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NiFe Oxide as Anodic Material for the Electrocatalytic Oxidation of Glucose to Glucaric Acid

Rosalba Passalacqua[a]*, *Lidia Caterina Pellicano*[a], *Salvatore Abate*[a], *Siglinda Perathoner*[a], *Gabriele Centi*[a]

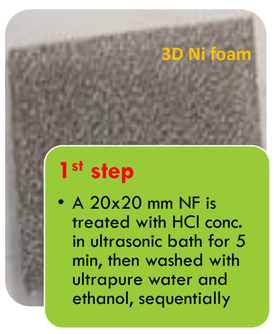
[a]Department of ChiBioFarAM (Industrial Chemistry) University of Messina, ERIC aisbl and INSTM/CASPE

Viale F. Stagno d'Alcontres 31, Messina 98166, Italy

**rpassalacqua@unime.it*

A new frontier for the chemical industry is the highly selective transformation, by electrochemistry, of raw materials from biomass into intermediates. Demonstrating its feasibility is one of the objectives of the EU PERFORM project, focusing on specific investigations regarding targeted paired reactions such as glucose electro-oxidation to glucaric acid, a first step in the adipic acid electrochemical synthesis[1].
Here we report, the preparation of nanostructured NiFe oxide catalysts ($NiFeO_x$/NF), synthesized from nickel foam (NF), via layered double hydroxide (LDH) nanosheets intermediates, as efficient catalysts for glucose (Glu) electrochemical oxidation[2].

Preparation of $NiFeO_x$/NF catalysts

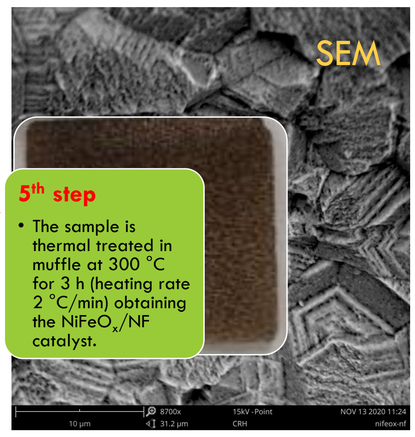


1st step
- A 20x20 mm NF is treated with HCl conc. in ultrasonic bath for 5 min, then washed with ultrapure water and ethanol, sequentially

2nd step
- The cleaned NF is immersed in a 0.2M Fe^{3+} solution and sonicated for 30 min

3rd step
- 10 mL of a 1.5 M $CO(NH_2)_2$ solution and 20 mL of ethanol were added, the mixture containing NF is put in autoclave and heated to 160 °C for 24 h (heating rate 2 °C/min)

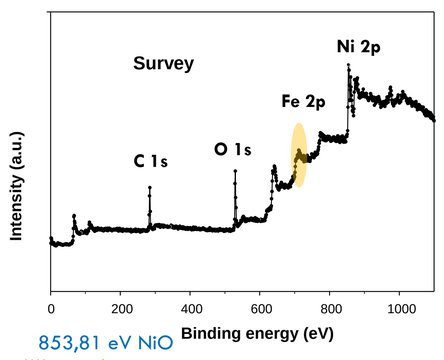
4th step
- The sample is removed from the autoclave, washed with ultrapure water and ethanol obtaining the hydroxide nanosheets $NiFe(OH)_x$/NF

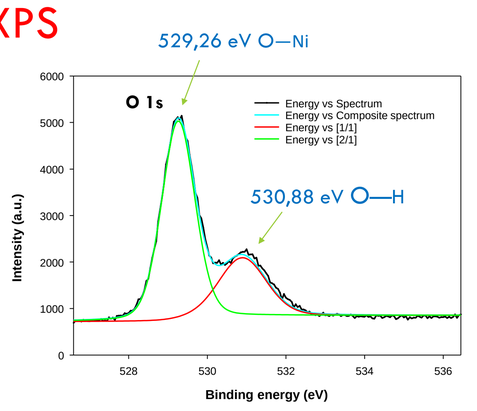


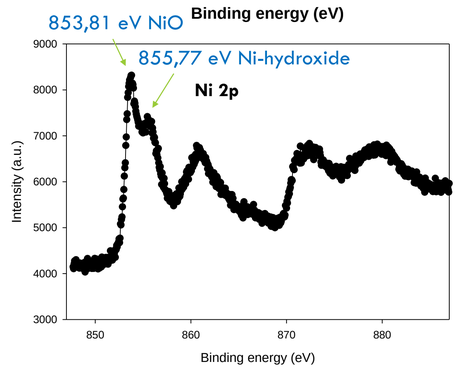
5th step
- The sample is thermal treated in muffle at 300 °C for 3 h (heating rate 2 °C/min) obtaining the $NiFeO_x$/NF catalyst.

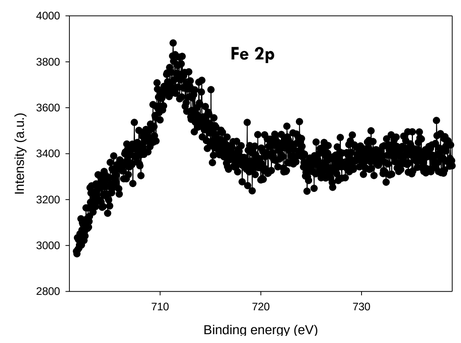
Catalyst characterization

XPS







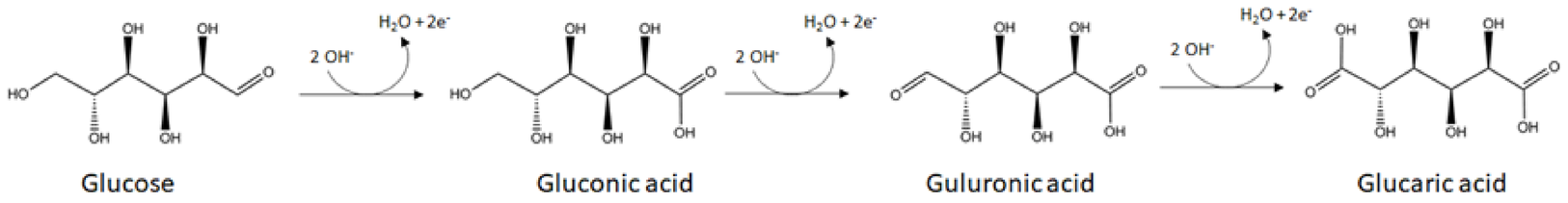


Weight % Table

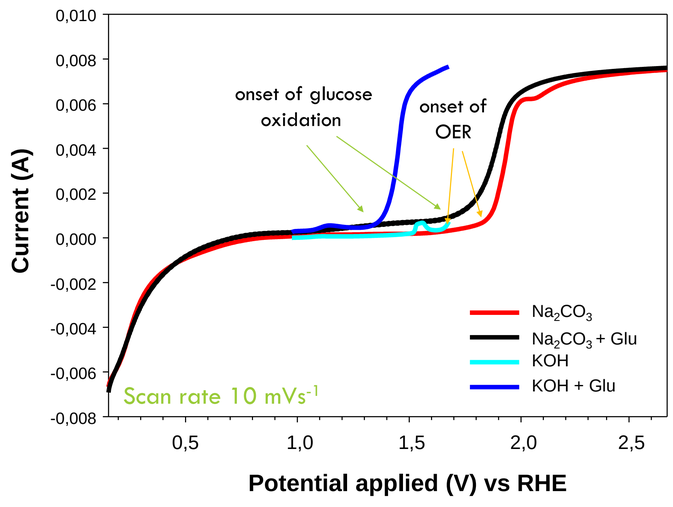
C1s	O1s	Fe2p3	Ni2p3
15,75	13,30	9,22	61,72

NiFeOx/NF testing: D-glucose oxidation reaction

Probable Reaction pathway



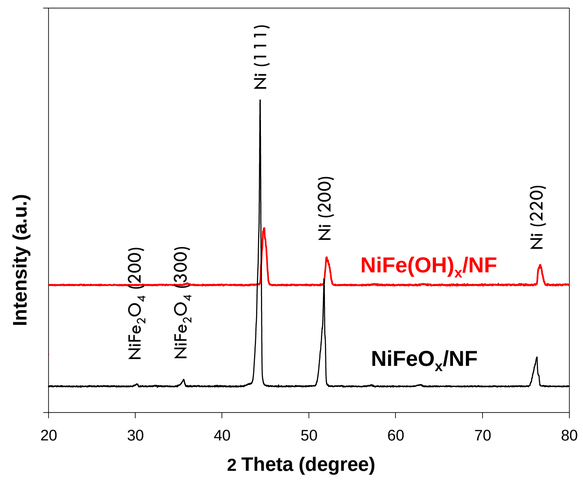
Linear Sweep Voltammetry (LSV)



Electrochemical tests conditions:

- PGSTAT30 with a three-electrode set-up
- Batch cell
- rt
- Counter E: Pt wire
- Reference E: saturated Ag/AgCl
- Working E: NiFeOx/NF sample
- Electrolyte: 1M KOH Ar purged or 0,1M Na_2CO_3 in air

XRD



During the reaction, the formation of Gluconic Acid initially takes place, then its conversion according to Scheme 1. For a screening of the capability of the prepared NiFeOx/NF catalysts, the amounts of GA produced in batch by Glucose oxidation was determined by IC.

IC conditions:

MagIC Net Metrohm
Column: Metrosep Organic Acids
Eluent: 0.5 mM H_2SO_4
Flow: 0.5 mL/min
Pressure: 5.34 MPa
Injection Volume: 20 µL

Catalyst	Electrolyte	pH	Reaction Time/h	Gluconic Acid/ppm
NiFeOx/NF	KOH 1M	13,52	2	238
NiFeOx/NF	Na_2CO_3 0,1M	10,95	2	143

Conclusions

- A new electrochemical approach inspired to green chemistry, allowing both energy saving and biomass valorization, is under investigation under the PERFORM project.
- NiFeOx/NF catalysts have been successfully prepared and characterized.
- The NiFeOx/NF catalysts have been demonstrated effective in D-glucose oxidation reaction under different reaction conditions, with better results for KOH electrolyte.
- Tests will be performed with the best screening catalysts in flow cell by the partners of PERFORM to evaluate the conversion capability under different conditions and selectivity toward glucaric acid.

References
1. *Molecules* 2020, 25, 3712.
2. *Nature Communications*, 2020, 11:265.

Acknowledgments
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