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Advances in Inorganic Nano Sunscreens for UV Protection and Biocompatibility Enhancement

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Abstract:
Excessive ultraviolet (UV) radiation increases skin disease risk, which is a global health issue. Sunscreen is an effective way to reduce UV radiation. While inorganic nano ZnO and TiO2 have occupied partial sunscreen market in recent years due to their high efficiency and good photostability, there is still improvement space in sunscreen efficiency, biological safety and customization. This paper reviews the recent literature on inorganic nano sunscreen modification. To improve sunscreen performance, the inorganic nanoparticles can be loaded on scaffold materials or doped with specific metal elements. For their biological safety, modification with certain natural polymers to eliminate reactive oxygen species (ROS) and the size/structure control to decrease skin penetration are effective. Adding dark-colored materials and skin-care materials to inorganic nano sunscreen formulations helps them better commercialization. However, most of these studies are still in the laboratory stage, requiring further research. Hopefully, more enhancement mechanisms and available modification materials are expected to be developed in the future.

Keywords: Inorganic nano sunscreen, ultraviolet protection, nanoparticle dispersion, metal doping, sunscreen biocompatibility.

1. Introduction

Ultraviolet (UV) radiation, given by the sun and many artificial light sources, is usually everywhere in everyone's daily life. As a type of electromagnetic wave, UV contains certain amount of energy, inducing human skin to produce health-beneficial Vitamin D in a moderate intensity. Excessive UV, however, is a significant environmental factor to cause dermatological conditions, sunburn, skin allergy and skin cancer [1]. Thus, it's important to reduce intense UV exposure to skin for skin disease prevention.

Sunscreen is one of the most effective methods to reduce UV injury nowadays. Different types of sunscreens have been developed from early 20th century, which can be divided into two categories, chemical sunscreens like Octinoxate, Avobenzone and Bemotrizinol, and physical sunscreens like TiO_2, ZnO and CeO_2 [1]. These sunscreens are capable to block UV-A and/or UV-B, but there remain drawbacks, such as photosensitivity of chemical ones and white cast of physical ones [2,3].

Nanotechnology has been developed rapidly for decades. And in recent years, nanoparticles have been applied in sunscreen development for their unique physical and chemical properties. Nano TiO_2 and Nano ZnO, with enhanced UV absorption and scattering and less white cast, are widely used for commercial nano sunscreens [4]. Some popular products contain nano TiO_2 and/or ZnO, for example, Anthelios Mineral One SPF 50+ (La Roche-Posay), Eryfotona Actinica SPF 50+ (ISDIN) and Natural Sun Cream SPF 50 (Isntree). Although they are well commercialized, efficiency problem and safety concern are revealed over time. Typically, there are two main directions to solve the issues, inorganic-inorganic composite and inorganic-organic hybrid. Common materials in cosmetics and biomedical field like kaolinite, lignin and cellulose show their potential in nano sunscreen development. Through different mechanisms, nano sunscreen can have better UV block effect, lower toxic side effects and etc.

By systematically analyzing recent advances in nanotechnology-based sunscreens, this study identifies the key factors that influence the efficacy and safety of nano sunscreens, hopefully providing practical ideas for the design of new products that are not only efficient and safe but also multifunctional and customizable.

2. Sunscreen Enhancing Mechanism of Inorganic Nanoparticles

The main sunscreen mechanisms of TiO_2 and ZnO are UV absorption and UV scattering. TiO_2 and ZnO are natural n- type semiconductors, so they have semiconductor properties such as the band gap. TiO_2, with a high refraction index, has a band gap which mainly depends on the crystal structure such as 3.0 eV for rutile phase and 3.2 eV for anatase phase. ZnO has a band gap of 3.3 eV, which allows ZnO to provide broad-spectrum protection from UV-A and UV-B. Research in recent years has shown that both TiO_2 and ZnO mainly achieve the sunscreen effect through the mechanism of UV absorption through the semiconductor band gap, while the proportion of UV scattering is relatively small [5].

Nanotechnology promotes huge enhancement on the effectiveness of inorganic sunscreens. According to Mie theory, a particle has stronger UV absorption and scattering when its size fits UV wavelength, where the scale coefficient $x = 2\pi r/\lambda \approx 1$. Nanoscale TiO_2 and ZnO have suitable sizes to absorb and scatter UV, making them behave better than micron-scale ones [6]. Size reduction to nanoscale also reduces the scattering in the visible light region significantly, greatly ameliorating the white cast issue of traditional physical sunscreens. On the other hand, larger specific surface area provides more active sites per unit mass or unit area to interact with UV, improving sunscreen efficiency as well.

Although nanotechnology has improved inorganic sunscreens, it has also introduced new problems to be solved, the most significant among which is biotoxicity. The nano-scale size and the photocatalytic activity of inorganic sunscreen nanoparticles may cause cytotoxicity and many other damages [7]. For inorganic nano sunscreens, it is important to meet diverse needs from consumers as well, and the flexible modification allows them to achieve it.

3. Enhancement of Inorganic Nano Sunscreen's UV Protection Capability

Many countries and regions have different requirements for the maximum content of nano TiO_2 and nano ZnO in sunscreen products. The EU and China, for example, require the content to be no more than 25%. Under the restriction, only a single active ingredient of ZnO or TiO_2 in a product is hard to achieve the target sunscreen effect, and the inevitable aggregation of inorganic nanoparticles greatly reduce their sunscreen effect as well. Based on these, sunscreen performance can be enhanced mainly from several aspects. The dispersibility of nanoparticles should be improved, exposing more active sites and enhancing the scattering reflection of the particles. The absorption spectrum of inorganic nano-sunscreen particles should be adjusted to a suitable width to cover the UV-A and UV-B, and even the visible blue-violet light region.

3.1 Reduction of Sunscreen Nanoparticle Aggregation

Due to the high specific surface energy, nanoparticles tend to aggregate during production and use, which reduces their ability to shield against UV rays. One effective way to reduce the nanoparticle aggregation and recover their UV shielding is to load them on some scaffold carriers. Wang et al. loaded nano ZnO to Kaolinite, a widely used inorganic material in cosmetics and biomedicine, to reduce the aggregation based on bidirectional dispersion. As a result, the composite material decreases the UV transmittance of nano ZnO significantly from 32% to 18% [8] (Fig. 1).

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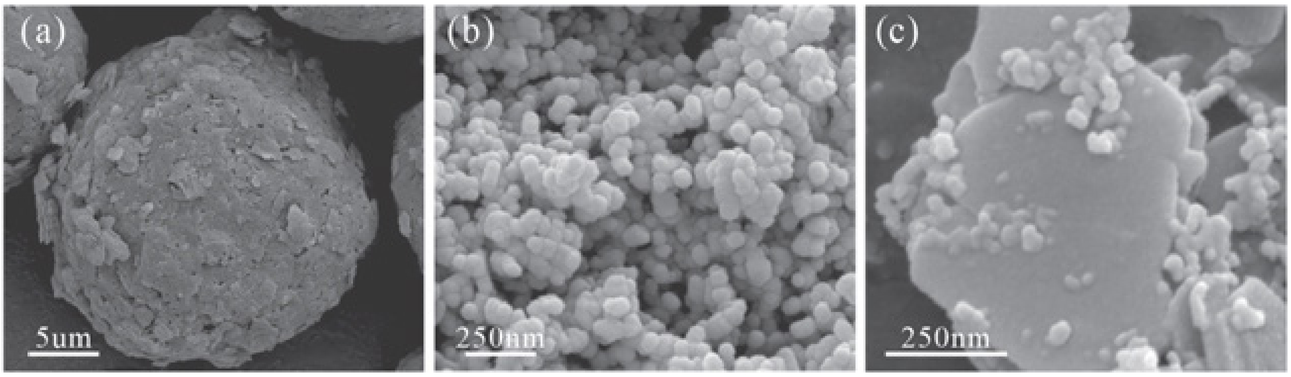

Fig. 1 SEM images of kaolinite loading to enhance the sunscreen effect of nano zinc oxide (a) flake kaolinite aggregated into spheres (b) Nano-ZnO by direct precipitation method (c) Nano-ZnO@Kaolinite [8]

Similar to inorganic carriers, loading inorganic nanoparticles onto natural polymer derivatives with certain mechanical strength can also improve UV protection in different ways. For example, cellulose is a natural polymer composed of glucose monomers. One of its derivatives, microcrystalline cellulose (MCC), is often used as a loading material in cosmetics and medicine. In Yu et al.'s experiment, nano TiO2 was coated onto MCC surface in disorder via the low-temperature hydrolysis method, resulting in photon localization, which means confining the incident photon in the TiO2 nanoparticle, to enhance the UV absorption. In addition, the crystal size and crystal structure of nano TiO2 significantly improved the UV absorption and scattering [9] (Fig. 2A). Attaching nano-TiO2 to cellulose nanofibers (CNF), a mesh cellulose derivative, can also enhance the UV absorption ability of TiO2 [10] (Fig. 2B).

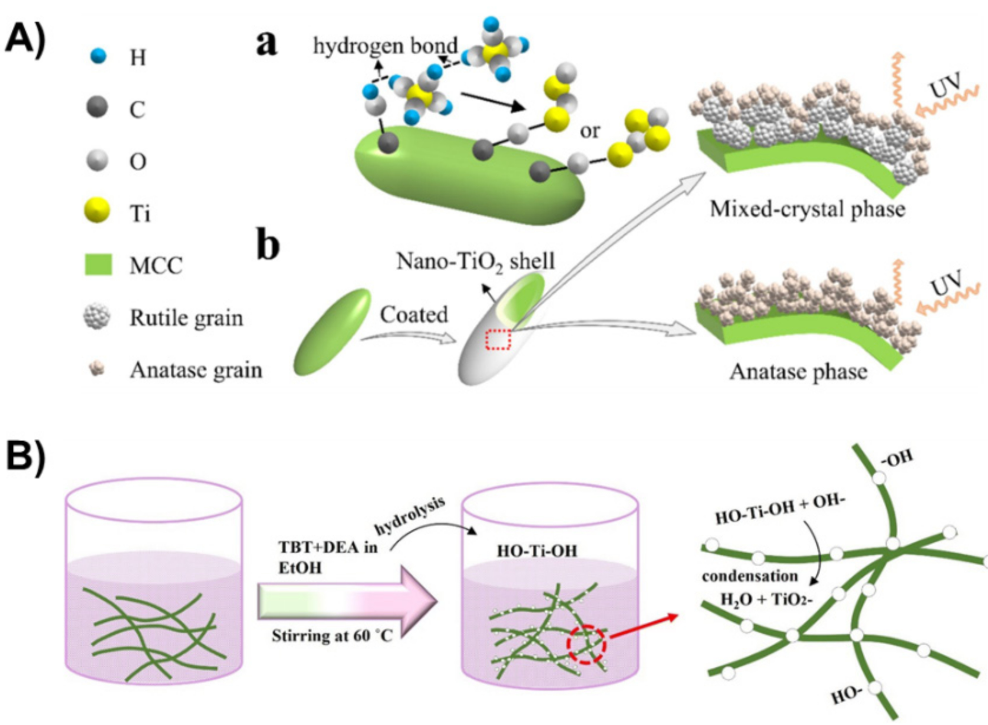

Fig. 2 The structure of inorganic nano sunscreen particles loaded onto organic scaffolds to improve sunscreen performance.

A) The disordered loading structure of Nano-TiO2@Microcrystalline cellulose leads to photon localization [9].

B) The structure of Nano-TiO2@Cellulose Nanofiber [10]

Surface modification is another way to reduce the aggregation of sunscreen nanoparticles, and certain physical or chemical properties can be used to estimate the effect. Takekawa et al. found that the sunscreen layer flatness would enhance the UV shielding ability of nano TiO2, and pseudo-HLB (hydrophilic-lipophilic balance), a calculated value based on chemical structure, is the key factor to predict the flatness [11].

3.2 Change in the Band Gap of Inorganic Sunscreen Nanoparticles

Doping other elements into inorganic nanoparticles can improve their sunscreen effect by changing the lattice structure. Ansari et al. doped cobalt into the nano ZnO lattice to partially replace zinc ions, reducing the effective band gap to better fit UV adsorption, and the ultraviolet absorption rate was positively correlated with the cobalt concentration [12]. Torbati and Javanbakht produced a multi-component nanoparticle of nano ZnO, antibacterial Ag+ and stable phase Zn2TiO4, whose crystal structure and band gap are close to those of pure TiO2 nanoparticles, but the SPF value is improved [13]. It should be noticed that this study emphasized the photocatalytic activity of the composite nanoparticles, which is not conducive to improving sunscreen products.

In addition to TiO2 and ZnO, CeO2 has been discovered as an active ingredient in sunscreen in recent years. Similarly, CeO2 enhances its absorption of mid-band ultraviolet light as its particle size decreases. Li et al. doped CaO into CeO2 to form oxygen defects and reduce the particle size, thereby improving CeO2's UV absorption efficiency and covering the entire ultraviolet band [14]. Due to carrier limitations, the absorption band of CeO2 doped with NiO is blue-shifted from 351 nm of pure CeO2 to 246 nm and 276 nm, and the band gap energy of NiO/CeO2 is closer to 3.1 eV, which enhances ultraviolet absorption [15].

Unlike inorganic metal doping, organic modification achieves the self-doping of inorganic nanoparticles by redox reactions. Lignin is a common plant aromatic polymer with functional groups absorbing UV rays. Lignin sulfonate is a good dispersant in cosmetics industry and has low biological toxicity [16]. Yu et al. used lignin sulfonate to hydrothermally esterify the surface hydroxyl groups of nano TiO2, forming Ti4+/Ti3+ self-doped and oxidized semiquinone structures to broad the absorption spectrum and strengthen the UV absorption (Fig. 3). Also, lignin sulfonate increased the surface roughness of nanoparticles to enhance UV scattering. The electrostatic repulsion of the sulfonic acid group helped TiO2 particles disperse. The above multiple effects improved the sunscreen performance of TiO2 by 30-60% synergistically [17].

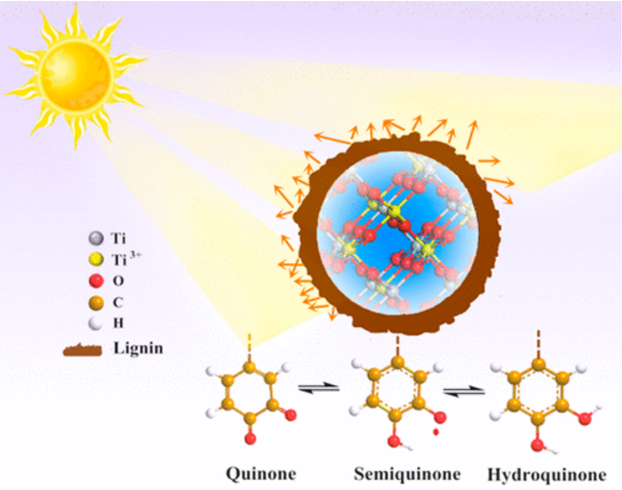

Fig. 3 Organically modified inorganic nanoparticles Lignosulfonate@Nano-TiO2 redox self-doping to enhance sunscreen performance [17]

4. Biocompatibility Improvement of Inorganic Nano Sunscreens

Nanotechnology has made much progress on inorganic sunscreens, but new research shows that there are some potential biosafety problems in inorganic nano sunscreens. The nano-scale particle size of these inorganic nano sunscreens may allow the particles to penetrate stratum corneum, then inducing cytotoxicity and other damages. In addition, inorganic nano sunscreens exhibit certain photocatalytic activity under UV exposure, possibly producing excessive amount of ROS, which leads to cell damage [7]. It is vital to solve them.

4.1 Size and Structure Design to reduce the skin penetration

Some studies have shown that nano ZnO has certain toxicity caused by size and solubility, and although the toxicity of nano TiO2 is much lower than that of other metal oxides, there are still related works showing its biological toxicity [18]. In order to solve the issue, a combination of micron & nano (MicNo) is adopted to retain the advantages of both micron particles and nanoparticles. Entekno Materials has developed a MicNo-ZnO sunscreen, which retains the transparency and high UV absorption efficiency of nano ZnO. Genç et al. tested the MicNo-ZnO toxicity in HaCaT cells, finding that its cytotoxicity and genotoxicity were much lower than those of nano ZnO, which reflected the properties of micron ZnO [19] (Fig. 4).

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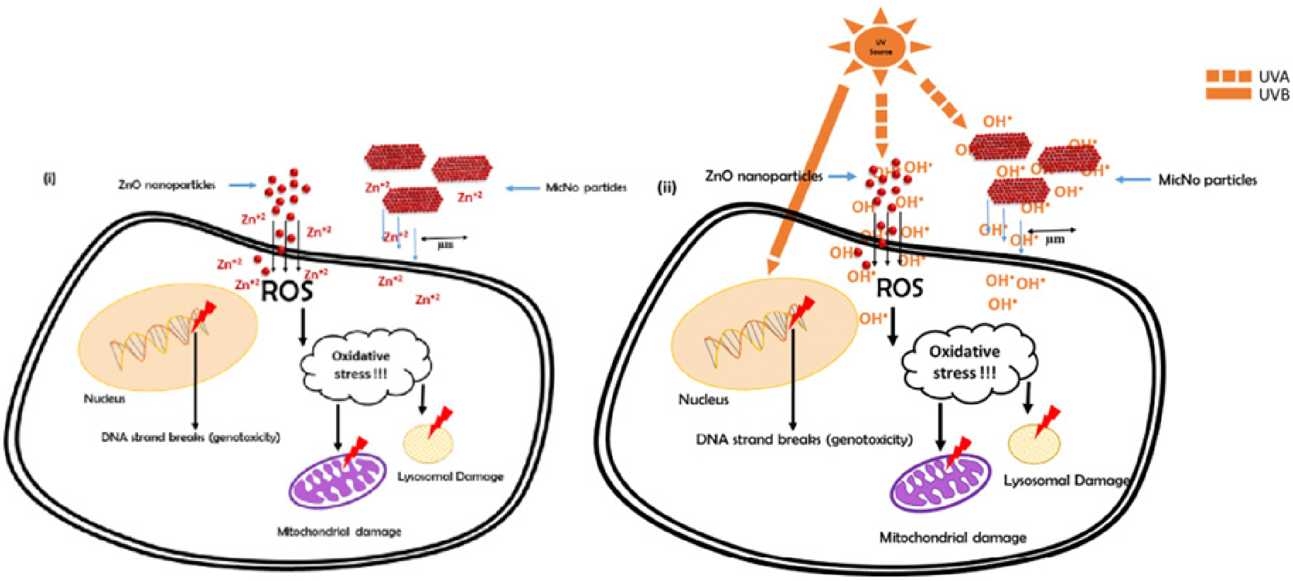

Fig. 4 Mechanism of improving biological safety of micro & nano patterned inorganic sunscreen [19].

4.2 Synergism/Antagonism of Metal Oxides to reduce toxicity

For inorganic nano sunscreens, doping or mixing of metal oxides not only influence their UV absorption and scattering, but also influence their biotoxicity by various mechanisms. As reported, nano ZnO and nano TiO2 both have possible biotoxicity, and there may be antagonism and synergism when they are mixed in certain conditions, thereby reducing the mixture's toxicity. Liang et al. found that the combination of ZnO and TiO2 reduced ZnO skin toxicity in a human skin model for TiO2 promoted ZnO aggregation and suppressed Zn2+ dissociation [20]. Ghiazza et al. introduced iron ions onto the TiO2 surface to replace Ti4+ in the lattice, which both reduced the photocatalytic activity and inhibited cytotoxicity and genotoxicity of TiO2 significantly [21].

4.3 Reduction of the Photocatalytic Activity

Inorganic nanoparticle TiO2 has photocatalytic activity and will produce a large amount of reactive oxygen species (ROS) under UV lights. As signal molecules, ROS plays an important role in organisms, such as cell proliferation and repair, but excessive ROS will lead to oxidative stress, causing cell dysfunction, protein damage, DNA damage, and diseases eventually [7]. There are several ways to reduce photocatalytic damage: reducing the generation of ROS by inhibiting the photocatalytic activity of nanoparticles or reducing the contact between nanoparticles and photons, or eliminating the generated ROS by introducing substances that quench free radicals.

Some organic polymers not only have the ability to enhance the ultraviolet absorption efficiency of inorganic nano sunscreens, but also can reduce the harm of ROS generated by ultraviolet irradiation to the human body through different mechanisms. Polydopamine (PDA), a biocompatible synthetic polymer, has strong capabilities of adhesion and ROS scavenging, while phycocyanin (PC) has antioxidant, anti-inflammatory, and anti-tumor properties. Zheng et al. prepared PC-PDA-@Nano-TiO2 via spontaneous oxidative polymerization, and formed a dense PC-PDA shell on nano TiO2 surface [22]. The synergistic effect of PC and PDA reduced the photon absorption of TiO2 and hindered the carrier migration in TiO2 as well as scavenged free radicals, which reduced ROS concentration in several ways. (Fig. 5)

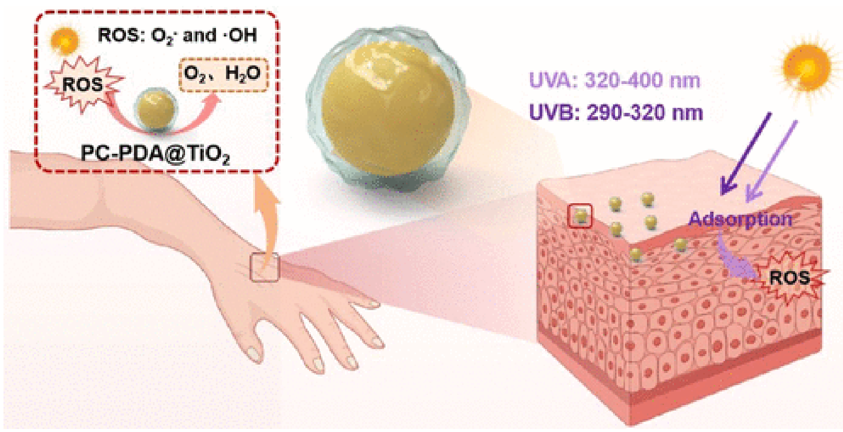

Fig. 5 The structure of Phycocyanin-& Polydopamine-@NanoTiO2 and its mechanism of eliminating ROS [22].

Li et al. used a type of lignin derivative, quaternary ammonium alkali lignin (QAL), to coat nano TiO2 via self-assembly, and the two were bound together by intermolecular hydrogen bonds [23]. QAL can quickly capture free radicals generated by TiO2, thereby reducing the harm of photocatalysis to the skin. In addition, the hydrophobicity from the outside of the lignin micelles can also improve the compatibility of TiO2 with hydrophobic creams. (Fig. 6) The CNF in nano-TiO2@CNF could also scavenge hydroxyl radicals, and compared with the traditional inorganic metal oxide coating method to reduce photocatalysis, it significantly reduced the photocatalytic activity of TiO2 while maintaining a high UV absorption efficiency [10].

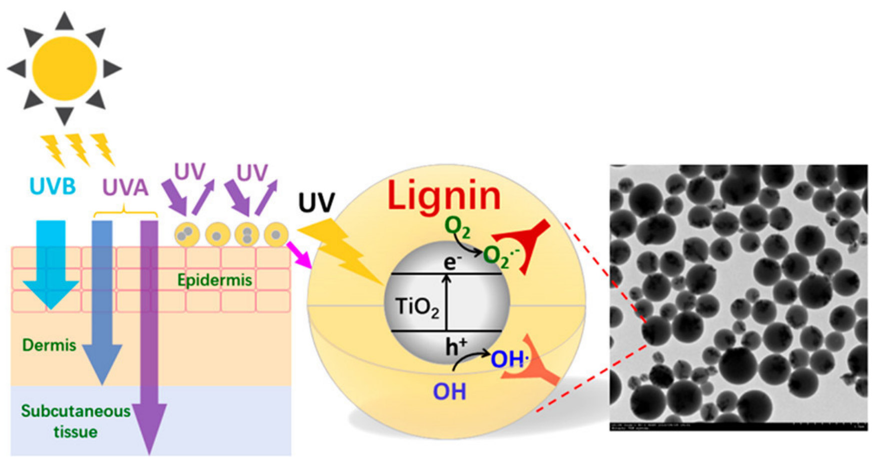

Fig. 6 Mechanism of reducing ROS generation and eliminating ROS by Lignin Colloidal Sphere@Nano-TiO2 [23].

The photocatalytic activity of ZnO is relatively smaller than that of TiO2, but it is still not negligible. Wei et al. prepared ZnO/de-alkali lignin (DL) composite nanosheets. The specific surface area of DL flakes enhanced its ability to eliminate free radicals [24]. However, whether the sheet-like ZnO/DL can be used in sunscreen cosmetics needs further testing.

Unlike TiO2 and ZnO that cannot remove ROS by themselves, nano CeO2 can achieve the oxidation state cycle of Ce3+/Ce4+ through simple surface modification, such as PEGylation, to simulate superoxidase and peroxidase, and then achieve the ability to remove ROS, which makes it a promising material in the nano sunscreen field [25].

5. Beyond Effectiveness and Biocompatibility

Except for the basic demands for UV protection and biocompatibility, consumers consider many other factors as well when choosing sunscreen products, such as cosmetic elegance and multifunction [3]. Several inorganic and organic modifications are capable to develop these aspects, which sometimes attract more attention from consumers.

5.1 Aesthetics of Inorganic Nano Sunscreen

TiO2 and ZnO powders both appear white under daily light, and it has been well solved by nanotechnology in

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commercial sunscreens. Even so, it is difficult to apply them to dark skin for aesthetic reasons. Dark skin is also more sensitive to high-energy visible light (blue-violet light) and needs broad-spectrum sunscreen. Therefore, dark pigments are added to the formula or nanoparticles are modified and compounded with color change as the orientation to meet the needs of dark skin.

Iron oxide will appear black, red, yellow and many other colors according to different oxidation states. Mixing iron oxide with TiO2 or ZnO in different proportions can prepare sunscreens of various chromaticities [26]. And iron oxide enhances the absorption of visible blue-violet light, improving the broad-spectrum of sunscreen, and is widely used in dark sunscreens, such as the Invisible Tinted Mineral Facial Sunscreen (Buttah Skin). M oreover, some organic polymers that can be used in sunscreen products are dark themselves, and can also be used to prepare sunscreens of different colors with multiple efficacies. For example, melanin-like polydopamine (PDA) is bonded to SiO2 to form black nanoparticles, which can be mixed with white TiO2 in different proportions to produce sunscreen lotions of different shades [27] (Fig. 7). The two nanoparticles together absorb UV and remove ROS efficiently.

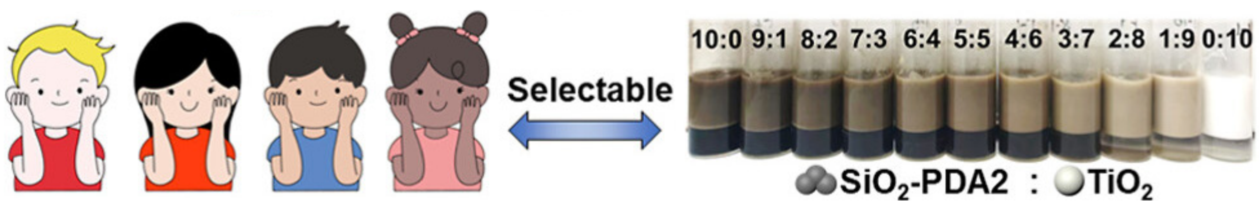

Fig.7 Gradient adjustment of colored organic polymer melanin-like polydopamine to improve the appearance color of inorganic nano sunscreen [27]

5.2 Multifunctional Nano Sunscreens

With the development of society and technology, consumers are no longer satisfied with the single function of sunscreen, but tend to prefer "n-in-one" sunscreen that meet multiple functions, such as antibacterial function, moisturizing, anti-aging, and freckle removal.

It can be seen that the modification of inorganic sunscreen nanoparticles will synergistically enhance other functions while improving UV resistance, stability, and biosafety. Hasnu et al. prepared CeO2-NiO composite nanomaterials from tea processing waste using a green method [15]. While having excellent anti-UV-B radiation efficacy, it also has significant antioxidant and antibacterial effects. However, since the antibacterial mechanism of this material involves the induction of ROS, the biological toxicity test is very important, therefore further research is required if it is used in sunscreen cosmetics. Badalkhani et al. added nano-TiO2 and nano-methylene bis-benzotriazolyl tetramethylbutylphenol (MBBT) to a nano-lipid carrier (NLC) containing the antioxidant γ-oryzanol to achieve a composite effect, that is, SPF=34, and have good biocompatibility, while achieving anti-aging by delaying photoaging [28].

6. Conclusion

This article systematically reviews the research on inorganic nano sunscreens in recent years, including the improvement of UV protection ability, the reduction of biological toxicity and the functional development. The UV protection performance of inorganic nano sunscreens is mainly improved in terms of broad spectrum and high efficiency, and particle dispersion and lattice structure are the keys to achieve the goals. For improving dispersibility, both inorganic and organic have commonly used carriers in medicine and cosmetics, such as kaolinite, microcrystalline cellulose, cellulose nanofibers, etc. Inorganic-inorganic composites mainly change the lattice structure and the semiconductor band gap energy by doping with other metal elements, such as cobalt, silver and iron, to broaden the absorption spectrum and improve the efficiency. Inorganic-organic composites can achieve broad spectrum through self-doping by redox, such as lignin sulfonate reducing partial Ti4+.

The biosafety of inorganic nano sunscreens is also a highly focused issue. Nanoparticles have a special size for a risk of entering cells and causing adverse reactions. The risk can be minimized through micron-nano combination. Inorganic sunscreen nanoparticles have the photocatalytic capability to generate excessive reactive oxygen which is harmful to health. Some natural organic polymers can capture the generated reactive oxygen to help increase the biocompatibility of the sunscreen materials, such as lignin, cellulose and their derivatives.

With the continuous development of sunscreen nanotechnology, consumer demand for sunscreen is not only for basic sunscreen function, but also for aesthetics and versatility, which have gradually become key factors for consumers to consider. By adding iron oxide or some dark organic polymers to the basic formula, the color of sunscreen can be adjusted to non-fake white status, meeting the needs for different skin colors. According to a certain optimization ratio, the integration of sunscreen, moisturizing, whitening, antioxidant, antibacterial and other cosmetic formulas can achieve multifunctionality.

This article provides research ideas for performance improvement in the future field of inorganic nano sunscreen cosmetics from the perspective of effective ingredient compounding, and connects with the future trend of personalized precision medicine.

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