Laser-Assisted Preparation of Polycarbonate/TiO_2 Mesh and its Hydrophobicity Improvement Using Octadecylamine

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ABSTRACT— **The present research aims to combine rapid and modern laser drilling with loading octadecylamine (ODA) to produce a hydrophobic polycarbonate/TiO_2 mesh. The laser drilling first created hierarchical microholes on the polycarbonate/TiO_2 film using ablation process. The polycarbonate/TiO_2 mesh was then immersed in ODA solution. The morphological and structural investigation indicated that ODA shells cover the PC-TiO_2 mesh surface. X-ray photoelectron spectroscopy (XPS) was utilized for investigating the change in element/chemical state after laser drilling and ODA treatment. The wettability of the produced polycarbonate/TiO_2 mesh was changed from hydrophilic to hydrophobic, in a way that the water contact angle was changed from 45 to 122.4°.**

KEYWORDS: Hydrophobic, Laser drilling, Polycarbonate, TiO_2.

I. INTRODUCTION

In the last decade, polymeric meshes have been noticed due to their biocompatibility, mechanical properties and light weight [1]. They are widely utilized to develop materials for tissue engineering, drug delivery and filtration. They can also be employed for electronic devices preparation, electromagnetic interface shielding and designing electrodes for electrochemical applications [2]. These polymeric structures can be generated from different polymer nanocomposites [3]. Polymeric nanocomposites, including polymer matrix and nanofiller, are attracting attention due to their unique chemical properties and light weight. They have significant mechanical and physical stability due to the presence of large interfacial area between the polymer matrix and nanofillers. The presence of nonofillers such as carbon-based and semiconductor nanoparticles (NPs) can enhance the mechanical and physical properties of polymers. The presence of nanofillers leads to an increase in chemical activation of the polymer matrix due to their high surface area. Polymeric nanocomposites can be produced through different methods such as mechanical mixing, solution dispersion and the sol-gel method [4]-[7].

Polycarbonate (PC) is an attractive polymer with remarkable transparency in the visible spectral range. It is introduced as a great replacement for glasses in different applications. It is used as an accurate optical element [8,9]. It has shown significant properties such as high chemical resistance, good heat resistance, facile shapeable and water resistance [10]. However, PC has demonstrated poor physical properties, such as low hardness and photocorrosion resistance, which limit its applications. Therefore, it needs to be modified for improving or changing its properties, which can be realized by adding nanofillers [11]-[15].

Nanofillers can be organic or inorganic in nature, in which semiconductor nanomaterials such as SiO_2 and TiO_2 are the most common nanofillers for developing polymer properties. They are easily available or synthesized. Adding nanoparticles (NPs) can provide thermal and mechanical stability. Among these, TiO_2 NPs gained attention due to their non-toxicity, availability, corrosion resistance and cost-effectiveness properties. It is also chemically inert and possesses a high refractive index and high hardness. TiO_2 nanofiller affords unique optical, electrical and physicochemical features even at low concentrations, making polymer-TiO_2 nanostructures as a practical material for applications in solar cells, gas sensors, photocatalysis and drug delivery [16-18]. TiO_2 can be found in three different metastable phases of rutile, anatase, and brookite, which define its properties. For example, different phases of TiO_2 have different bandgap values; rutile phase has a bandgap of 3 eV, while anatase has 3.2 eV [19]. It possesses low free energy of surface and can improve the wettability behavior of the surface [20]. Degussa P25 TiO_2 has been a standard material, which is a biphasic TiO_2, including mixed anatase and rutile phases. These nanoparticles possess significant properties, including small particle size, large specific surface area and high crystallinity [21].

Surface wettability is an important behavior of polymer materials that has attracted a lot of attention in material research. Surface wettability is also well-known as a significant characteristic of materials for specifying their applications, which strongly depends on surface properties such as chemical compounds and surface morphology. Surfaces with different wettability degree lead to unique properties and applications such as corrosion resistance, self-cleaning, liquid separation, de-icing and non-fouling. Wettability degree is determined by measuring the contact angle (CA) between the wetting liquid and the solid substrate. The CA less than 90° is shows a hydrophilic surface, while a hydrophobic surface is introduced with a CA higher than 90° [22]. In comparison with hydrophilic surfaces, hydrophobic surfaces have attracted a lot of attention in basic research. They can be applied in particular application fields such as self-cleaning, antibacterial, preparation of waterproof and anti-fogging glass, anti-adhesion and oil/water separation. Different materials such as mesh, polymeric films and textiles can be employed to fabricate hydrophobic surfaces [23]-[26]. However, the number of hydrophilic surfaces is more than hydrophobic surfaces. A hydrophobic surface can be manufactured by using low-surface-energy materials or by creating a rough structure on the materials with low-surface energy. According to reports, there are different approaches for fabricating hydrophobic surfaces, such as anodic oxidation, electrodeposition, chemical vapor deposition, sol-gel, laser ablation and spin-coating [27]-[33]. Octadecylamine (ODA) possesses low surface energy due to its long alkyl chain and has been widely used to improve hydrophobicity [34, 35]. For instance, in 2020, Sun et al. produced a hydrophobic coating (ODA-polyphenol film) through one-step spraying co-deposition [36]. They reported that the ODA modification enhances the hydrophobicity of poly(tannic acid) and poly(pyrogallol). The ODA was also used for the treatment of nylon by Du et al in 2015 [34]. They immersed nylon fabric treated by poly(acrylic acid)/tetraethylorthosilicate at room temperature for 2 h. The reaction between amino groups of ODA and carboxyl groups of nylon enhanced the hydrophobicity of the nylon. Fig. 1 shows a schematic of ODA structure on the mesh substrate.

Recently, laser processing has been widely employed for modifying surface properties of a wide range of materials [37]. It can be used to modify the surface of polymers and change the surface energy and CA value [38]. Laser ablation can provide a controllable and cost-effective technique for fabricating stable micro/nano hierarchical structures on different materials and drill targets with narrow damaging effects for producing mesh. Laser beam drilling, a thermal energy machining process, is the most attractive technique for drilling wide range of materials such as ceramics, metals and polymers. It is introduced as a high-speed and accurate method for modifying materials with high efficiency [39-42]. Different types of lasers such as CO_2, Nd:YAG and fiber laser have been utilized for this purpose [42]-[44]. The laser beam drilling has been successfully used for preparing polymer meshes. For instance, Abdulwahab et al. created micro-holes on the PC and poly methyl methacrylic using CO_2 laser source [45]. They investigated the effect of laser power, exposure time and focal plane position on the holes. In another report, a femtosecond pulsed laser was used to from controlled micro-hole arrays on the PC sheet [46].

The laser beam drilling is based on the laser ablation mechanisms, which consists of two vaporization and melt expulsion processes. The surface electrons of the target are excited by focusing and hitting the laser beam, leading to generate heat. Light intensity and target thickness are effective in the absorption of light. The heat creates melting or vaporization of the target, forming a plasma plume. The melted or vaporized materials are removed by plasma expansion, making accurate holes. The scraped materials deposit around the holes. Therefore, it can be used to produce a mesh sheet [47, 48].

In this report, a hydrophobic polymeric mesh was provided through a simple and low-cost method of laser drilling followed by ODA coating. The TiO_2 NPs were firstly dispersed in the PC matrix by magnetic stirring and PC-TiO_2 nanocomposite films were acquired after drying at 40 °C. The laser drilling method was utilized to obtain PC-TiO_2 mesh. This process provided hierarchical microholes on the film. The ODA solution was then used to change the wettability of PC-TiO_2 mesh. The presence of ODA with shell-like morphology did not close the holes of the polymeric mesh and created a hydrophobic surface.

II. EXPERIMENTAL PROCEDURE

A. *Material and Instruments*

TiO_2 NPs with anatase and rutile phases were fabricated from the Degussa Co. and used as nanofiller. PC was also purchased from General Electric Company of America. The crystalline structure of PC-TiO_2 film was surveyed using X-ray diffraction (XRD, ITALSTRUCTURE) pattern. Wettability of PC and PC-TiO_2 nanocomposite film was examined by measuring water contact angle. The surface chemical features were identified via X-ray photoelectron spectroscopy (XPS, X250i), in which all the binding energy was calibrated by the characteristic carbon feature C1s = 284.7 eV. The surface changes in PC and PC-TiO_2 mesh after ODA treatment were determined by scanning electron microscopy (SEM, JEOL-JSM-840A) images.

B. *Preparation of PC-TiO_2 Nanocomposite Film*

To fabricate PC-TiO_2 nanocomposite film, 1 g of PC was dissolved in 30 ml of dichloromethane by magnetic stirring at ambient conditions. Different masses of TiO_2 NPs (0.01, 0.02, 0.03 and 0.05 g) were dispersed in 10 ml dichloromethane by sonication for 60 min. When the TiO_2 NPs suspension was stirred, the PC solution was slowly added to it, preventing the precipitation of TiO_2 NPs and penetrating PC into the spaces between the NPs. Afterwards, the prepared suspension was poured into a circular glass template with diameter of 7 cm and dried at 40 ºC for 6 h.

C. *Preparation of PC-TiO_2/ODA Mesh*

A fiber laser (RFL-P30Q) with maximal power of 30 W, wavelength of 1064 nm and frequency of 20 kHz was used to drill the PC-TiO_2 nanocomposite film. The holes were designed with a diameter of 160 µm and the edge-to-edge distances of 340 and 400 µm. A laser beam with power of 9 W, scanning speed of 3000 mm/s and 2 scanning loops was focused on the PC-TiO_2 surface and the polymeric nanocomposite was hacked line-by-line with scanning line distance of 0.01 mm. Finally, a regular arrangement of holes was obtained and the samples were named as PC-TiO_2-laser.

To improve hydrophobicity of PC-TiO_2 mesh, ODA ethanolic solutions with different weight percentages 0.01 wt.%, 0.02 wt.%, 0.03 wt.% and 0.05 wt.% were prepared. The obtained solution was then heated at temperature of 60° C and the PC-TiO_2 mesh was immersed in ODA solution for different times of 5, 10, 15, 30, 45, 60 and 90 min. At last, the PC-TiO_2 mesh impregnated with ODA was dried at ambient conditions for 2 h and named as PC-yTiO_2/ODA-x-laser (y and x are TiO_2 weight and immersing time, respectively). For comparison, PC-TiO_2 immersed in ODA was also prepared and labeled as PC-yTiO_2/ODA-x.

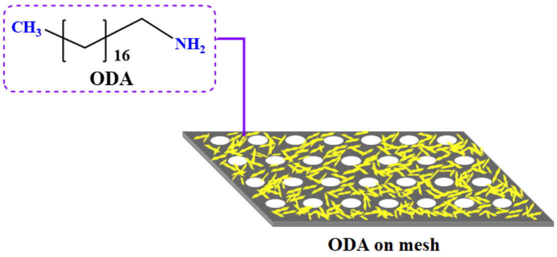


Fig. 1. The structure of ODA on the mesh substrate.

III. RESULTS AND DISCUSSIONS

As mentioned, PC-TiO_2 nanocomposite films with different weight percentages were prepared. Among these samples, preliminary observations revealed that the film containing 5 wt.% TiO_2 was brittle, while the 1 wt.% and 2 wt.% samples did not form a mesh structure under laser irradiation, and pores did not appear in some regions. This behavior can be attributed to the fact that, at low concentrations, the samples behave similarly to pure polycarbonate. Therefore, the sample including 0.03 g TiO_2 was selected as the best sample.

Figure 2 indicates the XRD patterns of PC-TiO_2, PC-0.03TiO_2/ODA-90 and PC-0.03TiO_2/ODA-90-laser nanocomposite films. The observed peak with the maximal intensity at about 17.1° reveals the amorphous nature of PC [49, 50]. The peaks located at 25.4°, 37.9°, 48.2°, 54.2°, 55.2° and 63° are due to the presence of anatase phase of TiO_2 (JCPDS 04-0477). Two peaks with low intensity at 27.5° and 69.1° are attributed to the rutile phase of TiO_2 (JCPDS 21-1276). It is clear that anatase is the main phase of TiO_2 NPs. The low intensity of TiO_2 peaks is related to the low concentration of TiO_2 NPs in the PC matrix. The XRD profile of PC-TiO_2/ODA is similar to PC-TiO_2 and a tiny shift can be observed in the PC peak (from 17.1° to 17.9°), suggesting no change in the PC-TiO_2 structure. The degree of sample crystallinity has declined in PC-TiO_2/ODA-laser XRD pattern, which is probably due to material ablation.

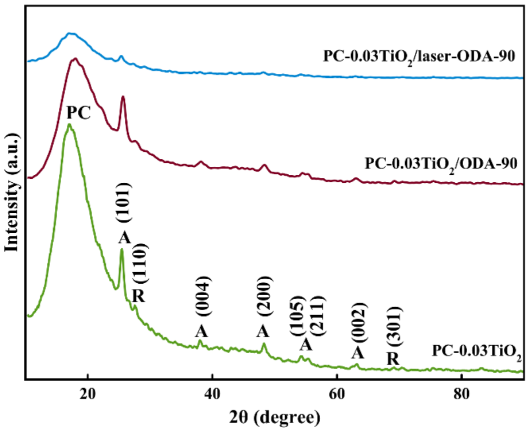


Fig. 2. XRD pattern of PC-0.03TiO_2, PC-0.03TiO_2/ODA-90 and PC-0.03TiO_2/ODA-90-laser.

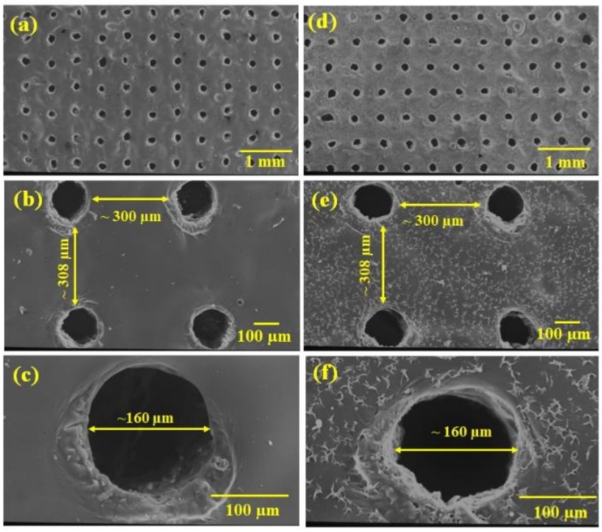
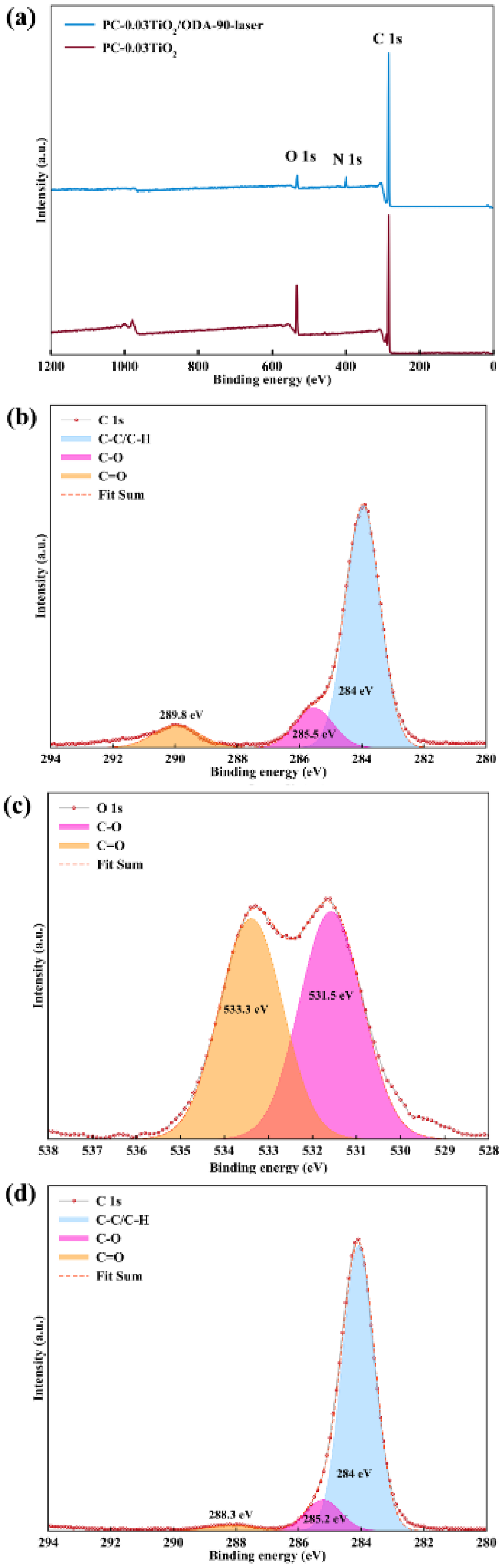


Fig. 3. SEM images of (a-c) PC-0.03TiO_2 mesh and (d-f) PC-0.03TiO_2/ODA-90-laser.

SEM images at different magnifications have been used to identify the morphology of samples. Fig. 3(a-c) indicates a regular array of holes on the flat PC-TiO2. A cover of ODA shells on the PC-TiO2 mesh can be observed in Fig. 3(d-f). It is clear that holes are not closed with ODA.



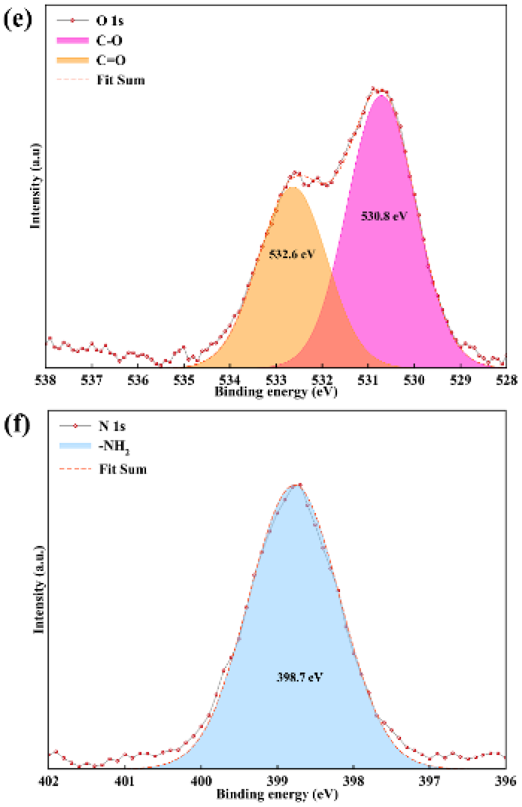


Fig. 4. (a) XPS spectra of PC-0.03TiO$_2$ and PC-0.03TiO$_2$/ODA-90-laser, high-resolution XPS spectrum of (b) C1s and (c) O1s of PC-0.03TiO$_2$, high-resolution XPS spectrum of (d) C1s, (e) O1s and (f) N1s of PC-0.03TiO$_2$/ODA-90-laser.

XPS analyses were employed to identify chemical bindings of PC-0.03TiO$_2$ and the PC-0.03TiO$_2$/ODA-90-laser. As depicted in Fig. 4(a), the survey XPS spectra reveal that the C1s and O1s, detected at 284 eV and 531 eV, are the main elements of PC. After loading ODA, the N1s has also appeared in survey XPS spectra of PC-0.03TiO$_2$/ODA-90-laser film, which appears at binding energy of 399 eV. Adding ODA and the laser drilling process introduces a reduction in the O1s peak. The high-resolution spectra of C1s and O1s are shown in Fig. 4(b-e), in which, the C1s consists of three Gaussian functions corresponded to the C-C/C-H (284 eV), C-O (285.5 eV) and C=O (289.8 eV). It is clear that the intensity of C-O and C=O groups are reduced after ODA treatment, providing hydrophobic surface. However, the main compound in both PC-0.03TiO$_2$ and PC-0.03TiO$_2$/ODA-90-laser is C-C/C-H bonds instead of carbon-oxygen bands. The O1s is also separated by two peaks at about 531 eV and 533 eV which are related to C-O and C=O groups [51]. As summarized in Table 1, the percentage of C=O is reduced after adding ODA and laser processing. As shown in Fig. 1(f), the N1s spectrum only includes a peak at 398.7 eV, assigned to the -NH_2 [52, 53].

Table 1. The percentages of different carbon and oxygen bonds.

Sample	C1s			O1s	
	C-C/C-H	C-O	C=O	C-O	C=O
PC-0.03TiO_2	76.75 %	13.76 %	9.49 %	50.81 %	49.19 %
PC-0.03TiO_2/ODA-90-laser	87.20 %	10.24 %	2.56 %	60.10 %	39.90 %

Table 2. Water contact angle values for nanocomposite films.

Sample	Water contact angle (degree)
PC-TiO_2	90
PC-TiO_2-laser	45.4
PC-TiO_2/ODA-laser-15	103.9
PC-TiO_2/ODA-laser-30	97.2
PC-TiO_2/ODA-laser-45	117.5
PC-TiO_2/ODA-laser-60	113.7
PC-TiO_2/ODA-laser-90	122.4

Wettability of the nanocomposite films was identified through measurement of the water contact angle (WCA) by the sessile-drop method and compared with each other in environmental conditions. The average contact angle of five droplets was measured for each nanocomposite film and reported in Table 2. Before laser drilling, the WCA value of the prepared PC-TiO_2 film was 90°. The WCA of the PC-TiO_2 film after laser drilling was obtained as 45.4°, indicating hydrophilic behavior of PC-TiO_2-laser sample. The chemical composition of the surface and surface roughness of solid samples strongly affected on the WCA values. It is obtained that the initial WCA of PC-TiO_2 was reduced after laser drilling, which is due to surface roughness enhancement. The presence of ODA on the laser-drilled PC-TiO_2 can also improve hydrophobicity by increasing the WCA value. The hydrophobicity of the mesh was enhanced with increasing immersing time and the best result was obtained for PC-TiO_2 mesh immersed in ODA for 90 min. In fact, the ODA creates long hydrocarbon chains on the surface of laser-drilled PC-TiO_2 and enhances the hydrophobicity [54].

IV. CONCLUSION

In the present study, laser drilling was used as a fast and simple method to create a polymeric mesh with uniform holes. To prepare polymer nanocomposite film, polycarbonate was used as a matrix for dispersing anatase and rutile phases of TiO_2 NPs through a simple physical mixing method. According to SEM images, a regular array of holes was created on the PC-TiO_2 film without any crack. The presence of ODA shells was also confirmed through SEM images and XPS analyses. The laser drilling provided a rough surface and adding ODA cover created long hydrocarbon chains on the surface of PC-TiO_2 mesh, changing the hydrophilic surface to hydrophobic. The as-prepared hydrophobic mesh can be used as a filter to remove nonpolar pollutants such as oil from water.

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