“DESIGNING METAL OXIDE NANOSTRUCTURE FOR ELECTROCHEMICAL WATER SPLITTING”

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IN

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Submitted By

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Certificate

This is to certify that the dissertation entitled **"DESIGNING METAL OXIDE NANOSTRUCTURE FOR ELECTROCHEMICAL WATER SPLITIING"** being submitted by **Hemant Kerwal (Roll no. 301702013)** in partial fulfillment for the award of the degree of Master of Science in Chemistry, Thapar Institute of Engineering and Technology, Patiala is a bonafide work carried out under the esteemed supervision and conception of Dr. Raj Kumar Das and that no part of this thesis has been submitted for the award of any degree.

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Candidate's Declaration

I hereby declare that the thesis entitled **"DESIGNING METAL OXIDE NANOSTRUCTURE FOR ELECTROCHEMICAL WATER SPLITTING"** is an authentic record of my work carried out in partial fulfillment for the award of the degree of Master of Science in Chemistry, Thapar Institute of Engineering and Technology, Patiala under supervision of Dr. Raj Kumar Das, Assistant Professor, School of Chemistry and Biochemistry (SCBC), Thapar Institute of Engineering and Technology (TIET), Patiala during January 2019 to July 2019. All the information in this dissertation has been presented in accordance with the academic rule and ethical conduct. This is to further state that, no part of dissertation has already been or is being currently submitted to any other University or College in any form for the award of such degree.

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It is certified that the above statement made by the student is correct to the best of my/our knowledge and belief.

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HEMANT KERWAL

DEDICATED

TO

MY BELOVED FAMILY

(Mr. Manohar Lal Kerwal and Mrs.Indu Bala)

CONTENTS

ABSTRACT

Fuel cell technology is in demand nowadays owing to the urgent need of more eco-friendly and everlasting energy sources. Hydrogen fuel has great potential in electricity and transportation. Water splitting is one of the most proficient techniques for production of hydrogen. Electrolysis of water requires efficient catalysts which can work at lower overpotential to exhibit reactions like HER (Hydrogen Evolution Reaction) and OER (Oxygen Evolution Reaction). Earlier noble metal like Pt, Ir, Ru were used as electrocatalysts. However, in the present study, non-precious metals like Ni, Fe etc were used to synthesize an economical electrocatalyst by a hydrothermal process. The purity and crystallanity of the prepared samples was verified by XRD. BET analysis revealed that the samples had reasonable surface area. The catalyst works at lower overpotential for HER and OER. The sample made with sucrose is better as it shows better water splitting efficiency. The study explicates the simplistic approach to synthesize NiFe based NPs having excellent activity toward water splitting for generation of hydrogen.

CHAPTER 1

INTRODUCTION AND REVIEW OF LITERATURE

Nowadays, there is a enhanced need to find environment friendly energy sources. From the last 50-150 years, the current resources of energy are fossil fuels (oil, coal, gases, etc.), however they are hazardous towards environment due to emission of greenhouse gases [1]. Thereby, scientists are exploring different types of clean, abundant and renewable alternate source of energy [2]. Recently these alternative sources are slowly substituting the conventional sources of energy by these.

In this perspective, hydrogen represent an excellent example of a fuel that gives rize to zero carbon emission. But the current large-scale production of H_2 is done by steam reforming of fossil fuels [3]. In this aspect water provides the best alternative to produce hydrogen. Water is composed of oxygen and hydrogen and is one of the most abundant molecule in the universe, so hydrogen production by electrolysis of water has become a new emerging area. Such methods attracted lot of attention as it produces highly pure hydrogen and there is no need of any sophisticated experimental techniques.

H_2 and O_2 operated fuel cell is the best choice it only produces water as the combustion product. The process of water splitting consists of two distinct half-cell reactions: hydrogen evolution reaction (HER) at cathode and oxygen evolution reaction (OER) at anode [4]. However, the most efficient catalyst are based on noble metals (Pt/C for HER catalysts and Ru- or Ir-based catalysts for OER) [5] which are less abundant and high cost limiting their practical use.

So, there is an urgent need to seek more abundant alternative of noble metals for sustainable hydrogen production. Therefore, use of abundant earth metal (Fe, Ni, Co etc.) are been used for achieving high OER and HER activity [1]. Also, the different carbons materials such as nanotubes, graphene, carbon nanospheres etc. with variable compositions has also emerged as new generation electrocatalyst.

WATER SPLITTING

Water splitting is a process that produces of hydrogen and oxygen by direct water dissociation. But water decomposition is thermodynamically endothermic and requires a substantial amount of energy. The required energy can be supplied the mainly through three different ways: heat, light and electricity [6].

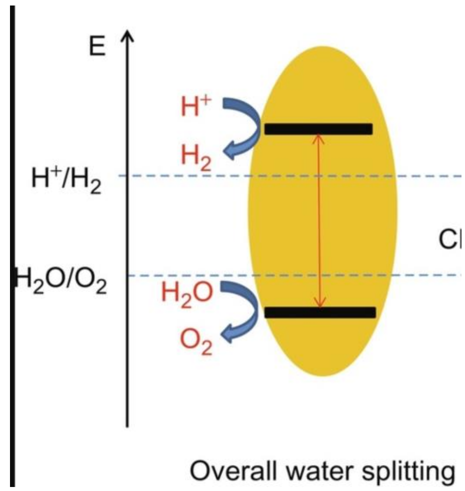

Figure1: Showing overall water splitting reactions

Overall water splitting:

Cathode : $4H^+ + 4e^- \rightarrow 2H_2$ (Water Reduction reaction)

Anode: $2H_2O \rightarrow 4H^+ + 4H^+ + O_2$ (Water Oxidation Reaction)

Overall reaction: $2H_2O \rightarrow 2H_2 + O_2$

PHOTOCHEMICAL WATER SPLITTING:

H_2 generation from water in presence of sunlight represents an attractive route to produce hydrogen in a eco-friendly manner. Till date a significant number of earth abundant, low-cost metal oxide materials have been reported to posses high catalytic activity in presence of light [7].

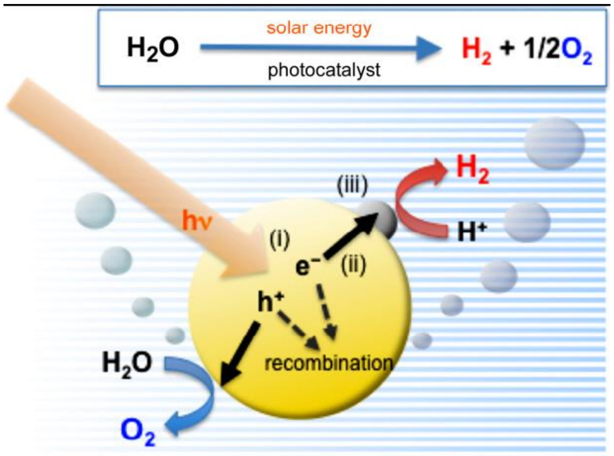

Figure 2: Photochemical water splitting using solar energy.

ELECTROCATALYTIC WATER SPLITTING

Electrocatalytic water splitting comprises of two half reactions (OER and HER). The OER, which is a four-electron process, is more kinetically sluggish among these two [8]. The electrochemical water splitting is demostrated in the **Figure1**.

In acidic medium, water under goes oxidation as following:

$2H_2O \rightarrow O_2 + 4e^- + 4H^+$.

The generated electrons are then transported through the external circuit proton passes through the solution. The electrons and protons then reacts together produce hydrogen at the

$4e^- + 4H^+ \rightarrow 2H_2$.

Theoretically water splitting involves potential difference of 1.23 at least. However, due to slow kinetics, much greater amount of activation energy must be supplied. This term 'overpotential' (η) which represents the amount of extra energy need to be supplied to get the required current density. In order to get low overpotential, suitable catalysts are required. Such catalysts accelerated the electron transfer process to reduce the overpoteial [9]. Till date, precious metals (platinum at the cathode and IrO_2 or RuO_2 at the anode) are reported to possess best catalytic activity. Whereas under basic conditions, first row transition elements act as excellent HER and OER catalysts [10].

The basic reaction for HER and OER are as follows:

HER: $4H_2O + 4e^- \rightarrow 2H_2 + 4OH^-$

OER: $4OH^- \rightarrow O_2 + 2H_2O + 4e^-$

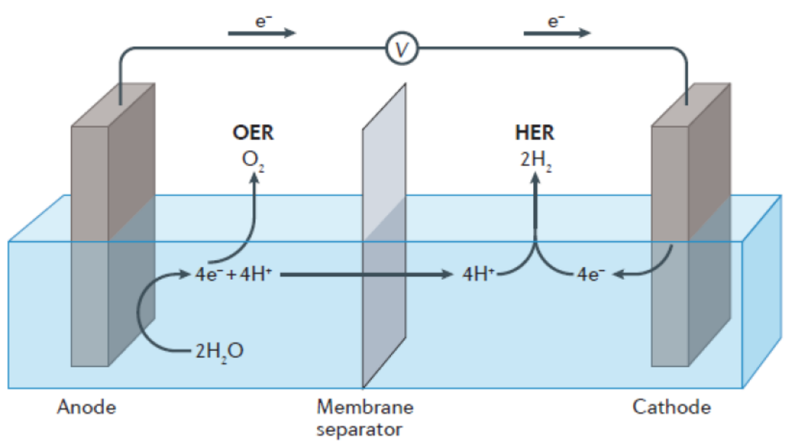

Figure 3: Showing HER and OER processes [11].

The electrochemistry of the water splitting reaction [14]

The water splitting reaction can be divided into the following half-reactions:

$2H^+ + 2e^- \rightarrow H_2$ (For HER), with $E_o = 0$ **(eq. 1.2)**

$E = E_o + 0.059\times2 \log [H^+]^2 = E_o + 0.059 \log [H^+] = 0\text{-}0.059pH$ **(eq. 1.3)**

$2H_2O \rightarrow O_2 + 4H^+ + 4e^-$ (For OER), with $E_o = 1.23$ **(eq. 1.4)**

$E = E_o + 0.059\times4 \log [H^+]^4 = E_o + 0.059 \log [H^+] = 1.23\text{-}0.059pH$ **(eq. 1.5)**

PARAMETERS USED TO EVALUATE THE CATALYTIC ACTIVITY

For estimating and comparing the catalytic activities of catalyst the broadly known parameters are described below. In this section, the advantages and disadvantages related with each of these parameters are as following:

Overpotential (η): Electrochemical reaction does not work at expected potential only by considering thermodynamic condition having no kinetic barrier are used[11]. Due to these barriers, a supplementary driving force given by appling additional potential will affect these electrochemical process, this is overpotential (η). There are three source of overpotential in case of OER and HER which is the activation and concentration overpotential, and the overpotential because of uncontributed resistance, which is due to electrochemical interfaces [12]. The material possess intrinsic property which helps in electrode reaction catalysis and may change from material to material. Therefore, this can be reduced by selected an effective catalyst. In HER, the activation overpotential, which is also known as onset overpotential, and it is highly important in comparison to others, as the kinetics of the HER works faster in comparison to OER [13]. The calculation is done by plotting a graph of the polarization curve which can be attained by a plot between overpotential and current density.

$$E_{applied} = 1.23 + \eta_{anode} + \eta_{cathode} + iR \qquad \textbf{(eq. 1.6)}$$

Tafel Slope:-

The Tafel plot is obtained by a plot between log(j) and η. The dependence b/w overpotential and the logatrithm of current density, is known as Tafel slope and below is the equation of tafel slope:-

$$\frac{d\log(j)}{d\eta} = 2.303RT/\alpha nF$$

Due to the high capacitive of the catalyst, the scan rate has becomes a serious problem and at this the polarization curve was determined and is further used for Tafel plot. To determinine the exchange current density which lead to significant error because the exchange current density is determined by logarithmic scale at zero overpotential and linear fit was extraploted toward the current density. In such a case the catalyst would have a large exchange current density that has a high overpotential. This would not have been possible as there is a large exchange current density which means that across the catalytic surface the transfer of electrons will be easy and need very less amount of activation energy. Hence, as a result the overpotential will be low.

The Tafel slope is the parameter that tells about the performance of an electrocatalyst. The equation for the steady state current density is as follows:

$$j = j_o e\ \alpha F\eta\ /RT \qquad \textbf{(eq. 1.7)}$$

with j_o = exchange current density, α = transfer coefficient, η = overpotential, F = Faraday constant, R = gas constant and T = temperature.

If we take napierian logarithms the equation becomes

$$\ln j = \ln j_o + \alpha F\eta/RT \qquad \textbf{(eq. 1.8)}$$

and if we convert it to natural logarithms

$$2.303 \log j = 2.302 \log j_o + 2.303\alpha F\eta/RT \qquad \textbf{(eq. 1.9)}$$

And dividing by 2.303

$$\log j = \log j_o + \alpha F\eta/2.303RT \qquad \textbf{(eq. 1.10)}$$

This can be arranged as

$$\eta = a + b \log j \qquad \textbf{(eq. 1.11)}$$

a and b are constants and the above expression is called as the Tafel equation. If we plot a graph between values of logarithm of current density at different values of potential and overpotential a linear region is obtained. This slope shows that how with change in given potential range electrocatalytic catalyst performs. Therefore, to compare different catalysts it is a useful parameter: the catalyst is better if tafel slope is smaller, so small icrease in overpotential is required for the increased current densities and also that even the small changes in the overpotential can "tune" the current density.

Stability: The electrocatalyst was subjected to test the stability at a higher scan rate by CV cycling, that is called as the accelerated degradation test, and to chronoamperometric or chronopotentiometric analyses. For HER, several thousands of cycles were carried out ones the polarization begins from 0V vs NHE done by accelerated degradation test. Whereas in case of OER, the degradation test requires a number of cycles which is from 250 to 1000. In very rare cases a report with extreme stability beyond 1000 cycles [4].

Faradaic Efficiency: It is one of the parameter which quantitatively is used for determination of HER and OER. It is the efficiency of an electrocatalyst when an external circuit effect the electrode reaction when by crossing the interface to electroactive species which lead to transfer of electron in this case HER or OER is used [12].

Turnover Frequency (TOF): At a known overpotential turnover frequency (TOF) is used to evaluate an electrocatalyst. The TOF for electrocatalyst can be calculated by following equation:

$$\mathbf{TOF} = \frac{I N_A}{A F n \Gamma}$$

where I = current,

NA= Avogadro constant,

A = geometrical surface area,

n = the number of electrons transferred,

and Γ is the surface or total concentration of catalyst.

CATALYSTS CATALYSTS

PRECIOUS METAL CATALYSTS

For hydrogen evolution, (Pd, Ru, Pt, Rh, and Ir) were proved to be an excellent OER and HER catalysts. The use of such type of rare and costly metals is not advisable as it increases the cost for production of hydrogen and oxygen, so the production on large scale is hindered.

NON PRECIOUS CATALYST

Catalyst base on abundant and less expensive element such as first row transition metals are recently examined either the HER or the OER. Now days, bifunctionally active Fe-based materials are been studied for water splitting. Martindale and Reisner recently reported a catalyst which consists of Fe only which is highly efficient water splitting used for half reactions in alkaline conditions and the formed catalysts showed greater activity than from a reported paper of bifunctional Co and Ni catalysts. Catalysts are one of the key factors for efficient energy conversion and storage. In past serval years water spitting had been tried using earth abundant elements (Co, Ni and Fe) as a catalyst for water splitting. Using cobaloxime WRCs hydrogen was produced using water and has been reported more than 9000 TON's and up to 1000 TON's has been found for oxygen production using cobalt based WOCs. More than 2700 TON's in case of hydrogen production and water oxidation was done though nickel oxide film. For water splitting iron is the most interesting element and abundantly available metal for water splitting [15].

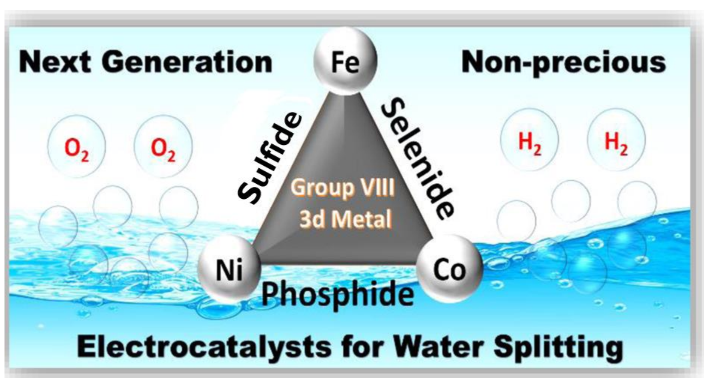

Figure 4: Non precious metals (3d metals)for electrocatalyst for water splitting [1].

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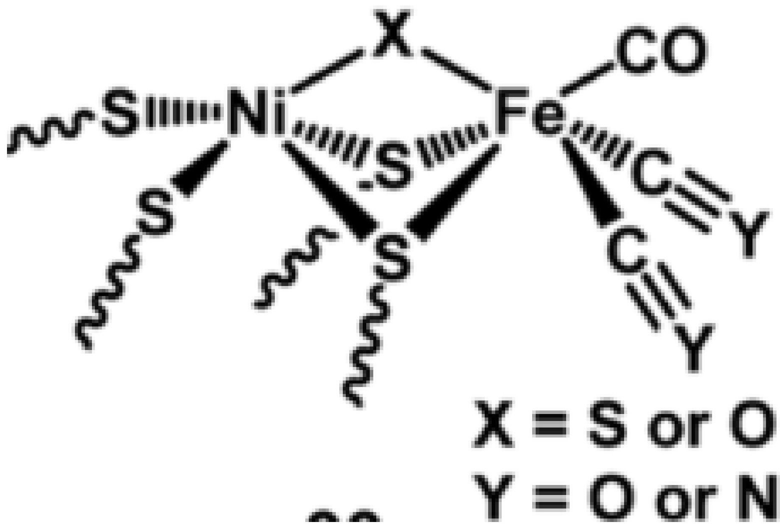


Figure 5: Non precious metal complex Ni,S,Fe complex [1].

OBJECTIVES

1) Developing highly active electrocatalyst based on non-precious metals.
2) To prepare high performance redox electrocatalyst with high surface area, charge mobility, high conductivity, more active sites and stability with low overpotential values for HER and OER.
3) To prepare mixed metal oxide Nanostructure as an electrocatalyst by solvothermal method.
4) Characterization of the prepared catalyst by phsico-chemical techniques like powder X-ray diffraction (powder XRD) and BET (Brunauer Emmett Teller).
5) Application of the prepared catalyst for water splitting to produce hydrogen and oxygen.

CHAPTER 2

MATERIALS AND METHODS

MATERIALS:

Inorganic salts, Iron(III)chloride (FeCl3),Nickel chloride (NiCl2), Urea, Sucrose, Iron(II)nitrate nonahydrate[$Fe(No_3)_3.9H_2O$], Nickel(II)Nitrate hexahydrate [$Ni(No_3)_2.6H_2O$], NaOH,Nafion117 from Sainenergy Fuel Cell India Pvt Ltd, Ethanol (CH_3CH_2OH) from RANKEM and Carbon Fibre Paper (CPF) were purchased from Fuel cell store.

METHODS:

X- ray diffraction studies (XRD)

From the XRD diffraction pattern of the samples it can be observed that the Ni-Fe Metal Oxide are present in the CNS. The broad peaks in the pattern are due to the amorphous carbon present in the sample which arise due to the polymerization and condensation of the sucrose. By using Rigaku Ultima IV fully automatic high resolution X-ray diffractometer system with Theta-Theta (θ-θ) Goniometer Powder X-ray diffraction patterns of the prepared catalysts were recorded. The generator operates at 20-60 kV with rated tube current of 2-60 mA. The samples were scanned in the range of $2\Theta=0\text{-}90^o$ at the scanning speed of 2 min.), ant type of carbon source present in the microspheres is completely removed by calcinations.

Brunauer Emmt Telleret (BET)

It is use to determine the structure of the a synthesized Ni-Fe Metal Oxide products and was checked using different characterization techniques. Through BET the surface area and pore diameter and pore volume of the synthesized sample and exact morphology can be determined.

CATALYST PREPARATION:

Without sucrose: For the formation of catalyst Nickel(II)Nitrate hexahydrate [$Ni(NO_3)_2.6H_2O$] (1.5mmol, 436.23mg) and Iron(II)nitrate nonahydrate [$Fe(NO_3)_3.9H_2O$] (1mmol, 404mg) were dissolved in 100mL of distilled water. Then NaOH was added dropwise into the solution with continuous stirring till pH 13 is attained. The solution was warmed at 80^0C for 60 minutes, and the resultant precipitate was collected by centrifugation. The collected precipitate was further washed serval times with distilled water and last washing was done with ethanol, and finally dried at 60^0C overnight in hot air oven. The dried precipitate was calcinated in air at 350^0C for 2hours inside a tube furnace at heating rate 2.5^{o}C min^{-1} and the obtained product was NiFe-nanoparticles and labeled as 1.

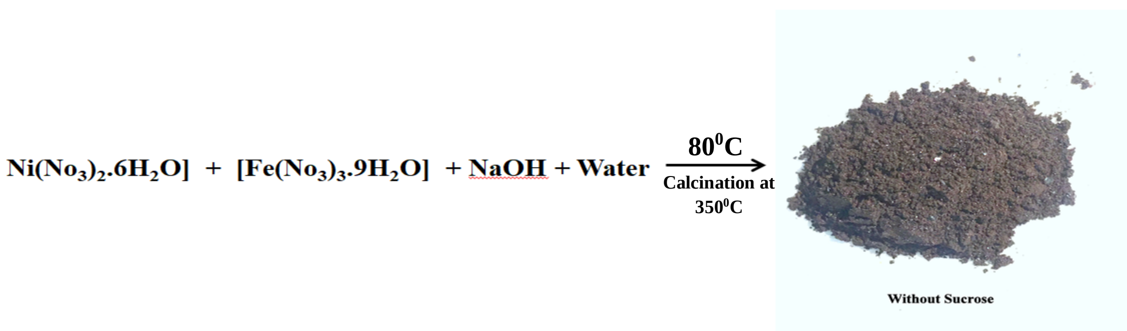


Figure 6: Preparation of nanoparticles without sucrose.

With sucrose: In distilled water(30mL) $FeCl_3$(0.65mmol, 105.43mg) and $NiCl_2$(0.65mmol, 84.23mg) were dissolved with continuous stirring to form complex. The formed solution was added dropwise to sucrose solution formed by adding sucrose (8.6g) in distilled water (100mL). The solution was transferred to autoclave and placed at 160^0C for 20 hours. Through hydrothermal process powder is formed which is further calcinated in air at 400^0C for 2 hours inside a tube furnace at heating rate 2.5^{o}C min^{-1} and fine powder was obtained and labeled as $1_{\textbf{sucrose}}$.

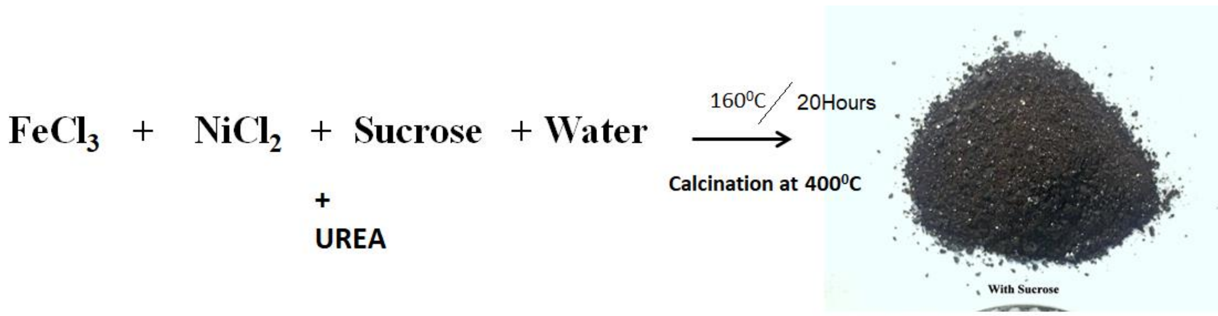


Figure 7: Synthesis of sample using sucrose.

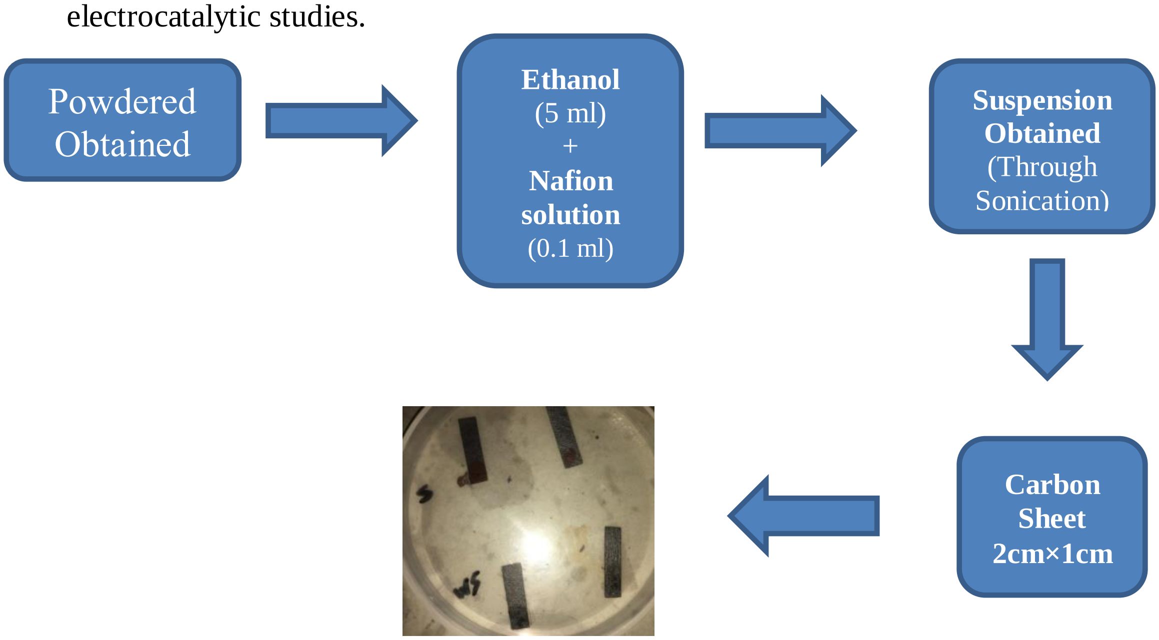
ELECTRODE PREPARATION

WORKING ELECTRODE PREPARATION

For the all working electrodes, crystals of all these Metal Oxides Nanostructure (1 mg) were added to ethanol(5ml) and Nafion(0.1ml) solution in an eppendorf. The mixture was sonicated for 1 hour and the suspension obtained was evenly spread over 2cm×1cm carbon sheet. It was then oven dried for overnight giving the desired results.

Preparation of 1M KOH used as an electrolyte

For the preparation of 500ml of 1M KOH, in a volumetric added 28.0582 gm of KOH. After that water was added up to the mark on volumetric. Mixed it well to obtain a clear solution for further electrocatalytic studies.



Scheme 1: Explains the detailed formation process of the Ni-Fe Complex solution. Firstly, sucrose, Ni-Fe precrussor and urea was dissolved in aqueous solution with continous stirring and then the solution by kept in autoclave. Condensation and polymerization of sucrose in water by hydrothermal process led to formation of carbon microspheres. Further these submicrospheres are calcinated in tube furnane and multishelled NiFe-NP's are (**Figure 8).**

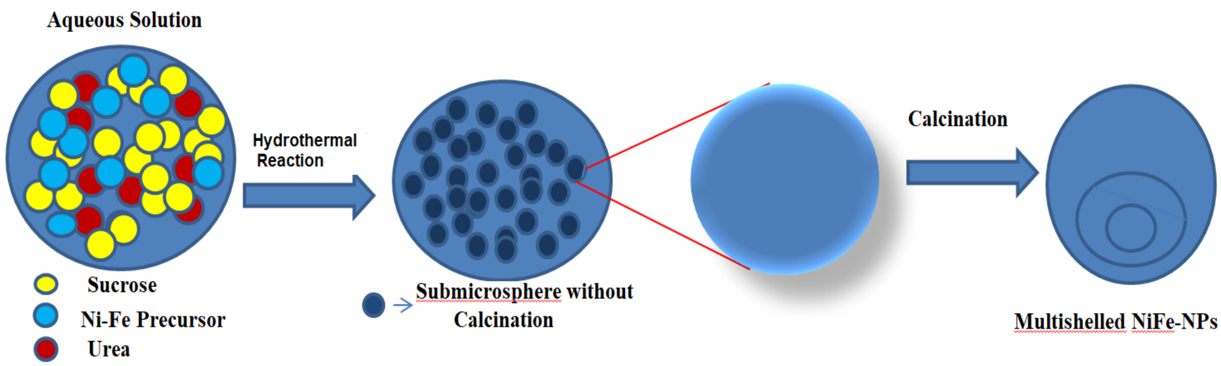


Figure 8 : Scheme explaining the synthesis of nanoparticles.

CHAPTER 3

RESULTS AND DISCUSSION

XRD Analysis

The mixed valence Metal Oxides Nanostructures $1_{Sucrose}$ & 1 were synthesized by reaction of $FeCl_3$ with $NiCl_2$, $FeCl_2$, Urea respectively, and Sucrose Solution. solvothermally under 160^0C for 12 hours. Then these Metal Oxide Nanostructure have been deposited in carbon paper by drop casting methods.

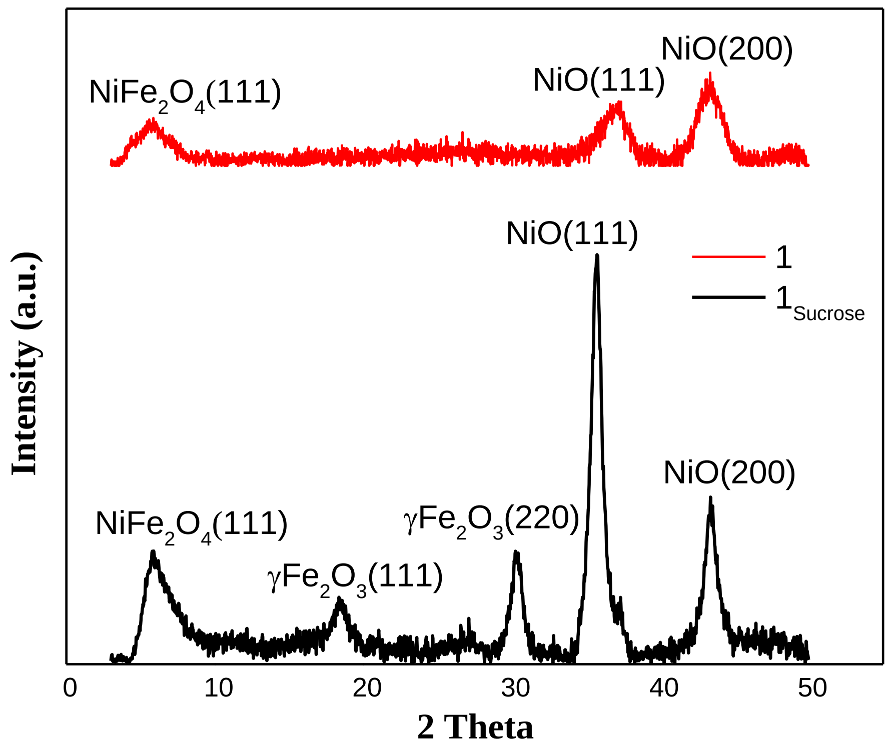
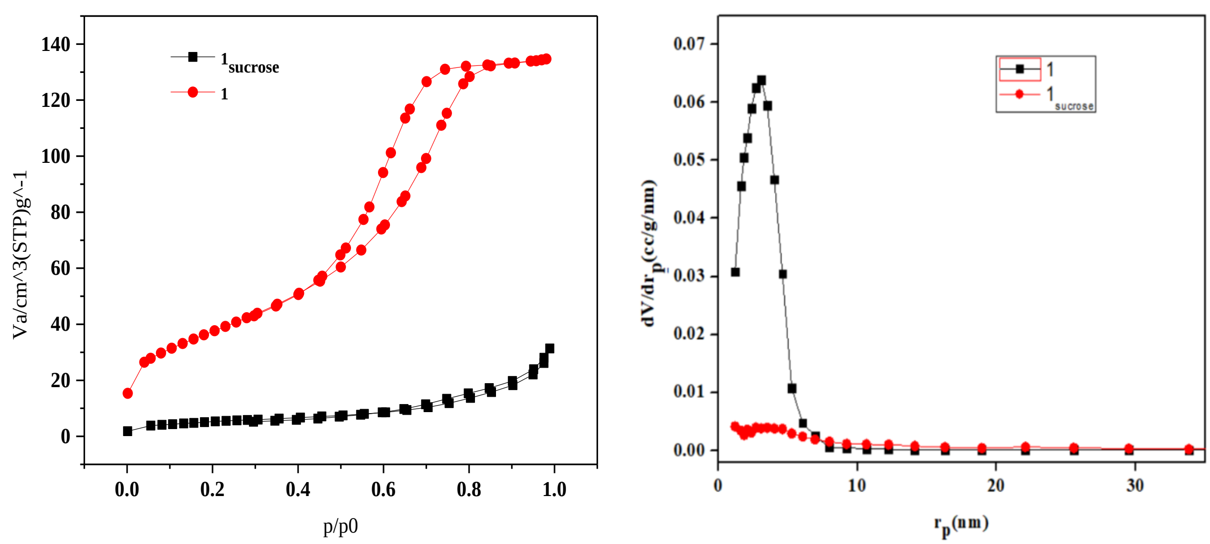


Figure 9: XRD Diffraction Pattern of sucrose and without sucrose.

The powder XRD studies were performed for the prepared mixed valence metal oxide nanostructures. In sample 1; the representative diffraction peaks at 37.12° and 43.18° correspond to (111) and (200) planes of NiO, respectively (JCPDCS No. 47-1049) and peak at 5.9° corresponding to $Ni_2Fe_2O_4$. In case sample $1_{sucrose}$; the characteristic peaks at 35.62° and 43.41° correspond to (111) and (200) planes of NiO, respectively (JCPDCS No 47-1049) , the peak at 5.8° corresponding to $Ni_2Fe_2O_4$ while diffraction peaks at 18.33° and 30.41° correspond to (111) and (220) planes of γ–Fe_2O_3, respectively (JCPDCS No. 39-1346).

BET ANALYSIS

Surface area of the as prepared catalyst were analyzed by using BET. In this analysis N_2 desorption-adsorption was performed to determine the surface are of prepared catalysts. Initially samples were preheated in a vacuum oven at 100°C for about 5-6 hours to eliminate any kind of additional foreign particle before N_2 adsorption/desorption.



(a) **(b)**

Figure10. (a) N_2 adsorption and desorption isotherms of as prepared Ni, Fe-based catalysts and **(b)** pore size distribution of prepared Ni, Fe-based catalysts

The isotherms are supposed to be prevented from any other adsorbents on the surface. The specific surface area of **1**$_{sucrose}$ and **1** were calculated ~19.3 m^2/g and ~13.4 m^2/g respectively. By using BJH method the pore size distribution of prepared catalysts was obtained. The total pore volume obtained for catalyst **1**$_{sucrose}$ is ~0.0485 cm^3/g and the corresponding mean pore diameter is 10.066 nm. For catalyst **1** the pore volume calculated is ~0.2083 cm^3/g and the corresponding mean pore diameter is ~6.228 nm. **Figure10(a)** shows N2 adsorption and desorption isotherms of **1**&2suggesting the mesoporous nature of prepared catalysts corresponding to IV adsorption isotherm. (**b)** shows pore size distribution of **1**$_{sucrose}$ & **1**. It shows that both halve similar external surface area but **1**$_{sucrose}$ hase significantly higher pore volume compare to the other.

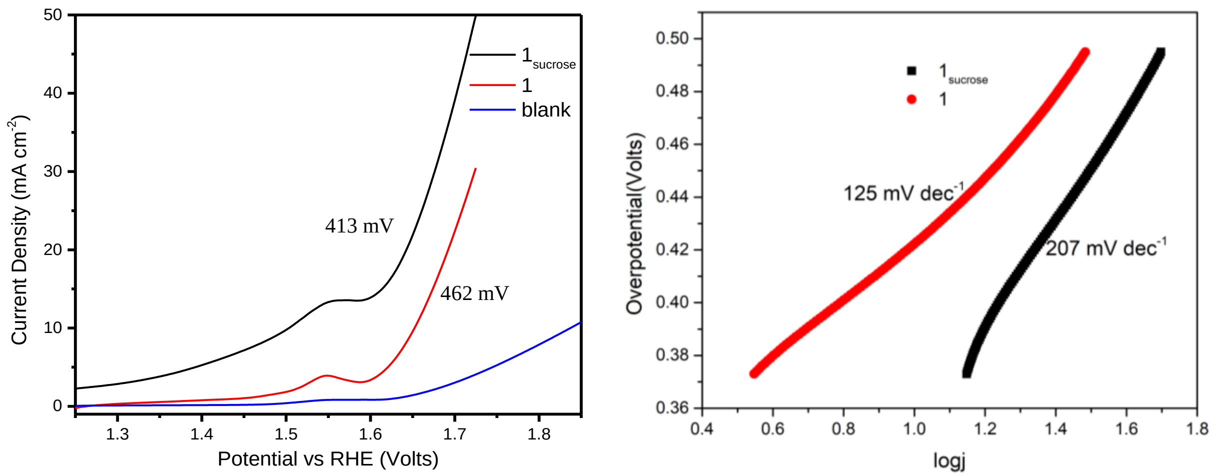
CATALYST	**Surface Area (m^2g^{-1})**	**Pore Diameter (nm)**	**Pore Volume (cm^3g^{-1})**
[1$_{SUCROSE}$]	19.3 m^2/g	10.066 nm	0.0485 cm^3/g
[1$_{WITHOUT\ SUCROSE}$]	13.4 m^2/g	6.228 nm	0.2083 cm^3/g

Table.1. Comparison of **1**$_{sucrose}$ and **1** through BET Surface Area Analyzer.

ELECTROCHEMISTRY ANALYSIS

OER ANALYSIS

The electrocatalytic oxygen evaluation reaction (**OER**) activities of these as prepared mixed Metal Oxides Nanostructures were evaluated by recording the linear sweep voltammetry (**LSV**) polarization curves using a typical three electrode system having the Metal Oxides Nanostructures as working electrodes, Ag/AgCl/KCl as reference electrode and Pt as counter electrode using 1M KOH solution as the electrolyte with a scan rate of 10 mVs^{-1}. Fig.1a shows the LSV polarization curves comparing the **1** & **2**. These Metal oxide Nanostructure require overpotential values of **413 & 462 mV** respectively **(Figure 9a)**. Thus, it suggests that catalyst **1** exhibits superior activity followed by **2**.



(a) **(b)**

.Based on these observations we suggest the following mechanism for OER. First the divalent cation undergoes oxidation to its trivalent state followed by oxidation of water by the trivalent cation .

Figure11.**(a)** The OER polarization curves and **(b)** the corresponding, Tafel plots of Ni/Fe, Fe/Fe-based catalyst in 1.0 M KOH.

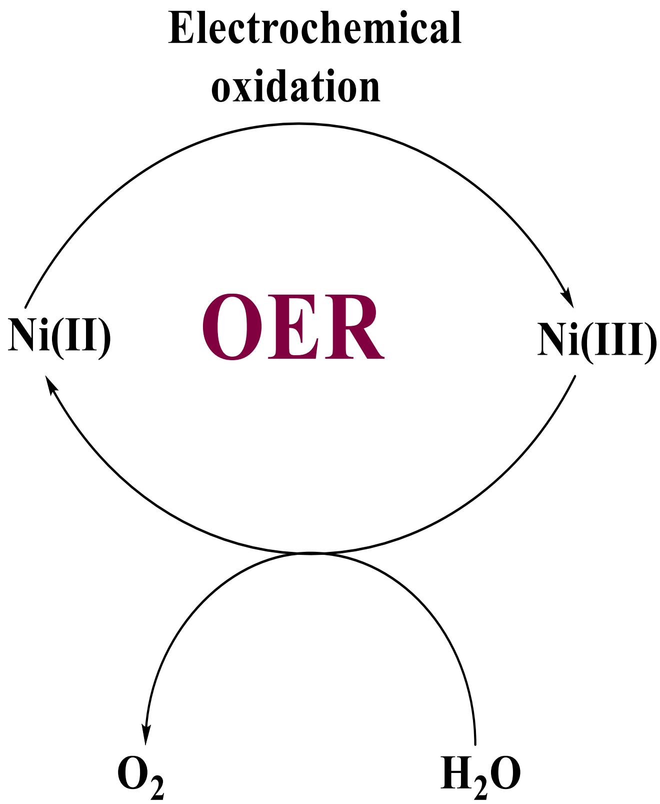
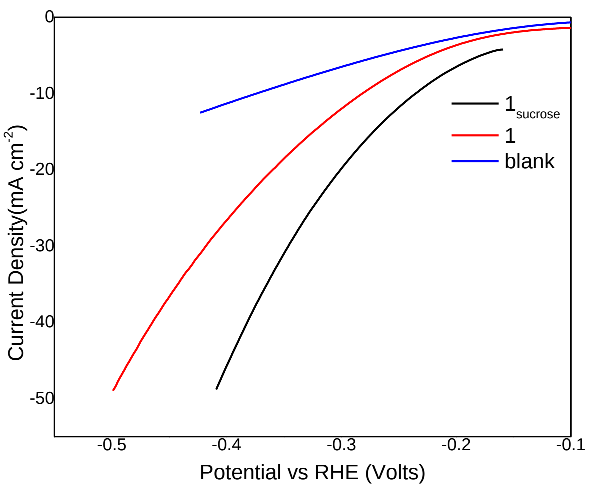


Figure 12. Scheme showing mechanism for OER.

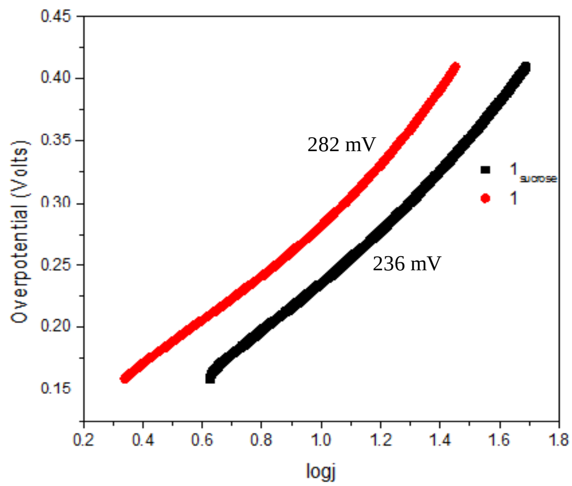
Such assumptions are validated by the OER activities of these MOFS. The related Tafel plots are shown in Fig.12.b and from the Tafel equation, yielding Tafel slopes of 413 & 462 mV dec^{-1} for $\mathbf{1_{sucrose}}$ **&** **1** respectively. The Tafel slope values suggest that **1** catalyst exhibits the best OER kinetics.

HER ANALYSIS

The electrocatalytic hydrogen evaluation reaction (**HER**) activities of these as prepared mixed Metal Oxides Nanostructures were evaluated by recording the linear sweep voltammetry (**LSV**) polarization curves using a typical three electrode system having the Metal Oxides Nanostructures as working electrodes, Ag/AgCl/KCl as reference electrode and Pt as counter electrode using 1M KOH solution as the electrolyte with a scan rate of 10 mVs^{-1}. **Fig.11a** shows the LSV polarization curves comparing the $\mathbf{1_{sucrose}}$ & **1**. These Metal Oxides Nanostructures require overpotential values of 236&282 mV respectively (**Fig. 11a**). Thus, it suggests that catalyst **1** exhibits superior activity followed by **2** .



(a)



(b)

Figure 13.(a) The HER polarization curves and **(b)** the corresponding, Tafel plots of Ni/Fe, Fe/Fe-based catalyst in 1.0 M KOH.

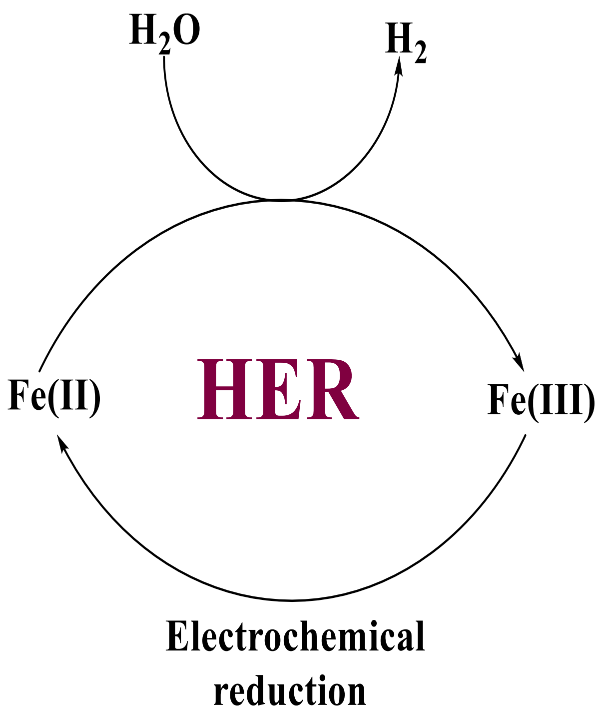


Figure 13. Scheme showing mechanism for HER.

Among two divalent ions Fe(II) is the strongest reducing agent as a result the Fe-base catalyst shows lowest overpotential for **HER**.. Such assumptions are validated by the HER activities of these MOFS. The related Tafel plots are shown in **Fig.17.b** and from the Tafel equation, yielding Tafel slopes of 236&282 mV dec^{-1} for $1_{Sucrose}$ &1 respectively. The Tafel slope values suggest that **1** catalyst exhibits the best **HER** kinetics.

WATER SPLITTING ANALYSIS

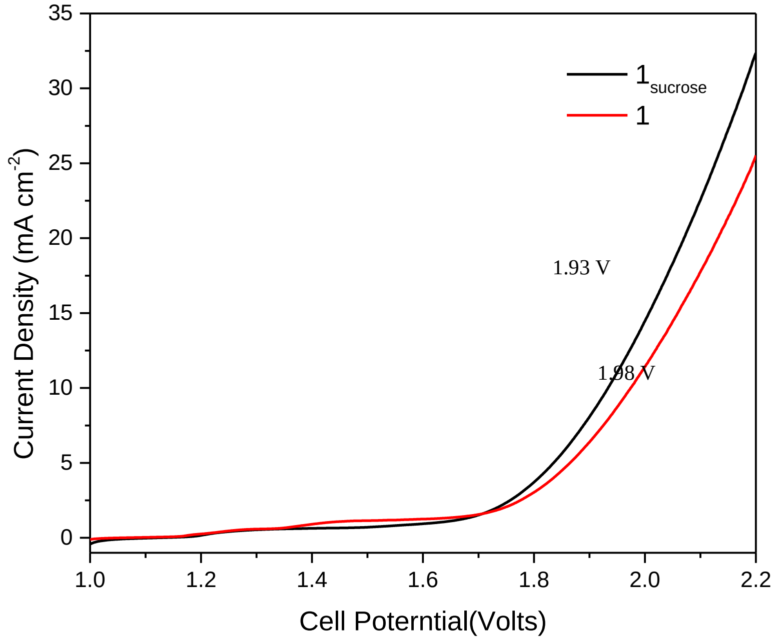


Figure 13: Linear Sweep Voltammetric curves of water splitting of the prepared catalyst.

Inspired by all the above electrochemical studies, we studied the performance of catalyst $\mathbf{1}_{\text{Sucrose}}$ for water splitting in 1 M KOH. **Fig.13.(a)** shows water splitting polarization curve for the catalyst $\mathbf{1}_{\textbf{sucrose}}$**.** The curve suggests that for catalyst **1** the potential achieved is 1.93 V for current density of 10 mA cm^{-2}. For catalyst **1** the potential obtained is 1.98 V a. From the data it can be observed that catalyst **1** gives smaller potential value compared to **2** Therefore, the curve shows better activity for the catalyst $\mathbf{1}_{\textbf{sucrose}}$ which is probably due to its higher pore volume..

CHAPTER 4

CONCLUSION

Water splitting plays a very important role in replacing non-renewable sources of energy by eco-friendly and cost effective techniques. Water splitting leads to generation of hydrogen which can be used in various fields as fuel. Among the two samples, the one formed with sucrose shows better result by exhibiting excellent electrocatalytic performance in HER and OER. $1_{sucrose}$ has lower overpotential value both in case of HER and OER compare to **1**. was 413mV. The water splitting potential obtained in case $1_{sucrose}$ was 1.93V better than catalyst 1 with 1.98V, clearly determines that $1_{sucrose}$ is better electrocatalyst for water splitting as the metal used are non precious and abundant on earth.

WORK TO BE DONE

1. Electron microscopy measurements to find out particle size and morphology
2. Stability of these catalysts under electrochemical environment
3. Faradaic efficiency analysis.

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