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Overview on natural dyes and their IR-spectra - Part VI: Algae based pigments

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ABSTRACT

Algae are water based species with different colorations as green or red. Color pigments and dye extracts gained from algae are offered as natural based plant dyes. Algae based materials are usually rarely considered for dyeing of textiles in comparison to synthetic or conventional plant based dyes. Nevertheless, algae are a sustainable and natural source of bio-based substances and an increasing use might be a promising development in future. The current paper is the last one of a series of papers reporting on natural dyes and materials with special view on their infrared spectra. This last overview paper is dedicated to available special types of algae based natural pigments and related other algae and lichen based products which can be useful for coloration processes. Besides, general information on dye origin and possible application, especially infrared spectra of dye and reference materials are supported. The IR spectra are discussed in respect to sample composition and chemical structure. By this, the actual paper can be useful for persons searching for alternative coloration materials or working in the field of material analytics and spectroscopy.

Keywords
Algae,
Pigment,
Infrared spectroscopy,
Scanning electron microscopy,
Spirulina,
Chlorella,
Dulce,
Orcein

1 Introduction

Natural dyes are often categorized by their origin into plant based and animal based dye products [1-3]. In textile industry conventionally, synthetic dyes are used mainly because of color properties, fastness and cost reasons [3]. Nevertheless, in current days the discussion on use of natural dyes and pigments increases. This discussion is mainly based on a future proposed demand on sustainable processes and bio-based materials [4]. Additionally to the coloration of textiles, natural dyes can be used to implement antibacterial properties on textile materials to reach green functional textiles [5-7]. Other types of textile functionalization by dye applications are as well reported, a prominent example is the implementation of UV-protective properties with natural dyes [8, 9]. Algae are also living organism performing photosynthesis, so dyes and pigments from algae could be counted to natural dyes. However, conventional overviews on natural dyes often do not contain as well algae based products and their use as color pigments. By extraction of different types of algae a broad range of different types of natural dyes and pigments can be produced and their application for textile dyeing is discussed in scientific papers broadly [10-13]. Most algae based color products are used for food coloration and food products [14, 15]. Nevertheless, for industrial dyeing procedures of textiles algae based materials are rarely considered even if algae are fascinating sustainable natural materials and algae are even seen as a keystone for development of a new sustainable and blue economy [16]. For this, the significance of the current paper is also to improve the visibility of those algae materials in the field of textile applications. The actual overview paper is mainly related to algae based dye products and some algae reference materials. It is the sixth overview paper on natural dye products and their infrared spectra. The first five overview papers offer a broad overview on different plant based and insect based natural dyes [17-21]. The combined presentation of review information on algae materials and the presentation of measured infrared spectra, offers the advantage that not only literature based information is presented. Also, measurement data are provided which can be a helpful tool for material science, analytical investigation and quality control. In literature often, single IR spectra of medium quality and limited discussion are presented. With this background an aim of the current overview is to support a broad range of IR-spectra recorded from various products. The presented IR spectra are systematically presented and discussed in respect to chemical structures and composition of the investigated products. The infrared spectroscopy is a broadly used method in organic chemistry for analysis and identification of compounds [22-24]. It is as well used in polymer science and for identification of polymer containing materials as textiles [25-28]. Most algae exhibit green coloration and are often offered as food additives or food colorants [29-31]. Due to limited light fastness of green algae coloration, the use on textiles may be limited. The green colored component in algae is the Chlorophyll, which is the main plant dye responsible for photosynthesis. The chemical structure of Chlorophyll is presented in Figure 1. Chlorophyll can be also assorted to the category of metal complex dyes, because it contains a centered magnesium ion Mg^{2+} surrounded by four complex bonded nitrogen atoms [32]. Additionally to the green Chlorophyll, a broad range of different colored components can be extracted from different algae, as e.g. the red colored Astaxanthin [33-35]. Instead of using algae extracts for coloration, also complete algae material can be grinded and used as color pigments for textile prints [36, 37]. Prominent examples are here microalgae as Spirulina and Chlorella.

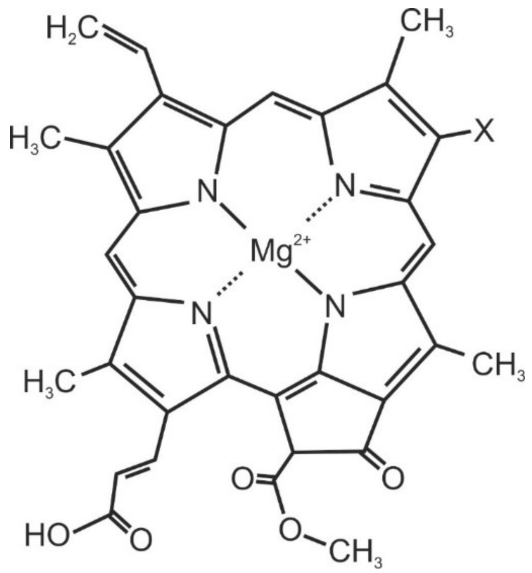

Fig. 1 Chemical structure of Chlorophyll C1 and C2 (C1: X = CH_2-CH_3 ; C2: X = CH=CH_2). The main green colored component in lgae.

2 Materials and Methods

2.1 Materials

For the actual overview, two algae based color pigments Spirulina Green and Spirulina Blue are considered. Both pigments are supplied by Kremer Pigment GmbH (Aichstetten, Germany). The pigment Spirulina Blue is an extract of the blue component from the Spirulina algae. As reference materials, two algae materials offered as food additives are investigated. These food additives are gained from the company Narayana Verlag GmbH (Kandern, Germany). The purpose of these food additives is not food coloration. They are dedicated as vegan source for protein and several essential minerals [38, 39]. As further algae based materials Irish Moss, Dulse red algae, Wakame algae and brown algae are considered. For comparison, also the dye Orcein as lichen based natural material is reported. The natural dye Orcein is supplied by Carl Roth GmbH (Karlsruhe, Germany). An overview on presented algae based materials is given in Table 1 together with the supplier and a reference from supplier leading to further product information like safety data or recommendation for application.

Table 1. Investigated algae based materials.

No.	Content	Form	Supplier	References
B1	Spirulina Green Pigment	powder	Kremer	[40]
B2	Spirulina Food Additive	powder	Narayana	[41]
B3	Chlorella Food Additive	powder	Narayana	[42]
B4	Spirulina Blue Pigment	powder	Kremer	[43]
B5	Irish Moss / Carageen moss	Dried plant	Kremer	[44]
B6	Dulse / red algae / food additive	Dried pieces	Narayana	[45]
B7	Wakame / food additive	Dried pieces	Narayana	[46]
B8	Brown algae / Laminaria digitata / Kelp / food additive	Dried powder	Nature Ingredients	[47]
B9	Orcein / Natural Red 28	powder	Carl Roth	[48]

2.2 Analytic Methods

The infrared spectra (IR spectra) of investigated algae materials and references are recorded with a FT-IR spectrometer IRTracer-100 from Shimadzu (Japan) using a Specac Golden Gate ATR unit. Scanning electron microscopy (SEM) is done by using TM4000Plus Tabletop microscope from Hitachi (Japan). Photographic images with enhanced magnification are taken by using a Dino-Lite Premier Digital Microscope (ANMO Electronics Corporation, Taiwan).

3 Discussion of Dyestuffs

3.1 Spirulina based pigments & Microalgae Materials

Spirulina algae materials are used as food and food additives to support a broad range of different essential minerals, vitamins and amino acids to the consumer [38, 39, 49]. The use of Spirulina platensis for dyeing of cotton fabrics is described. Here, recipes containing Spirulina in combination with aluminum sulfate as mordant are reported. Further components added are alginates and glycerin [37]. In this recipe the added alginates have also the purpose to act as binder for the color pigments on the textile surface. Advantageous is this approach, because with alginates as bio-based binder a completely biobased coloration recipe can be realized [37]. A microscopic image taken from the Spirulina green pigment (product BV1) is presented in Figure 2. This pigment is supplied as fine powder. The pigment particles are not separated from each other during sample preparation for taking the SEM image.

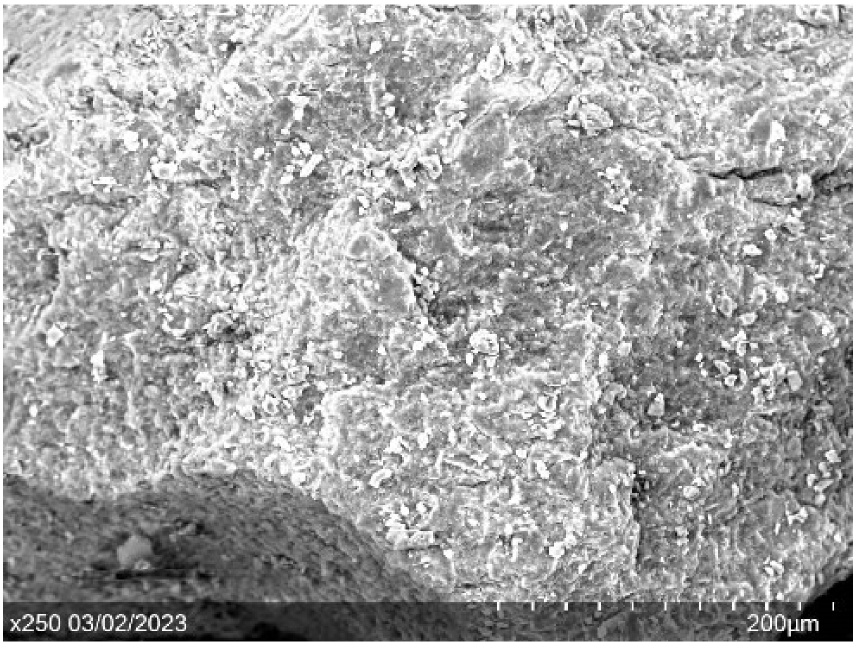

Fig. 2 SEM image of pigment powder from Spirulina green pigment (product B1).

According to supplier information Spirulina Blue contains Phycocyanin extract as blue pigment, 30% trehalose and 5% sodium citrate as stabilizers. The chemical structures of phycocyanin and trehalose are presented in Figures 3 and 4, respectively. The molecular structure of phycocyanin is built up by four heteroaromatic ring systems, which are conjugated with each other. Further, two carboxylic acid groups are present in this structure (Figure 3). However, these acid groups are not part of the conjugated system of the chromophore. The chemical structure of trehalose is built up by two connected sugar units (Figure 4). An excellent report on dyeing of wool and cotton fabric with phycocyanin under consideration of different mordanting agent is given by Moldovan et al. [50].

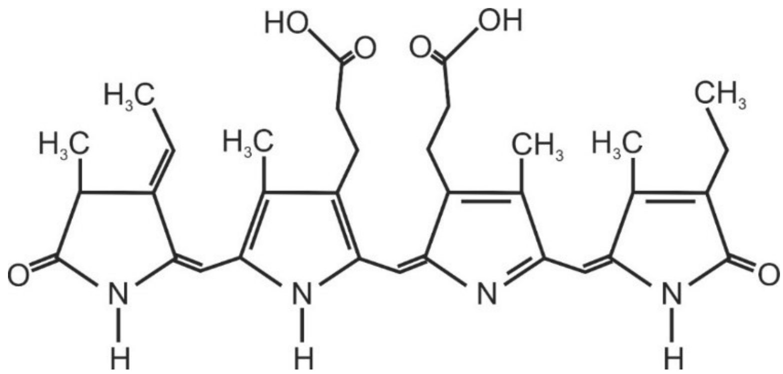

Fig. 3 Chemical structure of Phycocyanin.

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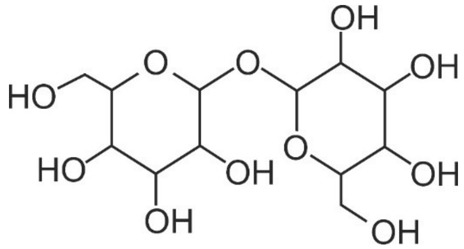

Fig. 4 Chemical structure of Trehalose.

Microscopic images of dry pigment Spirulina Blue are supported in Figure 5 for different magnifications. In contrast to Spirulina Green, the pigment particles of Spirulina Blue are separated during preparation for the microscopic measurements. For this, the pigment particles can be clearly identified and described. The shape of Spirulina Blue pigment particles can be described as spheres or broken spheres. The diameter of the spheres is between around 10 to 30 micrometers. The observed particle shape for Spirulina blue can be the result of a spray drying procedure for encapsulation of Spirulina extracts under presence of trehalose [51].

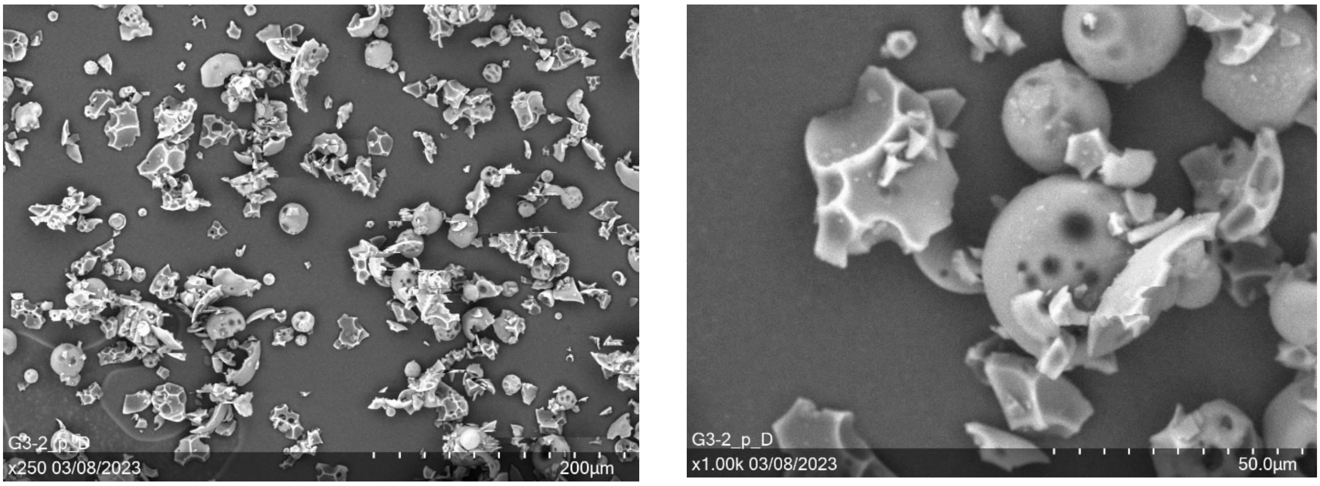

Fig. 5 SEM images of pigment powder from Spirulina blue pigment (product B4) – left: low magnification; right: high magnification.

The infrared spectrum recorded from the pigment Spirulina Green is presented in Figure 6. This measured IR spectrum is in quite good accordance to an infrared spectrum reported in literature. However, this IR spectrum from literature is recorded in transmission and the presented figure quality is medium [52]. Another transmission IR spectrum is reported in literature for Spirulina Fusiformis. Also, here a good similarity to the actually recorded IR spectrum can be stated even if the earlier reported IR spectrum is only published in medium image quality [53]. A good similarity is also given to an IR spectrum reported for Spirulina powder but the position of the main signal is shifted [54]. Spirulina as an alga is built up by several components like carbohydrates and proteins. For this, a discussion of the infrared spectrum related to the green dye Chlorophyll and its chemical structure is not possible. The broad signal at 3280 cm^{-1} is related to O-H and N-H stretching vibrations of hydroxy and amino groups, respectively. The weak signal at 3063 cm^{-1} can be either assigned to C-H stretching vibration of an aromatic system or an overtone vibration related to an amide group from protein [23]. The three signals at 2961 cm^{-1}, 2924 cm^{-1} and 2871 cm^{-1} are assigned to C-H stretching vibration of aliphatic units as $-CH_2-$ or $-CH_3$. The strongest peak at 1635 cm^{-1} is related to C=O stretching vibration from amide groups in proteins [22]. This signal is also named as Amide I [23]. Please, remark that the assignment of the signal around 1635 cm^{-1} is typical for C=O stretching vibration of amide groups in proteins, even if different groups discuss this signal for IR spectra recorded from Spirulina to different types of vibration [52, 53]. The strong signal at 1539 cm^{-1} can be assigned to the amide group from protein (signal Amide II) and is related to a combination of stretching vibration of the C-N bond with a deformation vibration of C-N-H [22]. The medium signal at 1453 cm^{-1} can be assigned to out-of-plane bending vibration of $-CH_3$ methyl groups [54]. The strong signal at around 1030 cm^{-1} can be assigned to C-O stretching vibrations.

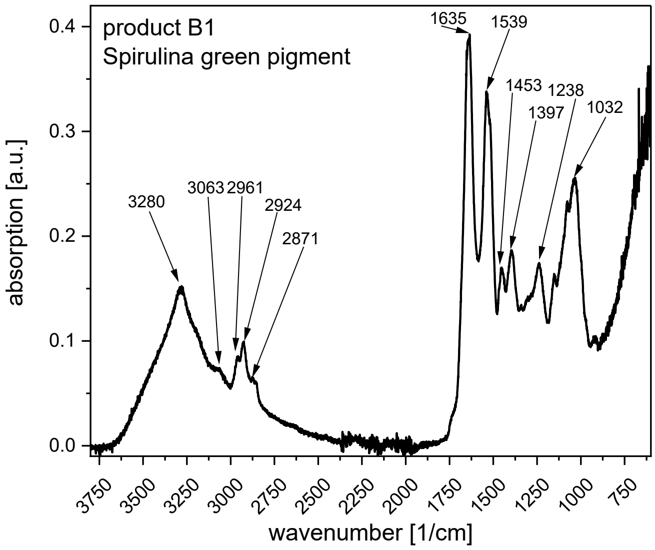

Fig. 6 Infrared spectrum of pigment Spirulina green (product B1).

The IR spectrum of the pigment Spirulina green is compared to the IR spectra of two microalgae offered as food additive (Spirulina and Chlorella) (see Figure 7). The IR spectra of Spirulina green and the Spirulina food additive exhibit the same pattern with similar signals. By this, Spirulina materials can be identified using IR spectroscopy. However, some of the signals in the fingerprint area of the IR spectra appear at slightly different wavenumbers. Probable the different Spirulina materials are built up by the same components but eventually the ratio of these components differ for the different product, so different signal positions might be explained for the IR spectra. The IR spectrum recorded from the Chlorella food additive is nearly similar to those of the investigated Spirulina products. Also, a good similarity to an infrared spectrum reported for Chlorella vulgaris in literature is given [55]. However, the IR spectrum of the Chlorella product exhibits a small shoulder signal at 1735 cm^{-1}. This shoulder signal is also reported in literature for Chlorella material and other green algae [56, 57]. Such a signal is typical for a C=O stretching vibration in an ester group. A component related to this can be ester of fatty acids present in the algae material.

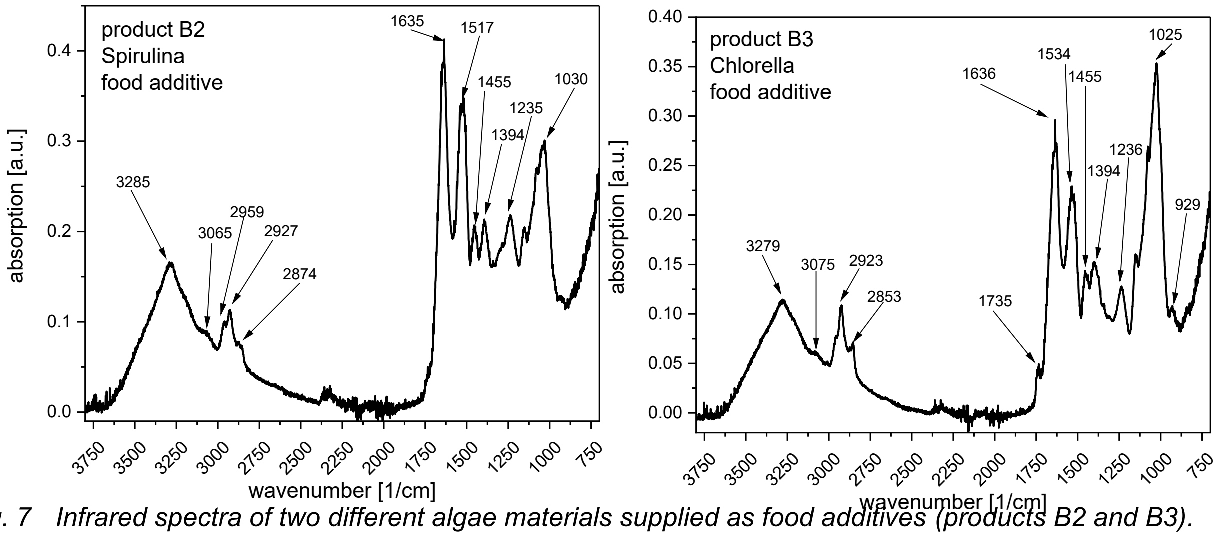

Fig. 7 Infrared spectra of two different algae materials supplied as food additives (products B2 and B3).

The IR spectrum recorded from Spirulina blue pigment (product B4) is presented in Figure 8. The IR spectra of Spirulina blue and Spirulina green pigments are clearly different from each other (compare Figures 6 and 8). The broad signal at around 3290 cm^{-1} can be assigned to O-H stretching vibrations from hydroxy groups in trehalose. The two signals at 2933 cm^{-1} and 2877 cm^{-1} are related to C-H stretching vibrations of different aliphatic units. The both signals at 1653 cm^{-1} and 1539 cm^{-1} can be assigned to amide groups which are part of the ring systems of the phycocyanin molecular structure - signals Amide I and Amide II [23]. The strongest peak at 988 cm^{-1} is assigned to C-O stretching vibrations.

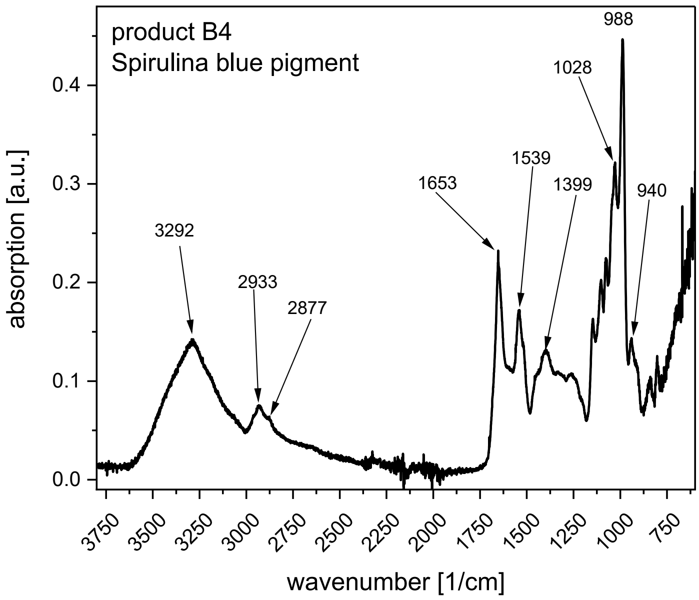

Fig. 8 Infrared spectrum of pigment Spirulina blue (product B4).

3.2 Macroalgae materials

For comparison and reference to above discussed microalgae materials, in this section three different macroalgae materials are discussed with their IR-spectra. One product is offered for usage as marbling base or binder for water-based paints [44]. The other both products are offered for food purposes. The macroalgae product B5 (Irish moss) is offered named as Pearl moss, Carragheen or Carrageen. Photographic image with enhanced magnification and SEM images of different magnifications are presented in Figures 9 and 10. The characteristic topography of the water based species can be observed in the images recorded with low magnification, while with higher magnification the structure of plant cells gets visible.

Fig. 9 Irish moss (product B5) – left: photographic image with higher magnification; right: SEM image with low magnification.

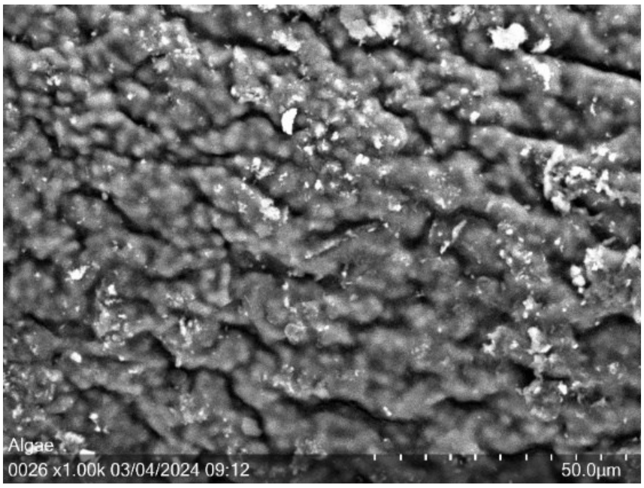

Fig. 10 Irish moss (product B5) – SEM image with higher magnification.

The IR spectrum recorded from Irish moss (product B5) is presented in Figure 11. This IR spectrum exhibits several signals which are quite similar to the IR spectra of the microalgae Spirulina and Chlorella. Also for product B5, the both amide (protein/based) signals at 1635 cm^{-1} and 1539 cm^{-1} are clearly present. The broad signal around 3300 cm^{-1} is broader, probable because of the presence of different types of components with hydroxy groups and with N-H units. The strong signal with three peaks at 1145 cm^{-1}, 1069 cm^{-1} and 1026 cm^{-1} can be assigned to C-O stretching vibrations probable related to cellulosic based components building up the structure of the macroalgae.

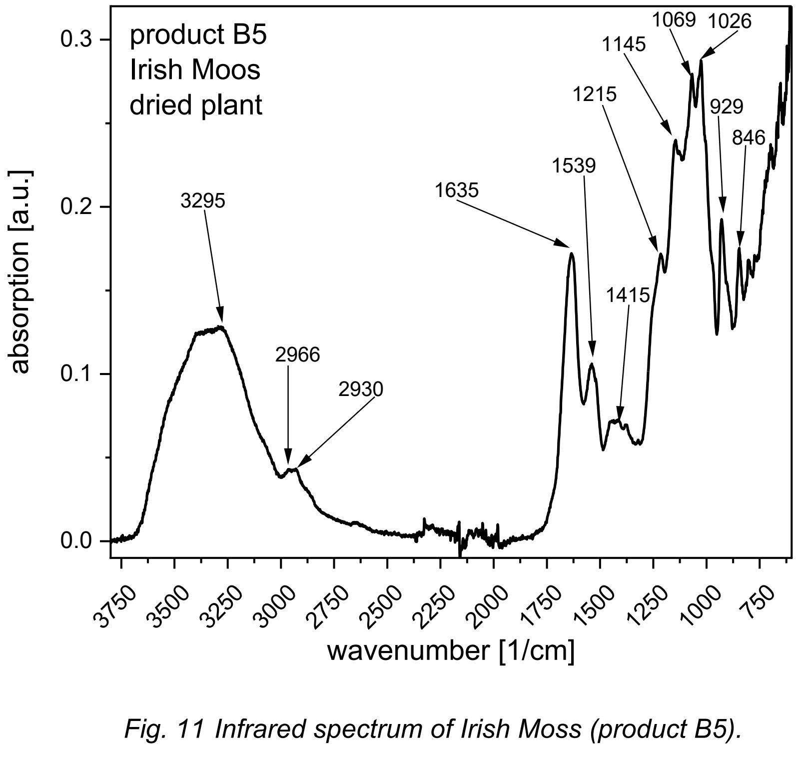

Fig. 11 Infrared spectrum of Irish Moss (product B5).

The red seaweed dulse (*Palmaria palmata*) is broadly used as food additive and an effective protein source [38, 58-60]. The photographic image with enhanced magnification taken from Dulse product B6 is presented in Figure 12. The red to violet coloration is clearly visible, together with the flake like structure of this product. From one of these flakes, a SEM image is recorded in higher magnification (Figure 12). In this SEM image, the plant cells are clearly visible on the surface of this product.

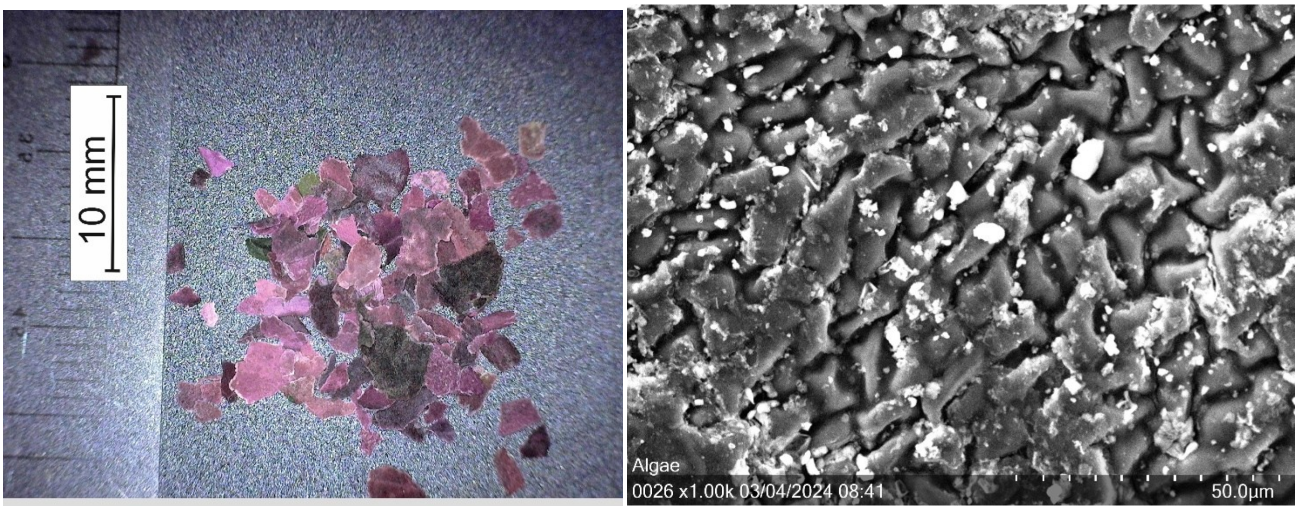

Fig. 12 Dulse, red algae (product B6) – left: photographic image with higher magnification; right: SEM image recorded in higher magnification.

The IR spectrum of Dulse is presented in Figure 13. It is clearly different from the IR-spectra presented for the other products above. The broad signal around 3300 cm^{-1} is distributed into several peaks at 3381 cm^{-1} and 3260 cm^{-1} leading to the assumption that different types of OH units are present in this product. The amide related signal at 1637 cm^{-1} is weaker, so a lower protein content can be estimated. Finally, the strongest signal is recorded at 1032 cm^{-1}, which can be assigned to C-O stretching vibrations. This is probable related to a higher content of cellulosic components or other carbohydrates building up the structure of this macroalgae.

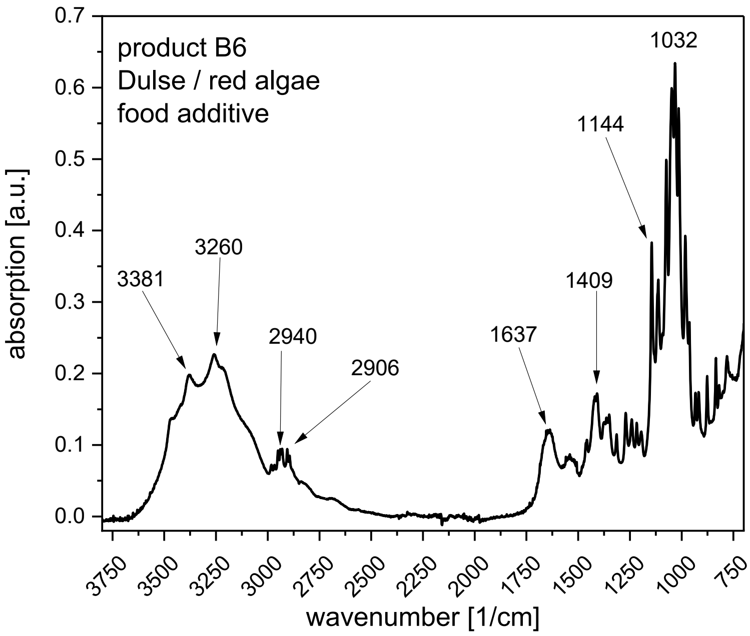

Fig. 13 Infrared spectrum of Dulse (red algae) (product B6).

As third macroalgae product in this overview dried pieces of Wakame seaweed are considered. This product is offered for food purposes [46, 61, 62]. A SEM image showing the magnified topography of the Wakame product is presented in Figure 14. In this image, small and regular structures can be observed, which could be assigned to plant cells.

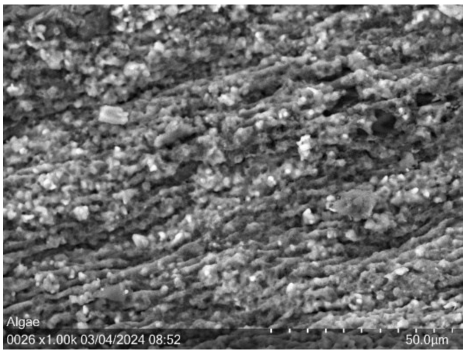

Fig. 14 SEM image of a dried piece from Wakame algae (product B7).

An IR spectrum recorded from the Wakame product is presented in Figure 15. This IR spectrum is quite comparable with the IR spectra presented above for Spirulina and Chlorella products, so for these products a differentiation between macroalgae and microalgae is not possible by using IR spectroscopy.

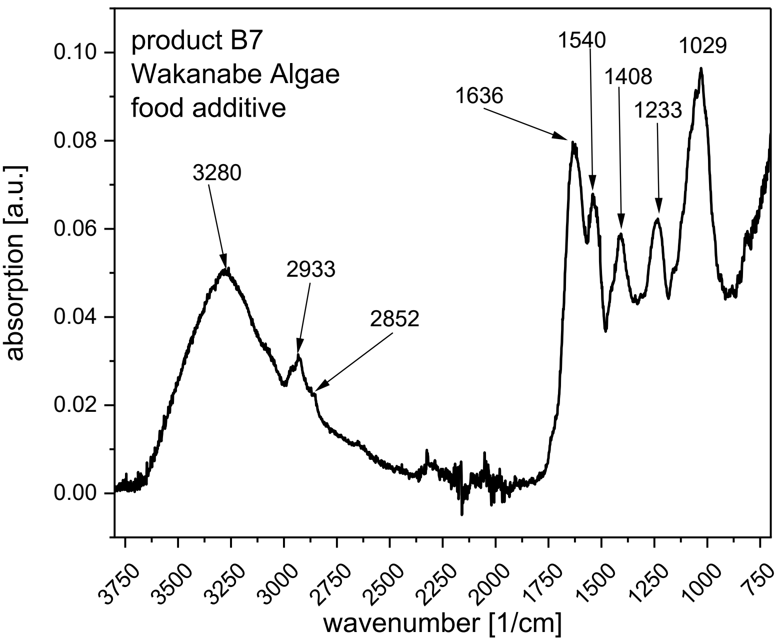

Fig. 15 Infrared spectrum of Wakame algae (product B7).

Finally, with product B8 a powder from brown algae is considered. This algae product is also named Kelp and from the algae *Laminaria digitata*. It is offered as food supplement and especially the high content of the amino acid phenylalanine and the chemical element iodine is mentioned [47]. The IR spectrum of product B8 is presented in Figure 16. This IR spectrum is quite unique among the other reported IR spectra, mainly because of the average signal at 1740 cm^{-1}. This signal can be assigned to C=O stretching vibrations of ester group and could be a hint on the content of fatty acids in this algae product B8.

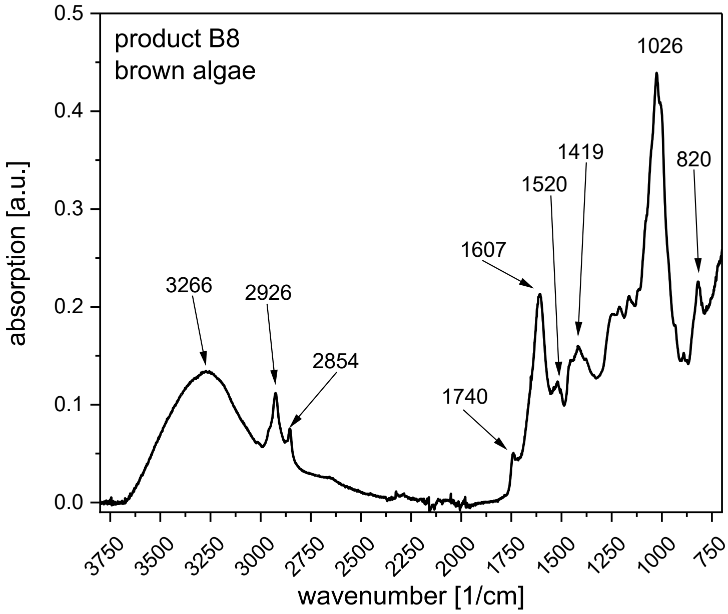

Fig. 16 Infrared spectrum of brown algae powder / Laminaria digitata (product B8).

3.3 Orcein

Orcein is a natural dye obtained by extraction from lichen, similar to litmus [63, 64]. Lichens are organisms that represent a symbiosis of algae and fungi. The fungus supplies the algae with water and minerals, while the algae part supplies sugar from photosynthesis. Orcein was also used to dye textiles in the Middle Ages [63, 65]. Nowadays, orcein is commonly used to stain biological preparations [63]. Orcein is also named as CI Natural Red 28 [1]. A study reports on the presence of 14 dye compounds present in Orcein. These dye compounds are mainly related to derivatives of 7-amino-2-phenoxazone, 7-hydroxy-2-phenoxazone and 7-amino-2-phenoxazime [66]. An overview on the chemical structures of these color components of Orcein is given in Figure 17.

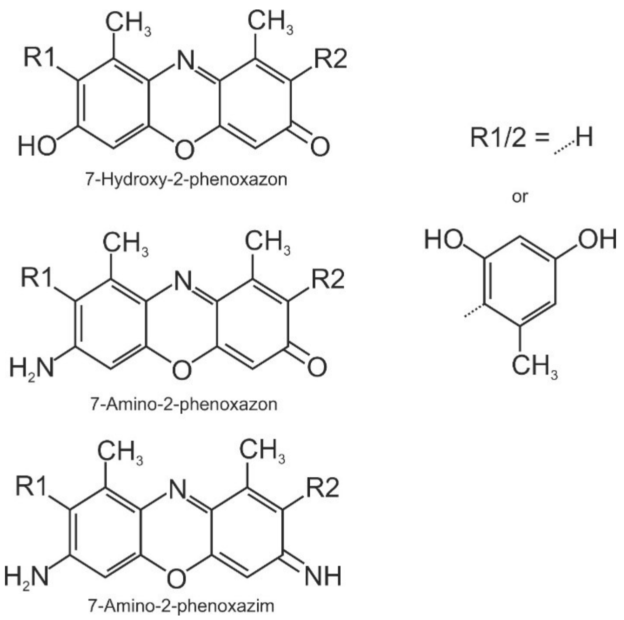

Fig. 17 Chemical structures of different basic units of the natural dye Orcein.

The IR spectrum of dye Orcein (product B9) is shown in Figure 18. A broad signal around 3190 cm^{-1} can be assigned to O-H stretching vibrations and N-H stretching vibrations from the dye molecule. Peaks related to C-H stretching vibrations are nearly not detected. The strong peak at 1585 cm^{-1} can be assigned to C=O stretching vibration of keto groups attached to the aromatic ring systems building up the chromophore [23]. The signal at 1448 cm^{-1} can be related to a C=N stretching vibration of the C=N-H group attached to the chromophore [23]. The strongest signal at 1140 cm^{-1} can be assigned to C-O stretching vibration from hydroxy groups attached to aromatic units.

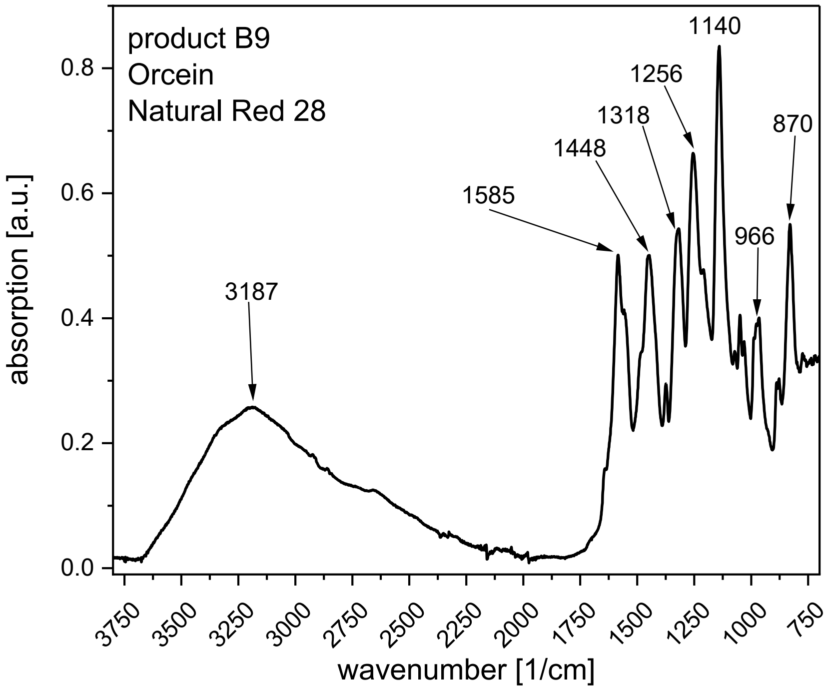

Fig. 18 Infrared spectrum of natural dye Orcein (product B9).

4 Conclusions

Algae materials and products find a broad range of different applications. However, there use for dyeing processes of textile materials is less considered, even if there are interesting reports in scientific literature. In the current overview, nine algae related products are presented, also under consideration to their use for coloration purposes. For all products, the IR spectra are recorded and presented. These IR spectra are mostly dominated by vibrations caused from amide groups, due to the protein content of the algae products. Additionally, significant signals are related to the presence of cellulosic components or other carbohydrates. However, the specific distinguishing between different types of algae materials is challenging by infrared spectroscopy. Nevertheless, the presented overview and IR spectra can be valuable for purposes in the field of pigment analytic, algae and quality control. Overall, the use of algae materials even for textile treatment processes and dyeing might have a prospering future, due to increasing demands for sustainable product and fully bio-based materials.

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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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