LANTHANUM ORTHOFERRITE-CHITOSAN NANOCOMPOSITE FOR REACTIVE BLACK 5 DYE REMOVAL

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DEDICATION

This thesis is dedicated to my mother, my wife, and my family who taught me that the best kind of knowledge to have is that which is learned for its own sake.

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ABSTRACT

Presence of reactive dyes such as reactive black 5 (RB5) in wastewater severely interfere the photosynthetic function of plants due to low light penetration, leading to reduction in the concentration of dissolved oxygen used by aquatic organisms to breathe. This will also affect the quality of freshwater used in our everyday lives as reactive dyes are known to be toxic and carcinogenic to human if consumed in large quantity. One promising way to eliminate dyes in wastewater is by photocatalysis process by perovskite-like nanosized material such as lanthanum orthoferrite ($LaFeO_3$). However, due to susceptibility of $LaFeO_3$ to agglomerate because of high interparticle surface energy, scattering the nanoparticles onto a support material is believed to be an effective way. Thus, a new $LaFeO_3$-chitosan nanocomposite, LC15 was successfully fabricated in this work based on chemical precipitation methods. Characterization using X-Ray diffraction analysis showed that there was no change in crystallinity of $LaFeO_3$ nanoparticles when integrated with chitosan, while the Fourier Transform Infrared Spectroscopy confirmed the formation of $LaFeO_3$-chitosan nanocomposites by strong hydrogen bonding. Transmission electron microscopy verified the nanocrystalline structure of synthesized $LaFeO_3$ while field emission scanning electron microscopy and energy dispersive X-Ray spectroscopy demonstrated good distribution of $LaFeO_3$ on chitosan matrices along with changes in elemental composition of LC15 nanocomposites. Brunauer–Emmett–Teller and Barrett-Joiner Halenda analyses exhibited reduction in specific surface area and increased average pore radius of LC15 compared to pristine $LaFeO_3$, while UV-vis diffuse reflectance spectroscopy revealed reduction of band gap value for LC15. Apart from that, both adsorption and photocatalytic activity LC15 were also studied by varying the pH of synthetic wastewater, loading of nanocomposites, and initial concentration of RB5 dye. These studies were important to understand the behaviour of the sample and to determine the optimal condition for maximum synergistic action of $LaFeO_3$-chitosan nanocomposite onto RB5 dye. Following that, the reusability study was also performed in order to recognize the ability of LC15 nanocomposite to be used in real life application. Finally, the photocatalytic pathways for total removal of RB5 dye were also proposed based on species trapping experiment. Based on this study, LC15 nanocomposite showed the most prominent characteristics with high synergistic removal of RB5 dye at optimum conditions (pH 6, 2g/L loading and 30 mg/L of initial RB5 dye concentration). Moreover, the reusability experiment confirmed the stability of the nanocomposite with no dramatic changes occurred to their chemical structure, while the involvement of reactive oxygen species and positive vacant holes were established in species trapping experiment.

ABSTRAK

Kehadiran pencelup reaktif seperti hitam reaktif 5 (RB5) dalam air kumbahan mengganggu fungsi fotosintetik tumbuhan kerana penembusan cahaya yang rendah, menyebabkan pengurangan oksigen terlarut yang digunakan oleh organisma akuatik untuk bernafas. Ini juga akan mempengaruhi kualiti air tawar yang digunakan dalam kehidupan seharian kita kerana pencelup reaktif diketahui beracun dan karsinogenik kepada manusia jika terhadam dalam kuantiti yang banyak. Salah satu cara yang terbaik untuk menghapuskan pencelup dalam air sisa adalah dengan proses pemangkinan cahaya oleh bahan perovskit bersaiz nano seperti lanthanum ortoferit ($LaFeO_3$). Walau bagaimanapun, disebabkan kecenderungan $LaFeO_3$ untuk bergumpal kerana tenaga permukaan antara partikel yang tinggi, penyebaran partikel nano ke bahan sokongan dipercayai merupakan cara yang berkesan. Oleh itu, komposit nano $LaFeO_3$-kitosan, LC15 telah berjaya dihasilkan dalam kajian ini berdasarkan kaedah pemendakan kimia. Pencirian menggunakan analisis pembelauan sinar-X menunjukkan bahawa tiada perubahan terhadap pengkristalan partikel nano $LaFeO_3$ apabila digabungkan dengan kitosan, manakala spektroskopi jelmaan inframerah Fourier mengesahkan pembentukan komposit nano $LaFeO_3$-kitosan dengan ikatan hidrogen yang kuat. Seterusnya, mikroskopi elektron penghantaran mengesahkan struktur kristal nano $LaFeO_3$ yang disintesis, manakala imej mikroskop elektron imbasan pancaran medan dan spektroskopi penyebaran tenaga sinar-X menunjukkan taburan $LaFeO_3$ yang sekata pada matriks kitosan bersama dengan perubahan komposisi unsur komposit nano LC15. Analisis Brunauer – Emmett – Teller dan Barrett-Joiner Halenda menunjukkan pengurangan kawasan permukaan tertentu dan peningkatan radius purata LC15 berbanding $LaFeO_3$, sementara spektroskopi refleksi serapan UV-Vis menentukan pengurangan nilai sela jalur untuk LC15. Selain itu, kedua-dua aktiviti penjerapan dan fotobermangkin LC15 juga dikaji dengan mengubah pH air sisa sintetik, muatan komposit nano, dan kepekatan awal pencelup RB5. Kajian-kajian ini penting untuk memahami sifat sampel dan untuk menentukan keadaan optimum untuk tindakan sinergi maksimum komposit nano $LaFeO_3$-kitosan ke pencelup RB5. Setelah itu, kajian penggunaan semula juga dilakukan untuk mengenali kemampuan komposit nano LC15 untuk digunakan dalam aplikasi sebenar. Akhirnya, laluan fotobermangkin untuk penyingkiran pencelup RB5 juga dicadangkan berdasarkan eksperimen pemerangkapan spesies. Berdasarkan kajian ini, komposit nano LC15 telah menunjukkan ciri-ciri paling menonjol dengan penyingkiran pencelup RB5 yang tertinggi pada keadaan optimum (pH 6, muatan 2 g/L dan 30 mg/L kepekatan awal pencelup RB5). Tambahan lagi, eksperimen kebolehgunaan semula mengesahkan kestabilan komposit nano tanpa perubahan dramatik terhadap struktur kimianya, sementara penglibatan spesies oksigen reaktif dan lubang kosong positif dikenal pasti dalam eksperimen pemerangkapan spesies.

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LIST OF ABBREVIATIONS

$LaFeO_3$	-	Lanthanum Orthoferrite
TiO_2	-	Titanium Dioxide
RB5	-	Reactive Black 5
COD	-	Chemical Oxygen Demand
BOD	-	Biological Oxygen Demand
AOPs	-	Advanced Oxidation Processes
UV	-	Ultraviolet
UV-vis	-	Ultraviolet-Visible Light
LED	-	Light Emitting Diodes
mL	-	Millilitre
mg/L	-	Milligram per Litre
cm^{-1}	-	Per Centimetre
ppm	-	Parts per Million
nm	-	Nanometre
S_{BET}	-	Specific surface area by BET method
Max_r	-	Maximum pore radius distribution (BJH)
Avr_r	-	Average pore radius (BJH)
V_p	-	Specific pore volume measured at $p/p^0 = 0.99$

LIST OF SYMBOLS

Wt%	-	Weight percent
eV	-	Electronvolt
λ	-	Wavelength
θ	-	Bragg's angle
π	-	Pi
E_g	-	Band Gap Energy
e^-	-	Electron
h^+	-	Positive Vacant Hole
R^2	-	Correlation Coefficient
k	-	Rate constant
hv	-	Photon energy
C_0	-	Initial Concentration
C_t	-	Concentration at Given Time
°C	-	Degree Celsius

CHAPTER 1

INTRODUCTION

1.1 Research Background

Textile industries normally use large amount of water in their processes, especially in drying and washing processes. Approximately 21 to 377 m^3 of water is consumed for each tonne of textile product, while the chemical consumption made up between 10 to 100 percent of the weight of the clothes. Annually, it is estimated that 7 x 10^5 tonnes of dyestuffs are produced, leading to 280,000 tonnes of textile effluents containing dyes discharged into the aquatics (Van Hoa *et al.*, 2016; Asghar *et al.*, 2015). As a result, the large amount of coloured effluent having high level of Chemical Oxygen Demand (COD) (150-10,000 mg/L), Biological Oxygen Demand (BOD) (100-4000 mg/L) and instable pH discharged negatively impacted aquatic flora and fauna. This occurs as presence of dyes in wastewater severely interfered the photosynthetic function of plants due to low light penetration, thus reducing the concentration of dissolved oxygen used by aquatic organisms to breathe. Subsequently this will also affect the quality of freshwater used in our everyday lives as certain dyes are known to be toxic and carcinogenic to human if consumed in large quantity over period of times (Holkar *et al.*, 2016).

Widely used in textile industries, reactive dyes are chosen due to their high stability with respect to washing. These water soluble and non-biodegradable compounds comprised of one or more azo bonds (-N=N-) proved to be difficult to be treated as they have low absorbability. Among them, Reactive Black 5 (RB5) dyes has been one of the most utilized dye, contributing to more than 50% of total reactive dyes demand (Semiz, 2019; Garg *et al.*, 2016). As reactive dyes are designed to resist degradation, these aromatic and heterocyclic compounds need proper treatment strategies to meet the requirements set by the government, which in Malaysia is the Department of Environment (DOE) under Ministry of Energy, Science, Technology, Environment and Climate Change. According to the Fifth Schedule of Acceptable Conditions for Discharge of Industrial Effluent for Mixed Effluent of Standard A and B from Environmental Quality (Industrial Effluents) Regulation 2009, the allowable limit for wastewater containing colorant or dyes were set at 100 to 200 ADMI with BOD_5 at 20°C less than 40 mg/L.

According to Koyuncu and Güney (2013), there are two conventional methods for treatment of textile industry wastewaters, namely the end-of-pipe treatment and segregation methods. As the name applied, the end-of-pipe treatment is carried out at the end of the mixed wastewater stream, while the later is by segregating the wastewater streams and applying different treatment steps to each stream. Differ as they may be, both treatments comprised of either physical, chemical, biological or mixture of them in efficiently treat the wastewater. Methods such as adsorption, membrane separation/filtration, coagulation-flocculation, chemical oxidation, ozonation, ultraviolet (UV) treatment and use of bacteria are among commercially available methods (Semiz, 2019; Katheresan *et al.*, 2018; Çınar *et al.*, 2017; Erdem *et al.*, 2016; Wojnarovits and Takacs, 2008). Comes with sets of advantages and disadvantages that will be discussed in depth later, they are being improvised year after year by many researchers around the world to find the ultimate solution for efficient dye wastewater treatment.

Among that, a branch of chemical treatment known as advanced oxidation process, or simply known as AOPs is potentially to be one of the best treatment available till date for total degradation of organic compounds, including reactive dyes. AOPs are widely recognized as highly efficient treatments for recalcitrant wastewater that employ the degradation of organic pollutants by forming hydroxyl radicals which are highly reactive and non-selective (Oller *et al.*, 2011). These include the treatment of wastewater with ozone, UV light, and Fenton-like treatment that uses reagent such as hydrogen peroxide, H_2O_2 to carry out degradation of organic compounds. Besides that, photocatalysis is another AOPs that can be deployed for RB5 dye wastewater treatment (Garg *et al.*, 2016).

Being studied either as homogenous (liquid state oxidants) or heterogenous (solid state oxidants) process, many researches have been carried out in order to developed most advanced photocatalysts since the 1970s As for this research, it will be focusing on the application of heterogenous photocatalysts. In general, heterogenous photocatalysis is a process to degrade, destroy and permanently remove organic contaminants in wastewater that occurs when certain wavelengths of light interact with light-reactive materials called photocatalysts – a suspension of nanoparticles or nanocomposites, usually wide-bandgap semiconductors. The activation of these metal oxides promoted the photo excitation of electrons from the valence band to the conduction band, which generates ·OH free radicals via secondary reaction that will subsequently degrades the organic contaminants found (Ayati et al., 2014).

However, the application of photocatalytic nanoparticles in wastewater treatment may not be favourable by many as the nanoparticles tend to agglomerate, especially if the catalysts used are in amorphous form, thus reducing the capability to carry out catalytic process. Thus, by combining the powerful degradation mechanism of photocatalysis and the importance of adsorption capability for efficient wastewater treatment, the synthesis of photocatalyst-adsorbent nanocomposite could be a promising method that can adsorb and degrade organic compounds such as RB5 dye in the presence of UV/visible light irradiation (Peng *et al.*, 2016). This synergistic combination will not only preserve the advantageous components possessed by both treatment, but will also overcome drawbacks such as low absorptivity and rapid recombination of photogenerated electrons (Keane *et al.*, 2011). Moreover, the combination will significantly increase the surface area of the composite, providing more active sites for adsorption and degradation of pollutants (Gao *et al.*, 2016). Finally, the introduction of multiple functional groups will promote ionic exchange between the composites and specific pollutants, increasing their adsorption efficiency and thus, increasing the percentage of adsorbed pollutants to be degraded by photocatalyst process (Mansur *et al.*, 2014).

1.2 Problem Statement

One of the most promising and most studied photocatalyst within AOPs treatment is titanium dioxide, TiO_2. TiO_2 is chosen by many researchers as it possesses several good properties such as low-cost, high chemical stability, commercially available, non-toxic, and environmentally friendly. However, pristine TiO_2 can only absorbed light in ultraviolet region due to their large band gap of 3.2 eV. Multiple researches were carried out to narrow the band gap, thus allowing the TiO_2 particles to be activated by visible light (Leong et al., 2014).

On the other hand, perovskites, a collective name for materials with orthorhombic crystalline structure of ABO_3 (A=typically rare-earth elements; B=transition metals) is a promising candidate of photocatalysts. Lanthanum orthoferrite, $LaFeO_3$ is an example of compound with perovskite structure that had been studied for their photocatalytic activity in wastewater treatment. $LaFeO_3$ possessed a narrow band gap of 1.86 to 2.36 eV, making it efficient in visible light region as compared to TiO_2. Furthermore, its stability and non-toxicity properties made it a promising material in wastewater treatment (Thirumalairajan *et al.*, 2013).

However, the susceptibility of $LaFeO_3$ to agglomeration hinders its application in wastewater treatment researches and studies. This is mostly due to its high surface energy that interacts between the particles. Thus, by scattering the nanoparticles onto a support material that concurrently act as an effective adsorbent is one of the effective ways to reduce the agglomeration of the nanoparticles. Besides, the support material will also provide heterojunction for electron and holes that limit the charge recombination. There have been many support materials uses to support the nanoparticles (Peng et al., 2016).

One of the perfect candidates to achieve this goal is the application of chitosan. Chitosan is the most important derivative of chitin, which is the second most abundant natural polymer behind cellulose. Mainly extracted from crustaceans such as shrimp and crabs, chitin can also be found in the exoskeleton of arthropods or in the cell walls of yeasts and fungi as ordered crystalline microfibrils. Chitosan possessed excellent non-toxic, anti-microbial, biocompatible and biodegradable properties that are proven to be advantageous for wastewater treatment (Shukla et al., 2013).

Chitosan has been widely studied for water and wastewater treatment due to high functional groups content (acetamido group, both primary hydroxyl and secondary hydroxyl group and amino group). The presence of these functional groups subsequently contributed to good adsorption capability of this material. These also provide a good base for interaction with other materials such as metal oxides where multiple chemical bonds can be made to effectively improves the desired parameters, such as lowering the band gaps or improving distribution of nanoparticles in heterogenous photocatalysis studies (Saravanan *et al.*, 2018; Al-naamani *et al.*, 2017; Ahmed *et al.*, 2017).

In this study, $LaFeO_3$-chitosan nanocomposites were prepared by chemical precipitation method. This method is selected due to its simplicity, required no specialized equipment, time-saving and easily replicated. The ratio of chitosan and $LaFeO_3$ were varied accordingly in order to determine the difference in physiochemical properties and synergistic adsorption/photocatalytic performance of the nanocomposites.

The dominants active species generated during the photocatalytic process were identified via scavenger experiment. Previous studies on the scavenger experiment have shown that the dominant active species could be different for each dye molecules and photocatalyst (Chiu *et al.*, 2019). Therefore, this study is important to establish the mechanism of photodegradation of RB5 dye by nanocomposite $LaFeO_3$-chitosan. Apart from that, the operational parameter for photocatalytic degradation such as pH, nanocomposite loading and initial concentration of RB5 dye is further investigated to establish highly efficient photocatalytic degradation of RB5. This research is important in that it provided additional knowledge on the functions of chitosan adsorbents in enhancing $LaFeO_3$ exceptional photocatalytic degradation of organic pollutants such as dyes to improve the feasibility of AOP technologies in wastewater treatment.

1.3 Objectives of Study

The aim of this study is to fabricate $LaFeO_3$-Chitosan nanocomposites for synergistic act of adsorption and photocatalytic degradation of Reactive Black 5 dye from synthetic textile wastewater under visible-light irradiation. The fabrication of the nanocomposites is also aimed to reduce the risk of $LaFeO_3$ agglomeration due to their high surface energy.

In order to achieve that, there are three specific objectives of this study, which were:

1. To assess the effects of $LaFeO_3$:chitosan ratio prepared via chemical precipitation method on physicochemical properties and photocatalytic degradation of RB5 dye in synthetic textile wastewater.
2. To determine the effect of various operating parameters (pH, catalyst loading and initial concentration of RB5 dye) towards the chosen nanocomposite performance, and subsequently the reusability of the nanocomposite.
3. To determine and deduce the removal mechanism or pathways of RB5 dye by chosen $LaFeO_3$-chitosan nanocomposite via scavenging experiments.

1.4 Scopes of Study

1. Synthesizing $LaFeO_3$ using gel combustion method via citric acid route at 200°C without any subsequent calcination process at high temperature.

2. Characterizing the physicochemical properties of $LaFeO_3$ using several characterization analysis such as X-ray diffractometer (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Transmission Electron Microscopy (TEM), field emission scanning electron microscopy (FESEM), Energy-dispersive X-ray spectroscopy (EDS), Brunauer-Emmet-Teller (BET), and UV-vis diffuse reflectance spectroscopy (UVDRS) analysis

3. Fabricating $LaFeO_3$-chitosan nanocomposites via chemical precipitation method by varying the ratio of $LaFeO_3$:chitosan ($LaFeO_3$/chitosan in weight percent (wt%): 85/15, 75/25, 65/35 and 55/45), followed by preliminary studies to select the best two nanocomposites.

4. Characterizing the physicochemical properties of chitosan and chosen $LaFeO_3$-chitosan nanocomposites using several characterization analysis such as X-ray diffractometer (XRD), Fourier Transform Infrared Spectroscopy (FTIR), field emission scanning electron microscopy (FESEM), Energy-dispersive X-ray spectroscopy (EDS), Brunauer-Emmet-Teller (BET), and UV-vis diffuse reflectance spectroscopy (UVDRS) analysis, and subsequently determine the most suitable nanocomposite for effect of operating parameters evaluation.

5. Evaluating the synergistic adsorption-photocatalytic removal of RB5 dye by chosen $LaFeO_3$–chitosan nanocomposite by varying the operating parameters, including the pH of synthetic RB5 dye wastewater (pH 3, pH 6, and pH 9), nanocomposites loading (1g/L, 2g/L and 3g/L) and initial concentration of RB5 dye (30 mg/L, 50 mg/L and 70 mg/L). It was then followed by reusability experiments (5 cycles of reusability).

6. Determining and proposing the possible mechanism involved in removal of RB5 dye by carrying out radicals' scavenging experiment.

1.5 Significance of Study

In this study, $LaFeO_3$ will be integrated with chitosan to produce high performance nanocomposite for Reactive Black 5 dye removal via synergistic adsorption/photocatalytic activities. Findings of this study will provide the knowledge to the scientific community on chitosan roles in improving the physicochemical properties of $LaFeO_3$. This study will also provide additional knowledge on the exceptional synergistic performance possessed by $LaFeO_3$ and chitosan nanocomposite at optimum ratio under visible light irradiation, and the reusability of it. By providing these insights, it is believed that the real-life application of heterogenous photocatalysts may soon become a reality in the near future.

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LIST OF PUBLICATIONS

Indexed Journal

1. **Aizat, A**., Aziz, F., Mohd Yusop, M. Z., Jaafar, J., Yusof, N., Wan Salleh, W. N., Ismail, A. F. 2019. Decolorization of Reactive Black 5 dye using gel combustion synthesized $LaFeO_3$ nanoparticles. *Malaysian Journal of Fundamental and Applied Science,* 15(3*),* pp 462-466.

2. **Aizat, A**., Aziz, F., Mohd Sokri, M. N., Sahimi, M. S., Yahya, N., Jaafar, J., Wan Salleh, W. N., Yusof, N., Ismail, A. F. 2019. Photocatalytic degradation of phenol by $LaFeO_3$ nanocrystalline synthesized by gel combustion method via citric acid route. *SN Applied Sciences,* 1(91).

Book Chapter

1. **Aizat, M. A.** and Aziz, F. 2019. Chitosan Nanocomposite Application in Wastewater Treatments in Ahsan, A. and Ismail, A.F. (eds.) Nanotechnology in Water and Wastewater Treatment. Theory and Applications. Oxford. Elsevier, pp. 243-265.