METAL OXIDES INCORPORATED BAUXITE HOLLOW FIBRE PHOTOCATALYTIC MEMBRANE FOR BISPHENOL A REMOVAL FROM AQUEOUS SOLUTION

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ABSTRACT

The demand for advanced water treatment technologies is increasing for the treatment of high-strength wastewater, including complex water pollutants. Removal of bisphenol A (BPA) from water has presented a major challenge for the water industry. Membrane separation has the advantages of simplicity, high speed, and high efficiency, and has received extensive attention around the world. It is well known that membrane materials and membrane processes are two of the key factors affecting the separation process. The selection of suitable membrane materials is of great significance to produce effective dual-function ceramic membrane which possesses filtration and photocatalysis features in a single unit of membrane. In this study, naturally existing bauxite was selected as a ceramic material because of its availability and the presence of iron (III) oxide (Fe₂O₃) and titanium dioxide (TiO₂) which have potential to be used as photocatalyst. In the first stage of this work, bauxite powder was subjected to thermal treatment at different temperatures. In the second stage of the study, a hydrophilic, asymmetric bauxite hollow fiber membrane (BHFM) was fabricated by phase inversion and sintering method. To study the morphologies of BHFM, the bauxite loading and sintering temperature was varied from 45 to 55 wt% and at temperature ranging from 1250 to 1450 ºC. Then, in the third stage, the yielded membrane was subjected to the surface modification to lift up the photocatalytic properties using titanium dioxide (1wt% of $TiO₂$) and copper oxide (1wt% of CuO) particles via hydrothermal method for the removal of BPA. $TiO₂$ and CuO particles were modified on the surface of 50 wt% BHFM by varying the hydrothermal time of 2.5h, 5.0h and 7.5h. In the fourth stage of the study, the photocatalytic membrane was further evaluated for the photocatalytic efficiency in degradation of BPA, which was present in water. The finding of this study showed that the powder treated at 800 ºC possessed good photocatalytic degradation as it was able to degrade up to 75% of 5 mg/L BPA. 50 wt% BHFM which spun at bore fluid flow rate of 10 mL/min, air gap of 5 cm, and sintering temperature of 1300 °C induced good mechanical strength of 98.2 MPa, stable permeate water flux (PWF) of \sim 281.4 L/m²h and moderate BPA degradation rate of less than 70%. The pristine BHFM and modified $TiO₂$ and CuO BHFM with hydrothermal time of 5.0h showed promising finding with almost even distribution of modified particles on the membrane surface. The experimental results of photocatalytic activity test showed that the BPA degradation of 96.8% was achieved by CuO BHFM under visible light irradiation, while for UV light irradiation, $TiO₂$ BHFM possessed the degradation rate of 90.3% for 360 minutes. Three intermediate products were determined which were 4-(2-hydroxy-2-propanol)phenol, 4-isopropenylphenol and dihydroxybenzene. All the findings in this study are helpful for understanding the process of photodegradation and to become a promising potential treatment to degrade BPA to provide water safety for living organisms.

ABSTRAK

Permintaan terhadap teknologi rawatan air termaju semakin meningkat untuk rawatan air dengan pencemaran tinggi termasuk bahan cemar yang kompleks. Penyingkiran bisphenol A (BPA) dari air memberikan cabaran yang besar dalam industri air. Pemisahan membran yang mempunyai kelebihan dari segi kesederhanaan, kelajuan yang tinggi, dan kecekapan yang tinggi telah mendapat perhatian luas di seluruh dunia. Secara umumnya, telah diketahui bahawa bahan membran dan proses membran adalah dua faktor utama yang mempengaruhi proses pemisahan. Pemilihan bahan membran yang sesuai sangat penting untuk menghasilkan membran seramik dwi fungsi yang berkesan dengan mempunyai ciri penapisan dan fotomangkin dalam satu unit membran. Dalam kajian ini, bauksit yang wujud secara semula jadi telah dipilih sebagai bahan seramik kerana ketersediaan bahan tersebut dan adanya ferum (III) oksida (Fe₂O₃) dan titanium dioksida (TiO₂) yang berpotensi untuk digunakan sebagai fotomangkin. Pada peringkat pertama kajian, serbuk bauksit menjalani rawatan terma pada suhu yang berbeza. Pada peringkat kedua kajian, membran serat berongga hidrolik, asimetrik menggunakan bauksit (BHFM) dihasilkan dengan menggunakan kaedah penyongsangan fasa dan pembakaran. Untuk mengkaji morfologi BHFM, muatan bauksit dan suhu persinteran dipelbagaikan dari 45% berat hingga 55% berat dan pada suhu antara 1250 ºC hingga 1450 ºC. Pada peringkat ketiga kajian, membran terhasil dilakukan pengubahsuaian permukaan untuk meningkatkan sifat fotobermangkin dengan menggunakan zarah TiO₂ (1% berat TiO₂) dan tembaga oksida (CuO) (1% berat CuO) melalui kaedah hidrotermal untuk penyingkiran BPA. Zarah TiO₂ dan CuO diubahsuai pada permukaan 50% berat BHFM dengan mempelbagaikan masa hidroterma 2.5 jam, 5.0 jam dan 7.5 jam. Pada peringkat keempat kajian, membran fotobermangkin selanjutnya dinilai untuk kecekapan fotobermangkin dalam penurunan BPA yang terdapat di dalam air. Hasil kajian menunjukkan bahawa serbuk bauksit yang menjalani rawatan terma pada suhu 800 ºC mengalami penurunan fotobermangkin yang baik kerana mampu menurunkan sehingga 75% dari 5 mg/L BPA. 50% berat BHFM yang diputar pada kadar aliran bendalir gerek 10 mL /min, jurang udara 5 cm, dan suhu persinteran 1300 °C memberikan kekuatan mekanikal yang baik pada 98.2 MPa dan fluks air telapan stabil (PWF) ~ 281.4 L / m 2 j dan kadar penurunan BPA sederhana kurang daripada 70%. BHFM yang tidak diubahsuai dan TiO₂ dan CuO BHFM yang telah diubahsuai dengan masa hidroterma 5.0 jam menunjukkan penemuan yang memberangsangkan dengan sebaran zarah yang diubahsuai hampir sama rata pada permukaan membran. Hasil eksperimen ujian aktiviti fotobermangkin menunjukkan bahawa penurunan BPA 96.8% dicapai oleh CuO BHFM di bawah penyinaran cahaya tampak, sementara untuk penyinaran cahaya UV, TiO² BHFM menguasai kadar penurunan 90.3% selama 360 minit. Tiga produk perantaraan ditentukan iaitu 4- (2-hidroksi-2-propanol) fenol, 4 isopropenilfenol dan dihidroksibenzena. Semua penemuan dalam kajian ini bermanfaat untuk memahami proses fotopenurunan dan menjadi rawatan yang amat berpotensi dalam menurunkan BPA untuk keselamatan air bagi organisma hidup.

TABLE OF CONTENTS

TITLE PAGE

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

LIST OF SYMBOLS

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Background of Research

The presence of contaminated wastewater containing endocrine disrupting compounds (EDC), in particular bisphenol A (BPA), has had an effect on both living organisms and the environment. It can also induce immunotoxic, mutagenic, genotoxic, hepatotoxic, teratogenic, neurotoxic and carcinogenic effects, even at nanomolar level (Pfeifer et al. 2015). Despite BPA's negative impact on the human body, it is one of the most commonly produced and used compounds worldwide with annual production expected to reach 10.6 million metric tons in 2022. Its annual growth rate between 2016 and 2022 is approximately 4.8% (Industry Experts, 2016). Because of the wide usage of polycarbonate plastics and epoxy resins in industry and households, BPA is a prevalent contaminant in the environment and its concentration, especially in the aquatic environment, is constantly increasing (Cleveland et al. 2014; Bilal et al. 2019; Grelska and Noszczyńska 2020). It enters these ecosystems mainly through the effluents of wastewater treatment plants (WWTPs), where by lack of efficient systems of its removal, BPA may contaminate drinking water sources downstream (Zielinska et al. 2019). Taking into account that BPA possesses an ecological risk, there is an urgent necessity to eliminate it from the environment.

Currently, there are different methods of BPA wastewater treatment including physical and chemical process such as adsorption, membrane technologies, oxidation, coagulation/flocculation and photocatalysis. The benefits of these approaches are that they are capable of extracting a wide variety of dyes and fast processes (Pearce et al., 2003). On the other hand, these methods are expensive and the aggregation of concentrated sludge often causes difficulties to remove the contaminants (Li and Guthrie, 2010; Pearce et al., 2003). In general, biological

treatment methods are more efficient and environmentally friendly (Pearce et al., 2003).It has been reported that microorganisms are able to degrade BPA molecule efficiently under anoxic conditions and the intermediate products (amines) could be detoxified under aerobic environment (Grekova-Vasileva et al., 2009). The aromatic compounds were then degraded under aerobic condition (Stolz, 2001), to produce catechol compound and eventually to $CO₂$, water and ammonia (Van der Zee and Villaverde, 2005). Among the studied method, removal of BPA through photocatalysis degradation is found to be the most competitive one because it does not need a high operating temperature and several coloring materials can be removed simultaneously (Crini, 2006). The versatility of photocatalysis is due to its high efficiency, economic feasibility and simplicity of design (Chen et al, 2010).

Advanced oxidation processes (AOPs) have attracted increasing attention for water and wastewater treatment. AOPs differ from conventional physical and biological water treatment processes as they generate hydroxyl radicals, which are the strongest oxidant after fluorine in aqueous solutions. AOPs are able to degrade toxic and refractory pollutants into simple and harmless inorganic molecules without generating secondary waste. In addition, recalcitrant organic contaminants can also be eliminated by degradation of these compounds under certain exposure to the sunlight. The generation of hydroxyl radicals can be initiated by primary oxidants (hydrogen peroxide, ozone, and wet air oxidation), energy sources (UV light, ultrasonic and heat) or catalysts (for example, titania, zinc oxide and Fenton reagent). Several drawbacks of AOPs still need to be addressed before industrial introduction at large scale, including the high cost for chemicals either as oxidants or energy sources, and the potentiality to handle large amounts of wastewater (Leong et al., 2014).

Semiconductor photocatalysts offer advanced oxidation processes (AOP) and are able to degrade a wide range of ambiguous refractory organic pollutants in waste water effectively, which has drawn much attention worldwide. Semiconductor photocatalysts like TiO₂, ZnO, Fe₂O₃ and Cu₂O are capable of generating highly reactive species under irradiation to mineralize organic compounds. Among the possible technologies to accomplish this task, novel and economical advanced oxidation techniques based on catalytic or chemical photooxidation are emerging as a promising alternative. Semiconductor mediated photocatalytic oxidation has been accepted as a promising alternative to the conventional methods because most of the pollutants can be completely mineralized to $CO₂$ and $H₂O$ with suitable catalysts (Zangeneh et al. 2015).

Commercial membranes are produced from two distinct classes of material: polymers consisting of organic material (e.g. polysulfone, regenerated cellulose, poliamide and polyvynilfluoride) or inorganic materials (mainly ceramics) (Coutinho et al., 2009). Ceramic membranes have advantageous properties when compared to polymeric membranes such as higher mechanical, chemical and thermal stability, which are basic requirements for adequate cleaning protocols and, consequently, higher membrane lifetime (Gebreyohanneset al., 2006; Mantzavinos and Kalogerakis, 2005). Furthermore, depending on the used materials, they can present a higher hydrophilicity (Hofs et al., 2011; Coutinho et al., 2009). The improvement of membrane hydrophilicity and fouling reduction through the use of membrane coatings with nanoparticles are currently a challenge (Chen et al., 2003).

1.2 Problem Statements

Bauxite is among the most important ore of aluminium and the existence of bauxite in Malaysia that has been discovered was reddish-brown color which indicated that it was naturally composed of heterogeneous material and comprises of more than one aluminum hydroxide minerals (Abdullah et al., 2016). Bauxite commonly comprises various metal oxides, including $Fe₂O₃$, $Al₂O₃$, $TiO₂$, $SiO₂$, and CaO and among these oxides, $Fe₂O₃$ and TiO₂ are the commonly used photocatalysts. Previously, researchers have demonstrated bauxite's capability as an adsorbent for removing pollutants (Yan el at, 2020; Shi et al, 2020(a); Shi et al, 2020(b)). Based on the above elicitation, bauxite was considered as the low-price and easily available material to be used as photodegradation photocatalysts. The evaluation and application of natural bauxite minerals as heterogeneous photocatalyst for degradation of organic pollutants are not investigated so far.

Ceramic membranes were successfully applied for treatment of various wastewaters including those from domestic and industrial sectors (Bhattacharya et al., 2015). One of the limiting factors for ceramic membranes includes fouling. These could be overcome by use of nanocomposite membrane which results in low fouling due to presence of inorganic nanomaterial, thus increasing membrane productivity. Metal oxide nanoparticles are widely used for removal of harmful contaminants and heavy metals from water. Nanocomposites are formed by combining one or two nanomaterials having unique properties resulting in desirable properties. The usage of membrane filtration for water treatment is on the rise due to increasingly stringent regulations regarding environmental safety. However, morphological control of the membrane surface to improve its photocatalytic reactivity for the degradation of organic pollutants remains a challenge.

Titanium dioxide $(TiO₂)$ and copper oxide (CuO) are the most studied photocatalysts due to their particular advantages, includes easy availability, low cost and high chemical stability due to its high oxidant capacity of the photogenerated holes, which gives a high photocatalytic activity (Fujishima et al., 2000). TiO₂ has high chemical stability and excellent biocompatibility along with photocatalytic and other optical and electrical properties. Use of $TiO₂$ in separation process arises from its anti-fouling and antimicrobial characteristics (Oun et al., 2017). TiO₂ as photocatalyst is known to remove estrogens and bisphenols. CuO on the other hand is used for removal of contaminants from wastewater and has wide application in the field of gas sensors, catalysis as well as show antibacterial activity. CuO was impregnated on surface of activated carbon for removal of endocrine disruption compound (EDC) like atrazine, caffeine and diclofenac respectively from drinking water. To encounter this challenge, immobilization nanoparticles by incorporating them into membrane while retaining the high functionality of the powdered form is a good solution.

To the recent days, the development of cost-effective ceramic hollow fibre membrane which exhibit high hydrophilic membrane surface, high porosity, and rather uniform pore size distribution, high water flux, lower fouling, and longer membrane lifetime by utilizing economical and abundant bauxite solely to replace

the usage of pure alumina are limited and not widely studied. Therefore, with all these aforementioned pros, it makes bauxite as a very proficient raw material for cost-effective membrane development. Photocatalytic ceramic membranes have attracted considerable attention for wastewater treatment. Among the photocatalysts used, titanium dioxide (TiO_2) and copper oxide (CuO) are considered efficient because of their photoactivity, physical and chemical stability, low cost and easy access. However, the requirement of separation of the suspension catalyst particles hinders the wide application of photocatalytic reaction systems, thus, a submerged ceramic membrane photocatalytic reactor using membrane was developed to separate the catalyst from the effluent, however, the problem of membrane fouling happened occasionally. The above problems can be avoided in photocatalytic reactors where the catalytic particles are immobilized on a support or carrier. Hence, it is promising to employ the $TiO₂$ and CuO on the membrane for removal of recalcitrant compound like BPA in the water. Further membrane structure modifications could still be tested to enhance the membrane efficiency and the performances evalution of the fabricated membrane towards photodegradation of organic contaminants can be studied.

1.3 Research Objectives

The main objective of this study is to develop hydrophilic, photocatalytically active hollow fibre membrane from bauxite powder for degradation of bisphenol A in the wastewater. The specific objectives of the research are as follows:

- 1. To examine the effect of thermal treatment on the characteristics of raw bauxite powder and photocatalytic activity of bauxite powder.
- 2. To study the influence of different bauxite loading and sintering temperatures on the development of microfiltration bauxite based hollow fibre membrane (BHFM) in terms of structural, physical, chemical and filtration properties.
- 3. To investigate the composition of photocatalyst of sintered BHFM by surface functionalization of titanium dioxide $(TiO₂)$ and copper oxide (CuO) through hydrothermal method and to correlate the photocatalytic efficiency of modified BHFM on the degradation of synthetic BPA wastewater under UV and visible light as well as the membrane repeatibility study of BHFM.

1.4 Research Scope

The present study is carried out to investigate the degradation of BPA from wastewater by photocatalytic BHFM. The scopes of study have been identified and are listed below:

For Objective 1:

- (a) Investigating the effect of thermal treatment by exposing raw bauxite powder under heat temperature ranged from 600 °C to 1000°C on the characterization of bauxite powder.
- (b) For physical characterization, observing the morphological structure of the bauxite using FESEM and EDX mapping.
- (c) Analyzing the chemical composition contains in bauxite as well as the energy gap and effective surface area for further application through XRD, XRF, BET, AFM, MIP and UV-VIS-NIR.

For Objective 2:

- (a) Varying the ceramic content from 45 wt% to 55 wt% of bauxite to observe the morphological structure of the BHFM upon selection of the best membrane.
- (b) Fabricating bauxite based hollow fibre membrane (BHFM) by using phase inversion based spinning technique, followed by the sintering process at the temperature of 1250° C to 1450° C.
- (c) Characterizing the morphology of BHFM via SEM, EDX and AFM, mechanical strength, contact angle, mercury intrusion test and XRD.
- (d) Evaluating the performances of BHFM characterization by conducting pure water permeation test and solute rejection test.
- (e) Conducting photocatalytic performance on fabricated BHFM under both visible and UV light irradiation with various BPA concentrations (10 mg/L, 20 mg/L and 30 mg/L).

For Objective 3:

- (a) Conducting the surface modification of BHFM (selected BHFM based on Objective 2) to improve the photocatalytic properties of the membrane using hydrothermal method by varying the duration of hydrothermal reaction form 2.5h, 5.0h and 7.5h using titanium dioxide $(TiO₂)$ and copper oxide (CuO)
- (b) Characterizing the morphology of decorated $TiO₂$ BHFM and CuO BHFM in terms of morphology using SEM, elemental composition using EDX, crystallinity phase using XRD, surface roughness using AFM, mechanical strength, contact angle measurement and water permeation test.
- (c) Developing photocatalytic membrane reactor (PMR) using both visible and UV light to observe the photocatalytic performance of modified $TiO₂$ and CuO BHFM (with hydrothermal time 5.0h) on the degradation of BPA at concentration of 10 mg/L, 20 mg/L and 30 mg/L.
- (d) Comparing the BPA degradation rate between decorated $TiO₂ BHHM/CuO$ BHFM with pristine BHFM and commercial ceramic membrane representative - pristine and decorated CuO – alumina hollow fiber membrane (AHFM)
- (e) Identifying and determining the intermediate product through high pressure liquid chromatography (HPLC) for degradation of BPA using CuO BHFM.
- (f) Conducting reusability test of modified BHFM by exposing used membrane under the UV light and observe the performance of the membrane after several times of usage.

1.5 Novelty of Study

The outcome of this study is going to provide an early insight on the potential of ceramic photocatalytic membrane as a promising technology for complete degradation of BPA in the water. The hybrid function of this BHFM allows the separation and degradation to occur simultaneously which latter on contribute to rapid treatment process. High distribution of photocatalyst on the surface of BHFM after surface functionalization gives great advantage as it can be used under both UV

and visible light exposure and even under poor indoor lightning, thus prolong the lifetime of BHFM due to less fouling towards the membrane. The information gained in this study is able to give a new shine in the direction of solving the global crisis on the depletion of water source which getting worst while the demand of clean water keep increasing. Thus, the recovery of clean water using photocatalysis of BHFM is gaining serious considerations with the development of the novel recovery technologies.

1.6 Organization of Report

This thesis is organized into eight chapters which describe original works on the fabrication of photocatalytic ceramic hollow fibre membrane from bauxite with different bauxite loading and sintering temperature for removal of BPA and its detrimental effects in the wastewater.

Chapter 1 briefly introduces on the general information of the research and rising issues that led to this study. Four objectives of the study are decided and the scopes of study are completed upon attaining all the objectives. Then, this chapter is resume with thesis outline and chapter summary. **Chapter 2** discusses on a comprehensive literature review regarding bauxite in Malaysia and its availability and existing component in this mineral. This chapter also deliberates about the development of ceramic membrane and photocatalysis process as well as previous study of the degradation BPA using various technologies. In **Chapter 3**, all the materials, instruments and methodologies used throughout this study is discussed. Complete research framework and comprehensive illustrated working procedures from thermal treatment of the raw bauxite powder, fabrication of BHFM with different ceramic loading, characterization techniques and BHFM performance evaluation are describe in details.

All the result and discussion are covered from Chapter 4 till Chapter 7. **Chapter 4** focuses on the characterization of bauxite powder as new potential photocatalyst and the effect of thermal treatment on the bauxite powder behaviour is revealed in this chapter. **Chapter 5** presents the feasibility study of bauxite based hollow fibre membrane (BHFM) and the effect of various ceramic loading and sintering temperature towards the characterization of the pristine membrane in terms of morphological structure and chemical composition. The best membrane to be further used in this study is also being decided in this chapter.

In **Chapter 6,** the surface functionalization conducted on the membrane surface to enhance the photocatalytic properties of fabricated BHFM is elaborated. Two types of nanoparticles that responsible in this study which is copper oxide (CuO) and titanium dioxide (TiO₂) are decorated and the changes due to their deposition are comprehensively discussed in this chapter. **Chapter 7** revealed the effect of BPA concentration and different light source (visible and UV) towards BPA degradation. The effectiveness of modified nanoparticles at the outer layer was investigated in presence of UVA light and the BPA degradation rate is determined using Langmuir-Hinshelwood model. The intermediate products of BPA have been identified by using high performance liquid chromatography (HPLC) analysis. The selfcleaning ability of BHFM was also studied as well as the reusability of BHFM throughout this study. The best performance BHFM on the removal of BPA is revealed in this chapter.

To conclude this thesis, general conclusions on this study and recommendation for future direction have been listed in **Chapter 8.**

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- 1**. Ismail, N. J.**, Othman, M. H. D., Kamaludin, R., Esham, M. I. M., Ali, N. A., Rahman, M. A., & Bakar, S. A. (2019). Characterization of Bauxite as a Potential Natural Photocatalyst for Photodegradation of Textile Dye. Arabian Journal for Science and Engineering, 44 (12), 10031-10040. [IF = 1.711]
- 2. **Ismail, N.J.**, Othman, M.H.D., Abu Bakar, S. Jaafar, J., & Rahman, M. A. (2020). Fabrication of Ceramic, Hollow-Fiber Membrane: The Effect of Bauxite Content and Sintering Temperature. Clays and Clay Minerals. $68, 309-318$. [IF = 1.679]
- 3. **Ismail, N. J**., Othman, M. H. D., Abu Bakar, S., Sheikh Abdul Kadir, S. H., Abd Aziz, M. H., Pauzan, M. A. B., A Rahman, M. (2020). Hydrothermal synthesis of TiO2 nanoflower deposited on bauxite hollow fibre membrane for boosting photocatalysis of bisphenol A. Journal of Water Process Engineering, 37, 101504. $[IF = 3.465]$
- 4. Esham, M., Othman, M., Ismail, A., Rahman, M., Jaafar, J., & **Ismail, N.J.** (2019). Effect of sintering temperature of bauxite hollow fiber membrane on flexural strength and water permeability. Malaysian Journal of Fundamental and Applied Sciences, 15, 190-193.
- 5. Raffi, A. A., Rahman, M. A., Salim, M. A. M., **Ismail, N. J.,** Othman, M. H. D., Ismail, A. F., & Bakhtiar, H. (2020). Surface treatment on polymeric polymethyl methacrylate (PMMA) core via dip-coating photopolymerisation curing method. Optical Fiber Technology, 57, 102215.
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BOOK CHAPTER

- 1. Othman M.H.D., Tai Z.S., Usman J., **Ismail N.J**., Rahman M.A., & Jaafar J. (2021) Oily Wastewater Treatment. In: Inamuddin, Ahamed M.I., Lichtfouse E. (eds) Water Pollution and Remediation: Organic Pollutants. Environmental Chemistry for a Sustainable World, vol 54. Springer.
- 2. Othman M.H.D., Adam M.R., Kamaludin R., **Ismail N.J.,** Rahman M.A., & Jaafar J. (2021) Advanced Membrane Technology for Textile Wastewater Treatment. In: Zhang Z., Zhang W., Chehimi M.M. (eds) Membrane Technology Enhancement for Environmental Protection and Sustainable Industrial Growth. Advances in Science, Technology & Innovation (IEREK Interdisciplinary Series for Sustainable Development). Springer.