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Review Article

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Review of recent nano TiO_2 film coating methods, assessment techniques, and key problems for scaleup

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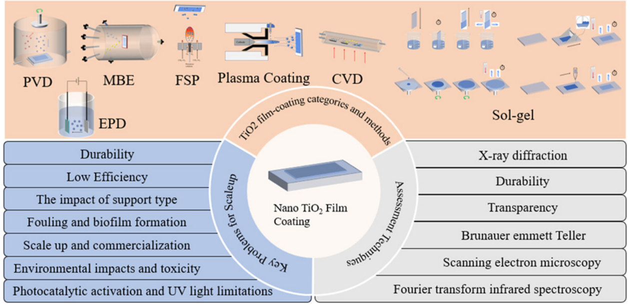
Abstract: The growing demand for green and sustainable development positions TiO_2 film coatings at the forefront of novel technologies. Applications of TiO_2-based materials in photocatalytic, optic, energy, electronic, aerospace, automotive, decorative, and other fields have accelerated on a large scale. Various coating processes are widely used in order to prepare nano TiO_2 filmcoated supports for different purposes. Each technique offers numerous advantages, such as the potential to be produced in large quantities and surface areas, acceptable adherence and reactivity of the resulting product, cost-effectiveness, simplicity, *etc.* This work provides a comprehensive overview of the most recent as well as commonly applied wet and dry TiO_2 deposition methods in terms of principals, advantages, applications, coating assessment techniques, required equipment, and key technical problems for mass production and commercialization such as coating durability, fouling, cost-effectiveness, environmental impacts, *etc.* This information provides a basis to better understand the new and conventional coating processes and the relative contribution rate of the influencing factors that are necessary to control the physicochemical properties of resulting coated substances for the applications aimed.



Graphical abstract

Keywords: nano TiO_2, film deposition, wet and dry methods, coating assessment, key problems

1 Introduction

Nanomaterials exhibit enhanced characteristics due to their high surface area-to-volume ratio and quantum effects. Generally, when materials are produced at the nano scale, the following happens at the surface: (a) A higher percentage of their atoms are exposed on the surface compared to those atoms inside the bulk material, (b) A higher surface energy (particles free energy) will be available due to bigger surface area, and (c) hence, these will enhance the reaction rates of the chemical components [1]. Moreover, what possibly can occur at the volume are: (a) A shorter wavelength which causes high frequency and energy, consequently, (b) atoms blue shift will take place in terms of their optical spectra absorption, (c) change in magnetic properties since materials in nanoscale can have minor scale than their magnetic domain (for ferromagnetic such as iron, cobalt, *etc.* [2], and (d) for the free electrons scheme, energy spacing raises averagely, followed by a reduction in the atom's quantity, which improves the catalytic characteristics of nanomaterials [1].

Therefore, the preparation of nanoparticles (NPs) has been among the hot-spot research areas owing to their novel properties, and investigation in this line of research

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is currently ongoing [3]. Nanomaterials have a wide range of applications, such as in medical science, electronic devices, industrial and agricultural sectors, and so on. Among which, applying advanced oxidation processes (AOPs) *via* nanomaterials (especially semiconductors) for environmental remediations has drawn much attention from the research works. Nano TiO_2 photocatalysis, as one of the most promising AOPs, uses UV energy (photoinduced processes) to enhance the pollutant degradation efficiency of water purification systems [1]. Studies on the photocatalysis ability of TiO_2 and its different applications have been ongoing over the past four decades, from hydrogen generation as fuel in 1972 by Fujishima for the first time [4], until recently, which has been studied for disinfection of SARS virus and COVID-19 during the pandemic [5]. Applications of nano TiO_2 in the removal of water and air pollution [6–11] have been exemplified elsewhere.

Titanium (Ti), with the atomic number of 23 is the nineth most abundant material (and seventh metal) on earth. It is available in nature as its oxidized form (TiO_2). Yearly around four million tons of TiO_2 are being produced as white pigment to be utilized in different industries. Its three known phases (or polymorphs) include anatase, brookite, and rutile which the latest is also the most stable phase. In their most thermodynamically stable forms, brookite occurs from 11 to 35 nm in size, while below this range of size would be anatase, and above this range is rutile. Figure 1 shows the titanium dioxide crystal structures in its three various forms, schematically [12].

For water treatment purposes, although nano TiO_2 has been extensively used as suspension form (in slurry-type reactors) with outstanding efficiency; however, it will need post-separation and secondary treatment, which is a costly process. Different attempts have been made for the easy removal of suspended form catalysts such as magnetic nano-structured photocatalyst [13] since TiO_2 is paramagnetic (unlike magnetic materials such as iron, nickel, cobalt, and steel). This technique is similar to polymer coating magnetic NPs, which are being used in medical science [14,15]. Immobilization of nano TiO_2 for photocatalytic contamination removal or self-cleaning applications [16] on various kinds of support such as zeolite [17], cement [6,8], bricks [12,18], silicalite-1 [19], borosilicate glass spheres [7], sintered glass [20], low iron float glass [21], glass wafers [22], ceramics [23,24], polypropylene nano composites [23,25,26], polymers [27–29], rubber [30,31], aluminum [32], *etc.*, can prevent such post separation requirements. In this way although the efficiency will decrease to half or lower [33] compared to suspended form, secondary treatment would not be needed.

The selection of the immobilization method and its success rate greatly depends on the type of substrate and the final product application. Besides, other parameters such as cost-effectiveness, scaleup feasibility, simplicity, technology, and required equipment can play important roles. Considering these criteria, a wide range of substrates are examined as support to be coated *via* different immobilization methods. In fact, an ideal support media for an efficient coating and performance must provide a good affinity and adhesion *via* high surface energy [34], durability, recyclability, thermal stability, and resistance to high temperature (for annealing and calcination purpose) [33]. It also must be transparent to ultraviolet (for efficient photocatalytic activity) while UV resistance, with largescale applicability, compatibility with the method used, resistant to degradation *via* TiO_2 oxidation ability, and able to offer sufficient surface area for the NPs and contaminants contact maximization, beside a base for well adherence and crack free layers of coatings [7,29].

Generally, the immobilization can take place in three ways including cementing, thermal bounding (hot press such as for polymeric supports [35,36]), or film coating [8,25,37]. Compared with coating and cementing techniques, in thermal bounding methods (such as melt mixing

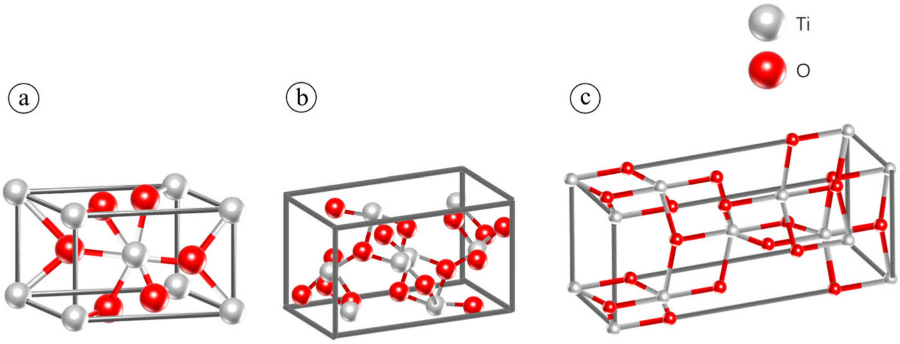

Figure 1: TiO_2 crystal structures in its three various forms: (a) rutile, (b) brookite, and (c) anatase.

via screw extruder [38], solution mixing [26,39], and *in situ* polymerization [40] less dissolution, erosion, and leaching of semiconductor will happen; however, other problems such as degradation of the substrate *via* embedded semiconductors might arise [33]. As for the cementing, besides the abovementioned problems, UV transparency, durability, and available surface area to volume can avoid largescale applicability of such methods. Film-coating however seems to be a more promising technique when it comes to water quality enhancement reactors.

Taking into consideration the variety of supports applied as substrates for different purposes, various nano TiO_2 coating methods are studied and developed. To achieve the right functionality and efficiency would require explicit materials and methods selection, tailored to specific protocols. Especially, the morphology and properties of the final NP coated product is inseparable from the coating method used. Hence, the key in preparing the most desirable coating lies in method selection. From practical point of view, the choice of coating method would be challenging in the absence of a comprehensive reference to compare different methods. Nevertheless, a relatively lesser number of research works have focused on the TiO_2 coating methods and mechanisms in detail. This review aims to introduce and compare different film-coating methods applied to fix TiO_2 NPs on a wide range of supports. Moreover, it illustrates their advantages and disadvantages, key problems, and assessment methods, technically. Using 214 references, this work adds value to the body of science in this field *via* offering helpful guidelines about the coating process and methods, that can be a practical source for scholars to provide the TiO_2 materials they aim for, with a desired coating technique.

2 Film-coating in brief

Nanomaterial film coating refers to the application of a thin film coating made of nanomaterials onto a surface (support). In other words, it is the immobilization of NPs as a thin layer on a surface to change, control, or enhance the physiochemical properties of the support or just for the purpose of the film application. The deposition of thin films on supports can be done through different methods to fix NPs onto the surface of materials at a thickness of 0.1–1,000 nm [41]. Nanomaterial film coatings have applications in various industries, such as electronics, environment, photocatalysis, aerospace, automotive, healthcare, energy, *etc.* They are employed to enhance the performance, durability, and functionality of surfaces, leading to improved product quality and enhanced capabilities based on the requirements. But if the application of the film itself is the main target, then the support will just provide a surface for the immobilization of the NPs. Nanomaterials used in film coating offer several advantages such as:

2.1 Enhanced functionality

Nanomaterials can provide improved functionality to the coated surface. For instance, they can enhance the surface's mechanical, electrical, thermal, or optical properties, depending on the specific characteristics of the nanomaterial used [42,43].

2.2 Barrier properties

Nanofilm coatings can create an exceptional barrier against moisture, gases, or other environmental factors by preventing the permeation of substances, thereby protecting the underlying surface [44–46].

2.3 Self-cleaning and antimicrobial properties

Certain nanomaterials such as TiO_2 possess self-cleaning properties, preventing the accumulation of dirt, dust, or other contaminants on the coated surface [47]. Additionally, they exhibit antimicrobial properties, inhibiting the growth of bacteria or other microorganisms on the surface.

2.4 Controlled release

Nanofilm coatings can be engineered to enable the controlled release of substances such as in agents or drug carriers where the coating can release substances at a controlled and sustained rate [48–50].

Based on the purpose of coating, the method can be selected depending on multiple criteria such as the available technology, cost, size, application, *etc.* In a more general perspective, there is a great deal of research on thin film preparation for a wide range of NPs *via* different techniques, depending on the coating purpose, the kind of nanomaterial used, the applied support surface, the required properties, and the available technology. These methods and materials can be such as vacuum evaporation technique used for vanadium pentoxide (V_2O_5) coating on glass substrate for film structural characteristics analysis [51], high target utilization sputtering, which is a plasma assisted method used to coat indium tin oxide (ITO) on glass and polyimide for solar cells with high transparency to visible light [52], plasma assisted molecular beam epitaxy (MBE) for ZnO film coating on sapphire substrate [53], chemical bath deposition, which is a wet method to grow ZnO film on glass substrates for gas sensors [54], electrolytic deposition of cerium oxide on stainless steel for corrosion avoidance [55], cadmium oxide thin film fabricated *via* Langmuir–Blodgett technique [56], liquid–liquid interfacial methods used to produce thin films of gold and other metals [57], liquid-phase deposition of TiO_2 NPs applied on steel in flow boiling equipment for a better heat transfer [58], and matrix assisted pals laser evaporation which is a laser deposition based method used for TiO_2 coating on gas sensors [59]. Also, some advanced techniques described in the literature are pulsed laser deposition (PLD), radio frequency (RF) magnetron sputtering, reactive ion beam assisted deposition, vacuum arc deposition [29], and much more. However, here in this review, we will only focus on the most frequently applied methods of TiO_2 film coating for different applications.

3 TiO_2 film-coating categories and methods

TiO_2 film coatings can be achieved through various techniques to ensure the uniform deposition of nanomaterials onto the surface, resulting in a thin layer with desirable properties and advantages. The coating on the support surface for TiO_2 (just to differentiate from the coatings on NPs), can be categorized into two main groups including dry particle coating [60] and wet chemical synthesis [19] with various methods within these two categories. Dry methods are such as physical vapor deposition (PVD), atmospheric plasma [34], chemical vapor deposition (CVD) [61], atomic layer deposition (ALD), photo-assisted deposition (PAD) [62], fluidized bed deposition, and MBE method, while wet methods are such as sol–gel [63], incipient wetness impregnation (IWI) method [19], and electrophoretic deposition (EPD). Another categorization method broadly divides the coating methods into physical, chemical, and sol–gel methods. Here the ten most frequently used techniques for TiO_2 coating are briefly explained and exemplified.

3.1 Dry TiO_2 coating methods

In this category, dry TiO_2 particles are vaporized, electrocuted, or sprayed to be deposited on the surface and no solution-based reactions are involved. Below are the top seven methods with several techniques within each, to coat titanium NPs dryly, on different support surfaces.

3.1.1 PVD

This method is a thin film deposition technique through the physical transfer of material atoms from a solid or liquid source to a substrate, without any chemical reactions involved [64,65]. As a vacuum-based method, PVD generally involves either one of the two primary processes: evaporation or sputtering. In evaporation, the material to be deposited (in this case, TiO_2) is heated to a high temperature, causing it to vaporize and then condense. In sputtering, energetic ions (such as Argon) as plasma are used for bombarding the titanium source to dislodge and eject Ti atoms from the target material surface [66,67]. The schematic diagram of this method is shown in Figure 2. PVD is carried out in a vacuum environment to eliminate interference from air molecules and reduce contamination. This is crucial for the control and quality of the thin film being deposited. The vaporized or sputtered material is then directed toward a substrate, where it condenses in the presence of oxygen or other oxidizing agents such as ozone, and forms a thin film.

Five different PVD techniques include thermal evaporation, electron beam evaporation, magnetron sputtering, laser ablation (PLD), and ion beam sputtering. In PLD, a laser is applied to ablate the Ti target, vaporizing it which later reacts with O_2 to form TiO_2 on the support surface, applied for dense and nanostructured film deposition [68–72]. Each method has

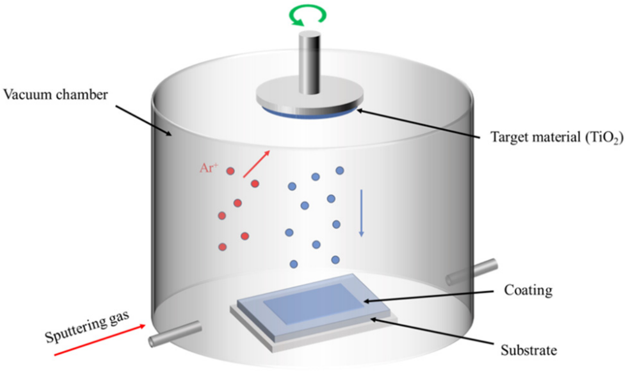

Figure 2: Schematic illustration of the PVD process.

its advantages and is chosen based on the specific requirements of the application. PVD is widely used in industries such as semiconductor devices, electronics and protective coatings, optics and antireflection, aerospace, and automotive for purposes like decorative coatings, and hard coatings. It is a versatile process that allows for the deposition of a wide range of materials onto diverse substrates, including flat surfaces (for evaporation) or complex geometrics (for sputtering). Although high cost, oxygen control, and slow deposition rate are reported to be the disadvantages [73–75], PVD is favored for its ability to produce thin films with excellent adhesion, uniformity, durability, and control over thickness. Coating properties such as its thickness can be controlled by deposition rate/time adjustment, substrate temperature control, oxygen pressure, and the amount of power supplied to the sputtering target or source of evaporation.

3.1.2 Plasma coating methods

Plasma is a highly ionized (using strong voltage) gas (normally air, argon, or oxygen), which contains free ions and electrons [76]. Plasma coating applies a plasma jet under the atmospheric pressure to deposit nano TiO_2 film on the support surface. The plasma is usually produced *via* an atmospheric pressure plasma jet or a dielectric barrier discharge. For TiO_2 coating *via* this method, after the Ti-based precursor vaporization, it will be injected into the plasma. Finally, the high energy of the plasma gas causes the precursor breakdown, resulting in Ti particle's oxidization, followed by TiO_2 film deposition on the support surface. The schematic diagram of the method is shown in Figure 3. Atmospheric plasma treatment prior to the coating procedure can enhance the NPs decoration on the substrate surface and increase the final product durability [77]. This technique is helpful for coating on supports with low surface tension such as polymers; however, the increased surface tension and plasma-assisted hydrophilic features can greatly reduce during the time [29]. Different techniques are practiced within the plasma coating method such as plasma spray coating and RF plasma coating.

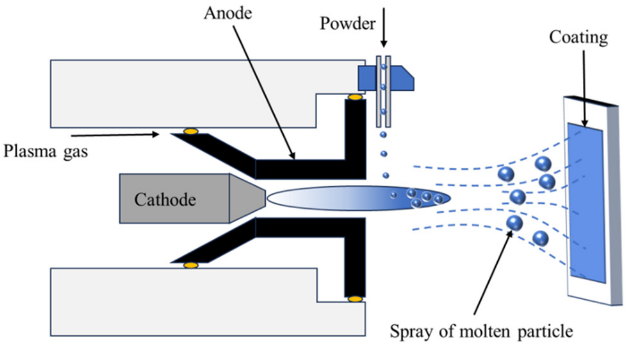

Figure 3: The working principal schematics of plasma spray coating method.

In the plasma spray coating method, a high-temperature plasma arc melts the TiO_2 powder injected into it. The melted nano TiO_2 particles will be driven by the plasma arc (sprayed) toward the support surface, where it condenses quickly to form a robust film [78,79]. This kind of coating is more suitable for wear resistance, corrosion protection, thermal stability, and barrier applications. The high deposition rate in this method produces high adhesion strength and hardness of the coated film. RF plasma coating method applies RF power to produce the required plasma field which causes precursor (mixed with oxygen) to be activated and react with oxygen to form and deposit thin and uniform TiO_2 film [80]. By regulating factors such as plasma power, precursor injection rate, support temperature, and the type of gas applied, the coating features can be adjusted. This method is known as a low-temperature method which offers thin and uniform coating, even on complex geometries, that makes it appropriate for thermo-sensitive supports; however, it is relatively complex, costly, and might require high maintenance. This technique is widely used in photocatalysis, antimicrobial surfaces, anti-corrosion, and optical coatings, or wherever precise control over thickness and uniformity is vital.

3.1.3 CVD methods

In the CVD method, a precursor gas containing Ti (such as $TiCl_4$, $Ti(OiPr)_4$) chemically reacts on the substrate surface in a heated vacuum chamber (from 200 to 700°C) to deposit (condenses) a thin film in the vapor phase. The process is illustrated graphically in Figure 4. Besides the thermal CVD, which uses the precursor in the presence of an oxygen source (such as O_2, or H_2O vapor) as a reactant to form the film, other six CVD techniques within this method are as follows.

Atmospheric pressure CVD (APCVD), in which the process is carried out at the atmospheric pressure, is a simple and cost-effective method while it might have limitations in terms of coating quality or uniformity. This technique is more applicable for industrial use or coating of large surfaces [47]. Low-pressure CVD (LPCVD), which can be performed under a reduced pressure, enhances the coating uniformity and quality. This method is more suitable for high-performance applications such as the manufacturing

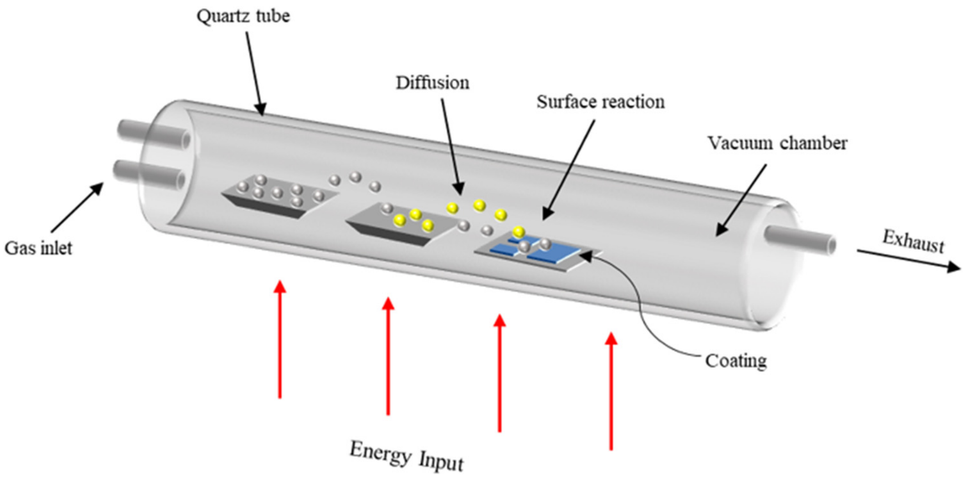

Figure 4: Schematic diagram of TiO_2 thin film preparation by CVD technology.

of semiconductors, coating for optical purposes, as well as for advanced photocatalysis applications [81,82]. Metal organic CVD (MOCVD) method as another type uses a metal-organic precursor (such as $Ti(OiPr)_4$) to form the film. This process has the advantage of having a precise control over the film deposition rates as well as its composition. The main applications of this method are for high-quality films or nanostructured coating [83].

Hybrid physical CVD (HPCVD) is another technique which combines and leverages principles of both PVD and CVD methods. In the PVD part of it, titanium is sputtered or vaporized from a solid Ti metal or its alloy and directed toward the support surface, where it can condense and deposit a thin film. At the same time (the CVD part), oxygen gas will be injected inside the deposition chamber. This causes the reactions between the O_2 and Ti atoms to form high-quality TiO_2 film *in situ*, on the support surface. This hybrid and low-temperature method offers precise control over the component ratio, morphology, thickness, and other properties of the film as compared to both PVD and CVD. The method is known as complex, slow, and costly, which makes it not suitable for large-scale applications, unless economically justified [35,36].

Plasma enhanced CVD (PECVD) or plasma assisted CVD (PACVD), on the other hand, as a low-temperature method, is applicable for thermal sensitive supports, which cannot withstand high temperatures [84]. The technique uses plasma (ionized argon gas) to enhance the chemical reactions (precursor breakdown) that produce the film. The vaporized precursor is ionized in the plasma field and forms TiO_2 film after reacting with oxygen. It can lower the reaction temperature while raising the deposition rates. It is mostly applied in the production of electronics, solar cells, photocatalysis, coatings for optical purposes, or wherever a coating at low temperature is required. If this process is carried out under low-pressure plasma conditions, the method is called low-pressure PECVD as a kind of PECVD. Laser chemical vapor deposition (LCVD) is a process that combines CVD principles with the laser ablation method [85,86]. It uses laser to break down the Ti precursor exposed to oxygen in order to directly deposit titanium dioxide on the support surface. This technique can be applied where small areas with precise coatings are required.

Although CVDs are known as low-temperature methods for precise control of film thickness, high purity, excellent adhesion, uniformity, scalability, and versatility, in general, they are costly with high technologies required, complexity, and can introduce toxicity [33].

3.1.4 ALD method

This technique allows for precise and controlled growth of thin films at the atomic level [87]. Although the method works based on the CVD principles, the key characteristic of ALD is its ability to deposit materials in a self-limiting chemical reaction [88], one atomic layer at a time, that is, in each deposition cycle, a pair of precursors react with the substrate in a sequential manner alternatively. This self-limiting behavior ensures that only one layer of material is deposited per cycle which typically is about 0.1–0.3 nm of TiO_2 film. After each precursor is introduced (Ti-containing precursor and then water vapor or O_2), the reaction byproducts (such as HCl) and excess precursor are purged from the reaction chamber to ensure that only the desired reaction products remain on the substrate surface. By repeating the deposition cycles (sequential exposure of the support to Ti precursor and O_2), precise control can be achieved at the atomic level. This technique generally operates in low to moderate temperature ranging from 100 to 300°C, which makes it more suitable for thermal-sensitive supports. The specific steps are shown in Figure 5.

Although ALD is known as slower process [88] (comparatively, as the final coating characteristics is time-sensitive to the steps of each cycle), which requires costly equipment (for maintaining precise control over temperature, pressure, chemicals flow rate, as well as purging cycles), with finite ultrathin coating thickness (suitable for small scale coating), it is extensively used in semiconductor manufacturing, photovoltaics, dielectrics, supercapacitors, batteries (to enhance their charge storage, lifetime, and stability), micro-electro-mechanical systems, surface protection (as corrosion/water-resistant coating), and other fields wherever accurate control over pure, thin, and uniform film properties is crucial, especially for complex surfaces such as three dimensional or porous supports.

3.1.5 Flame spray pyrolysis method (FSP)

For this technique (as a rapid thermal decomposition method), Ti precursor (as aerosol or vapor) will be introduced into a high-temperature flame (normally made by a fuel such as hydrogen) to form TiO_2 and deposited onto a support surface [89,90]. The schematic diagram of the method is shown in Figure 6. This technique is applicable for large-scale coatings with high deposition rates, to produce thin and uniform films [89,91,92]. Factors such as the flame temperature, concentration of the precursor, and the distance between the flame and the support surface can regulate the final product properties. Some advantages of films produced

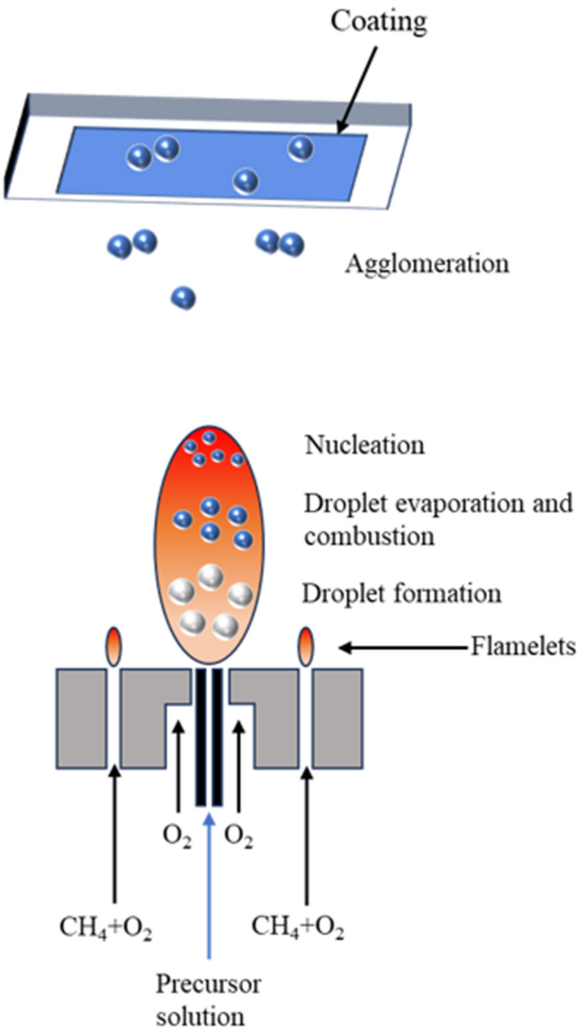

Figure 6: Graphical diagram of the preparation of TiO_2 films by FSP method.

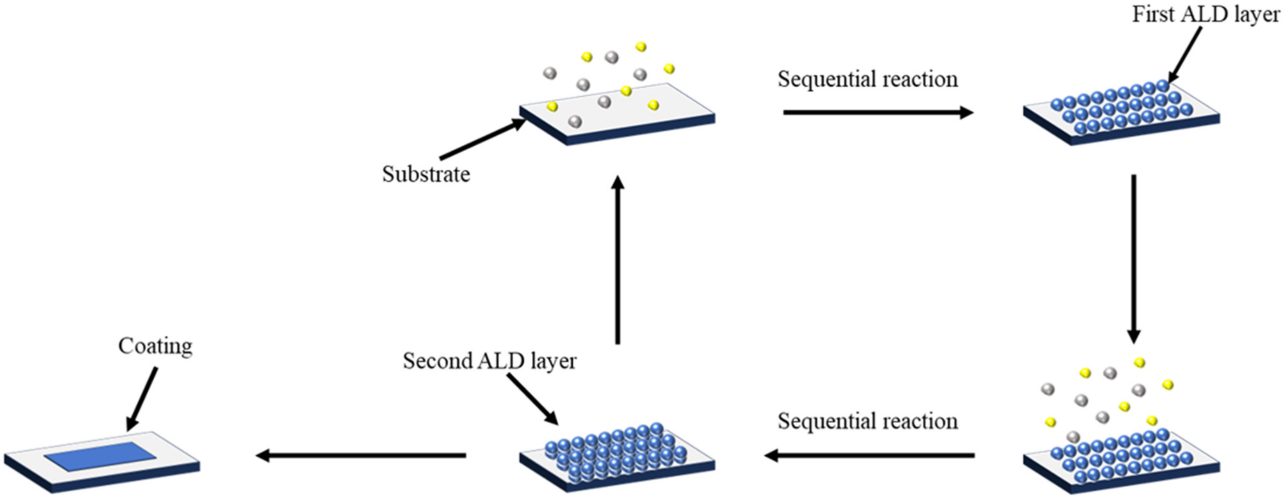

Figure 5: Schematic illustration of ALD cycle.

via the FSP method can be such as high purity, nanostructure control, scalability, and versatility [92].

3.1.6 MBE method

MBE is a vacuum-based deposition method which is carried out through elemental Ti evaporation in the presence of O_2 gas. Two beams of Ti and O_2 are heated in different effusion cells and pointed to the support surface (normally kept at a certain temperature), where they interact and condense to form a crystalline nano TiO_2 layer [53]. This process can provide a precise control to enable an epitaxial growth of the film, by which it grows in conjunction with the crystal structure of the support surface. The process is illustrated graphically in Figure 7. The technique is known for its capability to form a well-defined smooth film at the atomic level, with high purity, and tuned stoichiometry (Ti to O_2 ratio). The coatings *via* MBE can be tailored for special applications such as sensors, electronic/optoelectronic products, or advanced research projects where crystalline quality is crucial. Besides high cost, and complexity, another disadvantage of this method can be, support type limitation since temperature control of the support is necessary, and the material properties of the support can impact the final product quality.

3.1.7 Fluidized bed deposition method

TiO_2 NPs are fluidized in a bed in this method and then coated on the support surface. The technique is applicable for coating powders or irregular shape supports [93–95]. The principle of this method is to have a bed (container) of suspended particles as powder which can behave like liquid using an upward flow of gas (air flow through a perforated plate) for fluidization from the bottom. The targeted support will be immersed into the fluidized bed, where the suspended NPs can be collided and deposited on it. If the support is heated, or any binder or adhesive is applied, the particles will better adhere to the surface. In this dry method, the coating thickness is controlled *via* deposition time and support movement within the fluidized bed, to build a uniform coating [93]. Further heat treatment (of around 500°C) will result in an improved bonding strength, crystallinity, phase transformation, and can remove any residual moisture or organic matter. Simplicity, cost-effectiveness, applicability for complex geometrics, and thicker coating with acceptable adhesion (especially if the support is preheated or if any binder is applied) are features of fluidized bed coating. This technique is suitable for large-scale and industrial applications such as in corrosion-resistant coatings. Particle agglomeration possibility is reported if the NPs are not well-dispersed in the fluidized bed [96].

3.2 Wet chemical TiO_2 coating methods

In wet chemical synthesis methods normally, reactions are involved within chemical solutions and therefore, are also known as chemical solution deposition methods. The technique/s can be carried out *via* applying a Ti precursor solution onto a heated support for the deposition of TiO_2 film [97–99]. Although the techniques are generally simple and low in cost, they might result in less precise film fabrication, comparatively [100,101]. The three most frequently applied wet coating methods with their sub-categories are explained and illustrated as follows.

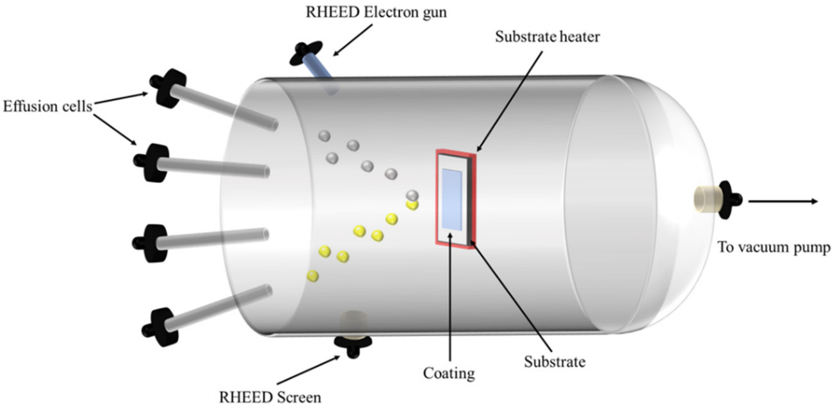

Figure 7: Schematic diagram of TiO_2 thin film preparation by MBE method.

3.2.1 Sol–gel coating methods

Among the well-known wet chemical methods for large-scale film coating are the sol–gel-based methods [34]. Prior to the coating, a liquid precursor is transformed into the sol (solution containing the NPs) or the gel (the denser phase of the sol whose formulation methods are explained elsewhere, stoichiometrically [102]) will be provided. Then, a thin layer of the gel would cover the support surface through methods such as dip coating [32,103], spin coating [104–110], spray coating [34,111], or Dr. blade [11], which are separately explained below. Most of these coating methods are followed by a hydrothermal treatment step (annealing) at high temperatures (around 550°C and above in muffle) to crystallize the coated NPs to the desired crystal type and remove the intermediates. Multiple cycles of coating and annealing can be performed to maximize the Brunauer–Emmett–teller (BET) surface area, anatase crystal rates, and adhesion. This mechanism is graphically presented in Figure 8. Some literature reported an enhanced coating features if UV light exposure is applied prior to the hydrothermal step as PAD [62] method.

3.2.1.1 Dip coating

Gently immersing the support into the gel, holding it deep into the gel for a certain period of time (around a minute), and lifting it out at a low speed are the principles of the dip coating method, as the process is illustrated graphically in Figure 9a. It has been reported that dip coating is preferred over spin coating due to higher adherence and efficiency [81,82], although is challenging for large samples [34]. When using dip coater equipment, parameters such as immersion speed, pulling speed, gel viscosity, and support type can greatly impact the coating thickness and homogeneousness [112,113]. In some cases, due to the shape or the size of the support, such as crushed glass pieces [63], adding the gel into a container containing the support and draining it after, could be applied. If multiple layers of coating are required, then repetitive cycles can form a layer-by-layer film on the surface (an optimum of 5–6 layers of TiO_2 coating on glass support was recommended by Gong *et al.* [9]). A less viscous gel in multiple rounds of coating will result in a more uniform and crack-free film with less agglomerations [102,114].

3.2.1.2 Spin coating

For the spin coating method, after the substrate is thoroughly washed and dried, it will be fixed on the spinner, and then an adequate amount of TiO_2 gel would be placed (or dropped) on top of the support surface (at the center) before being rotated (spun) horizontally at certain rpm (rapidly) and time (800 rpm for 25 s was reported by Wang *et al.* [32]). This will disperse a thin uniform layer of the coating solution due to centrifugal force [81], the specific steps are shown in the Figure 9b. The spin coating generally includes two rounds of rotation at the wet and drying phase (at 200–2,500 rpm), each followed by a holding period of 10–20 s [27]. The higher rotation speed (3,000–5,000 rpm) will result in a thinner layer, especially since a considerable amount of the sol will be thrown away

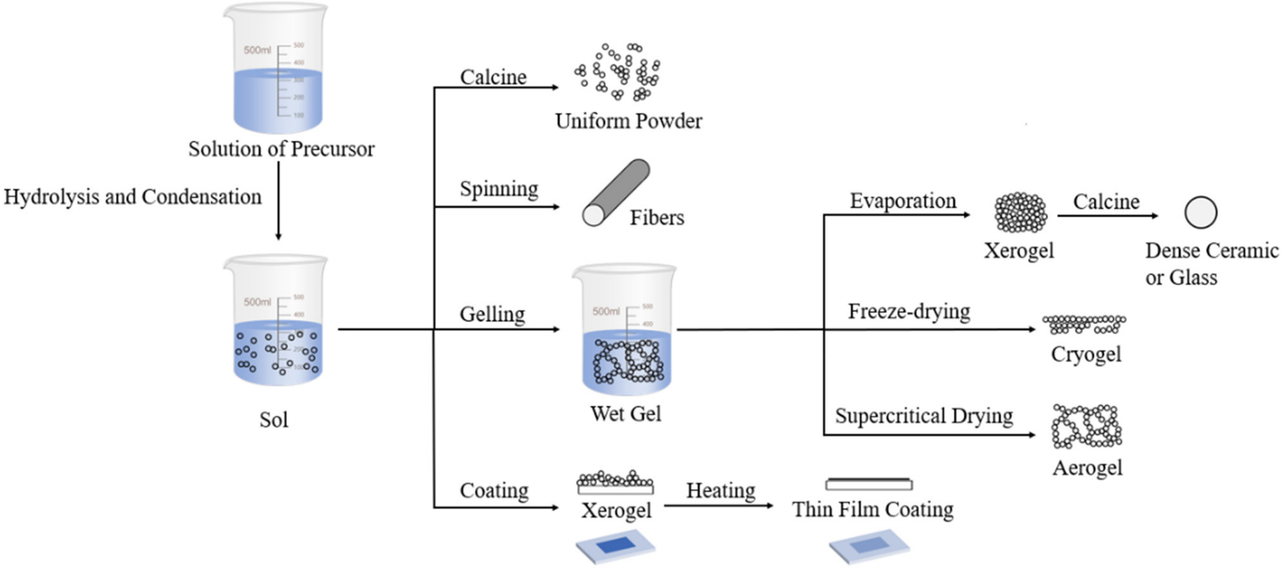

Figure 8: Sol to gel and to film conversion steps: air drying, oven and muffle, multiple annealing, gel to xerogel, and then dens film.

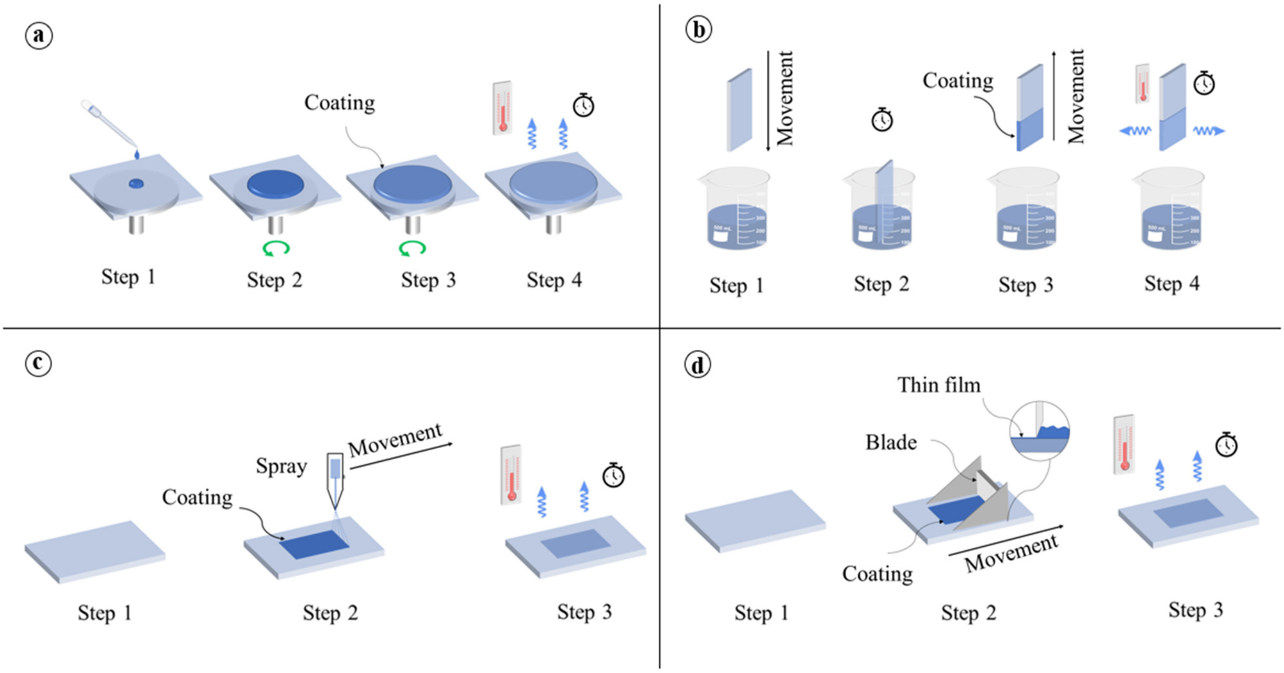

Figure 9: Different sol–gel coating methods: (a) Spin film coating method, (b) dip coating method steps for film deposition, (c) spray film coating method, and (d) Dr. Blade film coating method steps.

from the support surface during the spinning step [115–117], although other parameters such as the viscosity of the sol are also important in controlling the thickness [118].

3.2.1.3 Spray coating

The spray coating usually is carried out using a fine nozzle sprayer at a certain discharge rate, pressure, and distance. In this method, a precursor solution containing TiO_2 NPs is sprayed onto a heated substrate. The solvent evaporates, leaving behind a deposited TiO_2 film. The process is illustrated graphically in Figure 9c. To avoid nozzle blockage, a very thin gel (or sol) with low viscosity must be used to coat every corner of the surface with the desired thickness. This method is more suitable for non-flat samples [34].

3.2.1.4 Dr Blade

Dr blade or tape casting method is a technique in which the moving substrate can have a controlled thickness of the coated material (started from around 20 micron thickness of the slurry [119]) since the blade meters the gel to produce a uniform thickness on the moving substrate. The detailed steps of this method are shown in Figure 9d. This method can be applied to coat large surfaces [11].

3.2.2 IWI method

This method takes place *via* impregnation of a porous support using liquid Ti precursor but to use just enough of it so that the support pores are filled (or wet), but not the surface. Knowing the pore volume in this method is critical for deciding the amount of the precursor solution required. For impregnation, the support is immersed into the precursor, or the precursor is added dropwise in an orderly manner. The amount of precursor is accurately measured to make sure that it just fills the support pores, without any extra precursor being left on the support surface [120,121]. This will also minimize the precursor waste. After drying the substrate in oven, it will further go through the calcination process (of about 500°C) for TiO_2 crystallization and phase transformation. Porous supports with high surface area will have a controlled film thickness through this technique and are applicable in various fields such as water/air purification, contamination removal, surface modification, and self-cleaning.

3.2.3 EPD method

EPD is a coating technique which employs an electric field to charge TiO_2 particles (as suspended), and drive toward a conductive substrate (electrode). This migration of charged particles leads to the deposition of a uniform and thin film coating [122]. For this method, after preparing the sol (TiO_2 suspension as electrolyte solution) the particles are positively/negatively charged through pH modification or *via* using stabilizers, which causes them to migrate when an electric field is available. In this process, two electrodes (negative and positive) are required in the sol for the migration of charged particles. The support (submerged in TiO_2 sol) will be used as cathode (negative), and another counter-electrode (a plate made of stainless steel or platinum) will serve as the anode (positive). After anodization, a direct current (DC) electric field is applied in the sol, driving the charged NPs toward the support surface [123,124]. Applying the right charge type (−/+) is necessary to ensure the correct direction of particles migrating toward the substrate. The schematic diagram of this method is shown in Figure 10. The TiO_2 particle accumulation rate depends on multiple parameters such as the DC current voltage and its duration, the concentration of NPs in the sol, and the distance between the two electrodes [125]. After deposition, the coated support is dried followed by annealing process at a high temperature (around 500°C) for crystallization.

Besides simplicity, the method offers a cost-effective, dense, durable, and uniform coating, with an even distribution of NPs that is applicable to coat on supports with complex geometries. However, the applied substrate in this method must be conductive (like metal) for the migration of charged NPs, otherwise, it might require pretreatment steps. Also, to maintain TiO_2 particle suspension stability is critical in this technique, to ensure the coating quality [126]. The usual applications of nano TiO_2 coating *via* other methods (such as optical, environmental remediation, energy saving, protective layers, *etc.*) are also available *via* this technique.

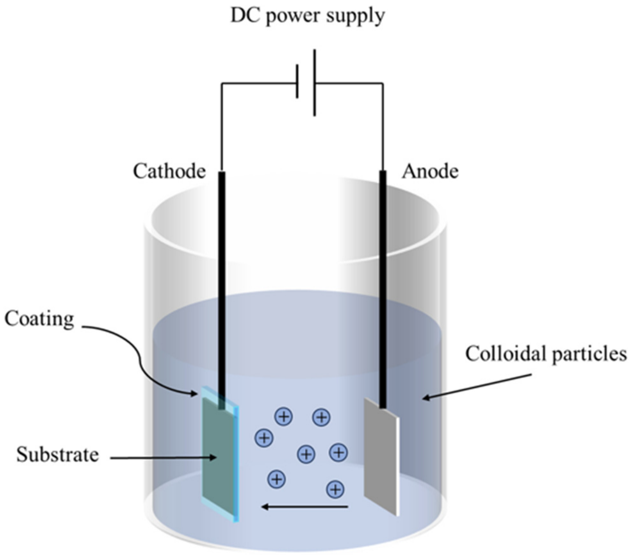

Figure 10: Diagram of EPD apparatus.

3.3 Summary of TiO_2 Coating Methods

See Table 1.

4 Coating characteristics assessment

Once the coating is done, a set of tests is required to examine the coating morphology, obtained NP type and size, roughness and cracks of the film, surface area, adherence, defects, *etc.*, which are important as influencing factors on the mechanical and chemical desired properties of the coating, based on the targeted application. The commonly used testing equipment and techniques are as follows.

4.1 X-ray diffraction (XRD)

XRD is a powerful and widely applied non-destructive method for structure characterization [127], phase composition identification, and crystallinity level (in terms of size, structure, and orientation) of coatings with highly accurate and reliable results. These are necessary parameters for analysis and coating assessment which influence the properties of the final product during the film fabrication, for the targeted application. For this test, an X-ray tube produces X-ray beams and illuminates them at the coating surface (sample). Due to the interaction of X-ray beams with the material crystal lattice, the beams will diffract in certain directions according to the atomic planes (or lattice planes) space of the crystal structure. This will cause the diffracted X-ray beams to form a pattern which can be distinguished by a detector. Through analyzing the diffraction pattern features (such as peaks, intensity, and angles) the arrangement of atoms in the crystal can be identified [128–130]. The frequently used term of 2θ in this test is referring to the angle between the incident and diffracted X-ray beams, as a comparison basis to classify the diffraction pattern intensity. Moreover, the internationally approved diffraction features information (such as ICCD) provides standard databases of distinct XRD patterns for comparison and phase identification of the coated material

Table 1: Summary of the TiO_2 coating methods with their sub-methods, advantages, disadvantages, and applications, comparatively

Method	Description	Applications and advantages
PVD (PLD, electron beam and thermal evaporation, magnetron and ion beam sputtering)	Evaporation or sputtering of atoms, and condensation on substrate in vacuum environment	Wide range of application: optical, wear resistant layers, photocatalysis; thin film and good adhesion, slow and high cost
Plasma coating (plasma spray, RF plasma coating)	Vaporized Ti is injected into highly ionized gas, which oxidizes the particles and drive toward support, for supports with low surface tension	Antimicrobial, optic, anticorrosion, thermo-sensitive supports, barrier, made at low temperature, costly, controlled thickness, high adhesion
CVD (thermal CVD, MOCVD, HPCVD, LCVD APCVD, LPCVD, PECVD	Gaseous Ti precursor reacts on substrate in heated vacuum to deposit vaporized thin film	For electronics, and precise control applications, thin film, high purity, excellent adhesion, low temperature, costly.
ALD	Self-limiting chemical reaction, one atomic layer at a time based on CVD principles	Low temperature, thin film, slow, costly, precise control industries, semiconductors
FSP	Uses fuel for high-temperature flame decomposing vaporized precursor into film	High deposition rates, high temperature, thin film, scalable
MBE	Vacuum-based, elemental Ti evaporates in presence of O_2 beam, pointed to the support to interact	Smooth ultra-thin film, high temperature, sensors, electronic/optoelectronic products
Fluidized bed deposition	Suspended particles using an upward gas flow fluidized to coat the support	Thick coating, for large scale, corrosion resistance
Sol–gel (dip, spin, spray, and Dr. blade coating)	Liquid precursor is transformed to sol and then gel, followed by thermal treatment for crystallization	Low cost, simple, thick coating, for photocatalysis, self-cleaning
IWI	Impregnation of a porous support *via* enough liquid Ti precursor so just pores are filled, but not the surface	High temperature, low cost, self-cleaning, water/air purification, surface modification
EPD	migration of charged particles *via* pH modification, and drive toward conductive substrate in electric field	Low cost, high temperature, simple, dense film, wide range of application

[131] (such as the relative amounts of anatase or rutile phases in case of TiO_2).

The average crystal size can be identified *via* the Scherrer equation (equation (1)) from the broadening of the diffraction peaks. Based on equation (1)

$$D = K\lambda/(\beta\cos\theta), \tag{1}$$

where D shows the domaining average crystallite size, K is constant, used as a shape factor (normally equal to 0.9), λ presents the X-ray radiation wavelength, β (in radians) presents the full width at half maximum of the peak, and θ is the Bragg angle, which is the diffraction angle corresponding to the peak [132].

Normally, the larger peak width shows a smaller crystal size. The coated TiO_2 level of crystallinity can be extracted from the diffraction peaks sharpness as well as intensity. The sharper and the more well-defined peaks, show the more well-crystallized films, while the more broad peaks indicate less crystallized films [133]. XRD can also be used to identify any lattice strain in the crystal. This phenomenon is referred to the deformation of the crystal structure, caused by stress or any defect, that is assessed from diffraction peaks broadening. Besides, the diffraction peaks relative intensity can be used for orientation and crystallographic plane analysis [134]. Some peak standards of XRD patterns, corresponding to anatase and rutile TiO_2, can be mentioned as: 25.3°, 38°, 48°, 55°, and 62.5° corresponding to 101, 004, 200, 105, and 204 crystal planes of anatase, respectively, and 27.4°, 36.1°, 41.2°, 54.3°, and 56.7° corresponding to the 110, 101, 111, 211, and 220 crystal planes of rutile, orderly, all at 2θ values.

The known limitation of the XRD test for film coating assessments is resolution imperfection when the film thickness is low (generally lower than 100 nm) because the primary analysis goal of this method is the bulk crystal structure but not the films. This might cause a weak diffraction signal which leads to difficulties in TiO_2 phase identification, roughness, and amorphous surface layers unless the thicker film is being analyzed.

4.2 Scanning electron microscopy (SEM)

SEM is an important method applied for morphological and structural characterization such as surface morphology, delamination, grain structure, thickness, integrity, and uniformity of films. The method offers high-resolution images of the surface as well as the cross-sectional structure of the film, providing valuable information about the coating. The principles of SEM work are based on scanning an illuminated beam of electrons with high energy at the substrate surface. Reflection of different signals emitted from the surface after the electron beam strikes are then detected by various detectors, and employed to form high-resolution images [135,136]. These reflected signals might be including secondary electrons (used for surface morphology analysis such as roughness, porosity, and distribution of particles) or backscattered electrons employed in composition and structure assessment [137]. SEM can also be coupled with energy dispersive X-ray spectroscopy or characteristic X-ray applied for elemental composition identification, and impurity assessment [138].

As a non-destructive method, SEM provides quick and efficient high-resolution images for TiO_2 coating examination to assess both surface and cross-section properties of films, which is vital to the analysis of coating characteristics. However, it also has its drawbacks and sensitivities such as thin film features detection limitations, conductivity requirement for imaging, which needs coating surface modification with conductive materials, and limited areas imaging, which may not represent the entire film surface.

4.3 BET

BET measurement is a commonly used method to test the specific surface area and pore structure of materials or coatings. This technique applies N_2 gas as adsorbate to determine how much of it is adsorbed on the surface of the object at certain temperature that is the liquid nitrogen temperature. For this examination, the film is exposed to N_2 gas stream at −196°C, and the amount of gas adsorbed is tested at different relative pressures. Then, based on adsorption isotherm, the BET formula (equation (2)) is used to compute the specific surface area in m^2/g [139].

$$P/\left(P_0 \times \left(1 - \frac{P}{P_0}\right)\right) = 1/(V_m \times C) + (C - 1) \times \left(\frac{P}{P_0}\right)(V_m \times C), \tag{2}$$

where P/P_0 is the adsorbate gas (N_2) relative pressure of equilibrium to the saturation vapor pressure at the test temperature, V_m indicates the monolayer of the adsorbed N_2 gas quantity, and C is the constant related to the adsorption heat between the N_2 gas and solid surface.

Combined with other methods such as Barrett-Joyner-Halenda (BJH) the BET test can be applied to calculate the pore size range, distribution, as well as total pore volume (void space) of the film coatings [140]. This non-destructive method provides accurate and reliable measurement for the coating's specific surface area; however, it might not be suitable for heterogeneous coatings or very thin films with thicknesses of less than a few nm, due to limitations in N_2 gas absorption.

4.4 Fourier transform infrared spectroscopy (FTIR)

FTIR is applied for composition verification, phase recognition, and to identify the presence of different chemicals in the coatings. This will enable to detect contaminants or any functional groups (such as when using surfactants, doping, hydrophobicity assessment, *etc.*) involved on the deposition surface, for coating quality assessment. This spectrophotometry method uses the infrared light spectrum to analyze the vibration peaks (of Ti–O, O–H, *etc.*) as well as other films' characteristics [141,142].

4.5 Durability

Durability test for coatings is necessary to know their life cycle and possible treatment/recoating requirements [143]. This result will ensure that the film maintains its performance during the desired lifetime under targeted environmental conditions. It shows the film's ability to tolerate criteria including abrasion, corrosion, weathering, UV, and chemical exposure while keeping its physical and/or chemical properties [144,145]. Different examinations have been applied to assess the coating's durability, taking into consideration the targeted application. Here the eight most frequently practiced durability testing techniques for TiO_2 coating are introduced with the details explained in Appendix A including the Abrasion resistance test: such as Taber Abraser, and scratch test; Weathering and UV exposure test: such as accelerated weathering, and outdoor exposure; Corrosion resistance test: such as salt spray test, and electrochemical impedance spectroscopy (EIS); Chemical resistance test: such as immersion, and surface interaction assessments; Photocatalytic stability test: such as degradation efficiency, reusability, and temperature cycling tests; Adhesion test: such as cross-hatch adhesion, and pull-off adhesion tests; Mechanical durability test:

such as impact resistance assessment; and finally, Hydrophobicity test such as contact angle measurement.

4.6 Transparency

Since opacity can cause reduction in the transmission of light, transparency test for TiO_2 coating takes place where optical properties and clarity is a necessary criterion for the coating application. These applications can be self-cleaning windows, solar panels, optical coatings, and transparent conductive layers where transparency can impact the applicability of the final product. The seven common transparency test techniques for TiO_2 film coating are including: First, the UV-vis Spectrophotometry that is a test of transmittance and absorption of the light spectrum in which high transmittance (400–700 nm) shows a good transparency while UV blockage can be observed when high absorptions occur in the range of 100–400 nm [146,147]; second method is optical coherence tomography (OCT) imaging which measures the thickness as well as uniformity of the coating [148–150]; Refractive index measurement (RIM) uses a refractometer or ellipsometer, which quantifies how much of the light transmitted or refracts through the film (for TiO_2 this index is 2.5–2.9) is the third method [151]; fourth is the film thickness test through optical profilometry or contact profilometry which measures the coating thickness as it impacts transparency [152]; fifth is the visual coating inspection, which offers a quick evaluation of its optical clarity and cloudiness; sixth is the coatings' color assessment method that applies the CIE Lab system to check the color changes or coatings' tints *via* colorimeter which quantifies the L, a, and b values as the film lightness and chromatic components [153]; and finally seventh is the light scattering test *via* dynamic light scattering (DLS) or light scattering goniometers which evaluates scatterings' angle to check the potential opacity [154–156].

4.7 Summary of TiO_2 coating characteristics evaluation methods

See Table 2.

Table 2: Summary of coating assessment methods, equipment, and application areas

Method		Applications
XRD		Crystal structure, phase composition, lattice parameters, and stress state. Disadvantage: Has resolution limitation when the film thickness is low
SEM		Surface morphology (roughness, porosity, and distribution of particles), delamination, grain structure, thickness, integrity, and uniformity of films. Disadvantage: Thin film features detection limitations, conductivity requirement for imaging which needs coating surface modification with conductive materials, and limited areas imaging
BET		Specific surface area and pore structure, calculate pore size range, distribution, as well as total pore volume (Combined with other methods such as BJH). Disadvantage: Not suitable for heterogeneous coatings or very thin films with thickness of less than few nm due to limitations in N_2 gas absorption
FTIR		Composition verification, phase recognition, to identify the presence of chemicals in the coatings
Durability test	Abrasion	Quantifies the film ability to tolerate mechanical wearing or friction using abrasion or scratch tests
	Weathering/UV	Evaluate the ability of film to withstand harsh weather conditions & sunlight exposure in real scenarios
	Corrosion	Assess their resistance abilities to the environments including salt or humidity
	Chemical	It shows the ability of film to resist degradation in environment exposed to chemicals
	Photocatalytic	It evaluates the photocatalytic stability of the coating, including degradation efficiency and reusability
	Adhesion	It tests the possibility of coating damage, *i.e.*, peeling, cracking, and delamination under stress
	Mechanical	It tests the resistance of the film to impact/deformation when coating is subjected to mechanical stress
	Hydrophobicity	It tests the ability of films to repel water droplets when applied for self-cleaning
Transparency	UV-vis	A test of transmittance and absorption of the light *via* spectrophotometer
	OCT	To measure the thickness as well as uniformity of the coating
	RIM	Uses refractometer to test the amount of light transmitted/refracted through the film
	Thickness	It measures the coating thickness by optical or contact profile method
	Visual coating	It offers a quick evaluation of its optical clarity and cloudiness
	Coating color	It applies the CIE Lab system to check the color changes or coatings' tints
	DLS	It evaluates scatterings angle to check potential opacity

5 Key problems of coating

Considering the type of application expected for TiO_2 coating, the possible challenges which the coating might face may come from a variety of reasons before, during, and after the coating, as listed below. These can range from the choice of applied materials such as support type or chemicals used (before coating) to the selected coating methods (during coating), and even during the practical application (after coating), which can lower the final product/system efficiency.

5.1 Durability

TiO_2 film coatings, normally, must withstand different conditions, such as harsh environmental exposure (to UV, humidity, temperature, and flow rate), and chemical/electrical interactions. During time, these conditions will decrease the effectiveness and structural integrity of the film. Therefore, improving the chemical and mechanical strength of the film is vital. This will necessitate to develop a more stable coating through different techniques such as applying hybrid coatings with improved properties, such as to combine titanium dioxide with other materials including silica, carbon, *etc.* Some coating methods might require surface modification to enhance the affinity of the film with the support surface, which is called functionalization [27]. These techniques are post modification, applying surfactants, or pre-treatment (for instance surface etching for efficient adherence *via* H_2SO_4 or SO_3) [157] which act like anchor points to better decorate the NPs in a matrix or skeleton, on the targeted surface [27]. Besides the choice of deposition methods, material selection (like precursors and substrates) is also vital. Employing the wrong substrates can impact the photocatalytic activity, and may cause effectiveness reduction due to residues by the synthesis of photocatalyst, and the film contamination by intermediates produced through different reactions [157]. For example, organic polymers are transparent, light, flexible, cheap, environmentally resistant, and available; however, due to their low surface free energy and thermodynamic characteristics, only low temperatures (up to 170°C [9]) coating methods could be applied on them; while nano TiO_2 crystallization requires temperature of around 500–550°C for anatase phase transformation and acceptable adherence [7,9,23]. Besides, most polymers are highly degradable under UV light radiation [29], due to a process called photo-aging which causes the loss of structural integrity through a decreased molecular weight induced by UV [158]. Moreover, the weak van der Wall force between the NPs and the polymers along with hydrophobic nature and low wettability [27,159] of the surface, ease the coating detachment [160].

5.2 Low efficiency

Film coating's efficiency might be limited by parameters including surface area, NP size, TiO_2 phase and crystallinity level, coating thickness, and the presence of pollutants [70,161,162]. To achieve a coating with the most desired properties through selecting the best match method and chemical compounds is the first step to minimize future efficiency issues. Developing a self-cleaning TiO_2 coating, which is capable of resisting the formation of biofilms over the application time, is a necessary action to maintain high efficiency. Moreover, nano TiO_2 doping *via* employing other elements (such as nitrogen, sulphur, and carbon) can enhance its efficiency under the visible light spectrum, which will improve its performance under natural daylight [163–165]. For other applications such as optical, protective barrier, *etc.*, where certain properties such as transparency or thickness are the priority, control over the fabrication process would be the key to enhance the coating efficiency and minimize maintenance or recoating requirements. Coating heterogenicity or weak adhesion will lead to delamination and localized degradation, which will reduce the coating efficiency and life cycle of the film. Applying more advanced methods such as EPD or plasma spraying which ensures uniform deposition, strong adhesion, and longevity is recommended, if the cost-effectiveness allows.

5.3 Scale up and commercialization

The scale-up of nano TiO_2 film production for practical, outdoor, and industrial applications is a well-known challenge compared to lab-scale production [166]. Here the production cost-effectiveness, reproducibility, and scalability are to be taken into consideration. This will be difficult when the film coating goes through precise processes such as coating uniformity or curing, which requires full control over the ambient condition. For this purpose, cost-effective coating methods (such as sol–gel or spray pyrolysis) or materials (supports or hybrid compounds) are preferred, while these solutions might also need to be optimized for commercially viable large-scale production [167].

Moreover, standardization and automation of processes can cause cost reduction and/or coatings properties enhancement. This will help to unlock the full potential of TiO_2 film coatings in diverse applications.

5.4 Fouling and biofilm formation

When it is exposed to the outdoor environment, TiO_2 film coatings can face the problem of fouling, which is the accumulation of biofilms or byproducts remaining from oxidation/reduction processes on the coating surface. The unwanted settled and accumulated byproducts or “foulants” are inorganic ions and compounds, microorganisms, carbonaceous species, colloids, metals, and solutes on the surface or within the substrate structure caused by interactions between contaminants and support [6,168]. This is more common in aquatic or wastewater environments of bioreactors, which reduces the efficiency. This phenomenon can decrease the catalyst’s active surface area, interrupt the photocatalytic activities, and lead to shorter intervals of maintenance requirements.

Developing coatings with antibiofouling ability, through surface modification or self-cleaning properties (such as hydrophobic coatings, doping *via* silver or other materials, *etc.*), can help to minimize this problem. Some scholars reported that a dynamic cleaning systems such as a periodic UV exposure, can help to better manage the expected photocatalytic activities [169,170]. It was suggested that occasionally applying pure water instead of contaminated samples in the reactor for photocatalysis can completely remove the degradable intermediates and foulants from the coated film [7].

5.5 Photocatalytic activation and UV light limitations

Technically, TiO_2 particles on the film require UV light for activation. However, for industrial and/or outdoor applications, there may not always be enough natural UV available. Besides, an evenly illuminated light might not irradiate at the coating surface, which impacts its overall effectiveness. Doping TiO_2 with other materials such as N_2 or C can, to some extent, improve its expected efficiency. Using solar panels to convert a bigger portion of the light spectrum to UV is another option. In terms of transparency, some support such as polymers (due to degradation over time or due to additives which are used as UV stabilizers to reduce the photooxidative process [171]) or glass (owing to iron compounds [172]) are opaque to ultraviolet radiation [29]. In polymers, this opacity will increase since C–C and C–H bonds can be broken down to lower molecular weight compounds by the photon’s energy from the UV light [173]. Using supports such as low iron glass [174] can increase the UV penetration for enhanced TiO_2 activation.

5.6 Environmental impacts and toxicity

While titanium dioxide itself is considered as a non-toxic element, when it comes to nano-scale, it can pose human health risk or environmental risks, if not properly handled during production or application [175]. This is a concern for commercialization and scale-up production of TiO_2-coated materials. Following safety protocols and standards for handling and disposal of TiO_2 needs to be taken seriously into consideration, to ensure its eco-friendliness and minimized risks.

5.7 Impact of support type

Nanomaterial film coating is the immobilization of NPs as a thin layer on a surface to change, control, or enhance the physiochemical properties of the support surface or just for the purpose of the film application. However, the substrate will also have an effect on the TiO_2 thin film in turn. Although there are various types of substrates, the selection of support type is based on the application, requirements, and method used for preparing TiO_2 films. Nanostructured TiO_2 films could be grown on different types of substrates such as glasses (quartz glass, sodium-lime glass, borosilicate glass), metal plates (such as stainless steel and aluminum), as well as ceramic tiles, rubber sheets, and even some substrates with poor thermal stability like polymers [176–180]. Surface tension, thermal stability, scalability, availability, transparency and transmittance, cost-effectiveness, UV resistance ability, porosity, durability, coating method, chemical stability, and available technology are of important parameters to consider when selecting the support type [181–185].

In fact, the substrate not only affects the formation of crystal phase of TiO_2 films but also the microstructure, and surface morphology of the films, greatly, which all will have impact on the performance of the final product (such as photocatalytic [186,187]). For instance, the roughness of the substrate is of great significance when preparing TiO_2 thin films. Substrate roughness promotes the retention of TiO_2 colloid. The rougher the substrate, the more TiO_2 can be carried on the prepared film, and the thicker it will be [188]. This also indirectly affects the photocatalytic performance of the TiO_2 film.

Krýsa *et al.* found that after preparing titanium dioxide films using three different types of glass with different Na contents as substrates, the higher the Na^+ content in the glass, the worse the photocatalytic activity of the titanium dioxide photocatalyst. This is because sodium ions in the glass used as the substrate diffuse onto the titanium dioxide film during calcination, and sodium ions prevent the formation of the photoactive anatase phase, thereby affecting the photocatalytic activity of the titanium dioxide film [189,190].

Qiu *et al.* prepared TiO_2 films on glass, ITO glass, and P-type single crystal silicon. They observed the surface morphology of the titanium dioxide films through atomic force microscopy. It was found that nearly circular particles of about 80 nm were uniformly arranged on the glass surface. For P-type monocrystalline silicon, they observed perfect round particles within the range of 50–150 nm, and the surface roughness is larger than the other ones. They found slender particles closely packed on ITO, with a grain size of approximately 20 nm and a relatively small surface roughness. It can be seen from this that the surface topography, including the shape and arrangement of particles, also largely depends on the substrate [186].

Harizanova *et al.* prepared TiO_2 films on silicon wafers, ordinary glass, and ITO glass using the sol–gel method. They found that the optical behavior of the TiO_2 films obtained on ITO substrates was significantly different. The coatings reveal very high transparency in the visible spectral range up to 85%, despite the increase in the number of layers and the heat treatment, reaching the transparency of bare ITO substrate. However, as the number of layers and annealing temperature increase, the transparency of TiO_2 films on ordinary glass decreases. This indicates that the optical properties of titanium dioxide films obtained from different substrates are also different [191].

Daoud *et al.* found in their research that the thermal stress caused by the difference in the coefficient of thermal expansion between the substrate and the film would lead to the formation and propagation of microcracks along the grain boundaries after crystallization. In their research, they used glass ceramics and aluminum as substrates to prepare titanium dioxide films and reached the above conclusion by comparing the thermal expansion coefficients of the three materials and titanium dioxide (anatase phase) [188]. Overall, the importance of the support type must be taken into account for any coating considering its application as well as all the abovementioned parameters as an influencing factor.

6 Concluding remarks and future perspectives

Development of TiO_2 coating methods will continue to be among the hot topics, taking into consideration its versatile and exceptional photocatalytic, antibacterial, UV protection, and optical properties which will unlock new applications. As research works will continue, TiO_2 coating methods are expected to have more broad applications in a variety of fields, from environmental to energy, electronics, and health in order to enhance the quality of life. Some of the future key drivers in advancements of film coating technologies for its widespread adoption of coatings might be as follows.

1. Development of low-cost coating technologies: novel techniques for applying TiO_2 coating uniformly onto large surface areas will enhance its scalability and decrease the process costs, making the TiO_2 film coating technologies more viable, commercially.
2. Nanostructured and 3D coating: Advancements in nanotechnology can lead to the formation of nano TiO_2 coated supports having complex geometries and 3D nanostructured with improved surface area which causes enhanced efficiency. These new coatings can offer new opportunities in fields such as textile, building, and electronic materials.
3. Automation and optimization: Automation in processes (from production to application) and optimization of deposition methods, chemicals used, and support types can cause cost/time savings with more efficient methods for mass production and scaleup. TiO_2 coatings can be integrated with an automated surface cleaning system, which their functionality can be optimized if applying AI to respond to the changes in the environment intelligently.
4. Sustainability and green engineering: As the world moves toward sustainable development, TiO_2 film coating can have a leading role in decreasing environmental contaminants of industrial activities such as through low-carbon manufacturing. Advancements in low-energy processes in TiO_2 film coating production, such as low-temperature methods, can help in carbon reduction associated with such processes. TiO_2-based coatings can be applied in green roofs or carbon

capturing technologies, which can help CO_2 conversion into useful materials.

5. Longevity and recyclability: Material modifications will be a hot spot research area for scalable TiO_2 coatings production, and integration with emerging technologies, cost-effectively. This can lead to produce durable coatings which can last longer under harsh environments for outdoor/practical uses. Efforts need to be made toward production of TiO_2 coatings with simple recyclability and/or re-manufacturability, so that at the end of the coatings' lifecycle, they can be reused as more economic products.
6. New fields and applications: The wide range of TiO_2 coatings applications will bring this technology into new industries. Advancement of coating features for its different usage can bring it to the key areas where TiO_2 film deposition is likely to evolve and bring substantial impacts in the future. These might further expand the TiO_2 film coating scopes in a variety of industries as below.

6.1 Photocatalytic applications

The ability of nano titanium dioxide to decompose different chemical compounds and biological contaminants as a catalyst when it is activated by UV offers a vast potential for its diverse applications. Marketable building materials including photocatalytic concrete for the reduction of air pollution, tiles with self-cleaning ability, or smart windows with enhanced optical properties and self-cleaning abilities are already in use, as the fields where TiO_2 applications can be expanded in. The future of TiO_2 coatings can be scaled up for air/water treatment in industrial purification systems. Products such as anti-fog, self-healing, and self-cleaning windows for vehicles or aircraft might be future active areas of research *via* such coatings.

6.2 Enhanced bandgap energy under visible light

The photocatalytic activities of nano TiO_2 are dependent on the UV light intensity, that is just a small portion of the sunlight and consequently, is limited in practical large-scale use. Research works will most likely concentrate on enhancing its photocatalytic efficiency using natural visible light, improving its reactivity for various fields. This might be possible through doping with other elements to enhance its bandgap energy and develop its light absorption range to visible light as a broader spectrum. Such hybrid combination of semiconductors can have an improved activity, with higher charge separation ability, for an efficient catalyst under natural daylight.

6.3 Energy harvesting fields

TiO_2's capability in generating electricity from sunlight through photocatalytic and photovoltaic effects has caused such coatings to be explored for application in solar panels. It has already been employed in dye-sensitized solar cells as the photoanode material. The concentration of the research work is to improve TiO_2-based solar cells in terms of their efficiency, stability, and cost-effectiveness. Advancements may bring more economically affordable solar energy with higher efficiency to the end-users. This might also play a great role in supercapacitors, batteries, or energy storage devices in general, with enhanced charge storage and cycling stability.

6.4 Healthcare and biocompatibility

As biocompatible materials, TiO_2 coatings exhibited antimicrobial and pathogen removal properties. Their application in the medical field can develop considerably. Such coating and material can be employed to heal wounds, antivirals, implant devices, use in antibacterial or self-sterilizing surfaces, or even to integrate with bandages, prosthetics, biosensors, and food processing plants. It has been studied to be applied in photodynamic therapy for cancer treatment, in which through the generation of reactive oxygen species it can eliminate the cancer cells. Moreover, TiO_2 might emerge for drug delivery or imaging agents to enhance public health.

6.5 Smart coatings and sensors

TiO_2 coatings can also play an active role in the smart materials market. This could include devices like sensors which are able to respond to environmental stimuli such as for detecting gases or pollutants. Another innovative coating can be self-healing TiO_2 that will be able to repair itself after mechanical damage or wear, for long-term applications. Moreover, TiO_2 is being further researched for applications in transparent and conductive coatings for touchscreens, displays, and smart windows.

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Appendix A

TiO_2 coating durability can be tested through different methods. Below are the eight common techniques, in brief, used for this purpose.

A1 Abrasion resistance test

This method quantifies the film's ability to tolerate mechanical wearing or friction using abrasion or scratch tests, that is important for coatings that are subjected to friction.

A1.1 Taber Abraser test

The test is carried out *via* applying an abrasive wheel which rotates to wear the film and then quantifies the amount of change or loss in the coating's surface. The weight difference or deformation after certain rotation rounds will be measured to calculate the abrasion resistance of the film [192,193].

A1.2 Scratch test

Using a sharp object (such as a needle), a line is drowned across the film surface in a controlled load manner to measure the film's scratch resistance and solidity. This can indicate that how difficult or easy the surface might be damaged when a mechanical force applied to simulate the real environment situations [194–196].

A2 Weathering and UV exposure test

Harsh weather conditions and UV or sunlight illumination can damage films during time [197]. Exposing TiO_2 films to such an environment is a test to assess their ability to withstand real scenarios.

A2.1 Accelerated UV exposure

Films will be exposed to UV light *via* xenon arc weathering hollow or fluorescent UV lamp which in fact simulates the long-term exposure of the film outdoor [198]. After a certain period of illumination time (<1,000 h), the changes in the film's physical appearance such as color, cracks, or its photocatalytic ability can be evaluated.

A2.2 Outdoor exposure

Also, films can be examined in real environmental conditions for certain periods (up to a few years based on feasibilities). Afterward, criteria such as changes in color, cracks appearance, functionality effectiveness, and degradation level can be assessed.

A3 Corrosion resistance test

Films applied as anticorrosion should be examined to assess their resistance abilities to the environmental criteria including salt or humidity.

A3.1 Salt spray test

Exposing the TiO_2 film to salt mist for a certain time (<1,000 h) in a chamber is called salt spray test in which the film's physical appearance can be monitored for corrosion marks, delamination, or coating degradation after the exposure. This method is normally used for metal coatings assessment [199,200].

A3.2 EIS

It evaluates the electrical impedance of TiO_2 film exposed to various electrochemical environments, such as saline or acidic solutions. The value of impedance indicates the protective ability of the film, including corrosion resistance or being a protective barrier [201–203].

A4 Chemical resistance test

TiO_2 films might be applied in different environments and hence exposed to various chemicals. This test shows how good the film can resist degradation in such environments.

A4.1 Immersion tests

Immersing TiO_2 film in a variety of chemicals (solutions with different acidities, oils, solvents, *etc.*) during certain times (1–3 days) can be performed in this method to evaluate the coating for degradation marks such as swelling, change in color, delamination [204].

A4.2 Surface interaction test

TiO_2 film can be exposed to smaller amounts of chemical compounds under a more controlled condition (such as spraying or wiping) to examine their effect on the film properties.

A5 Photocatalytic stability test

For photocatalytic applications of TiO_2 film, it is necessary to evaluate how stable is the coating functionality during time. For this purpose, there are three methods to employ.

A5.1 Degradation efficiency

It is the exposure of TiO_2 film-coated support to UV light while it is in use for contamination removal and evaluate the degradation rates during time to monitor if any changes in terms of degradation efficiency will happen [63].

A5.2 Reusability test

This test is in fact the films' photocatalytic effectiveness stability measurement after certain cycles of use [63].

A5.3 Temperature cycling test

If the TiO_2 film is supposed to be applied in a high-temperature environment, monitoring its characteristics after certain cycles of heating and cooling is a measure of its thermostability [205].

A6 Adhesion test

A firm adhesion of the film onto the support surface is a measure of durability [206,207]. The possibility of damages to the coating such as peeling, cracking, and delaminating when it is stressed is called the adhesion test [208]. There are two methods to measure these parameters as follows.

A6.1 Cross-hatch adhesion test

This technique is carried out based on the ability of a tape to remove the TiO_2 film when it is sticked on the support and pulled off from its surface. For this test initially, network shape cuts are made on the film's surface and then the tape is applied. The amount of film that remains on the support or the tape surface can be a measure to determine the coatings' adhesion strength [209].

A6.2 Pull-off adhesion test

In this method, the amount of force required to remove the film from the support surface is used as the direct measure of the adhesion strength. This test uses a special device which is attached to the coating surface and then pulled [210].

A7 Mechanical durability test

When the coatings are mechanically stressed, different properties of the film such as impact resistance or deformation tolerance would play important roles in the overall durability and functionality of the coating. The below test can measure the resistance ability of the film when it is under physical stress.

A7.1 Impact resistance test

This method calculates the resistance ability of the coating against being cracked or detached when an external force is applied. For this test, normally a swinging weight is released to apply a controlled force of a falling object on the coating. Then, the damage resulted from this force will be assessed to measure the resistance ability of the coating [204,211].

A8 Hydrophobicity testing

When TiO_2 film is being applied for self-cleaning purposes, its hydrophobic properties in repelling water droplets will be a critical element for its functionality. The hydrophobicity of TiO_2 coating can be reduced during time, due to wearing or exposure to the pollutant.

A8.1 Contact angle test

This technique is a measurement of water droplet contact angle when it is dropped on the film surface before and after being exposed to ultraviolet, abrasion, or weathering. The higher the contact angle of the droplet, the better the hydrophobicity of the film [212–214].