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Fabrication of self-cleaning bio-based plastic with antimicrobial properties via solution casting technique

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Abstract. PLA/TiO_2 composite materials were created using solution casting approaches in this investigation. Water absorption and antimicrobial were examined by evaluating the interactions between the essential components of the film-forming materials. The overall performance of nanocomposites with varying TiO_2 concentration was investigated. Fourier Transform Infrared (FTIR) Spectroscopy, a water absorption test, and antimicrobial analysis were used to conduct the studies. The water absorption of bioplastics was shown to be reduced when TiO_2 concentration was increased up to 5.0 wt%, with the lowest water saturation point of 17.93%. This is because the interaction between PLA and TiO_2 lowers the number of free OH groups in the resultant bioplastics. As a consequence, there was a decrease in water absorption-related deterioration, such tensile property degradation of the bioplastics. Furthermore, antibacterial activity enhanced under UV-A irradiation with a TiO_2 nanoparticles concentration of 5.0 wt%, and 1.0 wt% of the nanofilms displayed inhibitory zones of 10.49 ± 0.22 and 12.61 ± 0.78 mm and 6.83 ± 0.19 and 6.96 ± 0.62 mm for E. coli and S. aureus, respectively. Overall, the performance of the nanofilm with a higher TiO_2 concentration outperformed the pure film. Nonetheless, both nanocomposite membranes complied with the requirements of food packaging films.

1. Introduction

The widespread manufacturing and consumption of conventional plastic materials in a variety of industrial activities has a significant impact on the fossil fuel sources and environment. Bio-based polymer alternatives have emerged as a result of the development of renewable resource production [1]. Bio-based plastic made from agricultural waste fibre is an appealing option since it is a sustainable and cost-effective resource. The hydrophilic characteristic of natural fiber-based bio-composites, contrary, will result in a high moisture content of bio-based polymer, lowering its thermal and physical properties and so constraining the usage [2]. As a corollary, the 'lotus effect' characteristic are being used to overcome the sensitivity of natural bio-based polymers to moisture content and water. Poly (lactic acid)

(PLA), a synthetic bio-based polymer, is being explored globally for biomedical and consumer applications due to the increased demand for renewable materials that are sustainable alternatives to petrochemical-derived products [3, 4, 5].

Product from the polymerization of lactide or lactic acid which commonly referred as PLA, is the most typical carboxylic acid synthesized by microbial carbohydrates fermentation in nature [6]. Nonetheless, PLA's applicability has been limited owing to its hardness, degradation rate, low thermal distortion temperature [7]. Another approach for resolving these challenges would be to enrich PLA with inorganic nanoparticles such as traditional nanoclay, carbon nanotubes, zinc oxide, and anatase (A-TiO_2) [6, 8, 9, 10, 11, 12].

Antimicrobial packaging would be another type of packaging that is beneficial to both customers and food and hygiene products. Hence, innovative packaging can assist to extend product shelf life by deactivating or inhibiting the growth of fungus and microbes that are detrimental to human health [13]. It is critical to create antimicrobial and biodegradable materials. Titanium Dioxide (TiO_2) nanoparticles powder were synthesized in this study under optimal conditions to create the smallest and purest anatase phase crystallite. TiO_2 is a widely used material for self-cleaning photocatalytic surfaces. This material is also employed in antimicrobial surface investigations due to its antimicrobial characteristics. TiO_2 decomposes organic contaminants and induces water dispersion on the surface through photocatalytic activity and completing the self-cleaning approach. To improve its application, the powdered TiO_2 nanoparticles were disseminated with bio-based plastic to create a formulation with the distinctive 'lotus-effect' characteristic. The 'Lotus-effect' is defined as the capacity to self-clean as a result of hydrophobic tendencies [14]. This means that the interfaces will oppose contaminants such as solid particles, organic liquids, and biological impurities with a hint of liquid droplet [15].

Furthermore, surfaces with antimicrobial characteristics are necessary to minimize infections and reduce/kill bacteria induced by these microorganisms. Therefore, it is essential for the bio-based plastic to have the antimicrobial properties as well. The superhydrophobic and antimicrobial solution was modified by preparation four different weight ratio of TiO_2 nanoparticles were to be dispersed into PLA which are 0.1, 1.0 and 5.0 wt% to fabricate the self-cleaning bio-based plastic. Hence, in this studies, the interaction of molecular between the fundamental elements with TiO_2 nanoparticles was investigated using Fourier transform infrared spectroscopy (FTIR). Moreover, a water adsorption study was performed to estimate the weight gain after immersion to quantify the amount of water absorbed. Subsequently, the inhibitory zone technique was used to evaluate the composite membrane's antimicrobial performance against Staphylococcus aureus (S. aureus) and Escherichia coli (E. coli).

2. Materials and methods

All of the chemicals used were analytical grade and were acquired from Sigma-Aldrich. They were utilized precisely as they were acquired, with no further purification. The chemicals used in the research are Dichloromethane (DCM), and Hexadecyltrimethylammonium bromide (CTAB). Apart from this, materials such as polylactic acid (PLA), were bought from Sigma-Aldrich. Dionized water was utilized in the preparation of all aqueous solutions. Table 1 shows the list of compounds used in this study, as well as their properties.

Table 1. List of the chemicals that have been acquired, along with their properties.

Chemicals	Purchase	Properties				
		Chemical Formula	Molecular Weight ($gmol^{-1}$)	Boiling Point (°C)	Melting Point (°C)	Appearance
DCM	Sigma-Aldrich (M) Sdn Bhd, Malaysia	CH_2Cl_2	84.93	39.75	-97.00	Colourless liquid

CTAB	Sigma-Aldrich (M) Sdn Bhd, Malaysia	$C_{19}H_{42}BrN$	364.45	235.00	218.00 to 247.00	Crystalline white powder
PLA	Sigma-Aldrich (M) Sdn Bhd, Malaysia	$(C_3H_4O_2)n$	60,000.00	-	175.00 to 220.00	Amorphous white resins

2.1. Fabrication of self-cleaning bio-based plastic using solution casting method
During the bio-based plastic preparation process, four distinct weight ratios of TiO_2 nanoparticles were to be dispersed into PLA: 0.1, 1.0, and 5.0 wt %. Approximately 5.0 g of PLA was weighed and added into 25 mL of DCM. The solution is vigorously stirred at 900 rpm with a magnetic stirrer until the PLA is completely dissolved into a uniformly viscous and clear solution. In a 1:1 ratio, 0.1 wt% TiO_2 nanoparticles powder was combined with CTAB and 25 mL of DCM. The solution is then allowed to dispersed in an ultrasonic machine for around 20 minutes. After 20 minutes of vigorous stirring at 700 rpm, the mixture was added to 25 mL of dissolved PLA. The well blended solution was then placed into a petri dish. Following that, the solution was exposed to ambient for 48 hours up until the DCM solvent in polymeric coating was adequately volatilized, resulting in the formation of PLA/TiO_2 nanocomposite films with varying weight percentages.

2.2. Fourier Transform Infrared Spectroscopy Analysis
The interactions between the main components of the PLA/TiO_2 and pure PLA film materials were analyzed using Fourier transform infrared (FTIR) analysis. FTIR spectroscopy (FTIR-650, Suzhou Leiden Scientific Instrument Co., Ltd., Suzhou, China) was used to characterize the film samples at 23°C and 62.3% RH. In 32 scans at 4 cm^{-1} spectral resolution, spectra in the range 4000–650 cm^{-1} were acquired using automated signal gain.

2.3. Water Absorption Analysis
The samples were subjected to water absorption studies in accordance with ASTM D570-98, a modification which is an immersion approach in distilled water for up to 60 minutes at 21.5°C. The measurements were collected at 10, 20, 30, 40, 50, and 60 minute intervals, respectively. All of the samples were weighed immediately as they were removed from the water and the water was wiped away from the sample's surface. By calculating the weight gain after immersion, the amount of water absorbed was determined to the nearest 0.0001g.

2.4. Antimicrobial Analysis
The inhibitory zone technique was used to assess the antibacterial efficiency of the synthesized membrane against Staphylococcus aureus (S. aureus) and Escherichia coli (E. coli). To fully activate photocatalytic capabilities of TiO_2, the size of diameter of the films were prepared into 10 mm and exposed to UV-A light for 48 hours. A 8W of Philip Cleo fluorescent lamp was applied at its highest intensity of 360 nm for 6 hours (UV-A region) for For UV radialization technique. The vertical lighting distance was adjusted to 10 cm. To determine the impact of light source on TiO_2's antimicrobial characteristics, the study was done under UV light irradiation and non-irradiation settings. In order to have a specified amount of water molecules on the surface of TiO_2, the atmosphere must be kept at a high relative humidity. To generate a stable log phase, the bacteria were housed in a solid incubator at 37°C for 24 hours. Three gradients were diluted with physiological saline after the test bacteria were

activated, and with a concentration of 103–104 (105–106 CFU/mL), 100 L of microbial cell suspension was equally dispersed across the solid medium's surface using an applicator until the surface dried. Thereafter, the disc membrane was arranged into the centre of the substrate. Subsequently, the medium was cultivated for 48 hours at 37°C in a constant-temperature incubator. The diameter of the inhibition zone was used to determine the bacteriostatic effect. The study was carried out at least three times.

3. Results and discussions

3.1. Fourier Transform Infrared Spectroscopy Study

The interaction of molecular between the base components with TiO_2 nanoparticles were investigated using FTIR. According to FTIR analysis as shown in figure 1, a pure PLA film and a PLA/TiO_2 film were utilized as drawing representations. Figure 1 depicts the FTIR spectra of TiO_2 nanoparticles, pure PLA, and PLA/TiO_2 film. Stretching vibrations of TiO_2's surface O–H groups create tiny peaks in the TiO_2 spectra about 1630 cm^{-1} and 3440 cm^{-1} [16]. The stretching of C–O–C bonds also causes the vibration band at 1110 cm^{-1} [17]. In the pure PLA spectrum, the absorption peak at 1750 cm^{-1} is due to the stretching vibration of aldehyde or ester carbonyl C=O [18], while the peak at 1080 cm^{-1} is due to the stretching vibrations of and C–O bonds and C–O–C bonds [19]. The tensile vibration of the distinctive hydroxyl group of TiO_2 nanoparticles causes the band at roughly 3420 cm^{-1} in the PLA/TiO_2 film's spectra, which displays a little shift from 3440 cm^{-1}. The tensile vibration of TiO_2 nanoparticles' distinctive hydroxyl group, which displays a minor change from 3440 cm^{-1}. This is most likely owing to the partial integration of a considerable number of hydroxyl groups presents on TiO_2 surfaces into the composite membrane, leading to creation the hydroxyl group vibration peak in the synthesized membrane spectrum.

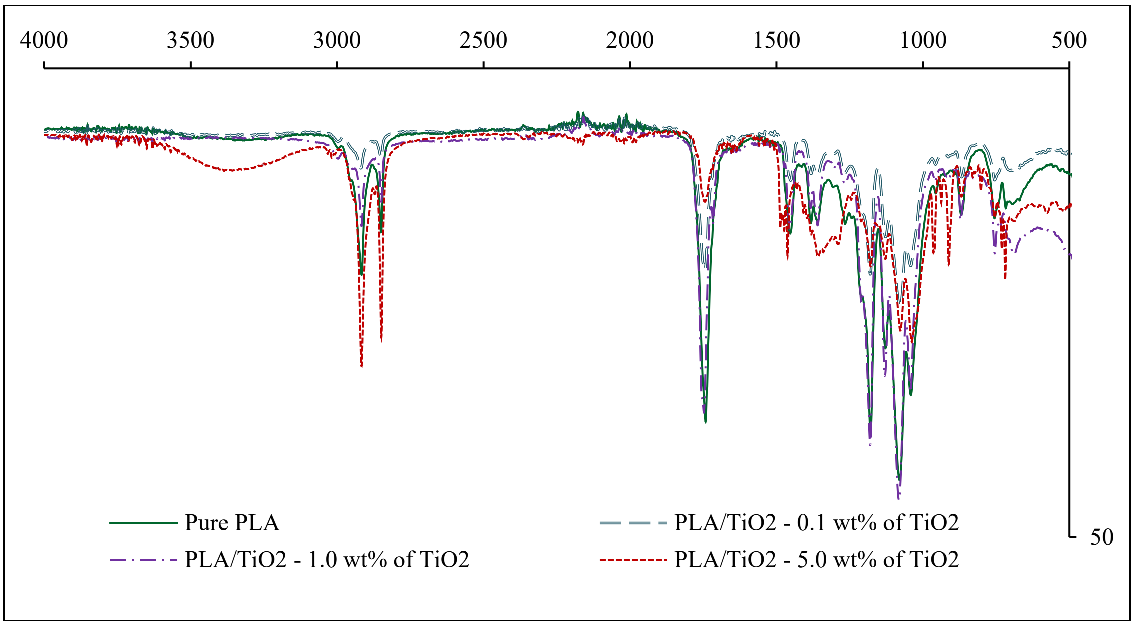

Figure 1. Combined FTIR spectra of pure PLA, PLA/TiO_2–1.0 wt% and PLA/TiO_2–5.0 wt%.

The signal at 1750 and 1080 cm^{-1} remained unaltered after the addition of nanoparticles, which might be attributed to the physical blending of PLA with TiO_2. However the intensity of the absorption peak and the region under the peak of the PLA/TiO_2 specimens increased when compared to pure PLA. A similar rise in peak intensities was found by Li et al. [20]. Apart from the surface hydroxyl group present on the TiO_2 nanoparticles, the PLA/TiO_2 nanocomposite films displayed no new peaks, showing that the components were literally physically combined. Furthermore, no functional group modifications occurred, and the films' underlying structure was not impacted.

3.2. Water Absorption Behaviour Study

The quantity of water absorbed by the bioplastic film samples at three different PLA/TiO_2 compositions is shown in figure 2. All of the films absorbed water quickly in the beginning and then gradually levelled out, achieving saturation after about 30 minutes of immersion. In comparison to the PLA/TiO_2 films, the pure PLA film absorbed more water. Water absorption increases as immersion duration increased, but dropped as PLA/TiO_2 concentration increased, with the lowest water saturation point of 17.93% achieved with the addition of 5.0 wt% TiO_2. PLA's intrinsic hydrophilic nature is primarily responsible for the mixes' water absorption.

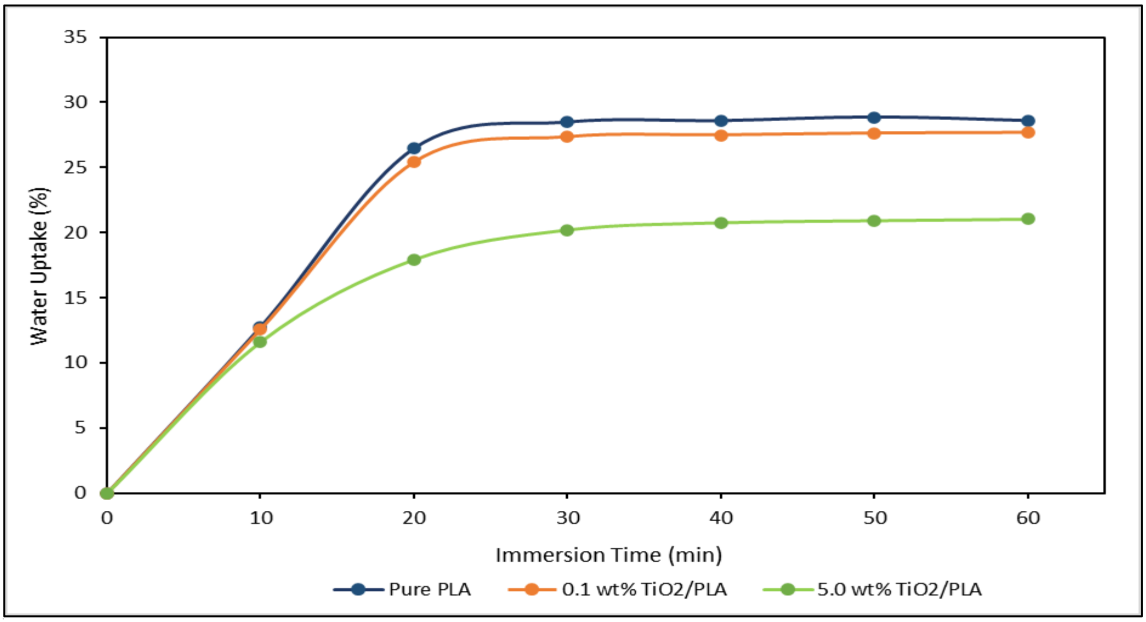

Figure 2. Water absorption of pure PLA, PLA/TiO_2–0.1 wt% and PLA/TiO_2–5.0 wt%.

The reduction in water absorption with increasing TiO_2 concentration may be explained using a general mixes rule. The final qualities of a blend of two or more polymers are a weighted mean of the material constituents' properties, according to the rule. Water saturation points of 25.4% and 17.93% were achieved with the addition of 0.1 and 5.0 wt% TiO_2, respectively. These values are still within the range of each pure material constituent's saturation point. The interaction between PLA and TiO_2 results in a reduction of free OH groups in PLA/TiO_2 bioplastics, as indicated in the FTIR result in figure 1.

3.3. Antimicrobial Study

The films' antimicrobial characteristics were tested on two types of model bacteria, gram-positive (S. aureus) and gram-negative (E. coli), over two distinct illumination conditions, which are under a UV-A (360 nm) irradiation and fluorescent lamp source. As shown in table 2, the antibacterial impact of the nanocomposite film grew progressively as the number of TiO_2 nanoparticles increased, with the maximum inhibition ratio obtained at a TiO_2 concentration of 5.0 wt%. The nanofilm's inhibitory zones for E. coli and S. aureus were 10.49 ± 0.22 and 12.61 ± 0.78 mm, respectively, with a TiO_2 concentration of 5.0 wt%. For E. coli and S. aureus, the inhibition zones of films containing 0.1 wt% TiO_2 were 6.83 ± 0.19 and 6.96 ± 0.62 mm, respectively. It is commonly acknowledged that TiO_2 and different TiO_2-based photocatalysts can only become active when exposed to light. Toniatto et al. discovered that following UV irradiation, which activated the TiO_2 nanoparticles, the bactericidal impact of PLA/TiO_2 nanofilm against S. aureus was high, and that the effect was controlled by the time in accordance with TiO_2 concentration [21]. Under UV irradiation, Fonseca et al. investigated the antibacterial impact of

PLA/TiO_2 nanocomposite films containing 8 wt% TiO_2 nanoparticles and observed that the killing effect on bacteria and fungi was 94.3% and 99.9%, respectively [22]. His findings indicated that the antibacterial activity of PLA/TiO_2 nanocomposites might be attributed to the TiO_2 nanoparticles, which is similar with He et al. [23]. The experimental results reveal that TiO_2 has substantial oxidizing capabilities under long-wavelength UV light (320–400 nm), resulting in microbial mortality. When the irradiated TiO_2 nanoparticles come into contact with the microbes, active species such as hydroxyl radicals, hydrogen peroxide, and superoxide anions are produced, that could passivate the microorganisms through the process of cell lysis, which affects the genome and other intracellular molecules. As a result, increased TiO_2 concentrations produce more ROS and have a significantly higher antibacterial rate as illustrated on figure 3. In their investigation, Li et al. proved that the illumination conditions influenced the bactericidal impact of the composite films [25, 26].

Table 2. E. coli and S. aureus inhibitory zones as a function of TiO_2 nanoparticle concentration in PLA with varied weight percentages.

Sample(s)	Inhibition zone (mm)					
	E.coli (ATCC 25922)			**S. aureus (ATCC 43300)**		
	Trial 1	**Trial 2**	**Trial 3**	**Trial 1**	**Trial 2**	**Trial 3**
1.0 wt% TiO_2/PLA	6.64	7.01	-	7.68	6.6	6.6
5.0 wt% TiO_2/PLA	10.66	10.11	10.71	12.61	11.82	13.39

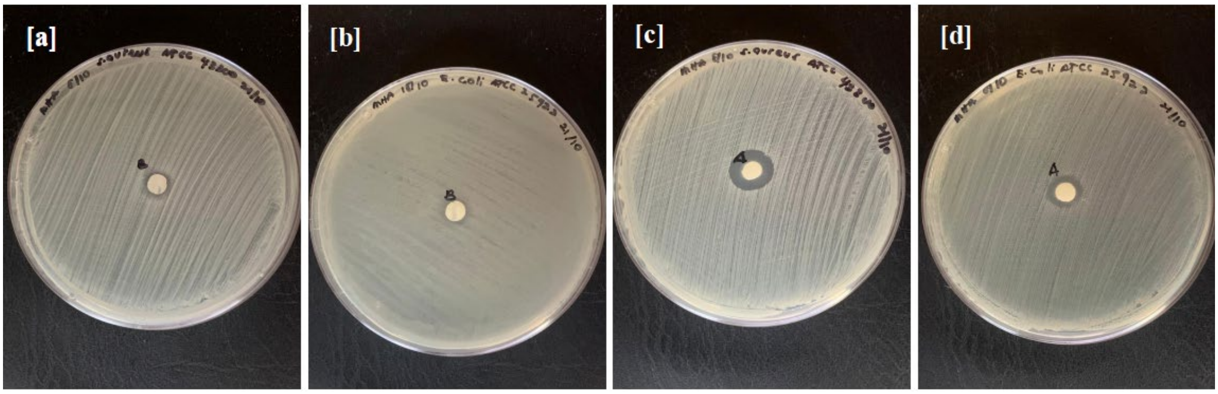

Figure 3. Study of antibacterial characteristics on two types of model bacteria, gram-positive (S. aureus) and gram-negative (E. coli); (a) 0.1 wt% PLA/TiO_2 tested with S. aureus, (b) 0.1 wt% PLA/TiO_2 tested with E. coli, (c) 5.0 wt% PLA/TiO_2 tested with S. aureus, (d) 5.0 wt% PLA/TiO_2 tested with E. coli.

The findings of this investigation indicate that TiO_2 nanoparticles are effective at inhibiting both Gram-negative and Gram-positive bacteria. The inhibitory zones created by the PLA/TiO_2 nanocomposite films for S. aureus, on the other hand, are greater than those formed for E. coli. Salarbashi et al. [26] found that following the bacterial inhibition test, the vitality of E. coli was greater than that of S. aureus as shown in figure 3, which validates our conclusion. They came to the conclusion that this was due to the different compositions of Gram-positive and Gram-negative bacteria's cell walls. Gram-negative bacteria's cell wall consisted of a thin peptidoglycan layer and an inner and outer membrane, while gram-positive bacteria's cell wall composed of a thick peptidoglycan layer with a thick wall thickness that effectively blocked TiO_2 penetration through into cell membrane. Because the structure was complicated, bacteria might be protected against a variety of chemical agents [27, 28].

Furthermore, the photocatalytic reaction of TiO_2 was accomplished by direct contact between test bacteria and free radicals. Hence, TiO_2 was completely stimulated and photocatalysis happened over its surface. Because of the physical swirling and mixing, TiO_2 nanoparticles easily aggregated in the PLA molecules, lowering the photocatalytic response. As a result, the nanofilm's inhibitory effect was successful.

4. Conclusion

Solution casting was used effectively in this investigation to generate PLA/TiO_2 films. The bioplastics' water absorption was shown to decrease as the TiO_2 concentration increased, with the lowest water saturation point of 17.39% reported at the greatest TiO_2 addition (5.0 wt%). This is due to the interaction of PLA with TiO_2, which lowers the number of free OH groups in the resultant bioplastics. As a result, there was less water absorption-related degradation, with improved preservation of tensile strength and stiffness and a larger elongation increase following water immersion. Moreover, UV-A irradiation increased antibacterial activity, with films with 5.0 wt% TiO_2 content exhibiting inhibitory zones of 10.49 ± 0.22 for E. coli and 12.61 ± 0.78 mm for S. aureus, respectively. The PLA/TiO_2 films with a TiO_2 concentration of 5.0 wt% demonstrated the best overall functioning. The nanofilm outperformed the pure PLA film in terms of performance. Future research is needed to determine the usefulness of the films as a packing material for regular food.

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