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Abstract

The strong integration of TiO_2 nanoparticles (NPs) into the membrane surface to impart antibacterial functionality is often challenging due to the agglomeration of nanoparticles and often requires a labour-intensive, multi-step process. . To tackle this challenge, we developed a practical, simple, and scalable method to firmly adhere TiO_2 NPs on a commercial polyether sulfone (PES) membrane using a one-step dip coating process harnessing polydopamine as an adhesive agent. The process was optimized with regards to the reaction time. A range of analytical methods were utilized for a thorough examination of the surface chemistry and the structural properties of the produced membranes. The experimental results revealed that the modification at the optimum reaction time of 30 min enhanced the hydrophilicity of the PES membranes as confirmed by the decrease of contact angle. As a result, the modified membranes exhibited a significant improvement in membrane permeability with 12 times higher water permeation flux (962 LMH for pDA-f-TiO_2-PES30) compared to the pristine PES membranes (79.9 LMH). The static adsorption of BSA on the surface of membranes was reduced from (60 $\mu g/cm^2$ for pristine PES to 21 $\mu g/cm^2$ for pDA-f-TiO_2-PES120).

The modified PES membranes displayed a higher flux recovery ratio (97%) and fouling reversibility (98.62%) than pristine PES membrane (37.63%). Also, the coated PES membranes bestowed a good antibacterial property relative to the pristine one. The membranes showed better physical and chemical stability compared to unmodified PES membranes. Thus, the proposed coating method proved to be a practical and effective TiO_2 immobilization approach for improving membrane lifespan and its fouling resistance.

Keywords: Ultrafiltration membranes, Polydopamine, Titanium dioxide, Anti-fouling property, Anti-bacterial

1. Introduction

Water scarcity is one of the most important issues worldwide. Currently, rapid urbanization, industrialization, population growth, and climate disruption exacerbate the problem of water scarcity. The worldwide demand for fresh, clean, and abundant water has increased significantly over that from the past. Therefore, sustainable technologies are required urgently to overcome this global challenge [1, 2]. Among the adapted strategies, membrane-based separation technologies have been extensively developed to extend and renew water resources by producing a high-quality water due to their exceptional properties such as low operating cost, lower energy consumption, high separation efficiency, reliability, and simplicity, which make them more efficient than conventional water treatment technologies [3-5]. As part of membrane separation technology, ultrafiltration (UF) membranes with higher membrane flux, higher anti-fouling property and excellent stability are urgently required as UF membranes are extensively used for wastewater treatment [6-8], industrial wastewater treatment [9], reverse osmosis (RO) pre-treatment and other food separation applications, chemical and biochemical industries [10-12].

In comparison to other commercialised polymers, polyethersulfone (PES) is a thermosetting polymer used for several ultrafiltration applications due to its excellent chemical properties, high thermal stability, outstanding mechanical strength, and film-forming properties [13-20]. However, the strongly hydrophobic character of PES results in membranes which are easily fouled by adsorption of pollutants during separation and purification processes, which ultimately results in decreasing water permeation flux, then affects membranes' separation performance [13]. Membranes with anti-fouling surfaces have considerable potential to improve the water permeation and performance of this polymer [21].

Until now, different modifiers through different methods have been introduced to improve membrane's flux or enhance its antifouling properties. To improve the separation performance of membranes, several strategies have been introduced. For instance, the incorporation of hydrophilic and antimicrobial nanoparticles (NPs) into membranes has received significant interest due to facile processing and predictable separation performance [5, 22]. These nanoparticles include silver nanoparticles (AgNPs), gold nanoparticles (AuNPs), carbon nanotubes (CNTs), graphene oxide (GO), zinc oxide (ZnO), zeolite and titanium dioxide (TiO_2) [23-28]. Titanium dioxide (TiO_2) has been identified as an effective antifouling nanomaterial for organic foulants in wastewater treatment processes due to its outstanding properties such as high hydrophilicity, antibacterial properties, stability, non-toxicity, photocatalytic activity and low cost [29]. However, the uniform dispersion and firmly attaching/binding of TiO_2 NPs on the membrane surface are a crucial factor for constructing membranes with stable and long-lasting high performance. Tightly bonding TiO_2 NPs on the surface of membranes can result in a high surface hydrophilicity and reduced resistance to water permeation, whilst uniformly dispersed TiO_2 NPs can help decrease the likelihood of blocking membrane pores. A common shortcoming in existing studies relates to the agglomeration of TiO_2 NPs, resulting in less enhancement in membrane performance and anti-fouling properties. In many cases, the NPs are enclosed by the polymer matrix, rendering them barely useful in modifying the surface hydrophilicity. Thus, covalent cross-linking and self-assembly approaches are usually adopted in thin film hybrid membranes to try and address the above-mentioned problems. However, these approaches rarely modify the surface of PES membrane effectively due to the weak binding force between PES membranes and TiO_2 NPs.

While TiO_2-based modifications offer numerous advantages, some challenges persist, such as the potential detachment of TiO_2 from the membrane surface over prolonged operation and the possible reduction in membrane mechanical strength due to excessive nanoparticle loading[30-32]. These limitations highlight the need for further research into optimizing nanoparticle loading, improving long-term stability, and exploring alternative deposition methods that ensure strong nanoparticle adhesion while maintaining membrane integrity. By addressing these research gaps, this study contributes to the ongoing development of high-performance, antifouling UF membranes, advancing sustainable water treatment technologies for future applications. Therefore, it is required to find a scalable method to attach TiO_2 NPs on the top surface of the membrane surface.

Dopamine (DA), as a mussel-inspired bio-glue, is currently applied as a promising agent for membrane surface modification owing to its outstanding properties including self-polymerization on any substrate, higher anchoring capability and the possibility for further functionalization [33-36]. It is also regarded as extremely versatile platform to improve the adhesion of the nanoparticles and other polymeric materials on the membrane surface. DA undergoes the oxidative self-polymerization process in an alkaline environment to generate a surface-adhering polydopamine (pDA) film for different organic and inorganic materials [34]. pDA remains reactive due to the existence of amine and hydroxyl functional groups, which makes it suitable for further functionalization and modification for various applications [37]. A variety of membranes including reverse osmosis (RO), nanofiltration (NF), and ultrafiltration (UF) membranes have been modified with pDA coating through hydrogen bonding, π-π stacking, the action of van der Waals forces and chelation [38]. This allows the grafting of different polymeric functional groups with their antifouling moieties such as zwitterionic polymers, hyper-branched polyglycerol, and nanomaterials [39-44]. Kasemset et al.[45] have studied the effect of deposition pDA on the performance of polyamide (PA) reverse osmosis membranes for applications of oil/water. The results showed that membrane flux decreased with increasing dopamine solution concentration and polydopamine deposition time. Jiang et al[46]. have discovered the fundamental surface characteristics of pDA-coated PVDF membranes. The pDA could stick robustly on the surface of PVDF membrane and effectively decrease the water contact angle. Ou et al.[47] have deposited successfully a uniform TiO_2 films on different polymer substrates (PE and PTFE) pre-modified by pDA coating and investigated the biocompatibility of modified materials.

However, to the best of our knowledge, limited studies have focused on one-step binding approach for TiO_2 NPs onto PES membrane surface using dopamine, without requiring silane coupling agents or other cross-linkers. This study distinguishes itself from previous research work in the following ways: 1) simplified process: unlike conventional methods that involve additional chemical coupling agents, our approach leverages the strong adhesion properties of pDA to immobilize TiO_2 NPs directly onto the membrane surface, eliminating unnecessary complexity 2) cost-effectiveness: by avoiding chemical coupling agents, the proposed method reduces material costs, making it a more economical alternative 3) environmental sustainability: the elimination of additional chemicals not only minimizes process costs but also reduces environmental impact, contributing to a more sustainable and resource-efficient methodology 4) scalability: unlike previous studies that relied on deposition techniques involving dead-end cells [48], which are complex and costly, our method utilizes a facile in situ dip-coating oxidation polymerization approach, enhancing practicality for large-scale applications.

This study proposes a simple and scalable approach for modifying PES UF membranes using a one-step, in situ dip-coating oxidation polymerization of DA to immobilize TiO_2 NPs onto the membrane surface robustly. Different coating times (15, 30, 60, and 120 minutes) were investigated. The modified membranes were characterized using various surface characterization techniques, and their performance was evaluated in terms of permeation flux, rejection, antifouling properties, anti-adhesion capability, and antibacterial efficacy. Furthermore, this study assessed the physical and chemical stability of the modified membranes, an aspect rarely reported in previous literature.

2. Materials and Methods

2.1. Materials

Commercial PES membranes were purchased from Sterlitech Corporation (USA). TiO_2 NPs (Powder, anatase type, 99.8% metals basis, 25 nm size) and DA hydrochloride were obtained from Sigma Aldrich, Australia. Bovine serum albumin (BSA) was used as foulant models during UF experiments and were purchased from (Sigma Aldrich, Australia). NaOH (purity ≥ 99, Merck KGaA Company, Germany). Deionized water and ultrapure water were used in the experiments.

2.2. Modification of PES UF membranes

PES membranes were immersed in ultrapure water overnight prior to the experiment while TiO_2 NPs (0.05 wt%) were first dispersed in a solution of Tris-HCl buffer (15 mM, pH 8.5) and then sonicated to prevent glomeration ultrasonication. Subsequently, DA (2 wt%) was added to the 100 mL of the prepared solution to obtain the DA-TiO_2 hybrid. PES membranes were immediately dip-coated into DA-TiO_2 solution and mixed by shaking (as explained schematically in **Scheme 1 (SI)**). The reaction proceeded at room temperature (RT) for different intervals; 15, 30, 60, and 120 min, denoted as pDA-f-TiO_2-PES15, pDA-f-TiO_2-PES30, pDA-f-TiO_2-PES60 and pDA-f-TiO_2-PES120, respectively. The selected coating times were chosen based on preliminary experiments and literature findings to systematically investigate the effect of deposition duration on membrane performance. The rationale for choosing these specific time intervals is as follows: **15 and 30 minutes:** To examine the early-stage adhesion of polydopamine (pDA) and assess whether a sufficiently uniform and functional layer is formed for effective TiO_2 nanoparticle immobilization. **60 and 120 minutes:** To explore the effect of prolonged deposition, which can lead to thicker coatings that may enhance hydrophilicity and antifouling properties but could also increase resistance to water permeation. Afterward, the coated PES membrane was rinsed with deionized water at least four times and placed in the oven for 10 minutes. All the modified PES membranes were kept in deionized water (DI) for 24 hours before use.

2.3. Membrane characterization

2.3.1. Membrane Surface Characterization

The surface structure of the modified and pristine PES membranes was characterized by X-ray photoelectron spectroscopy using an AXIS Nova spectrometer (Kratos Analytical Inc., Manchester, UK) with a monochromated Al Kα source at a power of 180 W (15 kV × 12 mA) and a hemispherical analyzer operating in the fixed analyzer transmission mode. Moreover, the chemical compositions of membranes were collected using Fourier transform infrared spectra (FTIR, PerkinElmer, USA). Additionally, the surface morphology and mapping of membrane was examined by scanning electron microscopy-electron dispersive energy (SEM-EDS, FEI Magellan 400 FEG SEM, Hillsboro, USA). Finally, the hydrophilicity of membrane surface was determined using a contact angle measurement (CA) (OCA15EC, Dataphysics, Germany). Results were collected from the average of at least ten values at different areas for each membrane sample.

2.3.2. Permeation test

The water flux of the pristine PES and hybrid PES membranes was determined using a HP4750 dead-end ultrafiltration cell (Sterlitech Corporation, USA) as shown in Figure S1. Circular pieces of membrane samples with 25 mm diameter and 14.6 cm^2 effective membrane area were cut from the coated membranes and stored in double deionized water (DDI) for 60 min before use. Then, the filtration cell was filled with DDI (to volume 300 ml) and then attached to 5 L water reservoir. During the filtration test, the membranes are first compacted at pressure 0.15 MPa for 60 min to get a steady filtration. Once the steady state was achieved, the water flux (J_{w1}) was measured at 0.1 MPa pressure by measuring the permeate weight with the time using an electronic balance connected with a computer. The data from the electronic balance was connected to a computer using LabVIEW software. Three membrane samples were tested to calculate the averages of water flux values.

2.3.3. Fouling measurements

Dynamic fouling mode was utilized to study the anti-fouling performance of the coated PES membranes [49]. The pristine and modified PES membranes are first compacted with deionized water at 0.15 MPa to get a stabilize flux. After 60 min filtration, the water flux was measured at pressure at 0.1 MPa. Afterwards, BSA protein solution (1000 ppm) was tested as foulant to accelerate the fouling onset for an hour and pressurized to 0.1 MPa. The flux (J_{BSA}) was recorded at the last 10 minutes of the run. The concentrations of BSA in the permeate and feed sides were spectroscopically determined at 280 nm using Varian Cary 100 Bio UV-VIS spectrophotometer. The rejection ratio (%R) of the membrane was calculated by the following equation [50]:

$$R(\%) = \left(1 - \frac{C_p}{C_f}\right) \times 100 \qquad (1)$$

Where R(%) represented the rejection coefficient of the membrane, C_f and C_p (mg/mL) are the concentrations of feed solution and permeate stream, respectively.

The concentration of sodium alginate in the solution was calculated based on the calibration curve prepared in advance. Then, the fouled membranes underwent in cleaning process. In physical cleaning, the fouled membranes were rinsed with deionized water for 30 min and the flux was measured from the Labview software at 100 MPa. In chemical cleaning, the fouled membrane was rinsed with solution of NaOH (2g/l) for 20 minutes, following by rinsing with deionized water three times to remove the NaOH solution, then the flux was measured from the Labview software at 100 Mpa. For each cleaning step (physical and chemical), the flux was measured (J_{w2}) at 100 Mpa. Three sequential fouling cycles were applied for each membrane.

To determine membrane performance, flux recovery ratio (% FRR) was calculated using the following equation [51, 52]:

$$FRR\ (\%) = \frac{J_{w2}}{J_{w1}} \times 100 \qquad (2)$$

Where the (J_{w2}) and (J_{w1}) are the flux after cleaning process and before fouling process.

To study the membrane fouling in detail, total fouling resistance (R_t), reversible fouling resistance (R_r) and irreversible fouling resistance (R_{ir}) were determined using the following equations respectively[53].

$$R_t = \frac{J_{W1} - J_p}{J_{W1}} \times 100 \qquad (3)$$

$$R_r = \frac{J_{W2} - J_p}{J_{W1}} \times 100 \qquad (4)$$

$$R_{ir} = \frac{J_{W1} - J_{W2}}{J_{W1}} \times 100 \qquad (5)$$

Where J_p is the flux of BSA foulant model and R_t is the sum of R_r and R_{ir}.

2.3.4. Static protein adsorption

The adsorption capacity of the membrane surface was determined using protein adsorption experiments. BSA was used as model foulant. Circular pieces of membranes with diameter of 25 mm were cut from the coated membranes and washed with double deionized water several times before use. Then, the circular pieces of membranes were placed in filtration cell by exposing the filtration surface of membrane to BSA solution (500ppm). The filtration cell was then incubated in a shaker with 150 rpm speed at 25°C for 24 hrs. The concentration changes of BSA were measured and the adsorption capacity was calculated based on the concentration changes of BSA using equation 6 [49, 54]:

$$Q = \frac{(C_a - C_b)V}{A} \quad (6)$$

Where Q (μg/cm^2) represents the adsorbed protein, C_a (g/L) is the initial concentration of BSA, C_b (g/L) is the final concentration of BSA, A (cm^2) is the area of membrane sample, and V (L) represents the volume of the BSA solution.

The concentration of BSA macromolecules was measured using an ultraviolet-visible spectroscopy (mini-1240, Shimadzu, Japan) at absorbance 280 nm. The obtained results were the average of the protein adsorption on four samples of membrane. After static protein fouling, the flux of the fouled membrane was recorded, and the flux was recorded again after cleaning cycle. The flux recovery ratio after static protein was calculated by recording the flux before and after fouling tests.

2.3.5. Antibacterial test

The antibacterial properties of the membrane were analyzed by the plate counting method [55]. Briefly, 1.8 × 2.5 cm^2 rectangular membrane samples were prepared and sterilized with 80% ethanol prior to the test. The sterilized membrane samples were immersed into broth LB medium cultured with Escherichia. coli BL21 (OD600 = 0.1) and incubated at 37°C and 250 rpm shaking (Thermoline Scientific Equipment Pty Ltd., Sydney, Australia) for 24 hours. The membrane samples were collected after 12 hours of incubation and washed with 7.5 mL phosphate-buffered saline (PBS, pH 7.4) for 20 min to remove unattached cells. The washed membranes were then transferred into 7.5 mL fresh PBS and sonicated (Power Sonic 405, Thermoline Scientific Equipment Pty Ltd., Sydney, Australia) for another 20 minutes to extract the attached cells. The optical densities of the extraction solutions were recorded with a spectrometer (DR 5000™ UV-Vis Spectrophotometer, Hach, Australia). The extraction solutions were diluted with filter-sterilized PBS to normalized OD600 of 0.0001 and spread on LB agar plates. The plates were incubated overnight at 37°C and the number of colonies was counted.

2.3.5. Stability evaluation

To evaluate the coating stability, two modes of stability have been done: the first mode is physical stability and the second one is chemical stability. Concerning physical stability, the unmodified and modified PES membranes were immersed into DDI water bottle. Then, the samples with the water bottle were placed in into a water path under continuous shaking at a speed of 100 rpm at 30 °C for 15 days.

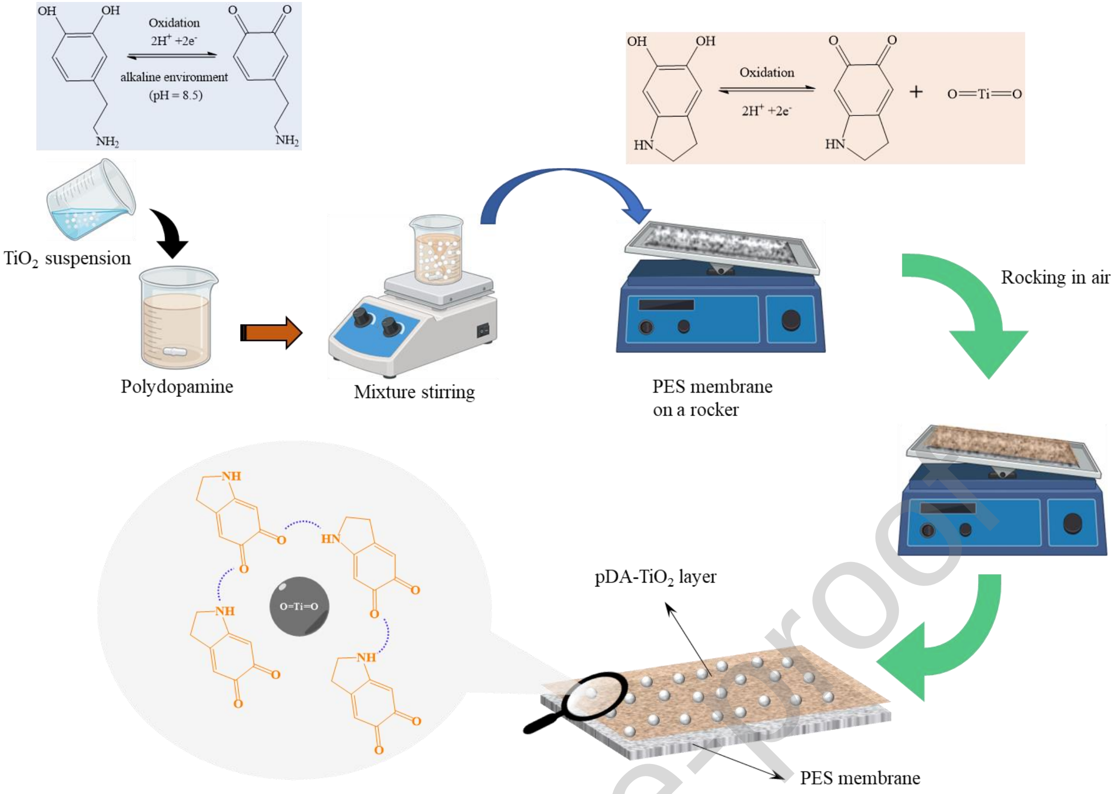
The chemical stability was done by immersing the pristine and modified PES membranes in different membrane washing agent (NaOCl solution concentration = 500 ppm, pH=9.5, sodium hydroxide solution NaOH at pH=13 and hydrochloric acid solution HCl at pH=2) respectively.

Each chemical stability test lasted for 15 days. The membranes after physical and chemical washing were characterised by CA and membrane performance and the results are tabulated.

3. Results and discussion

3.1. Mechanism of in-situ polymerization process

The procedure for in-situ polymerization of pDA-f-TiO_2 onto the PES membrane surface is depicted in **Scheme 1 (SI file).** As shown in Scheme 2, the TiO_2 NPs were immobilized/coated well on the surface of the PES membrane using polydopamine as an adhesive agent. The reaction has occurred at room temperature (25 °C). The coating time was varied from 15 min to 120 min. **Scheme 2** shows the mechanism to link pDA and TiO_2 NPs without using any cross-linkers, silane coupling agent or using any chemical agent. the oxidation of catechol in dopamine to O-quinone (dopamine quinone) by alkaline pH-induced oxidation (pH=8.5). Its intramolecular cyclization, oxidation to dopaminechrome, formation of 5,6-dihydroxyindole (DHI) and further oxidation to 5,6-indolequinone (IDQ). It is notable that the mixture of dopamine, quinone and indole may co-exist in solution after the first steps since the oxidation and cyclization may be complete. The mixtures of these units undergo various pathways to form the PDA, which is later connect with metal oxide (TiO_2) NPs to enhance the adhesion of this NPs onto PES membrane surface.



Scheme 2. The mechanism of linking of polydopamine (pDA) and titanium oxide nanoparticles TiO_2 NPs on the surface of PES UF membranes. Three steps were observed in the scheme as presented in different colors.

3.2. Membrane morphology

Fig. 1 shows the membrane photographs and SEM top surface morphologies of membranes before and after the coating of pDA-f-TiO_2 layer on the surface of PES UF membrane. As observed in **Fig.1A,** during the process of oxidative self-polymerization of DA-f-TiO_2, the color of the modified PES membrane surface was changed from white clear (transparent) to light grey after 15 and 30 min coating time and then change further to dark grey color after prolonged coating times of 60 and 120 min. This color change indicates successful grafting of the pDA-f-TiO_2 layer on the surface of the PES membranes.

It also evident from **Fig.1B** that the pore size of PES membrane was decreased progressively with increasing modification time due to the polymerization of dopamine, which forms a coating on the membrane surface. The decrease in pore size as modification time increases is significant because it directly impacts the membrane's permeation flux, which refers to the rate at which fluid passes through the membrane. As the pore size decreases, the resistance to fluid flow increases, which could reduce the permeation flux. This relationship between pore size and permeation flux is important to understand for optimizing membrane performance in various filtration applications.

Upon higher magnification, **Fig. 1C** reveals the presence of TiO_2 NPs visibly anchored on the membrane surface at 15 and 30 min coating times. However, at longer coating times (60 and 120 min), the TiO_2 NPs are no longer distinctly visible, possibly due to increased coverage and densification of the pDA-f-TiO_2 layer. The white circles in Fig.1C highlight TiO_2 NPs within the PES membrane surface, supporting the hypothesis that prolonged coating times lead to an increased amount of pDA-f-TiO_2 on the membrane surface. The stability and formation of the pDA-f-TiO_2 layer on PES membranes can be attributed to non-covalent interactions, including hydrogen bonding, π-π interactions, and electrostatic interactions. The observed reduction in flux for membranes with longer coating times (60 and 120 minutes) correlates with the measured decrease in pore size, validating that a thicker coating layer leads to higher resistance to water transport.

To further verify the presence of TiO_2 NPs on the surface of PES membranes, SEM images, EDX scan images and EDX mapping were taken for pristine PES and modified PES membranes and the results are shown **in Fig. 2.** As is seen in **Fig. 2A**, pristine PES membrane shows no trace of TiO_2 NPs. However, the modified PES membranes showed that there are several particles are anchored on the surface after modification. These images indicate the successful coating of pDA-f-TiO_2 layer on the surface of PES membranes This result is in agreement with the reported literature[56]. The coating of pDA-f-TiO_2 layer onto PES membranes surfaces was further verified by EDX-Ti-mapping analysis and the results are also shown in **Fig. 2B and 2C**. The Ti peak was not detected in the EDX spectrum of the pristine PES membrane; however, the Ti peak was detected for the modified PES membranes at **0.45 keV** as shown in **Figure 3.3b-d Middle row.** The coated PES membranes also showed the main peaks at 0.27 keV and 0.52 keV, which corresponded to **C** and **O** peaks. The EDX results confirmed the successful coating of pDA-f-TiO_2 layer on the top of PES membranes.

The EDX image could determine the elements that present in the membranes and usually be used to analyse the distribution of a chosen element. The results of EDX mapping are shown in the **Fig. 2C.** The EDS Ti Maps of the modified membranes clearly show where the Ti rich particles are located. The areas with a high concentration of Ti in the EDX map well match the locations of particles in the SEM image. Furthermore, the green dots in the Ti EDX map of the pristine membrane represented only the noise from the spectrum background. The SEM images, EDX scan images, and EDX mapping indicate that the whole PES membranes were fully modified since the modification layer of pDA-f-TiO_2 is distributed uniformly on the inner pores of membranes. The EDX scan images signifies that the whole PES membrane was successfully modified since the modification layer of dopamine is evenly distributed on the inner pores.

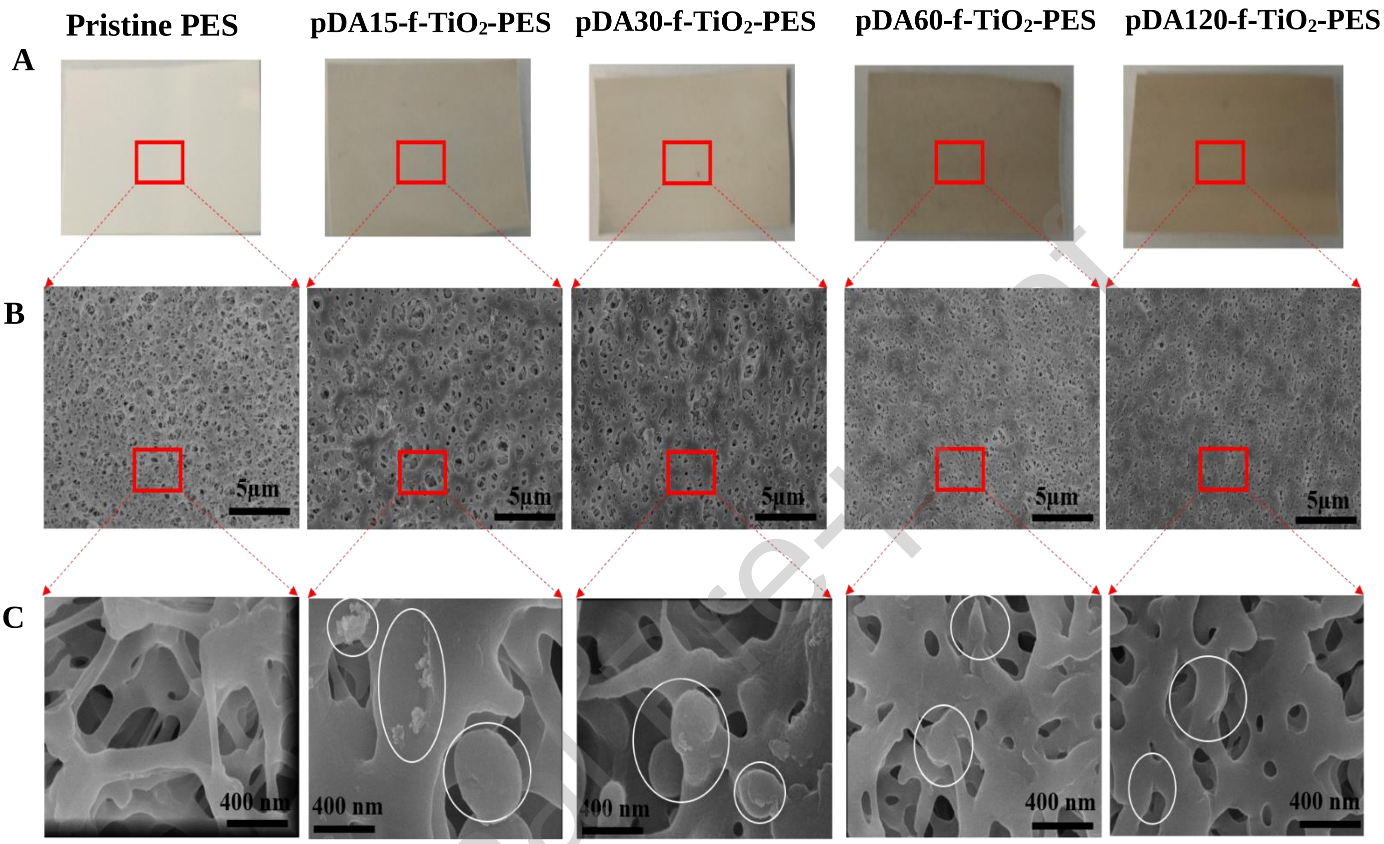


Fig. 1. Photographs and SEM morphology images of top surface PES UF membranes after coating with layer of pDA-f-TiO_2. (A) Photographs of membrane before and after modification with layer of pDA-f-TiO_2. (B,C) SEM top surface morphologies of membranes before and after modification with layer of pDA-f-TiO_2. All the SEM images were taken using Magellan.

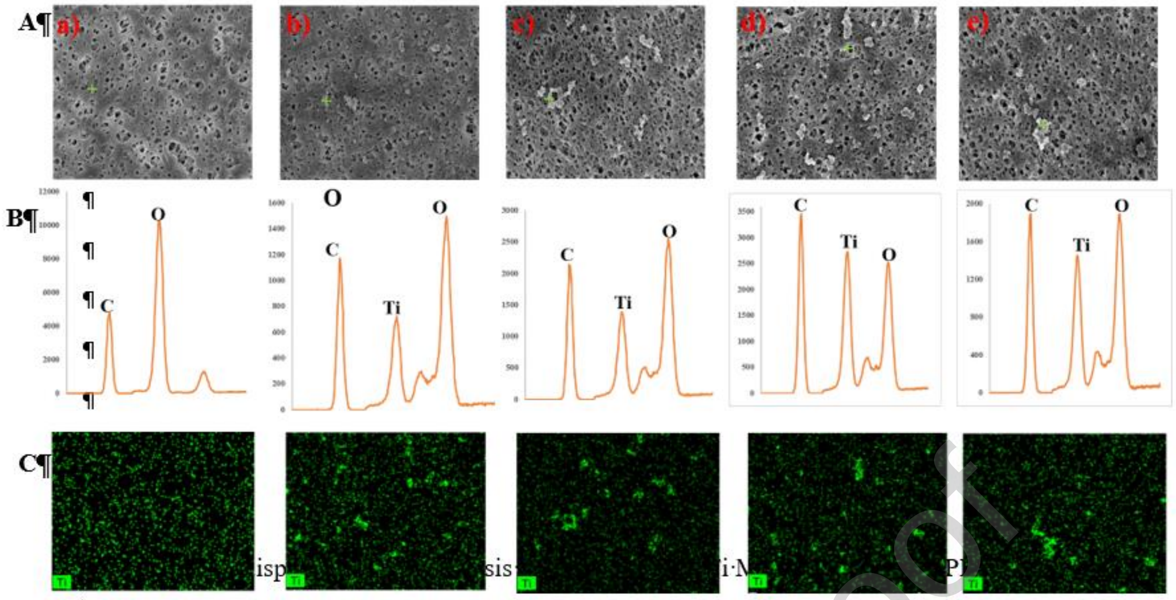


Fig. 2. Energy dispersive X-ray analysis (EDX) and EDX Ti Maps of a) pristine PES b) pDA-f-TiO_2-PES15 c) pDA-f-TiO_2-PES30 d) pDA-f-TiO_2-PES60 e) pDA-f-TiO_2-PES120 UF membranes. (A) SEM top surface morphology of pristine and modified PES membranes. (B) EDX spectrum of pristine and modified PES membranes to confirm the presence of TiO_2 on PES surface. (C) EDX Ti maps.

3.3. Chemical compositions of membrane surface

To check the chemical structure of pristine PES and modified PES membranes, FTIR and XPS were undertaken, and the results are presented. The FTIR spectrums of the unmodified and modified PES membranes are presented in **Fig. 3.** The absorption bands for each membrane, which correspond to the PES structure, are observed at 1244 cm^{-1} (aromatic ring stretching vibrations), 1488 cm^{-1} (C-C bond stretching), and 1580 cm^{-1} (benzene ring stretching) respectively [57]. The thickness layer coating of pDA-f-TiO_2 is smaller (in nanometer) than the detecting-depth of FTIR measurement (10-14 µm), so the functional groups of pDA and TiO_2 layer did not have high intensity in FTIR spectrum. This result in line with the previously reported in the literature [58].

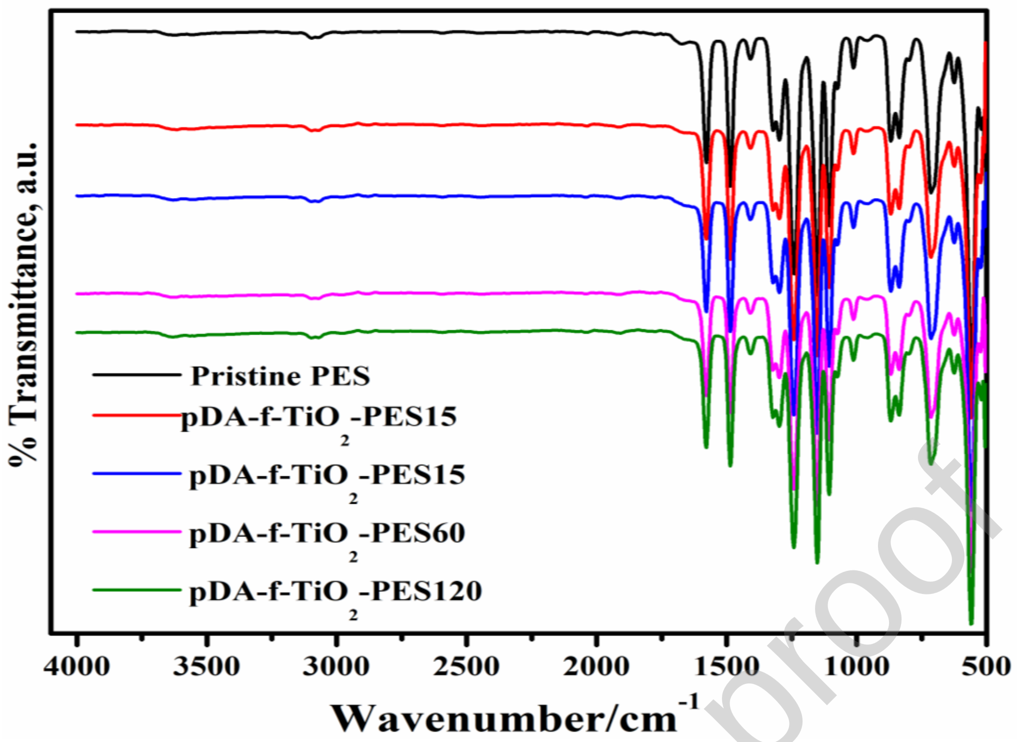


Fig. 3. FTIR spectra for pristine and modified PES UF membranes.

The XPS analysis was conducted to check the elemental chemical composition of pristine and modified PES membranes and the results are shown in **Table S1 and Fig. 4** where the pristine PES membrane shows the presence of C1S (81.04), followed by the elements of O1s (13.95) and S2p (3.23). There are no N1s and Ti2p observed in pristine PES membranes. However, the PES/pDA-f-TiO_2 membranes display the peaks of O 1S and N 1S and Ti2p, which implies the readily coating of pDA-f-TiO_2 layer on the surface of PES membranes. The content of nitrogen (N1s) element was increased after modification from (2.21 to 4.50), indicating that the modified PES membranes were indeed covered with pDA. Whilst the content of titanium (Ti2p) was decreased from (0.44 to 0.2), indicating that the membranes are fully covered with pDA layer after 120 min coating. All this evidence confirmed the successful coating of PES membranes with pDA-f-TiO_2 layer. The O-CO group was found to be the most abundant in the modified PES membranes, indicating the presence of pDA and TiO_2 within the membrane surface.

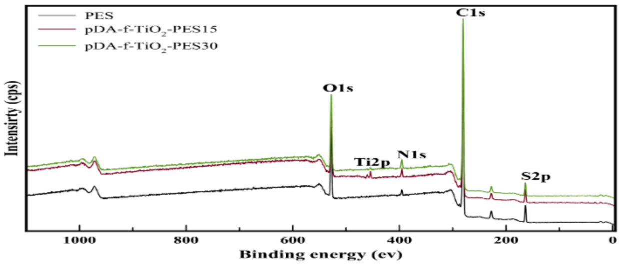


Fig. 4. X-ray photoelectron spectroscopy (XPS) wide spectra of pristine and pDA-f-TiO_2 modified PES UF membranes at 15 and 30 coating times.

3.4. Membrane hydrophilicity, permeating properties, and static adsorption of membranes

The membrane hydrophilicity is a vital aspect to improve the antifouling performance of membrane [59].The hydrophilicity of pristine and modified PES UF membranes were determined using contact angle (CA) measurement as illustrated in **Fig. 5.** The results revealed that the contact angle for unmodified PES membranes was 61° due to the intrinsic hydrophilic characteristic of the PES membrane. After coating the membrane with a layer of pDA-f-TiO_2, the modified PES membranes exhibited significant decrement in CA (25.9º, 24.8º,20.6º and 13.1 º after 15 min, 30 min, 60 min and 120 min dip-coating) respectively. The significant reduction in contact angle was due to an increase in surface hydrophilicity of PES membrane due to the presence of hydrophilic polydopamine and TiO_2 NPs. Longer coating times resulted in more pDA-f-TiO_2 layer deposited on the surface of PES membranes, which is consequently led to increasing the hydrophilicity of membranes. The combination of polydopamine and TiO_2 can provide a good membrane performance. These results are similar to the data reported in other studies [46, 47, 60-62]. It can be observed Contact angle measurements confirmed that hydrophilicity improved with increased coating time up to 30 minutes, beyond which further increases in coating time resulted in diminishing returns. This trend can be explained by:

- **pDA Layer Growth:** Within the first 30 minutes, polydopamine self-polymerizes and forms a uniform hydrophilic layer, reducing the contact angle significantly and enhancing water permeability.
- **Excessive Coating (≥60 min):** Longer deposition times lead to the formation of a denser, more compact layer, which may restrict water flow through the membrane pores, causing a decline in flux despite increased hydrophilicity. The performance peak at 30 minutes

suggests an optimal balance between enhanced surface hydrophilicity and minimal resistance to water transport.

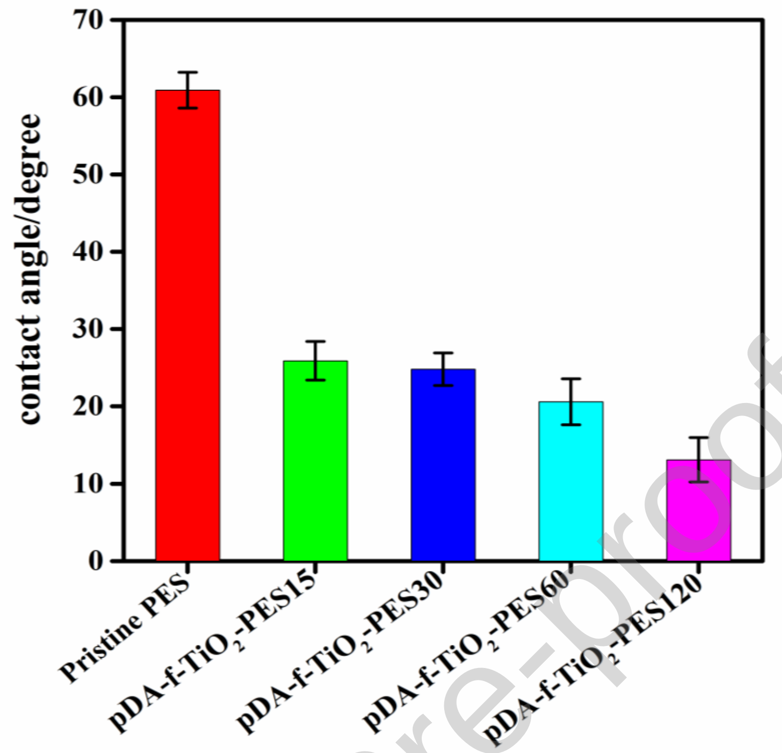


Fig. 5. Contact angle measurements of pristine and modified PES UF membranes. The bar height is the average of five contact angle measurements for each membrane and the error bars represent ± one standard deviation.

The results of water flux (J_{w1}) and rejection of BSA for pristine and hybrid PES UF membranes are illustrated in **Fig. 6.** It can be seen that the water flux (J_{w1}) for unmodified PES membrane was 79.9 LMH. After coating the PES with hydrophilic layer of pDA-f-TiO$_2$, the water flux was increased remarkably from 79.9 LMH for pristine PES to 962 LMH for pDA-f-TiO$_2$-PES30 This increase in water flux is attributed to the incorporation of hydrophilic groups, such as hydroxyl (OH) and amine groups (NH), which likely enhance the membrane's wettability and permeability [63, 64].

A further increase in coating the membranes with pDA-f-TiO$_2$ layer for 60 and 120 min, the water flux was reduced moderately to 584.6 LMH for pDA-f-TiO$_2$-PES60 and 478.4 LMH for pDA-f-TiO$_2$-PES120. However, both values were still higher than the pristine PES membrane’s flux. The reduction in flux at longer coating times is attributed to the complete coverage of the membrane surface with the pDA-f-TiO$_2$ layer, which results in a decrease in the pore size and an increase in transport resistance. This aligns with the findings in the literature [65].

Fig. 6 also shows the results of % BSA rejection. BSA was used as a macromolecule organic , and its rejection in UF membranes is influenced by factors beyond just size exclusion. The hydrodynamic radius of BSA is ~7 nm[66], which means it wouldn't typically be rejected through size exclusion alone. However, other mechanisms, such as oligomerization, adsorption, or electrostatic interactions, could contribute to its rejection in the modified membranes[67]. It is evident from the results that the modified PES membranes showed a higher % retention rate of **BSA (96% for M4),** compared to unmodified PES membranes (81%). This increased rejection due to the reduction in pore size resulting from the increased coating time of the pDA-f-TiO2 layer, which improves the ability of the membrane to adsorb or interact with the BSA, facilitating its rejection.

The enhancement of **hydrophilicity** (increasing the interaction with water) and the reduction in **pore size** due to the coating, combined with **BSA adsorption**, were the two main factors contributing to the increase in BSA rejection.

Interestingly, there is an optimized pDA-f-TiO_2 modification time (degree) for obtaining the best membrane performance with regard to pure water flux. During pDA-f-TiO_2 modification, the pure water flux initially increases as the coating time increases, reaching its peak at 962 LMH for the membrane modified for 30 minutes. This increase is likely due to improved hydrophilicity, a reduction in water permeation resistance, and possibly a larger effective pore size at this stage.

After the 30-minute mark, as the coating time increases ($\geqslant$ 60 minutes), the water flux decreases moderately. This can be attributed to the increase in hydraulic resistance caused by the thicker pDA-f-TiO_2 layer, which reduces the effective pore size and increases resistance to water permeation. This behavior aligns with findings in similar studies[68], such as the one using atomic layer deposition (ALD) for modifying PVDF membranes, where a similar initial increase in water flux is observed, followed by a decline as the coating thickness increases[68]. Based on these results, the 30-minute coating time appears to be the most suitable, balancing both enhanced water flux and BSA rejection.

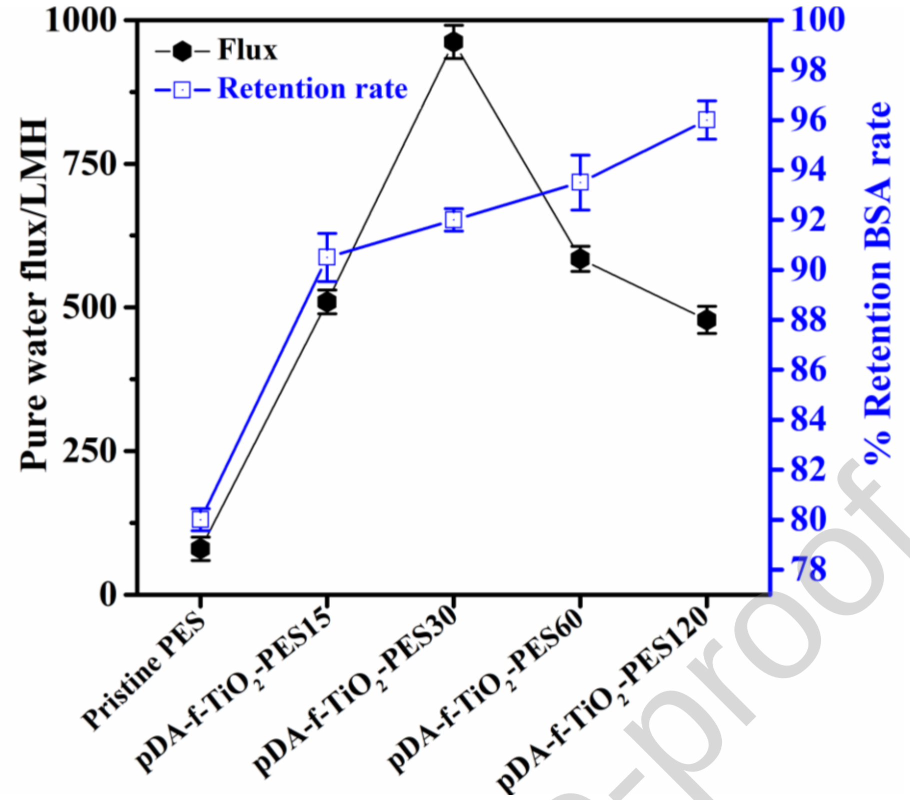


Fig. 6. Pure water flux and % BSA Rejection rate of pristine PES and modified PES UF membranes. The flux reached a maximum point at 962 LMH for 30 minutes coating then reduced significantly to 584.6 LMH and 478.4 LMH for 60 and 120 minutes coating, respectively. The bar height (+/-) represents the standard deviations of five measurements of flux and rejection were taken for each membrane sample.

The flux recovery ratio (FRR) is a key parameter for assessing membrane reusability and antifouling performance, in which higher % FRR reflects improved antifouling performance. **Fig.7** shows the results of %FRR of unmodified and modified PES membranes. The observed FRR across multiple cleaning cycles highlights the significant antifouling benefits of membrane surface modification. The modified PES UF membranes exhibited a much higher FRR (97.1% after 120 minutes of coating) compared to the unmodified membranes (42.1%), indicating superior resistance to fouling. This improvement is primarily attributed to the enhanced hydrophilicity of the coated membrane surface, as evidenced by the contact angle measurements (Fig. 5).

A higher FRR suggests that the modified membranes require less intensive cleaning to restore their original permeability, leading to several practical benefits. Reduced fouling and easier cleaning cycles help maintain membrane integrity over time, extending its lifespan and delaying the need for replacement. Improved cleaning efficiency minimizes the frequency and intensity of chemical cleaning, lowering operational costs associated with cleaning agents and labor. A consistently high FRR ensures stable membrane performance across multiple filtration cycles, which is essential for industrial applications such as wastewater treatment and water purification. Additionally, lower consumption of cleaning chemicals reduces chemical waste discharge, making the process more environmentally sustainable.

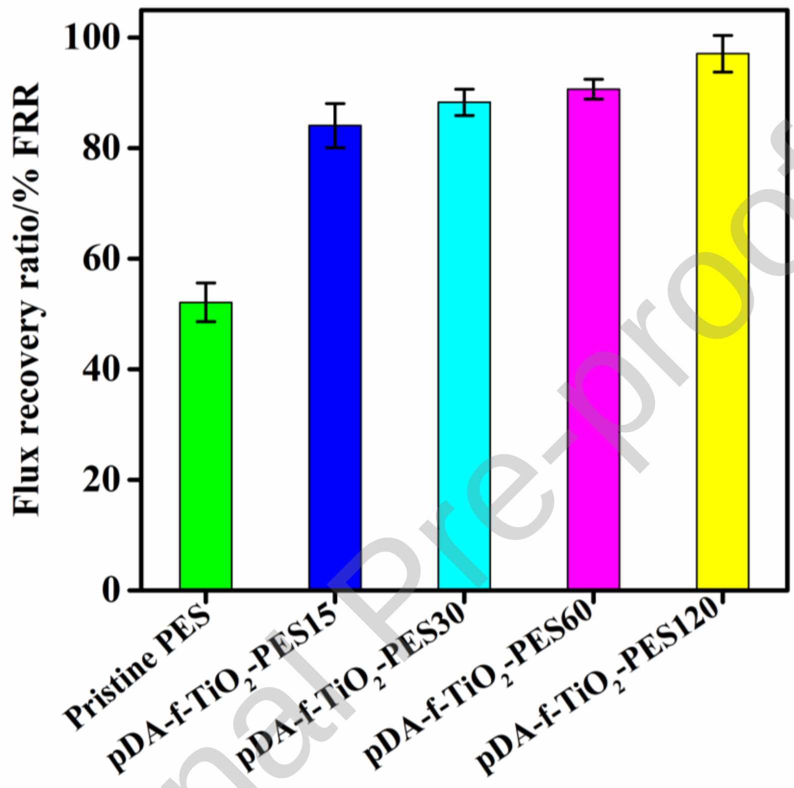


Fig. 7. % Flux recovery ratio (% FRR) of PES and modified PES UF membranes. The bar height is the average of five %FRR measurements, and the error bars represent +/- one standard deviation.

The amount of adsorbed BSA macromolecules was evaluated in static protein (BSA) adsorption test and the result is shown schematically in **Fig. 8**. It was clearly observed the adsorption amount of BSA is related to the hydrophilicity of membrane and to the morphology. PES membrane has higher BSA adsorbed amount due to the hydrophobic nature of PES polymer. In hydrophobic surfaces, the affinity between the membrane surface and protein is weakened, which allow the protein to easily adhere on the membrane surface and dominate the boundary layer. However, the modified PES membranes have less amount of BSA adsorbed (21 μg/cm^2 after 120 min of coating) than pristine PES (60 μg/cm^2). The significant decrement in BSA adsorption amount indicates that the modified PES membranes are more hydrophilic.

Hydrophilic surfaces can create a tightly bounded water layer, which represents as physical barrier to inhibit the adhesion of BSA molecules on membrane surfaces [69-72].

The flux recovery ratio (% FRR) was calculated again after BSA adsorption and the results are shown in **Fig. 8**. It can be noted that the modified PES membranes showed higher % FRR than pristine PES membrane counterpart. The results of % FRR after protein adhesion was roughly similar to the %FRR after cleaning cycles, indicating that the PES membranes are hydrophilic.

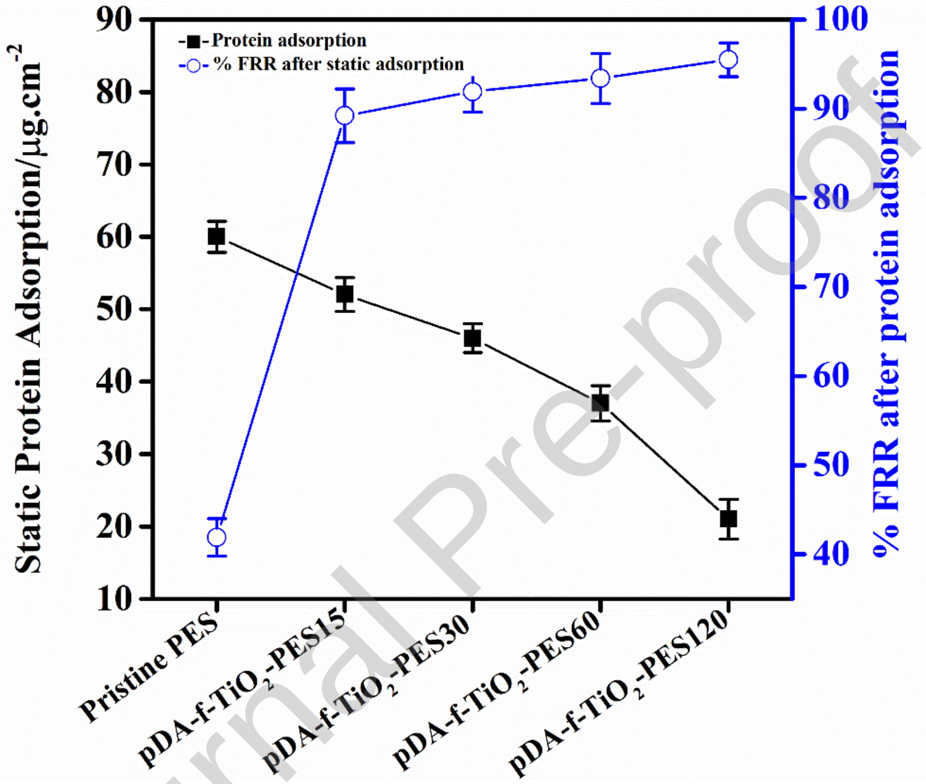


Fig. 8. Static protein adsorption and % FRR after protein adsorption of pristine PES and modified PES membranes. 1 g/l of BSA solution was used in this study as foulant model. The bar height is the average of five measurements, and the error bars represent +/- one standard deviation.

3.5. Anti-fouling performance, and fouling resistance analysis of membranes

The membrane fouling was quantitatively studied, in terms of total fouling resistance (R_t), which is caused primarily by the adsorption of protein on the membrane surface or inside the pores. The total fouling resistance is consisted of two fouling resistances that are reversible fouling resistance (R_r) and irreversible fouling resistance (R_{ir}). R_r occurs due to the loose attachment of foulants on the membrane surface, while R_{ir} arises due to the adsorption of foulants.

As seen in **Table 1,** the discrepancies in the results of R_t were not remarkable for all PES membranes. However, the variations in the R_r and R_{ir} were pronounced. The hybrid PES membranes showed a significant increase in the R_r ratio and gradual decrease in the R_{ir} ratio as as the pDA-f-TiO_2 coating time increased. In contrast, the pristine PES membranes showed the highest Rir ratio and the lowest Rr ratio. These results indicate that the pDA-f-TiO_2 layer enhanced the antifouling properties of PES membranes. The presence of polydopamine and TiO_2 improved hydrophilicity, reducing the adhesion of organic foulants on the membrane surface[73, 74].

Table 1. Fouling resistance for PES and modified PES UF membranes.

Membrane Samples	**%R_t**	**%R_r**	**%R_{ir}**	**%(R_r/R_t)**	**%(R_{ir}/R_t)**
Pristine PES	95.61±2.31	36.17±1.92	59.44±1.88	37.83	62.16
pDA-f-TiO_2-PES15	98.94±1.99	69.23±1.44	29.71±1.65	69.97	30.02
pDA-f-TiO_2-PES30	99.39±1.31	78.62±1.29	20.77±1.73	79.10	20.89
pDA-f-TiO_2-PES60	98.95±1.54	84.62±1.34	14.33±1.55	85.51	14.48
pDA-f-TiO_2-PES120	98.64±1.66	97.28±1.53	1.36±1.89	98.62	1.39

3.6. Anti-bacterial performance of the modified membranes

The antibacterial properties of pristine PES and hybrid PES membranes were evaluated, with the results presented in **Table 2** and **Fig.9**. Bacterial attachment to the membranes surface occurs through several mechanisms including electrostatic interaction and hydrophobic interaction [75]. It is well-established that bacterial attachment on a surface can occur via a layer of adsorbed protein; therefore, surfaces that resist protein adsorption should also exhibit reduced bacterial attachment.

In this study, pristine PES, pDA-f-TiO_2-PES15, and pDA-f-TiO_2-PES120—were selected for antibacterial testing, and the data are summarized in Table 2. Compared to the pristine PES membranes, the pDA-f-TiO_2-PES120 membranes demonstrated significant antibacterial activity, with the total number of recovered viable E. coli reduced to approximately 34% of that on the PES membranes (100%), corresponding to an E. coli inactivation rate of 66%.

The primary mechanism behind this bactericidal effect is the presence of TiO_2 NPs and the amphoteric nature of pDA on the membrane surface. TiO_2 NPs generate reactive oxygen species (ROS) such as hydrogen peroxide (H_2O_2), hydroxyl radicals (•OH), and superoxide anions (O_2^-), which exhibit potent oxidative properties. These ROS attack bacterial cell walls and membranes, inducing lipid peroxidation, protein oxidation, and DNA damage, ultimately leading to bacterial cell death [76, 77]. Specifically, •OH generated by TiO_2 interact with the lipids in bacterial membranes, disrupting their structural integrity and permeability. Additionally, H_2O_2 diffuses across the bacterial membrane and interacts with intracellular components, further accelerating bacterial inactivation[78-84].

Beyond the ROS-based mechanism, the amphoteric nature of pDA contributes to antibacterial activity. The primary and secondary amine groups in pDA carry a positive charge, allowing electrostatic interactions with the negatively charged bacterial cell membranes. This interaction leads to membrane destabilization and increased permeability, further impairing bacterial viability [85]. The synergistic effect of TiO_2-generated ROS and the hydrophilic amine groups of pDA enhances bacterial inactivation, preventing biofouling and improving membrane performance [58]. These findings suggest that integrating TiO_2 and pDA on PES membranes significantly enhances antibacterial properties through a dual-action mechanism—ROS-mediated oxidative stress and electrostatic membrane disruption—making them promising candidates for biofouling-resistant filtration applications.

Table 2. The total no of viable cells of pristine and modified PES membranes.

Sample	**PES**	**pDA-f-TiO_2-PES15**	**pDA-f-TiO_2-PES120**
Number of colonies	289±0.32	156±0.44	99±0.77
Normalized viable cells (E. coli)	100%	53%	34%

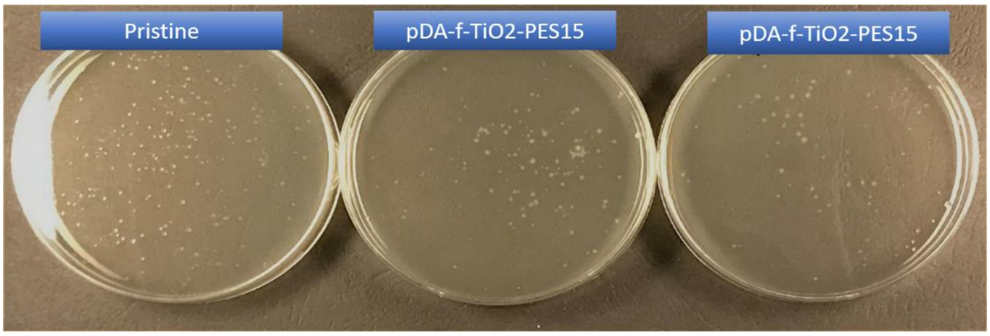


Fig. 9. Photographs (snip chat) showing the bacterial culture plates of E. coli of pristine and modified PES UF membranes.

3.7. Stability Evaluation of the modified membranes

To evaluate the durability of the membrane under long-term operation, a stability test was conducted in two parts: physical stability and chemical stability. The physical stability test involved immersing pristine and modified PES membranes in deionized (DDI) water. The membrane samples, placed in bottles, were subjected to continuous shaking at 100 rpm in a water bath at 30 °C for 15 days.

For chemical stability, the membranes were immersed in different membrane-cleaning agents, including NaOCl solution (500 ppm, pH = 9.5), sodium hydroxide solution (pH = 13), and hydrochloric acid solution (pH = 2). Each chemical stability test lasted for 15 days. After both physical and chemical treatments, the membranes were characterized by contact angle (CA) measurements and membrane performance analysis, with the results presented in the Electronic Supplementary Information **(S2-S7).**

Regarding the physical stability, the results of contact angle, pure water flux, % BSA rejection, % FRR and protein adsorption of pristine and modified PES membranes did not change significantly in comparison with the membranes before washing with water. This suggests that the hydrophilic coated layer did not peel off during the immersion in water for 15 days. These results indicate that the coated pDA-TiO_2 layer is tightly embedded on the PES membrane surfaces. See **Tables (S2-S4).**

After immersion, the membranes were analyzed for changes in **contact angle, pure water flux, BSA rejection, FRR, and protein adsorption** (Tables S2–S4). The results showed that the **contact angles of the pristine and modified PES membranes remained stable**, confirming the strong adhesion of the pDA-TiO_2 layer to the membrane surface. Similarly, **water flux values remained consistent**, indicating no significant structural changes due to water exposure.

Regarding **BSA rejection performance (S3 table)**, the pristine PES membrane exhibited a slight decrease from **80.0% to 79.9%** after immersion, which is within experimental uncertainty. The modified membranes showed **marginal reductions in BSA rejection** after 15 days, with the highest rejection observed for **pDA-f-TiO_2-PES120 (96.0% before and 95.5% after immersion)**. The **minor decline in BSA rejection across all modified membranes suggests that the membrane structure remained stable**, and the functionalized TiO_2 nanoparticles were firmly bound to the membrane surface. These results confirm that prolonged exposure to water does not compromise the protein-rejection capabilities of the membranes.

For chemical stability, the results of contact angle, water flux, and BSA rejection are shown in Tables S5–S8. The modified PES membranes exhibited excellent stability under acidic conditions (HCl), as evidenced by the minimal changes in contact angle and water permeation flux. This stability can be attributed to the inherent resistance of polydopamine (pDA) in acidic, neutral, and weakly basic environments [86, 87].

However, for sodium hydroxide (NaOH), the water flux of the modified PES membranes was increased with **prolonged** washing time. The **increas**e indicates that the coated layers **were** not completely stable in a strong alkaline agent, **as** polydopamine disintegrates in a strongly alkaline environment. After immersing in NaOH solution for 15 days, the flux of all modified PES membranes increased by approximately 1.5 times compared to their initial flux, suggesting partial degradation of the coating layer.

Similarly**, when washed** with NaOCl, the water flux of the modified PES membranes significantly increased, almost 3 times higher than the initial flux. **This suggests that** polydopamine undergoes reaction with NaOCl solution, producing a complex mixture of products [60]. **Possible interactions occurred** between the pDA-f-TiO_2 layer with the original membrane material in hypochlorite**-rich environment**, which may have contributed to membrane structural damage or degradation, leading to increased permeation flux.

Despite these changes in water flux, the contact angle of the modified membranes did not show significant discrepancy after immersion in NaOH or NaOCl, indicating that the membranes retained their hydrophilic characteristics even after chemical exposure.

Regarding BSA rejection (Table S8), the results show that all modified PES membranes retained high protein rejection efficiency after acidic treatment. However, after prolonged exposure to NaOH and NaOCl, the BSA rejection slightly declined due to potential degradation of the coating layer and increased pore size. The loss in rejection efficiency was more pronounced after exposure to NaOCl, correlating with the observed increase in water flux.

3.8. **Environmental Impact of TiO_2 Nanoparticle Leaching**

A critical concern during long-term membrane operation is the potential leaching of TiO_2 NPs, which may pose environmental risks. If TiO_2 NPs detach from the membrane surface, they can enter treated water streams, potentially impacting aquatic ecosystems and end users. Studies have shown that excessive exposure to TiO_2 nanoparticles can lead to oxidative stress in aquatic organisms due to the generation of reactive oxygen species (ROS) such as H_2O_2, HO•, and O_2^-. These ROS can cause cellular damage in microorganisms, algae, and higher organisms, disrupting aquatic food chains. Additionally, TiO_2 NPs can adsorb and transport contaminants, affecting water quality.

Long-term monitoring of TiO_2 release in operational conditions is necessary to assess the extent of leaching, which can be achieved through various techniques that incorporate in-line approach such as nanoparticle tracking analysis or off-line approaches like Inductively Coupled Plasma Mass Spectrometry (ICP-MS)[88]. In any case, to mitigate potential environmental risks, it is important to ensure the strong adhesion of TiO_2 NPs to the membrane surface through stable polymeric binding, such as the one proposed in this study

3.9. Comparison with other studies

The modified PES membranes featuring a hydrophilic layer of pDA-TiO_2, as investigated in this study are compared with some previously published hybrid UF membranes functionalised with TiO_2 NPs and the results are presented in **Table 3**. As we can see, A noteworthy observation is the scarcity of studies, addressing the stability and antibacterial properties associated with binding TiO_2 particles onto the surface of polymeric membranes.

In this work, both the stability and the antibacterial effects of the modified PES membranes are thoroughly discussed. The results show that the modified PES membrane displays exceptional stability under acidic, salty, and physical stress conditions. However, it is noteworthy that the as-prepared membrane exhibits susceptibility in strong alkaline environments. This vulnerability is mainly attributed to the stability of the polydopamine coating, which remains very strong in acidic, neutral, and weak basic solutions but undergoes disintegration in robustly alkaline environments. This finding emphasizes the importance of understanding the environmental conditions in which the modified membrane can effectively function and highlights the need for further considerations in applications involving strong alkaline environments.

Furthermore, some studies have showed that the hybrid membranes functionalised through a coating process may exhibit a lower water permeation flux compared to the pristine polymer membranes. This discrepancy is usually ascribed to potential blocking of membrane pores by the coating layer. However, our results contradict this trend, as we observed that the water permeation flux in the modified PES membranes was even higher than that in the virgin PES membranes after a 2 hrs coating. This suggests a unique and effective modification process in our study that enhances rather than impedes water permeability.

Table 3: Comparison with some relevant hybrid UF membranes previously published

Membrane	Method	Flux (LMH) Polymer/hybrid	Rejection (%)	%FRR	Antibacterial test	Stability	References
pDA-KH550-TiO_2/PVDF	Crosslinking then dip-coating of TiO_2	0/650	98.86%, 99.34%, 99.13%, and 99.52%, SDS/petroleum ether/H_2O emulsion, SDS/n-hexadecane/H_2O emulsion, SDS/1,3,5-trimethylbenzene/ H_2O emulsion, and SDS/diesel oil /H_2O	-	-	Higher stability results	[56]
TiO_2/PVDF	Sol gel coating	100/148	98.77% of RB5 dye color	81%	-	-	[89]
pDA/Psf	Blending	285/428	90% for BSA	80% for 1 cycle, 72% for 8 cycles	-	-	[30]
pDA-TiO_2/PES	Self-assembly	22.5	84% for BSA	32%	-	-	[90]
TiO_2/PVDF	Spin coating	1700	96% for toluene/water emulsion	-	-	-	[91]
TiO_2/PES	Low temperature	4707/1080	76%	93%	-	-	[92]

	hydrothermal coating						
TiO_2/PVDF	Phase inversion	76.99/392.81	99% for SDS/MB	91.35 %	-	-	[93]
TiO_2/pDA/Psf	Grafting coating	280/160	90% for BSA pollutants	80%	-	-	[94]
pDA-TiO_2/PES in this study	**Dip coating**	**79.9/962**	**97% for BSA**	**97.1% for 3 cycles**	**53% no of** Normalized viable cells (E. coli) compared to 100% for pristine PES	**Higher Stability in acidic And alkali conditions**	**This study**

4. Conclusions

In this study, we introduced a versatile and facile method to modify PES membranes by directly attaching TiO2 NPs firmly via a one-pot reaction using in-situ dip coating of a hydrophilic pDA-f-TiO_2 layer. This modification significantly reduced the membrane pore size, as evidenced by SEM analysis, and enhanced membrane selectivity. The coating improved the hydrophilicity and permeated flux of the membranes across different coating intervals (15, 30, 60, and 120 min). Additionally, the modification effectively diminished the adhesion of organic macromolecules, such as BSA, on the membrane surface, resulting in higher antifouling performance and stronger antibacterial properties compared to pristine PES membranes. Stability tests confirmed that the coated layer remained stable after prolonged use, demonstrating the long-term viability of this coating process. Overall, this modification technique offers a cost-effective and scalable solution that can be easily implemented in industrial applications.

Further work should focus on scaling up the in-situ dip coating method for industrial applications. It would be essential to explore the uniformity and reproducibility of the coating process when applied to larger-scale membrane production. Additionally, pilot-scale testing is recommended to assess membrane performance in real-world industrial settings, such as water treatment or wastewater filtration systems. These tests will help determine the practicality, efficiency, and economic feasibility of implementing this modification at an industrial level.

While the coated layer showed good stability under typical conditions (physical or chemical conditions), further studies should examine its performance under extreme operating conditions, such as high temperatures, high pressures, and strong alkaline or acidic environments. Understanding the long-term durability of the modified membranes in such conditions is critical for their broad industrial adoption.

One limitation of this work to take into consideration is the instability of the coating layer in highly alkaline environments. The pDA-f-TiO_2 coating may degrade or lose its functionality under such harsh conditions, restricting its potential application in industries that deal with strong alkalis (e.g., chemical processing and heavy-duty wastewater treatment). Thus, future work should focus on improving the chemical stability of the coating, possibly by introducing crosslinking agents or modifying the coating composition to improve resistance to harsh chemical environments.

Conflicts of interest

Authors have no conflicts of interest to declare.

Author Contributions

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

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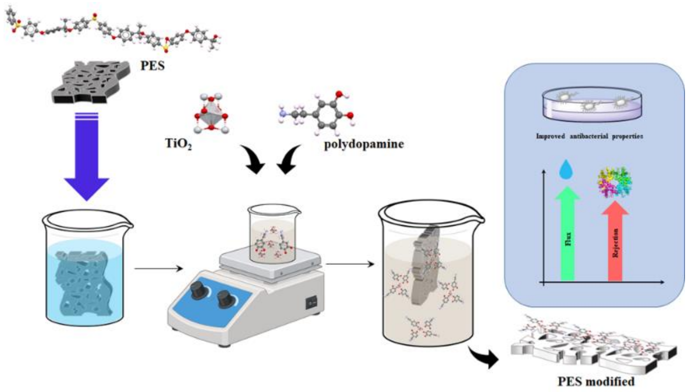
Graphical abstract



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Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Highlights

- PDA strongly fixed TiO_2 NPs onto the surface of the PES UF membrane
- Hydrophilicity, BSA rejection, and flux recovery ratio were improved
- Modified membrane had exceptional chemical and physical stability
- The modified PES membranes exhibited good antibacterial properties