FORMATION OF TiO_2 NANOTUBULAR STRUCTURE IN FLUORINATED ETHYLENE GLYCOL ELECTROLYTES CONTAINING ADDITIVES BY ANODISATION

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by

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DECLARATION

I hereby declare that I have conducted, completed the research work and written the thesis entitle "Formation of TiO_2 Nanotubular Structure in Fluorinated Ethylene Glycol Electrolytes Containing Additives by Anodisation". I also declare that it has not been previously submitted for the award of any degree or diploma or other similar title of this for any other examining body or University.

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Mustaffa Ali Azhar Bin Taib

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LIST OF SYMBOLS

%	Percentage
<	Less than
>	More than
°	Degree
°C	Degree Celsius
° C/min	Degree Celsius per minute
[ ]	Concentration
θ	Bragg angle
2θ	Diffraction angle
λ	Wavelength
$^{\bullet}O_2^-$	Superoxide radical
$^{\bullet}OH$	Hydroxyls radical
$^{\bullet}OOH$	Hydroperoxyl radical
at%	Atomic percent
A	Ampere
Å	Angstrom (10^{-10} m)
cm	Centimetre
d	Thickness
E_c	Conduction band
E_g	Bandgap energy
E_v	Valence band
e^-	Electrons
e^-_{CB}	Conduction band electron
eV	Electron volt
g	Gram
h	Hour
h^+	Holes
hv	Photon energy
h^+_{VB}	Valence band hole
J	Current density
L	Litre

M	Molarity
m	Meter
mA	miliampere
mg	miligram
min	Minute
mL	Millilitre
mm	Millimetre
MW	Megawatt
nm	Nanometer (10^{-9} m)
µm	Micrometer (10^{-6} m)
ppm	Parts per million
s	Second
T	Temperature
V	Voltage
Vö	Oxygen vacancies
wt%	Weight percent

LIST OF ABBREVIATIONS

a.u.	Arbitrary unit
AAO	Anodic aluminum oxide
ads	Adsorption
AM 1.5	Air Mass Solar Spectrum (1000 W/m^2)
AR	Aspect ratio
ASEAN	The Association of Southeast Asian Nations
BSE	Backscattered electrons
CB	Conduction band
DC	Direct current
DEG	Diethylene Glycol
DI	Deionized water
DMSO	Dimethyl Sulfoxide
DNA	Deoxyribonucleic acid
DSSC	Dye-sensitized Solar Cells
EDX	Energy Dispersive X-ray
EFTEM	Energy Filtered Transmission Electron Microscopy
EG	Ethylene Glycol
ESCA	Electron Spectroscopy for Chemical Analysis
ESI	Electron Spectroscopic Imaging
FESEM	Field Emission Scanning Electron Microscopy
FiT	Feed-in Tariff
FRL	Fluoride-rich layer
FSTNTs	Free standing TiO_2 nanotubes
FTIR	Fourier Transform Infrared
FWHM	Full Width High Maximum
GHG	Greenhouse gases
HRTEM	High Resolution Transmission Electron Microscopy
ICSD	Inorganic Crystal Structure Database
ISO	International Organisation for Standardization
J-V	Current density-voltage
J-t	Current density-time transient

LSV	Linear sweep voltammetry
MB	Methyl blue
MBIPV	Malaysia Building Integrated Photovoltaic
MO	Methyl orange
min	Minute
NTs	Nanotubes
NREPAP	National Renewable Energy Policy and Action Plan
PDF	Powder Diffraction File
PBR	Pilling-Bedworth Ratio
PEC	Photoelectrochemical
pH	Hydrogen potential
PL	Photoluminescence
RO	Reverse osmosis
SAED	Selected Area Electron Diffraction
SE	Secondary electrons
SEM	Scanning Electron Microscopy
SHE	Standard Hydrogen Electrode
TEM	Transmission Electron Microscopy
TNTs	TiO_2 nanotubes
UV	Ultraviolet
UV-Vis	Ultraviolet- Visible Spectrophotometer
VB	Valence band
XPS	X-ray Photoelectron Spectroscopy
XRD	X-ray Diffraction

PENGHASILAN STRUKTUR NANOTIUB TiO_2 DI DALAM ETILENA GLIKOL MENGANDUNGI PENAMBAH MELALUI PENGANODAN

ABSTRAK

Rangkaian tiubnano TiO_2 (TNTs) telah menarik minat yang signifikan sebagai calon yang paling sesuai untuk aplikasi tindakbalas terfotoaruh. Komposisi elektrolit adalah salah satu faktor yang penting untuk pembentukan oksida melalui penganodan. TNTs dihasilkan dengan elektrolit etilena glikol (EG)/ammonium fluorida (NH_4F) yang mengandungi pelbagai bahan tambahan (H_2O, H_2O_2, KOH, LiOH and Na_2CO_3) sebagai penyedia O^{2-} dan/atau OH^-. Ciri-ciri yang disiasat termasuklah morfologi, struktur oksida nanotubular yang terbentuk dan penghablurannya. TNTs yang terbentuk dalam EG/NH_4F/H_2O_2 menghasilkan struktur berumput (ketebalan dinding ~ 10 nm) disebabkan punaran kimia yang tinggi di hujung permukaan tiub. TNTs yang terbentuk dalam elektrolit EG/NH_4F/KOH sebahagiannya adalah berkristal dengan panjang tiub purata 6.1 µm. Ion-ion OH^- menghadkan punaran permukaan yang berlebihan di hujung tiub. Sementara itu, penambahan Na_2CO_3 dalam elektrolit EG/NH_4F berjaya membentuk TNTs bebas berdiri (FSTNTs) akibat evolusi gas yang membantu melemahkan lekatan filem anodik pada Ti. FSTNTs mengandungi kristal nano anatase. TNTs berumput menunjukan kecekapan pennyahwarna fotokatalitik MO tertinggi (90.7%) selepas 2 jam disebabkan keupayaan fasa anatase untuk kekal pada 600 ^oC di hujung struktur berumput.