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Dyeing properties, coloring compounds and antioxidant activity of *Hubera nitidissima* (Dunal) Chaowasku (Annonaceae)

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ABSTRACT

An evaluation of the coloring potential of the different parts of selected plants from New Caledonia has been performed. Among them *Hubera nitidissima*, an Annonaceae, showed an intense yellow color on fibers. This is the first report of such a coloring potential for a *Hubera* sp. In this study we present dyeing results obtained with extracts from the leaves of *H. nitidissima* on linen, silk and wool, color characteristics, dye light-fastness and chemical identity of coloring compounds. The absorbance, *K*/*S* index and CIELab coordinates of dyed fibers were measured. Color strength values and fastness properties of the dyed fabrics were high: 7/8 in Blue scale rating. The dyed fibers were analyzed by liquid chromatography hyphenated with UV–Vis spectroscopy and mass spectrometry using UHPLC-DAD-HRMS (ESI-QTOF) instrumentation. We identified two xanthones: mangiferin and homomangiferin together with quercetin glycosides. The hydroalcoholic extract from the leaves was screened for its free radical scavenging properties (DPPH) and showed significant antioxidant activity (26.5 μg/mL). The presence of mangiferin in the Annonaceae family is attested here for the first time. As a result of our study, *H. nitidissima* appears as an excellent source of light-fast yellow dye with interesting antioxidant properties.

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1. Introduction

Until the last third of 19th Century, dyeing matter came from the natural world, especially from plants [1] but later on, with the invention of synthetic dyes, natural colorants have been neglected. Nowadays interest is growing in finding nature friendly sustainable technologies that can be used as alternatives to fossil-based raw materials [2]. This is creating a return to the use of products coming from renewable resources such as natural dyes [3]. The use of natural dyes is also on the rise because they offer diverse and unique shades on different natural fabrics and they are found to be UV protective [4], deodorizing, antifeedant [5], antimicrobial and antioxidant [6–8]. Furthermore, the interest for the assessment of antioxidant properties of natural compounds increases due to their role in minimizing harmful effects of oxidative stress and because of their uses in medicine, food and cosmetics [9,10]. The importance of natural dyes in cultural heritage led to take into account their use throughout the Pacific islands. In New-Caledonia, plants are a major part of the Kanak culture and played a role in economic and cultural exchange [11] as well as in the rich local pharmacopoeia. Little is known about the natural dyes of New Caledonia despite attention of a few researchers. Rageau established an informal list on the dyeing plants present in New Caledonia in 1957 but he didn't take this part in his manuscript published in 1973 concerning the medicinal plants of New Caledonia [12]. Zepernick wrote an article in 1967 about the tinctorial plants of Polynesia [13]. Formerly, in the Kanak civilization, people were admittedly little dressed but used beaten bark cloth and plaited mats to make women's traditional skirts, men's traditional hats, traditional finery or traditional barter "money". The more transformed the objects were, and particularly, dyed, the more they were valued. A lot of such traditional artifacts were found to be dyed but, with the contact of European culture, those practices and knowledge disappeared. In this context, our

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aim was to evaluate the coloring potentialities of the New Caledonian flora which is almost 80% endemic. Thus, following a screening based on the coloring potential of different parts of selected plants, an Annonaceae, *Hubera nitidissima* was selected for further investigation because its extract gave an intense yellow color on different fibers. The Annonaceae is represented in New Caledonia by 5 genera: 12 species including 11 endemic [14], and they are known to contain a wide range of bioactive compounds such as antioxidant and antibacterial [15]. Some Annonaceae were previously investigated as natural dyes for their flavonoids and tannins contents [1] but the genus *Hubera* (ex. *Polyalthia* [16]) is unknown for dyeing properties. Here we present for the first time the coloring potential of a *Hubera* sp., *H. nitidissima* (Dunal) Chaowasku. This species is distributed along the Australian coasts (Northern Territory, Queensland and New South Wales) [17,18] as well as in Vanuatu, Papua New Guinea and New Caledonia [19] where it is the only species of this genus to grow. This plant is known in Northern New Caledonia for the use of its bark to make traditional Kanak customary money [20]. In Vanuatu its wood is used for traditional hut post [21]. Leaves of *H. nitidissima* contain different isoquinoline alkaloids [19] and essential oils [22].

The purpose of this research was to characterize *H. nitidissima* leaves as a tinctorial raw material. Firstly, we studied the dyeing efficiency through bath dye exhaustion and spectrocolorimetry. Then, we evaluated the dye light-fastness properties using spectroscopic approach. The original colors and the color changes during light fading have been recorded in CIELab (1976) color coordinates. We have identified the dye components in extracted dyestuff by liquid chromatography hyphenated with UV–Vis and mass spectrometric detection – a powerful method for chemical characterization in dye analysis [23]. The samples dyed with fresh and dry leaves were analyzed with an UHPLC-DAD-HRMS system after the re-extraction of the main colorant and compared to the hydroalcoholic extract from leaves. We were also interested in the antioxidant activity of the leaf extract: the 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging assay was practiced for the assessment of antiradical properties.

2. Materials and methods

2.1. Materials

2.1.1. Plant material

Leaves of *H. nitidissima* (Dunal) Chaowasku were collected in June and November 2011 (authorization number: 60912-203-2011/jjc) from the North Province of New-Caledonia (South Pacific) at the Creek Hervouet (S 21°19,290′, E 165°04,659′, alt. 54 m). A voucher specimen (reference: MT18) identified by Jérôme Munzinger is kept at the Herbarium of Nouméa (NOU). One part of the harvested plant material was air dried and grounded into powder before extraction; the other part was used fresh, directly after collecting.

2.1.2. Fibers and chemicals

Wool yarn, silk and linen were purchased from Shop Text Ponsard (Paris, France). Potash alum ($KAl(SO_4)_2{\cdot}12H_2O$, medicinal) obtained from Cooper (Melun, France) was used for mordanting the fibers. The standard of mangiferin (98%) was obtained from Sigma–Aldrich (Steinheim, Germany). Ascorbic acid (reagent grade) and 2,2-diphenyl-1-picrylhydrazyl (DPPH, ≥ 85%) were purchased from Sigma–Aldrich (Saint-Quentin-Fallavier, France).

Extractions were done using distillated water and absolute ethanol furnished by Unilab (Ajax Finechem Pty. Ltd., Auckland, New Zealand). Dimethylsulfoxide (DMSO, 99.7+%, Extra Dry) for extraction and solubilization purposes was bought from Acros Organics (Geel, Belgium). Methanol (MeOH, Chromasolv HPLC gradient grade) and oxalic acid dihydrate (OxA, ACS reagent, ≥99%) were obtained from Sigma–Aldrich (Saint-Quentin-Fallavier, France).

Chromatographic separations were done using acetonitrile (MeCN, HPLC supra gradient) purchased from Biosolve (Dieuze, France), water (H_2O, HPLC gradient grade) supplied by Fischer Scientific (Illkirch, France) and glacial acetic acid (99.9+%) for anion suppression, purchased from Acros Organics (Geel, Belgium).

2.1.3. Instrumentation

The dye exhaustion was measured at a maximum wavelength of 450 nm using an Anthelie Advanced 2 UV–Vis spectrometer (Secomam, Ales, France).

The color CIELab coordinates (L^*, a^*, b^*) as well as reflectance spectra of the dyed samples were measured in the wavelength range 360–740 nm (8°, D65 illuminant) with CM-2500d spectrocolorimeter (Konica Minolta).

The light-fastness test of dyed fibers was conducted on Xenotest 150S+ (Atlas) having an air-cooled xenon lamp (24 °C, 2200 W).

The spectroscopic analyses of extracts were performed with 1290 Infinity UHPLC-DAD-HRMS (ESI-QTOF model G6530A, Agilent Technologies).

The assay of the antioxidant properties was performed by measuring the absorbance of DPPH using a microplate reader (SpectroBiotekEpoch).

2.2. Methods

2.2.1. Dyeing

After having soaked the fibers in water, they were submitted to pre-mordanting method. Fibers were put in a warm solution of potash alum at 0.3% w.o.f (weight of fiber). This bath was kept at 60 °C for 45 min and left at room temperature for cooling. Mordanted fibers were rinsed with tap water to remove the remaining, non fixed alum. For the dyeing bath, a M:L (material to liquor ratio) at 1:40 was used for fresh and dry leaves. The dyeing bath was first prepared as an infusion of leaves in distilled water at 60 °C for 45 min. After cooling the bath to 30 °C, the mordanted wet fibers were all put in the dyeing bath which was heated again to 60 °C and kept at this temperature for 45 min with manual agitation. The fibers in bath were allowed to cool down, and then left immersed in the dyeing bath for 12 h at room temperature. The dyed fibers were then rinsed with tap water and left for drying at room temperature.

2.2.2. Determination of dye exhaustion

According to previous works [8] the determination of dye uptake was done by measuring the absorbance of the fresh leaves dyeing bath solution before and after dyeing. The dyebath was cooled to room temperature prior to measures. The percentage of dye exhaustion (DE) was calculated according to the given formula:

$$DE = [(A_0 - A_1)/A_0] \times 100 \quad (1)$$

where A_0 and A_1 are absorbances of the dye bath, before and after dyeing respectively.

2.2.3. Color characteristics

The color strength and color depth of the dyed samples were determined by light reflectance technique. Colorimetric and absorption measurements were performed before and after the light-fastness test in order to evaluate sensitivity to light of the textiles dyed with fresh and dry matters. The absorbance of samples dyed with fresh matter was recorded before and after the light-fastness test with an average of six measurements. The colors are given in CIELab coordinates: L^* corresponding to the brightness (100 = white,

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