TITANIUM DIOXIDE NANORODS IN REGENERATED CELLULOSE MEMBRANE FOR PHOTOCATALYTIC DEGRADATION OF PHENOL

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To my beloved parents
(Mohamed B. Md Yusuf and Ramenas Bt Abdullah)
and friends who gave me inspiration, encouragement and endless support throughout the success of my study.
May this thesis be an inspiration and guidance in the future.

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ABSTRACT

Polymer-inorganic nanocomposite membrane was successfully prepared via incorporation of nitrogen doped (N-doped) titanium dixide (TiO_2) anatase/rutile mixed phase nanorods in the cellulose microfiber by using phase inversion technique. The use of the non-toxic solvent-based system and recycled newspapers as the cellulose source in this study provides a significant contribution towards the development of a green technology system. The incorporation of N-doped TiO_2 nanorods that have been calcined at 400°C (T400) in regenerated cellulose membrane matrix has altered significantly its morphological and physicochemical properties, as revealed by Fourier Transform Infrared (FTIR), Field Electron Scanning Microscopy (FESEM), Transmission Electron Microscopy (TEM), Atomic Force Microscopy (AFM), UV-vis spectroscopy, Thermal Gravimetric Analysis (TGA), X-ray Diffraction (XRD) and X-ray photoelectron spectroscopy (XPS) analysis. The UV-vis spectroscopy and XPS analysis confirmed that the highly visible light absorption capability of the prepared regenerated cellulose/titanium dioxide (RC/TiO_2) nanocomposite membrane is due to the existence of nitrogen as dopant in the TiO_2 lattice structure at 396.8, 397.5, 398.7, 399.8, and 401 eV. It was found that 0.5 wt % of N-doped TiO_2 nanorods (T400) is the best loading in the regenerated cellulose/titanium dioxide (RC/TiO_2) nanocomposite membrane with desirable morphological, physicochemical and photocatalytic properties. The RC/TiO_2-0.5 exhibited the highest photocatalytic activity of 96 % and 78.8 % in degradation phenol after 360 minutes under visible and UV lights irradiation. From the findings, this study promotes the use of RC/TiO_2 nanocomposite membrane as a new green portable photocatalyst in the field of wastewater treatment without any residue of photocatalyst in the reaction system.

ABSTRAK

Membran polimer-bahan tak organik nanokomposit telah berjaya dihasilkan dengan menggabungkan titanium dioksida (TiO_2) fasa bercampur anatasa/rutil yang telah didopkan dengan nitrogen (N-doped) bersama-sama dengan mikrofiber selulosa dengan menggunakan teknik fasa penyonsangan. Penggunaan sistem berasaskan pelarut-bukan toksik serta penggunaan akhbar kitar semula sebagai sumber selulosa dalam kajian ini adalah sangat penting untuk ke arah pembangunan sistem teknologi hijau. Gabungan N-doped TiO_2 nanorod yang dikalsinasi pada 400°C (T400) sebagai nanokomposit di dalam membran selulosa terjana semula telah merubah sifat morfologi dan fizikokimia seperti dibuktikan melalui analisis Inframerah Transformasi Fourier (FTIR), Mikroskopi Medan Pengimbas Elektron (FESEM), Mikroskopi Transmisi Elektron (TEM), Mikroskopi Daya Atom (AFM), UV-vis spektroskopi, Analisis termal gravimetrik (TGA), Pembelauan sinar-X (XRD) dan spektroskopi sinar-X fotoelektron (XPS). Analisa UV-vis spektroskopi dan XPS telah mengesahkan bahawa keupayaan penyerapan cahaya nampak yang sangat tinggi adalah disebabkan oleh kewujudan nitrogen sebagai pendopan di dalam struktur kekisi TiO_2 dan tenaga pengikatannya dikesan pada kedudukan 396.8, 397.5, 398.7, 399.8, dan 401 eV. Didapati bahawa berat peratusan TiO_2 nanorod (T400) sebanyak 0.5 merupakan jumlah kandungan terbaik dalam membran selulosa terjana semula/titanium dioksida (RC/TiO_2) nanokomposit membran dengan ciri-ciri morfologi, fizikokimia, dan sifat fotopemangkinan yang diinginkan. Sampel RC/TiO_2-0.5 menunjukkan aktiviti fotopemangkinan yang paling tinggi dengan peratusan degradasi fenol pada 96% dan 78.8% selepas diradiasikan di bawah cahaya nampak dan UV selama 360 min. Hasil dapatan menerusi kajian ini menggalakkan penggunaan RC/TiO_2 membran nanokomposit sebagai fotomangkin mudah alih hijau baru bagi merawat air sisa tanpa meninggalkan sisa fotomangkin di dalam sistem tindak balas.

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LIST OF ABBREVIATIONS

AFM	-	Atomic Force Microscopy
ATR	-	Attenuated total reflection
BET	-	Brunauer, Emmett and Teller
CA	-	Cellulose acetates
CMF	-	Cellulose microfiber
CNF	-	Cellulose nanofiber
DS	-	Degree of swelling
FESEM	-	Field Emission Scanning Electron Microscopy
FTIR	-	Fourier Transform Infra-red
FWHM	-	Full-width-at-half-maximum
LED	-	Light-emitting diode
MCC	-	Microcrytalline cellulose
MF	-	Microfiltration
MWCNT	-	Multiwall carbon nano tube
NF	-	Nanofiltration
NMMO	-	N-methylmorpholine N-oxide
NMP	-	1-methyl-2-pyrrolidone
NOM	-	Natural organic material
PMR	-	Photocatalytic Membrane Reactor
PVDF	-	Polyvinylidene fluoride
PWF	-	Pure Water Flux
RC	-	Regenerated cellulose
RCM	-	Regenerated cellulose membrane
RNP	-	Recycled Newspaper

RO - Reverse osmosis
SEM - Scanning electron microscopy
TEM - Transmission Electron Microscopy
TGA - Thermal gravimetric analysis
UF - Ultrafiltration
UV - Ultra violet
WC - Water content
WCA - Water contact angle
XPS - X-ray Photoelectron Spectroscopy
XRD - X-ray Diffraction

LIST OF SYMBOLS

A - Membrane surface area (m^2)
E - Band gap energy (eV)
C - Speed of light (3.00×10^{17} nm/s)
C_0 - Initial concentration of pollutants
C_t - Concentration of the pollutants at specific time
H - Planck’s constant (4.1357×10^{-15} eV s)
J - Pure water flux (PWF) ($\mathrm{L\ h^{-1}\ m^{-2}}$)
$\mathrm{r}f$ - Membrane mean pore size (nm)
R_a - Surface roughness (nm)
v - Frequency (cm^{-1})
W_w - Weight of wet film (g)
W_d - Weight of dry film (g)
ρ_H - Density of water ($0.998\ g/cm^3$)
ρ_c - Density of cellulose ($1.5\ g/cm^3$)
$\Delta \mathrm{P}$ - Load pressure (Pa).
λ - Wavelength (nm)
λ_{max} - Wavelength maximum (nm)
ε - Porosity (%)
η - Water viscosity (8.9×10^{-4} Pa s)
hv - Photon energy
Eg - Band gap energy

CHAPTER 1

INTRODUCTION

1.1 Background of Study

The ability of titanium dioxide (TiO_2) semiconductor to degrade organic and inorganic pollutants comes from redox environment that is generated from photoactivation, and this makes it intensively utilized as a photocatalyst in wastewater treatment (Chun *et al.,* 2000; Fan *et al.,* 2011; Manilal *et al.,* 1992). The photoactivation of TiO_2 photocatalyst occurs when the absorption of UV irradiation onto TiO_2 particles surface takes place. The UV irradiation absorption can be equal or higher than the band gap value of 3.2 eV for anatase or 3.0 eV for rutile (Ouzzine *et al.,* 2014; Scanlon *et al.,* 2013). TiO_2 exists in three distinct polymorphs, which are anatase, rutile (both tetragonal crystal systems), and brookite (orthorhombic crystal system) (Devilliers 2006; Wu *et al.* 2012). A previous study on the band gap alignment of rutile and anatase TiO_2 has proven that the mixed phase of anatase/rutile TiO_2 has synergistic effects and higher photocatalytic activity as compared to pure phase of either in anatase or rutile (Scanlon *et al.* 2013). Degussa P25 and Aeroxide TiO_2 P25 are the common commercial mixed phases of anatase/rutile TiO_2, containing about 80 % anatase and 20 % rutile. The reason for the synergistic effects of the mixed phase of anatase/rutile TiO_2 nanoparticles in photocatalytic properties, however, still remains elusive. It is believed that the mixed phase of anatase/rutile TiO_2 can improve the charge carrier separation through electron trapping in rutile and consequently reduce the electron recombination. As a result, the formation of radical species for oxidation of substrate molecules can be maintained (Ohtani *et al.,* 2010).

Today, membrane technology has expanded broadly and has been applied in various applications and industries such as in water and wastewater treatment, petrochemical, pharmaceutical, and water desalination. The introduction of membrane technology in such applications and industries have given a lot of advantages such as low energy consumption, low chemicals consumption, production of water of stable quality almost independent on the quality of the treated water, automatic, flexible and stable operation, low maintenance cost, as well as easy to scale up by simple connecting additional membrane modules (Mozia, 2010). Typical membrane processes involved separation operation by means of filtration including reverse osmosis, micro-, ultra-, and nanofiltration, gas separation, pervaporation, and ion exchange membrane processes-electrodialysis (Baker, 2004). Membrane processes are already utilized in many applications but currently, with new membrane system such as photocatalytic membrane, has its own prestigious reputation in the separation and purification technology (Hou *et al.,* 2013; Patsios *et al.,* 2013; Kumakiri *et al.,* 2010; Mozia *et al.,* 2008; Choo *et al.,* 2008). Based on previous studies, TiO_2 is used as a photocatalyst and has been integrated with specific membrane materials (Chin *et al.,* 2006; Moustakas *et al.,* 2014; Shi *et al.,* 2012; Sun *et al.,* 2012).

The combination of membrane and photocatalytic technology creates fascinating approach in order to ensure that water and wastewater treatments become more effective. The developments of photocatalytic membranes technology have been broadly studied. Many studies are focused on the type and modification of the catalyst and membrane, the optimization of photocatalytic membrane reactor and system in terms of design and performance, detailed fundamental of photocatalytic membrane, factors that affect the photocatalytic activity in terms of percentages of photodegradation, and the efficiency of the systems and processes (Mozia, 2010; Zeng *et al.,* 2010; Mozia *et al.,* 2008).

Cellulose is one of the membrane materials that gives a great promise in nano-photocatalytic membrane. In addition, cellulose is considered as a good candidate for host material of nanoparticles due to the ability of improve the stability, retain the special morphology, and control the growth of nanoparticles (Zeng *et al.,* 2010). Environmental friendly, nanostructured, high porosity, thermal insulator and very high impact strength material have also made cellulose becomes one of the potential materials in many fields. There are various potential applications such as in electronics, chemistry, mechanics, engineering, energy production and storage, sensors, medicine, nanotechnology, military and aerospace, oil and gas recovery, thermal insulation and household uses. Regenerated cellulose membrane (RCM) has been extensively commercialized in the field of membrane science and technology, which involved various membrane separation processes such as microfiltration, ultrafiltration, nanofiltration, reverse osmosis, gas separation, pervaporation and many more (Dogan and Hilmioglu, 2010; Fukuzumi *et al.,* 2009; Ma *et al.,* 2012; Ramesh Babu and Gaikar, 2001; Singh *et al.,* 2008; X. Xiong, *et al.,* 2010; Q. Yang *et al.,* 2011; Zhu *et al.,* 2012). Cellulose films showed an average Young's modulus of 14 GPa (Henriksson and Berglund, 2007). A study has shown that a sheet-shaped material prepared from bacterial cellulose has Young's modulus more than 15 GPa across the plane of the sheet (Yamanaka et al. 1989). The nanoscale of cellulose fibers is approximately 10 to 100 nm have a web-like network microstructure make cellulose one of the most high porous material (Takagi 2011).

It is believed that the performance of membrane processes can be improved by introducing nanomaterials in the membrane matrix. The overviews from recent studies about the knowledge of nanoparticles in modification of TiO_2 and cellulose-based membrane matrix in nanoscale offer great promises in the wastewater treatment industry. In addition, the understanding and modification of microstructures of photocatalytic membrane gives a positive impact towards development of high performance and effective wastewater treatment.

1.2 Problem Statement

Titanium dioxide (TiO_2) is one of the semiconductors that has been widely used as a photocatalyst in water and wastewater treatments due to its chemical stability, low cost, excellent optical and electronic properties, as well as high photocatalytic activity (Li *et al.,* 2009; Liu *et al.,* 2010). Compared to rutile and brookite, anatase has shown the highest photocatalytic activity in the degradation of various organic pollutants in wastewater treatment. Most of the previous studies have focused on the preparation of single-phase TiO_2 nanostructures (Ao *et al.,* 2008a; Li *et al.,* 2009; Li *et al.,* 2013; Liu *et al.,* 2010; Liu, 2012). In addition, a recent study revealed that the combination of anatase/rutile mixed phase exhibited excellent photocatalytic activity compared to its single constituents (Apopei *et al.,* 2014; Kalashnikova *et al.,* 2013; Scanlon *et al.,* 2013; Xiong *et al.,* 2014; Zhang *et al.,* 2010). The excellent photocatalytic activity is due to the synergy effect between anatase and rutile, which promotes interfacial electron transfer from rutile to anatase (Apopei *et al.,* 2014).

A major drawback of TiO_2 pure is its large band gap, which means it can only be activated under UV region ($\lambda \leq 387$ nm), thus limiting the practical efficiency for solar applications (Pelaez *et al*., 2012). Therefore, it is important to develop photocatalyst that can be utilized under visible light. Recently, many studies have been conducted to improve the photoabsorption features of TiO_2 under UV and visible light irradiation (Khan *et al.,* 2014; Ruzimuradov *et al.,* 2014). Currently, increasing attention has been paid to the doping of TiO_2 with non-metal atoms since it provide a promising way to avoid deteriorating thermal stability of the TiO_2 lattice structure (Kumar and Devi 2011). An effective way to narrow the band gap is to dope TiO_2 with non-metal elements such as B, S, C, N, F, Cl, Br and I (Hu *et al.,* 2014; Wang *et al.,* 2012). However, nitrogen has been found to be one of the promising non-metal dopant materials for TiO_2 lattice to induce visible absorption (Lee *et al.,* 2014; Viswanathan and Krishanmurthy 2012; Selvaraj *et al.,* 2013). The main reasons of utilizing nitrogen as the dopant material are due to its comparable atomic size with oxygen, small ionization energy, eco-friendly, higher stability and simple synthesis methods (Viswanathan and Krishanmurthy 2012; Zhang *et al.,* 2013; Gai *et al.,* 2012). In a present study conducted by Hu and co-worker (2014), N-doped anatase/rutile TiO_2 hybrid material was synthesized in low-temperature by direct nitridization in order to enhance photoactivity under UV and visible light irradiations (Hu *et al.,* 2014). The substitution of lattice oxygen via nitrogen doping in TiO_2 lattice crystals leads to narrowed band gap and facilitated visible light absorption capability (Viswanathan and Krishanmurthy 2012). On top of that, nitrogen doping also inhibited the recombination of the photoinduced carriers and therefore increased the quantum efficiency of TiO_2 photocatalyst (Hu *et al.,* 2014; Ruzimuradov *et al.,* 2014; Viswanathan and Krishanmurthy 2012). Basically, this approach has expanded the versatility of TiO_2 photocatalyst in a broader range of UV and visible regions. The combined effects of N-doping and synergistic effects have improved the photocatalytic activity of TiO_2 in the mineralization of hazardous pollutants.

Recently, there are several advanced synthesis method have been applied to prepared visible light active TiO_2 included Flame Spray Pyrolysis (FSP), sputtering technique, Angle Deposition (AOD) technique, Successive Ion Layer Adsorption and Reaction (SILAR), and Flame Spray Pyrolysis (FSP) (Inturi *et al.,* 2014; Asahi *et al.,* 2001; Xie *et al.,* 2014; Xie *et al.,* 2013). The common defect of these techniques is the requirement of complicated and relatively expensive equipment. The development of novel TiO_2 photocatalyst with enhanced UV and visible light activity via economic, simple and direct synthetic method has become necessitous. Sol-gel is one of the most prominent methods used to prepare mixed phase of anatase/rutile TiO_2 nanoparticles due to its simplicity and low equipment requirements. The preparation of TiO_2 from sol-gel had some advantages, such as the production of high purity nanocrystalline through precipitation and the flexibility to control the synthesis process (You *et al.,* 2014). There are three main chemical reagents required in the preparation of TiO_2 via sol-gel method, which are a precursor or the starting material for Ti source, an acid catalyst, and a solvent as dispersing media. The common precursors used for the preparation of anatase nanocrystalline are titanium-n-butoxide (Ao *et al.,* 2008; Li *et al.,* 2009; You *et al.,* 2014), titanium (IV) isopropoxide (Cimieri *et al.,* 2013; Ananth *et al.,* 2014), and tetrabutyl orthotitanate (You *et al.,* 2012). The highly crystalline TiO_2 nanoparticles can be prepared via sol-gel method, and followed by heat treatment that ranges from 0 to 600 °C (Li *et al.,* 2009; Liu, 2012). It has been reported that the high quality of mixed phase of anatase/rutile TiO_2 nanoparticles, which contributes to high photocatalytic activity, can also be obtained by manipulating the types of reagents and heat treatment conditions (Bakardjieva *et al.,* 2005; Mahshid *et al.,* 2009; Wu *et al.,* 2012).

However, to the best of our knowledge, there is hardly a report on the simple and direct preparation of nitrogen doped (N-doped) anatase/rutile mixed phase TiO_2 nanostructures (Peng *et al.,* 2013; Schütz *et al.,* 2012).

The rapid developments in synthetic polymer have provided great benefits to the modern society. Microporous and mesoporous synthetic membranes have attracted considerable attention for water treatment due to their excellent thermal, chemical, and mechanical stability, and their reusability after burning over conventional polymeric membranes such as polysulfone (PSf), polyamide, polyvinylidene fluoride (PVDF), and polytetrafluoroethylene (PTFE) (Choi *et al.,* 2006). However, waste from synthetic polymer is difficult to degrade and dispose of, resulting in severe urban environmental consequences. As environment sustainability is concerned, the world has urged the demand to search for alternative sources in order to prevent and control white pollution. The white pollution will lead to detrimental effects on soil structure, water and nutrient transport, as well as crop growth, thereby disrupting agricultural environment and reducing crop production (Liu *et al.,* 2014). Among the alternatives, polymer recycling and return to biologically-based renewable polymers may be emphasized (Qi *et al.,* 2009; Rodrigues Filho *et al.,* 2008).

Cellulose is unquestionably the most abundant naturally occurring reproducible organic compound and it will become the main chemical resource in the future (Schurz 1999). The versatility of cellulose to be applied in various applications is due to the low cost, strong hydrophilicity properties, fascinating structure, biocompatible, and derivable properties (Yang *et al.,* 2011). In recent years, the green comprehensive utilization of cellulose resources has drawn much attention from governments and researchers (Qi *et al.,* 2009). Previous studies have utilized various sources of cellulose in the fabrication of RCM such as cotton linter, softwood pulp, and microcrystalline cellulose (Ichwan and Son, 2011; Mahmoudian *et al.,* 2012; Mao *et al.,* 2006). To the best of our knowledge, there is no research has been attempted in the preparation of RCM by utilizing old recycled newspaper as cellulose sources. This would be an environmental friendly approach since tonnes of newspapers are discarded every year. Therefore, it is important to assess the feasibility of old recycled newspaper as cellulose sources in the preparation of green and low cost RCM.

The development of photocatalytic membrane as the new treatment technology can enhance the effectiveness of water and wastewater treatment. The combination of photocatalyst and membrane is believed to improve the efficiency and effectiveness in water and wastewater treatments. Various studies have been done to incorporate TiO_2 nanoparticles in various membrane matrices such as polyvinylidene fluoride (PVDF), polyvinylidene fluoride-grafted-polyacrylic acid (PVDF-g-PAA), polyvinylidene fluoride/sulfonated polyethersulfone (PVDF/SPES), polysulfone (PSf), polyethersulfone and γ-alumina membrane (Bae and Tak, 2005; Damodar *et al.,* 2009; Emadzadeh et al, 2014; Moustakas *et al.,* 2014; Rahimpour *et al.,* 2011; Yang *et al.,* 2007; You *et al.,* 2012). The introduction of TiO_2 into these polymeric membranes can improve the hydrophility, self-cleaning, anti-fouling, anti-bacterial and photocatalytic properties of polymeric membranes (Damodar *et al.,* 2009; Shi *et al.,* 2013; You *et al.,* 2012).

The feasibility study of RC/TiO_2 nanocomposites membrane in water and wastewater treatment has been studied by previous studies. For example, Zeng et al. (2010) proposed TiO_2 immobilization in cellulose matrix for photocatalytic degradation of phenol under weak UV light irradiation (Zeng *et al.,* 2010). Tianrong *et al.,* (2012) developed a novel inorganic–polymer hybrid membrane by the incorporation of nano-TiO_2 into regenerated cellulose (RC) with high performance for dehydration of caprolactam by pervaporation. Furthermore, Zhang and co-workers prepared bacterial cellulose/TiO_2 composite membrane doped with rare earth elements and its photocatalytic properties have been evaluated (Zhang *et al.,* 2011). The resultants composites membrane has high strength, ultrafine nanoporosity, and water absorption characteristics, whereas the photocatalysis efficiency was significantly enhanced after TiO_2/BC membrane was doped with rare earth ions. Furthermore, the obtained RC/TiO_2 nanocomposites membrane also exhibited high UV- vis light absorption (Morawski *et al.,* 2013). Moreover, reusable photocatalytic titanium dioxide-cellulose nanofiber films show the potential for degradation of organic molecules in natural water sources (Snyder *et al.,* 2013).

However, the study on immobilization and incorporation, as well as dispersion of N-doped TiO_2 mixed phase anatase/rutile nanoparticles in RCM has not yet been reported. Therefore, it is important to study the potentiality, feasibility and compatibility of both materials; cellulose and N-doped TiO_2 anatase/rutile mixed phase in the field of photocatalytic membrane. The preparation of this membrane will improve the photodegradation of water pollutants under broad range of UV and visible light irradiations. Furthermore, this approach is a truly green process and cost-effective in terms of its development, preparation and application. The knowledge of modification of titanium dioxide and cellulose-based membrane in nanoscale needs to be considered, high-performance photocatalytic membrane reactors need to be developed. The preparation of membrane nanocomposites with proper methods and techniques is crucially important in the development of high-performance photocatalytic membrane in removal of pollutants in water and wastewater.

1.3 Objectives of Study

Based on the research background and the problem statements aforementioned, the objectives of this study are:

1) To evaluate the effect calcination temperature on physicochemical and photocatalytic activity of N-doped anatase-rutile mixed phase TiO_2 nanorods via a direct and simple sol-gel method.

2) To access the effect of different step of pretreatment for the extraction cellulose microfiber from old recycled newspaper as a cellulose source for RC/TiO_2 nanocomposite membrane fabrication.

3) To study the effect of loading ratio of TiO_2 to RCM matrix on physicochemical properties and photocatalytic membrane activities.

1.4 Scope of Study

In order to fulfil the objectives of the study, the following scopes of work have been drawn:

1) Synthesize and characterize N-doped anatase-rutile mixed phase TiO_2 nanorods via a direct and simple sol-gel method.

The aim of this part is to prepare N-doped TiO_2 anatase/rutile nanorods assembled microspheres with high photocatalytic properties in UV and visible regions via a direct sol-gel method by manipulation of calcination temperature. The calcination temperatures are varied from 200, 400, 600, and 800 °C. In this part, titanium-n-butoxide, $Ti(OBu)_4$ was used as the Ti precursor, nitric acid as the catalyst, and isopropanol as the dispersing media. The photocatalytic activity of the prepared TiO_2 nanoparticles was evaluated under UV and visible light irradiations. The physicochemical and structural characterization of high photocatalytic of the prepared TiO_2 was evaluated by x-ray diffraction (XRD), Brunauer, Emmett and Teller (BET) surface area, field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM) and atomic force microscopy (AFM), Fourier transform infrared (FTIR), UV-Vis spectroscopy and x-ray photoelectron spectroscopy (XPS). The best TiO_2 was then used as photocatalyst nanocomposites in RCM.

2) Development of RC/TiO_2 nanocomposite membrane.

This part involves the extraction of cellulose microfiber from old recycled newspaper as a cellulose source using different steps of chemical pretreatment. Then, the resultant treated cellulose microfiber is further utilized in the preparation of flat sheet cellulose/TiO_2 anatase/rutile mixed phase nanocomposite membrane by manipulating the loading ratio of TiO_2 to cellulose dope solution (0.1 to 0.5 wt %). Scanning electron microscopy (SEM), atomic force microscopy (AFM), transmission electron microscopy (TEM), Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD) spectroscopy and thermal gravimetric analysis (TGA) were carried out to investigate the physicochemical and structural properties of RC/TiO_2 nanocomposite. On top of that, the physical characteristics of for the resulting membrane were studied for its pure water flux, water content, water contact angle, porosity and pore size.

3) Investigating the photocatalytic activity of RC/TiO_2 nanocomposite membrane.

In this part, phenol aqueous solution was used as model water pollutants. The model water pollutant was irradiated using ultraviolet (UV) lamp (Vilber Laurmat, $\lambda = 312$ nm, 30 watt) and visible lamp (light-emitting diode (LED), $\lambda > 420$ nm, 30 watt). In addition, the effect of loading ratio of TiO_2 to RCM matrix on photocatalytic membrane activity was evaluated. The photodegradation of phenol was monitored using UV-visible spectroscopy at the wavelength of 296.35 nm.

1.5 Significance of Study

The search for alternatives to preserve the environment becomes crucial. Now, sources for production of cellulose microfiber from recycled newspaper become a green solution for environmental preservation. Using recycled newspaper as a cellulose source is considered economic and the added value to industrial and urban residues is increasing significantly. The preparation of cellulose as the membrane from recycled resources (recycled newspaper) is considered ‘‘green’’. Cellulose is well-recognized as a renewable and biodegradable natural polymer with good mechanical properties in its natural or derivative form. Thus, it is a perfect candidate as a green membrane material. In addition, titanium dioxide is a well-known photocatalyst due to its stability, chemical structure, biocompatibility and physical properties. The incorporation of N-doped TiO_2 anatase/rutile mixed phase in cellulose membrane matrix will improve the elimination of hazardous pollutants in water and wastewater under UV and visible light irradiations. The study of the interaction of TiO_2 in cellulose membrane at the nanoscale level will provide promising knowledge and contribution towards photocatalytic membrane development.

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