



# Physicochemical characteristic of regenerated cellulose/N-doped TiO<sub>2</sub> nanocomposite membrane fabricated from recycled newspaper with photocatalytic activity under UV and visible light irradiation

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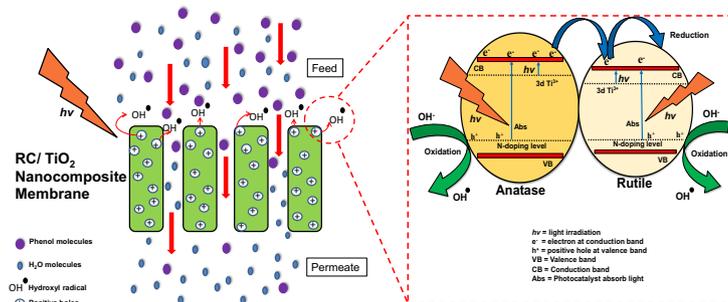
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## HIGHLIGHTS

- Fabrication of novel green photocatalytic membrane.
- The utilization of recycled newspaper as the cellulose source.
- Conversion of dense to porous membrane structure by the addition TiO<sub>2</sub> nanorods.
- RC/TiO<sub>2</sub>-0.5 shows highest catalytic activity under UV and visible light irradiation.
- Potential to be applied as a photocatalytic membrane for wastewater treatment.

## GRAPHICAL ABSTRACT



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## ABSTRACT

The use of recycled newspaper as sustainable cellulose resource for the fabrication of green organic/inorganic hybrid photocatalytic membrane via phase inversion method was highlighted in this study. The incorporation of N-doped TiO<sub>2</sub> nanorods as a nanocomposite in regenerated cellulose membrane matrix to great extent has altered its morphological and physicochemical properties, as revealed by FESEM, AFM, FTIR, XRD, XPS, and UV-visible spectroscopy analyses. FTIR analysis suggested that there is a strong interaction between the hydroxyl groups of regenerated cellulose (RC) and the TiO<sub>2</sub> nanorods through hydrogen bonding interactions. The UV-visible spectroscopy and XPS analysis confirmed that the highly visible light absorption capability of the prepared RC/TiO<sub>2</sub> nanocomposite membrane is due to the existence of nitrogen as dopant in the TiO<sub>2</sub> lattice structure. The resultant membranes showed a significant photocatalytic performance in the degradation of phenol in aqueous solution under UV and visible light irradiation, respectively. It was found that 0.5 wt% of TiO<sub>2</sub> nanorods was the best loading in the regenerated cellulose membrane (RCM) with desirable physicochemical and photocatalytic properties. This study promotes the use of RC/TiO<sub>2</sub> nanocomposite membrane as a new and green portable photocatalyst in the field of wastewater treatment without leaving any photocatalyst in the reaction system. It is crucial to emphasize that the use of a non-toxic solvent-based system in this study provide a significant contribution towards the development of a green technology system.

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<b>Abbreviations</b>		$Q$	volume of the permeate water per unit time ( $\text{m}^3 \text{s}^{-1}$ )
PWF	pure water flux	$W_w$	weight of the wet membrane (g)
RC	regenerated cellulose	$W_d$	weight of the dry membrane (g)
RCM	regenerated cellulose membrane	$\rho_H$	density of water ( $0.998 \text{ g/cm}^3$ )
RC/TiO <sub>2</sub>	regenerated cellulose/titanium dioxide	$\rho_c$	density of cellulose ( $1.5 \text{ g/cm}^3$ )
FESEM	field emission scanning electron microscope	$r_m$	membrane mean pore radius (nm)
EFTEM	energy filter transmission electron microscopy	$l$	membrane thickness (m)
FT-IR	fourier transform infrared	$\Delta P$	load pressure (Pa)
XPS	X-ray photoelectron spectroscopy	$R_a$	mean surface roughness (nm)
XRD	X-ray diffraction spectroscopy	$C_0$	initial concentration at time $t = 0$
AFM	atomic force microscopy	$C_t$	concentration at time interval
<b>Symbols</b>		<b>Greek symbols</b>	
$E_g$	band gap energy	$\varepsilon$	membrane porosity
$J$	water flux ( $\text{L m}^{-2} \text{ h}^{-1}$ )	$\theta$	water contact angle
$V$	volume of permeate (L)	$\eta$	water viscosity ( $8.9 \times 10^{-4} \text{ Pa s}$ )
$\Delta t$	time (s)		
$A$	area ( $\text{m}^2$ )		

## 1. Introduction

Currently, the advent of inorganic–organic nanocomposite membrane which combines the processability of polymers and the superior properties of inorganic materials has captured the attention of researchers, owing to the unique advantages of this novel membrane in comparison to the conventionally-made polymeric membrane. Previous works have shown that the incorporation of inorganic materials such as in TiO<sub>2</sub>, ZnO<sub>2</sub>, and Al<sub>2</sub>O<sub>3</sub> in polymeric-based membranes could potentially improve membrane stability and separation performance [1]. For example, the utilization of TiO<sub>2</sub> particles in a membrane modification has been shown that the TiO<sub>2</sub> have a good stabilization, is a hydrophilicity agent, anti-bacterial, has anti-fouling character, and photocatalytic properties [2–4].

Nowadays, photocatalytic technology has become one of efficient and green approaches for the elimination of hazardous pollutants in wastewater treatment [5–7]. TiO<sub>2</sub> has recently attracted substantial attention and has been proven to be the most promising catalyst with strong oxidation activity. However, for large scale applications, TiO<sub>2</sub> as a photocatalyst has two main drawbacks; (1) its photocatalysis oxidation rates for many target pollutants are too slow to be practically applied, and (2) it tends to agglomerate and difficult to separate in the purpose of catalyst recycling [8]. This issue can be solved by introducing nanomaterials in membrane matrix, in addition to offering great promises in the wastewater treatments [9–12].

Cellulose is one of the potential candidates for supporting TiO<sub>2</sub> nanoparticles due to its superfine networks structure. The advantages of superfine networks are they do not only provide mechanical support but also help to disperse the inorganic nanoparticles and to improve the particles stability, retain the special morphology, and control the growth of nanoparticles by providing a template surface for nucleate precipitation [13–15]. The nanoscale of cellulose fibers is approximately 10–100 nm in the form of a web-like network microstructure, which makes cellulose one of the most highly porous materials [16]. In addition, cellulose nanofibers can act as an attractive matrix material for the suspension of photocatalytic particles due to their desirable mechanical and optical properties [17]. There is a good compatibility between the TiO<sub>2</sub> nanoparticles and cellulose chain. The interaction is due to covalent bonds between TiO<sub>2</sub> nanoparticles with cellulose chain that can improve the rigidity of the polymer

chain and increase the energy to break down polymer chain [17]. The feasibility of regenerated cellulose (RC)/TiO<sub>2</sub> nanocomposites membrane in water and wastewater treatment has been extensively studied. For instance, Zeng et al. (2010) proposed TiO<sub>2</sub> immobilization in cellulose matrix for photocatalytic degradation of phenol under weak UV light irradiation [13]. Zhu et al. (2012) developed a novel inorganic–polymer hybrid membrane by the incorporation of nano-TiO<sub>2</sub> into RC with high performance for dehydration of caprolactam by pervaporation [18]. Zhang and co-workers prepared bacterial cellulose/TiO<sub>2</sub> composite membrane doped with rare earth elements, and evaluated its photocatalytic properties [8]. They found that, the resultant composites membrane has high strength, ultrafine nanoporosity, and water adsorption characteristics, whereas the photocatalysis efficiency was significantly enhanced after the TiO<sub>2</sub>/Bacterial cellulose membrane was doped with rare earth ions. Furthermore, the obtained RC/TiO<sub>2</sub> nanocomposites membrane also exhibited high UV–vis light absorption [19]. Moreover, reusable photocatalytic titanium dioxide–cellulose based films showed the potential for degradation of organic molecules in natural water sources [20,21].

Previous studies have utilized various sources of cellulose in the fabrication of regenerated cellulose membrane (RCM) such as cotton linter, soft wood pulp, and microcrystalline cellulose [22–24]. To the best of our knowledge, there is still no recorded study on cellulose from recycled newspapers in photocatalytic membrane application in the literature. The RCM made of recycled newspaper has a great interest towards sustainable future and control the white pollution [25]. The main drawback of pure TiO<sub>2</sub> is its wide band gap (3–3.2 eV). Particularly, it absorbs only the UV part of solar radiation that accounts for only 4% of the total solar radiation, leaving most of the visible light irradiation [26]. Therefore, it is important to develop photocatalyst that can be utilized under visible light. Among the reported photocatalysts with visible-light response, TiO<sub>2</sub> doped with nitrogen has been extensively studied because of its comparable atomic size with oxygen, small ionization energy, eco-friendly, relatively high stability and simple synthesis methods [27–29]. Therefore, it is important to study the potential, feasibility and compatibility of both materials; cellulose from recycled newspaper and N-doped TiO<sub>2</sub> nanorods for the preparation of photocatalytic membrane. The application of this photocatalytic membrane can overcome the difficulty in recollecting and removal of TiO<sub>2</sub> suspension in water after photocatalytic treatment. Furthermore, this approach is a truly green process

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