## SULFIDATION OF IRON - BASED NANOMATERIAL AS CATALYST FOR WATER SPLITTING USING HYDROTHERMAL

## **SKRIPSI**

Diajukan kepada

Universitas Pendidikan Ganesha

Untuk Memenuh<mark>i</mark> Salah Satu Persyaratan dalam Menyelesaikan

Program Sarjana Pendidikan Fisika

Oleh

Ni Luh Ayu Ardi Lestari NIM. 2013021006

DIES

# PROGRAM STUDI S1 PENDIDIKAN IPA JURUSAN FISIKA DAN PENGAJARAN IPA FAKULTAS MATEMATIKA DAN ILMU PENGETAHUAN ALAM UNIVERSITAS PENDIDIKAN GANESHA SINGARAJA

## SKRIPSI

# DIAJUKAN UNTUK MELENGKAPI TUGAS DAN MEMENUHI SYARAT-SYARAT UNTUK MENCAPAI GELAR SARJANA PENDIDIKAN

Menyetujui

Pembimbing I,

Pembimbing II,

I Gede Arjana, S.Pd., M.Sc., RWTH NIP. 19911226 202012 1 009

Jumes

Dr. Idu Bagus Putu Mardana, M. Si. NIP. 19640827 199102 1 001

Skripsi oleh Ni Luh Aryu Ardi Lestari ini Telah dipertahankan di depan dewan penguji Pada tanggal. 15 Februari 2024

Dewan Penguji.

I Gede Arjana, S.Pd., M.Sc., RWTH NIP. 19911226 202012 1 009

Dr. Ida Bacus Putu Mardana, M.Si. NIP. 19640827 199102 1 001

Drs. Putu Yasa, M.Si. NIP. 19611104 198703 1 002

Putu Widiarini S M.Sc. 'd NIP. 19890327 201903 2 020

(Ketua)

(Anggota)

(Anggota)

(Anggota)

Diterima oleh Panitia Ujian Fakultas Matematika dan Ilmu Pengetahuan Alam Universitas Pendidikan Ganesha guna memenuhi syarat-syarat untuk mencapai gelar sarjana Pendidikan

Pada:

Hari

: Selasa

Tanggal

: 20 Februari 2024

Mengetahui,

Ketua Ujian,

Dr. 1 Wayan/Puja Astawa, S.Pd., M.Stat.Sci, NIP. 19690116 199403 1 001 Sekertaris Ujian,

Prof. Dr. Ni Ketut Rapi, M.Pd. NIP. 19630830 198803 2 002

Mengesahkan atika dan Ilmu Pengetahuan Alam Dekan DEKAN Dr. 1 Wayan Sukra Warpala, S.Pd., M.Sc. NIP. 19671013 199403 1 001

#### PERNYATAAN

Dengan ini saya menyatakan bahwa karya tulis yang berjudul "Sulfidation Of Iron - Based Nanomaterial As Catalyst For Water Splitting Using Hydrothermal" beserta seluruh isinya adalah benar-benar karya sendiri dan saya tidak melakukan penjiplakan dan pengutipan dengan cara-cara yang tidak sesuai dengan etika yang berlaku dalam masyarakat keilmuan. Atas pernyataan ini, saya siap menanggung resiko/sanksi yang dijatuhkan kepada saya apabila kemudian hari ditemukan adanya pelangaran atas etika keilmuan dalam karya saya ini atau ada klaim terhadap karya saya ini.

Singaraja, 19 Februari 2024

Yang membuat pernyataan,

Ni Luh Ayu Ardi Lestari

#### ACKNOWLEDGEMENT

The writer expresses her highest gratitude to the Almighty God for blessing, love, opportunity, health, and mercy so that the writer could finish this bachelor thesis with the title **"Sulfidation of Iron - Based Nanomaterial as Catalyst for Water Splitting Using Hydrothermal"**. In arranging this thesis, a lot of people have provided motivation, advice, support, and even remark that had helped the writer. In this valuable chance, the writer aims to express her gratitude and appreciation to the following people:

- 1. I Gede Arjana, S.Pd, M.Sc. RWTH as the first supervisor and mentor for his willingness to take the time to guide, provide advice, motivation, enthusiasm and facilitate not only in completing the thesis but also while being a student in the physics education study program.
- 2. Dr. Ida Bagus Putu Mardana, M.Si. as supervisor II as well as mentor for his willingness to take the time to guide, provide advice, motivation, enthusiasm and facilitate not only in completing the thesis but also while being a student in the physics education study program.
- 3. Drs. Putu Yasa, M.Si, as Lecturer Examiner I for his willingness to provide guidance, advice and feedback during the examination process until the completion of the thesis.
- 4. Putu Widiarini, S.Pd., M.Pd., M.Sc as Lecturer Examiner II as well as academic advisor for his willingness to provide guidance, advice, motivation not only during the examination process to the completion of the thesis but also in lecture life.
- 5. Professor Chien-Kuo Hsieh as the writer's Advisor at Ming Chi University of Technology for his willingness to provide guidance, input and direction during the thesis writing process as well as facilitating during the experimental process until completion.
- Laboratory Assistant at Ming Chi University of Technology for his willingness to provide guidance and feedback during experimental process until completed.
- 7. Lecturers of Physics Education study program who cannot be mentioned one by one who have provided knowledge and insight.

- 8. Parents (I Putu Sidiasa and Ni Putu Ayu Sartikawati) who have dedicated all their time and energy to the writer. Thank you for all material and nonmaterial support and for being the best parents who has been with the writer throughout the lecture process and the writing of this thesis until completed.
- 9. Laboratory seniors as mentors who always provide insight and support during the experimental process.
- 10. To the writer's best friend and partner (I Gusti Ayu Mutiara Sandhy and Ni Putu Devi Kristina) who has helped me during my struggle as a student in the Department of Physics and Science Teaching and helped a lot in completing this thesis.
- 11. All the writer's friends in Bali and Taiwan for all the support and advice given to the writer.
- 12. Colleagues who cannot be mentioned one by one who have provided help and support in completing this thesis.

The writer realizes that this thesis is not perfect. For that, for the improvement of this thesis, expect all constructive criticism and suggestions from various sides. Hopefully this thesis can be useful for all of us, especially for the development of the world of education.



Writer

| AC  | KNOWLEDGEMENT  | i    |
|-----|--|------|
| AB  | STRACT   | iii  |
| CO  | NTENT  | iv   |
| FIC | GURE CONTENTS  | vi   |
| TA  | BLE CONTENTS   | viii |
| AP  | PENDIX CONTENTS                                      | ix   |
| СН  | APTER I  | 1    |
| INT | FRODUCTION   | 1    |
| 1.1 | Background   | 1    |
| 1.2 | Problem Limitation                                   | 8    |
| 1.3 | Problem Formulation                                  | 8    |
| 1.4 | Hypothesis   | 9    |
| 1.5 | Aims of Research                                     | 9    |
| 1.6 | Significance of study                                | 9    |
| СН  | IAPTE <mark>R</mark> II                              | 10   |
| LIJ | FERAT <mark>U</mark> RE REVIEW                       | 10   |
| 2.1 | Water Splitting                                      | 10   |
| 2.2 | Iron Hydroxide                                       | 15   |
| 2.3 | Sulfidation Iron – Based                             | 17   |
| 2.4 | Chemical Bath Deposition                             | 19   |
| 2.5 | Hydrothermal   | 20   |
| 2.6 | X – Ray Diffaction (XRD)                             | 23   |
| 2.7 | Field Emission Scanning Electron Microscope (FE SEM) | 25   |
| 2.8 | Electrochemical Measurement                          |      |
|     | 2.8.1 LSV  |      |
|     | 2.8.2 EIS  |      |
|     | 2.8.3 ECSA   |      |
| CH  | APTER III  | 32   |
| EX  | PERIMENTAL METHODS                                   | 32   |
| 3.1 | Types of Research                                    | 32   |
| 2 2 | Time and Place of Research                           |      |

### CONTENT

| 3   | 3.2.1       | Re   | search Time  | 32 |
|-----|-------------|------|--|----|
| 2   | 3.2.2       | Re   | search Place   | 32 |
| 3.3 |             | Ex   | perimental Variables                                   | 32 |
| 3.4 |             | Ex   | perimental Process                                     | 32 |
| 3.5 |             | Ex   | perimental Tools                                       | 33 |
| 3.6 |             | Ex   | perimental Procedure                                   | 34 |
|     | 3.5.        | 1 M  | aterials and Reagents                                  | 34 |
|     | 3.5.        | 2 Sy | ynthesis of Fe(OH) <sub>2</sub> /NF                    | 34 |
|     | 3.5.        | 3 Sy | ynthesis of Fe <sub>x</sub> S <sub>y</sub> /NF         | 35 |
| 3.7 |             | Saı  | mple Characterization                                  | 37 |
| 3.8 |             | Ele  | ectrochemical Measurement                              | 37 |
| 3.9 |             | Da   | ta Analysis  | 37 |
| СН  | APT         | ER   | IV.  | 38 |
| RE  | SUL         | TS / | AND DISCUSSION   | 38 |
| 4.1 |             | Re   | sult of Research                                       | 38 |
|     | 4.1.        | 1    | Synthesis Result of Nanomaterial Iron Sulfide          | 38 |
|     | 4.1.        | 2    | Characterization Result Iron Sulfide Using XRD         | 39 |
|     | 4.1.        | 3    | Characterization Result Iron Sulfide Using FE SEM      | 40 |
|     | 4.1.        | 4    | Electrocatalytic Activity Result Using Electrochemical |    |
|     | Mea         | asur | em <mark>ent</mark>                                    | 42 |
| 4.2 |             | Dis  | scussion   | 48 |
|     | 4.2.        | 1    | Synthesis Result of Nanomaterial Iron Sulfide          | 48 |
|     | 4.2.        | 2    | Characterization Result Iron Sulfide Using XRD         | 49 |
|     | 4.2.        | 3    | Characterization Result Iron Sulfide Using FE SEM      | 50 |
|     | 4.2.        | 4    | Electrocatalytic Activity Result Using Electrochemical |    |
|     | Mea         | asur | ement  | 52 |
| СН  | APT         | 'ER  | <b>V</b>   | 55 |
| CO  | NCL         | LUS  | ION  | 55 |
|     | 5.1 Summary |      |  |    |
|     | 5.2         | 2Sug | ggestion   | 56 |
| RE  | FER         | EN   | СЕ   | 57 |
| AP  | PEN         | DIX  | Κ  | 64 |

### **FIGURE CONTENTS**

| Figure 1. Classification of global energy1  |
|---|
| Figure 2. Electrochemical setup and the corresponding I-V polarization curve for  |
| (a) Oxygen Evolution Reaction, (b) Hydrogen Evolution Reaction, and (c)           |
| Corresponding full cell4  |
| Figure 3. Desired characteristics of a good HER electrocatalyst12                 |
| Figure 4. Molecular structures of ferrous hydroxide16                             |
| Figure 5. Molecular structures of ferric hydroxide17                              |
| Figure 6. Typical colors andrange of suitable temperature to form iron sulfide    |
| scale   |
| Figure 7. Schematic diagram of a typical chemical bath deposition (CBD) setup 20  |
| Figure 8. Illustration of a cylindrical-chamber autoclave                         |
| Figure 9. Schematic of typical hydrothermal method equipment23                    |
| Figure 10. Schematic of the experimental principle for X-ray diffraction25        |
| Figure 11. Bragg reflection on a set of N atomic planes                           |
| Figure 12. Principle of Operation of FESEM  |
| Figure 13. Schematic Diagram of FESEM   |
| Figure 14. I-V curve for the full water splitting reaction                        |
| Figure 15. Electrochemical Impedance Spectroscopy                                 |
| Figure 16. ECSA   |
| Figure 17. The flow chart of FexSy /NF experimental prosess                       |
| Figure 18. Schematic diagram synthesis of the Fe(OH) <sub>2</sub> /NF             |
| Figure 19. Schematic diagram synthesis of Fe <sub>x</sub> S <sub>y</sub> /NF      |
| Figure 20. Schematic diagram electrochemical measurements                         |
| Figure 21. Synthesis result of iron hydroxide                                     |
| Figure 22. Synthesis result of iron sulfide                                       |
| Figure 23. XRD patterns of iron sulfide and iron hydroxide40                      |
| Figure 24. The morphological structure of iron hydroxide using SEM with different |
| magnifications, (a) 10000x. (b) 25000x. (c) 50000x                                |
| Figure 25. The morphological structure of iron sulfide (0.0125M sodium sulfide    |
| nonahydrate) using SEM with different magnifications, (a) 10000x. (b) 25000x. (c) |
| 50000x  |

| Figure 26. The morphological structure of iron sulfide (0.025M sodium sulfide     |
|---|
| nonahydrate) using SEM with different magnifications, (a) 10000x. (b) 25000x. (c) |
| 50000x42  |
| Figure 27. The morphological structure of iron sulfide (0.05M sodium sulfide      |
| nonahydrate) using SEM with different magnifications, (a) 10000x. (b) 25000x. (c) |
| 50000x42  |
| Figure 28. The morphological structure of iron sulfide (0.1M sodium sulfide       |
| nonahydrate) using SEM with different magnifications, (a) 10000x. (b) 25000x. (c) |
| 50000x  |
| Figure 29.(a) LSV for OER with IR compensation. (b) Tafel slope for OER. (c)      |
| OER performance comparison between tafel slope (mV/dec) and overpotential         |
| (mV)44  |
| Figure 30 .(a) LSV for HER with IR compensation. (b) Tafel slope for HER. (c)     |
| HER performance comparison between tafel slope (mV/dec) and overpotential         |
| (mV)45  |
| Figure 31. Electrochemical impedance spectra measured at overpotential of 100     |
| mV, (a) for OER. (b) for HER  |
| Figure 32. Electrochemical Active Surface Area in five scan rate (10,30,50,70 and |
| 90) mV/s, (a) for OER. (b) for HER47  |
| Figure 33. Cyclic voltammograms iron hydroxide                                    |
| Figure 34. Cyclic voltammograms sampel 1  |
| Figure 35. Cyclic voltammograms sampel 2  |
| Figure 36. Cyclic voltammograms sample 3  |
| Figure 37. Cyclic voltammograms sample 4  |
| Figure 38. Cyclic voltammograms iron hydroxide67                                  |
| Figure 39. Cyclic voltammograms sample 167  |
| Figure 40. Cyclic voltammograms sampel 267  |
| Figure 41. Cyclic voltammograms sample 367  |
| Figure 42. Cyclic voltammograms sample 467  |

## **TABLE CONTENTS**

| Table 1. Overall and half cell reaction of water electrolysis              | 10 |
|--|----|
| Table 2. Most Common Iron Sulfide Scales in an Oilfield                    | 18 |
| Table 3. Experimental tools  | 33 |
| Table 4. Analysis teqhnique of Fe <sub>x</sub> S <sub>y</sub> /Fe(OH)2 @NF | 37 |
| Table 5.Comparison table of electrochemical measurements for OER           | 47 |
| Table 6. Comparison table of electrochemical measurements for HER          | 48 |
| Table 7. Fitting curve of cyclic voltammograms for HER                     | 68 |
| Table 8. Fitting curve of cyclic voltammograms for OER                     | 69 |
| Table 9. Fitting curve of EIS for HER                                      | 70 |
| Table 10. Fitting curve of EIS for OER                                     | 71 |



## **APPENDIX CONTENTS**

| Appendix 1. Weight of material and reagents             | 65 |
|---|----|
| Appendix 2. Cyclic voltammograms curve for OER          | 66 |
| Appendix 3. Cyclic voltammograms curve for HER          | 67 |
| Appendix 4. Table fitting curve of cyclic voltammograms | 68 |
| Appendix 5. Fitting curve for Impedance                 | 70 |

