EFFECT OF PROCESSING PARAMETER ON HYDROLYSIS METHOD TO PRODUCE TITANIUM DIOXIDE FOR THERMAL SPRAY PROCESS

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2022

EFFECT OF PROCESSING PARAMETER ON HYDROLYSIS METHOD TO PRODUCE TITANIUM DIOXIDE FOR THERMAL SPRAY PROCESS

This report is submitted in accordance with requirement of the University Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Hons.)

by

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2022

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Sesi Pengajian: **2021/2022 Semester 2**

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as partial fulfilment of the requirement for Degree of Manufacturing Engineering (Hons). The member of the supervisory committee is as follow:

....

(Dr. Toibah Binti Abdul Rahim)

ABSTRAK

Titanium dioksida (TiO_2) adalah serbuk putih yang digunakan untuk pelbagai aplikasi. Ia telah menjadi bahan yang paling terkenal kerana sifat uniknya, yang dibolehkan dalam penggunaan pigmen, salutan, makanan, dan yang paling penting, ianya salah satu komponen yang paling memberangsangkan dalam mekanisme fotokatalisis. Salah satu aplikasi fotokatalisis alam sekitar yang paling terkenal adalah untuk pembersihan udara atau sebagai lapisan untuk permukaan bangunan. Pemangkin foto aplikasi untuk bahan lapisan menunjukkan hasil yang menjanjikan kerana permukaan yang dilapisi dengan titanium dioksida boleh bertahan dari kulat selama bertahun-tahun. Kajian ini bertujuan untuk menganalisis kesan parameter proses seperti kadar kelajuan semasa mengacau dan masa hidrolisis terhadap proses hidrolisis semasa menghasilkan TiO_2. Serbuk TiO_2 yang disintesis akan digunakan sebagai bahan untuk proses semburan haba. Semasa kaedah hidrolisis, titanil sulfat dihidrolisiskan dengan air suling pada tempoh pengacauan yang berbeza (4 jam, 6 jam, dan 8 jam) pada 350 rpm dan kadar kelajuan yang berbeza (60 rpm, 125 rpm dan 350 rpm) selama 8 jam . Mendakan putih yang terbentuk daripada proses hidrolisis akan dikeringkan di dalam ketuhar selama 10 jam pada suhu 120ºC. Semua sampel telah dianalisis menggunakan XRD, PSA, FTIR, dan SEM. XRD mengesahkan hanya struktur anatase dalam semua serbuk TiO_2 yang disintesis. Keputusan menunjukkan bahawa apabila sampel dikacau selama 8 jam pada 350 rpm mempunyai zarah begumpal paling terbaik dengan saiz zarah 15.51 µm dan saiz kristal 19.28 nm, menjadikannya paling sesuai untuk aplikasi semburan haba. Imej PSA dan SEM menunjukkan bahawa peningkatan kadar kacau akan meningkatkan proses penggumpalan dan menghasilkan taburan saiz yang lebih sekata. Imej SEM juga menunjukkan bahawa zarah-zarah tersusun padat dan mempunyai bentuk sfera apabila kadar pengacauan ditingkatkan. Kajian ini menunjukkan kepentingan parameter proses terhadap pembentukan struktur anatase bersaiz mikron yang bergumpal secara semula jadi dan membolehkan TiO_2 sebagai bahan penyemburan untuk proses semburan haba dan membolehkan proses fotokatalisis dan lapisan yang stabil dan memberangsangkan.

ABSTRACT

Titanium dioxide (TiO_2) is a white form powder primarily used for various applications. It has become the most famous substance due to its unique properties, which are allowed as pigments, coatings, foods, and most importantly, one of the most promising components in the photocatalysis mechanism. One of the most famous environmental photocatalysis applications is for air purification or as a coating for surfaces such as buildings. Application photocatalyst for coating faces shows promising results since the surface coated with titanium dioxide can sustain as mould free for many years. This study aims to analyze the effect of processing parameters such as the stirring speed and stirring time on the hydrolysis method to produce TiO_2. The as-synthesized TiO_2 powders will be used as the feedstock for thermal spray process. During the hydrolysis method, the titanyl sulphate was hydrolyzed with distilled water at different stirring times (4hrs, 6hrs, and 8hrs) at 350 rpm and different stirring speeds (60 rpm, 125 rpm and 350 rpm) for 8hrs. The white precipitate was formed from the hydrolysis process would be dried in an oven for 10hrs at 120^{o}C. All the samples were characterized using XRD, PSA, FTIR, and SEM. XRD confirmed the presence of anatase as the only phase in all the synthesized TiO_2 powders. The results show that when the sample was stirred for 8 hours at 350 rpm, the most agglomerated particles were produced with a 15.51 µm particle size and a 19.28 nm crystallite size, making it the best fit for thermal spray application. PSA and SEM images reveals that increased of stirring rates would enhance the agglomeration process and result in a more even distribution of sizes. SEM images also show that the particles are packed most densely and form spherical shapes with better agglomeration when the stirring rates are increased by increasing the stirring time and speed. This study demonstrated the significance of processing parameters on the development of naturally agglomerated micron-size anatase structures using TiO_2 as a feedstock for the thermal spray process, which can allow a stable and better performance during coating or photocatalysis applications.

ACKNOWLEDGMENT

In the name of ALLAH, the most gracious, the most merciful, with the highest praise to Allah that I manage to complete this final year project successfully without difficulty.

I want to express my gratitude to my respected supervisor, Dr Toibah Binti Abdul Rahim, for the tremendous mentoring throughout the project. Also, for her kind supervision, advice, and guidance, I was exposed to meaningful experiences throughout the study.

Last but not least, I would like to say thank you to my friends who gave me much motivation and cooperation in completing this report and always be there for me through thick and thins. They had given their great suggestion and comments throughout my research. Thanks for the great friendship.

Finally, I would like to thank everyone important to this FYP report and express my apology that I could not mention each one of you personally.

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LIST OF ABBREVIATIONS

1D	-	One-dimensional
3d	-	Three dimensional
Ag	-	Silver
Al_2O_3	-	Aluminium oxide
$AlCl_3$	-	Aluminum chloride
AOPs	-	Advanced Oxidation Process
ASEAN	-	Association of Southeast Asian Nations
BTX	-	Xylene
C	-	Carbon ion
C_4H_9OH	-	Butyl alcohol
CaO	-	Calcium Oxide
CB	-	Covalence Band
Cl^-	-	Chloride ion
Cl_2	-	Chloride
CO_2	-	Carbon Dioxide
Cr-MCM	-	Chromium Doped Mesoporous Crystalline
MolecularCVD	-	Chemical Vapour Deposition
DCAC	-	Dichloroacetate chloride
DFT	-	Density-functional theory
EDTA	-	Ethylenediamine tetraacetic acid
Eg	-	Example
EPR	-	Electron spin Resonance Spectroscopy
EY	-	Eosin Y
F^-	-	fluoride ion
FAO	-	Food and Agriculture Organization
Fe	-	Iron
Fe_2O_3	-	Ferric Oxide
Fe^{3+}	-	ferric ion

$FeCl_2$	-	Iron chloride
$FeCl_3$	-	Iron (III) chloride
$FeSO_4$	-	ferrous sulphate
$FeTiO_3$	-	Iron titanium oxide
FTIR	-	Fourier-Transform Infrared Spectroscopy
FTO	-	Fluorine-doped Tin Oxide
H^+	-	Hydrogen ion
H_2	-	Hydrogen
H_2O	-	Water vapour
H_2O_2	-	Hydrogen peroxide
H_2S	-	Hydrogen sulphide
H_2SO_4	-	Sulphuric Acid
HCl	-	Hydrochloric acid
HNO_3	-	Nitric Acid
HVOF	-	High Velocity Oxygen Fuel
IQAir	-	Air Quality
IR	-	Infrared radiation
MB	-	Methylene Blue
MgO	-	Methylglyoxal
N_2SO_4	-	Natrium Sulphate
NaCl	-	Natrium Chloride
NaF	-	Natrium Fluoride
NHE	-	Standard Hydrogen Electrode
NO	-	Nitrogen monoxide
NO_2	-	Natrium Dioxide
Np-TiO_2	-	Nanoparticulate titania
O^-	-	Super Oxide Radical
O^{2-}	-	Oxide anion
O_2	-	Oxygen
OECD	-	Organization for Economic Co-operation and Development
OH	-	Hydroxyl ion

PCA	-	Photocatalytic Activity
PCO	-	Heterogeneous Photocatalytic Oxidation
PEC	-	Photoelectrochemical
PFCs	-	PhotoFuelCells
pH	-	Potential Hydrogen
PM	-	Particulate Matter
POWS	-	Photocatalytic Overall Water Splitting
PSA	-	Particle Size Analyzer
P-type	-	Positively charged semiconductor
PVD	-	Physical Vapour Deposition
RB	-	Rose Bengal
RH	-	Organic pollutant degradation
ROS	-	Reactive Oxygen Species
SEM	-	Scanning Electron Microscopy
SiO_2	-	Silicon Dioxide
SO_4^{2-}	-	Sulphate ion
SPD	-	Spray Pyrolysis Deposition
SPS	-	Spray Pyrolysis Synthesis
SPS	-	Suspension Plasma Spray
TB	-	Tuberculosis
TCE	-	Trichloroethylene
Ti	-	Titanium
$Ti (OC_4H_9)_4$	-	Titanium butoxide
$Ti (Oi\text{-}Pr)_4$	-	Titanium isopropoxide
$Ti (SO_4)_2$	-	Titanium sulphate
Ti^+	-	Titanium cation
$TiCl_4$	-	Titanium tetrachloride
TiO_2	-	Titanium dioxide
TiO_2-PN	-	Titanium dioxide-based photocatalytic nanoparticles
TiO_6-	-	Octahedra in TiO_2
$TiOCl_2$	-	Titanium oxide ion
$TiOSO_4$	-	Titanyl sulphate
TRIRA-ESS	-	Transient Infrared Absorption-Excitation Energy

		Scanning Spectroscopy
TTIP	-	Titanium tetraisopropoxide
UV	-	Ultraviolet
UV-A	-	Ultraviolet Radiation
VB	-	Valence Band
$VOCl_3$	-	Vanadium oxytrichloride
VOCs	-	Volatile organic compounds
XRD	-	X-ray diffraction analysis
ZNO	-	Zinc Oxide

LIST OF SYMBOLS

%	-	percent
±	-	plus, and minus
µ	-	micro
Å	-	Angstrom (0.1 nanometer)
Atm	-	Standard atmosphere (101,325 Pascals)
C_{max}	-	maximum supersaturation
C_{min}	-	critical level of nucleation
C_s	-	solubility level of bulk
CuKα	-	Copper K-alpha
8.04 keVD	-	crystallite size
e	-	Electron
eV	-	electron energy
F	-	Concentration ration of effective acid
g	-	gram
g/L	-	gram per liter
g/m^3	-	gram per meter cubic
GHz	-	Giga Hertz
h	-	Planck's constant (6.62607004 x 10^{-34}
m^2 kg/s)h^+	-	holes photon
hr.	-	hour
K	-	Kelvin
K	-	Particle Size Factor
m	-	meter
m^2 kg/s	-	joule-seconds
mL	-	milliliter
n	-	nano
ºC	-	Degree Celsius
r*	-	critical radius

r_p	-	grown particle
rpm	-	revolutions per meter
v	-	Incoming light frequency
V	-	Volt
wt. %	-	weight percent
β	-	Full width at half maximum at the intensity peak
θ	-	Bragg angle
λ	-	wavelength

CHAPTER 1

INTRODUCTION

This chapter discussed the research's background and the problem statement, which summarizes an issue to be conveyed or a condition to be improved because of this research.Aside from, the objectives (the study's purpose or aims), scopes of study, and significance or importance of the study to industry and environment are included.

1.1 Research Background

Titanium dioxide (TiO_2) is a white powder based on nearly all human-created pigments (Manuel et al., 2021). Mineral ilmenite and mineral rutile are examples of this mineral. These are the same minerals that go into making titanium metal. TiO_2 can reflect visible and ultraviolet light; it is non-toxic and has no reactivity with other compounds (Pawar et al., 2018). When it comes to TiO_2, one of its significant advantages is that it can be used in various ways. There is a study conducted on the synthesis of TiO_2 using the hydrolysis method. As a result, various objects have a longer life span because of this uniqueproperty. When TiO_2 is used in paints, it can increase the life of plastics and protect wood and metal. It also can be manufactured in a much more delicate powder that is transparent and more effective at guarding against UV rays, making it a better option (Guo, 2017). These properties elevate its role in sunscreen formulations and make it a more pleasant application experience (Trivedi and Murase, 2017). Photocatalysis is utilized in environment and energy due to efficient photoactivity, high stability, and safety for the environment and humans (Moma and Baloyi, 2019).

Rutile, anatase, and brookite crystalline forms of titanium dioxide ionically link titanium (IV) and oxygen. They have three distinct crystal structures (Hanaor and Sorrell, 2010; Mironyuk et al., 2020; Blvd, 2002). Due to the high demand and widespread use of TiO_2, there are many methods to produce fine powder of TiO_2, such as hydrolysis, sol-gel, solvothermal, sonochemical or microwave-assisted, deposition, electrophoretic deposition, spray pyrolysis, and oxidation (Mironyuk et al., 2020; Nyamukamba et al., 2018). The most generally used photocatalyst for organic compound oxidative breakdown is anatase structure. This anatase phase is an effective photocatalyst for solar-energy conversion because of its high sensitivity to light. The crystalline structure and morphology of TiO_2 have a significant role in the uses of the material. Hence it is critical to creating synthetic methods that can produce TiO_2. The TiO_2 sizes and shapes must also be regulated. Titanium sulphates and organic have been used to produce anatase nanoparticles. These procedures have flaws since the final products will always contain chemical impurities or minor accessory phases. Anatase-rich TiO_2 nanoparticles are formed in organic hydrolytic processes, while brookite-rich nanoparticles cannot be eradicated by changing the reaction conditions. The use of anatase nanoparticles in photocatalytic reactions and other chemical processes could be compromised if brookite is present in small concentrations.

Titanium dioxide anatase nanocrystals were prepared in this study by hydrolysis method. Nano particles of anatase TiO_2 will be produced by meticulous reaction conditions. In this work, the effect of hydrolysis reaction temperature and stirring speed will be studied. X-ray diffraction analysis (XRD), Fourier-transform infrared spectroscopy(FTIR), scanning electron microscopy (SEM), and Particle size analysis (PSA) are used to characterize the synthesized TiO_2 powders which prepared for feedstock materials for thermal spray process.

1.2 Problem Statement

In this urban era, fast technological and world growth will negatively impact the environment, notably air pollution. Air pollution is primarily produced by traffic, fossil fuelcombustion, industrial emissions, and indoor air pollution, among other factors. According to IQAir, Malaysia was placed 50th in the world in 2019 with a PM2.5 rating of 19.36 g/m^3, placing its annual average in the 'moderately' polluted range. To be classified as moderatelypolluted, a city or country must have a PM2.5 reading of between 12.1 and 35.4 g/m^3, which Malaysia at the lower end of this range. However, as can be seen, there are months throughout the year that experience significant spikes in pollution. Such, both in terms of theyearly average rating and the causes and appropriate measures that may arise in terms of pollution levels compared to other countries in south-east Asia. Malaysia came in significantly higher than many of its ASEAN (Association of South-East Asian Nations) neighbors, surpassing countries such as Thailand, which came in 28th place in 2019, Vietnam (15th place), and Indonesia (6th place). Malaysia faces a plethora of pollution andhaze-related issues every year. After a year, its neighbors provide perspective on the progress made over the last few decades of industrial growth and that, despite numerous roadblocks on the path to cleaner air, pollution levels could eventually be significantly reduced. Thus, the effective way to combat air pollution is to employ photocatalysis.

Due to its unique redox characteristic, it is exceptionally effective in removing environmental pollutants. It is widely used in the oxidative elimination and disinfection of air pollutants (Reddy et al., 2011). Photocatalysts can also be used to coat TiO_2 using the thermal spray method. The feedstock material (powders, liquid precursors, dispersions) is injected into an enthalpic source (flame, plasma) via a carrier gas, where the particles are melted (the liquid precursor decomposed, respectively), after which they are accelerated and impacted on the substrate, where they solidify (Toma et al.,2008). Also, there are various functions, including wear and corrosion resistance and sliding properties and thermalconductivity. By applying a suitable coating, these properties can be tailored to meet specific requirements. Coatings range in thickness from 100 µm to 400 µm, depending on the material and application (Muzika et al., 2021). Anatase is mainly used for photocatalysis mechanisms because it is more stable than brookite (Ohtani et al., 1985; Murakami et al., 2009; Ismail et al., 2010; Kandiel et al., 2020; Addamo et al., 2006; Augugliaro et al.; 2009;Zhang et al., 2012), which is the structure can be produced by hydrolysis method. By hydrolysis method, the powder in micron size and naturally agglomerated will produce during synthesis for photocatalyst application for coating using thermal spray method since nanoparticles distribution tends to clog inside the hoses. However, the use of nanoparticle size for photocatalyst gives better performance. Usually, micron-sized agglomerated powder of nanostructured materials is used as spray materials. However, this aggregation requires a dry spray process which will cause extra processing costs. Therefore, a synthesis method that can produce naturally agglomerated powders in micron-sized is used in this study to eliminate the requirement of a dry spray process to produce spray materials for the thermal spray process. Also, this work will contribute to increasing the effectiveness of photocatalysts, which are a vital tool for reducing air pollution.

1.3 Objectives

The objectives as follows:

a) To analyze the effect of different stirring speeds and stirring time on hydrolysis method to produce titanium dioxide for thermal spray process.

b) To characterize the as-synthesized TiO_2 on the structural or phase, morphology, physical and chemical properties using SEM, XRD and FTIR.

1.4 Scope of Study

a) The titanium sulphate and distilled water mixed at 80 °C for 4,6 and 8 hours on the magnetic hot plate stirrer with different stirring speed of 60, 125 and 350 rpm rates, respectively.

b) Then the obtained white precipitate is washed with distilled water five times, and dried in a drying oven at 120℃ for 10 hr.