Nickel based electro-catalyst: toward the electrochemical valorization of glycerol in alkaline medium

Kara A. Hughes, Mohamed S. E. Houache, Elena A. Baranova

Department of Chemical and Biological Engineering, University of Ottawa, 161 Louis-Pasteur Ottawa ON K1N 6N5 Canada

Ni Electro Can
NSERC Discovery Frontiers
Laboratoire de Génie Électrochimique
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LGE
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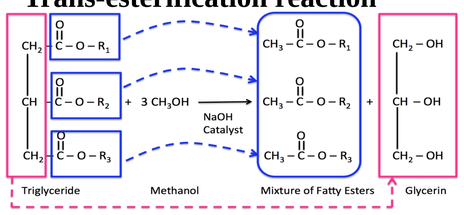
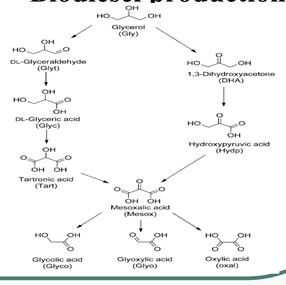
Introduction

Produced 600 million litres in 2017

Trans-esterification reaction



By-product glycerol – 10 wt%
Biodiesel production



Electrochemical transformation of glycerol into value-added chemicals

Objectives

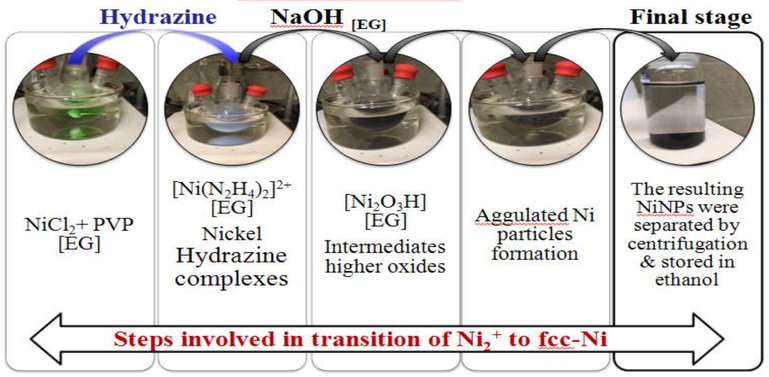
- ✓ Synthesising efficient nano-structures of nickel;
- ✓ Determining the effect different amounts of a capping agent – polyvinyl pyrrolidone (PVP) has on nanoparticles;
- ✓ Investigating electrocatalytic activity for (GOR) in an alkaline medium.

Methodology

I. Synthesis of Ni nanoparticles (NiNPs)

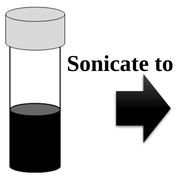
Reaction → $2Ni^{2+} + N_2H_4 + 4OH^- \rightarrow 2Ni + N_2 + 4H_2O$

Ratio: $\frac{[N_2H_5OH]}{[NiCl_2]} = 20$

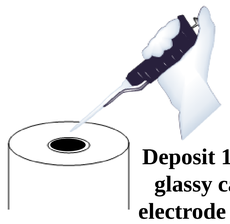


II. Ink Preparation

- **6 mg NiNPs**
- **1 µl H_2O**
- **200 µl Isopropanol**
- **100 µl Nafion**



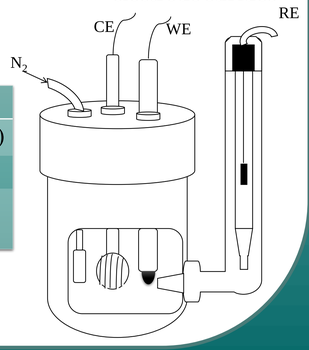
Sonicate to disperse



Deposit 10 µl on glassy carbon electrode surface

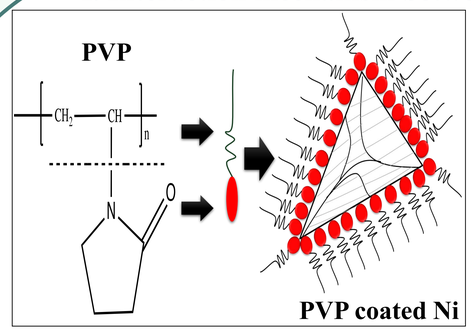
III. Electrochemistry Tests

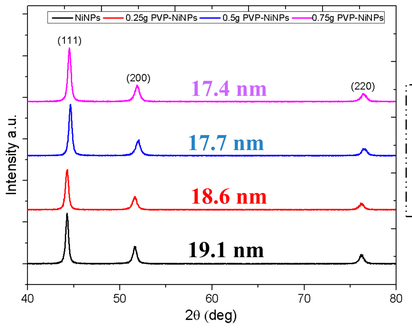
Working Electrode	NiNPs+ PVP
Reference Electrode	Mercury /Mercury Oxide(Hg/HgO)
Counter Electrode	Platinum Mesh
Electrolyte	▪ 1.0 M KOH ▪ 1.0 M KOH + 0.1 M Glycerol



Results and Discussion

I. Interaction mechanism between PVP & NiNPs





The corresponding XRD spectrum for the resultant particles illustrates **3 characteristic peaks for nickel**. This revealed that the resultant particles were pure **face-centred cubic nickel (fcc)**

II. Electro-oxidation of glycerol on Nickel nanoparticles

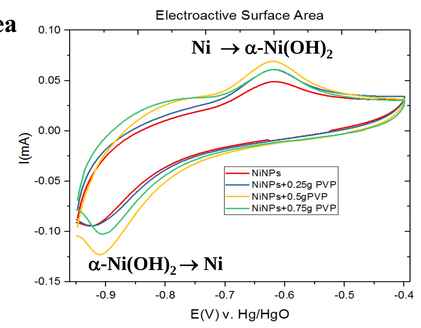
Determination of Electrochemical Surface Area

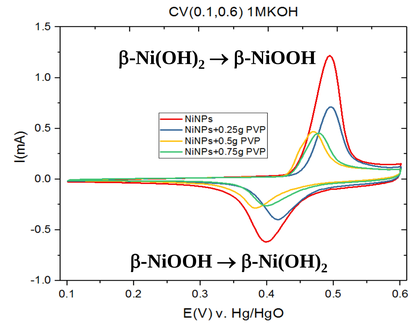
Formation of $\alpha - Ni(OH)_2$ on GC electrode in 1M KOH v= 50 mV/s

- ECSA from peak between **[-0.7V to -0.5 V]**

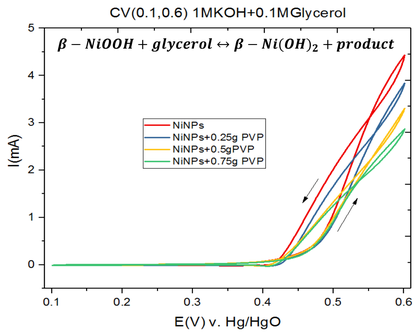
$$A_{easa} = \frac{Q}{514\mu C/cm^2}$$

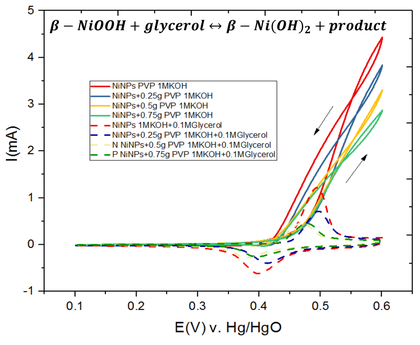
NiNPs	ECSA
No PVP	0.33±3.2%
0.25g PVP	0.48±4.7%
0.5g PVP	0.75±1.4%
0.75g PVP	0.41±7.2%

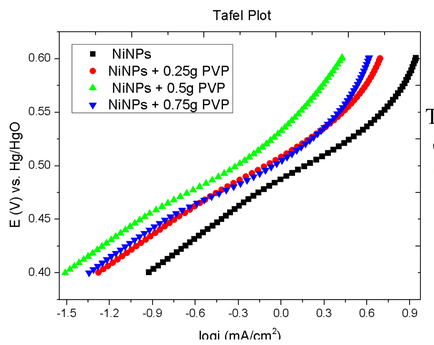




CVs of as-prepared electro-catalysts in N_2-saturated 1M KOH solution ***v= 50 mV/s***



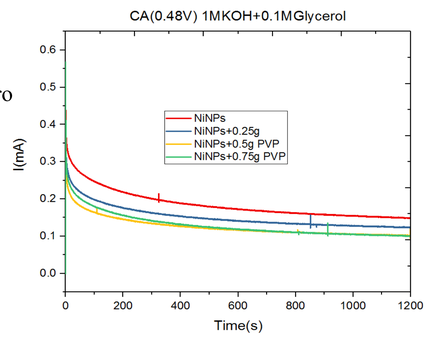


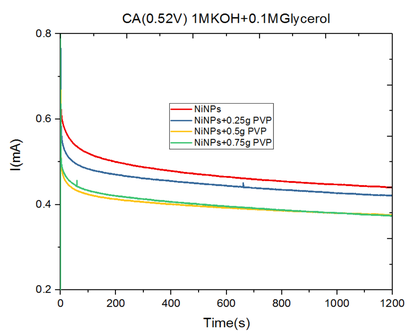


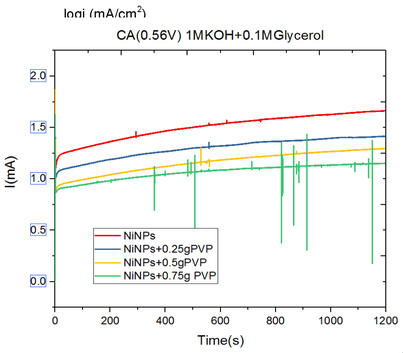
Tafel plots of as-prepared catalysts in 1 M KOH + 0.1 M glycerol solution ***v= 1 mV/s***

CAs of as-prepared electro catalysts at different potentials for 20 min

Stability test







Conclusion

- ✓ The resultant NiNPs by the hydrazine reduction were characterized by XRD and found to be pure crystalline nickel with face-centered cubic (fcc) structure.
- ✓ Increasing the amount of PVP added increased the time taken for the nanoparticles to form, for the synthesis. However, increasing the concentration of NaOH used reduced the reaction time dramatically.
- ✓ The nanoparticles with higher amounts of PVP had lower current densities and slightly smaller crystallinity.

Recommendations for Further Work

- ✓ Coupling CA with in-situ PM-IRRAS to identify glycerol electro-oxidation products on the catalyst surface and in the bulk solution.
- ✓ The effect of different molecular weights of PVP can be investigated to determine which one is the best for the this synthesis.
- ✓ Bimetallic nanoparticles such as Ni_xPd_{x-1} could be synthesised and electrochemically examine for use in glycerol oxidation.

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Acknowledgments

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