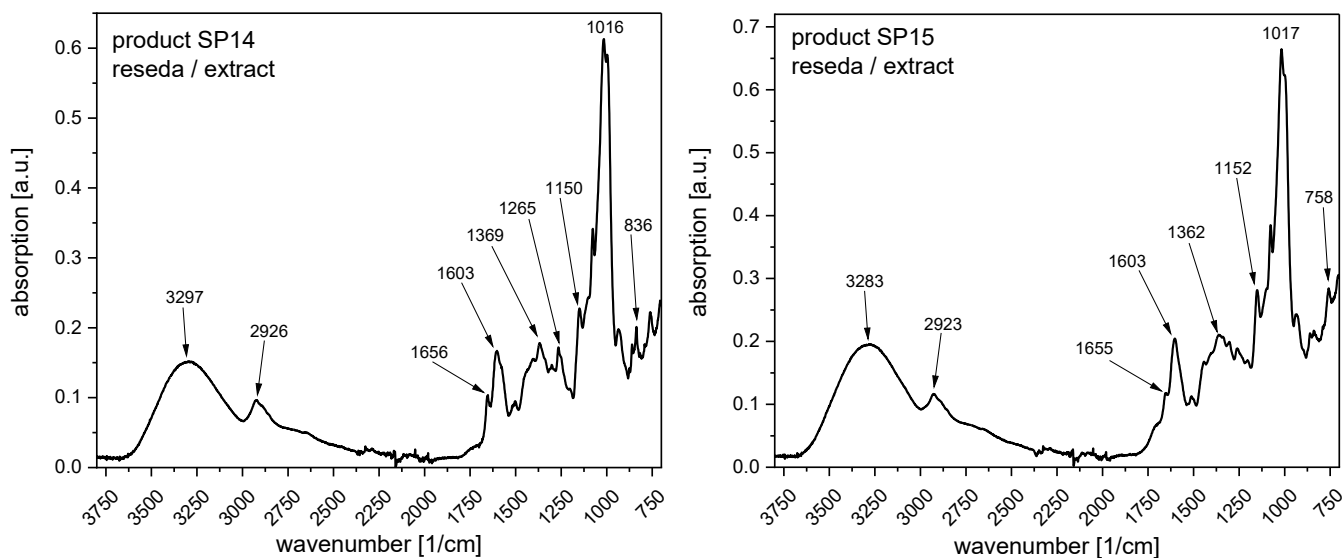


Fig. 17 IR-spectra recorded from two powders of reseda extract.

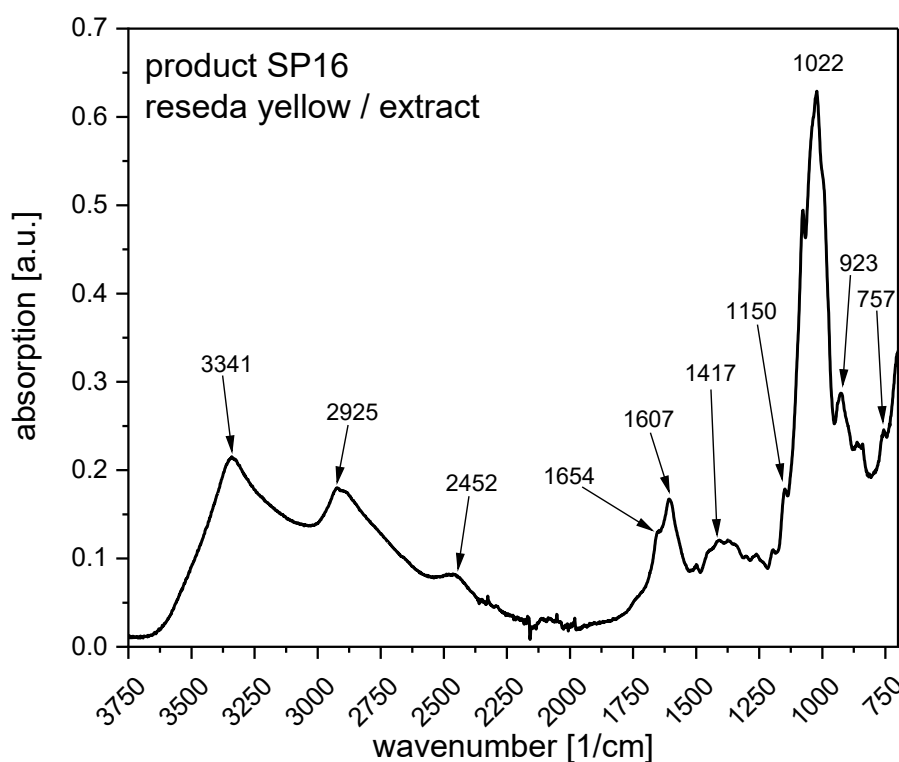


Fig. 18 IR-spectrum recorded from the product ResGelb (Reseda yellow).

The IR spectra of three catechu products are presented in the figures 19 and 20. These products are supplied by three different companies and product SP19 is part of a ready made recipe for home-dyeing purposes. The IR spectra of these three products are nearly identically and also similar to the earlier published IR spectrum recorded from catechu extract [43]. These catechu products are nearly identical and the identification of catechu extracts by IR spectroscopy is clearly possible.

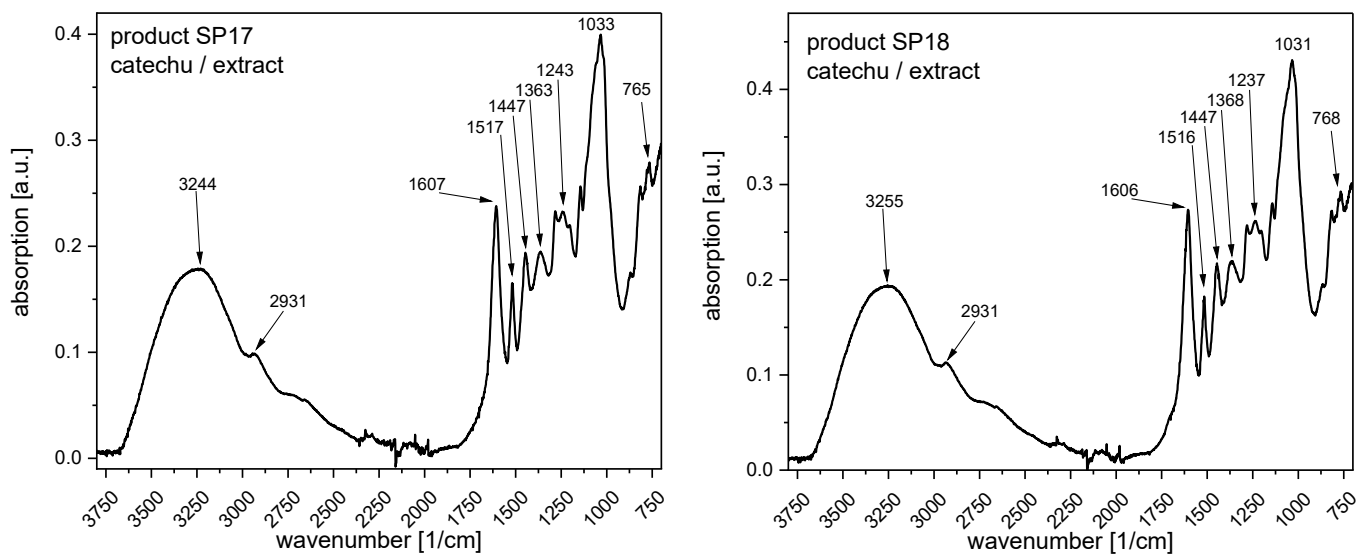


Fig. 19 IR-spectra recorded from two powders of Catechu extract.

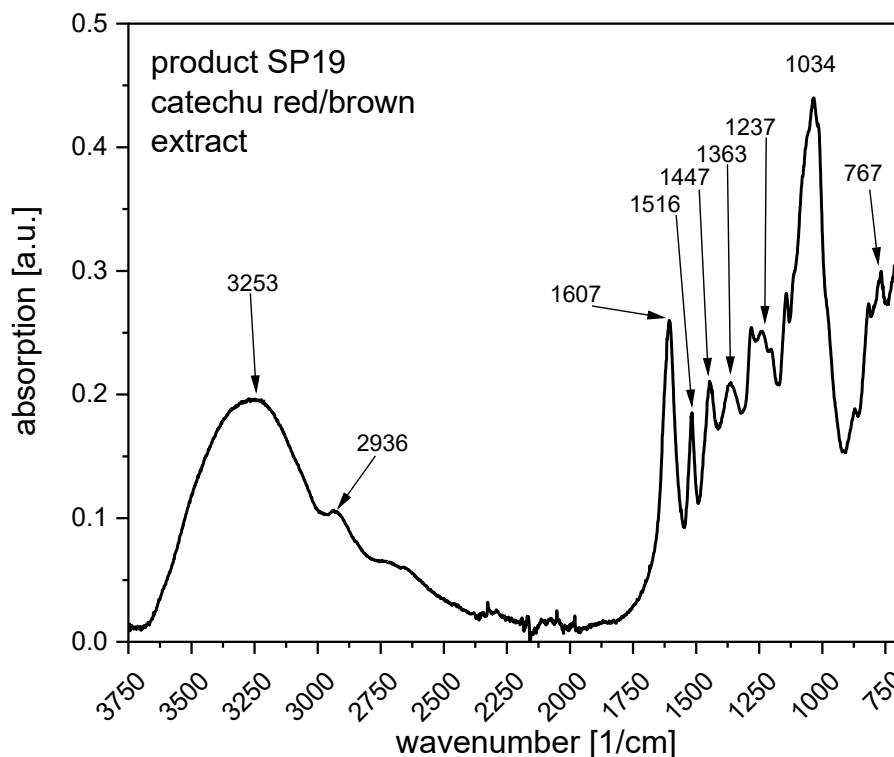


Fig. 20 IR-spectrum recorded from the product Catechu RotBraun (Catechu red brown).

The color product SP20 is supplied under the name Yeliona as a yellow colored natural dye. According to supplier information, this product is produced from extracts of the plants from *Tegetas erecta* and *Butea monosperma*. This plant is also named tesu flowers. Extracts from tesu flowers can be used for dyeing of silk by use of aluminum based mordant [135]. One main color component in *Butea monosperma* is butein

C.I 75760 C.I. Natural Yellow 28 [1]. Compare the chemical structure in Figure 21. The chromophore of butein is based on two benzene rings connected by an enol unit with attached keto group. As auxochromic groups, four hydroxy groups are present. The formation of an intramolecular hydrogen bridge bond between the hydroxy group of the enol unit and the keto group is most likely.

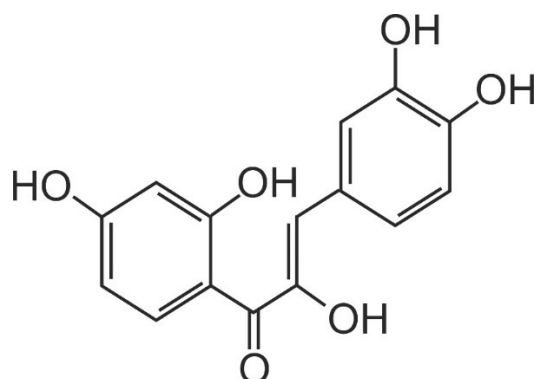


Fig. 21 Chemical structure of butein one main color component in tesu flowers.

The IR spectrum of Yeliona product SP20 is shown in Figure 22. The signals in the IR spectrum can be partly related to the chemical structure of butein (Figure 21). The broad signal at around  $3306\text{ cm}^{-1}$  is assigned to O-H stretching vibrations of the hydroxy groups. The signal at  $2927\text{ cm}^{-1}$  is related to C-H stretching vibrations from aliphatic units. However, the molecular structure of butein does not exhibit aliphatic units, so this signal is obviously related to a by-component in the color extract. The both signals at  $1632\text{ cm}^{-1}$  and  $1693\text{ cm}^{-1}$  are assigned to C=O stretching vibrations, as C=O groups are present in the molecule of butein. The strongest signal at  $1016\text{ cm}^{-1}$  can be assigned to C-O stretching vibrations of the hydroxy groups attached to the chromophore.

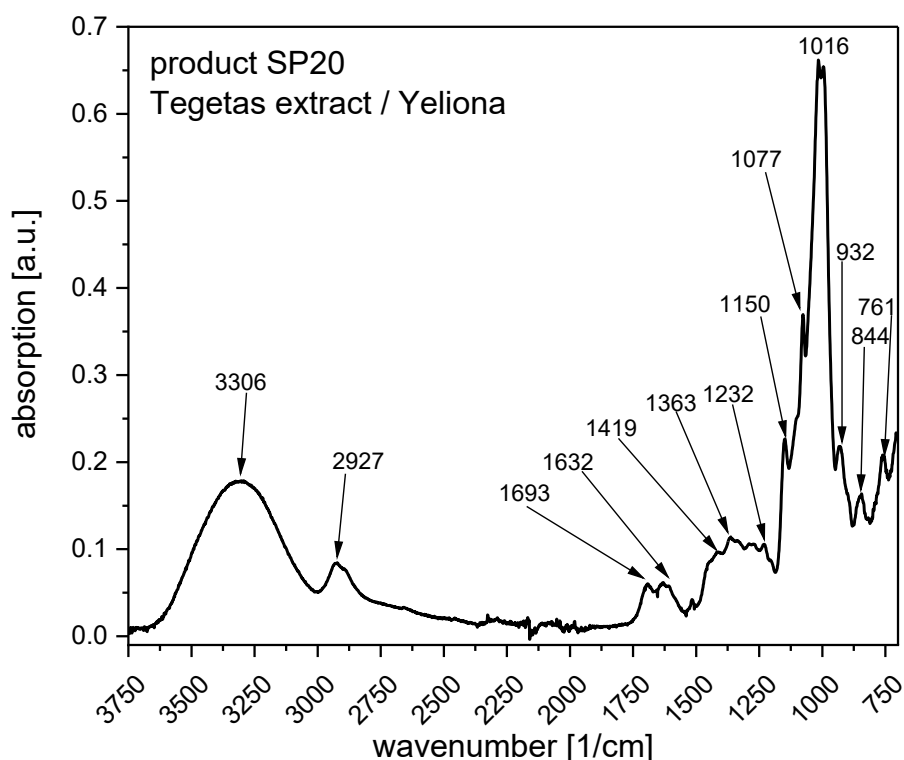


Fig. 22 IR-spectrum recorded from the natural dye product: Yeliona.

The color product SP21 is supplied under the name Karrel. According to supplier information this product is produced by extraction of the plant *Terminalia chebula* (common name Myrobalan). The proposed coloration is in the range of greenish to yellow. The extract of *Terminalia chebula* contains tannins and is therefore recommended for use bio-mordant in combination with other natural dyes. The supplier mentions also antibacterial, antimicrobial and astringent properties for textiles dyed with Kareel product. One main component of the extract of *Terminalia chebula* is chebulic acid – compare chemical structure in Figure 23 [136]. Chebulic acid exhibits a quite large molecular structure containing a sugar unit in the center. To this sugar unit several hydrophilic aromatic units are connected via ester bonds. Three of these aromatic unit are the esters of gallic acid. The application of *Terminalia chebula* natural dye on wool fiber materials is described in combination with mordants based on aluminum, iron and tin [137].

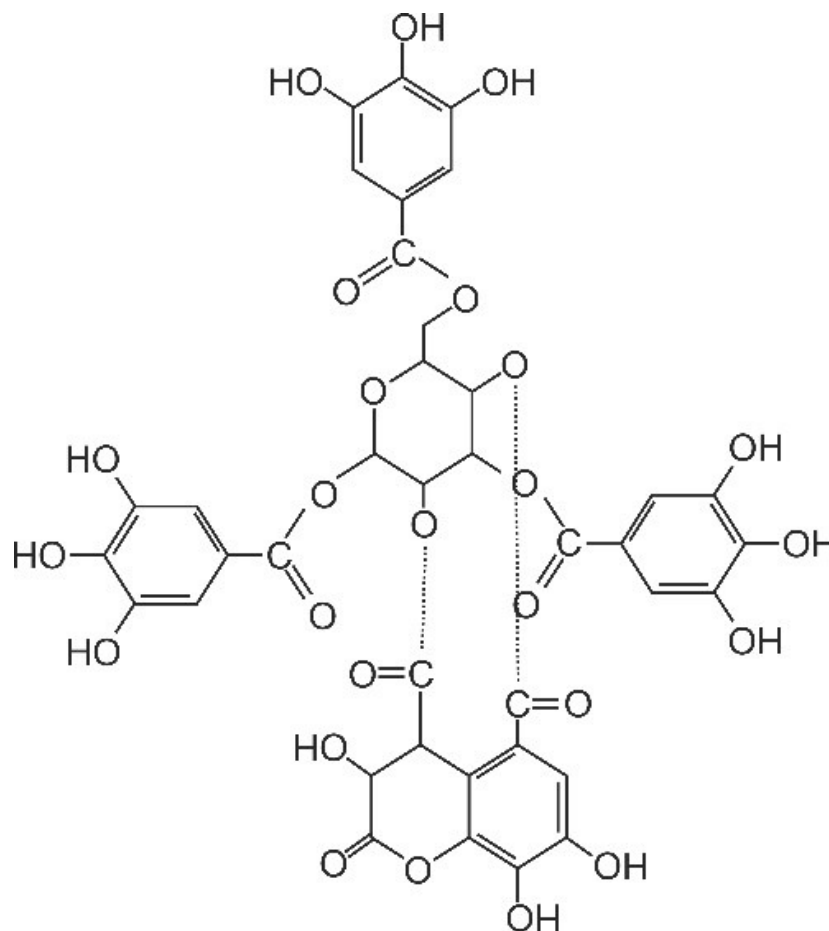


Fig. 23 Chemical structure of Chebulinic acid as one color component of Myrobalan.

The IR spectrum of the color product Karrel is presented in Figure 24 and can be compared with an IR spectrum reported in literature for a *Terminalia chebula* dye [137]. The IR spectrum in literature is of lower quality and exhibit some differences compared to the Karrel product, probable because of different composition of these natural products resulting from different plant origin or different extraction processes. The broad signal at around  $3188\text{ cm}^{-1}$  can be assigned to O-H stretching vibrations of hydroxy groups. The weak signal at  $3081\text{ cm}^{-1}$  is related to C-H stretching vibrations of aromatic units. The weak shoulder signal at  $2947\text{ cm}^{-1}$  is assigned to C-H stretching vibrations of aliphatic units. The signal at  $1695\text{ cm}^{-1}$  is related to C=O stretching vibrations of ester groups. Both strongest signals at  $1028\text{ cm}^{-1}$  and  $1195\text{ cm}^{-1}$  are related to C-O stretching vibrations from different groups as hydroxy or ester.

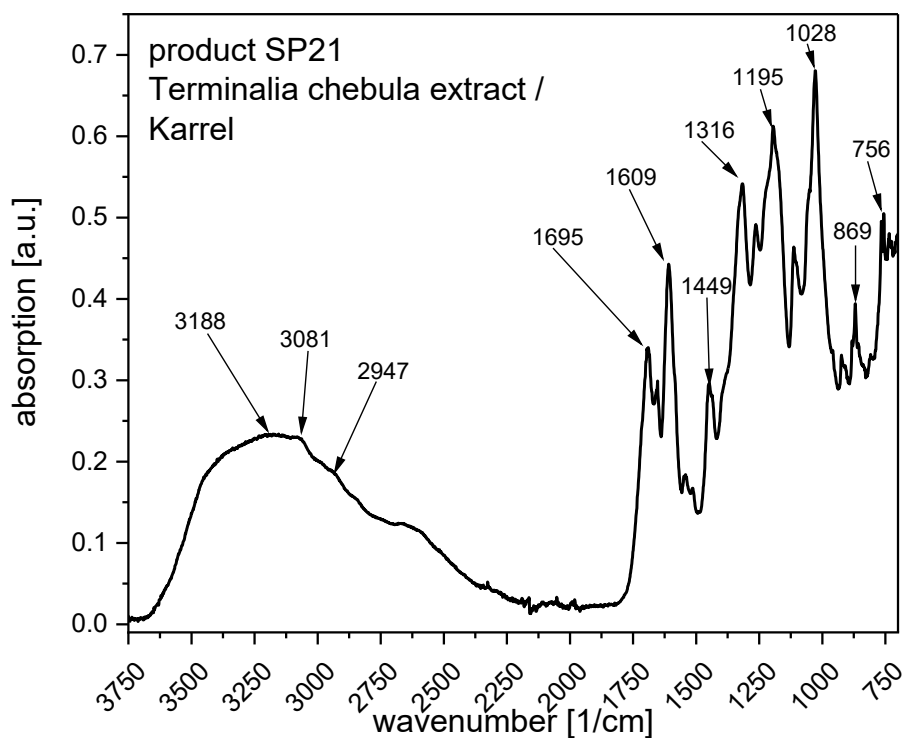


Fig. 24 IR-spectrum recorded from the natural dye product: Karrel.

The color product SP22 is distributed under the name “extract mixture green” and is produced as a mixture from different plant extracts [79]. The exact plant origin and chemical composition are not disclosed by the supplier. According to supplier information the coloration is in the range of petrol to olive. After application on textile fabrics this dye product leads to strong and brilliant green coloration. However, the light fastness of the achieved coloration is low. The IR spectrum of product SP22 exhibits several signals which can be set in relation to typical vibrations and functional units (Figure 25). The broad signal at  $3295\text{ cm}^{-1}$  is related to O-H stretching vibrations of hydroxy units. The average signal at  $2930\text{ cm}^{-1}$  is assigned to C-H stretching vibrations of aliphatic units. The signal at  $1723\text{ cm}^{-1}$  is assigned to C=O stretching vibrations of an ester group, while the signal at  $1603\text{ cm}^{-1}$  is assigned to a C=O stretching vibration of a keto unit. The signal at  $1445\text{ cm}^{-1}$  is probable related to deformation vibrations of  $\text{CH}_2$  and  $\text{CH}_2$  units. The strongest signal at  $1043\text{ cm}^{-1}$  is probable caused by stretching vibrations of C-O single bonds from hydroxy or ester groups.

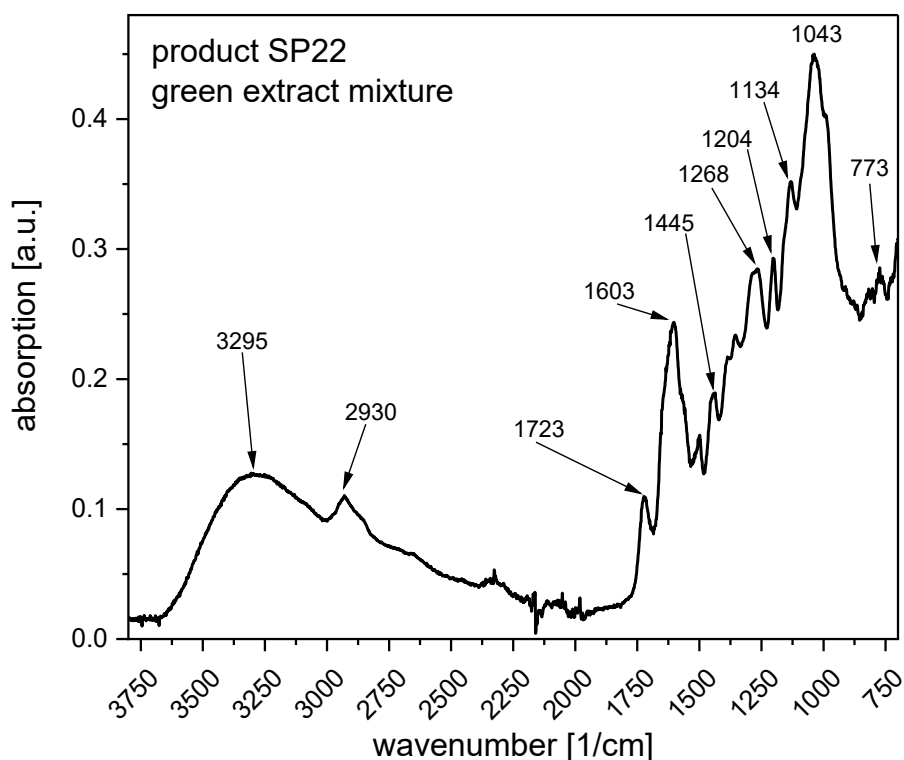


Fig. 25 IR-spectrum recorded from the natural dye product: green extract mixture.

An extract from sea buckthorn (*Hippophae rhamnoides*) leaves is supplied with product SP23. According to the supplier, with this extract a range of coloration from yellow and grey to black can be reached. As one main color component of sea buckthorn Quercetin is mentioned (compared the chemical structure in Figure 26) [1]. The chemical structure of Quercetin is quite similar to the structure of Butein (compare Figures 21 and 26).

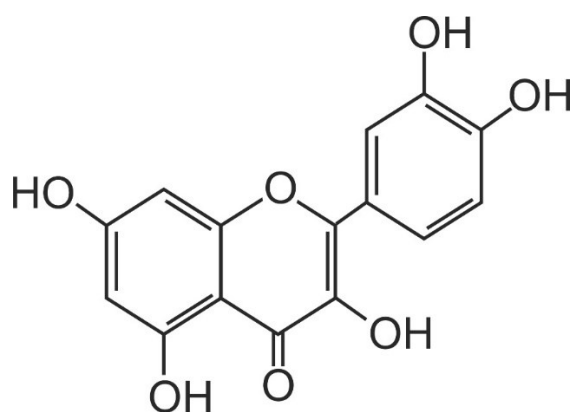


Fig. 26 Chemical structure of Quercetin as color component of sea buckthorn.

The IR spectrum of the extract powder from sea buckthorn leaves is given in Figure 27. The signals in the IR spectrum can be partly discussed in comparison to the chemical structure of Quercetin but several signals are caused by by-components in the extract. The broad signal at around  $3267\text{ cm}^{-1}$  is assigned to O-H stretching vibrations of hydroxy groups. The medium signal at  $2930\text{ cm}^{-1}$  is related to C-H stretching

vibration of aliphatic units. However, the molecular structure of Quercetin does not exhibit aliphatic units, so this signal is obviously related to the presence of by-components in the color extract. Analogously the signal at  $1718\text{ cm}^{-1}$  is related to C=O stretching vibrations from ester groups. As the molecule Quercetin does not contain any ester group, this IR signal is as well caused by a by-component in the extract. The clear signal at  $1606\text{ cm}^{-1}$  can be assigned to C=O stretching vibration of keto groups, which are part of the Quercetin molecule. The strongest signal at  $1019\text{ cm}^{-1}$  is related to C-O stretching vibrations of the hydroxy units bonded to the chromophore.

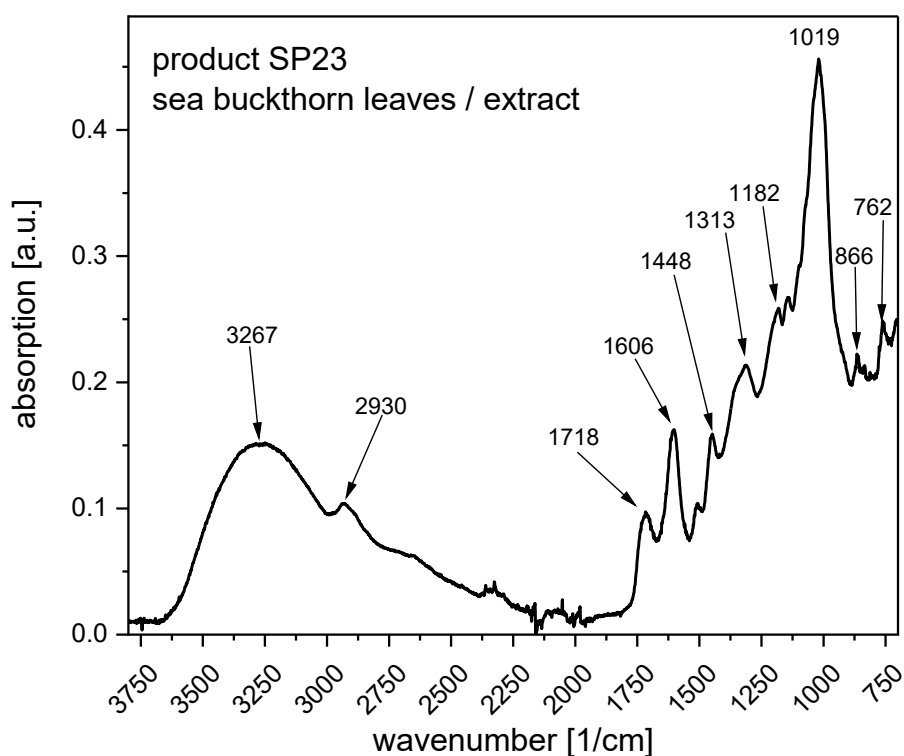


Fig. 27 IR-spectrum recorded from an extract from sea buckthorn leaves.

The color product SP24 is offered as extract from Yellow berries (*Rhamnus oleoides, fructi immaturi*). The color is mentioned by the supplier to be in the range of yellow to olive. According to literature, yellow berries are dried berries from different type of Rhamnus plants and the extract is also named as C.I. Natural Yellow 13 [1]. In other sources for yellow berries the common name Persian berries is mentioned and dyeing in combination with a broad range of different metal mordant agent is reported [138]. As color components in Natural Yellow, several components are mentioned as kaempferol, rhamnocitrin, quercetin, rhamnetin, rhamnazin and emodin (C175640, C175670, C175690, C175685, C175650, C175430, C175700) [112].

The IR spectrum of the yellow berry extract product SP23 is presented in Figure 28. Several main signals can be identified and discussed related to vibrations and functional groups. The broad signal around  $3262\text{ cm}^{-1}$  is related to O-H stretching vibrations of hydroxy groups. Both signals at  $2930\text{ cm}^{-1}$  and  $2852\text{ cm}^{-1}$  are assigned to C-H stretching vibrations of aliphatic units. The medium signal at  $1734\text{ cm}^{-1}$  can be assigned to C=O stretching vibrations from ester groups. Both peaks at  $1653\text{ cm}^{-1}$  and  $1594\text{ cm}^{-1}$  are related to C=O stretching vibrations probable from different keto units with different availability for bonding of intramolecular hydrogen bridge bonds.

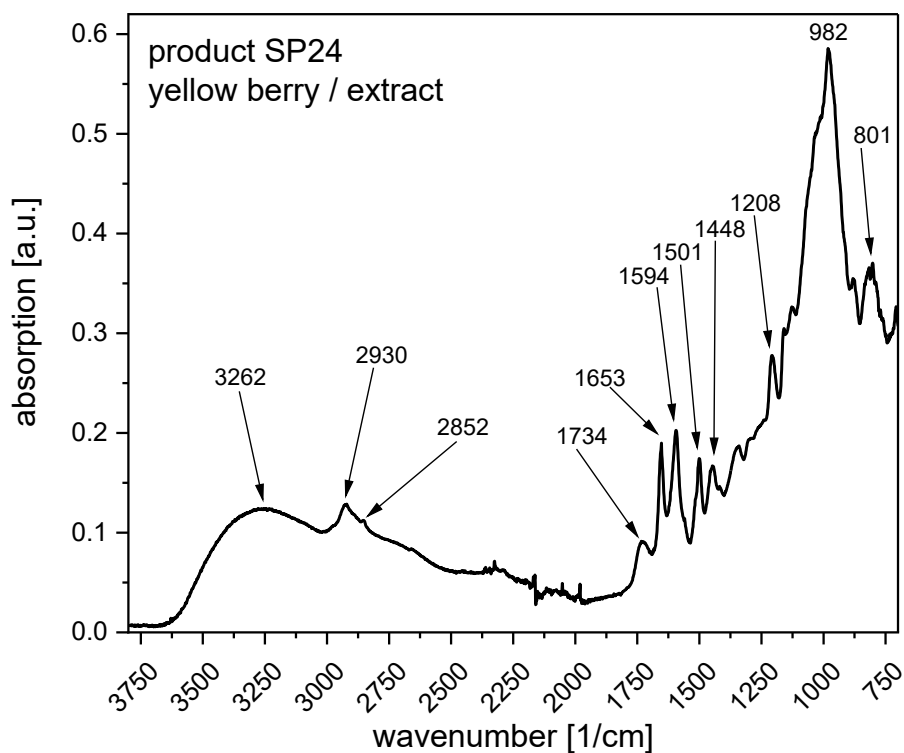


Fig. 28 IR-spectrum recorded from an extract from yellow berries.

### 3.4 Insect based dye materials

In the actual supplement, only two insect based natural dye products are presented. These products are from cochineal and lac dye. Both products are supplied by two different companies. The product SP25 is supplied a cochineal extract powder. In contrast, the product SP26 is distributed under the name LACROT (LacRed) as part of a ready-made recipe for home dyeing purposes.

IR spectra of cochineal products and Carmine were reported in the earlier overview papers intensively [42]. In comparison to these earlier reported spectroscopic data, the IR spectrum of the cochineal extract product SP25 is clearly different (Figure 29). These differences might be explained by the presence of several by-products as results from the extraction process. In any case, it can be stated that the clearly identification of cochineal extracts by IR spectroscopy is limited.

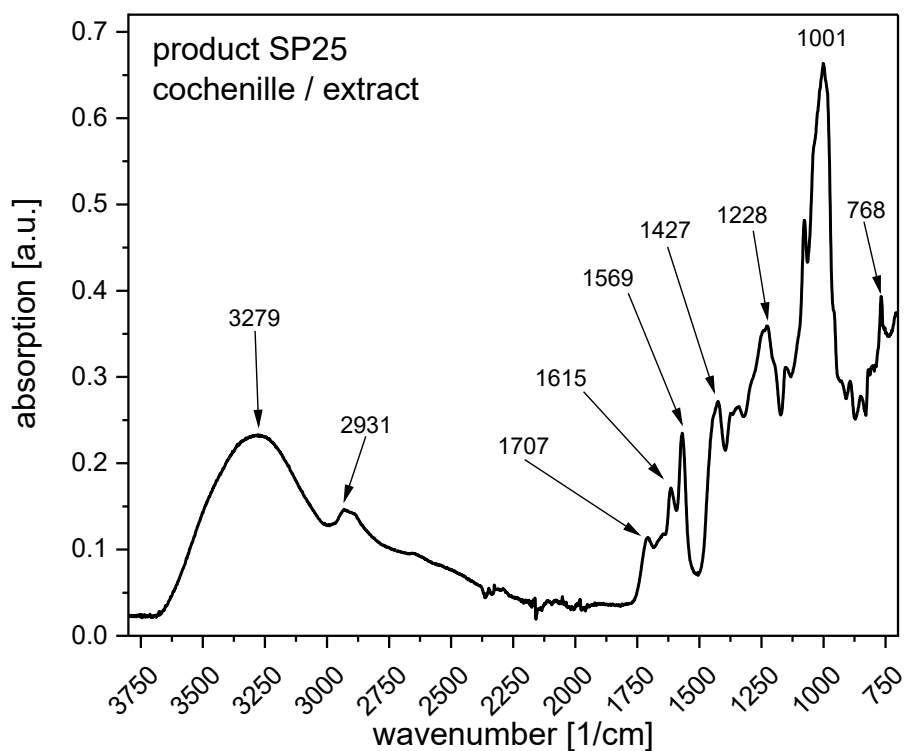


Fig. 29 IR-spectrum recorded from an extract from cochineal.

IR spectra of several lac dye products were reported in the earlier overview papers intensively [42]. In comparison to these earlier reported spectroscopic data, the IR spectrum of the product SP26 / LacRot is clearly different (Figure 30). These differences might be explained by the presence of several by-products as results from the extraction process. In any case, it can be stated that the clearly identification of lac dye products by IR spectroscopy is limited.

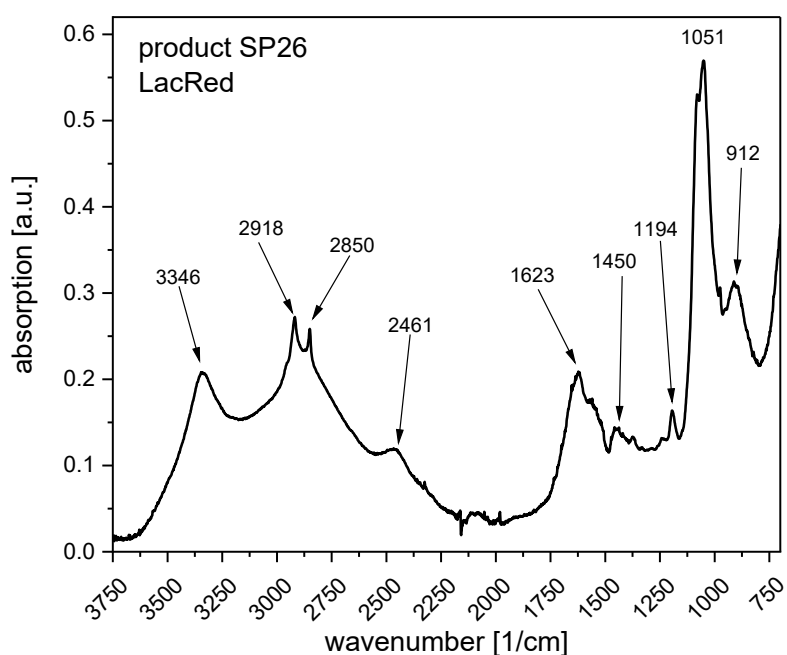


Fig. 30 IR-spectrum recorded from an extract from the product LacRot (lac dye red).

### 3.5 Asphalt

The product SP27 is a black powder and distributed under the name Asphalt for the purposes of wood treatment and coloration [84]. Asphalt is also named as C.I. Natural Black 6. It is described as natural product from raw oil reacted with oxygen from air used as color in paintings and soluble in hydrophobic solvents [1]. Usually asphalt types are distinguished between natural asphalt and artificial asphalt. Natural asphalt is described as the result of oxidation and polymerization of petroleum in interaction with microorganism [139]. Asphalt can be described as natural color product, because of its origin from a naturally occurring material. However, it is not from sustainable resources as plant or animal based color products. In fact, asphalt can be seen similar to different minerals which are supplied as natural color pigments [8, 140].

The IR spectrum of the Asphalt powder (product SP27) is presented in Figure 31. The signals in this IR spectrum can be assigned to several vibrations and related functional groups which are present in this mixture of organic compounds. The strongest peaks at  $2921\text{ cm}^{-1}$  and  $2854\text{ cm}^{-1}$  are assigned to C-H stretching vibrations of aliphatic units. The very small signal at  $3475\text{ cm}^{-1}$  can be related to O-H stretching vibration from a carboxylic acid. The medium signal at  $1601\text{ cm}^{-1}$  with shoulder at  $1654\text{ cm}^{-1}$  is related to C=O stretching vibration of oxidized carbon component, as functional groups e.g. keto or carboxylic acid. The strong peaks at  $1456\text{ cm}^{-1}$  and  $1375\text{ cm}^{-1}$  are related to different C-H deformation vibrations from aliphatic units  $\text{CH}_2$ . The weak signal at  $1032\text{ cm}^{-1}$  can be set in relation to C-O or C=S stretching vibration [45, 141].

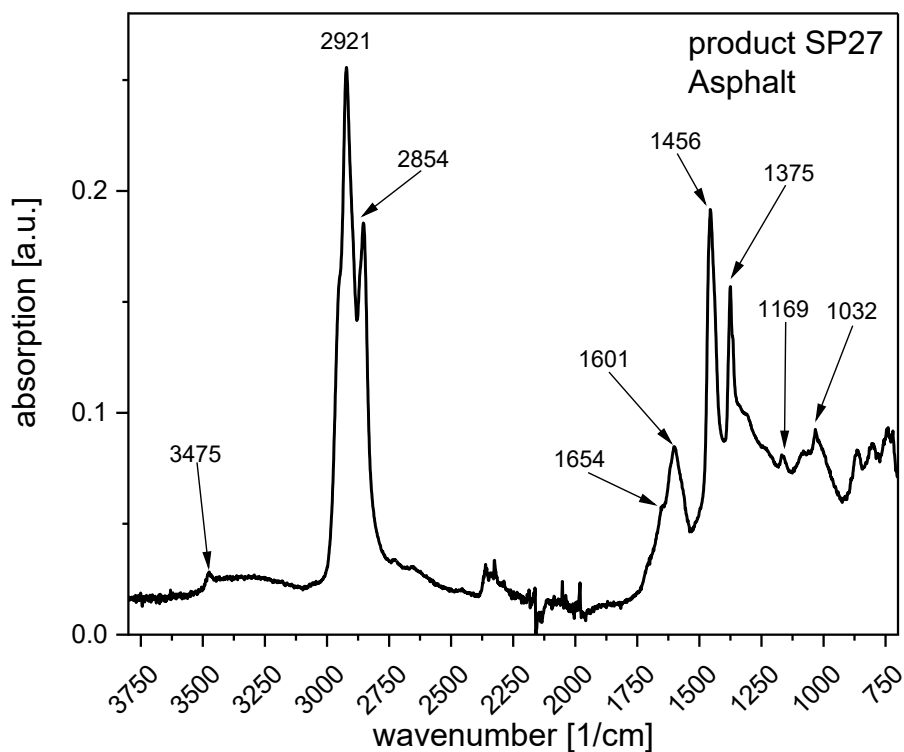


Fig. 31 IR-spectrum recorded from Asphalt powder.

### 3.6 Maya pigments

Maya pigments are built up by a combination of layered silicates with organic dyes. As organic dyes mainly indigo and indigo derivatives are used [87, 142]. Due to the incorporation of the organic dye into an inorganic matrix, an enhanced color stability against different influences results [143]. In the current supplement five different Maya pigments are reported which are supplied by the same company. The products MA1 to MA4 exhibit different types of blue coloration, while the product MA5 is a red pigment. The pigment product MA1 is named Maya blue and is built up according to supplier information by a magnesium aluminum layered silicate containing as organic dye the indigo derivative thioindigo. Thioindigo (Vat Red 41) is a synthetic dye, where the imide groups in the indigo molecule is replaced by a Sulphur atom (Figure 32) [144, 145]. Microscopic images of this pigment powder recorded in different magnifications are shown exemplarily in Figure 33. The silicate structure is here seen. In contrast, the product MA2 (Maya blue light) is built up by the organic dye indigo with magnesium aluminum layered silicate [89-92].

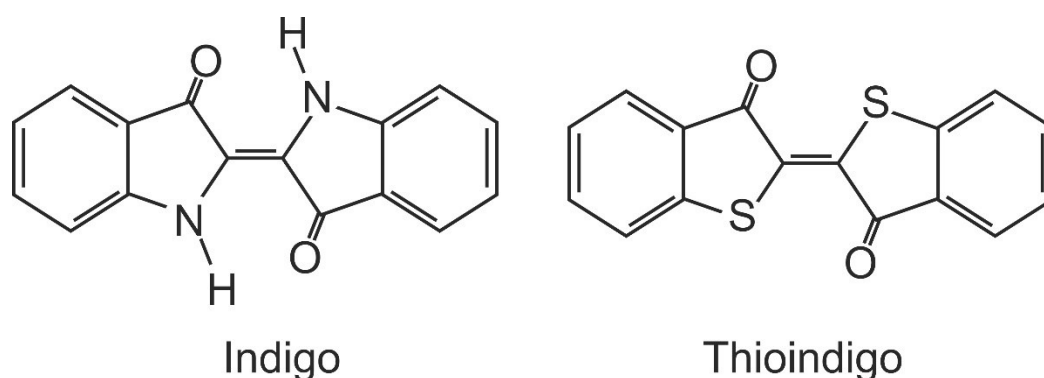


Fig. 32 Chemical structures of Indigo and Thioindigo.

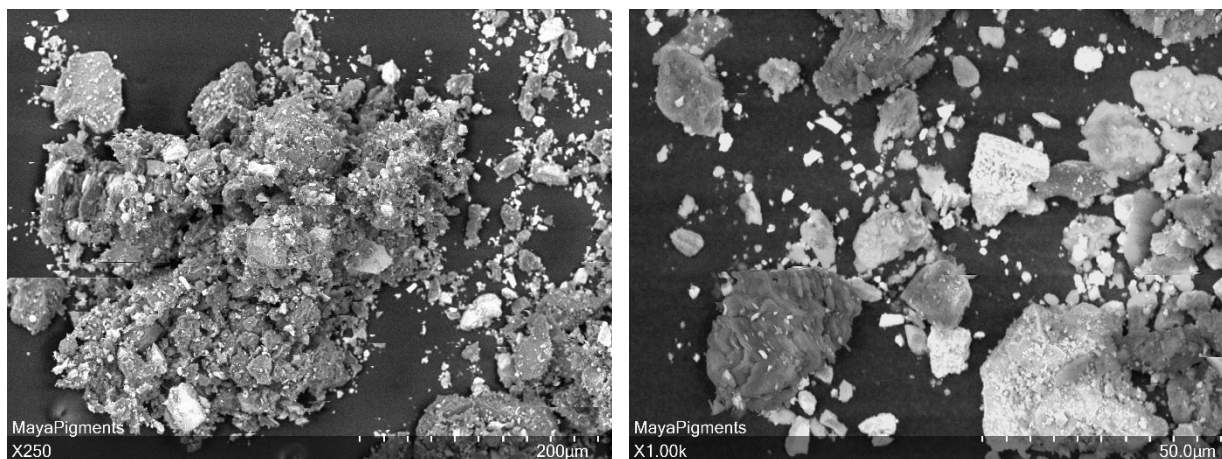


Fig. 33 SEM images of pigment Maya Blue, recorded in different magnifications.

The IR spectra of Maya pigments are the results of vibration of two components, the inorganic matrix and the embedded organic dye molecules (Figures 34 to 37). All IR spectra of actually considered Maya pigment products exhibit several weak and broad signals in the range from  $3300\text{ cm}^{-1}$  to  $3700\text{ cm}^{-1}$ , probable related to O-H stretching vibration of bonded water or N-H stretching vibration from embedded indigo molecules [85]. Further all IR spectra exhibit a strong signal in the range of  $955\text{ cm}^{-1}$  to  $983\text{ cm}^{-1}$ , which is probable related to a combination of Si-O and Al-O stretching vibrations from the inorganic matrix material. The weak signal at around  $1625\text{ cm}^{-1}$  can be assigned to the C=O stretching vibration of the keto

unit in the indigo molecule [40, 85]. Only, the IR spectrum of Maya Blue product MA1 exhibits a clear signal at  $1419\text{ cm}^{-1}$  (Figure 34). This signal might correspond to the presence of thioindigo in this pigment.

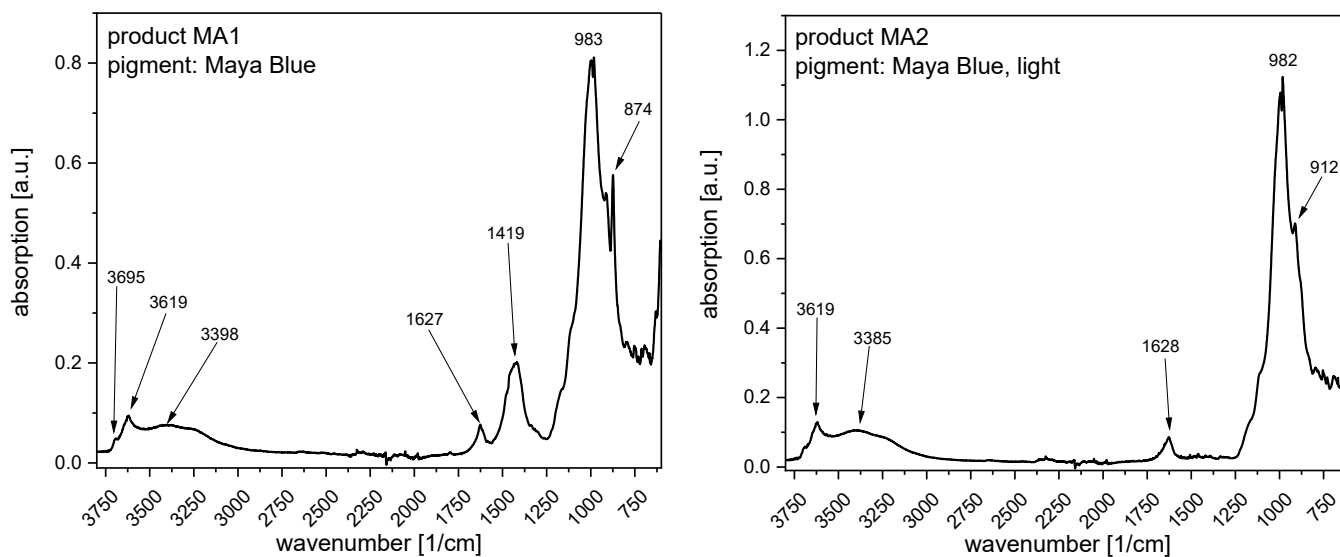


Fig. 34 IR-spectra recorded from two different types of Maya Blue pigment products.

According to supplier information, the product MA3 (Maya grey-blue B) is an indigo containing lithium magnesium sodium layered silicate, while the product MA4 (Maya deep blue) is an indigo containing magnesium aluminum layered silicate [89-92]. The IR spectra of these both products exhibit the main signals as the other Maya pigment products (Figure 35). The weak signal at  $1462\text{ cm}^{-1}$  can be assigned to a combined vibration of C-C stretching from aromatic C6 rings and C-H out-of-plane vibration of the embedded indigo molecules [40]. Product MA3 exhibits an additional weak signal at  $1706\text{ cm}^{-1}$  which is probably related to a C=O stretching vibration from a functional group different from indigo.

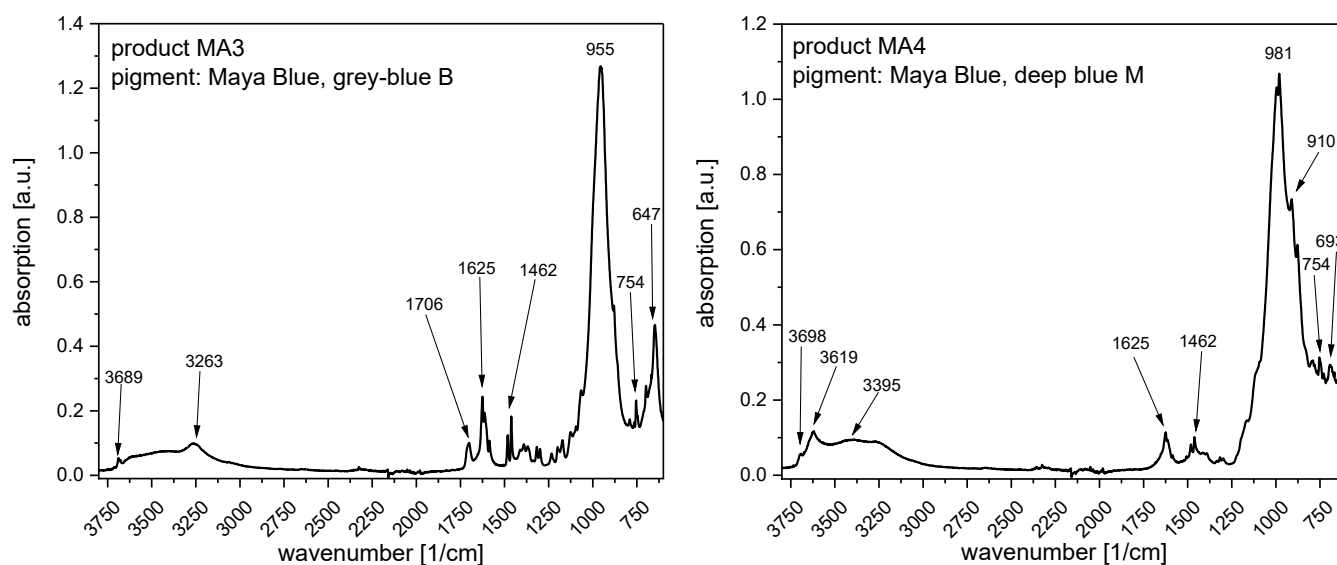


Fig. 35 IR-spectra recorded from two different types of Maya Blue pigment products.

The pigment MA5 (Maya red) is of red coloration and by this different to the other four products mentioned above. SEM images recorded from the powder of the pigment Maya red are shown in Figure 36 in different magnifications. According to the supplier, pigment MA5 is described as a PR 224 containing magnesium aluminum layered silicate and produced as absorption pigment from the mineral Attapulgite with PR 224 [webgae]. Attapulgite is a natural clay mineral [146]. PR 224 is probable CI Pigment Red 224, which is a synthetic red perylene pigment [147].

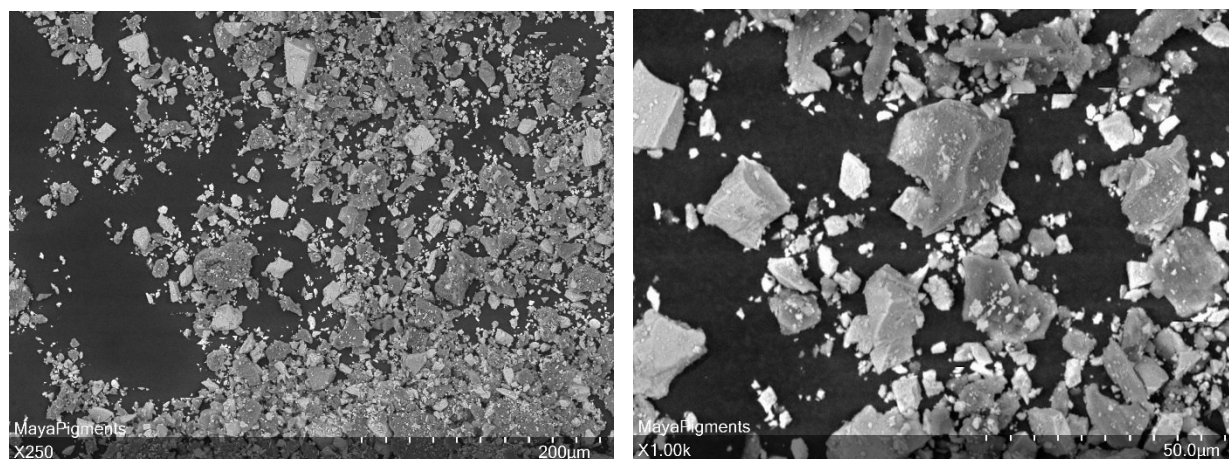


Fig. 36 SEM images of pigment Maya Red, recorded in different magnifications.

The chemical structures of Perylene and Pigment Red 224 are shown in Figure 37. Perylene  $C_{20}H_{12}$  is built by four aromatic six ring units. Pigment Red 224 (Perylene tetracarboxylic dianhydride)  $C_{24}H_8O_6$  contains two anhydride units attached to the Perylene structure. Pigment Red 224 is also used as vat dye [148].

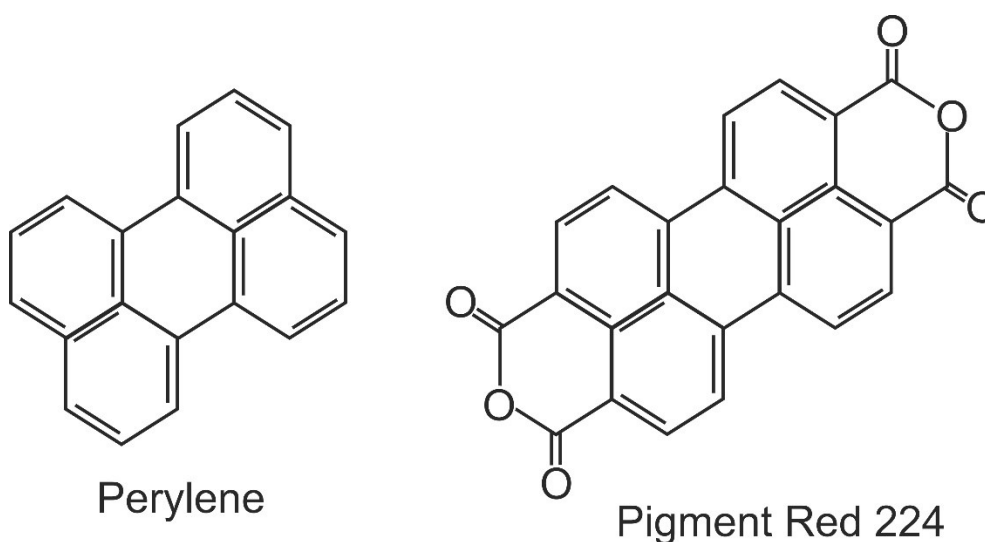


Fig. 37 Chemical structures of Perylene and Pigment Red 224.

The IR spectrum of the Maya red pigment product MA5 is presented in Figure 38. This spectrum is quite similar to the IR Spectrum of the product MA2 (Maya blue light). The weak signal at  $1635\text{ cm}^{-1}$  is probable related to the  $C=O$  stretching vibrations from the anhydride units of Pigment Red 224.

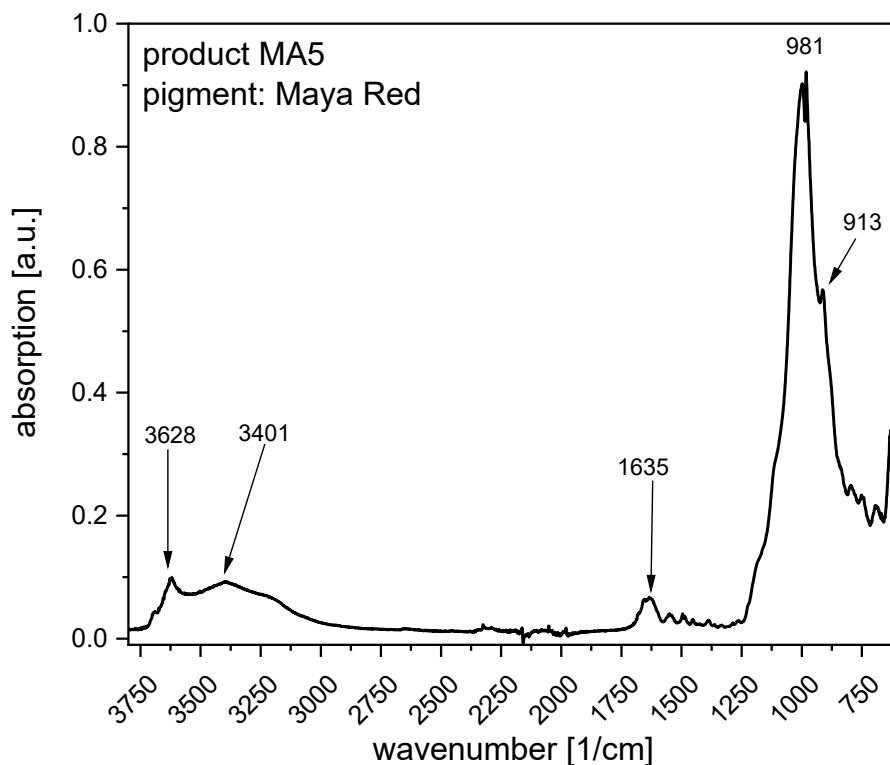


Fig. 38 IR-spectrum recorded from the pigment product Maya Red.

### 3.7 Natural mordanting agents

For application of natural dyes in most cases the use of mordanting agents is necessary to reach a sufficient coloration depth and good fastness properties [1, 94, 94]. As effective mordants, often water soluble metal salts are used [94]. However, due to environmental concerns, the use of metal containing components might be avoided. As alternative, bio-based natural mordanting agents are discussed, evaluated and used [149, 150]. Following some typically and commercially available natural mordanting agents are presented together with their IR spectra. The four products M1 to M4 are tannin products from different sources. The product M1 is supplied by the chemical supplier Carl Roth GmbH under the name tannic acid with a purity of > 86%. The chemical structure of tannic acid ( $M_w=1701$  g/mol;  $C_{76}H_{52}O_{46}$ ) is presented in Figure 39. Tannic acid contains a sugar unit as core to which several units are connected by ester bonds. The attached units are partly built up by gallic acid.

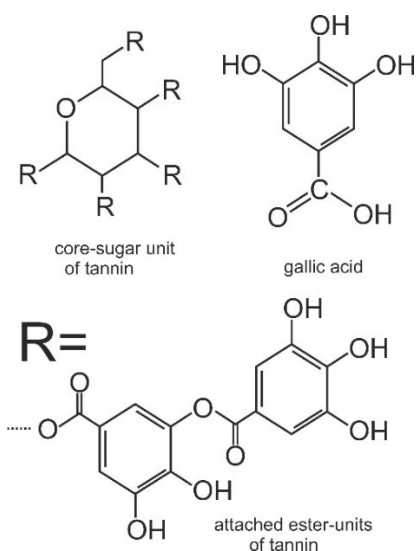


Fig. 39 Chemical structures of tannin and gallic acid.

The IR spectra of the four tannin products are presented in Figures 40 to 42. For tannin products M1, M2 and M4, the IR spectra are nearly identical even if they are from different suppliers, they are quite identical from their chemical composition. Main signals of the IR spectra are further discussed according to the chemical structure of tannin acid. The broad signal at around  $3300\text{ cm}^{-1}$  is related to the O-H stretching vibration of the hydroxy groups in tannic acid. Surprisingly, no signal around  $2990\text{ cm}^{-1}$  is determined, which would be related to C-H stretching vibrations. The both signals at around  $1700\text{ cm}^{-1}$  and  $1608\text{ cm}^{-1}$  are assigned to C=O stretching vibrations. Both signals may be related to ester groups, which might be different due to different chemical surroundings with and without the possibility to form intramolecular hydrogen bridge bonds to neighboring hydroxy groups.

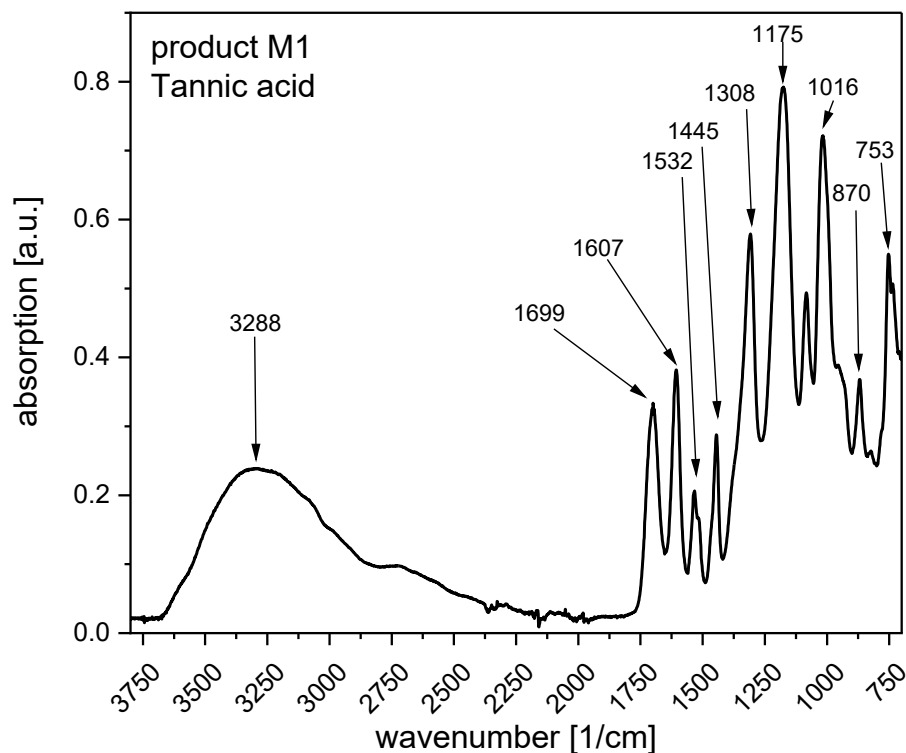


Fig. 40 IR-spectrum recorded from a Tannin acid product.

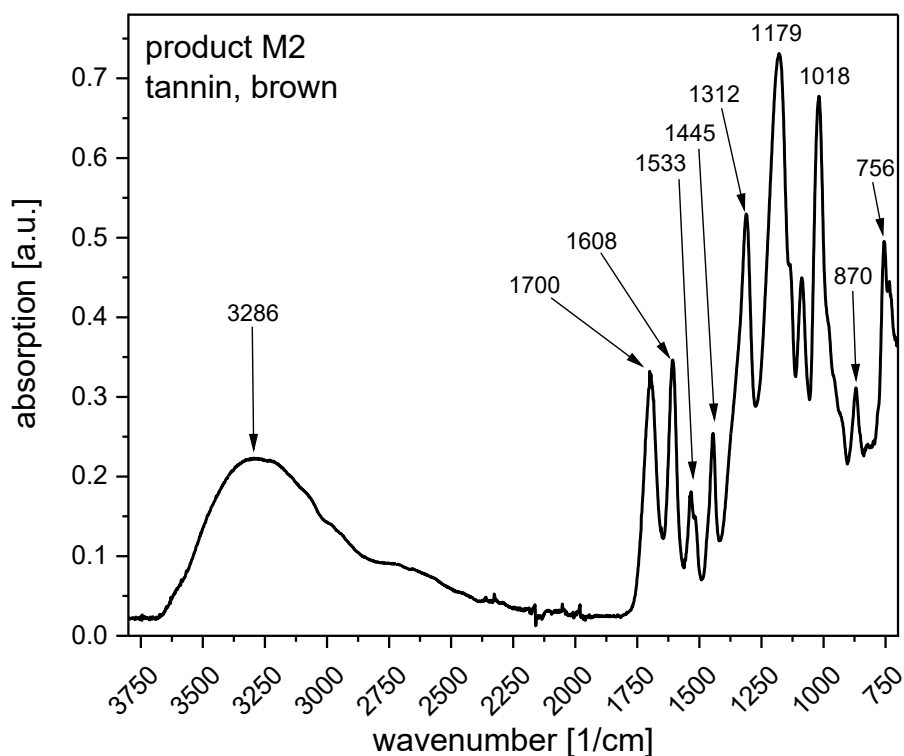


Fig. 41 IR-spectrum recorded from a Tannin product.

Tannin products M3 and M4 are supplied by the same German company and distinguished in the declaration as “tannin” and “tannin light” [83, 84]. The product M4 (tannin light) exhibits a quite similar IR spectrum as the two products M1 and M2. In contrast, the IR spectrum of product M3 exhibit clear differences (Figure 42). At around  $2940\text{ cm}^{-1}$  a clear shoulder signal is determined which is assigned to C-H stretching vibrations of aliphatic units. Surprisingly no clear signal around  $1700\text{ cm}^{-1}$  is determined, which would be clearly related to C=O stretching vibration of the ester groups from tannic acid.

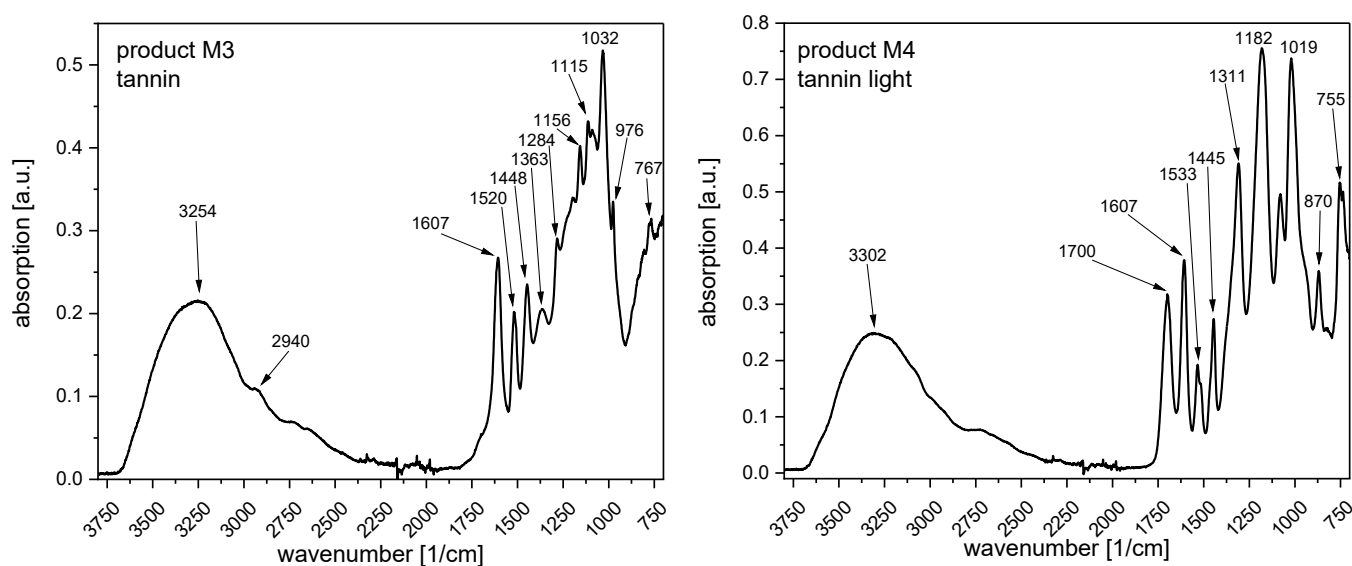


Fig. 42 IR-spectra of two different tannin products from the same supplier.

As alternative to tannin products, two suppliers offer a product named tara powder as natural bio-based mordanting agent. The IR spectra of both tara powders are presented in Figure 43. The IR-spectra of the two tara products are quite identical to each other and in main features also similar to the IR-spectra of the tannins. The main difference compared to the IR spectra of tannin, is the appearance of two signals around  $2852\text{ cm}^{-1}$  and  $2925\text{ cm}^{-1}$  for the tara products. These both signals are assigned to C-H stretching vibrations from aliphatic units and might be caused by by-components in these natural products. However, it can be stated that the tara products are certain chemically identical to the tannin products.

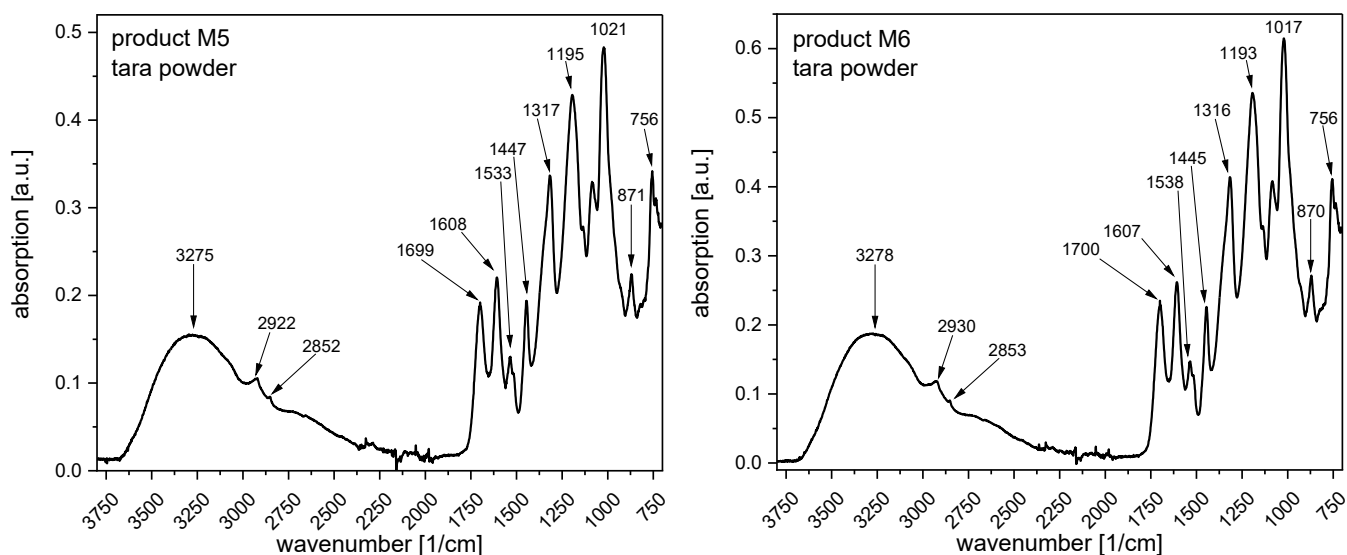


Fig. 43 IR-spectra of two different tara powder products from two different suppliers.

Similar to the two tara powder products, also the powder Myrobalane is offered as natural based mordanting agent. The IR spectrum of this product M7 is presented in Figure 44 and shows a high similarity to the IR spectra of the tara products. Obviously these three products are of quite similar composition even if they have different names and are supplied by different companies.

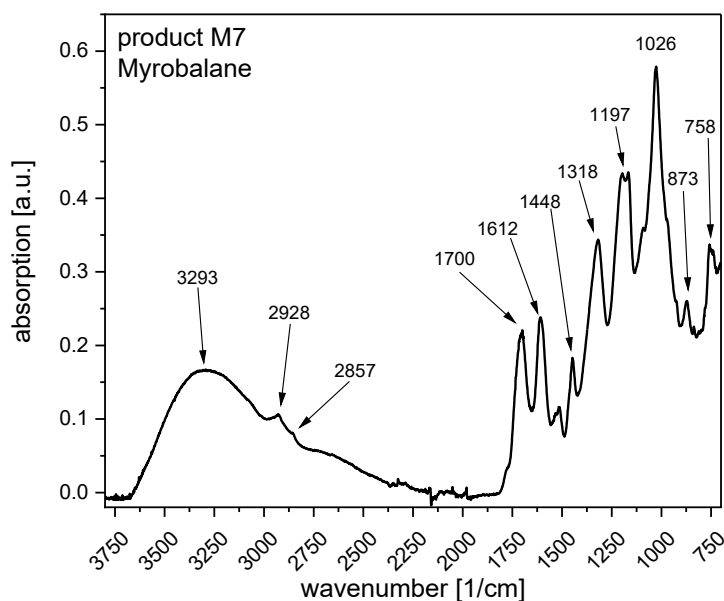


Fig. 44 IR-spectrum recorded from Myrobalane.

## 4 Conclusions

Altogether, 27 natural color components, 5 Maya pigments and 7 bio-based natural mordanting agents are presented and discussed together with their IR spectra. By this a comprehensive view on a broad range of different commercially available natural products is given. It is shown that the IR spectroscopy can be a helpful tool for product identification. However, due to the occurrence of by-components in natural products, this analytical method is also limited. Nevertheless, the current paper offers a broad overview on very different natural color materials and their application on textiles. The supported IR spectroscopic data are useful for material identification and helpful for people working in the fields of textile modification, dyeing, material research and quality control. This overview is also a valuable tool for educational purposes guiding interested persons into the field of natural dyes and infrared spectroscopy.

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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. All discussed color components, products and chemicals are purchased on the legal market. No donation was given or offered by the supplying companies.

## Conflicts of Interest

The author declares no conflict of interest.

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