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Near-infrared green camouflage of cotton fabrics using vat dyes

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Abstract: In order to match the reflectance profile of the greenish leaf at NIR region, four commercially available vat dyes were used to dye cotton fabrics. The reflectance of dyed fabric and the transmittance of dye liquor in alcohol solution were measured by using U-4100 spectrometer with an integrating sphere. The effects of the combination dyeing, dyeing concentration (% owf) and fabric weaves on the reflectance were also studied. The results show that the reflectance of the combination dyeing is determined by one of the dyes whose reflectance curve emerges at longer wavelengths. Vat Blue 13 plays an important role in green camouflaging. When the dyeing concentration of vat blue 13 is 1–2% owf, the reflectance of the dyed fabric almost overlaps that of greenish leaf in red shift region (region *A*). Fabric weave has a little effect on the reflectance for the same dyeing program.

Key words: Camouflage, cotton fabric, near-infrared, reflectance, vat dye

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INTRODUCTION

From a defensive standpoint, there is constant research to find sophisticated means of deceiving the military observer, i.e., to make personnel less visible, whether in daylight or by night. Concealment from daylight visual surveillance is a long-established practice in which different hues are employed to overcome the contrast of an object and its surroundings (Zhang, 2002). The hues found in most camouflage patterns are green, olive, khaki, brown, and black. However, with the advent of NIR surveillance equipments, such as night-vision devices, the image intensifier and NIR photography, it became necessary to take into account the IR reflectance of paints and garments in the 700–1300 nm waveband (Wang, 2003). It therefore became necessary for camouflage patterns to be painted or printed with selected dyes and pigments having NIR reflectance that matched the expected surrounding topography (Gupta *et al.*, 2001). For military personnel, it is necessary to wear the appropriate clothing that attempts to minimize the contrast between objects and their environment over the NIR region in addition to the visible region (Burkinshaw *et al.*, 1996). Results show that the NIR camouflage properties of dyed fabric have relations with the constitution of dyes to a great extent. Different fabrics can be dyed by using a variety of dyes. The same fabric can also be dyed by using different dyes to claim the requirement of NIR camouflage (Coleman, 1974). Given the sensitive nature of research into NIR camouflage, it is unsurprising that much of the work on the synthesis, formulation and application of colourants remains confidential.

In this paper, the reflectance profiles of different greenish leaves were measured at first, and then cotton and PET/cotton fabrics were dyed by using four vat dyes to resemble the characteristics of greenish leaves. The reflectance of dyed fabric in NIR region was studied. The effect of dyeing concentration and fabric weaves on the reflectance was also investigated.

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THE REFLECTANCE CHARACTERISTICS OF GREENISH LEAVES

It is obvious that the reflectance curve of different vegetation varies a little in the visible and NIR wavebands. Even for the same vegetation, the reflectances are also different during its growing phase (Zhang, 2002). Hence, the typical reflectance profile of greenish foliage (chlorophyll) is generally selected as a standard reference which we called the standard reflectance curve. We chose several different species of fresh greenish leaves and measured

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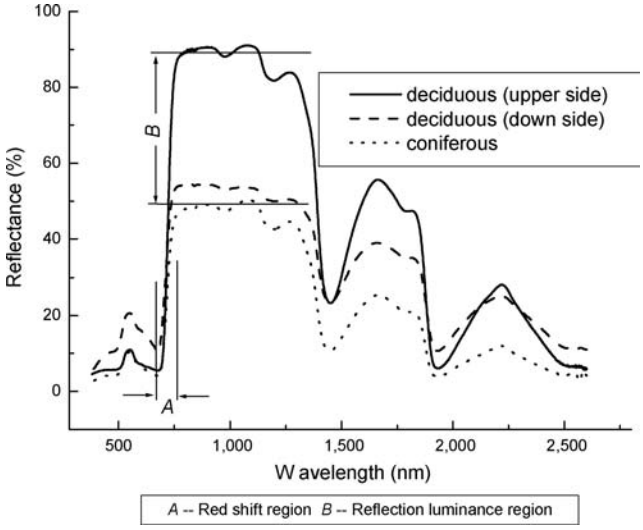


Figure 1 Reflectance curves of greenish leaves.

their reflectance curves, as illustrated in Figure 1. Only three reflectance curves are given (the deciduous tree leaf comes from *Populus tomentosa*, the coniferous needle belongs to *Cedrus deodara*), whereas other reflectance curves are within the scope of them. It can be seen that the reflectance curves have a characteristic shape bestowed by chlorophyll. They have a small increase in reflectance at around 540 nm, which is responsible for the green coloration. Reflectance then decreases slightly in the red region and rises steeply between 670 and 760 nm to a plateau at 780 nm, here we call it (the 670–780 nm waveband) the reflection red shift region (region *A*). At longer wavelengths, the reflectance remains fairly constant until about 1300 nm, where it decreases again, here we call it (the 780–1300 nm waveband) the reflection luminance region (region *B*). The level of the plateau is dependent on vegetation type: deciduous tree leaves have relatively high IR reflectance (about 90%), whereas coniferous needles are inclined to be more strongly absorbing in the IR region (the reflectance is about 50%). Beyond 1300 nm, there are two absorption regions around 1400 nm and 1900 nm, which correspond to the water. The reflectances of both sides for the same leaf are also different. The reflectance of upper side is larger than that of down side. Therefore, in order to obtain NIR green camouflage for cotton fabric, the reflectance curve of dyed fabric not only overlaps that of greenish leaf in region *A*, but also locates in the range of region *B*.

Table 2 **Vat dyes used in the dyeing experiments**

C.I. No.	Wavelength of max. Abs.	Chemical class
C.I. Vat Orange 1 (C.I.59105)	430 nm	Quinone
C.I. Vat Yellow 33 (C.I.65429)	420 nm	Antraquinone
C.I. Vat Black 27 (C.I.69005)	590 nm	Antraquinone
C.I. Vat Blue 13 (C.I.69845)	640 nm	Antraquinone

EXPERIMENTAL

Materials

The dyeing experiments were carried out on five different fabrics. Commercially obtained cotton fabric (sample 1#) and PET/cotton (65/35) plain fabric (sample 2#) were provided by the local dyeing plant. We fabricated other three fabrics (sample 3#-5#) on a SL7900 sampling loom (CCI Tech Inc.) using 34.3 tex bleached cotton yarn. All tested sample features are given in Table 1.

The IW (indanthrene warm) vat dyes were used in the experiment (see Table 2), including vat orange 1 (C.I.59105) 100%, vat yellow 33 (C.I.65429) 100%, vat black 27 (C.I.69005) 100% and vat blue 13 (C.I.69845) 100%, which were provided by Dystar Trading Co. Ltd.

Alkali (NaOH) and sodium carbonate (Na_2CO_3) were of analytical grade. Sodium hydrosulfite ($Na_2S_2O_4$) and sodium sulfate (Na_2SO_4) were obtained from the local dyeing plant.

Dyeing method

Figure 2 shows the procedure for dyeing cotton fabric with vat dyes. The reduction of vat dyes was carried out by adding two-thirds alkali and sodium hydrosulfite to the water solution at 80°C for 10 min. The concentrated solution of the leuco form was then diluted by adding it to cold water and the temperature of dye bath was adjusted to 50°C. The left alkali and sodium hydrosulfite were also added to the dye bath. Treated cotton samples were added to an aqueous solution containing x% owf vat dye with continuous stirring, and dyeing was continued at this temperature for 15 min. Half of sodium sulfate was added to

Table 1 **Parameters of fabric**

Sample	Material	Fabric weave	Linear density, tex		Density, thread/10 cm	
			Warp	Weft	Warp	Weft
1#	Cotton	2/1 twill weave	7.2	7.2	795	615
2#	PET/cotton	Plain weave	30.0	30.0	340	245
3#	Cotton	Plain weave	34.3	34.3	310	255
4#	Cotton	8-harness satin weave	34.3	34.3	330	310
5#	Cotton	Honeycomb weave	34.3	34.3	320	300

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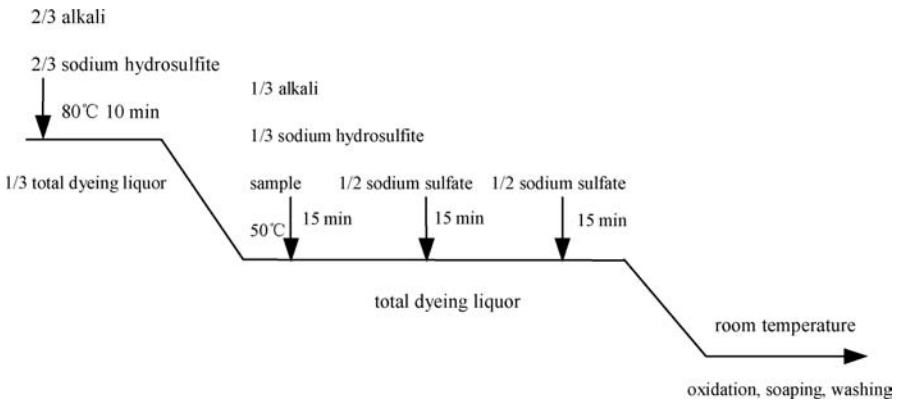


Figure 2 Dyeing program for the dyeing of samples.

the dye bath every 15 min. Oxidation of leuco vat dye can be achieved by exposing the sample to air for 25 min after an initial rinse to remove adhering liquor. The samples were then washed in an aqueous solution containing soap (2 g/L) and sodium carbonate (2 g/L) boiling for 10 min. Finally, the samples were washed with cold water and dried under ambient conditions.

The detailed dyeing conditions are given as follows: alkali 2 g/L, sodium hydrosulfite 0.8 g/L, sodium sulfate 2.2 g/L. The weight of each sample was 1 g and a total dye bath volume was 100 mL. The combination of dyes and dyeing concentration for different fabrics are presented in Table 3.

The dyeing experiments were performed in duplicate. Two samples were separately dyed and the measurements were performed on these samples. Three reflectance measurements were made on each sample rotating the samples 90° before each measurement. The average of the measurements of the two samples was recorded as the reflectance.

The K/S values of dyed sample at the maximum light absorption wavelength were measured by using Datacolor SF600 Plus colorimeter.

The reflectance measurements of dyed sample were performed between 400 nm and 700 nm under D65/10° illuminant by using a Datacolor SF600 Plus colorimeter using a measuring area with a diameter of 9 mm. These reflectances were used to calculate the CIE1976 L^*, a^*, and b^* colour coordinates. Comparison of the colour coordinates of green samples with the fresh greenish leaf of white birch (*Betula platyphylla*) allowed calculation of the variability of the colour of dyed fabric in the form of CIECMC colour difference, $\Delta E^*_{cmc}(2{:}1)$.

The reflectance curve in the 380–2600 nm waveband was measured by using a U-4100 UV-Visible-NIR spectrophotometer (Scan speed: 1200 nm/min at visible range, 1500 nm/min at NIR range. Detector: Photomultiplier (VIS), PbS (NIR). Inner face coated with $BaSO_4$. Incident angle on reflective sample: 10° on both standard and reference sides) with an integrating sphere (ø60 mm).

RESULTS AND DISCUSSION

Reflectance of the combination dyeing

We found that the reflectance of the combination dyeing had relation with the constitution of dyes at NIR region. The reflectances using dyeing formula No. 1, No. 2, No. 3, No. 4, and No. 5 (see Table 3) are presented in Figure 3. It is obvious that the reflectances of Vat Orange 1 (No. 1) and Vat Yellow 33 (No. 2) increase sharply within the range of 400–580 nm. The reflectances of Vat Black 27 (No. 3) and Vat Blue 13 (No. 4) increase rapidly in the corresponding range 600–750 nm and 620–800 nm, respectively. However, the reflectance of the combination dyeing (No. 5) increases in the 620–800 nm waveband, namely, the absorption edge almost overlaps that of Vat Blue 13. Furthermore, compared with fresh greenish leaf, the

Table 3 **Dyeing formula**

No.	Dyeing fabric	Combination of dyes and dyeing concentration (owf)
1	Sample 1#	C.I. Vat Orange 1 1%
2	Sample 1#	C.I. Vat Yellow 33 1%
3	Sample 1#	C.I. Vat Black 27 1%
4	Sample 1#	C.I. Vat Blue 13 1%
5	Sample 1#	C.I. Vat Orange 1 1%, C.I. Vat Yellow 33 1%, C.I. Vat Black 27 0.2%, and C.I. Vat Blue 13 1%
6	Sample 1#	C.I. Vat Blue 13 0.3%, 0.5%, 1%, 1.5%, 2%, 2.5%, 3%, 4%, 5%, 6%, 7%, 8%
7	Sample 3#, 4#, and 5#	C.I. Vat Blue 13 2%
8	Sample 1# and 2#	C.I. Vat Orange 1 5.5% and C.I. Vat Blue 13 2%

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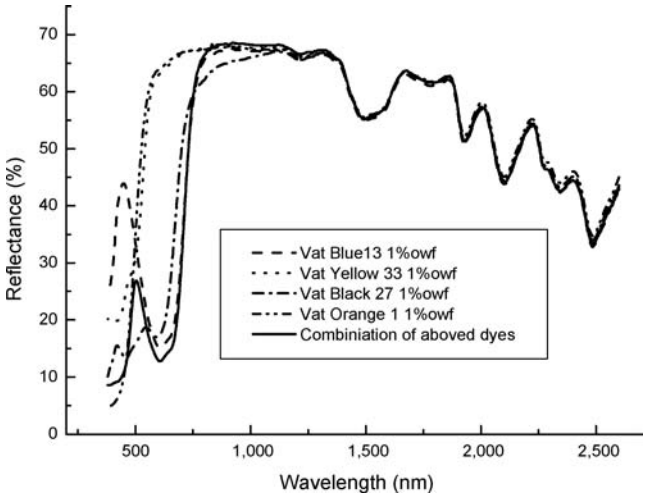


Figure 3 Reflectance curves of dyed fabric using single dye and combination dyeing.

reflectance of Vat Blue 13 is very close to that of greenish leaves at region *A*. So, Vat Blue 13 plays an important role in NIR camouflage.

Reflectance of cotton fabric before and after dyeing

The transmittance curves of four vat dyes in alcohol solution are given in Figure 4. It can be seen that there is an absorption peak around 430 nm for Vat Orange 1 in the visible range. There are two absorption peaks around 420 nm and 490 nm for Vat Yellow 33. The absorption peaks of Vat Black 27 occur at 450 nm and 590 nm. There is a broad absorption peak for Vat Blue 13 around 640 nm. Compared with Figure 3, the reflectance of dyed fabric is mainly determined by the absorption characteristics of the dyes in the visible range. It can be illustrated by the transmittance curve of Vat Blue 13 in alcohol solution in Figure 5. The reflectances of cotton fabric before and after dyeing are also presented. It is clear that the maximum absorption peak of dyed fabric at 606 nm is due to the absorption characteristic of Vat Blue 13.

With regard to NIR waveband, it is seen from Figure 4 that the absorption peaks of four vat dye liquors are at 976 nm and 1194 nm. The infrared is absolutely absorbed by dye liquor beyond 1380 nm. It is seen from Figure 5 that the absorption peak does not shift after dyeing, but the reflectances decrease to some extent, such as 1492 nm (O-H stretching 1st overtone), 1778 nm (C-H stretching 1st overtone), 2100 nm (O-H deformation and C-O stretching 1st overtone), and 2344 nm (C-H stretching and C-H deformation).

Effect of dyeing concentration on reflectance

We chose dyeing formula No.6 to study the effect of dyeing concentration on reflectance, and measured the K/S values of dyed fabric at absorption peak (610 nm). The relationship between K/S values and dyeing concentrations (% owf) is shown in Figure 6. As the dyeing concentration of Vat Blue 13 increases, the K/S value increases rapidly. It is obvious that the dyeing ability of Vat Blue 13 is very good.

Figure 7 presents the reflectances of dyed fabric using Vat Blue 13 at different dyeing concentrations. They are very similar to each other in shape in the visible range. The wavelength at absorption peak (610 nm) does not shift. As the dyeing concentration increases, the reflectance decreases gradually. The reflectance curves turn flat around 610 nm. The absorption edge in region *A* overlaps that of greenish leaves on condition that the dyeing concentration is between 1% and 2% owf. The average reflectance at region *B* is about 85%. So, the suitable dyeing concentrations of Vat Blue 13 are 1% owf for dyeing light colour and 2% owf for dyeing dark colour.

Effect of fabric weave on reflectance

In order to investigate the effect of fabric weave on reflectance, we fabricated three cotton fabrics, and then dyed

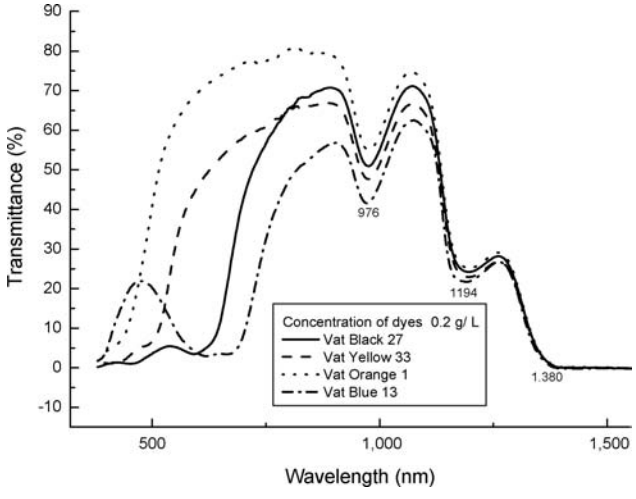


Figure 4 Transmittance curves of dye liquors.

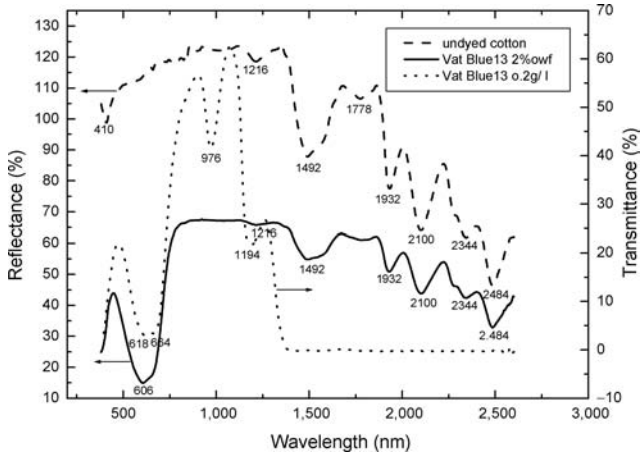


Figure 5 Transmittance curve of Vat Blue 13 and reflectance profile of dyed fabric.

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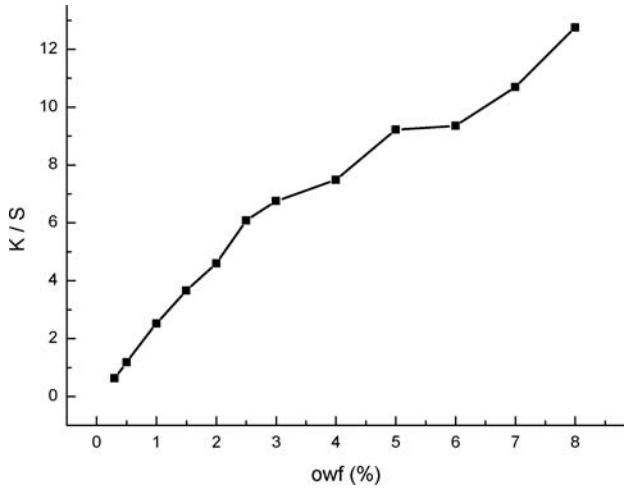


Figure 6 K/S values of Vat Blue 13 versus dyeing concentrations.

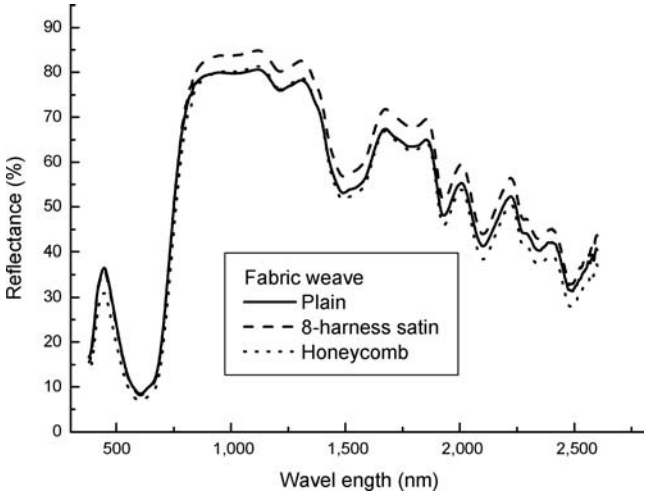


Figure 8 Reflectance curves of different fabric weaves.

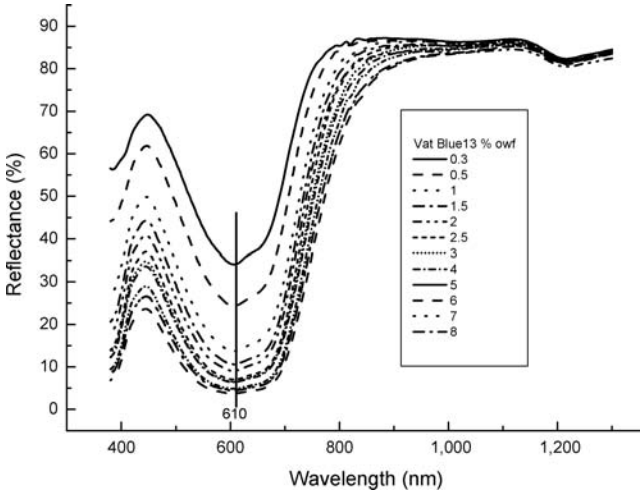


Figure 7 Reflectance curves at different dyeing concentrations.

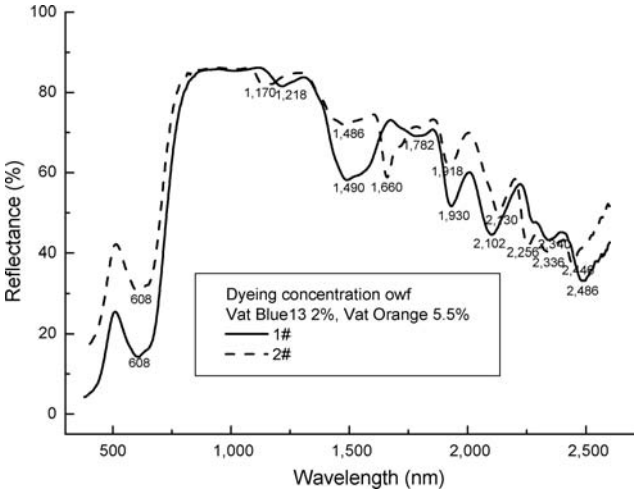


Figure 9 Reflectance curves of PET/cotton and cotton fabrics.

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them using the same dyeing formula No. 7. The reflectance curves are presented in Figure 8. The reflectances of plain fabric (3#) and 8-harness satin fabric (4#) are larger than that of honeycomb weave fabric (5#) in the visible range. There is a little discrepancy in the 850–1300 nm waveband, about 4%. This suggests that the fabric surface has a little effect on visible and NIR camouflage.

Effect of blend fabric on reflectance

The reflectance profiles of PET/cotton and cotton fabric are presented in Figure 9 by using dyeing formula No. 8. It is evident that the reflectance of PET/cotton fabric (sample 2#) is relatively larger than that of cotton fabric (sample 1#) for the same dyeing formula in visible range, but they almost overlap in region *B*. This suggests that PET fiber, whether dyed or not, has no effect on NIR camouflage, that is to say, the NIR camouflage of PET/cotton fabric is mainly determined by the characteristic of dyed cotton fiber. The typical absorption peaks of PET fibre are at 1170 nm (C-H stretching 2nd overtone), 1660 nm (C-H stretching 1st overtone), 1918 nm (C = O stretching 1st overtone), 2130 nm (N-H stretching and C = O stretching), respectively.

Reality of visible and NIR camouflage

In order to obtain a close spectral match at NIR region and a minimum color difference in visible range, we adjusted the dyeing recipe and produced the optimized reflectance curve. Figure 10 shows the reflectance curves of dark and pale green cotton fabrics by using Vat Blue 13 (1% owf for pale green, 2% owf for dark green), Vat Orange 1 (5% owf), and Vat Yellow 33 (3% owf). The reflectances are very close to each other, and they nearly overlap that of greenish leaves in region *A*. Meanwhile, they are located between deciduous leaf and coniferous needle in region *B*, which meet the requirement of NIR camouflage. We also found that the dyeing concentration of Vat Black 27 has a great effect on NIR camouflage beyond 0.5% owf.

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Table 4 **Color coordinates of dark green and pale green fabric and ΔE^*_{cmc} (2:1) compared with the greenish leaf of white birch ($L^* = 27.993, a^* = -3.229, b^* = 4.344$)**

Dyed cotton	X	Y	Z	L^*	a^*	b^*	$\Delta E^*_{cmc}(2:1)$
Dark green	4.51	5.63	3.96	28.28	−9.70	9.74	8.82
Light green	4.54	5.61	4.20	28.41	−9.85	8.64	8.29

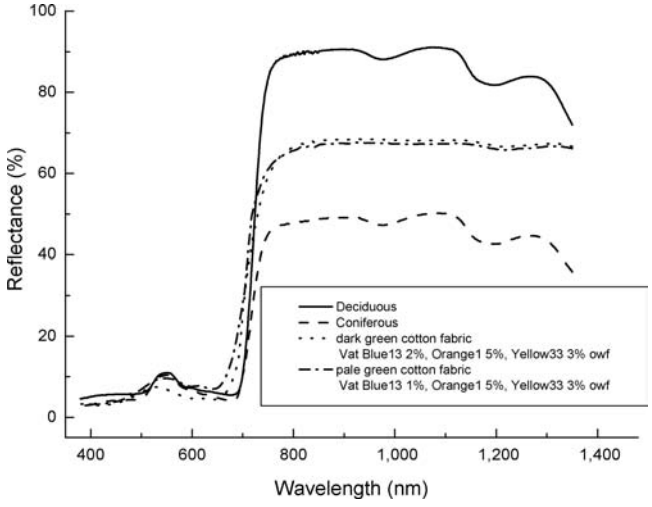


Figure 10 Reflectance curves of green cotton fabrics.

We measured the chromatic values (CIE1976 L^*, a^*, and b^*) of pale and dark green fabrics to verify the effect of visible camouflage. According to CIECMC colour difference, $\Delta E^*_{cmc}(2:1)$, the color differences between green fabrics and the white birch leaf ($L^* = 27.99$, $a^* = -3.23$, $b^* = 4.34$) are given in Table 4. Although the L^* value is very close to that of greenish leaf, the $\Delta E^*_{cmc}(2:1)$ is very large, so further improvement is needed in the future.

CONCLUSIONS

Cotton fabrics were dyed by using a combination of dyes (Vat Orange 1, Vat Yellow 33, Vat Blue 13, and Vat Black 27) to imitate the reflectance profile of greenish leaf on the basis of NIR camouflage theory. Results show that the reflectance of dyed fabric is markedly influenced by the constitution of dyes in visible and NIR region. The absorption edge of dyed fabric is determined by one of the dyes whose reflectance curve emerges at longer wavelengths. Vat Blue 13 plays an important role in NIR green camouflage. When the dyeing concentration of Vat Blue 13 is 1–2% owf, the reflectance of dyed fabric matches with that of greenish leaf in region *A*. Fabric weave has a little effect on the reflectance for the same dyeing formula. For PET/cotton blended fabric, the NIR camouflage property is mainly determined by the characteristic of dyed cotton fiber.

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