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Cellulose-titanium Dioxide Nanocomposite in Protective Coating of Paper Artwork

Gang Lin*

Jiaxing University, Jiaxing, Zhejiang, 314001, China

**corresponding author*

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Abstract: With the improvement in the quality of living standards, people are gradually pursuing art. As an essential material for artwork, the quality of paper determines whether the artwork can be preserved intact. This paper focuses on the preparation of cellulose-titanium dioxide nanocomposites and investigates the changes in the ageing resistance and water resistance of artworks after the application of the paper protection layer. The paper was analysed by SEM to determine the changes in fibre morphology with and without nanocomposites. The paper coated with nanocomposites was found to have very stable strength properties. When observing fading, we found that within one hour, the titanium dioxide coated paper faded about one time faster than the paper without any material. When looking at the change in tensile strength of the different papers after accelerated ageing, it was clear that over a period of 100 years, the tensile strength of the paper coated with nanocomposites is reduced by less than 2N/cm^2, while the tensile strength of the paper without the material is reduced by 10.7N/cm^2.

1. Introduction

Nano-titanium dioxide is a non-dissolution type and is an excellent inorganic photocatalytic antibacterial material. It has the advantages of odorless, non-irritating, good thermal stability, and high heat resistance. However, the fly in the ointment is that the environmental conditions for the action of nano-dioxide are more severe, and the sterilization effect can only be produced under the conditions of exposure to ultraviolet light. Organic/inorganic nanocomposite materials are new building materials produced by the development of nanotechnology. In recent years, they have become one of the hotspots of materials engineering science research. Combining with organic and inorganic materials can have more outstanding characteristics.

For cellulose-titanium dioxide nanocomposites, there have been many related researches abroad. Kumar t tested the applicability of the membrane in the filtration of 1, 4-dioxane by Brinell based on Fourier transform infrared spectroscopy, antibacterial property and intermolecular interaction of

PVDF / Ca electrospun membrane. After adding TiO_2, the filtration efficiency of the membrane was improved by 55% [1]. Haroun A described the preparation, characterization and dielectric evaluation of cellulose/titania nanoparticles. These nanocomposites are successfully prepared by emulsion technology in the presence of two different crosslinking agents such as epichlorohydrin and glutaraldehyde. Infrared spectroscopy, thermogravimetric analysis, and the dielectric measurement was studied. The results showed that TiO_2 was physically adsorbed and distributed among the cellulose fibers. Due to the polarization effect, the dielectric parameter increases in the case of epichlorohydrin, while the dielectric parameter is lower in the case of glutaraldehyde. The presence of TiO_2 enhances the AC conductivity [2]. Jadhav s found that pdnp has good dispersion on TiO_2 cell carrier, which enables the synthesis of biphenyl, acrylate, acetylene and prochiral ketone with low Pd loading (1mol%), which has excellent catalytic activity at relatively low temperature. It is convenient to further study the properties and dosage of base and the properties of solvent. [3]. Shakeel I prepared Ca / polyethylene glycol based mixed matrix membrane by solution casting method to better study the effect of iron nickel and titanium dioxide nanoparticles on methane permeation characteristics. [4]. Campbell s confirmed and characterized the product by Fourier transform infrared spectroscopy and thermogravimetric analysis, which proved the carbon dioxide adsorption capacity. The carbon dioxide adsorption and recovery experiment proved the high stability and recycling capacity of cellulose titanium dioxide in the carbon dioxide capture process [5]. Khalid a used agar disk diffusion method to test the antibacterial activity of nanocomposites against E. coli, and evaluated the wound healing effect of burn models in vivo by measuring wound area, contraction rate and histopathology, in order to study the potential of bacterial cellulose for wound healing. It was found that the inhibition rate of nanocomposites against E. coli and Staphylococcus aureus has been improved a lot [6]. Li Y prepared copper doped cellulose catalyst by hydrolysis precipitation method. The prepared photocatalyst can effectively decolorize organic dyes, reduce agglomeration and increase specific surface area under the synergistic effect of simultaneous adsorption and photocatalysis [7]. However, these research fields are not suitable for the general public, and there are still some controversies in their experimental methods, so they are not well popularized and applied.

The cellulose-titanium dioxide nanocomposite material not only prevents ultraviolet radiation, but also has a very good effect in sterilization and anti-pollution. The material is also very environmentally friendly, and is used on a large scale as an indispensable white dye in housing construction. In addition, it is also used as a protective layer in the aviation field and has many advantages such as heat resistance and waterproofing. The cellulose-titanium dioxide nanocomposite material is of great help to the improvement of paper performance in this research, so it can be used as a protective layer of paper art to protect the art from being damaged by environmental factors.

2. Application Methods of Cellulose-titanium Dioxide Nano composites in Protective Coatings for Paper Artworks

The experiment studied the preparation of cellulose-titanium dioxide Nano composite. SEM analyzed the changes in fiber morphology of paper with or without Nano composite material. Then by observing the fading of the paper dyed with volatile dyes under different aging times, and finally did an accelerated aging experiment to detect the change in the tensile strength of the paper after accelerated aging. The specific experimental research framework is shown in Figure 1:

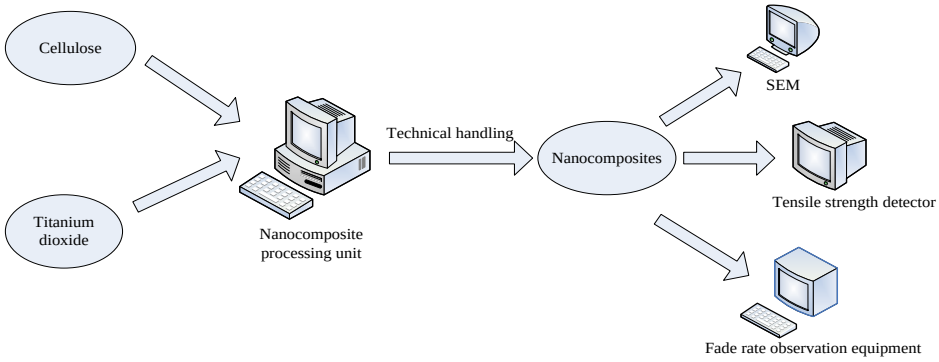


Figure 1. Framework diagram of the experimental study

2.1. Cellulose

Cellulose is a green, renewable, healthy and safe resource, which is widely present in almost all biomass materials in nature. Cellulose is the most widely distributed and biodegradable natural polymer material in nature. It is widely found in the roots, stems, leaves of trees, cotton, hemp and other plants, as well as some algae and bacteria. Most of the cellulose has not been developed and utilized by humans. The chemical structure of cellulose is $(C6H10O5)_n$, where n is the degree of polymerization [8]. In cellulose, the contents of carbon, oxygen, and oxygen are 44.44%, 6.17%, and 49.39%, respectively. The molecular structure of cellulose is shown in Figure 2. There is also a large amount of cellulose in most crop wastes. Every year, a large amount of crop stalks are burned directly in our country, which not only pollutes the natural environment, but also consumes resources. The use of cellulose for crop stalks creates an effective way for waste recycling and efficient use of land resources. Cellulose material is a glucan produced by connecting β-D-pyrrole and glucose via β-1,4-glycosidic bond. It is a kind of 0.5 rigid linear macromolecular material. There are a large number of hydroxyl groups in the molecular structure, and the hydrogen bond interactions generated within and between the molecular chains cause the molecular chains to cross polymerize and produce a large number of crystalline regions; this crystalline region can produce extremely high hardness. Due to the excellent thermodynamic properties of cellulosic materials, a series of advantages such as low acquisition cost and considerable resource reserves, it has been widely used in the fields of food, material processing and medicine [9].

At room temperature, dietary cellulose is not soluble in water nor in common organic solvents, such as ethanol, toluene, acetone, toluene, etc., but not in dilute alkaline solution, and soluble in copper ammonia solution and copper ethylenediamine aqueous solution. Therefore, even at room temperature, it is relatively stable, mainly because the cellulose molecule has internal hydrogen bonding [10]. Cellulose is also poorly pliable and is low in rigidity.

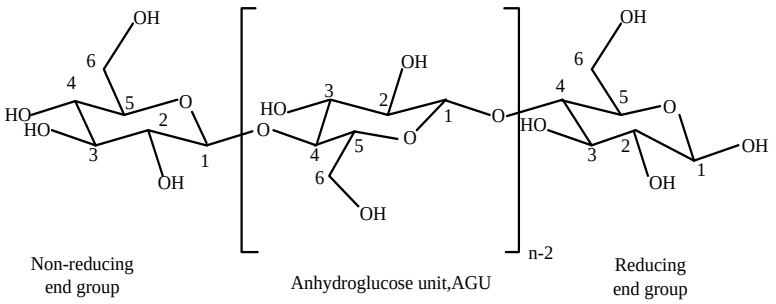


Figure 2. Chemical structure of cellulose

2.2. Nano Titanium Dioxide Material

Dioxide is a very critical type of metal oxide nanomaterial, and it has great research and application value in photocatalysis, solar cells, biological antifungal and lithium-ion power batteries. Using biological template technology, the natural fiber-oriented dioxide nanomaterial can not only accurately replicate the special structure of cellulose, but also can bring the inherent excellent characteristics of cellulose materials, including the huge specific surface area brought into the dioxide material.

2.2.1. Characteristics of Nanomaterials

The application of nanomaterials has many characteristics that are different from ordinary metal materials: surface effects, which are mainly due to the finer structure of the atomic particles that make up the nanomaterials and the large atomic surface area. By observing the surface structure, we can know that among the atoms distributed on the surface of the crystalline material, the number of atoms occupied is much smaller than the fraction of atoms randomly arranged on the surface. Due to these special properties, nanomaterials have produced super-high surface active compounds, microwave absorption properties, high oxidizability and superparamagnetism. At the same time, significant blue shift or red shift can be observed in the absorption spectrum of which is the main research object. In addition, nanomaterials possess some excellent properties such as chemical reaction properties, optical properties, chemical reaction kinetics, catalytic properties, photocatalytic properties, electro-photochemical properties, and specific physicochemical and mechanical properties.

The surface effect of nanomaterials is reflected in: when its particle size changes, the ratio of the number of atoms on the surface of the particle rises sharply, which will cause changes in the material properties. The reduction of particles will also lead to the increase of the surface properties and surface area of the particles, which will have higher chemical activity.

2.2.2. Titanium Dioxide

Titanium dioxide has three crystal structures: Rutile, Anatase and Brookite [11]. The formula for calculating the content of anatase and rutile phases in titanium dioxide is shown in formula 1:

$$\mathrm{R}_{\%} = 0.679\frac{P_r}{p_r + p_a} + 0.312\left(\frac{P_r}{p_r + p_a}\right)^2 \tag{1}$$

In formula 1, $R_{\%}$ is the percentage content of rutile phase in titanium dioxide, P_r and P_a are the diffraction peaks of 101 crystal plane and 110 crystal plane in rutile phase and anatase phase, respectively.

The physical parameters of the three types of titanium dioxide crystals are shown in Table 1.

The concentration of the titanium solution and the temperature of the acidity affect the reaction path of the hydrolysis the lower the concentration, the lower the acidity, and the higher the temperature, the easier the reaction. In order to increase the temperature, the reaction is often carried out under pressure, and the reduced particles can also be combined to make the product function better [12].

Under the condition of light irradiation, the surface hydrophilicity of titanium dioxide is caused by the change of its surface structure. Under ultraviolet light, the electrons in the titanium dioxide valence group vibrate in the carrier group. The electrons react with titanium, and the holes react

with the surface by combining oxygen ions to form fine trivalent titanium ions with vacant oxygen sites. During this period, the hydrogen in the gas is absorbed in stages and converted into chemical synthesis [13].

Table 1. Physical parameters of three types of titanium dioxide crystals

Key length and space group	Crystal Type		
	Plate titanium ore	Anatase	Rutile
Crystals	Oblique Square	Quadrilateral	Quadrilateral
Ti-O_1/nm	0.187	0.194	0.198
Ti-O_2/nm	0.204	0.194	0.198
Ti-O_3/nm	0.199	0.193	0.194
Ti-O_4/nm	0.194	0.193	0.194
Ti-O_5/nm	0.192	0.193	0.194
Ti-O_6/nm	0.200	0.193	0.194
Average bond length/nm	0.196	0.194	0.195
Space group	Pbca	C4/am c	$P4_2$/mmm

The formula for calculating the threshold of the wavelength of absorbed light is shown in Equation 2:

$$\lambda = 1240 / E_r \tag{2}$$

In Equation 2, E_r is the band gap value, which is the difference between the highest and lowest energy values in the conduction band.

Titanium dioxide's high resistance to ultraviolet radiation is due to its high reflectivity and high scattering rate. Its anti-ultraviolet intensity and its meaning are related to the particle size: the larger the particle size, the more ultraviolet rays are blocked and dispersed on the titanium dioxide, which is effective for sound waves and large waves [14]. As the particle size decreases, light can pass through the surface of the particle, and it can be observed through the invisible ultraviolet light that it invades the light wave area [15]. The sun protection method to improve it is to remove ultraviolet rays, usually to remove ultraviolet rays in the mid-wave space. It can be found that solar titanium dioxide has different meanings for different ultraviolet wavelengths [16].

It is possible to absorb ultraviolet light in titanium dioxide: the electrical production of nano-titanium dioxide consists of a set of carrier bands formed by zero-orbital valence electrons. When irradiated with ultraviolet light, its intensity is much higher than its aperture (about 3.2eV). As the light propagates, the electrons in the valence group explode in the carrier group. As a result, there are no electrons in the valence band, which creates a hole, making the electron-electron pair easy to move and powerful [17]. On the one hand, these electric-electron pairs can be combined in

various redox reactions to release energy in the form of heat and produce fluorescence. Or on the other hand, they can be separated in the crystal lattice, and electrons at other reaction sites are retained by surface groups. Under normal conditions, titanium dioxide activates surface water to form surface hydroxyl groups to trap free holes and form hydroxyl compounds. Free electrons quickly combine with the oxygen involved to form peroxide compounds, which will also kill surrounding bacteria and viruses. Visible, ultraviolet radiation, titanium dioxide will generate free radicals after the introduction of ultraviolet rays, accelerate skin aging and skin damage. Therefore, when using titanium dioxide as a sunscreen, these three basic methods should be reduced or eliminated to reduce or eliminate its light activity and minimize its harmful effects.

The lattice size can be calculated from the Xie Le formula, as shown in formula 3:

$$s = K\beta / (Nc\cos\gamma) \tag{3}$$

In Equation 3, s represents the lattice size, the K coefficient is 0.89, β is the wavelength of the ray, N is the full width at half maximum, and γ is the diffraction angle.

The calculation formula for calculating the forbidden bandwidth value:

$$W_{G\infty} = (1 - G_{\infty})^2 / 2G_{\infty} \tag{4}$$

In formula 4, $W_{G\infty}$ is the forbidden band value, and G_{∞} is the relative diffuse reflectance (that is, diffuse reflectance):

$$G_{\infty} = G'_{\infty} / G'_{\infty 0} \tag{5}$$

In formula 5, G'_{∞} is the absolute diffuse reflectance of the sample, and $G'_{\infty 0}$ is the diffuse reflectance of the reference sample.

Light absorption is based on Lambert Beer's law. The mathematical expression of Lambert Beer's law is shown in Equation 6:

$$W = \lg(1/Q) = Krc \tag{6}$$

Where W is the absorbance, Q is the transmittance (transmittance), which is the intensity of the emitted light than the intensity of the incident light.

2.3. Preparation and Modification Methods of Cellulose-titanium Dioxide Nanocomposite

Natural cellulose is a three-dimensional network hierarchical structure composed of many fine nanofibers and has a high specific surface area. A series of functional composite materials based on natural cellulose can be obtained by introducing guest materials into cellulosic materials, followed by different post-treatments. We deposited a titanium gel film on the surface of cellulose nanofibers, and obtained a rutile titanium dioxide/cellulose composite material. We use a combination of solvothermal and hydrothermal methods to use tetrabutyl titanate as the precursor. Ultrafine anatase titanium dioxide nanoparticles were grown uniformly on the surface of cellulose nanofibers, and anatase titanium dioxide/cellulose composite materials were also obtained.

There are certain differences in the chemical and physical properties of cellulose-titania nanocomposites prepared by different preparation methods. Common preparation methods are: hydrothermal method, sol-gel method, solvothermal method, etc. [18].

The hydrothermal method is to add the raw materials to water, seal it in a closed container, and then place it in a high temperature environment. The expansion and gasification of the water inside the container form a high temperature and high pressure reaction environment. The preparation

process of dissolving and recrystallizing materials with poor solubility at normal temperature and pressure in the supersaturated state of the reactor [19]. The hydrothermal method has the advantages of cheap raw materials, easy dispersion, complete crystal structure, high yield, diverse morphology, and can obtain metastable equivalents that are difficult to achieve by other preparation methods. However, it also has shortcomings such as long reaction period and lack of a complete theoretical system.

The sol-gel method is a liquid chemical wet method for preparing solid materials from small molecules [20]. It is a process suitable for reaction at relatively low temperature, with the advantages of very meticulous processing, low cost and simple equipment [21]. It can be used for film formation, or mold forming, and can also be used to prepare nano powders. The sol-gel method is a commonly used experimental method for preparing nanocomposites. In the specific preparation process, an alcohol solution is used as a co-solvent, an alkali solution or an acid solution is used as a catalyst, and the inorganic salt or metal alkoxide undergoes hydrolysis and condensation reaction to generate titanium dioxide gel [22]. The use of alcohol solution as a co-solvent in the reaction system can prevent the production of titanium dioxide from agglomerating, because the functional group -OH on the surface of the alcohol can condense with the -OH on the surface of the titanium dioxide particles generated in the reaction solution to remove water molecules. Therefore, the -OH on the surface of the titanium dioxide particles will not be able to undergo condensation and hydrolysis reaction with the -OH on the surface of other titanium dioxide particles [23].

Solvothermal method is a material preparation method developed based on hydrothermal method. Organic solvents or non-aqueous solvents are replaced by water in the hydrothermal process. Non-synthetic materials can be prepared in aqueous solutions. They are easily oxidized or sensitive to water, such as III-V semiconductor compounds, nitrides, chalcogenides, etc. [24].

The surface modification of nano-titanium dioxide was carried out by adding organic modifiers under the condition of adding hydrochloric acid, and finally the hydrophobic modified nano-titanium dioxide was obtained through subsequent processing work such as filtering, washing, drying and grinding [25]. In this experiment, the acidic conditions created by the acetic acid solution used to dissolve cellulose are directly used to simplify the specific process of organic surface modification of nano-titanium dioxide in an acid environment. The modification process as shown in the figure was designed, and the surface modification of nano-titanium dioxide was carried out with the organic surfactant sodium laurate [26].

Because nano-titanium dioxide is hydrophilic, it is difficult to disperse uniformly in organic substances such as chitosan. If it is not modified, after the prepared coating is allowed to stand for 24 hours, the nano-titanium dioxide will agglomerate and settle to the bottom of the coating. In this experiment, after the surface active agent sodium laurate was used to modify the nano-titanium dioxide, its dispersion stability in cellulose was significantly improved.

The main principle of surface modification of nano-dioxide is based on the presence of double light radicals on the surface of nano-dioxide. Adjacent light groups combine with each other through hydrogen bonding. The isolated hydrogen atoms have strong positive electrical properties, and they are easy to adsorb mutually between negatively charged molecules and undergo dehydration condensation reaction with substances containing light groups. It reacts with silane coupling agent and produces water esterification reaction with epoxy material [27].

3. Experiments on the Application and Research of Cellulose-titanium Dioxide Nanocomposites in the Protective Coating of Paper Art

3.1. Experimental Materials

Some materials and equipment will be used in the experiment. The specific materials and

specifications are shown in Table 2:

Table 2. Main experimental raw materials

Experimental materials	Manufacturers	Specification
Cellulose	Shandong Meikai Chemical Technology Co.	Deacetylation degree ≥ 90%, 50-800mPa-s
Nano titanium dioxide	Aladdin Chemical Reagent Co.	99.8%, anatase, 100nm
Hydrochloric acid	Zhejiang Sanying Chemical Reagent Co.	Analysis of pure
Distilled water	Laboratory homemade	Analysis of pure
Glycerol	Shanghai Zhan Yun Chemical Co.	Analysis of pure
YZ-139 Amphoteric starch	Hangzhou Paper Friends Technology Co.	Water content 6-9%, substitution degree 0.035%
SEM	Dongguan Jinghan Industrial Co.	1nm

3.2. Experimental Process

In this experiment, the content of nano-titanium dioxide was changed on the premise that the solid content of the cellulose-titanium dioxide nanocomposite was 8%. The main preparation route and process flow are shown in Figure 3. Measure 180 ml of 5% acetic acid solution in a three-necked flask, slowly add a certain quality of natural cellulose, stir for 20 minutes at a stirring speed of 500 rpm to fully dissolve it, and obtain an acetic acid suspension of the cellulose liquid. Then add a certain amount of glycerol (glycerin) as a plasticizer, and mechanically stir for 20 minutes to improve the flexibility of the cellulose molecular chain and increase its reactivity. Subsequently, after adding modified starch (MS) as an adhesive and stirring for 30 minutes, the suspension of modified nano-titanium dioxide was added to the reaction system. Finally, after the system is continuously stirred for one hour, the composite system is transferred to a special plastic cup, and a high-speed disperser is used for high-speed dispersion at a speed of 6000 rpm for half an hour. And during the reaction process, sodium hydroxide is used to adjust the pH value of the composite system to neutrality to prepare a cellulose-titanium dioxide nanocomposite material.

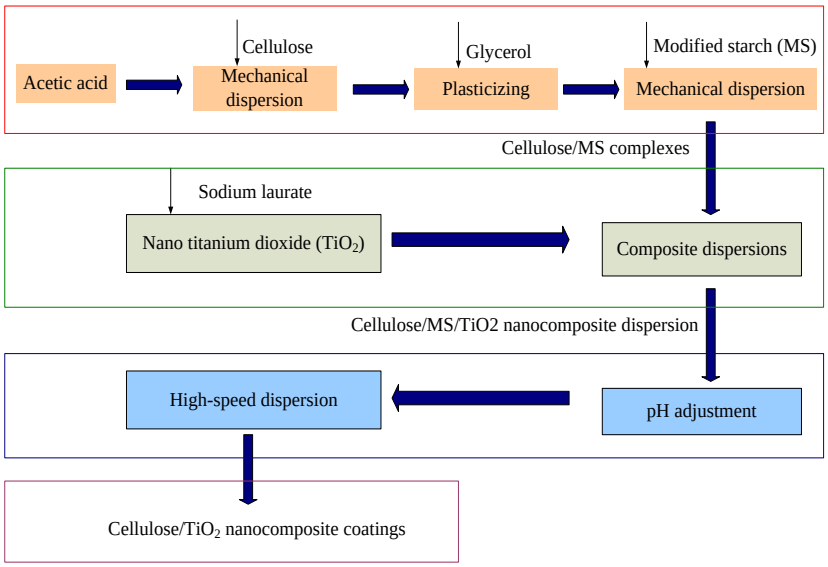
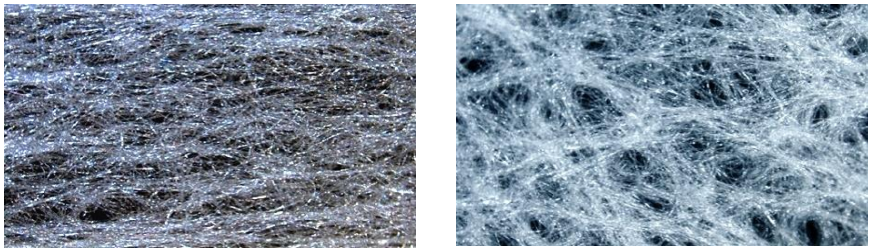


Figure 3. Flow chart for the preparation of cellulose/titanium dioxide nanocomposite coatings

3.3. Experimental Results

The scanning electron microscope is used to detect the two paper samples that are not coated with materials and those coated with nanocomposite materials to analyze the interaction of the coating components on the paper surface. The results of the detection are shown in Figure 4:



a、Paper sample of uncoated material　　b、Paper samples coated with nanocomposites

Figure 4. SEM analysis of different paper samples

Figure 4a is an SEM picture of an uncoated paper, which shows that the closely packed cellulose fibers are interwoven into a web without deformation. Figure 4b shows that the fiber morphology of the coated paper has not changed.

In order to test the degree of protection of nanocomposite materials for paper art, and to prove that the nanocomposite protective layer can have a certain immune effect against ultraviolet radiation, colored paper will be used as the target to detect the color fading of its color under ultraviolet radiation. Taking the pre-irradiation as a reference, comparing the three colored papers with uncoated materials, coated with nanocomposite materials, and coated with titanium dioxide, the fading rate of the paper under ultraviolet irradiation is measured at regular intervals [28]. The calculation formula 7 of the fading rate S(%) is as follows:

$$S = (A1 - A0) / A1 * 100\% \tag{7}$$

Among them, A1 is the absorbance of the paper before ultraviolet irradiation, and A0 is the

absorbance of the paper after absorbing ultraviolet rays.

We calculate the fading rate of the paper, and the specific fading situation is shown in Figure 5:

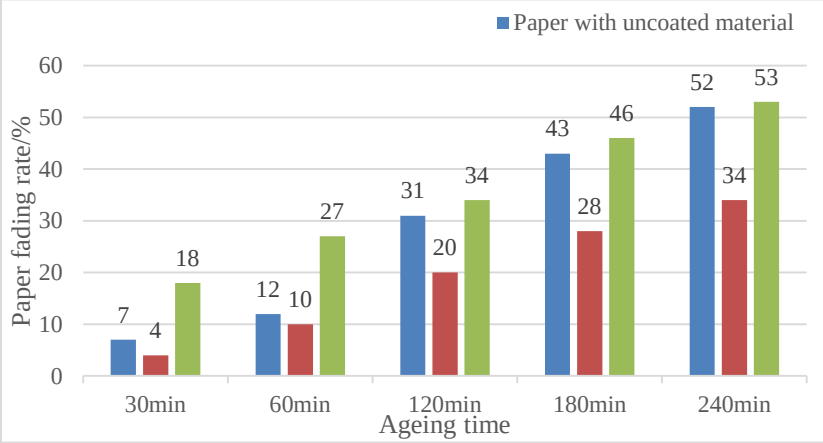


Figure 5. Fading of volatile organic pigments stained paper surface under different aging times

From the data in Figure 5, it can be reflected that the volatile dyes on the surface of colored paper are very easy to volatilize after exposure to ultraviolet rays. In addition, the color fading rate of uncoated colored paper is significantly higher than that of coated colored paper. The use of cellulose-titanium dioxide nanocomposite materials slows down the fading of the paper, which is mainly because the dyeing pigment will be incorporated into the cellulose and the titanium dioxide has the effect of absorbing ultraviolet rays.

The strength of the paper's aging resistance determines the degree of protection of paper art in the future. Therefore, experiments are needed to verify that nanocomposites have stable tensile strength for accelerated aging paper. Only in this way can nanocomposites be used as a protective layer for paper art [29]. The calculation formula of tensile strength (unit is N/mm2) is shown in formula 8:

$$\alpha = F_{max} / S_0 \tag{8}$$

In formula 8, F_{max} is the maximum force that the sample bears when it is broken, and S0 is the original cross-sectional area of the sample.

The experiment is to detect the tensile strength of the paper under different accelerated aging times for three papers processed in different ways. The specific experimental data has been presented in the form of a line graph, as shown in Figure 6 :

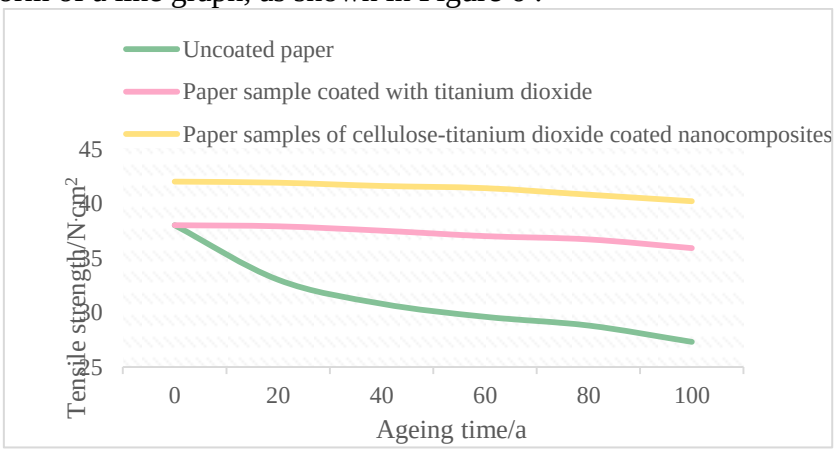


Figure 6. Changes in tensile strength of different papers after accelerated ageing

The fold-line graph of the tensile strength of the paper after artificially accelerated processing shows that the paper coated with the material still has a good tensile strength, while the tensile strength of the paper without the material is significantly weakened. The paper coated with cellulose-titanium dioxide nanocomposite still has a very stable tensile strength after 12 days of accelerated aging treatment (equivalent to one hundred years).

4. Discussion

This research is mainly to realize the preparation of cellulose-titanium dioxide nanocomposite material, and at the same time to verify the protective effect of this material on paper art. First of all, based on the background that paper art is not easy to preserve, improving the "shelf life" of paper art has become the primary goal. Because titanium dioxide is an indispensable material in modern art, titanium dioxide plays a huge role, such as making white pigments and acting as a blending agent to adjust hue and saturation.

A variety of chemical and physical methods are used to prepare the cellulose-titanium dioxide nanocomposite coating. The physical method includes hydrothermal method, and the chemical method includes sol-gel method and solvothermal method. The nanocomposite material prepared by combining multiple methods can not only be used in multiple forms, can be made into a thin film, but also can be made into a liquid coating, and it is also relatively complete in terms of functions. Such as antibacterial, anti-aging, reducing the damage of ultraviolet rays and polluted air [30].

Considering the importance of new materials for the long-term protection of artworks, accelerated aging experiments were carried out on paper. During the research, the degree of reduction in paper strength was analyzed according to the changes in the tensile strength of the paper caused by light and heat. In addition, volatile coatings are also used to study the fading of the surface of the dyed paper under different aging times.

5. Conclusion

In the process of paper artwork from production to preservation, there will be many uncontrollable factors that cause irreversible damage to the artwork. This research focuses on the preparation of cellulose-titanium dioxide nanocomposites, and nanocomposites have been proven to play a role in protecting paper art from external factors (such as ultraviolet rays, air, etc.). The cellulose-titanium dioxide nanocomposite is used as a protective layer on the surface of the paper in the papermaking process. The inner layer is cellulose (which can be used as a fixative), and the outer layer is titanium dioxide nanoparticles. Dioxide nanocomposite materials can protect paper from polluting gases, bacteria and molds. At the same time, experiments have also proved that paper coated with nanocomposites has excellent stability without affecting its tensile strength.

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Data Availability

Data sharing is not applicable to this article as no new data were created or analysed in this study.

Conflict of Interest

The author states that this article has no conflict of interest.

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