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Para o Desenvolvimento Ambiental, Cultural e Socioeconômico

Xosé Somoza Medina
(organizador)

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VOL IV

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PRÓLOGO

En este nuevo libro de la serie “Ciencia e Tecnologia para o desenvolvimento ambiental, cultural e socioeconómico” se han seleccionado diecisiete trabajos de gran calidad académica y capacidad de transferencia a la sociedad. Este último aspecto debe ser subrayado y puesto en valor. Un gran número de investigaciones publicadas en revistas de elevado nivel científico carecen de este impacto en la sociedad al desarrollar debates teóricos muy específicos que no tienen una traslación a la rutina diaria fuera de los laboratorios y aulas universitarias. En cambio, en todos los artículos que componen esta monografía se traslada de forma explícita la voluntad de las autoras y los autores de que sus investigaciones tengan un impacto real en la sociedad. Esta manera de actuar es una forma que tenemos las personas que nos dedicamos a investigar de devolver a la sociedad una parte de lo que se nos ha dado para poder dedicarnos profesionalmente a producir ciencia y tecnología.

Como en ocasiones anteriores, los trabajos publicados en este volumen se dividen en dos grandes apartados: Ciencia, con diez aportaciones, y Tecnología, con siete. En el primer apartado, Ciencia, sobresalen una serie de conceptos que muestran esa voluntad de transferencia a la sociedad, son innovación, gestión del conocimiento, y digitalización, aplicables principalmente a pequeñas y medianas empresas. El primer trabajo desarrolla el modelo de conocimiento e innovación sostenible en las PYMES, mientras que el siguiente capítulo estudia las condiciones necesarias para que surja la innovación y el tercero los sistemas de selección de personal en las PYMES a través del análisis de puestos. El cuarto capítulo analiza la innovación a través de un estudio de caso, concretamente el de una empresa familiar del sector de la construcción en México; el quinto traslada un diagnóstico de la transformación digital en las PYMES realizado en Bogotá y el sexto muestra como las características culturales afectan la planeación estratégica de las MIPyMES mexicanas. El séptimo trabajo estudia la capacitación digital de la demanda de turismo cultural en Michoacán. Los dos capítulos siguientes muestran nuevos modelos de gestión en las universidades, trasladables a las empresas, y el último trabajo desarrolla aspectos sobre la legislación mexicana en materia de protección de datos.

El segundo bloque de esta monografía, Tecnología, agrupa siete investigaciones aplicadas desde los campos de la ingeniería agrícola, geológica, o química. Los dos primeros trabajos son de agronomía, con investigaciones empíricas sobre residuos cítricos y maíz azul. El tercer trabajo analiza riesgos ambientales geológicos en la cuenca Inambari, en Perú y el cuarto trabajo las consecuencias de los incendios forestales en el Estado de Jalisco, México. Los tres capítulos siguientes desarrollan investigaciones de química aplicada, orientada a la electrólisis, las nanoestructuras o la metalurgia de las superaleaciones, que es el trabajo que cierra este volumen de “Ciencia e Tecnologia para o desenvolvimento ambiental, cultural e socioeconómico”.

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SUMÁRIO

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TECNOLOGÍA

CAPÍTULO 15

OXYGEN REDUCTION REACTION ON FENSEC MATERIALS, THEIR ELECTROLYTIC ACTIVITY IN ACID MEDIA

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ABSTRACT: A study of the oxygen reduction reaction (ORR) on FeNSeC electrocatalyst, derived from heat treatment of a chelate synthesized by a chemical reaction of dimethylglioxime, $FeCl_2$ and Na_2SeO_3 in aqueous media was investigated. The catalyst is composed of agglomerated heterogeneous particles with about 0.5 µm with irregular flakes forms. The ORR kinetics studied by rotating disk electrode (RDE) in a 0.5 M H_2SO_4 showed that catalytic activity towards the ORR occurs via a multi-electron transfer process (n = 4e$^-$) to water formation.

KEYWORDS: Oxygen reduction. Electrocatalyst. Tafel slope. Rotating disc electrode. Fuel cell.

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REDUCCIÓN DE REDUCCIÓN OXÍGENO SOBRE MATERIALES FENSEC, SU ACTIVIDAD ELECTROLÍTICA EN MEDIO ÁCIDO

RESUMEN: Se realizó un estudio de la reacción de reducción de oxígeno (RRO) con electrocatalizadores FeNSeC, derivados del tratamiento térmico de un quelato sintetizado por una reacción química de dimetilglioxima, $FeCl_2$ y Na_2SeO_3 en medio acuoso. El catalizador está compuesto por aglomerados de partículas heterogéneas de cerca de 0.5 μm en forma de hojuelas irregulares. La cinética de la RRO estudiada por electrodo de disco rotatorio (EDR) en una solución 0.5 M de H_2SO_4 mostró que la actividad catalítica hacia la RRO ocurre a través de un proceso de transferencia multi-electrónica (n = $4e^-$) hacia la formación de agua.

PALABRAS CLAVE: Reducción de oxígeno. Electrocatalizador. Pendiente de Tafel. Electrodo de disco rotatorio. Celda de combustible.

1 INTRODUCTION

Hydrogen and proton exchange membrane fuel cells (PEMFC) technologies offer a feasible alternative area of research because they represent an environmentally clean and friendly energy source, for applications in many human activities. At the present, one of the main challenges is to reduce the fuel cell cost by developing low-cost materials and components which include oxygen reduction catalysts, solid polymer membranes, gas diffusion electrodes and bipolar plates, Zhang et al. (2007). The oxygen reduction reaction in the cathode electrode is the restrictive reaction of the fuel cell efficiency. For this reason, much attention has been paid to catalysts obtained from the heat treatment around 600-1000 ºC, in an inert and reductive atmosphere of transition metal macrocycles, which are in general related to porphyrins (Takahiro et al. (2010), Tu et al. (2010), Sarakonsri et al. (2009), Richards et al. (2009), Faubert et al. (1996)) and phthalocyanines type (Ozaki et al (2010), Baker et al (2009), Li et al. (2010), Yu et al. (2009)). Literature associated to similar studies of this materials reported the presence of the coordination site N_4-Me (Me = Fe or Co), as a paramount importance in generating catalytic sites during the pyrolysis of the active centers in the catalytic reaction. According to this, Faubert et al. (1998, 1999) demonstrated that nitrogen is not required in the precursor's main structure. However, its presence plays an important role during the pyrolysis reaction of polyacrylonitrile with Co and Fe salts. The reported catalytic activity of these materials is similar to that reported for noble metals with limited stability. In the other hand, some other authors have reported that the catalytic activity highly depends of the type of carbon on which the precursor macrocycle is supported (Elizabeth et al. (2010), Fanny et al. (2008), Ja-Yeon et al. (2010), Cicero et al. (2008)). Furthermore, it is well known that the association of transition metals with chalcogens such as selenium enhances their catalytic activity (Suárez-Alcántara et al. (2006), Bron et al. (2001), Solorza-Feria et al. (1999)).

In the present study the electrocatalytic activity of such materials is presented towards the oxygen reduction reaction (ORR) by pyrolized material without carbon support as well as the evaluation of the influence of selenium in this synthesized material according to ORR activity. Besides, another important and attractive contribution of this work is to obtain materials at very low cost with catalytic activity comparable with big macrocycles of higher costs.

2 EXPERIMENTAL

2.1 MATERIALS

Dimethylglyoxime (DMG 99% Merck), $FeCl_2.4H_2O$ (99% Aldrich), Na_2SeO_3 (99% Aldrich), deionized water, H_2SO_4 (95-97% Aldrich), carbon Vulcan XC7TR, Nafion 5 wt% (Du Pont, 1100 EW) was purchased from Aldrich and the glassy carbon (GC) rod was supplied by Sygradur.

2.2 PRECURSOR PREPARATION

Chelate $Fe(DMG)_3(SeO_3)_2$ was prepared following the synthetic route developed by Jackels et al. (1973). Briefly, 3 mmol (0.348 g) of DMG were dissolved in deionized water at 100 ºC. Then, 1 mmol (0.198 g) $FeCl_2.4H_2O$ was added carefully, while stirring the solution for 30 min at the same temperature. Thereafter, 2 mmol (0.346 g) of Na_2SeO_3 were added slowly to a stirred solution for 30 min under reflux conditions. Then, the solution was concentrated and cooled. The precursor was separated by centrifugation and the resulted solid was washed with acetone and ethanol for three times. Finally the solid product was dried at 110 ºC, for 1 hour. Due to the good solubility of this precursor in water, it was not possible to develop kinetic studies in this stage.

2.3 CATALYST PREPARATION

The chelate $Fe(DMG)_3(SeO_3)_2$ was thermally treated for 2 hours at 600 ºC under nitrogen atmosphere. After the heat treatment, the catalyst was cooled down with nitrogen gas up to room temperature, washed several times with deionized water, acetone and ethanol and dried 1 hour in a furnace at 110 ºC. In order to test the reproducibility of this method, six repetitions were carried out with their consequent products (samples (1-5, 7)FeNSeC).

2.4 MORPHOLOGICAL AND CHEMICAL CHARACTERIZATION

The surface morphology of the cluster catalyst was examined using a scanning electron microscope (SEM, JEOL 3600), equipped with an energy dispersive X-ray (EDX) unit used to analyze the chemical composition at different points of the sample. Particle size was determined by transmission electron microscopy (TEM, JEOL/JEM 2000 EX). X-ray diffractions were recorded by a Siemens D5000 powder diffractometer, equipped with a Cu Kα radiation source.

2.5 ELECTROCHEMICAL PERFORMANCE

Discs with a cross-sectional area of 0.196 cm^2 were used as support for the thin films as well as working electrodes. The GC electrode surface was modified as follows: 1 mg of the catalyst and 0.2 mg of carbon Vulcan XC-72R were added to an alcoholic solution (0.40 mL) with 8 μL of commercial 5 wt% Nafion. The resulting mixture was sonicated for 5 min and 10 μL were pipetted onto the GC surface. The amount of catalyst on the disc was consequently 0.125 mg/cm^2.

The electrochemical measurements were performed by using the conventional single compartment of a three-electrode array. Linear voltammetry and rotating disc electrode (RDE) studies were carried out at 25 ºC. A platinum mesh (~4 cm^2) was used as counter electrode and Hg/Hg_2SO_4 0.5 M H_2SO_4 (MSE=0.68 V/NHE) as reference electrode. All the potentials are referred to normal hydrogen electrode (NHE). A potentiostat (EG&G, model 273A) was used for the steady-state experiments. Hydrodynamics experiments were recorded in oxygen-saturated 0.5 M H_2SO_4 under a rotation rate range of 200–1600 rpm at 5 mV/s. Prior to the electrochemical measurements, the solution was degassed with nitrogen for the working electrode activation. The electrode activation was carried out by scanning the potential between 0.83 V and 0.03 V/NHE for 20 min. afterwards, the electrolyte was saturated with oxygen in order to measure the ORR activity.

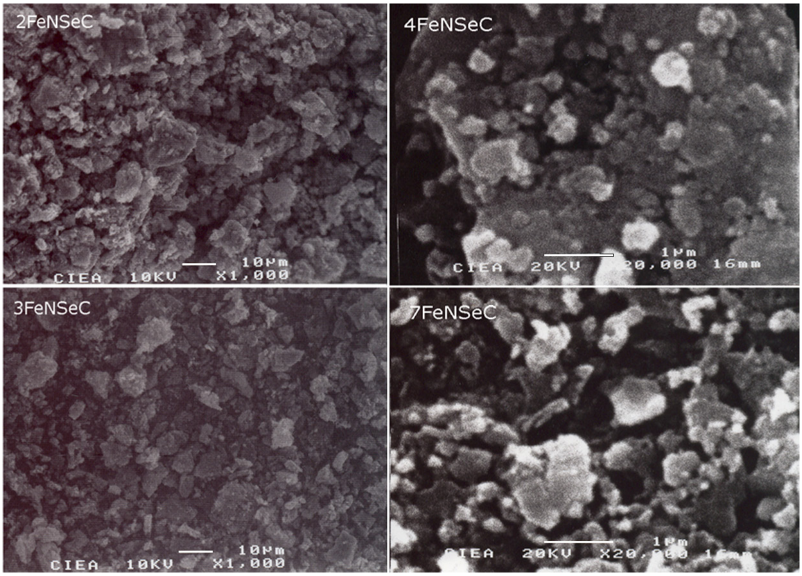
3 RESULTS AND DISCUSSION

3.1 CHARACTERIZATION OF FENSEC CATALYSTS

The black dust catalyst (FeNSeC) obtained by the thermal treatment procedure was characterized chemical and morphologically. Fig. 1 shows only the most representatives SEM photomicrographs of the 2-4,7FeNSeC catalysts treated at 600 ºC. Presence of agglomerates composed by irregular particles flakes-like is observed on 2 and 3FeNSeC SEM images of the left side of Fig. 1. At higher magnification (from 10 to

1 µm) the samples 4 and 7FeNSeC shows those agglomerates of particles smaller than 1µm (see images of right side in Fig. 1). Also, these agglomerates of the samples show homogeneous distributions of the FeNSeC catalyst with small particles of about 0.5 µm in size. As illustrated in Fig. 1, in the samples 4,7FeNSeC can be appreciated that some particles develop more brightness as a possible consequence of higher oxygen content on this sites. This indicates the presence of oxides in the sample as can be corroborated by the EDX analysis (Table 1, specific area and Fig. 3).

Fig. 1. SEM images of FeNSeC catalysts prepared by pyrolysis of Fe(DMG)$_3$(SeO$_3$)$_2$. Left side images (2,3FeNSeC) were taken using a 10 µm scale whereas in the right side can be observed the 4,7FeNSeC images with 10 times higher resolution (1 µm scale).



TEM image, shown in Fig. 2, depicts possible agglomerate composed by bigger particles of about 0.5 µm in size (dark ones) together with smaller particles (lighter ones).

Fig. 2. TEM image of the 2FeNSeC catalyst. This sample shows heterogeneous distribution of particle size. However, the size ratio of the darker and bigger particles is around 500 nm.

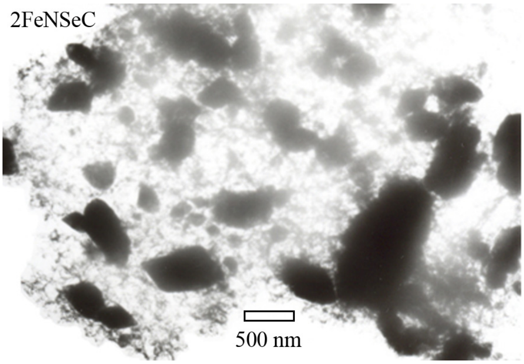
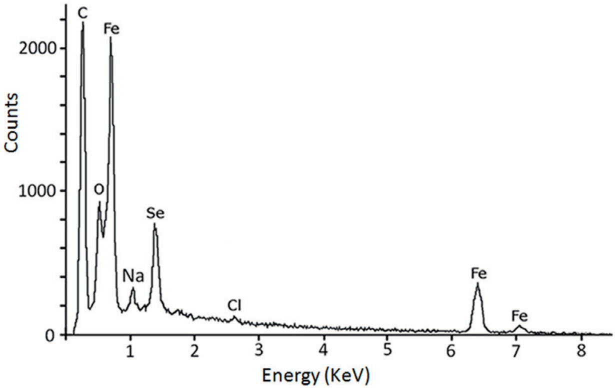


Figure 3 shows the EDX spectrum of 2FeNSeC catalyst where the characteristic signals for iron (~0.7, 6.4 to 7 keV) and selenium (~1.4 keV) are observed. Traces of sodium and chloride were also detected as remained impurities from the synthesis process. Moreover, signals attributed to nitrogen and oxygen are observed as well as at ~0.2 and 0.4 keV corroborate the chemical composition of the catalyst.

Fig. 3. EDX spectrum of 2FeNSeC catalyst. Labeled signals assigned to iron, selenium, and nitrogen atoms corroborate the chemical composition of the catalyst.



Some signals of the X-ray diffractogram of the synthesized 2FeNSeC, Figure 4a, matched in good agreement with some typical signals attributed to Fe_2O_3 (vertical lines pattern). However, is well known that this material is not catalytically active for the ORR. No peaks for Fe_2O_3 were detected in the 4FeNSeC sample, Figure 4b. On the other hand, when characteristic pattern signals from Fe_3C (Cohenite) are compared with the 4FeNSeC diffractogram a better fit is reached.

Fig. 4. X-ray difractograms: a) 2FeNSeC and b) 4FeNSeC catalysts compared with Fe_2O_3 and Fe_3C lines patterns, respectively.

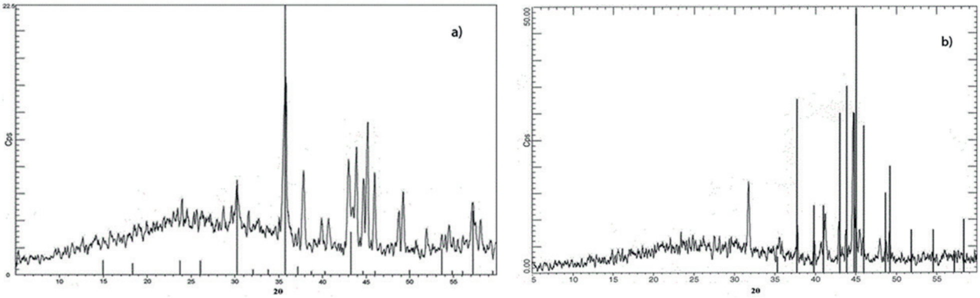


Table 1 shows the results of the EDX analysis. These results corroborate the high quantity of iron attributed to a strong interaction of the catalytic sites formed by Fe-N-Se-C particles. It was also observed that the presence of selenium favored the stabilization of the complex, in a similar form that occurs when incorporated to ruthenium as reported by Bron et al. (2001), or suppress the catalytic formation of graphite. Selenium can also coordinate to iron blocking the formation of Fe_3C, as reported by Tributsch et al. (2008).

In general, data of Table 1 show homogeneity in the distribution of the elements in the catalytic material (areas 1 and 2 with 1000 μm^2). On the other hand, the atomic percentage in specific areas (denoted as bright white particles in Fig. 1) indicate the presence of oxides, mainly from iron. Such behavior can be attributed to a clear increment in the amount of oxygen in all the samples.

Furthermore, sample 3FeNSeC contains the higher concentration of nitrogen. In contrast, sample 2FeNSeC shows the lower nitrogen concentration as well as different values among specific areas 1 and 2 comparable in this point with sample 7FeNSeC. However, it is not clear whether the relationship of the nitrogen content of the samples represents an advantage in catalytic activity or stability.

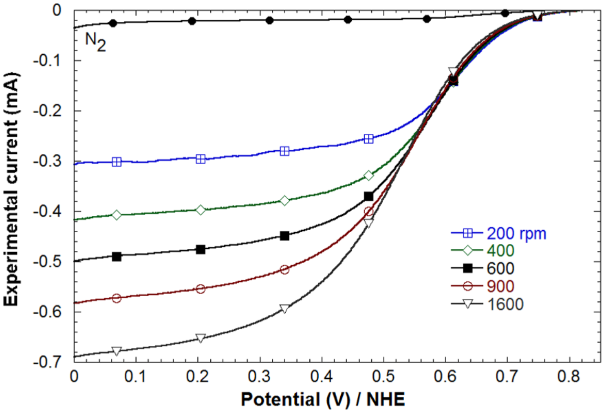
Table 1. Elemental atomic analysis (at%) obtained by EDX, for FeNSeC catalysts.

Material		Fe	Se	N	C	O
2FeNSeC	area	5.39	0.84	9.47	77.75	10.89
	specific	7.24	1.32	3.80	64.33	23.29
3FeNSeC	area	3.61	0.81	11.23	81.02	3.72
	specific	4.56	1.13	15.12	59.67	19.40
4FeNSeC	area	5.08	1.89	7.26	67.26	13.07
	specific	7.65	3.78	5.67	56.11	22.02
7FeNSeC	area	3.63	0.89	9.65	81.18	4.25
	specific	7.94	2.06	11.56	54.27	23.13

3.2 ELECTROCHEMICAL PERFORMANCE

The electrochemical activity of FeNSeC catalysts for the oxygen reduction reaction was determinated by linear voltammetry and the rotating disc electrode technique. Fig. 5 depicts the experimental polarization *i-E* curves for oxygen reduction on 2FeNSeC catalyst, where a dependence of the current with the rotation rates is observed at high overpotential.

Fig. 5. RDE measurements of oxygen reduction of 2FeNSeC in 0.5 M H_2SO_4 at a scan rate of 5 mV/s, 200-1600 rpm and 25 °C. The potential sweep is toward cathodic direction.



The catalytic currents mentioned above, as a function of the rotation speed (in rpm), fulfill the Koutecky-Levich equation:

$$1/i = 1/i_k + 1/i_l = 1/i_k + 1/B\omega^{1/2} \qquad (1)$$

Where i is the experimental current, i_k represents the kinetic current in the absence of any mass transfer effects, i_l, is the limiting current, $\omega = 2\pi f$ is the angular velocity, f is the angular frequency given in rps and 1/B is the Koutecky-Levich slope expressed as:

$$1/B = 1/[0.62nFACo_2(Do_2)^{2/3}\nu^{-1/6}] \qquad (2)$$

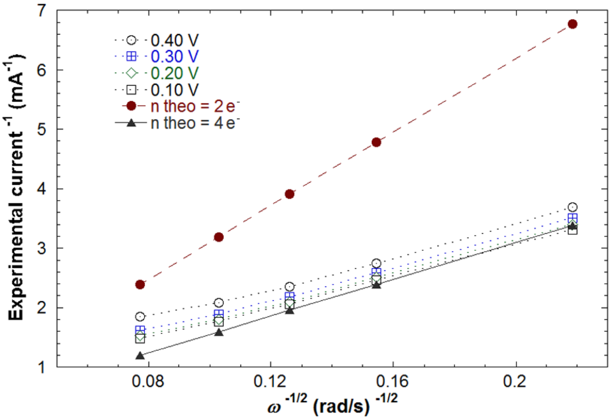
Where, Do_2 is the diffusion coefficient of oxygen (1.4×10^{-5} cm²/s) Hsueh et al (1983), Co_2 is the bulk concentration of oxygen (1.1×10^{-6} mol/cm³), ν is the kinematic viscosity of the electrolyte (0.01 cm²/s), n is the number of electrons transferred by oxygen molecule, F is the Faraday constant and A is the electrode area.

Fig. 6 illustrates the plot (1/i) against $\omega^{-1/2}$ to several potentials (where i is the experimental currents from Fig. 5). As can be appreciated in this graph, the slope of the experimental straight lines is similar with the theoretical slope calculated by means of the Levich equation when $n = 4e^-$ and A corresponds to 0.196 cm². According to this, it can be proposed that any variation of the experimental slopes with the theoretical one is due to a different electrochemical area (consider as a catalytic effective area). Considering this argument, the catalytic effective area was calculated for all the samples of FeNSeC catalyst from the Ec. (3):

$$A_{eff} = (60/2\Delta)^{1/2}\times(\nu^{1/6}/BnFC_{O2}D_{O2}^{2/3}) \qquad (3)$$

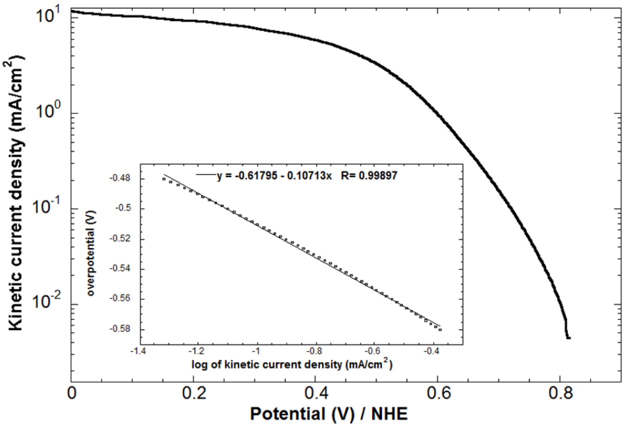
Where B = 3.006 vs. 3.034 (theoretical value for *n* = 4e$^-$), is the experimental average of the Koutecky-Levich slopes obtained in Fig. 6. Similar considerations were presented in the work of Alonso-Vante et al. (1995). The resulted areas from each catalyst were used for normalizing the experimental currents and recalculate the Koutecky-Levich slopes in order to obtain the limiting currents as Bard and Faulkner (1980) reported. The average kinetic current obtained in this form at different angular velocities was used in order to obtain the kinetic parameters as is indicated by Bard and Faulkner (1980).

Fig. 6. Comparison of experimental slopes with theoretical Koutecky-Levich slopes when *n* =4e$^-$ and 2e$^-$, with a geometric electrode area of 0.196 cm^2. Plot was obtained of 1/*i* vs. $\omega^{-1/2}$ at diffusional control potentials for oxygen reduction on 2FeNSeC in 0.5 M H_2SO_4 solution at 25 °C.



Although the mass-transfer-corrected Tafel slope were determine for all FeNSeC catalysts, only the 2FeNSeC sample plot is shown in Fig. 7 as representative material of its catalytic activity. As is depicted, in the inserted plot of in the same Fig. 7, the Tafel slope was essentially lineal into the overpotential range between -0.58 and -0.48 V corresponding to the potential values of 0.75 and 0.65 V, respectively vs. NHE. The kinetic parameters such as Tafel slope *b*, transfer coefficient *α*, and exchange current density i_0, were evaluated considering the same potential ranges for all the samples. These kinetic parameters from the Tafel slope are summarized in Table 2. When comparing with the deducted values of the Tafel slopes and the transfer coefficient parameters for all samples, it can be observed that similar values were achieved in each parameter column. This indicates that the synthesis reported here can be reproducible at least, with 1,2,4 and 7FeNSeC samples. In contrast, different values were observed with the exchange current density parameter. Moreover, those values are into the range of similar catalytic systems, Zhang et al. (2009).

Fig. 7. Corrected mass-transfer Tafel plot for the ORR on 2FeNSeC in oxygen-saturated 0.5 M H_2SO_4 at 25 °C is shown. In the inset curve can be appreciated the selected overpotential region where the Tafel slope was evaluated.



As can be appreciated, the data of the Tafel slope reported in Table 2 are close to 120 mV. This can indicated that at low overpotentials values, the oxygen reduction reaction is controlled by the first electron transfer to the adsorbed oxygen, Parthasarathy et al. (1991).

Table 2. Kinetic parameters deduced from the mass transfer-corrected Tafel plots of the ORR in 0.5 M H2SO4 at 25 °C.

Material	Tafel slops (-*b*) mV/dec	transfer coefficient (α)	exchange current density (j_0) mA/cm^2 ×10^{-6}
1FeNSeC	104.7	0.56	0.78
2FeNSeC	107.1	0.55	1.74
3FeNSeC	129.4	0.45	3.05
4FeNSeC	111.5	0.53	0.88
5FeNSeC	135.4^a	0.43^a	13.68^a
7FeNSeC	115.5	0.51	5.86
	113.6±9.7	0.52±0.04	2.46±2.1

^{a} value not considered for standard deviation.

4 CONCLUSIONS

The morphological characterization and EDX analysis did not provide a clear idea of where could be localized the active catalytic sites. Nevertheless, it is interesting to consider that the FeNSeC catalyst was not supported on any type of coal; for this reason, the active sites are being formed exclusively from the precursor materials during the heat treatment.

In general, the results indicated that all samples showed a reproducible catalytic activity for oxygen reduction reaction (ORR), which is similar to macrocycles reported in literature. Besides, those electrocatalyst can be obtained from low-cost raw materials and conventional synthesis procedure. Therefore, this work provided a promising topic of interest for further investigations involving materials with iron, selenium and nitrogen in such structures.

Finally, kinetic activity analysis demonstrated a lack of tendency among the results of composition of the materials and the values of the Tafel slope. This suggests that more studies need to be developed in order to understand better the relationship between the composition of the material and its catalytic activity.

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