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Usage of Natural Resources in Textile Wet Processes

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Abstract

Natural sources, whether plant-based or animal-based, are considered one of the richest sources that can be utilized in textiles field due to their renewable and sustainability. They are also environmentally friendly, non-toxic, and biodegradable. These natural sources contain numerous compounds that are beneficial for textile wet processing, such as dyeing and finishing. Examples of these compounds include flavonoids, organic acids. Purslane, tomato leaves, and bee propolis are from these natural sources. Previous studies have shown that the extracts from purslane and tomato leaves can be used in wool dyeing and to obtain various types of metal nanoparticles. Bee propolis can also be utilized in medical textile finishing, such as antibacterial, anti-inflammatory and in wound healing.

Key words: Purslane, tomato leaves, Natural sources, dyeing, medical textile finishing.

Introduction

Natural resources are materials from the nature that are used to support life and meet people's needs such as oil, natural gas, metals, soil, water, plants, animals etc... Plants and animals are from the most acknowledged renewable resource due to its richness, and renewability. [1] Plants and animals possess a wide variety of natural materials which has the potential to provide useful features in wet processes for textiles. In plants these useful features can be obtained from the extracts from different parts of plants like roots, stem, leaves, flowers and seeds. [2-22]

Bees, belong to the animal group, produce the propolis to use it for repairing and defending their habitats and creating protection against infections, this propolis also provide useful features in wet processes for textiles. Generally, the reason for giving these beneficial features is because they contain various chemical compounds such as flavonoids, acids (such as phenolic and amino acid), esters, terpenoids and steroids. These chemical compounds have the ability to provide color and different finishing properties for textile [23] Textile dying means the application of dyes or pigments on textile materials such as fibers, yarns, and fabrics. [24] Textile finishing is a method used to enhance existing properties or to impart new properties to the fabric such as anti-microbial finishing, UV-protection water repellent, flame retardant etc..[25] Medical textiles contribute to increase on quality of health service and improve the functionality of textiles used in hospitals. Medical textiles can reduce some medical problems due to infection spreading limitation. In order to prevent from hospital infections, these products can be manufactured with natural-based pharmaceutics materials by considering controversial effects of bacteria causing these problems. [26]

Purslane as an example to plant natural source

Portulaca oleracea is an annual medicinal plant from the family Portulacaceae. Flavonoids, alkaloids, fatty acids, terpenoids, and organic acids are active constituents of P. oleracea. The pharmacological properties of P. oleracea include antioxidant, anti-inflammatory, analgesic and wound healing. [27]

Purslane chemical structure

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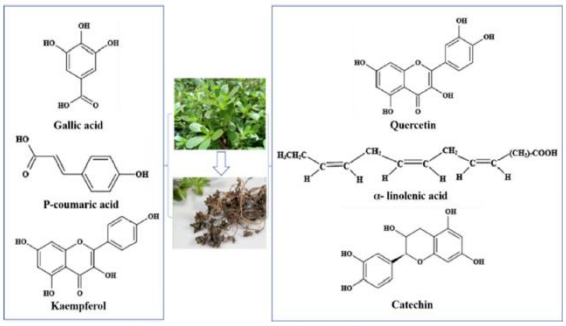
Portulaca oleracea L. contains about 184 compounds including phenolic acids, organic acids, flavonoids, and alkaloids, among them, ơ-linolenic acid, quercetin, p-coumaric acid, catechin, kaempferol, and tannic acid were the primary active ingredients of the observed medicinal properties of leaves. Quercetin is the flavonol responsible for purslane extract gray-yellow color.[28, 29]



The main active ingredients in Portulaca oleracea L

Purslane in textile dyeing

Portulaca oleracea L. plant is used as a natural colorant for wool fabric dyeing with a high color yield at optimum extraction and dyeing conditions. [29]

Extraction of Portulaca oleracea L. colorant

The natural colorant of Portulaca oleracea L. was extracted by water bath method, as the plant leaves were washed, dried then grind to powder. Then the extraction was carried out at different extraction conditions: - temperature (60–100 °C), time (0.5 h–2.5 h), solid (5 g of dried plant) to liquid (50 mL H2O). Then the obtained filtrate was evaporated at room temperature, and the extracted dye powder was stored at low temperature (4 °C). [30-32]

Using Portulaca oleracea L. extract in wool dyeing

The extract was used for dyeing wool fabric. Single factor optimization was used to explore the dyeing parameters, including pH (2–6), temperature (60–100 °C), dye concentration (1:20–1:40), and dyeing time (30–90 min). [30]

It was found that:

- The highest absorbance was at 90 °C temp, 75 min. time, pH 2 and L:R 1:35.
- Wool dyed sample gained brown color.

Undyed wool fabric Fabric dyed with Portulaca oleracea L

- The natural dyed wool fabric (NF) had given accepted K/S and fastness values when compared to the synthetic dyed wool fabric (SF).

Item	Colorfastness to				K/S	L*	a*	b*	C*	h°
	Wash	Dry rubbing	Wet rubbing	Light						
NF	4	4	3–4	3–4	21.1	49	9.63	23.05	24.98	67.33
SF	4-5	4–5	4–5	4–5	21.1	49.24	9.82	22.97	25.11	67.37

Purslane in textile finishing

Portulaca oleracea L. has analgesic, anti-inflammatory and neuro-protective effects and also has strong biological activities in antibacterial, anti-inflammatory, anti-allergic and anti-oxidation. [33] Natural dyes are extensively used in antibacterial finishing, UV protection, and other fields due to their non-toxic, environmental protection, and multi-functional properties including antibacterial, anti-ultraviolet, and insect repellent. [34, 35] It was found that the natural dyed wool fabric (NF) had given acceptable UV-protection and anti-microbial finishing when compared to the synthetic dyed wool fabric (SF).

UV protection performance of dyeing samples.

	Transmittance		UPF values	Antioxidant	Error
	T (UVA)	T (UVB)			
NF	0.47	0.43	229.05	25.3%	4.8%
SF	0.41	0.40	248.92	24.8%	6.4%

Antibacterial activity of fabrics against *S. aureus* and *E. coli*.

S. No.	*S. aureus*	*E. coli*	Antibacterial rates against *S. aureus* / *E. coli*	Error
WF			-/-	-/-
NF			47.8%/12.5%	6.8%/4.5%
SF			25.2%/11.8%	7.2%/4%

Using Purslane to obtain nanoparticles

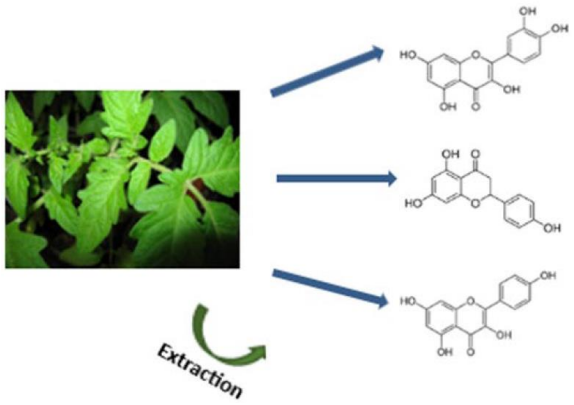
For the green synthesis of metallic nanoparticles, toxic chemicals are not introduced during the synthetic steps, which confer an entirely green route. Natural products such as plant extracts are considered to be effective reductants that also act as stabilizing agents. Plant extracts contain diverse primary and secondary metabolites that offer reducing power to change metallic ions to metallic nanoparticles based on redox reactions. Plant extract-mediated green synthesis has been popular due to its simple synthetic process, amenability to scale-up, economic effectiveness and environmentally friendly nature. [36] Purslane extract can be used as a green reductant to synthesis gold, silver/silver chloride, zinc oxide, cerium oxide and cupper nanoparticles without using any other chemicals. The preparation methods described at [31, 32, 37-39] Natural polyphenol compounds, flavonoids, serves as a main contributor to the green synthesis of metallic nanoparticles using plant extracts. [40]

Tomato leaves as an example to plant natural source

Tomato (Lycopersicon esculentum Mill.) belongs to the Solanaceae family, and it is one of the most widely consumed vegetables in the world. Tomato plants also have a variety of secondary metabolites including terpenoids, phenolic compounds, and glycoalkaloids. These bioactive compounds have been used as antibacterial, antifungal, and antiviral agents. Moreover, bioactive compounds found within tomato plants have been utilized coloring. The waste products include the lower leaves and stems that are regularly removed from the vines to improve the fruit production, and also whole tomato vines that are annually disposed of to landfill. It was speculated that tomato leaf waste represents a rich source of phytochemicals, phenolic compounds, flavonoids, and plant proteins that could be recovered for a broad range of applications. The concept of sustainability is used and applied in many areas in textile production. Natural dyes obtained from plants have been used in the field of textiles as it have ecological advantages such as being recyclable, biodegradable or decomposable in nature and also low cost and harmless to human health. [41]

Tomato leaves chemical structure

It was determined that the flavonoids in the leave structure, from the most to the least, were flavonol class components such as quercetin, naringenin, kaempferol respectively. Flavonol group components give yellow colours to the product they dye. Naringenin, a glycone of naringin and is used as a natural yellow dye in the dye industry, is particularly advantageous with its resistance to washing, abrasion and light. Kaempferol, on the other hand, is a flavonoid that can give a yellow colour and is missing an -OH group at the 3rd position compared to quercetin. [42-45]

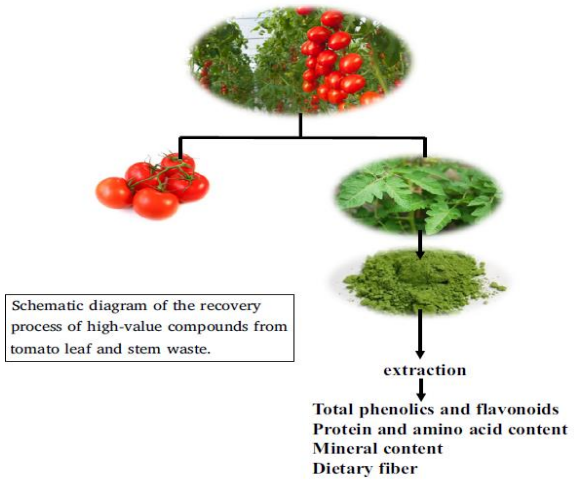


Tomato leaves in textile dyeing

In terms of sustainable production, it is especially important to be able to obtain natural dyes from agricultural and horticultural wastes that cannot be used as food sources. Tomato leaves and stems are one of the waste products that are disposed of in landfills after harvest and are suitable for evaluation in terms of sustainability. These products can be used for dyeing materials after harvest.

Extraction of Tomato leaves colorant

Aqueous extraction method was used to obtain the coloring extract from tomato leaves. Tomato leaves were collected and washed with distilled water. The leaves were kept in the oven at a temperature not exceeding 50 °C until they were dried, after that the leaves were grounded to powder. 10g of the obtained powder was boiled with 100 ml distilled water for 60 min at 80-100˚C, then the obtained yellow color aqueous extract was filtered then stored at 4˚C for further use. The extract pH value was in the range of '5-5.5'. [46]



Schematic diagram of the recovery process of high-value compounds from tomato leaf and stem waste.

Using Tomato leaves extract in wool dyeing

The wool samples were dyed with the prepared extraction by using infra-red dyeing method at a 1:20 bath ratio and dye concentration 20 g/l. Wool fabrics were dyed both without mordant and with three different mordant added dyes, separately. Mordant substances with concentration 1% (aluminium sulphate ($Al_2(SO_4)_3$, iron(II) sulphate heptahydrate ($FeSO_4 .{}_7H_2O$, copper(II) sulphate pentahydrate ($CuSO_4 .{}_5H_2O$) were added in the dye bath by mixing them with the dye extracts. The dyeing process started with 50°C degree and then increased to 90°C(20 min), then the machine actuated 40 min at 90°C; and then at a decreasing temperature 90 °C to 50°C for 20 min. When it reached 50 °C, the machine was stopped and the tubes were removed. Samples rinsed with cold water. It was found that:

- The dyed wool sample with tomato extract had yellowish-beige colours.
- Yellow tones were obtained when aluminium potassium sulphate was added to the dye bath.

- Brown tones were obtained when iron sulphate was added to the dye bath.
- Green-khaki tones were obtained when copper sulphate was added to the dye bath.
- When the mordant is not used in the extracts, the colour fastness of the samples was 4/5 and 5 values which are good levels.

Using Tomato leaves extract to obtain nanoparticles

Tomato leaves extract can be used as a green reductant to synthesis iron oxide nanoparticles (FeONPs) and Copper oxide nanoparticles (CuONPs) without using any other chemicals. The preparation methods described at [46, 47] Natural polyphenol compounds, flavonoids, serves as a main contributor to the green synthesis of metallic nanoparticles using plant extracts. [40]

Propolis as an example to animal natural source

Propolis is bee glue, collected by bees from various plant sources. The propolis is used by bees as a glue to seal the opening of the hives and to eliminate outside invaders. [48] Propolis extractions can be successfully used in textile medical finishing of woven and nonwoven fabrics such as cotton, polyester, polypropylene and tencel such as lyocel. [26, 49-52]

Propolis chemical structure

Propolis consisted of plants exudates, bees' salivary secretions, and beeswax, which make propolis a resinous and strongly adhesive natural substance. [53] Propolis main construction is polyphenolic compounds. The main polyphenols are flavonoids, accompanied by phenolic acids and their esters, phenolic aldehydes, steroids, alcohols, amino acids, ketones and approximately 180 flavonoids which were believed to confer the antibacterial effects of propolis. [54] Propolis is generally consists of 50 % resin, 30 % wax, 10 % essential and aromatic oils, 5 % pollen, 5 % other organic compounds, and mineral substances. [48]

Compounds	No. of kinds
Flavonoids	180
Hydroxy flavon	27
Minerals	22
Amino acids	17
Sinamil and sinamic acid derivatives	14
Benzoic acid and its derivatives	12
Hydroxy flavonones	11
Sekuterpen and triterpen hydrocarbons	11
Acids	8
Alcohols, ketones, phenols	8
Sugar	7
Sterols and steroid hydrocarbons	6
Esters	4
Chalcocites	2
Benzaldehyde derivatives	2

Propolis extraction methods

Propolis as a soluble extract [55]

While difficult to resolve in organic solvents, propolis easily dissolves in alcohols such as ethyl alcohol (ethanol), and propylene glycol.

1- Ethanolic extracts of propolis (EEP) were prepared by adding 2 g of propolis to 25 ml of (10-95%) ethanol in test tubes.

2- The test tubes were shaked at 70°C for 30 min, then after extraction, the mixture was centrifuged to obtain the supernatant. It was found that:-

- The appearances of the 10 to 50% EEP presented a yellow color and the 60% EEP was greenish yellow, and that as the percentage of ethanol increased this green color became deeper and deeper.
- Measurement of the absorption spectra of the propolis extracts showed that the absorptions were increased with increasing concentration of ethanol up to 80%, as the highest concentration of flavonoids were liberated from the propolis when using 80% ethanol.
- The extracts with 90 and 95% ethanol showed decreasing absorption.
- The 70 and 80% EEP demonstrated the greatest antioxidant and antibacterial activity.

Antimicrobial Activity of Ethanolic Extracts of Propolis to *Staphylococcus aureus*

Extracts of propolis	Zone of inhibition of microbial growth (mm)
10% ethanol	0
20% 〃	0
30% 〃	0.5
40% 〃	1.0
50% 〃	1.0
60% 〃	1.5
70% 〃	1.5
80% 〃	1.5
90% 〃	1.0
95% 〃	1.0

Propolis as a powder extract [52]

- Approximately 100 g of propolis were stored immediately in an ice box at 4°C for one week.
- Propolis was then dehydrated further under vacuum.
- The dried propolis was ground to a fine powder.

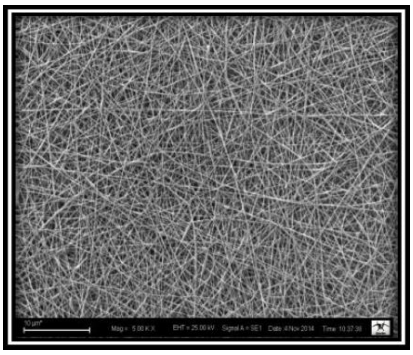
Propolis application methods on textiles

It is found that ethanolic propolis extracts can be directly applied to the fabric by conventional padding method. [49, 52] It is also found that the extracts can be applied to the fabric by other different methods as shown:-

Propolis electro spinning method [26]

- PVA polymer was dissolved in distilled water at 60 °C during 8 hours.
- Propolis was diluted with ethanol by concentration of 3% in weight.
- 3% propolis/ethanol solutions were added into PVA/water solutions drop by drop, and then the mixtures were stirred during 15 hours at room temperature.
- Homogenized solutions were electro-pun onto PP nonwoven surface with a potential differences between the tip and the counter electrode (collector) used to electros-pin the polymer /propolis solutions were 37 kV.
- Stationary collector covered with aluminum foil, placed 10 cm above the capillary tip, was used to collect the electro-spun fiber material. Feeding rate was under controlled during manufacturing and it was adjusted to 2.5 ml/h.

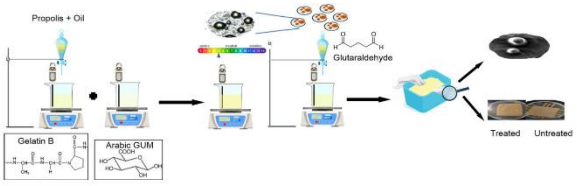
According to SEM micrographs, it is seen that propolis extract can be electro-spun by adding into PVA polymer solution.



Propolis encapsulation method [50]

Microcapsules containing propolis are synthesized by using gum arabic and Type-B gelatin according to complex coacervation method and glutaraldehyde is applied as crosslinking agent. Microcapsules were prepared via double emulsion method by using rice oil. Microcapsules were obtained from 10–30 ml propolis ethanolic extract, 30 ml rice oil, gum arabic, gelatin, acidic acid, sodium hydroxide and glutaraldehyde.

- Propolis emulsion was prepared by stirring rice oil with propolis extract at 1500 rpm.
- Gelatin and arabic gum solution were prepared by stirring gelatin and arabic gum at 40 °C in distilled water at 1500 rpm.
- At room temperature, propolis emulsion was added drop by drop in gelatin and arabic gum solution at 1500 rpm solution for 2 h, then glutaraldehyde in was added into the solution at 500 rpm at pH 4.
- Then, pH of the solution was increased up to pH 9 by adding NaOH to the solution and encapsulation process was terminated.
- 5% solution of microcapsules obtained was diluted with distilled water and the pH of the solution was set as 9 by NaOH, and an acrylate based commercial cross linker was added.
- The fabric was padded in the prepared liquor to transfer microcapsules then it was squeezed to a wet pick up of 85%, and then dried at 110 °C for 30 min.



It was found that:-

- After ten washing cycles, however inhibition zone disappeared, and bacterial growing is not observed on the microcapsule treated cotton fabrics. This is probably due to partly removal of microcapsules on the

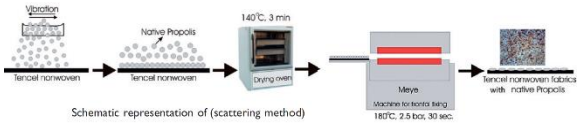
surface of cotton fabric after washing processes.

- The cotton fabrics treated propolis loaded microcapsule showed excellent antibacterial activity towards S. aureus and K. pneumonia microorganisms.

Scattering propolis method [52]

Propolis powder can be applied to the fabric by scattering method.

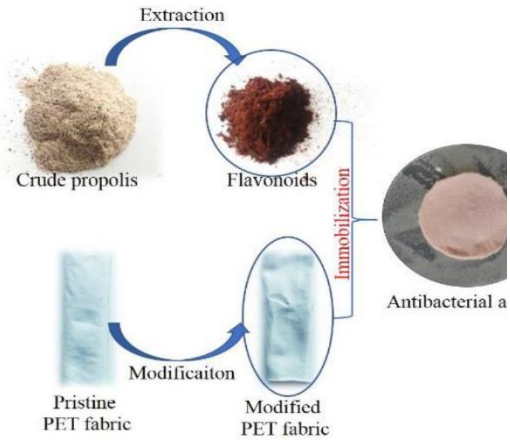
- Propolis powder was scattered by (125 g/1m^2) on the wet samples after squeezing in a uniform layer.
- The samples were dried at 140 °C for 3 min.
- The samples were inserted into the fusing machine at 180 °C for 30 s and 2.5 bar.



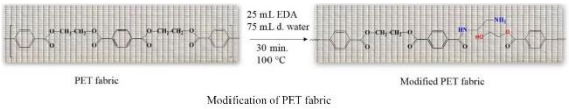
Schematic representation of (scattering method)

Fabric modified by amination method [51]

Phenols extracted from propolis were covalently bonded and immobilized on the surface of polyethylene terephthalate (PET) fabric. To do so, PET fabric was aminated, and then the phenolics were immobilized using polyethylene glycol diglycidyl ether (PEGDGE) as the crosslinking agent.

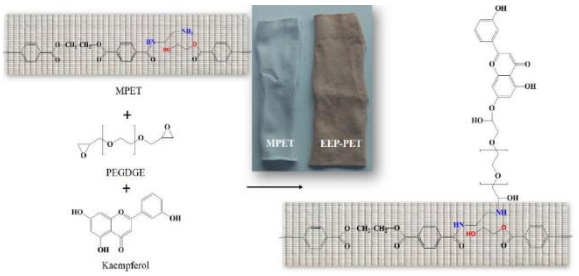
- 

- A mixture of 25 mL ethylenediamine (EDA) and 75 mL distilled water was prepared.
- A piece of PET fabric in dimensions of 5 cm x 15 cm was put in the mixture in a tightly closed glass bottle containing a magnetic bar.
- Then the bottle was placed in a water bath and stirred at 100 °C for 30 minutes on the hot plate.
- 4- Then, the heater was turned off, the temperature was allowed to decrease to 50 °C and the modified PET (MPET) was taken out.
- 5- The MPET thoroughly washed with distilled water and dried at 75°C in an oven.



Modification of PET fabric

- 6- EEP was Immobilized by utilizing amine groups of MPET, Poly(ethylene glycol) diglycidyl ether (PEGDGE) was used as crosslinking agent between MPET and EEP.
- 7- 1 gram of EEP was added to 75 ml tetrahydrofuran (THF), which is inert against the reactants, and stirred for 5 min.
- 8- MPET with 5 cm x15 cm dimensions was taken into the EEP solution.
- 9- 4 ml of PEGDGE was added dropwise and the reaction mixture was stirred for 1 hour at 25 °C.
- 10- The EEP immobilized PET fabric (EEP-PET) was washed with excess amount of distilled water and dried at 75°C after for 24 hours.



Immobilization of EEP on MPET (Kaempferol which is one of the EEP content was used as a representative). Inset is pictures of MPET and EEP-PET.

Propolis application as a hydrogel [56, 57]

The usage of hydrogels is one of the methods used to improve washing resistance and bring many functional properties in textile. Hydrogels is that they keep the wound site moist due to their high-water retention capacity, support wound healing, and allow the healing process to be monitored due to lack of their color. Hydrogel-coated textile materials are the preferred wound dressing after many surgical procedures due to their excellent properties. [58]

- 1- PVA transparent solution was obtained by adding 4% PVA to distilled water then stirring it at 90 ˚C for 2 hours at a stirring speed of 1500 rpm.
- 2- NaCMC solution was obtained by adding 4% NaCMC to distilled water then stir-

ring it at room temperature for 30 minutes at a stirring speed of 1500 rpm.

- 3- NaCMC solution was added drop wise to the PVA solution and the mixture was stirred at the same speed for another 2 hours.
- 4- Different amounts of propolis ethanolic extract (1, 2, 5, 10, 20 and 30 ml) were added to the resulting stock solution to form hydrogels.
- 5- For crosslinking of each solution on the fabric, 5% glutaraldehyde was added and stirred at 500 rpm for 24 hours, the hydrogel solutions ready for padding were obtained,
- 6- After all, the fabric is padded in the hydrogel solutions after setting the PH at 9 with NaOH for 10 minutes and then squeezed with a pick-up of 85%.
- 7- Finally the samples were dried at 110 ºC for 30 min in the oven.

And it was found that the antibacterial activity of the treated fabric increase by increasing the propolis ethanolic extract concentration.

Propolis medical finishing

Propolis is a well-known antibacterial agent with high ability to facilitate the wound healing process. [59] It is known that the ethanolic extracts of propolis have various pharmacological activities such as antibacterial, antiviral, an- tifungal, anaesthetic, anti-inflammatory, hypotensive, immunostimulatory, and cytostatic properties. Therefore, the medical applications of propolis preparations have led to an increased interest in its chemical composition as well as to its origin. [54] Propolis exhibites antifungal, antiviral, anti-inflammatory properties, as several studies have reported. Moreover, it has a considerable success to enhance the wound dressings' efficacy in different aspects. [59]

anti- bacterial finishing

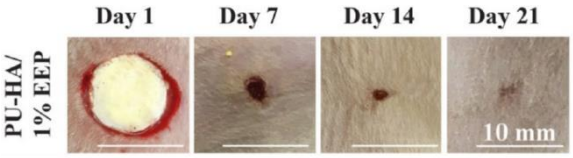
Propolis has approximately 180 flavonoids which were believed to confer its antibacterial effects. In addition, an increase of the ratio of phenyl propanoids over caffeic acid derivatives was associated with higher antibacterial activity. [49]

Zone of Inhibition (mm)			
Staphylococcus aureus, ATCC 25923	*Escherichia coli,* ATCC 25922	*Klebsiella pneumoniae* NRLLB4420	*Candida albicans*
9.3 ± 0.09	10 ± 0.9	11.66 ± 0.39	4.3 ± 0.1

Propolis is successful in producing textile products with anti-bacterial characteristics as it is able to inhibit the microbial cell division. The effective compounds in this are flavonoids, cinnamylidene acetic acid, benzyl p-coumarate and caffeic acid esters such as pinocembrin, galangin and pinobanksin. [60] Propolis is highly active against both gram-positive and negative bacteria, but for gnerally speaking, gram-positive bacteria were more sensitive to propolis than were gram-negative bacteria. [49, 50]

wound healing

The natural wound healing process in the body involves multiple biochemical and cellular reactions, which occur in four successive main stages: hemostasis (coagulation), then inflammation, followed by proliferation of new skin tissue, and finally remodeling of mature tissue. [61] Propolis was rich with flavonoids, phenolic acids and CAPE (caffeic acid phenethyl ester) therefore propolis is now being used for dealing with fungal, bacterial and viral infections as it has a healing effect on skin wound, burning wound, skin inflammation and other skin diseases. [62, 63] Due to its excellent antiseptic efficacy and antimicrobial properties, propolis has shown attractive advantages in wound dressings and skin wound, burns, and ulcers healing process and tissue repairing. [61, 63] The use of propolis in textile materials can make wound healing faster as it enable to produce bioactive materials for skin care that speed up cell proliferation. [64] Caffeic acid (ranged from 0.27% to 2.67%) in propolis extracts also proved to have potent antioxidant and anti-inflammatory activities, leading to wound healing. [52, 61]



Recommendations

Based on the aforementioned, we recommend the necessity of focusing and conducting further studies on the use of purslane, tomato leaves, and bee propolis as rich natural sources of flavonoids and beneficial organic acids in textile wet processing, whether in dyeing or finishing. This is to align with the trend towards sustainability and environmental conservation by reducing liquid waste from traditional wet processing methods.

Conflict of Interest

There is no conflict of interest in the publication of this article.

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