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Ultraviolet protection and antibacterial properties of silk fabric dyed with *Cinnamomum camphora* plant leaf extract

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In this study, natural dyes have been extracted from camphor plant (*Cinnamomum camphora*) dry leaves in alkaline medium and are then applied on to silk fabric by exhaust dyeing process. The UV protection, antimicrobial property, color strength values and colorfastness of the silk dyed with plant extract are studied. The results show that the fabric samples treated with extract have excellent UV protection properties and are very effective in blocking UVA and UVB radiations. The fabric dyed with natural extract shows antimicrobial properties, which is proved by bacterial reduction in quantitative tests. The color fastness to washing and rubbing is found very good to excellent and color fastness to light is poor. Silk fabric dyed for less time shows golden color, while the increased dyeing time shows reddish-brown color. The UV protection and antimicrobial performance of camphor plant leaf extract are found excellent.

Keywords: Antibacterial property, *Cinnamomum camphora*, Silk fabric, Ultraviolet protection

The dyes/colorants obtained from natural sources like plants, animals and minerals are termed as natural dyes[1]. Synthetic dyes are more toxic[2], non-biodegradable, allergic, and not environment friendly as compared to natural dyes. Natural dyes are consumed 10,000 tonnes per year which is equal to 1% of the total world synthetic dyes consumption[3, 4]. Petrochemical source is the origin of synthetic dyes and some of these dyes contain harmful carcinogenic amines[5]. Germany has banned the production of azo dyes because of their severe harmful effects on living organisms[6]. Natural dyes of some plants not only dye with unique and elegant colors, but can impart antibacterial, deodorizing and ultraviolet (UV) protective properties to fabrics[7].

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Camphor can be extracted from the wood of camphor laurel (*Cinnamomum camphora*) and other plants belonging to laurel family. It is a white, crystalline substance possessing pungent taste andstrong odor. Camphor tree is native to China, India, Mongolia, Japan, Taiwan and a specie of this class is also grown in Southern part of United States[8]. Camphor is a magic plant having many applications in pharmaceutical field, for example nasal decongestant, cough suppressant, topical analgesic, antiseptic, antispasmodic, antipruritic, anti-infective, anti-inflammatory, rubefacient, contraceptive, mild expectorant, etc[9]. The camphor is the main component found in the leaves of *Cinnamomum camphora* and minute quantities of cineol, linalool, eugenol, limonene, safrole, α-pinene, ß-pinene, ß-myrecene, α-humulene, p-cymene, nerolidol, borneol, and camphene are also obtained[8].

A white crystalline ketone (2-bornanone)[10] is known as camphor, found commonly in all parts of the *Cinnamomum camphora* tree. Traditionally, camphor has been used for the treatment of chest congestion and inflammatory diseases like rheumatism, sprains, bronchitis, asthma, and muscle pain[11]. Camphor is used for activation of some of TRP (transient receptor potential) producing warm sensation, the sensory nerves are excited and desensitized, relieving the pain, itch and irritation in applied area[11]. In radiotherapy camphor can be used as potential radio sensitizing agent[12]. A drug 714-X based on camphor is used for the treatment of some breast and prostate cancer patients[13]. A herbal preparation known as Padma 28 is based on camphor which is effective in treatment of chronic inflammatory diseases[14]. Atrazine (AT), a commonly used herbicides, causes DNA damage and biochemical changes in mice, which can be encountered by using *Cinnamomum camphora* leaves extract (CLE)[11]. The two components found in seeds of *Cinnamomum camphora*, namely cinnamomin and camphorinhaveshown their inhibitory effect against cultured carcinoma cells[15, 16]. Camphor containing plants are expected to play a vital role innutraceutical and pharmaceutical applications in future[17, 18].

The major environmental cause for skin damage is due to its exposure to ultraviolet radiation (UVR). UVB radiations are more harmful as compared to

UVA radiations. Long term UVB exposure to human skin creates wrinkles, and an increased risk of epithelial skin cancer[19]. Ultraviolet protection factor UPF is the protection provided by a material against UVR[20].UVRs can be blocked by using adequate clothing materials and the level of protection offered by them depends on various factors such as fiber chemistry and physics, yarn properties, fabric construction, weight, thickness and cover factor, its color and finishing treatments[21, 22]. Beside UV protection, many natural dyes obtained from various plants are known to have antimicrobial properties[15-23]. In the present study the ultraviolet protection and antimicrobial properties of silk fabric dyed with camphor plant leaf extract have been investigated.

Experimental

Materials

Silk fabric (72 g m^{-2}, plain weave) commercially degummed and bleached was used in this study. Camphor plant dry leaves were supplied from a botanical garden of Wuhan Textile University, Wuhan, Hubei, China. Laboratory grade caustic soda (NaOH) was used for the extraction of dyes from camphor plant leaves. Acetic acid (CH_3COOH) was used for maintaining *p*H during dyeing. A detergent (Alconox) was used for soaping off silk fabric after dyeing.

Instruments

Gyrowash machine (James H. Heal & Co. Ltd., England) was used to carry out the dyeing processes. Ultraviolet protection property of dyed fabrics was analyzed using Spectronic Camspec M550 double beam scanning spectrophotometer. IR spectrum of the powder form of plant sample was obtained using Perkin-Elmer System 2000 FT-IR Spectrometer.

Extraction

Green leaves of camphor plant were air dried in shade. Air-dried materials (20 g) of leaves wereground to a fine powder with an electric grinder. This powder was extracted with 0.8% NaOH solution (1000 mL) for 80 min at 95°C, with continuous agitation. The cooled alkaline extract was filtered through Whatman No. 1 paper (Whatman International Ltd, Maidstone, UK) and the filtrates were collected and cooled to room temperature. The filtrate was converted to fine powder with the help of spray evaporator. Extraction ratio was calculated using the following equation:

$$\text{Extraction ratio} = \text{ER} = [(W_0 - W_1)/W_0]\times 100\% \quad \ldots(1)$$

where W_0 is the weight of camphor plant leaves before extraction; andW_1, the weight of camphor plant leaves after extraction.

Dyeing of Silk

Powdered plant extract was applied on the bleached silk fabric by making their dispersion in water, on laboratory scale Gyrowash dyeing machine.

To investigate the effect of dyeing liquor ratio, silk fabric was dyed at six different liquor ratios. Camphor plant leaf extract was used 5% owf. The samples were dyed at 95°Cfor 60 min at *p*H 4 with different liquor ratios (1:10, 1:15, 1:20, 1:25, 1:30, and 1:35). Samples were washed off using detergent 2 g/L for 10 min at 70°C.

Silk fabric wasalso dyed in six sets of 5%owf of the camphor plant leaf extract at 95°C usingliquor ratio of 1:25 and *p*H of 4 for different time intervals (30, 45, 60, 75, 90 and 105 min). Samples were washed off using detergent 2 g/L for 10 min at 70°C.

Ultraviolet Protection Test (UPT)

AATCC 183 standard testing method was used for testing UPF of the conditioned silk samples. A double beam scanning spectrophotometer (Spectronic Camspec M550) with integrated sphere havingthe photometric range of 0-200% transmittance and photometric accuracy greater than 0.5% at 0.5A, 1A and 2A was used to determine UPF. UPF of the fabric can be determined by taking the ratio of irradiance (at a detector) of erythmally weighted UV without a specimen and also with specimen. The per cent blocking in UVA and UVB regions can bedetermined using the following equations:

$$\%\text{ UVA blocking} = 100 - \left(\frac{\sum_{315\text{ nm}}^{400\text{nm}} T.\Delta\lambda}{\sum_{315\text{ nm}}^{400\text{nm}} \Delta\lambda}\right) \quad \ldots(2)$$

$$\%\text{ UVB blocking} = 100 - \left(\frac{\sum_{280\text{ nm}}^{315\text{nm}} T.\Delta\lambda}{\sum_{280\text{ nm}}^{315\text{nm}} \Delta\lambda}\right) \quad \ldots(3)$$

Antibacterial Test

Bacterial Strains

The antibacterial activity of silk fabrics dyed with natural colorant from camphor plant leaves was investigated using AATCC 100-2004 test method. The test was carried out with *Staphylococcus aureus* (a grampositive bacterium), *Escherichia coli* (a Gram negative bacterium), and *Candida albican* (a Gram positive fungus). These three organisms are reference strains used for antimicrobial susceptibility testing. The strains were cultured on nutrient agar (Wuhan, Hubei, China) and incubated aerobically at 37°C overnight.

Percentage Reduction Test (AATCC 100- 2004)

The bacterial suspensionof a known concentration and specimens of test material were shaken, and the reduction in bacterial activity in standard test time (18 h) was measured. The efficiency of antimicrobial treatment was measured by calculating the reduction in bacterial concentration of treated sample with that of control sample expressed as a percentage reduction in standard test time, as shown below:

$$\% \text{ Reduction} = [(A - B)/A] \times 100 \quad \ldots (4)$$

where A and B are the surviving bacterialcells (CFU/mL) for the flasks containing control (blank silk fabric) and test samples (natural dye treated silk fabric) respectively.

Colorfastness

Color fastness to washing, light, and crocking of the dyed samples were measured according to ISO 105 C06-2010, ISO 105-B02: 1994, and AATCC Test Method 8-2007 respectively.

FTIR Study

The powder form of camphor plant leaf extract was analyzed by FTIR for identification of possible functional groups present in it.

Results and Discussion

The amount of dye powder obtained from 20 g leaves of camphor plant is calculated usingthe following formula:

$$\text{Extraction ratio (ER)} = [(W_0 - W1)/W_0] \times 100$$
$$\text{ER} = [(20 - 14.5)/20] \times 100 = 27.5\%$$

The ability of the plant to block or absorb UV radiations has been studied under UV spectrophotometer. The absorption percentage is found maximum at wavelength of 200-220 nm and goes down to 0 as the wavelength increases.The mean UPF and blocking of UV radiations by camphor plant extract dyed fabric samples in UVA and UVB regions are shown in Figs 1 and 2.

It is found that the fabric samples treated with camphor plant leaf extracts show excellent UV-blocking capabilities. Even at very low-application time and liquor ratio, the treated fabrics show tremendous UV-blocking properties. The UPF at dyeing time DT=20 min is considerably poor but as the dyeing time increases the UPF increases tremendously giving its maximum value at DT=60min as shown in Fig. 1(a). Although the UPF at liquor ratio of 1:10and 1:15 is observed to be moderate but as the liquor ratio is increased from 1:15 to 1:20 and so on, the UPF shows tremendous increase, giving its maximum value at liquor ratio LR = 1:30 [Fig. 1(b)]. The UPF results are much higher than that provided by commonly used synthetic UV protective agents. Camphor plant leaf extracts may be used to get excellent UV protection for fabrics.

As shown in Fig. 2, fabrics dyed with the plant extracts resultsin higher protection in UVB region than in UVA region. UVB is more dangerous than UVA radiations, and hence the main requirement of any UVR blocker is to block UVB radiations which cause greater erythmal damage and have more weightage in calculating UPF. Silk Fabrics dyed with camphor plant leaf extracts show tremendous blocking in UVB region thanin UVA region. The presence of flavonoids and phenolic contents in the camphor plant leaf extract contributes for providing UV-blocking properties. The flavonoids and phenolic compounds have been found to resist the plant tissues from damage caused by UV radiation during their growth. This is because of strong absorbance of UV

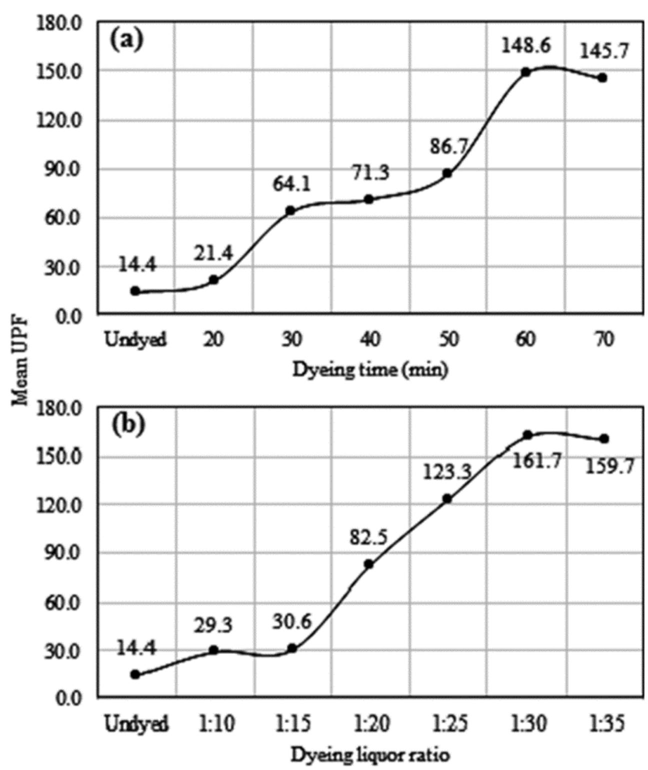

Fig. 1 — Mean ultraviolet protection factor (UPF) of fabric samples treated with camphor plant leaf extract (a) effect of change in dyeing time and (b) effect of change in liquor ratio

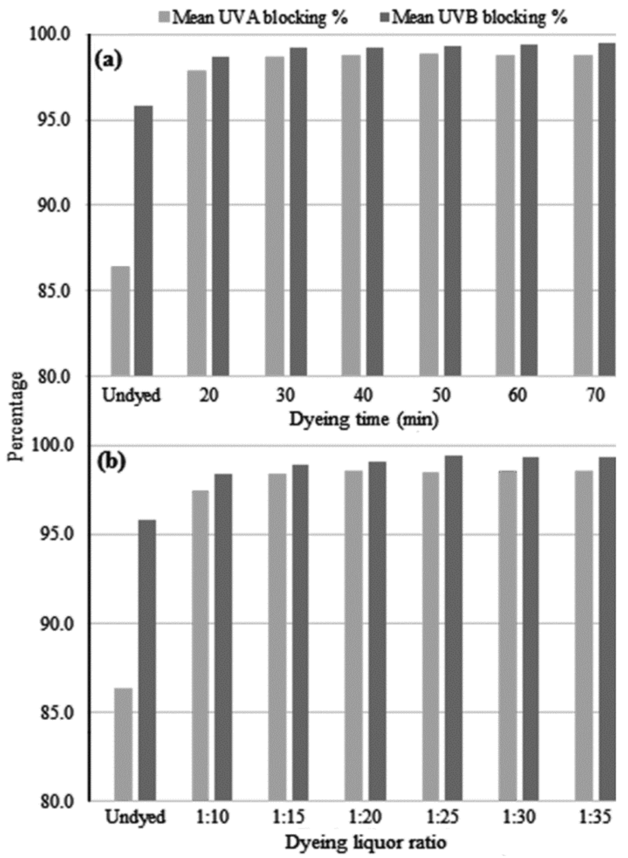

Fig. 2 — Blocking of UV radiations in UVA and UVB regions by camphor plant leaf extract (a) effect of change in dyeing time (b) effect of change in liquor ratio

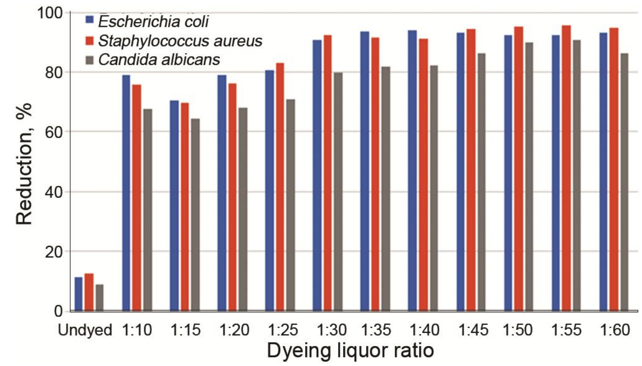

Fig. 3 — Effect of dyeing liquor ratio on the bacterial reduction % of *E. coli*, *S. aureus* and *C. albican*

Table 1 — Bacterial inhibition percentage for dyed and undyed silk fabrics

Sample	Dyeing conditions	*E. coli*	*S. aureus*	*Candida albican*
Standard	Undyed	11.2	12.6	8.8
1	L:R 1:10	78.9	75.7	67.83
2	L:R 1:15	70.5	69.7	74.5
3	L:R 1:20	78.8	76	78.65
4	L:R 1:25	80.7	82.9	81.83
5	L:R 1:30	90.6	92.3	85.65
6	L:R 1:35	93.7	91.4	87.34
7	L:R 1:40	94	91	89.87
8	L:R 1:45	93.2	94.5	94.01
9	L:R 1:50	92.5	95	96.15
10	L:R 1:55	92.3	95.4	95.89
11	L:R 1:60	93	94.6	94.43

radiations (particularly UVB radiations) by these compounds. The undyed samples show very little resistance to UVR in the range of UVA and UVB. The UVB resistance of undyed sample is satisfactory but the resistance to UVA is very low. The dyed samples show tremendous increase in blocking UVA to excellent extent and UVB is improved from satisfactory to excellent. The DT=30 min and liquor ratio of 1:15 are found to be sufficient to impart excellent UVA and UVB blocking property to silk fabric.

In the test against *E. coli*, *S. aureus* and *C. albican* in AATCC bacteriostasis broth, the inoculated control and test fabrics were evaluated for percentage bacterial reduction by cell counting. The results of the percentage reduction test are shown in Fig. 3 and Table 1. The reduction percentage for *E. coli*, *S. aureus* and *C. albican* correspond to the bacterial numbers on the respective control test of per milliliter. The reduction percentage for undyed silk sample is found to be 11.2 % for *S. aureus*, 12.6 % for *E. coli* and 8.80% for *C. albican*. The increase in liquor ratio gives more reduction in bacteria. The maximum value of reduction percentages for *E. coli*, *S. aureus* and *C. albican* are 94 % at L: R 1:40, 95.4 % at L: R 1:55 and 96.15% at L: R 1:50 respectively as shown in Table 1. It has been attributed by some researchers[24] that the bacterial inhibition is due to the gradual release of some active substances from the surface of fabric. The silk fabric dyed with camphor plant leaf extract shows excellent reduction in bacteria which may be attributed to the presence of ß-myrecene, α-humulene, p-cymene, nerolidol, cineol, linalool, eugenol, limonene, safrole, α-pinene, ß-pinene, borneol, and camphene found in its leaves[8].

The effect of liquor ratio on *K/S* values has also been studied in this research work. The maximum dye uptake is observed at liquor ratio 1:25. The liquor ratio is increased from 1:10 to 1:35 to study the effect. At liquor ratio of 1:25, the dye exhaustion reaches equilibrium, then there is no significant increase in dye uptake by further increase in liquor ratio. A longer dyeing time results in higher color strength (*K/S* values) until dye exhaustion reaches equilibrium, and there is no significant increase after further increases in dyeing time. At equilibrium state the majority of ionic sites is occupied by colorants and there are very little chances for further attachment. The optimum time for dyeing the silk fabrics is obtained as 60 min.

The fastness rating of silk fabric dyed with variation in dyeing time and liquor ratio is indicating that the washing fastness rating of silk fabric dyed with camphor plant leaf extract is very good to excellent (4 to 4-5). A probable explanation for the good wash fastness property is the presence ofphenolics and flavonoids in the leaf extract.The light fastnessis found poor to fair which is attributed to the low photo stability of the extract. The wet rub fastness and dry rub fastness are found good and excellent respectively.

The dye extract shows excellent UPF, UVA and UVB blocking when applied on silk fabric. Camphor plant extract gives excellent results in UV protection properties due to the presence of camphor, cineol, linalool, eugenol, limonene, safrole, α-pinene, ß-pinene, ß-myrecene, α-humulene, p-cymene, nerolidol, borneol and camphene in its leaves. The extractimparts excellent antibacterial activity against all three test organisms used, namely *E. coli*, *S. aureus* and *C. albican*. Color strength values and overall colorfastness valuesare found very good. It is concluded from the results that utilizing extracted natural colorants as dyeing materials significantly facilitates production of quality textiles having good dyeability, ultraviolet protection and antibacterial properties.

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