

# Overview on natural dyes and their IR-spectra – Part IV: Dyes originated from insects

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## INFO

CDATP, ISSN 2701-939X  
Peer reviewed article  
2024, Vol. 5, No. 1, pp. 107-119  
DOI 10.25367/cdatp.2024.5.p107-119  
Received: 20 February 2024  
Accepted: 28 April 2024  
Available online: 16 August 2024

## ABSTRACT

*Natural dyes were important in the past of textile industry. Due to actual trends towards bio-based and sustainable materials, also natural dyes might face a revival as part of a new sustainable clothing industry. According to this statement, the current paper gives an overview on selected natural dyes which are originated from insects. Altogether three different types of dyes are discussed gained from three different types of lice – cochineal, lac and kermes lice. Additional to an overview on dye properties and applications, a special view is done on the infrared spectra recorded from those dye materials. The IR-spectra are discussed in relation to chemical composition and structure of dye materials. They are a helpful tool for people working in the field of dye identification and quality control. The intension of this review series is to support an overview on natural dyes and their IR-spectra.*

## Keywords

natural dye,  
infrared spectroscopy,  
insect based dye,  
carmines,  
cochineal,  
lac dye  
kermes

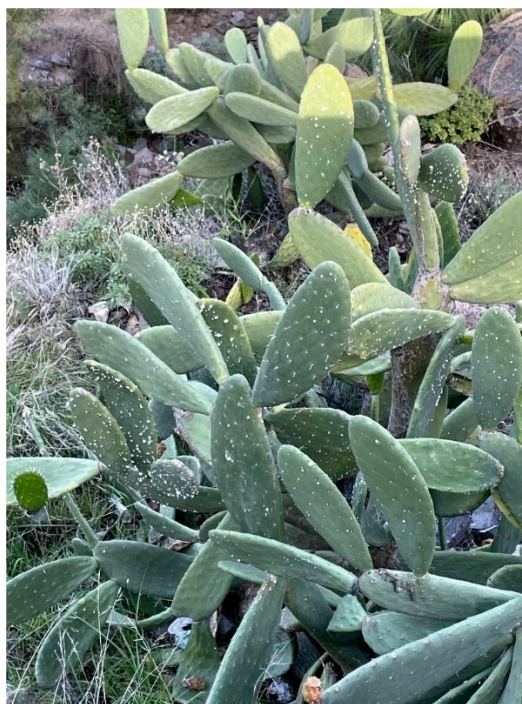
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## 1 Introduction

Natural dyes are nowadays often used in food industry to realize colorful food and drinks [1-4]. In this field of application, insect based red colored dyes play a significant role [5-9]. Beside food products, natural dyes are also used for cosmetics [10-13]. Insect based dyes are often related to different species of lice living in tropical and subtropical areas on cactus plants [14-16]. In fact, the lice can be seen as parasites infecting the cactus plant [17]. Figure 1 exhibits a photograph from such cactus infected with lice taken December 2022 on the island of Tenerife (Spain). The white spots on the cactus leaves are caused by the lice.



*Fig. 1 Photograph of cactus infected with lice – Tenerife (Spain).*

Before invention of synthetic dyes, those insect based natural dyes played a significant role in dyeing of textiles and clothes [18-20]. Nowadays, several investigations in literature are reported, as the dyeing of a broad range of different fiber types with cochineal dye [21-24]. However, the relevance in textile industry for this type of natural dye is relatively low in our times. Nevertheless, due to the actual trend to realize sustainable and fully bio-based products, natural dyes have a potential for a revival in future as part of fully bio-based and ecofriendly textile products [25-27]. Natural dyes might be seen as chance to establish sustainable and eco-friendly processes, what could be also a chance for the revival of traditional dyeing techniques [28,29]. With this background, the current paper gives an overview on natural dye materials gained from three different types of lice – cochineal, lac and kermes lice. This current overview paper is part of a paper series discussing different categories of natural dyes, available dye products and their infrared spectra. In addition to a view on the origin and some applications of the dyes, an intensive view is done on the infrared spectra of dye materials. Here a discussion of spectral signals is done in respect to dye composition, the chemical structure of the dye and of possible by-products. The infrared spectroscopy is a well-established analytical method in organic chemistry [30-32]. By this, it is a suitable tool for identification and investigation of textile fiber materials [33-36]. Also, IR spectroscopic investigations on leather and artificial leather materials are reported [37]. A summary of infrared spectra of prominent natural dyes is reported by Priyadharisini et al. However, these authors did not report IR spectra of natural dyes originated from insects [38]. Further, IR spectroscopy is used as tool to identify natural dyes on historical textile materials and fibers [39, 40].

In the current paper, a discussion of the spectra related to the chemical structure is supported. For this, infrared spectra are recorded from different dye products exclusively for this overview paper. The dye products investigated are gained from different suppliers as extracts, powders or even the complete lice. The three investigated dyes – cochineal, lac and kermes lice – exhibit similarities in their chemical structures. Their main chromophore is based on an anthraquinone unit. Further, the molecular structures of all three dyes contain at least one carboxylic acid as functional group. For this, they are soluble in water and exhibit a negative charge after deprotonation. These dyes can be seen as a kind of natural anionic dye or acid dye, which may especially be used for dyeing of protein based natural fibers like silk or wool. In addition to the overview on the dye products, one significant aim of this paper is to support IR-spectroscopic data and their discussion according to composition and chemical structure.

To support an intensive view on broad range of natural dyes, the current overview article is the number four in a series of natural dyes and their IR-spectra. The first three articles are related to plant-based

dyes, indigo and dyes from wooden materials [41-43]. The idea of this review series is to support an overview on natural dyes and their IR-spectra, which is actually rarely available in public literature. The supported data are helpful for people working in fields of dye identification and quality control.

## 2 Materials and Methods

### 2.1 Dye materials

Different natural dye stuff containing products are investigated in the current study. These products are originated from three different insects – cochineal, lac lice and kermes lice. They are gained as powder or dried insect pieces from five different suppliers – Kremer Pigmente GmbH (Aichstetten, Germany), Pflanzenfärbeshop (Hückelhoven-Baal, Germany), Das Wollschaf (Zweibrücken, Germany), Carl Roth GmbH (Karlsruhe, Germany) and AMA Herbal (India). All investigated materials are listed in Table 1 together with the related supplier and references of the suppliers giving additional information on application recommendation, dyeing recipes and safety issues.

Table 1. Investigated natural dye products.

No.	Content	Form	Supplier	References
A1	Carmine / C.I. 75470	Powder	Roth	[44]
A2	Cochineal / extract	Powder	Pflanzenfärbeshop	[45]
A3	Cochineal	Powder	Pflanzenfärbeshop	[46]
A4	Cochineal	Piece, dried	Wollschaf	[47]
A5	Lac dye	Powder	Wollschaf	[48]
A6	Lac dye	Powder	Kremer	[49]
A7	Lac dye	Powder	Pflanzenfärbeshop	[50]
A8	Lac dye, kerria iacca	Powder	Amaherbal	[51]
A9	Kermes lice	Piece, dried	Kremer	[52]

### 2.2 Analytic Methods

The infrared spectra (IR spectra) of investigated dye products are recorded with a FT-IR spectrometer IRTracer-100 from Shimadzu (Japan). This IR spectrometer is equipped with 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 done by using a TM4000Plus Tabletop Microscope from Hitachi (Japan).

## 3 Discussion of Dyestuffs

### 3.1 Cochineal

The main colored component in cochineal is carmine (also named carminic acid),  $C_{22}H_{20}O_{13}$  (Natural Red 4, C.I. 75470) with the molecular weight 492 g/mol [6,15,53]. Please, compare the chemical structure of carmine in Figure 2. Several extraction methods are described to gain carminic acid from the cochineal [54]. The chemical structure of carmine exhibits, in addition to the anthraquinone chromophore and the carboxylic acid group, also an attached glucose unit. Dissolved in water, the carboxylic acid group is deprotonated and this dye molecule is negatively charged. For this, carmine can be also taken as hydrophilic anionic dye stuff with especially high affinity to protein-based fibers like wool or silk [55-57]. As well the dyeing of polyamide fabrics is done with cochineal and here an intensive discussion of different mordanting processes is reported by Nateri et al. [58]. For cochineal application on cotton, beside conventional metal mordanting also mordanting procedures based on different approaches are reported – such as the application of chitosan or dendrimers [53,59-62]. Further unconventional methods are the use of microwave heating and the sol-gel technology [63,64]. Attempts are made to combine the dyeing of textiles with cochineal with textile functionalization, e.g. for UV protection or antimicrobial purposes [65,66].

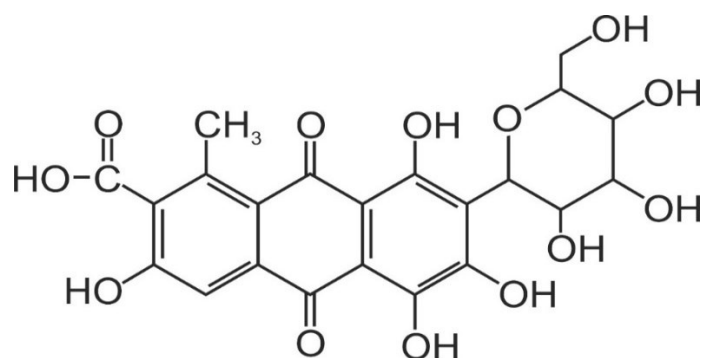


Fig. 2 Chemical structure of the dye carmine as main colored component in cochineal.

For IR-spectroscopic investigations, different types of cochineal products – powder, extract and the complete dried animal – gained from different suppliers are considered. Figure 3 presents a magnified photograph of dried cochineal lice (product A4). Those lice exhibit a dark red coloration and have a size of only few millimeters. A more detailed view on the surface of cochineal lice is supported with images recorded with scanning electron microscopy (Figures 4 and 5).

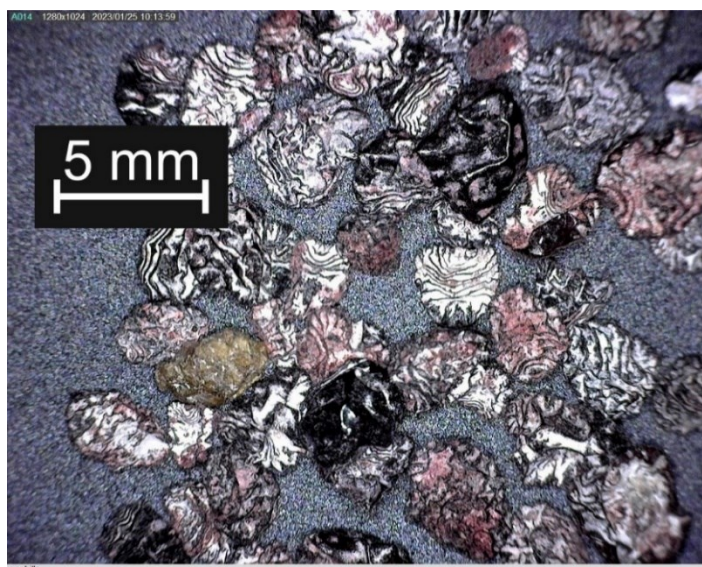


Fig. 3 Cochineal (product A4). Photographic image with higher magnification.

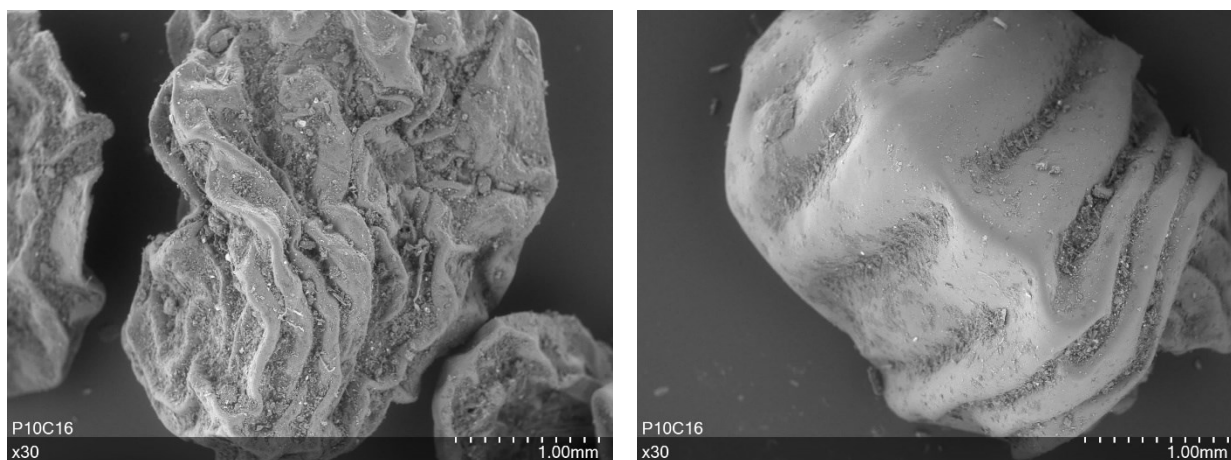


Fig. 4 Cochineal (product A4). SEM images taken in low magnification from different sides of cochineal lice.



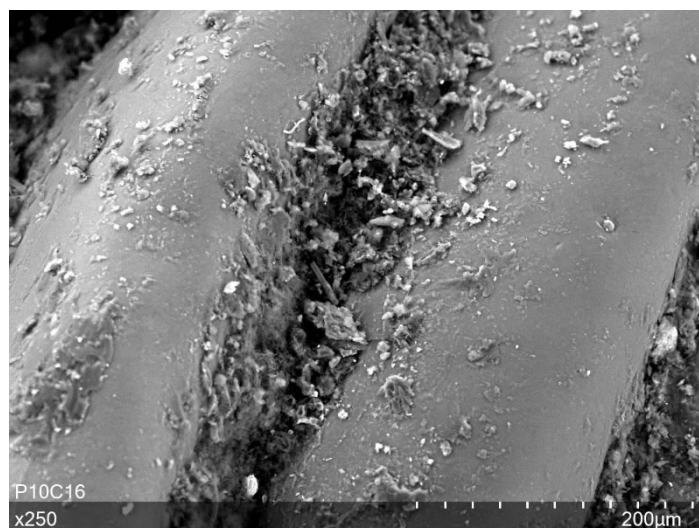


Fig. 5 Cochineal (product A4). SEM image taken in higher magnification.

Figure 6 supports a comparison of IR-spectra of four different cochineal products containing different purity and dye content. Product A1 is gained as carmine from the chemical supplier Carl Roth GmbH and is supposed to be the product with the highest dye content in the current investigation. Product A2 is as well a dye extract from cochineal. However, the method of extraction is not disclosed by the supplier. Due to the water solubility of the dye carmine, it can be expected that the extraction is done with water based solutions at elevated temperatures. The products A3 and A4 are from dried cochineal lice as powder or as complete dried animal, respectively.

The IR spectrum of product A1 carmine can be set in good accordance to the chemical structure of the dye carmine (compare Fig. 2). The broad signal at  $3318\text{ cm}^{-1}$  is related to the O-H stretching vibration of the many hydroxy groups in this molecule. The clear signals at  $2928$  and  $2853\text{ cm}^{-1}$  are related to C-H stretching vibration from aliphatic units, as  $-\text{CH}_3$ . [30, 31]. The signal at  $2928\text{ cm}^{-1}$  is related to the asymmetric vibration and the signal at  $2853\text{ cm}^{-1}$  is related to symmetric vibration [31]. The molecular structure of carmine exhibits only one C-H bond directly attached to the aromatic chromophore. A C-H stretching vibration related to this bond would be expected to appear in a spectral area around  $3050\text{ cm}^{-1}$ . However, in this area no clear signal is detected, probably because of low content of this C-H unit in the large molecular structure of the dye.

The chemical structure of carmine exhibits mainly two types of C=O double bonds – from carboxylic acid and from the keto groups as part of the anthraquinone chromophore. All three C=O units are part of the aromatic system and are neighbored by hydroxy groups. By formation of intramolecular hydrogen bridges to neighboring hydroxy groups, the C=O bonds are weakened and the position of related IR-signal would be shifted to lower wavenumbers. For this, the signal at  $1636\text{ cm}^{-1}$  is related to C=O stretching vibration and can be assigned to the carboxylic acid and keto groups [67]. The strongest peaks at  $1287\text{ cm}^{-1}$  and  $1248\text{ cm}^{-1}$  can be assigned to C-O stretching vibrations of hydroxy groups bonded to the aromatic unit or at the glucose unit. The peak at  $1564\text{ cm}^{-1}$  can be assigned to overtone and combination of the aromatic ring system. The peak at  $1408\text{ cm}^{-1}$  is probably assigned to C=C stretching vibration [31].

The IR spectrum of cochineal extract (product A2) is nearly similar to the spectrum of product A1 and is also in good accordance to an IR spectrum of cochineal dye reported in the literature [59]. All main signals appear. However, the intensity of the determined signals in the fingerprint area differs. The IR spectra of the two cochineal products A3 and A4 are mainly similar to each other but clearly different to the spectra of the two extract products A1 and A2. Of course, the complete cochineal lice contain many other compounds additional to carmine, so related to this mixture of different components, the related peaks show different intensity and also new peaks are determined.

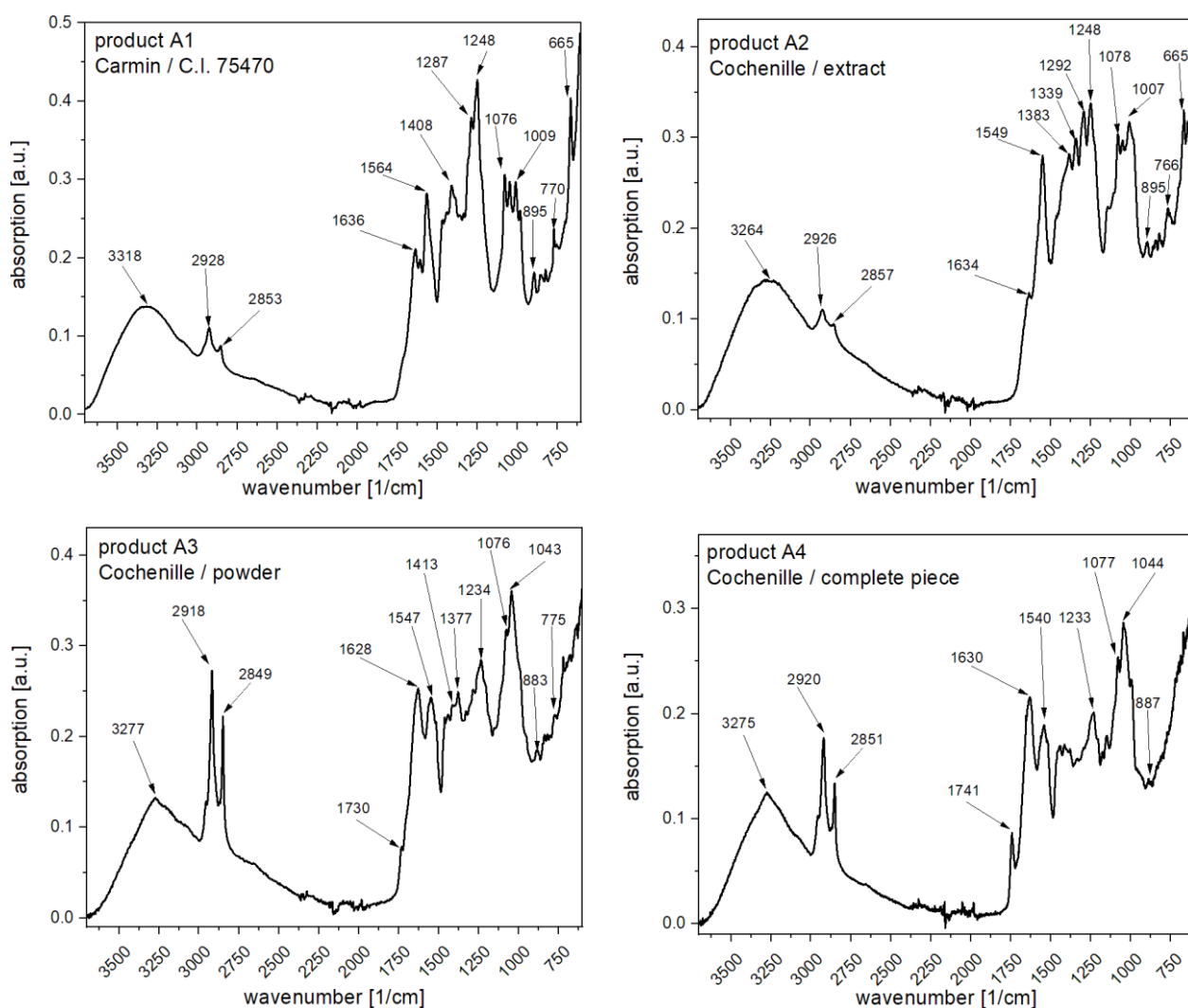


Fig. 6 Comparison of IR spectra of several cochineal / carmine products. Shown are the products A1 to A4 (compare Table 1).

The peak at  $1730\text{ cm}^{-1}$  (product A3) or  $1741\text{ cm}^{-1}$  (product A4) can be assigned to C=O stretching vibration of an ester group. This signal is probably related to the presence of an ester from fatty acids, which is insoluble in water and for this not dissolved in the extraction process. In literature this signal is also reported but mentioned to be stronger compared to the infrared spectra of product A3 and A4 [68]. Remarkable is also the strong intensity of the two clear peaks at around  $2920\text{ cm}^{-1}$  and  $2850\text{ cm}^{-1}$  related to C-H stretching vibration, which are also reported for the dye cochineal in literature [68]. These strong signals are probably caused by components in the cochineal lice containing high amounts of  $-\text{CH}_2-$  and  $-\text{CH}_3$  units, as e.g. proteins, chitin or fatty acids.

### 3.2 Lac dye

Lac dye is gained by extraction of lac lice [15]. An interesting approach for extraction of lac dye is using microwave devices [69,70]. The main colored component in lac dye is the laccaic acid (Natural Red 25) [14]. The chemical structure of laccaic acid is presented in Figure 7. The main chromophore of laccaic acid is an anthraquinone unit. Two carboxylic acid units are attached to this chromophore, making this dye soluble in water and lead to a negative net charge of the dye after solving in water. This natural dye is an anionic dye with exhibits by this a good affinity to protein fibers like wool, silk or regenerated protein fibers [71,72]. However, the properties of lac dye application can be significantly improved by mordanting [73]. As well applications on cotton, linen, viscose and synthetic fibers are reported for lac dye [74-77]. Three different types of laccaic acid (A, B and C) are distinguished, which differing only in the type of group R (Figure 7). Additionally, in literature also further types of laccaic acid (types D and E) are mentioned [78,79]. Lac dye is found in ancient manuscripts from 12<sup>th</sup> to 15<sup>th</sup> there it was used for colorful

illustrations [79]. Similarly, the use of lac dye and lac extract for the coloration of leather used for shoes is reported [80,81].

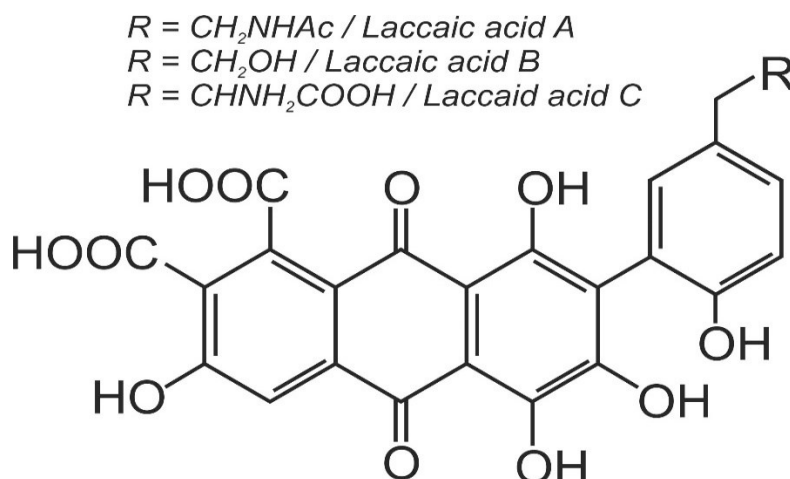


Fig. 7 Chemical structure of laccaic acids A, B and C as main colored compounds of Lac Dye.

In the actual overview, four different types of lac dye products supported by four different suppliers are investigated (products A5 to A8). The IR spectra of four investigated lac dye products (A5 to A8) are nearly similar and can be set in accordance to the chemical structure of laccaic acid (Figure 7 and 8).

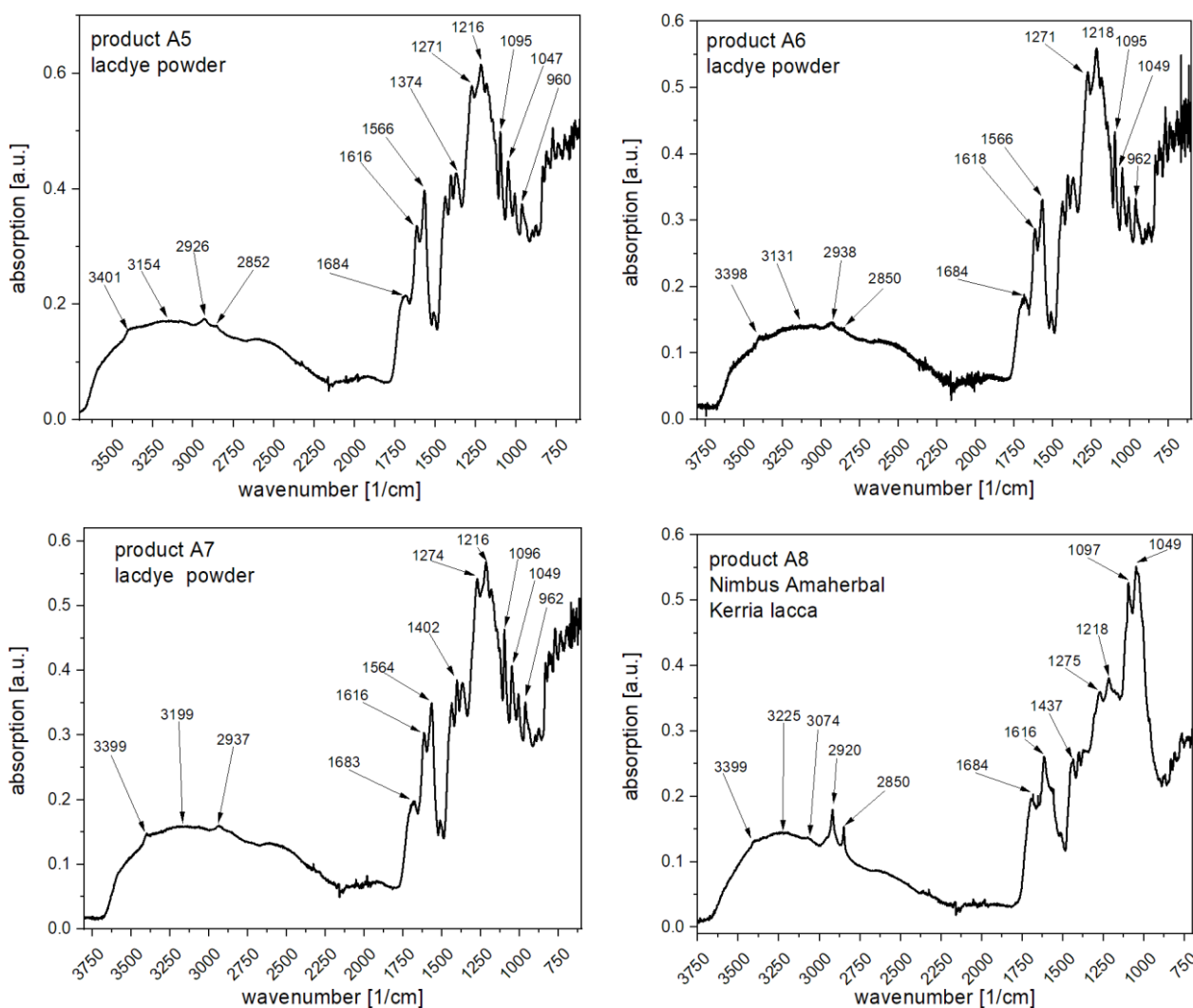


Fig. 8 Comparison of IR spectra of four Lac dye products. Shown are the products A5 to A8 (compare Table 1).

The very broad signal around  $3150\text{ cm}^{-1}$  can be assigned to O-H stretching vibration of hydroxy groups and N-H stretching vibration of amino groups. The small signal at around  $3400\text{ cm}^{-1}$  can be assigned to O-H stretching vibration of the carboxylic acid [30]. The small signals at  $2930\text{ cm}^{-1}$  and  $2850\text{ cm}^{-1}$  are related to C-H stretching vibrations of the aliphatic  $-\text{CH}_2-$  unit at the group R (compare Figure 7) [30]. The three signals at  $1684\text{ cm}^{-1}$ ,  $1616\text{ cm}^{-1}$  and  $1566\text{ cm}^{-1}$  can be assigned to C=O stretching vibrations from different functional groups, as the carboxylic acid neighboring the hydroxy group, the carboxylic acid neighboring the keto group and the both keto groups neighboring the hydroxy groups in the anthraquinone chromophore. Keto groups neighboring the hydroxy group form to them hydrogen bridges, which are weakening the C=O bond, so the IR-signal is shifted to lower wavenumber at around  $1566\text{ cm}^{-1}$  [67]. Compared to infrared spectra of laccaic acid and lac dye reported in literature, the spectra of the products A5 to A8 exhibit significant differences [68]. The infrared spectrum from literature exhibits as well the very broad signal reaching from  $2800\text{ cm}^{-1}$  to  $3600\text{ cm}^{-1}$ . However, in literature also the presence of a signal at  $1715\text{ cm}^{-1}$  is reported for lac dye [68]. Such a signal is not determined for any of the actually investigated lac dye products. This reported signal can be assigned to a C=O stretching vibration of an ester group and is probably caused by a by-product which is part of the measured sample reported in literature but not in the currently investigated dye products.

### 3.3 Kermes lice

The main colored component in kermes is kermesic acid (C.I. Natural Red 3) (chemical structure in Figure 9) [9]. The chemical structure of kermesic acid is based on an anthraquinone unit with attached carboxylic acid and four hydroxy groups. The deprotonation of the acid group leads to the formation of a negatively charged molecule with certain water solubility. It can be seen as anionic component with affinity to protein fibers as wool or silk. The application on those types of fibers is usually done in combination with mordants as e.g. aluminum or iron salts [82]. For historical textiles from wool and silk the presence of kermes dye together with cochineal dye is discussed [83]. Another dye constituent isolated from kermes lice is flavokermesic acid, which contains one hydroxy group less compared to kermesic acid [84].

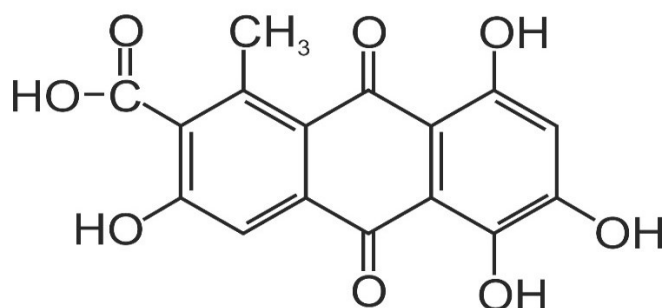


Fig. 9 Chemical structure of the dye kermesic acid as main colored component in kermes.

In the current overview, only one product of kermes is considered – a dried piece of kermes lice. Magnified photographs of the investigated kermes lice are presented in Figure 10. The dried kermes lice exhibit a red/brown coloration and a size of around 5 mm. Compared to cochineal lice, the kermes lice have diameters which are nearly double in size. A highly magnified microscopic image of a kermes lice is taken by scanning electron microscopy and presented in Figure 11.





Fig. 10 Kermes lice (product A9). Photographic image with higher magnification.

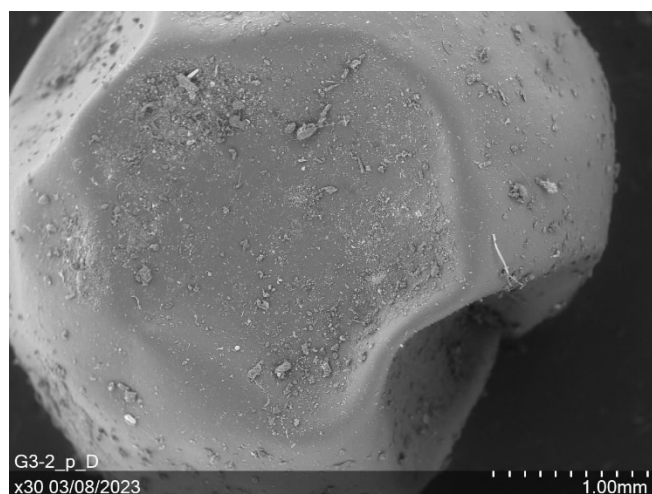


Fig. 11 SEM image of kermes lice (product A9).

The IR spectrum of the dried kermes lice (product A9) is presented in Figure 12. Additional to kermesic acid, the dried kermes lice contain a broad range of different components, as e.g. chitin, carbohydrates, proteins and fatty acids. Nevertheless, a discussion of the IR-spectrum with respect to the chemical structure can be made. Remarkable is that the broad signal from  $3200\text{ cm}^{-1}$  to  $3450\text{ cm}^{-1}$  exhibits several weak maxima. Here, the maximum at  $3436\text{ cm}^{-1}$  can be assigned to O-H stretching vibrations of carboxylic acid [31]. The other weak signals in that range are probably related to O-H stretching vibration of hydroxy groups and N-H stretching vibration from amine and amide groups. The weak signal at  $3064\text{ cm}^{-1}$  is assigned to C-H stretching vibration of hydrogen atoms bond to an aromatic ring system. The three signals at  $2955\text{ cm}^{-1}$ ,  $2919\text{ cm}^{-1}$  and  $2850\text{ cm}^{-1}$  are assigned to C-H stretching vibrations form aliphatic groups  $-\text{CH}_2-$  or  $-\text{CH}_3$ . Remarkable is the strength of the signals at  $2929\text{ cm}^{-1}$  and  $2850\text{ cm}^{-1}$ , which is a certain similarity to the IR spectra of cochineal products. Three signals at  $1781\text{ cm}^{-1}$ ,  $1700\text{ cm}^{-1}$  and  $1653\text{ cm}^{-1}$  are related to C=O stretching vibration of different functional groups, as carboxylic acid group from kermesic acid. However, this can be as well related to other component in the lice as chitin or proteins. Probably the signal at  $1653\text{ cm}^{-1}$  is related to a C=O stretching vibration from an amide group, which is present in chitin and proteins [31].

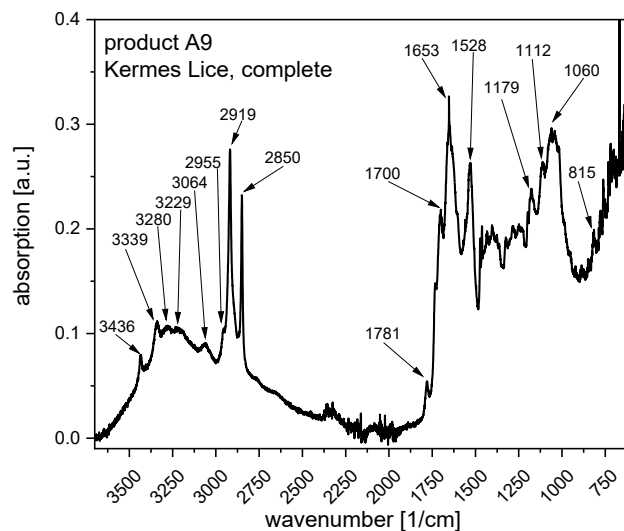


Fig. 12 IR spectrum of kermes lice product A9.

## 4 Conclusions

Several types of insect based natural dyes are available on the market. In current overview three categories of products gained from cochineal, lac and kermes lice are considered. These red colored dyes are all anionic dyes and exhibit an anthraquinone unit as chromophore. Their application on textiles is usually done in combination with mordanting agents. From those dye products recorded IR-spectra exhibit typical signals which are in accordance to the main colored component of the dye material. Nevertheless, the presence of other components in these natural products have to be considered. By comparison of the fingerprint area, the different dye products can be distinguished and identified. According to this, the current overview can be taken as helpful tool for dye identification and tasks in quality control.

## Acknowledgements

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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