



Ain Shams University
Faculty of Science
Chemistry Department

***Green Desulfurization of some Petroleum Organic
Sulfur Compounds Using Nano Layered Materials
Loaded with {Mo₁₃₂} Nanoball***

*Thesis Submitted for
Ph.D. Degree of Science
(Chemistry)*

*By
Asmaa Ahmed Abdelrahman Ahmed*

M.Sc. in Chemistry
(Inorganic Chemistry 2013)

*To
Chemistry Department
Faculty of Science
Ain Shams University
Cairo, Egypt*

2018



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Aim of the work

This study has aimed to produce ultra-low sulfur diesel fuel via oxidative desulfurization process. Two kinds of polyoxometalate - graphene oxide composites were used:

A- **{Mo₁₃₂}/GO composite.**

B- **{Fe₃₀Mo₇₂}/GO composite.**

The structural and chemical properties of the prepared materials were characterized using Fourier-transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), UV-vis., Raman spectroscopy, HR-TEM, TGA and SBET techniques.

So the plane of this work can be presented according to the following sequences:

- 1- Preparation and characterization of graphene oxide (GO).
 - 2- Preparation of polyoxomolybdate nanoballs {Mo₁₃₂}.
 - 3- Preparation of metal substituted polyoxomolybdate nanoball {Fe₃₀Mo₇₂}.
 - 4- Preparation of polyoxomolybdate and metal substituted polyoxomolybdate nanoballs graphene nanocomposite.
 - 5- Characterization of the prepared materials and composites with conventional techniques.
 - 6- Study the efficiency of the prepared material on oxidative – desulfurization of petroleum organosulfur compounds.
 - 7- Optimization of reaction conditions including (reaction temperature, H₂O₂ ratio (O/S) molar ratio, catalyst weight and time).
 - 8- Application of the more active prepared materials on petroleum fraction (gas oil) feed stock.
 - 9- Characterization of spent catalysts and reuse for several time.
-

Abbrev.	Description
2D	2-dimensional
3D	3-dimentional
A (in Arrhenius equation)	(Pre-exponential factor)
AC	Activated carbon
ADS	Adsorptive desulfurization
ASA	Amorphous silica-alumina
BDS	Biodesulfurization
BET	Brunauer-Emmett-Teller
BN	Boron nitride
BT	Benzothiophene
CNT	Carbon nano tube
DBT	Dibenzothiophene
DDS	Direct desulfurization pathway
DMDBT	Dimethyldibenzothiophene
DMF	Dimethylformamide
DTA	Differential thermal analysis
E _a	The activation energy (KJ/mol)
EDS	Extractive desulfurization

EPA	Environmental protection agency
EPODS	Extraction and photocatalytic oxidative desulfurization
g-C ₃ N ₄	Graphitic carbon nitride
GO	Graphene oxide
GPH	Graphene prepared by conventional Hummers' method
GPP	Graphene prepared by phosphoric acid
HDS	Hydrodesulfurization
HYD	Hydrogenation pathway
ICP	Inductive coupling plasma
IUPAC	International Union of Pure and Applied Chemistry
K	Rate constant of pseudo-first order
Me CN	Acetonitrile
MIPs	Molecularly imprinted polymers
MS	Mechanical stirring
MIL	Materials of Institute Lavoisier
MOF	Metal-Organic Framework
NMP	N-methylpyrrolidone
NMR	Nuclear magnetic resonance
O/S	The H ₂ O ₂ /DBT molar ratio

ODH	Oxidative-dehydrogenation
ODS	Oxidative desulfurization
OSC	Organic sulfur compounds
POMs	Polyoxometalates
ppm	Part per million
R	Universal gas constant
R ²	Regression coefficient
rGO/ZrP	Reduced graphene oxide /zirconium phosphate
RT	Room temperature
S ₀	The initial sulfur concentration
SEM	Scanning electron microscope
SO _x	Sulfur Oxides
St	The sulfur concentration at reaction time (t)
T	Temperature (K)
TBHP	Tertbutyl hydroperoxide
TEM	Transmission electron microscopy
TGA	Thermal gravimetric analysis
TMs	Transition metals
UV/VIS	Ultra-violet/visible

XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction
XRF	X-ray fluorescence spectroscopy

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