Functionalization of Cellulose Fibres with Oxygen Plasma and ZnO Nanoparticles for Achieving UV Protective Properties

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Low-pressure oxygen plasma created by an electrodeless radiofrequency (RF) discharge was applied to modify the properties of cellulosic fibrous polymer (cotton) in order to improve adsorption properties towards zinc oxide (ZnO) nanoparticles and to achieve excellent ultraviolet (UV) protective properties of cotton fabric. The chemical and physical surface modifications of plasma-treated cotton fabric were examined by X-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM). The mechanical properties of plasma-treated samples were evaluated, measuring strength and elongation of the fabrics. The quantity of zinc on the ZnO-functionalized cotton samples was determined using inductively coupled plasma mass spectrometry (ICP-MS) and the effectiveness of plasma treatment for UV protective properties of cotton fabrics was evaluated using UV-VIS spectrometry, measuring the UV protection factor (UPF). The results indicated that longer plasma treatment times cause higher concentration of oxygen functional groups on the surface of fibres and higher surface roughness of fibres. These two conditions are crucial in increasing the content of ZnO nanoparticles on the fibres, providing excellent UV protective properties of treated cotton, with UPF factor up to 65.93.

1. Introduction

UV radiation created by the Sun or artificial sources causes premature aging of the skin and changes to the functioning of the immune system [1]. The harmful effects on the skin and the eyes can become evident decades after being overly exposed to UV radiation. It has been proven that UV radiation is one of the prominent risk factors in the development of skin cancer. We can protect the skin from the negative effects of UV radiation by regularly using high factor sunscreens and wearing headwear that protects the ears and the neck and with long-sleeved clothing. With thinner materials that do not offer a high degree protection, we can use dark textiles or apply different UV absorbers [2].

Recently, we have been witnessing an increasing interest in the use of ZnO nanoparticles, due to the fact that decreasing the size of the particles increases their surface-to-mass ratio, consequently changing the fabrics’ chemical, mechanical, and optical properties [3–6]. Moreover, ZnO nanoparticles are an excellent blocker of UV radiation. Researches so far have proved that UV protection of cotton fabrics improves when being treated with ZnO/chitosan nanoparticles, increasing in intensity when treated with higher concentrations of nanoparticles of ZnO/chitosan [7]. UV protective properties of cotton fabrics depend on the size of ZnO nanoparticles [8]. Smaller particles of ZnO offer better protection from harmful UV radiation than larger particles. The application of ZnO nanoparticles to textiles is primarily carried out by means of wet-chemical procedures [3–5, 9–11], using synthetized [3–5, 9, 12–14] or ready-made ZnO nanoparticles [15]. Synthesized ZnO nanoparticles are applied to cotton fabrics by impregnating with a wet pick-up of 100%, drying, and curing or by treating the fabric in a bath for 10 minutes using a magnetic stirrer, drying, and curing [2, 4]. Textiles functionalized in such a manner offer sufficient, yet rarely excellent UV protective properties. Different synthesis conditions and the effect of different variables during
the synthesis affect the end product \([13, 16]\). It is therefore best to use ready-made nanoparticles available in the market when performing research on nanoparticle adsorption to textile substrates \([14, 15, 17]\). Another reason for the UV properties of functionalized textiles being sufficient but not excellent is a rather poor adsorption of ZnO nanoparticles to textiles. Acrylic and epoxy binding agents can be added to the treating bath to increase the adsorption of ZnO nanoparticles \([3, 12, 18, 19]\). Another solution would be to try changing the reactivity of the substrate by using chemicals \([4]\); however, ZnO nanoparticles would still retain a poor adsorption to textile substrates. When applying ZnO nanoparticles to a textile, it is important to select an appropriate method to ensure the optimal effect \([20]\). To achieve good protective properties, high concentrations of ZnO nanoparticles need to be used \([5, 6, 10]\), meaning that most of them remain in the bath, eventually being released to the environment where they cause pollution. The textile industry contributes immensely to the pollution of the environment, though they have started striving to use environmentally acceptable procedures for the production of textiles and their further treatments. Plasma treatment, for instance, is an environmentally-friendly technology that alters the surface properties of a textile, making it more adsorptive for nanoparticles. The consequence of an increased nanoparticle adsorption is the decreased content of nanoparticles in the bath \([21, 22]\). Plasma is the fourth state of matter or the state of gas in which a substantial part of molecules is dissociated and ionized. We use the so-called nonthermal plasma to treat textiles. It is a partially ionized gas that enables substrate reactions to take place without its thermal degradation \([23, 24]\). Plasma is created and maintained at a low and atmospheric pressure by using different systems of electrical power, such as the direct, radiofrequency, and microwave systems \([25, 26]\). Plasma treatment enables chemical modification of textiles by introducing new functional groups and active species, which alter the reactivity of the substrate. When plasma reacts with a substrate, new active species and functional groups are created on its surface. The longer we treat a substrate with plasma, the more we transcend into the sphere of etching, which leads to the increased nano- or microroughness of the surface \([27–30]\). Depending on the gas of choice, such as water vapour, oxygen, nitrogen or air, the concentration of carboxyl, carboxylic, and hydroxyl functional groups on the surface of a cotton fabric increases \([31–33]\). It has, however, been proven that for an increased adsorption of metal nanoparticles, the increased roughness of the substrate surface is favourable \([17, 32]\). The roughness of the surface is increased by means of plasma etching, where plasma causes desorption and elimination of by-products on the low on the substrate. To achieve a high degree of substrate roughness, we use the same gases as we do for achieving plasma functionalization; however, the treatment time and the reactor power must be higher \([34, 35]\). Because ZnO nanoparticles adsorb poorly onto cotton fibres, it is necessary to use high concentration of ZnO nanoparticles if we wish to achieve a sufficient protection against UV radiation. An increased adsorption of silver nanoparticles and titanium dioxide has so far been reached on plasma-treated cotton textiles \([22, 23]\), and an increased adsorption of ZnO nanoparticles by means of a low-pressure plasma is created in wet tetrafluoromethane \([36]\).

In this study, a cotton fabric was exposed to oxygen \((O_2)\) low-pressure RF plasma in different time intervals and later treated with ZnO nanoparticles. The purpose of the research was to increase the adsorption of ZnO nanoparticles and achieve excellent UV protective properties of cotton fabrics by using a low concentration of ZnO nanoparticles in the treating bath.

2. Experimental

2.1. Materials. Bleached and mercerised cotton fabric (weight: 119.2 g/m², number of warp threads: 52 threads/cm, number of weft threads: 26 threads/cm) was purchased from Tekstina d.d., Slovenia and ZnO nanoparticles (30 nm) were purchased from MK Impex Corp., Canada. Acetic acid was used from Sigma-Aldrich, ultrapure water was from MilliQ, Millipore, and ultrapure acids were from Merck, Suprapur.

2.2. Functionalization with Oxygen Plasma. Cotton samples were treated in low-pressure inductively coupled radiofrequency (IC-RF) plasma system \([36]\). The discharge chamber was a cylindrical Pyrex tube with a diameter of 27 cm and a length of 30 cm. Cotton samples were put onto a glass holder mounted in the centre of the discharge chamber. After closing the chamber, the pressure of 20 Pa was achieved by a double-stage rotary vane pump with a nominal pumping speed of 60 m³/h. A needle valve located on the aluminium plate opposite from the pumping side was used to adjust the pressure in the plasma reactor during continuous pumping. The valve was connected to a flask of \(O_2\) gas. During the experiments, a constant leak of \(O_2\) was established (partial pressure of approximately 60 Pa). A copper coil was connected to the plasma reactor and connected to a matching network. The network was connected to a radiofrequency generator. The generator operated at a standard industrial frequency of 27.12 MHz. The power was set to 400 W. The cotton samples were treated for 10, 20, or 30 s.

2.3. Functionalization with ZnO Nanoparticles. Functionalization of untreated and plasma-treated cotton samples was performed using 3% of ZnO nanoparticles, by dispersing ZnO powder in bidistilled water. The solution was stirred and simultaneously 1 mL/l \(\text{CH}_3\text{COOH}\) 30% was added dropwise. The solution was sonicated for 30 min and cotton samples were immersed into the solution at room temperature for 30 min. The ZnO-functionalized cotton samples were then foulard wrung with a wet pick-up of 100%, dried at 100°C for 5 min and cured in the oven at 150°C for 5 min.

2.4. X-Ray Photoelectron Spectroscopy. The surface composition of untreated and plasma-treated samples was studied using high-resolution X-ray photoelectron spectroscopy (XPS). The samples were mounted in a TFA XPS Physical Electronics XPS instrument. The base pressure in the XPS analysis chamber was approximately \(6 \times 10^{-8}\) Pa. The samples were excited with X-rays over a 400 μm spot with monochromatic Al \(K_{\alpha1,2}\) radiation at 1,486.6 eV. The photoelectrons...
were detected with a hemispherical analyser positioned at an angle of 45° to the sample surface. The energy resolution was approximately 0.5 eV. Survey-scan spectra were acquired at a pass energy of 187.85 eV with a 0.1 eV energy step. An additional electron gun was used to allow for surface neutralization during the measurements because the samples were insulators. The concentration of the different elements was determined using MultiPak v8.1c software from Physical Electronics, which was supplied with the spectrometer.

2.5. Mechanical Properties. The breaking strengths (N) and elongations (%) of the untreated and plasma-treated samples were analysed according to the ISO 13934-1:1999 standard. An Instron 6022 instrument was used for this purpose. Samples of cotton fabric (150 mm) were analysed along the warp direction using a preloading of 1 N and a speed of 100 mm/min. Samples were conditioned according to the ISO 13934-1:1999 standard method. The washing solution contained 4 g/L of SDC standard detergent. The solution was digested with a microwave-assisted digestion system (CEM MDS-2000) and a solution of 7 mL nitric acid and 1 mL hydrogen peroxide. The digested samples were cooled to room temperature and then diluted with 2% v/v nitric acid until their concentration was within the desired concentration range. The digestion procedure yielded clear solutions that were used in subsequent analyses.

2.7. UV Protection Factor Measurements. Untreated and plasma-treated ZnO-functionalized cotton samples were analysed for their UV protective properties on a Varian Cary 1E UV/VIS spectrophotometer containing a DRA-CA-301 integration sphere and Solar Screen software. The transmittance measurements and calculations of the ultraviolet protection factor (UPF) were carried out in accordance with the AATCC TM 183 standard. The UPF was calculated according to the following equation:

$$\text{UPF} = \frac{\sum_{\lambda=280}^{400} E_\lambda \cdot S_\lambda \cdot \Delta \lambda}{\sum_{\lambda=280}^{400} E_\lambda \cdot S_\lambda \cdot T_\lambda \cdot \Delta \lambda},$$

(1)

where $E_\lambda$ is the relative erythemal spectral effectiveness, $S_\lambda$ is the solar spectral irradiance, $T_\lambda$ is the spectral transmittance of the specimen, and $\Delta \lambda$ is the measured wavelength interval in nm.

The Australia-New Zealand Standard (AS/NZ 4399:1996) defines criteria for assessing the UV protective effectiveness of textiles and evaluation for labelling textile products with a protective function. The standard classifies textile products into three categories of protection, namely, excellent, very good, and good protection [37]. The values are in the range of 15 to 50 and the higher the value, the better the protection (Table 1).

2.8. Scanning Electron Microscopy. Untreated, plasma-treated, and ZnO-functionalized cotton samples were imaged using a scanning electron microscope (SEM; JEOL SEM type JSM-6060LV). All samples were coated with a thin layer of gold. The electron accelerating voltage was 10 kV.

2.9. Durability to Washing. The durability to washing was performed on untreated and plasma-treated ZnO-functionalized samples, washing them one and ten times in laboratory apparatus AATCC Atlas Launder-Ometer according to ISO 105-C06:1997 standard method. The washing solution contained 4 g/L of SDC standard detergent. The solution was heated to 40°C, and then the samples were inserted and washed for 45 minutes. After the washing, the samples were rinsed twice with deionized water for 1 min at 40°C and then dried at room temperature. The apparatus Launder-Ometer simulates the washing of the home washing machine. When 10 noncorroable stainless steel balls are added to the washing bath, one washing in Launder-Ometer corresponds to five home wash cycles.

### Table 1: Criteria and evaluation of UV protection effectiveness of textiles according to AS/NZ standard.

<table>
<thead>
<tr>
<th>Protection category</th>
<th>UPF value</th>
<th>% UV radiation blocked</th>
<th>UPF rating</th>
</tr>
</thead>
<tbody>
<tr>
<td>Excellent</td>
<td>40–50, 50, and more</td>
<td>More than 97.5%</td>
<td>40, 45, 50, 50+</td>
</tr>
<tr>
<td>Very good</td>
<td>25–39</td>
<td>95.9–97.4%</td>
<td>25, 30, 35</td>
</tr>
<tr>
<td>Good</td>
<td>15–24</td>
<td>93.3–95.8%</td>
<td>15, 20</td>
</tr>
</tbody>
</table>

### 3. Results and Discussion

Modification of surface properties of bleached and mercerized cotton fabrics in order to achieve excellent UV protective properties was performed by using oxygen plasma for different periods of time, that is, 10 s, 20 s, and 30 s, and by functionalization with ZnO nanoparticles. Information on the chemical composition and chemical bonds of the surface atoms of cotton samples after plasma treatment was obtained...
with XPS analysis. From the survey spectra, the surface composition was calculated and the results are presented in Table 2. On untreated cotton sample, small concentration of nitrogen (N) is observed which decreases after longer period of plasma treatment time. After plasma treatment, a lower concentration of carbon (C) and higher concentration of oxygen (O) were observed; however, no significant differences in chemical surface composition were observed for the samples that were treated for different periods of time. Due to the increased concentration of O and decreased concentration of C, the samples after plasma treatment have a higher O/C ratio. Increased O/C ratio after oxygen plasma treatment is due to the incorporation of oxygen containing functional groups onto the cellulose surface [38].

From the high-resolution XPS carbon C 1s spectra, the existence of chemical bonds among the surface atoms can be determined. Spectra C 1s (Figure 1) are presented for a case of untreated (Figure 1(a)) and plasma-treated samples (Figures 1(b)–1(d)). With the increasing plasma treatment time, the C-C bonds decrease and C-O bonds increase. The highest increase of C-O bonds is after treatment with oxygen plasma for 30 s. The O-C-O bonds increase by almost twofold after 10 s plasma treatment and then gradually decrease after longer plasma treatment time; however, the percentage of the O-C-O bonds remains higher than that of untreated samples. The same effect can be observed for O=C-O bonds.

Plasma treatment of cotton also changes the physical surface properties of fibres (Figure 2). Longer plasma treatment time causes more grooved and etched surface of the fibres, enhancing the roughness of the fibres surface. The microfibrillar structure of the fibres is more visible after plasma treatment. The upper individual microfibrils are stripped and separated from the fibres after plasma treatment and intertwined among each other on the fibres surface.

When such physical changes of surfaces occur upon plasma treatment, the mechanical properties of substrate have to be analysed. Any decrease of the textile mechanical properties would mean that the plasma etching effect was not limited only on the surface, but it occurred also in the bulk of the fibres. The results of the two most important mechanical properties, such as breaking strength and breaking elongation, were measured and are presented in Figures 3 and 4. From the results, it can be observed that untreated sample has the lowest value of breaking strength and elongation. Treating cotton fabric with plasma for 10 s increases the breaking strength for 18% and breaking elongation for 9%. Compared to 10 s plasma-treated sample, treating cotton fabric with plasma for longer time period does not dramatically increase the mechanical properties, and the changes are in the error limits. Here, it has to be emphasized that the mechanical properties did not worsen even after 30 s of plasma treatment. The reason for increased mechanical properties of plasma-treated cotton fabrics is probably due to the interlocking of microfibrils on the surface of fabric [31]. Plasma treatment enhances interyarn and interfiber friction due to etching [39]. Fabric breaks when most of the fibres in the yarn are broken. From SEM images, the ablation action of plasma on the fibres surface is visible. The rougher surface of individual fibres increases the friction between fibres within the yarn and also between the yarns. Due to this, slippage of fibres and yarns is decreased, leading to increased breaking strength. Increased breaking strength due to plasma treatment was also found for cellulose nonwoven fabric (viscose) and for protein-keratin fabric (wool) [40, 41].

The chemical and physical changes of plasma-treated fabrics are responsible for enhancing the adsorption properties of fibres towards ZnO nanoparticles (Figure 5). The quantity of Zn on plasma-treated samples increases after longer plasma treatment time due to the increased oxygen containing functional groups on the fibres surface (Figure 1) and increased surface roughness of fibres (Figure 2). Increased quantity of ZnO nanoparticles is also visible from SEM images of ZnO-functionalyzed samples (Figure 6). After longer plasma treatment time, nanoparticles are more evenly distributed and less of agglomerates are present on the surface of fibres.

Increased quantity of ZnO nanoparticles and their even distribution on the plasma-treated fibres increases the ultraviolet protection factor (UPF) of cotton fabric. In Table 3, the results of UV protective properties of untreated and plasma-treated samples are presented. The untreated and unfunctionalyzed sample does not provide a satisfactory UPF and consequently has an unrateable UPF rating. Functionalization of cotton fabric with ZnO nanoparticles provides good protection against harmful UV radiation, with UPF of 33.63 and UPF rating of 30. The textiles used as protection measures against UV radiation in countries with high UV index have to have excellent UV protection factor [42]. The excellent UPF was achieved on plasma-treated cotton fabric functionalized with ZnO nanoparticles (Table 3). Treating cotton fabric for 10 s with plasma increases the UPF from 33.63 to 58.52. Longer plasma treatment time increases the content of ZnO

### Table 2: The elemental composition (C, O, N), O/C ratio concentration and percentage of C-C, C-O, O-C-O, and O=C-O peaks presenting different chemical bonds of carbon atoms.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Elemental composition (at.%)</th>
<th>Atomic ratio O/C</th>
<th>C-C</th>
<th>C-O</th>
<th>O-C-O</th>
<th>O=C-O</th>
<th>O=C-O</th>
</tr>
</thead>
<tbody>
<tr>
<td>UN</td>
<td>68.2 30.3 1.5</td>
<td>0.44</td>
<td>40.4</td>
<td>45.9</td>
<td>13.6</td>
<td>5.0</td>
<td></td>
</tr>
<tr>
<td>P_10 s</td>
<td>55.3 43.4 1.3</td>
<td>0.78</td>
<td>12.4</td>
<td>52.6</td>
<td>24.9</td>
<td>10.0</td>
<td></td>
</tr>
<tr>
<td>P_20 s</td>
<td>55.0 44.0 1.0</td>
<td>0.80</td>
<td>14.3</td>
<td>56.0</td>
<td>21.1</td>
<td>8.6</td>
<td></td>
</tr>
<tr>
<td>P_30 s</td>
<td>54.9 44.2 0.9</td>
<td>0.81</td>
<td>12.4</td>
<td>63.7</td>
<td>18.9</td>
<td>5.0</td>
<td></td>
</tr>
</tbody>
</table>

UN: untreated, P_10 s: oxygen plasma-treated for 10 s, P_20 s: oxygen plasma-treated for 20 s, and P_30 s: oxygen plasma-treated for 30 s.
Figure 1: High energy-resolution XPS spectra C 1s obtained from the surface of cotton samples: (a) untreated, (b) plasma-treated for 10 s, (c) plasma-treated for 20 s, and (d) plasma-treated for 30 s.

Table 3: Ultraviolet protection factor (UPF), transmission ($T$) in the UVA and UVB region, ultraviolet radiation (UVR), UVA-, and UVB-blocking properties, and UPF rating of the cotton samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>UPF</th>
<th>$T$ (UVA) (%)</th>
<th>$T$ (UVB) (%)</th>
<th>$T$ (UVR) (%)</th>
<th>UVA % blocking</th>
<th>UVB % blocking</th>
<th>UPF rating</th>
</tr>
</thead>
<tbody>
<tr>
<td>UN</td>
<td>4.12</td>
<td>28.42</td>
<td>26.64</td>
<td>26.60</td>
<td>71.58</td>
<td>76.36</td>
<td>Unrateable</td>
</tr>
<tr>
<td>UN + ZnO</td>
<td>33.63</td>
<td>7.44</td>
<td>2.95</td>
<td>6.04</td>
<td>92.56</td>
<td>97.06</td>
<td>30</td>
</tr>
<tr>
<td>P$_{10}$ s + ZnO</td>
<td>58.52</td>
<td>6.73</td>
<td>1.59</td>
<td>5.16</td>
<td>93.27</td>
<td>98.41</td>
<td>50+</td>
</tr>
<tr>
<td>P$_{20}$ s + ZnO</td>
<td>61.86</td>
<td>6.41</td>
<td>1.46</td>
<td>4.90</td>
<td>93.59</td>
<td>98.54</td>
<td>50+</td>
</tr>
<tr>
<td>P$_{30}$ s + ZnO</td>
<td>65.93</td>
<td>6.13</td>
<td>1.53</td>
<td>4.727</td>
<td>93.87</td>
<td>98.47</td>
<td>50+</td>
</tr>
</tbody>
</table>

UN: untreated, P$_{10}$ s: oxygen plasma-treated for 10 s, P$_{20}$ s: oxygen plasma-treated for 20 s, P$_{30}$ s: oxygen plasma-treated for 30 s, and ZnO: functionalization with ZnO nanoparticles.
nanoparticles on fibres, increasing the UPF up to 65.93, giving the cotton fabric UPF rating 50+. Durability of treatments to washing was performed for one and ten home washing cycles. The results of UV protection factor of unwashed and washed samples are presented in Figure 7. After one performed wash, the UPF does not decrease extensively. The untreated sample retains the UPF rating of 30, while the UPF rating decreases for plasma-treated for 10 and 20 s from 50+ to 30 and 40, respectively. The sample treated with plasma for 30 s retains the UPF rating 50+ (UPF = 57.70), meaning that it has the best wash stability. However, since nanoparticles are mechanically bound to the cotton fabric, there are no chemical forces that would keep the nanoparticles on the fabric after ten washings. In our research, no binders were used in functionalization of cotton with ZnO nanoparticles, meaning that no chemical crosslinking between the nanoparticles and cotton was possible. Poor wash stability was also reported by other researchers [9, 43]. Nevertheless, using our system, higher quantity of ZnO nanoparticles was adsorbed onto plasma-treated samples, and furthermore excellent UV protective properties...
were achieved. A great advantage of the configuration of the experimental system used in this study is extremely low power density. The volume of the plasma reactor is about 15 L and the discharge power is 400 W so the specific power is solely 26 W L$^{-1}$. The energy consumption is therefore very low compared to the conventional plasma reactors. The electrodeless configuration and proper selection of plasma-facing materials allowed for a negligible loss of neutral reactive oxygen particles such as oxygen atoms (O-atoms) in the ground state. The resultant O-atom flux on the sample surface is therefore large enough to allow for the saturation of the textile surface with oxygen even in 10 s. Table 2 reveals that the concentration of oxygen on plasma-treated samples increased marginally for the sample treated for 30 s, only 0.8% as compared to the sample treated for 10 s. This value is within the accuracy of XPS. On the other hand, Figure 5 reveals well over 10% increase of the nanoparticles uptake from 10 to 30 s treatments. The discrepancy is explained by functionalization of fibres deep inside the fabrics at prolonged treatment times. The O-atoms diffuse into the interfibrillar...
space and cause functionalization of inner fibrils. The loss rate by heterogeneous surface recombination is therefore overcome by a large fluence. It is difficult to use this effect in conventional plasma reactors with electrodes since the ratio between dissociation and ionization fractions is not as favourable as in our reactor. In a conventional reactor, the ion density is rather large (as compared to the neutral O-atom density), so the textiles are subject to thermal loads caused by ion bombardment and surface neutralization; both effects are highly exothermic. A conventional reactor therefore does not allow for such deep functionalization since the required treatment time would be long enough to cause irreversible modifications of the surface fibrils due to heating.

4. Conclusions

Using textiles as a protection against harmful UV radiation caused by the Sun and other sources of UV radiation is crucial in reducing skin cancer. Since ZnO nanoparticles have excellent UV protective properties but have poor adsorption capacity towards textile materials, the aim of the research was to increase UV protection factor of cotton fabric using oxygen plasma. Cotton samples were treated with oxygen low-pressure RF plasma for different periods of time and functionalized with 3% of ZnO nanoparticles. The results showed that longer plasma treatment time causes higher adsorption of nanoparticles due to the increased concentration of oxygen containing groups on the fibres surface and because of the increased surface roughness of fibres. Plasma-treated cotton fabrics gained excellent UV protective properties with UPF rating 50+ and can be used as a protective measures in countries with high UV index.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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