Plasmonic colored nanopaper: a potential preventive healthcare tool against threats emerging from uncontrolled UV exposure

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Keywords: wearable devices, smart packaging, nanoplasmonics, nanophotonics, nanocellulose

Supplementary material for this article is available online

Abstract
Preventive healthcare is crucial to hinder or delay the onset of disease, furthermore it contributes to healthy and productive lifestyles and saves resources allocated to public health. Herein, we explore how the plasmonic coupling of silver and gold nanoparticles embedded within nanopaper allows for potential preventive healthcare tools based on a change in plasmonic color. Particularly, we selected UV radiation exposure as a potential threat to health to be monitored via plasmonic colored nanopaper (PCN). Uncontrolled UV radiation exposure is not only known to provoke epidermal damage, but also to trigger leaching of hazardous compounds from polycarbonate containers. In this context, we engineered UV-responsive PCN devices whose sensing mechanism is based on UV photodegradation of silver nanoparticles. Since absorbance and scattering of metal nanoparticles strongly depend on their size and inter-particle distance, the resulting PCN detectors are able to warn of the potential UV radiation-induced threat via a visually observable plasmonic color change with a yellowish/reddish transition. Epidermal experiments with tattoo-like PCN devices prove the resulting detectors can change in color upon safe dose of sun exposure. Moreover, PCN detectors stuck on polycarbonate containers also change in color after moderate sun exposure. This cost-effective and lightweight nanophotonic device leads to a versatile preventive healthcare tool.

1. Introduction
Given the increase in the number of deaths and the upsurge in economic burden derived from chronic diseases, preventive healthcare is nowadays a meaningful global public health topic [1]. For example, recently in Florida, a specialized network of affiliated primary care physicians who employed a program of personalized preventive health care reported reduced utilization of emergency room and urgent care services compared to nonmembers of such a network. The network also reported positive health care expenditure outcomes and improved health management [2]. Therefore, approaches intended for furthering lifestyle modifications and managing health behavioral changes are crucial to improve health outcomes.

Heat and energy provided by sun radiation are the cornerstone of life on Earth and its survival. Electromagnetic wavelengths reaching the Earth’s surface include near infrared radiation (750–1400 nm), visible light (380–700 nm) and ultraviolet radiation (100–380 nm), which can be divided into UV-A (320–380 nm), UV-B (280–320 nm) and UV-C (100–280 nm) subgroups [3]. On the one hand, moderate UV radiation exposure offers therapeutic health outcomes such as synthesis of adequate levels of vitamin D and beneficial modulation of blood pressure; furthermore, it is a valuable factor in mental healthcare [4]. On the other hand, uncontrolled UV radiation exposure can harm the skin and eyes as well as inhibit the immune system. In fact, UV radiation is the major and most preventable risk factor for the development of skin cancer [3], which affect millions of people annually [5]. Furthermore, uncontrolled UV exposure of polycarbonate food
packaging has been reported to provoke leaching of As and Sb from these containers at levels exceeding US EPA drinking water standards [6], which can be consumed inadvertently from bottled beverages. As a consequence, health risks from the consumption of bottled beverages increase upon UV/sun exposure. All in all, uncontrolled UV radiation represents a public health threat. Therefore, UV radiation monitoring is important to safeguard public health and promote preventive healthcare.

Flexible UV detectors have been recently engineered by using paper, dyes and polymer technology as well as nanoplasmonics based on silver nanoparticles (AgNPs) embedded within nanopaper. The former require several photoactive layers or extra filters to work properly in different skin types, increasing complexity and cost of these devices [7, 8]. Furthermore, their epidermal performance has not been reported. The latter are simpler and their epidermal behavior has been reported; however, as they display yellowish colors, their change in color may result difficult to monitor visually [9]. Herein, aiming at monitoring safe doses of UV exposure, we engineered plasmonic colored nanopaper (PCN) made of AgNPs and gold nanoparticles (AuNPs) as a potential preventive healthcare tool. Importantly, the physicochemical properties of AgNPs are currently employed in more than 200 consumer goods, including cosmetics and food products [10], whereas AuNPs have been studied in potential medical applications for centuries and they are generally reported to be biocompatible [11]. Moreover, nanopaper has been reported to be an advantageous optically transparent, flexible, lightweight and biocompatible optical sensing platform [12, 13]. On the one hand, AgNPs can undergo a photolysis via UV radiation, resulting in size decrease [10, 14, 15]. On the other hand, absorbance and scattering of metal nanoparticles strongly depend on their size and inter-particle distance [16–18]. In this context, we take advantage of these phenomena and investigate how modulation of the plasmonic coupling of UV-responsive AgNPs and AuNPs embedded within nanopaper leads to a versatile UV detection platform based on plasmonic color. The operational principle of this approach is depicted in figure 1. The resulting PCN is able to warn of the potential UV radiation-induced threat via a visually observable plasmonic color change, generally from yellowish to reddish color, so as the observer may take a proper action once the color change is spotted. We demonstrate that the resulting PCN detectors are useful as wearable devices in epidermal settings, as well as smart UV-responsive labels stuck on polycarbonate containers.

2. Experimental section

2.1. Reagents and equipment

All the reagents were of analytical grade and handled according to the material safety data sheets provided by the respective manufacturer. NaOH, AgNO₃, HAuCl₄ and Poly(ethyleneimine) (PEI) solution were acquired from Sigma-Aldrich (Toluca, Mexico State, Mexico). Bacterial cellulose nanopaper was purchased from Nano Novin Polymer Co. (Mazandaran, Iran). According to previous characterizations [12], the employed nanocellulose has an average fiber diameter around ∼45 nm, with a length of c.a. ∼10 μm. The utilized nanopaper also displays a crystallinity of 82% and a Young’s modulus of 17 GPa. All aqueous solutions were prepared in ultrapure water. Tattoo paper was from Silhouette America Inc. (Orem, Utah, USA). A spectrophotometer was employed to analyze UV–vis absorbance (Cytation 5, BioTek, Winooski, Vermont, USA). An Oriel Sol 3A solar simulator (Newport, Irvine, California, USA) was utilized to expose PCN to the respective UV light (total power, 1000 W m⁻²). The pictures were acquired via a mobile phone camera (Moto G 4 Plus, Motorola, Chicago,
Illinois, USA) and DroidCam 6.0.0 (Dev47Apps). SEM analysis was performed through an equipment JSM-7800F (Jeol USA Inc., Peabody, Massachusetts, USA)—the PCN samples were analyzed onto adhesive carbon tape. The particle size distributions were determined utilizing ImageJ 1.51j8 (Wayne Rasband, National Institutes of Health, USA). A total area of c.a. 1.44 μm² per sample was explored and a size analysis of 1120 particles per sample was recorded via the resultant SEM micrographs. Informed signed consent was obtained from the volunteers who kindly tested PCN devices.

### 2.2. Synthesis of PCN

PEI working solution was prepared by adding 500 mg of stock PEI solution [50% (w/v) in H₂O] in 5 ml of ultrapure water as a reducing agent. HAuCl₄ working solution was prepared at c.a. 0.9% (w/v) in ultrapure water as AuNPs precursor. AgNO₃ working solution was prepared at 0.1% (w/v) in ultrapure water as AgNPs precursor. 11 different types of PCN were synthesized by merging previously reported methods [9, 12].

The different PCN were synthesized according to the characteristics detailed in table 1. Firstly, AuNPs were embedded within nanopaper by soaking and stirring 15 square pieces of nanopaper (2.5 × 2.5 cm²) in 50 ml of boiled ultrapure water containing the corresponding AuNPs precursor volume (see table 1). The respective volume of PEI working solution (the same volume as the AuNPs precursor volume) was then quickly added into the reaction mixture and boiling was kept for 10 min. Nanopaper embedding AuNPs was then washed three times using ultrapure water to remove non-embedded AuNPs. After that, AgNPs were synthesized within AuNPs-decorated nanopaper by adding the respective AgNPs precursor volume in 50 ml of boiled ultrapure water containing the corresponding AuNPs precursor volume (see table 1). The resulting volume of PEI working solution (the same volume as the AuNPs precursor volume) was then quickly added into the reaction mixture and boiling was kept for 10 min. Nanopaper embedding AuNPs was then washed three times using ultrapure water to remove non-embedded AuNPs. After that, AgNPs were synthesized within AuNPs-decorated nanopaper by adding the respective AgNPs precursor volume in 50 ml of boiled ultrapure water and following previously reported methods [9, 12]. The synthesis of AgNPs was performed during 20 min. As the synthesis is carried out in the liquid phase, the resulting PCN is eventually dried following the aforementioned methods [9, 12]. Once PCN was dried, it was cut in form of circles using a punch tool of 6 mm diameter. The resulting PCN can be readily stuck on skin using tattoo paper. Alternatively, the PCN devices were stuck on polycarbonate containers using double-sided adhesive tape. To prevent undesired light interactions, the PCN devices were generally coated with aluminum foil before utilization. Previous research reports that plasmonic nanopaper is stable for at least 6 months [12].

In order to perform the studies of UV effect on PCN, the PCN devices were placed in a solar simulator at a distance around 17 cm from the light source. We also followed previously reported methods to avoid any possible change in refractive index due to humidity supplied by perspiration [9].

### 2.3. Safety

Importantly, no skin irritation was observed or reported by the volunteers.

### 3. Results and discussion

Plasmonic colors are structural colors arising from resonant interactions between light and metallic nanomaterials [19]. Suspensions of relatively small AgNPs (<25 nm) generally exhibit a yellowish color [20], whereas suspensions of spherical AuNPs (c.a. <95 nm) typically display a reddish color [21]. The respective colors are also displayed in noble metal nanoparticles embedded within nanopaper [12]. As we sought to optimize a UV-responsive device showing modulation in plasmonic color with a yellowish/reddish transition by embedding AgNPs and AuNPs within nanopaper, considering previous methods [9, 12], we synthesized
several PCNs with different AgNPs and AuNPs precursor volumes. The synthesis conditions are specified in the experimental section and table 1. The UV–vis absorbance peaks of the resulting nanocomposites are also itemized in table 1. As shown in figure 2, we characterized the resulting PCNs using UV–vis spectroscopy. On the one hand, we noted that most of the resulting nanocomposites displayed an absorbance peak of less than $\sim 453$ nm, particularly PCN1, 2, 3, 4, 5, 7, 9, 10 and 11, suggesting predominant scattering and absorbance of AgNPs in these materials. On the other hand, PCN8 had an absorbance peak at $\sim 522$ nm, suggesting a prevailing scattering and absorbance of AuNPs. However, PCN6 exhibited an absorbance peak at $\sim 490$ nm, suggesting a hybrid scattering and absorbance, that is, between those of AgNPs and those of AuNPs. In this context, PCN6 was put forward as an optimal candidate in terms of sensitive modulation in plasmonic color with a yellowish/reddish transition.

With our characterized PCNs in hand, aiming at testing their performance as UV detectors, we exposed the synthesized nanocomposites to controlled UV radiation using a solar simulator (at 1000 W m$^{-2}$) as detailed in the experimental section. Although naturally available doses of sun/UV radiation depend on many factors including cloud cover, ozone, altitude, seasons, Earth surface characteristics, latitude, land cover and time of day [22], a recommended/safe dose of sun/UV radiation to generate adequate levels of vitamin D may be around 15 min at 1000 W m$^{-2}$ [9, 23, 24]. Therefore, we manufactured tattoo-like detectors with all the resulting PCNs.

![Figure 2. UV–vis spectroscopy analysis and pictures of the resulting plasmonic colored nanopapers. (A) PCNs synthesized by varying AgNPs precursor volume (see table 1). (B) PCNs synthesized by varying AuNPs precursor volume (see table 1). Each spectrum represent the mean of at least three samples of the respective synthesis. A white background color was utilized to take these pictures.](image-url)
and as proof of concept, we performed experiments in epidermal settings during 15 min of solar simulator/UV exposure. 4 volunteers participated in this series of experiments, where we monitored the respective UV–vis spectral response of the utilized PCNs before and after solar simulator/UV exposure. As experimental evidence, pictures of the tattoo-like PCN devices and their UV-responsive character in epidermal conditions are shown in figure S1–2 is available online at stacks.iop.org/JPHOTON/1/04LT01/mmedia. The resulting UV–vis spectra are displayed in figures S3–S6.

To assess changes in the observed spectra quantitatively, we divided the area under the curve of the spectrum of the respective PCN upon sun exposure ($A_\text{f}$) into the original area under the curve of the respective spectrum ($A_0$), that is, before UV exposure. Figures S3–S6 also display the estimated $A_\text{f}/A_0$ relationships. The $A_\text{f}/A_0$ relationship supported visual observations related to changes in plasmonic color, confirming that generally PCN6 undergoes the most sensitive modulations in terms of scattering and absorbance. However, visually observable color of transparent nanoplasmonic substrates can strongly depend on a given background color [25]. In fact, the resulting PCNs are able to change from a yellowish color to a reddish color, or vice versa, depending on both, the PCN type and the background provided by the user’s skin color. For example, PCN4 looks originally yellowish onto the skin of volunteer 1 and reddish upon moderate UV exposure, whereas PCN6 looks originally reddish onto the skin of volunteer 3 and yellowish upon moderate UV exposure, see figures 3, S2 and S7. Therefore, we confirmed that the background color contributed by the explored skin also plays a critical role in the final visually observable plasmonic color [9]. As a consequence, the PCN devices can be potentially adapted according to different skin types in order to warn of a safe dose of UV exposure.

Overall, we also found a blue-shift in the studied PCN spectra upon UV exposure (see figures S3–S6), this suggest that the UV-responsive AgNPs embedded within PCNs undergo a size reduction [16]. Although perspiration (which consists in water, minerals, lactate, and urea) [26] is also able to release Ag ions from AgNPs [27, 28], previously performed control experiments confirm that perspiration itself is not able to trigger dramatic AgNPs size reduction leading to a color change in nanoplasmonic UV detectors. In fact this phenomenon only occurs in epidermal conditions upon UV exposure [9]. Encouraged by these results, we explored the nanoparticles size distribution of our PCN devices via scanning electron microscopy (SEM), as detailed in the experimental section. Particularly, we performed a SEM analysis of PCN4 and PCN6 behavior upon sun exposure in epidermal conditions (volunteer 1 and volunteer 3, respectively). Although it is hard to distinguish AuNPs from AgNPs in the resulting SEM micrographs, image processing and statistical analysis helped us to determine the respective particle size distributions (see details in the experimental section). Figure 3(B)(e)–(f) and (C)(e)–(f) display the resultant particle size distribution analysis ($n = 1220$). Although an important overlapping in the size distribution of the respective AgNPs and AuNPs may be present around 6–7 nm, it is clear that the main AuNPs particle size is around 7 nm before and after UV exposure, whereas the main AgNPs size is reduced from c.a. 5 to c.a. 2 nm. Therefore, we indeed found that the particle size distribution of AgNPs embedded within PCN experiments a size reduction. Interestingly, we discovered a balanced distribution between AgNPs and AuNPs in PCN6, that is to say, the percentage of the main AgNPs particle size is around 13%–14%, which may explain the aforementioned hybrid scattering and absorbance character of PCN6. Table 2 summarizes these observations. Figures 3(B)(g) and (C)(g) show the UV–vis spectral response of the explored phenomena, confirming the aforementioned blue-shift.

Recently, storage conditions for bottled beverages have been reported to be able to impact human health risk potentials related to inadvertent consume of hazardous compounds [6]. Considering that (a) leaching of As and Sb from polycarbonate containers at levels exceeding US EPA drinking water standards may occur upon 12 d of sun exposure [6], and (b) our UV detectors are intended for taking preventive actions; as proof of concept, we sought to find sensitive modulation of plasmonic color within 60 min of UV exposure. To this end, as detailed in the experimental section, we stuck all the available PCNs on polycarbonate containers and characterized their UV–vis absorbance behavior across 0, 15, 30, 45 and 60 min of UV exposure, see figure 4(A). The employed solar simulator operated at 1000 W m$^{-2}$, and we also determined the respective $A_\text{f}/A_0$ relationships. Figure S8 details this series of experiments. As shown in figure 4(B), PCN6 also displayed the most sensitive modulation in terms of changes in scattering and absorbance estimated by the respective $A_\text{f}/A_0$ relationship. Interestingly, in the explored conditions, the UV–vis spectral behavior of PC6 firstly displays a blue-shift (15 min) followed by a red-shift (30, 45, 60 min), see figure 4(C). This phenomenon is also caused by photodegradation of AgNPs and gradual predominance of AuNPs embedded within PCN6. Eventually, this leads to a plasmonic color transition from yellowish to reddish color, thus, demonstrating that PCN6 can be effectively exploited as a smart nanoplasmonic label warning of potentially harmful UV exposure in food packaging, see pictures in figure 4(C).

It is worth mentioning that the plasmonic color modulation of PCN devices unfolding in epidermal settings is not comparable to that occurring on polycarbonate containers, as the material interfaces are completely different. Firstly, perspiration exclusively combined with UV exposure may affect the studied phenomenon [9]. Secondly, as mentioned above, visually observable color of transparent nanoplasmonic substrates can strongly
Figure 3. Epidermal behavior of the resulting PCN devices. (A). Pictures of wearable PCN devices before and after 15 min of UV exposure. (B) Characterization of PCN4 in epidermal settings. B(a), B(b), PCN4 before (a) and after (b) UV exposure (volunteer 1). B(c)–(d), SEM micrographs of PCN4 before (c) and after (d) UV exposure. The scale bars represent 50 nm. B(e)–(f), Particle size distribution of PCN4 before (e) and after UV exposure (f). Bg, UV–vis spectral behavior of PCN4 before and after UV exposure. (C) Characterization of PCN6 in epidermal settings. C(a)–(b), PCN6 before (a) and after (b) UV exposure (volunteer 3). C(c)–(d), SEM micrographs of PCN6 before (c) and after (d) UV exposure. The scale bars represent 50 nm. C(e)–(f), Particle size distribution of PCN6 before (e) and after UV exposure (f). C(g), UV–vis spectral behavior of PCN6 before and after UV exposure.

Table 2. Features of the explored nanoparticle size distributions.

| Volunteer PCN | Main AgNPs particle size |  | Main AuNPs particle size |
|---------------|--------------------------|---|--------------------------|
|               | Before UV exposure       | After UV exposure | Before UV exposure       | After UV exposure |
| V1/PCN4       | ~5 nm (~15%)             | ~2 nm (~16%)       | ~7 nm (~10%)            | ~7 nm (~11%)       |
| V3/PCN6       | ~4 nm (~13%)             | ~2 nm (~24%)       | ~7 nm (~14%)            | ~7 nm (~7%)        |

* Percentage of the total nanoparticle population (n = 1120).
Figure 4. PCN devices as UV detectors on polycarbonate food packaging. (A). Pictures of PCN devices exposed across different UV exposure times. A transparent background (polycarbonate bottle) was utilized to take these pictures. (B). A/A₀ relationships estimated from the exposure of different PCN devices at different times. (C). UV–vis spectral response of PCN6 across different UV exposure times. Each spectrum represents the mean of at least three spectra.
depend on a given background color [25]. Therefore, this may explain different behavior in terms of plasmonic color modulation of PCN devices in the explored proof of concepts.

4. Conclusion

UV radiation monitoring is important to protect public health and further preventive healthcare. We studied how the plasmonic coupling of AgNPs and AuNPs embedded within optically transparent nanopaper can be utilized to engineer versatile UV detectors, where AgNPs operate as a UV-responsive agent and AuNPs function as a plasmonic color contrast agent. Given an optimized content of the embedded noble metal nanoparticles, we managed to achieve highly sensitive modulation of the resultant plasmonic color with a yellowish/reddish transition. Our lightweight and flexible PCNs were proven useful as a potential preventive healthcare tool against threats derived from uncontrolled UV exposure via wearable/adhesive devices, particularly to avoid either UV-induced skin damage or inadvertently consumption of hazardous agents released from bottled beverages upon UV exposure. As far as we are concerned, we are reporting the first application of nanophotonic devices based on plasmonic color devoted to healthcare.

Acknowledgments

The authors acknowledge the financial support from CONACYT (Mexico, Grant No. 293523, Apoyo al Fortalecimiento de la Infraestructura Científica y Tecnológica 2018; Grant No. 299058, Laboratorio Nacional de Micro y Nanofluídica). LF-A acknowledges financial support from Centro de Investigaciones en Óptica, AC (Academic Department). The authors also acknowledge the technical support provided by M C Albor-Cortes (SEM technician at CIO).

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