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BIOMASS RESIDUES AS A SOURCE OF NATURAL DYES FOR TEXTILE INDUSTRIES: THE CASE STUDY OF CORK POWDER

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ABSTRACT: The possibility of valorising cork powder, the most relevant residue of cork stopper producing activity, as a source of bio-dyes for the textile industry was evaluated in order to be an economically and environmentally sustainable way to replace chemical dyes by natural compounds. The aqueous extraction process of natural bioactive compounds from cork powder was optimized, modifying the temperature (30 °C and 100 °C) and the concentration of cork powder (5, 10 and 20 g/L) used in the extractions. The three textiles substrates used (cotton, cationized cotton and wool) presented a brownish hue after dyeing, more intense in the cotton with the cationization pre-treatment. The colouration obtained was uniform with a good light colour fastness performance. In general, the colour is more intense for textiles immersed in extracts obtained with higher concentration of cork powder and higher extraction temperature, but with more noticeable results in the wool and cationized cotton. The results show that cork powder can be highly valorised as a source of bio-dyes improving the implementation of the circular economy in the cork sector.

Keywords: biobased products, biomass, forestry, green chemistry.

1 INTRODUCTION

Cork is the bark of a special tree, the cork oak (*Quercus suber* L), that grows in the Mediterranean regions, and particularly in Portugal, which is the world leader of cork oak forests. The most important and noble product of the cork sector is the cork stopper, used worldwide as alcoholic beverages sealant. Several mechanical operations are required to transform the cork plank in this final product. During this process, it is produced a solid waste, the cork powder, usually valorised as fuel in biomass boilers.

With the aim of evolving towards a circular economy, the cork industry is currently very keen to develop processes that allow the valorisation of all its waste and by-products, with particular emphasis on cork powder, which is the most relevant residue of this activity.

The use of natural dyes in textile colouration has been growing in the last decade due to the increasing health concerns of consumers and environmental aspects. A significant amount of studies addressing the use of wastes as bio-dye sources, especially from the food and beverage industry [1–5] can be found in the bibliography. Studies with application of natural dyes extracted from plants in fabric colouration are also frequent [6–9], recreating ancient techniques. However, as far as we know, studies concerning the use of cork as colouring agent of textile substrates do not exist in the literature.

In the present paper, the possibility of valorising the cork powder residue as a source of bio-dyes to be used in the textile industry was evaluated. The aqueous extraction process of natural bioactive compounds from the cork powder was optimized. This process does not invalidate the traditional valorisation alternatives for the cork powder residue and is an economically and environmentally sustainable way to provide natural dyes to the textile industry.

2 MATERIALS AND METHODS

2.1 Raw material - cork powder

Cork powder was obtained from a natural cork stopper industry directly from the mechanical operation of natural cork, in order to avoid the contamination with chemical products used in the production of technical cork stoppers or during the surface treatment of the natural cork stoppers. M.A. Silva – Cortiças S.A. kindly provided the cork powder used in this study.

2.2 Textile samples

Three different types of fabric were used in the dyeing assays: wool (organic), cotton (natural) and cotton cationized. Tintex Textiles S.A. kindly provided the fabrics. The cationized cotton resulted from a previous treatment of the textile surface, which for reasons of confidentiality cannot be described.

Samples used for dyeing were obtained cutting circular shape portions, with approximately 4.6 cm of diameter, from fabrics.

2.3 Bioactive compounds extraction

A conventional solid-liquid extraction was performed to extract the cork powder bioactive compounds using tap water as solvent. Experimental conditions like temperature and ratio of powder cork/solvent were changed in order to optimize the separation of bioactive compounds. Aqueous extractions with temperatures of 30 °C and 100 °C were performed and the cork powder concentration used in the extractions were modified from 5 g/L to 20 g/L.

The extraction process was performed in a container equipped with a condenser in order to minimize losses by evaporation, during 3 h. A magnetic stirrer was used to promote the cork powder suspension in the water during extractions. In order to follow the amount of bioactive compounds extracted, samples were withdrawn at intervals of 30 min for quantification. The samples were

filtered to remove the suspended cork powder before being stored in the freezer.

After extraction the solid residue was separated by centrifugation (3000 rpm, for 10 min) followed by vacuum filtration (glass fibre filter with 0.47 mm of diameter and 0.2 µm porosity) and the filtrate was used in the dyeing experiments, anti-microbiological and phytochemical tests. The samples collected during extraction were also submitted to a vacuum filtration before being analysed.

The absorbance was measured in the range of 200–750 nm using a UV-vis spectrophotometer (Thermo Scientific, Evolution 201).

2.4 Qualitative phytochemical tests

A phytochemical inspection of constituents was made in the cork powder extract obtained, based on the work of Vesoul and Cock [10]. The presence of constituents as saponins, phenolic compounds, flavonoids, polysteroids, triterpenoids, cardiac glycosides, anthraquinones, tannins and alkaloids were examined.

2.5 Evaluation of antimicrobial activity

Antimicrobial activity of cork powder extract was determined using a modified Kirby-Bauer disc diffusion method. Two selected bacteria were tested: *Escherichia coli* (ATCC 10536) and *Staphylococcus aureus* (ATCC 29213). The bacteria were grown in NB fresh broth at 37 °C for 24 h and then 100 µL of this microbial suspension was spread onto Muller Hinton agar plates. Each antimicrobial assay was performed in triplicate. Standard discs (6 mm ϕ) of Penicillin G and kanamycin were obtained from Oxoid Ltd. and served as positive controls for antimicrobial activity and filter discs (6 mm ϕ) impregnated with 200 µL of distilled water were used as negative controls. The extract were tested using 6 mm ϕ sterilized filter paper discs impregnated with 200 µL of the test sample, allowed to dry and placed onto inoculated plates. The inoculated plates were incubated at 37 °C for 18 h and, after the incubation period, the diameters of the inhibition zones were measured with a ruler in order to evaluate the sensitivity of the bacteria in relation to the cork powder extract.

2.6 Dyeing process

In the dyeing process, the tissue samples were dyed in triplicate and a control sample was always used replacing the extract by water. Dyeing was carried out in polypropylene flasks with lid containing 20 mL of water (in the case of the control) or extract, both at 50 °C. Textile samples were introduced into the flasks and placed in an incubator at 50 °C for 100 min under mechanical agitation (320 rpm). At the end, the samples were removed with tweezers and placed on a clean absorbent paper to remove the excess of water/extract.

2.7 Colour fastness and colour measurement

Parameters as colour fastness to washing and to natural light and atmospheric agents are important to evaluate the stain suitability.

To evaluate the colour fastness to washing, after dyeing the samples were washed with cold (20 °C) and hot (45 °C) water, agitated mechanically (320 rpm) during 10 min.

To evaluate the colour fastness to light, after dyeing and washing, the samples were dried at room temperature with about half of its surface protected from light and atmospheric agents with a black card.

After one week, the colour of fabric samples were measured with a colorimeter using the CIELab coordinates system. To evaluate the colouration homogeneity obtained in the textile substrates and the colour fastness to natural light, six evenly distributed measurements were made on each side of the tissue, three of them on the side exposed to light and three others on the protected side.

3 RESULTS AND DISCUSSION

3.1 Cork powder extraction

Aqueous extraction of cork powder was carried out with the solvent at boiling conditions (about 100 °C) and at 30 °C. For each extraction temperature, three different cork powder concentrations were used: 5 g/L; 10 g/L and 20 g/L. During the extraction process, samples were withdrawn from the water bath where cork powder were immersed, in order to follow the extent of biocompounds extraction by UV-vis spectrophotometry. The absorbance scanning between 200 nm and 750 nm obtained from the samples collected (in intervals of 30 min), for all the cork powder concentrations used in the extraction processes at 100 °C are presented in Fig. 1. Figure 1 presents also similar information for all the extraction processes carried out at 30 °C. It should be noted that all the samples were diluted with the same dilution factor (1:50) before measuring the absorbance spectra.

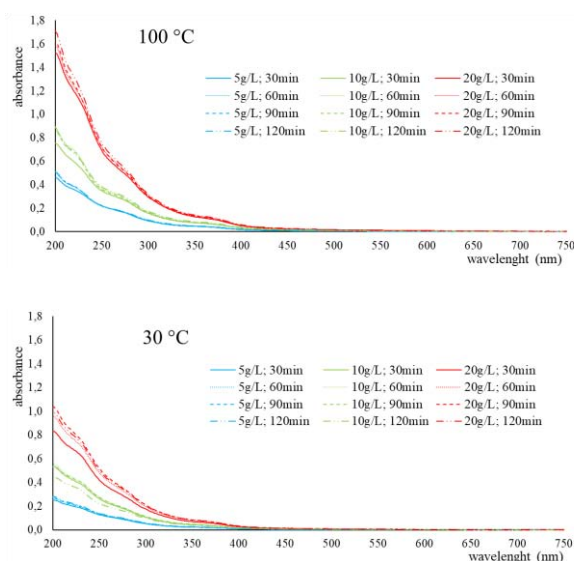


Figure 1: Absorbance spectra for cork powder extracts during the extraction process performed at different conditions.

The amount of cork powder used in the extraction have a great relevance for the level of biocompounds extracted in both temperatures. The effect of the temperature used to perform the extraction on the amount of compounds extracted is also relevant, obtaining greater absorbance values in the experiment the solvent was (nearly) boiling. However, extending the extraction more than 60 min is not advantageous because no significant modifications were observed in the absorbance spectra, even when a temperature of 30 °C was used.

3.2 Phytochemical tests

The phytochemical screening of the aqueous extract obtained from the cork powder indicated the visible presence of saponins, phenolic compounds (the majority water insoluble), flavonoids and tannins. Vestiges of anthraquinones in their free and combined forms were also registered.

3.3 Antimicrobial activity

The antimicrobial tests against the bacteria *Staphylococcus aureus* (gram negative) and *Escherichia coli* (gram positive) were conducted following the disk diffusion method, based on the Kirby-Bauer method. It was observed that the extract obtained from the cork powder did not inhibit the growth of these two bacteria, since the formation of inhibition zones was not observed. However, in the literature it was found references to the antimicrobial activity of cork, being this activity associated with the presence of phenolic content [11, 12]. Possibly, the extract used in the determinations is not enough concentrate, and consequently the compounds with antibacterial power, for example the tannins, may be present in amounts below the minimum inhibitory concentration.

3.4 Dyeing performance and final colour

Colorimetric analysis of the textile substrates performed at the end of the colour fastness tests allowed the evaluation of the dyeing quality. The CIE $L^*a^*b^*$ coordinates system used to measure the colour is stated in terms of three coordinates: L^* , a^* and b^* . The coordinate L^* represents lightness (or luminosity) and the value of 100 corresponds to white and 0 to black. The redness–greenness of colour is related to the coordinate a^* (higher positive values correspond to a colour closer to red while more negative values correspond to a colour closer to green) and the yellowness–blueness of colour is associated to the value of coordinate b^* (more positive closer to yellow and more negative closer to blue).

To express the difference in colour between the samples of the textile substrates in terms of a single value, the parameter ΔE^* was calculated as follows: $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$, where ΔL^* , Δa^* and Δb^* , represent the difference of coordinates L^* , a^* and b^* , respectively, between the sample dyed and the correspondent control (sample submitted to the dyeing process immersed in water). The parameter ΔE^* was calculated for all the samples of natural cotton, cationized cotton and wool dyed with the extracts obtained in different conditions.

The magnitude of the ΔE^* value obtained between two samples is related to the perception of colour differences between them [13]. If $\Delta E^* \leq 1.0$ a normal observer does not notice the colour difference. For ΔE^* values between 1 and 2, only an experienced observer notice the difference, but if ΔE^* is between 2 and 3.5 a normal observer already have the perception of the colour difference. If the value of ΔE^* is between 3.5 and 5 there is a clear difference in colour and for values greater than 5 the colours are different.

The parameter ΔE^* was also calculated between the portion protected and exposed to natural light in the same fabric sample to access the colour fastness to natural light and ambient agents. Using colour measurements made in different locations in both sides of the same fabric sample, the parameter ΔE^* was determined to evaluate the coloration uniformity obtained in the textile substrates.

The values of ΔE^* calculated for the natural cotton dyed samples were almost in all cases less than 1, indicating that a normal observer doesn't have the perception of any colour difference in the fabric. For the cationized cotton and wool samples, the majority of situations results too in values of $\Delta E^* \leq 1.0$, but in some cases the magnitude of ΔE^* is between 1 and 2 and in two wool samples ΔE^* is greater than 2. Therefore, only an experienced observer has the perception of the non-homogeneous in the cationized cotton and wool dyed samples. As, in general, the perception of colour difference in the same sample is not noticed by a normal observer, an average of the twelve values measured in both sides was used to represent the L^* , a^* and b^* coordinates.

The parameter ΔE^* was calculated for all the samples of textile substrates dyed with the extracts obtained from the powder cork extractions carried out at different temperatures and amounts of powder. The textile substrates used as controls during the dyeing operation were the reference for ΔE^* determination. As each fabric was dyed in triplicate the average value ΔE^* was calculated and used to represent the colour. In Fig. 2 the obtained values are presented in three bar graphics, one for each textile substrate: cotton; cationized cotton and wool. The error bars shown in the graphics represents the standard deviation of the data set of ΔE^* obtained for the three dyeing assays performed in the same conditions. The relative standard deviation (RSD) is for all cases smaller than 4.3 %, with exception for data set of dyeing tests performed with the extract obtained at 100 °C using an amount of 5 g/L of cork powder, in which RSD is 8.6 %.

As can be observed from Fig. 2, in all cases the colour of the fabrics after dyeing are different when comparing with the textile substrate before dyeing, because ΔE^* is greater than 5 [13]. However, the improvement in the colouration is clearly higher for the cotton with pre-treated surface with chemical cationization. The incorporation of cations in the cotton fibres results in a higher natural dye fixation and, thus, the textile colouration is more intense.

Although a general trend exists to have greater values of ΔE^* for textiles immersed in extracts obtained with higher amounts of cork powder and higher extraction temperatures (see Fig. 2), some remarks should be outlined.

When comparing the effect on the colourations obtained with extracts resulting from extractions carried out at different temperatures, the observations are the following:

- is more significant in the colouration of wool, mainly when small amounts of cork powder were used in the extractions. An increase of 24 % in the colour parameter ΔE^* for wool was observed when the extract obtained with 5 g/L of cork powder at 30 °C used in the dyeing process was changed to the one obtained at 100 °C.
- there is no significant modifications in the colour of the cotton (natural) samples. The variation in the colour parameter ΔE^* ($\leq 4\%$) calculated for the dyeing assays performed at 30 °C and 100 °C is comparable (or smaller) to the relative standard deviation (RSD) for dyeing tests made in triplicate.
- the modification from 30 °C to 100 °C of the extraction temperature, resulted in extracts that improved the colouring process of the cationized cotton with an increase of about 10 % in ΔE^* . The case of the extracts obtained with an amount of cork powder of 10 g/L is an exception

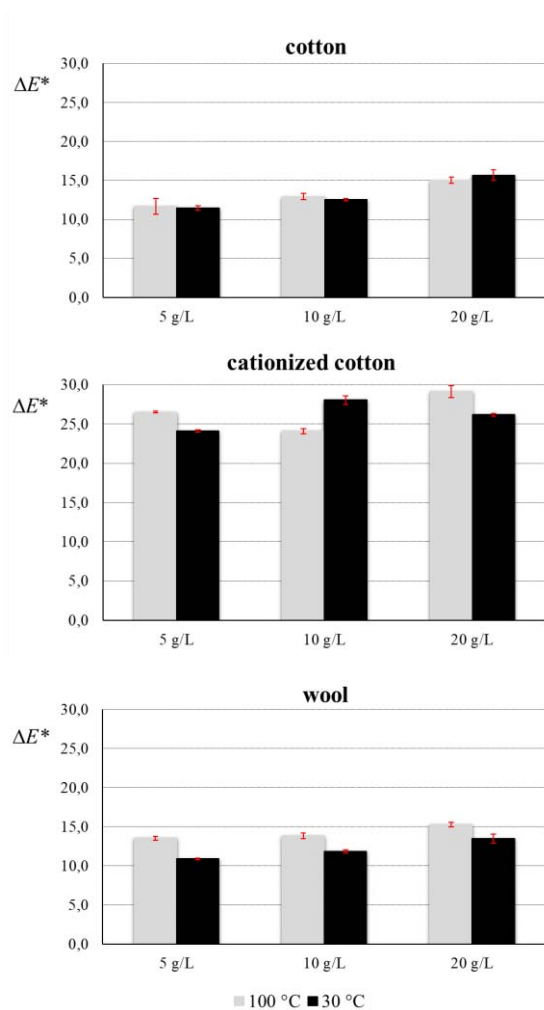


Figure 2: Colour difference observed between textile samples (cotton, cationized cotton and wool) and respective control, expressed by ΔE^* , after dyeing with the extracts obtained at 30 °C and 100 °C using different amounts of powder cork (5 g/L, 10g/L and 20 g/L).



















When comparing the effect on the textile colourations obtained with extracts resulting from extractions with different amounts of cork powder, the following considerations can be mentioned:

- the increase in the colouration when using greater amounts of cork powder is more pronounced for the cotton (natural) substrate and it seems that the effect is greater for lower temperatures of extraction. When the extraction is performed at 30 °C, increasing the amount of cork powder from 10 g/L to 20 g/L results in a colour parameter ΔE^* measured about 25 % higher in the cotton samples.
- the trend of greater ΔE^* measured in samples dyed with extracts obtained with higher cork powder concentration is not observed in the dyeing of the cotton with the pre-treatment of cationization.
- the increase in the wool colouration is not significant, being in all cases less than 14 %, but with better performance in the colour improvement for the extracts obtained at 30 °C.

In Table 1 the values of ΔE^* calculated for the samples dyed with the extracts obtained in all the conditions considered in this study are indicated below the rectangle filled with the colour created to represent the dyed fabric. The colour replication was possible after the coordinates

L^* , a^* and b^* being converted to the RGB coordinate system with the aid of the free software OpenRGB (Logicol S.r.l., 2018). Alongside the form filled with the colour obtained for the fabric sample dyed, it is presented a square filled with the colour recreated for the correspondent control.

Table I: Colour replication for all the textiles samples dyed with cork powder extracts and respective controls, after conversion of CIE $L^*a^*b^*$ coordinates into RGB coordinates.

	cork powder extract	cotton control sample	cotton cationized control sample	wool control sample
100 °C	5 g/L	 $\Delta E^*=11.66\pm1.00$	 $\Delta E^*=26.51\pm0.12$	 $\Delta E^*=13.53\pm0.27$
	10 g/L	 $\Delta E^*=12.93\pm0.40$	 $\Delta E^*=24.09\pm0.34$	 $\Delta E^*=13.82\pm0.35$
	20 g/L	 $\Delta E^*=15.03\pm0.38$	 $\Delta E^*=29.12\pm0.75$	 $\Delta E^*=15.26\pm0.26$
30 °C	5 g/L	 $\Delta E^*=11.46\pm0.28$	 $\Delta E^*=24.12\pm0.17$	 $\Delta E^*=10.89\pm0.10$
	10 g/L	 $\Delta E^*=12.54\pm0.15$	 $\Delta E^*=28.04\pm0.52$	 $\Delta E^*=11.84\pm0.24$
	20 g/L	 $\Delta E^*=12.05\pm0.27$	 $\Delta E^*=26.19\pm0.19$	 $\Delta E^*=13.49\pm0.50$

According to the colour replications presented in Table I, light brown colours were obtained in the textiles substrates dyed with cork powder extracts. However, the most intense brownish taint was observed in the cationized cotton samples, with the highest values of ΔE^* , revealing that the cotton surface pre-treatment enhanced the dye fixation in the cotton fibres, as referred before.

In Fig. 3 the colours for the three textile substrates, before and after dyeing, are expressed in terms of the colorimetric coordinates. As sketched in Fig. 3, lightness is denoted by L^* , red/green is represented by the coordinate component a^* and blue/yellow is represented by the coordinate component b^* .

From Fig. 3 is clear that all samples of cotton, cationized cotton and wool dyed with cork powder extracts are darker after dyeing, with L^* values for samples smaller than for the controls. However, the difference is markedly greater for cationized cotton and wool. In addition, the samples of cationized cotton and wool became more red after dyeing with the red/green coordinate component a^* exhibiting positive values. For the cotton samples, the coordinate a^* does not change significantly with dyeing and the negative values of a^* indicates a greenish tonality. The yellowness of the cationized cotton samples dyed is notably more intense comparatively to the controls as can be seen in Fig. 3 from the highest differences in the yellow/blue

coordinate b^* . The small difference observed in the dyed wool samples is due to the fact that the organic wool used in the dyeing process had already a more positive coordinate a^* , indicating a yellowish tonality (see colour reconstructions of wool controls in Table I).

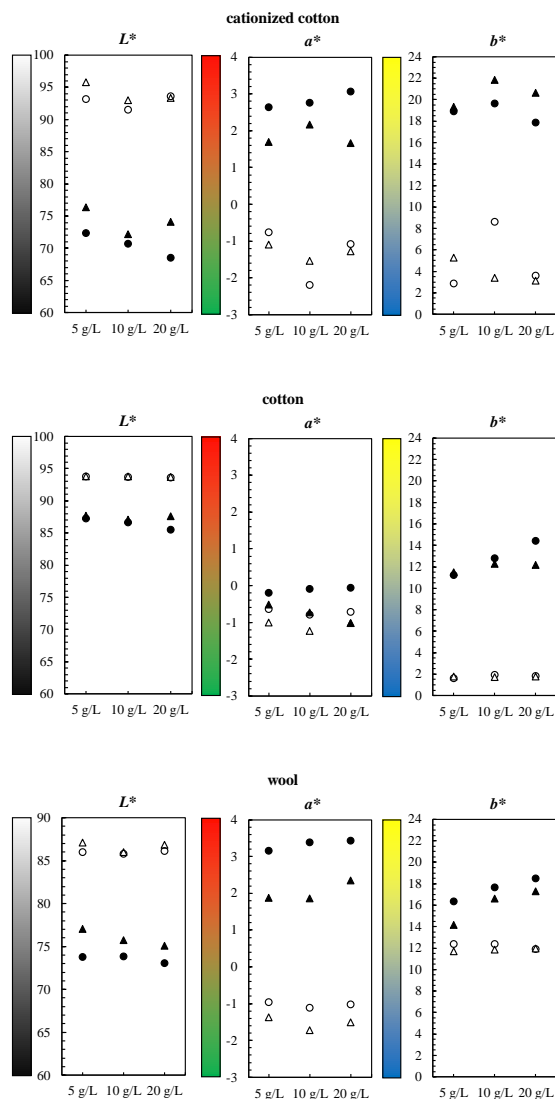


Figure 3: Coordinates $L^*a^*b^*$ measured for control samples (open symbols) and dyed samples (close symbols) for the three textile substrates used: cotton; cationized cotton and wool. The circles refer to extracts obtained at 100 °C and the triangles correspond to cork powder extractions performed at 30 °C.

5 CONCLUSIONS

The replacement of chemical dyes used in the textile industry by natural dyes obtained from bioproducts and residues are an up-to-date subject promoting environmentally sustainable and healthy processes.

The dye solutions used in this study were obtained from cork powder, the main residue in the cork stopper producing process, by means of eco-friendly solvents (water) as extraction agent. Samples of cotton (natural and with a pre-treatment of cationization) and wool remained immersed in the extracts obtained with different

conditions of temperature and cork powder amount. The final colour in the textile substrates was measured after washing (with hot and cold water) and natural light exposure.

A general trend seems to exist to have more intense colours for textiles immersed in extracts obtained with higher amounts of cork powder and higher extraction temperatures. However, this behaviour is more pronounced for the cotton (natural) and wool substrates.

The brownish colours obtained in the dyed fabrics appear to be interesting in a commercial point of view and could be incorporate in an earth tone colours palette.

This innovative process of obtaining bio-dyes from the cork powder residue contributes to respond to the increasing demand for natural dyes in the textile industry, improving the environmental sustainability of this sector. Nevertheless, this topic deserves further exploratory and systematized studies to consolidate improvements in colour/fastness properties.

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8 LOGO SPACE



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