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Improvement of VIS and IR camouflage properties by impregnating cotton fabric with PVB/IF-WS_2

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ABSTRACT

In order to examine the possibility to improve its camouflage properties standard cotton fabric with camouflage print was impregnated with poly(vinyl butyral), PVB and fullerene-like nanoparticles of tungsten disulfide, PVB/IF-WS_2. FTIR analysis excluded any possible chemical interaction of IF-WS_2 with PVB and the fabric. The camouflage behavior of the impregnated fabric has been examined firstly in the VIS part of the spectrum. Diffuse reflection, specular gloss and color coordinates were measured for three different shades (black, brown and dark green). Thermal imaging was applied to examine the camouflage abilities of this impregnation in IR part of the spectrum. The obtained results show that PVB/IF-WS_2 impregnation system induced enhacement of the materials camouflage properties, i.e. that IF-WS_2 have a positive effect on spectrophotometric characteristics of the fabric.

1. Introduction

Through the recent years demands for more improved functionalized textile materials are growing. The examples of their applications are vast, from self-decontaminating or self-cleaning materials with antibacterial features to materials with low electromagnetic absorption, and in many of them nanomaterials play the role of the functional constituent [1–8]. These materials can be classified as: protective-functional (environmental hazard protective, biological, chemical and radiation hazard, NBC protective), medical-functional (injury protective, therapeutic and rehabilitative, bio-sensing), sports-functional, vanity-functional, clothing for special needs and cross-functional assemblies (multifunctional performance protection, life support, comfort, communication) [9].

Camouflage protection is still one the main problems related to protection of military personnel and equipment in the so-called hostile environments. Dying of the material is therefore the key point of the camouflage protection. Due to rapid advancements of multispectral and high-resolution sensors capable of detection, identification, and recognition (DIR) of military targets, fusion of sensors, in different areas of the electromagnetic (EM) spectrum, and their deployment on a single platform make the DIR task more complicated for providing an effective solution [10]. Therefore, means of mathematical models have been proposed recently as well, in order to improve quantitative research of the spatial coloring process of digital camouflage pattern, for desired camouflage pattern spots and evaluation of the infrared camouflage effectiveness [11–13]. Nonetheless, in order to obtain multispectral camouflage protection camouflage textile materials are being impregnated with different polymers, organic and inorganic compounds which alter the optical behavior of the impregnated textile. The increased need for multifrequency camouflage (like laser radar, visual) compatible with mid-infrared (MIR) radiation has induced the development of various methods of thermal insulation layers, such as silica aerogel, Ge/ZnS multilayer, phase-changing materials, colored nanophotonic structure textile, as well as hierarchical metamaterials (HMMs) for multispectral signal control [14–18]. The application of thereof in thermal management is tightly connected with aesthetics, wearability and manufacturability - simple structural design. However, some of these solutions are expensive and not easily applicable in industrial manufacture.

The means for analyzing possible multispectral features of one material are part of standard laboratory equipment. The so-called

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passive sensors can detect solar radiation either reflected (UV-VIS-NIR) or radiated (thermal) energy from the target [10]. Therefore, standard UV-VIS-NIR spectrophotometers as well as IR thermography are used as techniques for multispectral material characterization. Even though IR thermography has great variety of applications such as monitoring temperature distribution on the surface layers of metal materials and ceramics, measuring fluid temperature and velocity fields in different turbulent flows and temperature gradients, thermal defectoscopy or for detection of boundary-layers transition zones in wind tunnel testing [19–22], naturally emitted energy, falling in the thermal infrared area of EM spectrum can be detected during day and especially well at night using IR cameras.

In this research a possibility was examined to alter the camouflage behavior of a standard military cotton textile by an impregnation system based on poly(vinyl butyral), PVB and WS_2 nanoparticles.

High transparency polymer, poly(vinyl butyral) was used in our previous studies as a convenient polymer matrix for impregnation, lamination and coating of other materials, having a positive impact on their mechanical and optical characteristics [23–29]. It is a ductile polymer which does not dissolve in water, so it has a good resistance to wetting. Beside this, PVB has excellent adhesive and film forming properties, since it shows fast solvent release and low solvent retention. It is generally often used in a variety of applications: in laminated glass, in temporary binders, for technical ceramic, inks, paints and coatings, primers, laminated composites, in ballistic protection, binders for reflective sheet and binders for magnetic media [28–31].

Tungsten disulfide, as other transition-metal dichalogenides, exhibits excellent mechanical properties, and due to them it may be used for a wide range of applications: aerospace and automotive technology, load bearing and release mechanisms, as solid lubricants, corrosion protection, reinforcement for polymer composites etc [32–35]. Tungsten disulfide nanoparticles in form of fullerene-like structure, IF-WS_2, exhibit exceptional resistance to high temperatures and due to that, it may find application even as a protective layer on the inner surface of the artillery barrels and nozzles, to reduce erosion and thermal load [36–38]. In our previous research fullerene-like nanoparticles of tungsten disulfide, IF-WS_2, were used as nanoreinforcement because their positive effect on spectrophotometric characteristics of military camouflage paints was shown [24–26,39]. Thus, even very low concentrations of IF-WS_2 enhance mechanical and wear resistance, as well as VIS camouflage behavior, while not affecting significantly the cost of final products. IF-WS_2 nanostructures have already been proven to significantly improve friction, wear and impact resistance when implemented in composites. The presence of such friction and wear resistant nanoreinforcement certainly improves the mechanical resistance and durability of the polymer impregnated fabric with this nanoreinforcement dispersed in, but this aspect is not the subject of this research. In this article, we have focused on the spectrophotometric properties and IR signature of the material, in terms of its camouflage potential.

Regarding all this, here we propose a method for impregnation of the cotton fabric with a standard camouflage print with PVB and PVB/IF-WS_2. The aim of this study is to compare the spectrophotometric characteristics and show the enhacement of the camouflage properties of the material and possible multispectral camouflage application.

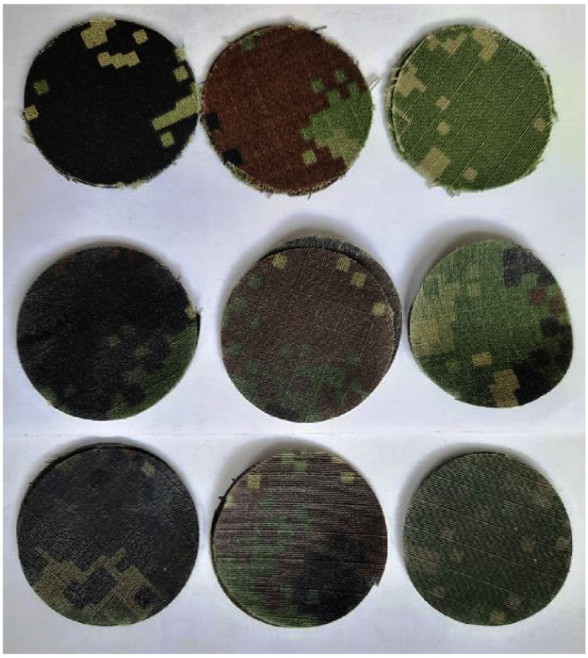


Fig. 1. Textile samples with and without impregnation, a) neat textile, b) with PVB impregnation and c) with PVB/IF-WS_2 impregnation.

2. Experimental

2.1. Samples preparation - impregnation of the fabric

As a base material, cotton fabric with camouflage print was used, intended for protective clothing. Cotton fabric with camouflage print was impregnated with ethanol solution of PVB (Mowital B30H, Kuraray GmbH) with and without nanoparticles of IF-WS_2 (NanoLub, ApNano, provided by SpeedUp International), previously ultrasonically dispersed (ultrasonic probe Badelin Sono Puls). Properties of the used raw materials, PVB and IF-WS_2, are given in Supporting information, in Tables S1 and S2. Automatic applicator (Byko drive) was used to apply the impregnation evenly onto the fastened fabric surface. The amount of applied PVB layer on the fabric was approximately 5 wt% regarding the mass of the fabric. Concentration of IF-WS_2 was 2 wt% regarding the mass of PVB. After solvent evaporation the fabric samples were cut into proper pieces for different measurements and analyzed regarding the effect of the added nanostructures on impregnated material properties. Fig. 1 shows the appearance of the samples for spectrophotometric measurements. FTIR analysis was performed in order to examine any possible chemical interaction of IF-WS_2, PVB and the fabric. Scanning electron microscopy (SEM) was applied to observe the samples before and after the impregnation. Specular gloss at 85° angle, which is important for fabric materials in the field of military camouflage, was measured. Diffuse reflection was measured on UV VIS NIR spectrophotometer on black, brown, and dark green shades of the examined fabric samples. IR thermography was applied to examine materials camouflage behavior in IR part of spectrum.

2.2. FTIR-ATR analysis

Fourier transform infrared spectra of the neat cotton fabric and the fabric impregnated with PVB and PVB/IF-WS_2 were obtained using a FTIR spectrometer Thermo Nicolet iS10. The Attenuated Total Reflectance (ATR) measurement mode was used with the sample mounted on a diamond crystal. Absorption spectra were recorded in the range of 400–4000 cm^{-1}.

2.3. SEM analysis

The fabric without and after the applied layer of PVB and PVB/IF-WS_2 was analyzed by field emission scanning electron microscopy (FE-SEM) on the Tescan Mira3 XMU electron microscope at 3.5 kV. The SEM analysis was performed in order to estimate the quality of fabrics impregnation and to observe the IF-WS_2 nanoparticles incorporated in PVB polymer matrix and impregnated fabric fibers, as well.

2.4. Camouflage properties - IR reflection, colorimetry and specular gloss

The spectrophotometric measurements were conducted using the UV/VIS/NIR spectrophotometer UV 3600 from a Japanese manufacturer Shimadzu with an integrating sphere For reflectance measurements samples were measured in the visible and near-infrared area of the electromagnetic spectrum (650–1000 nm) using UV Probe programme package, while color coordinates were measured in the visible part of the electromagnetic spectrum (380–780 nm) using Color program package with 10° and D65 observer. Color coordinates were determined in the CIE Measurement Lab space. Samples were placed horizontally in the sample compartment of the device, which has a 2 cm aperture diameter. Every sample was measured only once. Diffuse reflectance has ±1% measurement uncertainty, while measurement uncertainty for L^*, a^* and b^* coordinates are given in Table S3 of the Supporting information.

Specular gloss was measured with Elcometer 480 model T device; the angle was 85° which is a standard measurement angle for textile materials [40] with the measurement uncertainty ±0.2.

2.5. IR thermography

Two IR cameras that work in different spectral range were used in this research, FLIR SC620 and FLIR SC 7200. SC 7200 uses ”Altair” software for data acquisition and analysis, while SC 620 uses ”ThermaCAM 2.9″ software [41,42]. The thermal images were analyzed using “ThermaCAM Researcher Professional” software [43]. Technical characteristics of the two IR cameras used in the present research are given in Table S4 of the Supporting information.

In this experiment absolute black body TCB-4D was used, Controller black body 0411, produced by Inframet. Temperature rise was controlled by TCB software TAS-T [44]. It was heated at 36 °C, 50 °C and 75 °C. The heat exposure time was 10 min for each sample.

The overall goal of this type of analyses was to make comparative measurements and to obtain an insight of the processes between material of interest and electromagnetic energy in MWIR and LWIR spectral range of the electromagnetic spectrum.

3. Results and discussion

3.1. ATR-FTIR analysis results

Fig. 2 shows the FTIR spectra of cotton fabric without and with impregnations.

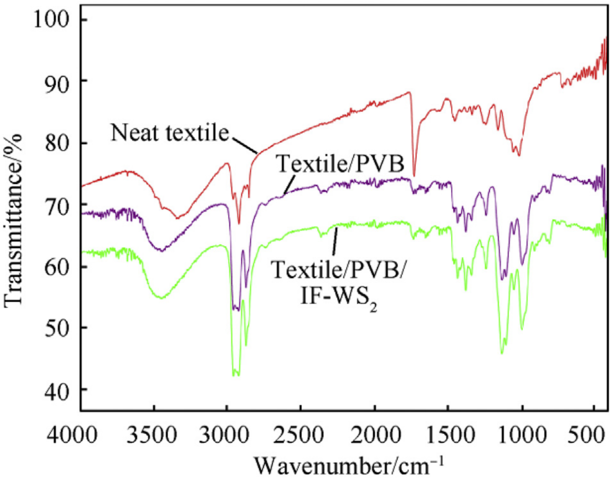


Fig. 2. FTIR spectra of fabric with and without impregnations.

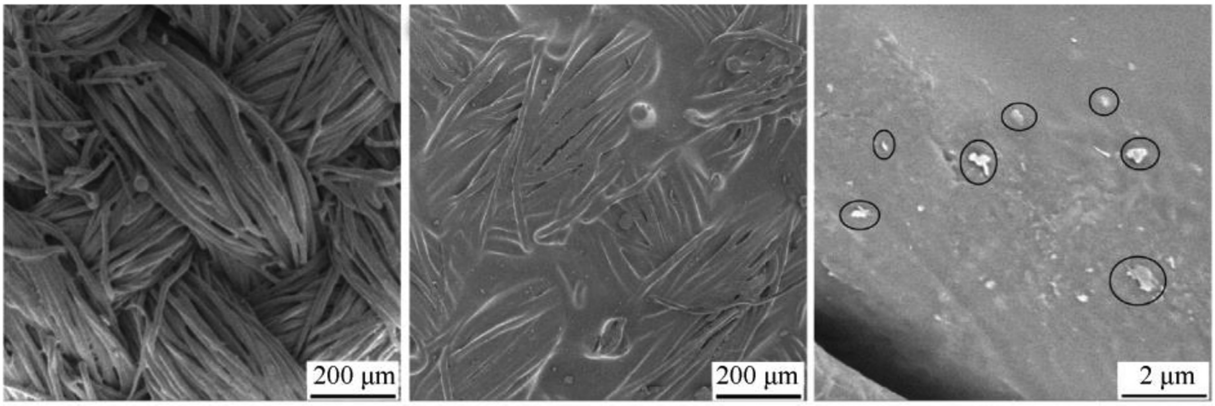
The observed FTIR spectra of these samples showed the characteristic peaks corresponding to cellulose structure [45], which is the main component of cotton fabric, as follows: the peak at 3300 cm^{-1} (actually starting from 3000 cm^{-1} to 3700 cm^{-1}), which originates from O–H stretching, the peak at about 1430 cm^{-1} from the aliphatic $-CH_2$ bending vibration of cellulose, the peak at 1335 cm^{-1} from the O–H bending of CH_2–OH group in cellulose, a wide peak at 2800–3000 cm^{-1} corresponds to C–H stretching. Cellulose contains $-CH_2-$ groups in the structure, but the peaks from symmetric and asymmetric stretching are not separated as sharp peaks. Although the used cotton fabric already has a military camouflage print, the components of the dye and other possible impurities are in too small concentrations to be observed in the spectrum.

From the FTIR spectra for the sample of fabric impregnated with PVB and fabric impregnated with PVB/IF-WS_2, it may be concluded that there is no interaction between this polymer and the nanoparticles, since the same peaks may be observed. In the both spectra the characteristic peak for the chemical structure of the PVB was registered: an intensive one at about 3480 cm^{-1} corresponding to the stretching of the OH group. Actually, this peak is wide due to different hydrogen bonds between O–H segments in the polymer structure of PVB molecules [29], so there is a distribution of vibrations in the range 3100–3700 cm^{-1}. Peaks registered at about 2960 cm^{-1} and 2870 cm^{-1} correspond to the symmetric and asymmetric vibrations of the CH_2 and CH_3 groups. The peaks at 1740 cm^{-1} correspond to the vibrational stretching of the ester group C¼O. The peaks at 1245 cm^{-1} and 1140 cm^{-1} originate from the valence vibrations of the ester group C–O. A prominent peak from the vibrational stretching C–OH is registered at about 995–1000 cm^{-1}. A low intensity peak approximately at 2350-2360 cm^{-1} is a consequence of the acidic solution in which PVB is synthesized.

This analysis confirmed the inertness of tungsten disulfide, i.e. the absence of a chemical reaction between the WS_2 nanoparticles/nanotubes and PVB, since there are no new peaks in the impregnation PVB/IF-WS_2 that have not already been observed in the previous spectrum.

3.2. SEM analysis results

The SEM images of the samples, shown in Fig. 3, present the neat fabric, the fabric impregnated with PVB only and the fabric impregnated with PVB containing dispersed nanoparticles of IF-WS_2. The observed images indicate that the fabric was well



(a) (b) (c)

Fig. 3. SEM images of the examined samples: a) neat fabric, b) fabric impregnated with PVB, and c) fabric impregnated with PVB/IF-WS_2 (nanoparticles are indicated by circles).

impregnated with the polymer matrix, PVB, and that the identified IF-WS_2 nanoparticles were well dispersed on the impregnated fabric fibers.

3.3. Diffuse reflection

Figs. 4–6 show graphs for diffuse reflection values for black, brown and dark green shade in the 650–1000 nm wavelength area without impregnation as well as with the PVB and PVB/IF-WS_2 impregnation.

Measurements of diffuse reflection show that for dark green and brown shade PVB and PVB/IF-WS_2 addition give decrease in values. This decrease is especially significant for the dark green shade and it gradually drops from values for standard camouflage material to material with PVB/IF-WS_2 in which case the values are the lowest. Nevertheless, all values are acceptable by the Serbian Military Standard [46].

In the case of brown shade, as stated, this decrease is observed as well, but it is not that obvious like for the dark green shade. Moreover, the values of diffuse reflection for materials with the above mentioned additives have similar values. In this case, as well, the values are in accordance with the standard [46].

However, for the black shade the insignificant increase has been observed. This increase ranges from 1 to 2%, which is of the measurement uncertainty's order [47] and therefore we can say that the values of the diffuse reflection for the black shade have not changed in the case of PVB and PVB/IF-WS_2 addition.

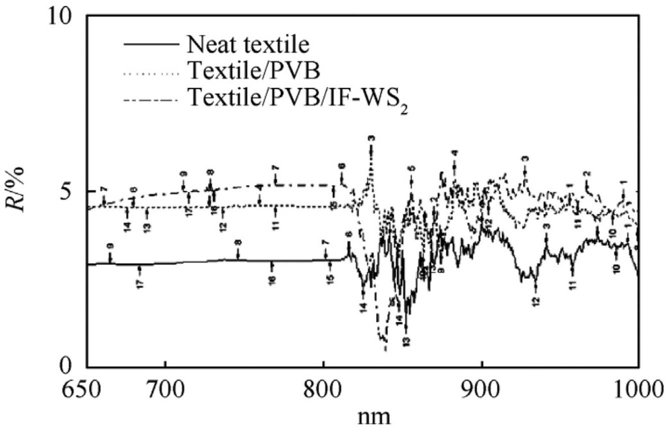


Fig. 4. Diffuse reflection for black shade with and without impregnation.

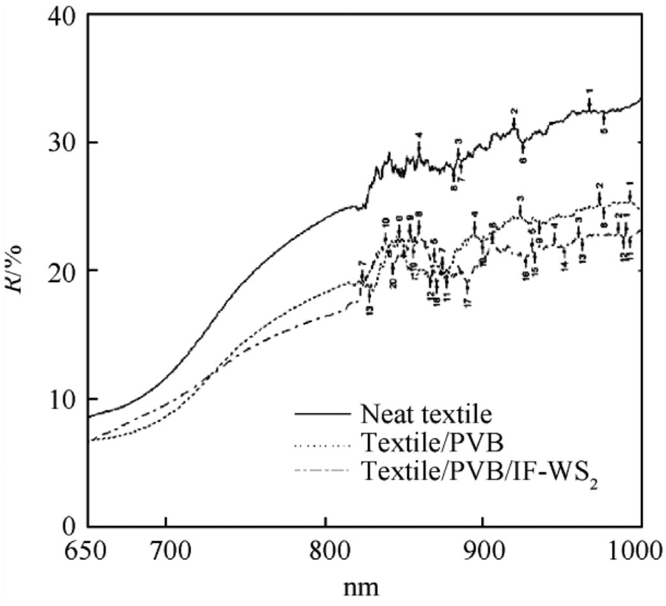


Fig. 5. Diffuse reflection for brown shade with and without impregnation.

3.4. Colour coordinates

Color coordinates defined in CIE Lab system for all three samples and three shades of interest were measured and the results are presented in Table 1.

The main characteristic these data provide is the so-called ΔE difference which is number that is calculated with Eq. (1):

$$\Delta E = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \quad (1)$$

If the ΔE is less than 1, the difference between two shades is not visible to the observer's eye. For the sake of this research we formed several ΔE differences and the results are presented in Table 2. ΔE_1 represents the difference between PVB impregnated material and standard cotton textile material. ΔE_2 is the difference between material impregnated with PVB/IF-WS_2 and PVB impregnated, while ΔE_3 is the difference between PVB/IF-WS_2 impregnated material and standard material.

There has been observed a change in the visual characteristics of the material which is proven by color coordinates measurements.

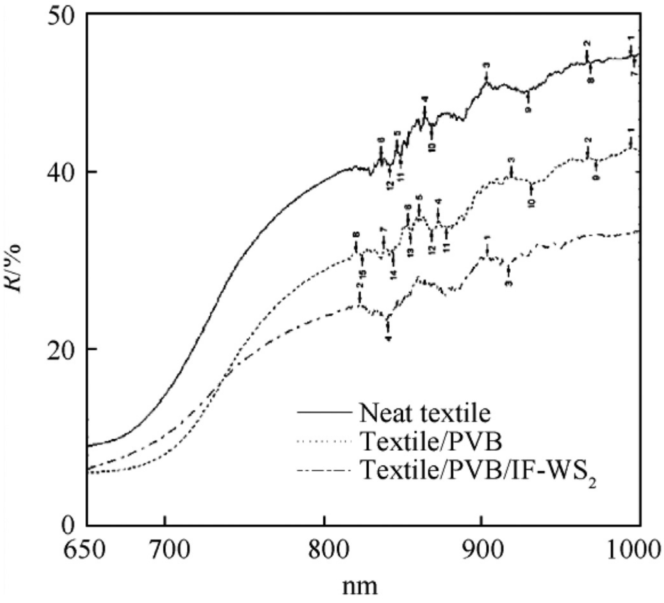


Fig. 6. Diffuse reflection for dark green shade with and without impregnation.

Table 1
Color coordinates defined in CIE Lab system for three shades of interest.

Shades	Standard material			With PVB impregnation			With PVB/IF-WS$_2$ impregnation		
CIE Lab	*L**	*a**	*b**	*L**	*a**	*b**	*L**	*a**	*b**
Black	20.01	0.03	−0.81	26.8	−0.25	−1.03	25.33	−0.02	−0.58
Brown	27.23	6.79	9.21	29.04	3.45	3.54	29.9	−0.38	3.78
Dark green	36.83	−8.76	12.58	31.48	−4.83	5.50	28.72	−3.03	3.92

Table 2
ΔE differences.

Shades	ΔE_1	ΔE_2	ΔE_3
Black	6.80	1.55	5.32
Brown	6.88	3.93	9.38
Dark green	9.71	3.65	13.18

Still, the colorimetry measurements show the least difference between two differently impregnated materials (Table 2) and for the black shade, which was expected.

3.5. Specular gloss

Specular gloss was measured in eight different spots due to anisotropy of textile materials. It was measured on fully camouflage printed samples which contain all shades needed by military demands. In order to present results in the most adequate way average values for every type of sample is shown in Table 3.

As seen in Table 3 specular gloss values are of the same order and very similar to each other, so the applied impregnation does not have any influence on it. Not less important is that all these values are in accordance with the Serbian Military Standard [48].

Table 3
Specular gloss values measured at 85°.

Sample	Specular gloss
Standard textile material	0.4
PVB impregnated	0.3
PVB/IF-WS$_2$ impregnated	0.4

3.6. IR thermography results

Thermographs of the examined fabric obtained with IR camera FLIR SC620 in LWIR range are given in Fig. 7, for the following temperatures of the black body: 36 °C, 50 °C and 75 °C.

Thermographs of the examined fabric obtained with IR camera FLIR SC7200 are given in Fig. 8, for the same temperatures of black body. In the recordings temperature fields are presented in shades of gray. On both figures, m1, m2 and m3 stand for neat textile, PVB impregnated and PVB/IF-WS$_2$ impregnated, respectively.

From these thermographs, temperature values were read and compared for the examined samples, in order to determine the relative temperature difference, as a measure of camouflage effect in IR spectrum range. Tables 4 and 5, show results obtained from IR cameras FLIR SC620 and FLIR SC7200.

From the presented IR thermography measurements results, it may be observed that smallest temperature difference, ΔT, was registered for the sample with tungsten disulfide nanoparticles. So, from the aspect of IR camouflage, the best results were obtained with PVB/IF-WS$_2$ impregnation regardless of the spectral range (at all three black body temperatures, 36 °C, 50 °C and 75 °C). These findings can be explained by the increase in density of the material and decrease in heat energy transmission. Also, most probable reason for this kind of behavior lies in the inherent structure of the used nanoparticles of tungsten disulfide: having a spherical, multi-

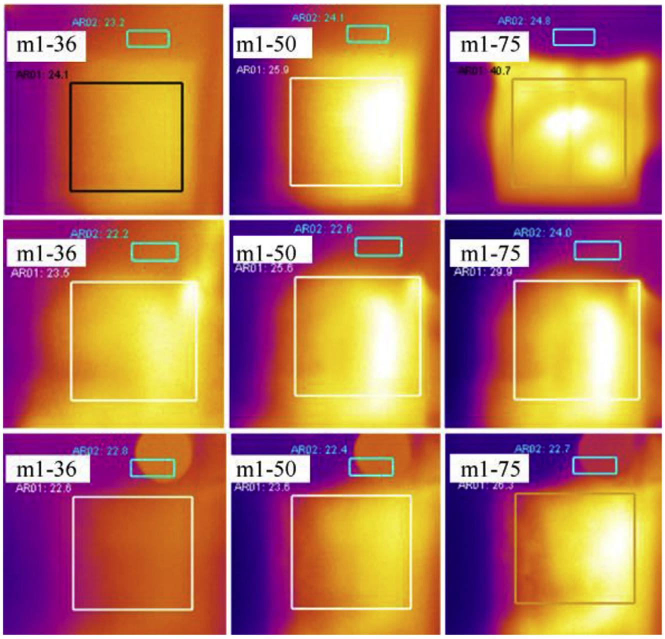


Fig. 7. Thermographs of the examined fabric obtained with IR camera FLIR SC620.

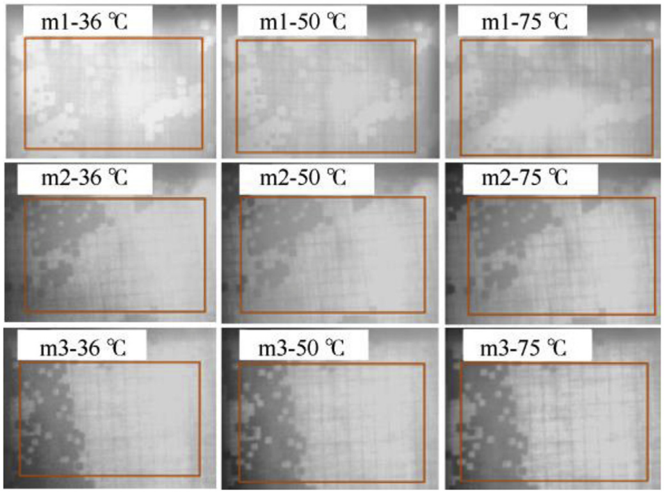


Fig. 8. Thermographs of the examined fabric obtained with IR camera FLIR SC7200.

Table 4
FLIR SC620 camera measurements.

	Standard material			PVB impregnated			PVB/IF-WS_2 impregnated		
$T_{black\ body}$/°C	36	50	75	36	50	75	36	50	75
T_{amb}/°C	17.8	17.8	17.8	19.8	19.8	19.8	21.8	21.8	21.8
T_{avr}/°C	24.0	25.8	40.1	23.4	25.7	30.1	22.5	23.6	26.3
$\Delta T = T_{avr}$- T_{amb}/°C	6.2	8.0	22.3	3.6	5.9	10.3	0.7	1.8	4.5

Table 5
FLIR SC7200 camera measurements.

	Standard material			PVB impregnated			PVB/IF-WS_2 impregnated		
$T_{black\ body}$/°C	36	50	75	36	50	75	36	50	75
T_{amb}/°C	17.8	17.8	17.8	19.8	19.8	19.8	21.8	21.8	21.8
T_{avr}/°C	25.3	29.8	40.3	23.7	26.4	32.2	23.0	24.6	29.0
$\Delta T = T_{avr}$- T_{amb}/°C	7.5	12.0	22.5	3.9	6.6	12.4	1.2	2.8	7.2

layer onion-like hollow structure, they behave as a specific nanometric thermal insulators. As a consequence, these results are promising for future multispectral camouflage applications.

4. Conclusion

Overall results have showed that impregnation of standard cotton textile material that is already printed with camouflage dyes with system of PVB/IF-WS_2 can enhance camouflage properties. FTIR analysis has confirmed the chemical inertness of IF-WS_2 in touch with the fabric and PVB, and SEM analysis has proven the presence of IF-WS_2 particles, as well as the good quality of fabrics impregnation with PVB. The existing properties of the material in UV/VIS range of spectrum are not affected with the impregnation and they still meet the adequate camouflage standards' requirements. Specular gloss did not change. Moreover, IR thermography shows good possibilities for broadening the camouflage spectral range to MWIR and LWIR range which in turn can lead to obtaining multispectral camouflage features of the textile material. The relative suppression ratio of the effective temperature difference between the material and the background was decreased by the addition of IF-WS_2. The most distinctive difference in color shade to the observer's eye was observed for green shade when the color coordinates were determined and compared, but this only means that in further research and application, the incorporation of the nanoparticles should be considered already in dyes production and original shades design. Furthermore, there is still enough space for extending this research in applying different IF-WS_2 concentrations.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.dt.2020.10.008.

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