

Methanol dehydrogenation and oxidation on Pt_{1-x}Ni_x/CNTs at low temperature: Effect of Ni addition



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ABSTRACT

This study reports the effect on catalytic activity resulting from Ni incorporation in Pt nanoparticles supported on carbon nanotubes (CNTs) for electrochemical methanol oxidation at low temperature in acidic conditions. Chemical composition, morphology and structure of the Pt_{1-x}Ni_x/CNTs (X = 0, 0.1, 0.2, 0.3, 0.4, 0.5) catalysts were studied by EDS, SEM, XRD, TEM and TGA. The catalytic activity of the prepared materials in methanol electro-oxidation reaction was investigated by cyclic voltammetry (CV) and chronoamperometry (CA). The results of catalytic activity of the nanostructured materials showed a volcano-type relationship between the Ni relative concentration current density. The enhancement of catalytic activity was attributed to changes in surface electronic structure of Pt nanoparticles that impacted in an increment of active sites for methanol dehydrogenation and oxidation processes. On the other hand, high concentration of Ni (concentration ≥ 30 at.%) in Pt nanoparticles caused a substantial decrease of the catalytic activity due to a depletion of active sites for the methanol dehydrogenation process. The highest catalytic activity was observed when the Ni relative concentration reaches 30 at.%.

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1. Introduction

Direct methanol fuel cells (DMFCs) have been used as attractive devices to convert chemical energy in electrical energy [1]. However, various drawbacks have limited their performance, such as poor kinetics of Pt/C catalysts [2,3], low abundance of Pt, and methanol cross-over from anode to cathode side through the membrane [4]. Particularly, the poor kinetics of the anodic reaction is one of the main issues for making DMFCs attractive [5]. Improving the anodic activity through the design of new catalytic materials combining both high activity in methanol dehydrogenation and more tolerance toward CO poisoning is a necessity [6]. However, the presence of Pt in the DMFCs anode cannot be discarded because the electro-oxidation process from methanol to CO₂ occurs through an initial dehydrogenation reaction step and Pt

is an excellent material for performing this essential step [7]. Therefore, research efforts have been devoted to the synthesis of catalysts combining ultra-low Pt loading with other noble metals (Ru, Rh, Pd, etc.), or to the formation of Pt-based bimetallic nanoparticles in form of alloys to increase the catalytic activity [8–10]. Also, to reduce the cost of Pt catalysts and to improve their activity, combination with other transition metals such as Fe, Co and Ni have been made demonstrating that their incorporation in Pt nanoparticles generally enhance the rate of electro-oxidation reactions [9–14].

In this respect, Ni addition to Pt nanoparticles has shown an enhancement of catalytic properties for the methanol electro-oxidation reaction [14,15], where the catalytic activity depends on different parameters such as the atomic ratio between the two metals, changes in crystalline structure and size of Pt-based nanoparticles [9,16]. These parameter modifications can be achieved using different synthesis strategies such as galvanic replacement [15], chemical reduction [17], polyol [18], colloidal [19] approaches, or Brust–Schiffrin method [20]. In particular, the Brust–Schiffrin method allows to control chemical composition,

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ORIGINAL ARTICLE

Synthesis and ammonolysis of nickel and cobalt tungstates and their characterisation



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Abstract The synthesis and characterization of NiW and CoW compounds are herein reported. The NiWO₄ and CoWO₄ samples, successfully synthesised by the hydrothermal method, were treated under NH₃ to obtain the metal nitride. The SEM micrographs show that this transformation is a topotactic process. Tungsten trioxide was also treated under NH₃ at similar operating conditions, and used as a reference. High nitrogen contents after ammonolysis were calculated, however, the percentages were below the theoretical values assuming the formation of pure NiWN, CoWN and WN. The XRD pattern indicates that WON is likely formed after ammonolysis of tungsten oxide whereas phase segregation was observed on the nickel and cobalt samples. Furthermore, the reactivity of the nitride samples as function of temperature was measured under argon and the results show that most of the nitrogen is removed from the cobalt and nickel samples whereas it was partially released from the tungsten specimen.

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1. Introduction

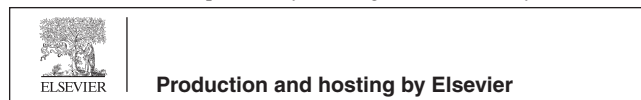
Metal nitrides are of interest due to their electronic and magnetic properties, their hardness and their application as catalysts (Oyama, 1996; Alexander and Hargreaves, 2010). In

terms of catalysis, much interest has centred upon the possibility of using certain metal nitrides as potential replacements for precious metal based catalysts, although caution should be exercised when drawing analogies with the activity of noble metals and nitrides should be recognized as interesting catalytic materials in their own right. Recently, attention has been focused upon the possibility of using certain interstitial metal nitrides as nitrogen transfer reagents wherein lattice nitrogen derived species could be directly transferred to organic target molecules potentially by-passing the requirement of ammonia as an intermediate reactant and indirectly utilizing N₂ as a reagent (Mckay et al., 2007, 2008; Hargreaves and McKay, 2009; Cairns et al., 2010). Such processes would be very attractive

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Enhanced photocatalytic activity of MWCNT/TiO₂ heterojunction photocatalysts obtained by microwave assisted synthesis

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ABSTRACT

Herein reported are the synthesis and the photocatalytic activity of MWCNT/TiO₂ heterojunction photocatalysts obtained by microwave-assisted method using titanium butoxide as precursor. The obtained materials were characterized by XRD, Raman, SEM, TEM, UV–vis, FTIR, surface area (BET) and photoluminescence. The characterization results showed that the addition of MWCNT did not provide structural changes on TiO₂. The photocatalytic activities of the synthesized materials were investigated using acid blue 9 dye as a model molecule. The results indicate an enhancement on photocatalytic activity by the addition of low amount of MWCNT (1 and 3 wt%). The improvement is attained to the synergic effect between TiO₂ and MWCNT which reduces the electron–hole pair recombination according with the photoluminescence study. In this way, a proposed schema for the enhanced photoactivity of the MWCNT/TiO₂ heterojunction photocatalyst is discussed.

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1. Introduction

Toxic, recalcitrant, and/or dyed organic compounds can be eliminated by various advanced oxidation processes. They are based on reactive oxygen species (ROS) production as hydroxyl radicals (OH•), which are effective for organic contaminants degradation [1]. The production of ROS in heterogeneous photocatalysis is based on the irradiation of semiconductors. The preferred semiconductor used in photocatalysis is titanium dioxide (TiO₂) [2–4], due to its chemical stability, superhydrophilicity, long durability, non-toxicity and low cost [5]. Unfortunately, the rapid recombination of photogenerated electron–hole pairs in the bulk semiconductor decreases the efficiency of photocatalytic reactions [6,7]. Several attempts have been made to increase the photocatalytic activity of semiconductors such as: controlling the morphology and

crystal phase; doping with transition metals or nonmetal elements; or coupling with secondary semiconductors/conductors [6,8–10]. Unfortunately, these methods involve compounds that are either thermally unstable, difficult to modify or even toxic. Another possible approach is by coupling TiO₂ with multiwall carbon nanotubes (MWCNTs), which provide a synergistic and cooperative effect that leads to enhancement of the overall photocatalytic performance [6,7,9,11]. In this regard, the improvement is attributed to the enlarged absorption region of TiO₂, the increment of surface area and the enhancement of electronic transfer; therefore, a reduction of electron in the TiO₂ bulk is achieved [12–15]. Different synthesis methods have been used to obtain the TiO₂–MWCNT materials (composites, hybrids, heterojunctions) [3,7,9,16–19]. Nevertheless, most of these methods require long preparation times (several hours or a day), involve multiple steps and have high thermal costs, which often result in structural damage in the MWCNT [14,15]. Moreover, microwave assisted synthesis is a novel technique that offers several advantages, such as simple and fast synthesis procedures, reduces the reaction time, offers faster kinetics, higher yield, uniform heating and minimal structural damage [20,21]. This method has been widely used to obtain pure TiO₂; nevertheless, there are few reports where microwave

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W and Mo doped TiO₂: Synthesis, characterization and photocatalytic activity

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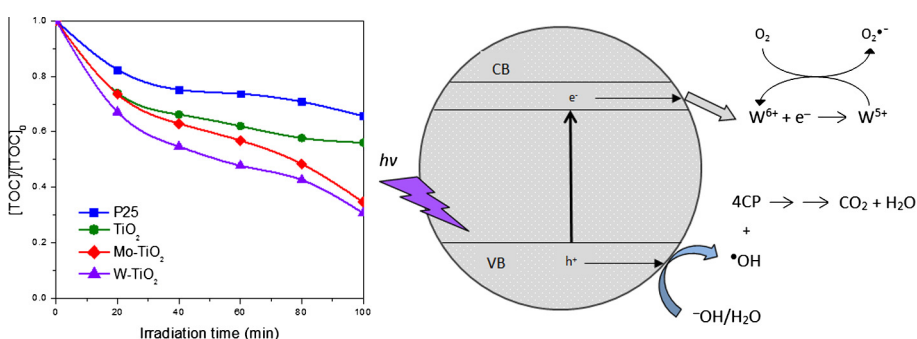
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HIGHLIGHTS

- Doping of TiO₂ with W or Mo leads to a higher photo-activity.
- Low content of W or Mo is related to the best performance of TiO₂ as photocatalyst.
- Addition of dopants eliminates the need of oxygen addition.

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ABSTRACT

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Characterization and catalytic performance of Co-Mo-W sulfide catalysts supported on SBA-15 and SBA-16 mechanically mixed

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ABSTRACT

A group of hydrodesulfurization (HDS) catalysts, based on transition metal sulfides (Co-Mo-W) and supported on mechanically-mixed mesoporous silicas (SBA-15 and SBA-16), have been synthesized and characterized by physicochemical methods (DRS-UV-vis, Micro Raman spectroscopy, XRD, SEM, HRTEM, EDS and catalytic activity). It has been demonstrated that the use of a mixture of silicas with two different porous structures has an advantage with respect to the use of the SBA-15 and SBA-16 separately for the preparation of supported catalysts for hydrodesulfurization reactions of refractory sulfur compounds, such as dibenzothiophene. This is because the presence of different porous structures has a positive effect over the diffusion processes of the precursors of the active phases on the support, and the whole result is a higher catalytic activity for the HDS reactions of dibenzothiophene, even more than that of the commercial catalyst used as a comparative model; this activity is also related with the stage of the synthesis process in which the mixture is done.

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1. Introduction

Currently, oil and its derivatives, natural gas and coal are the main sources of primary energy worldwide and this tendency will not change in the next two decades according to recent studies in energy topics.

Currently, oil and its derivatives are, along with natural gas and coal, the main sources of primary energy worldwide and, according to recent studies, this tendency will continue for the next two decades [1]. It involves a great quantity of challenges related to the separation of the fractions in the oil, specifically those associated with the removal of undesired elements such as nitrogen, oxygen, sulfur and metals, because of the environmental effects of those compounds. Hydrodesulfurization (HDS) focuses in the removal of sulfur-containing molecules in oil, typically by the use of catalytic materials. In the recent years, the research in this topic has focused its efforts in the development of new catalysts, which has to be effective in the removal of molecules of complex compounds of

sulfur such as thiophenes and benzothiophenes which are difficult to remove with conventional commercial catalysts.

The most of the catalytic materials used in hydrotreatment processes are based on transition metal sulfides, typically those of molybdenum and tungsten, promoted by cobalt or nickel and supported in inert materials. Alumina is the most common support material for HDS reactions for their mechanical properties, low cost and easy regeneration; however, this material has several disadvantages, mainly related to the presence of undesired interactions between transition metal and support which has promoted the research of alternative materials such as titania and silica, which latter have demonstrated alumina equally advantageous properties in terms of physical and chemical properties. Numerous previous studies have reported the convenience of using mesoporous silicas with cubic (SBA-16) or hexagonal (SBA-15) mesostructure as a support material for active species to be used in the hydrodesulfurization reactions since they have shown outstanding performance for the removal of sulfur atoms within the molecular structure of thiophenes and benzothiophenes.

Different mesoporous silica materials have been studied as supports for hydrodesulfurization catalysts, such as MCM, HMS and SBA materials, because of their ordered porous structure and high surface area. Particularly, many studies have been published

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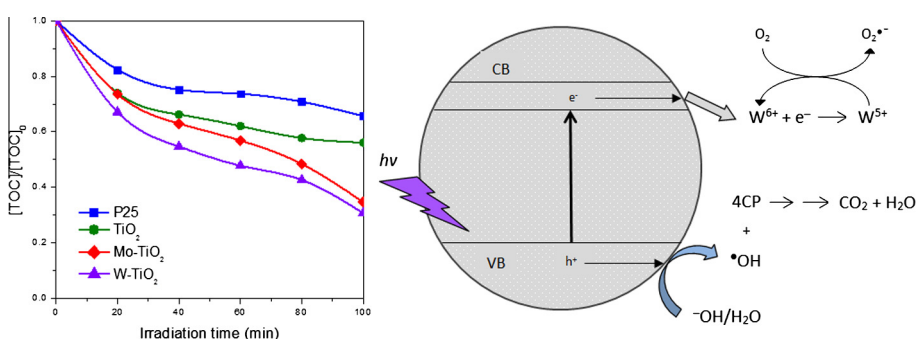
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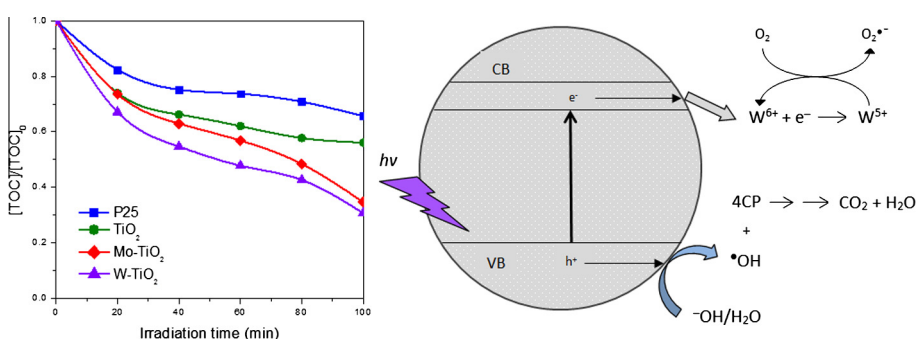
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ANÁLISIS DE CONTAMINACIÓN Y PROPUESTA DEL USO DE BIOGÁS PARA UN DESARROLLO ENERGÉTICO SUSTENTABLE EN MORELIA, MICHOCÁN, MÉXICO

José Juan Alvarado Flores¹

Jaime Espino Valencia²

RESUMEN

En este estudio, se analizó como afecta el grave problema de la contaminación actual en agua, aire y suelo debido a actividades antropogénicas de la ciudad de Morelia, Michoacán de Ocampo. Se ha realizado un análisis del estado actual de la tecnología de celdas de combustible, identificándose a la celda de combustible de óxido sólidos (SOFC), como un excelente candidato. Esto permitiría determinar la posibilidad de utilizar una celda SOFC como generador de energía eléctrica a partir de biogás compuesto principalmente de metano y bióxido de carbono como combustible.

PALABRAS CLAVE: Desarrollo energético sustentable, Biogás, Celda de combustible

1. INTRODUCCIÓN

La contaminación del aire, agua y suelo contribuye a la generación de enfermedades cardiovasculares y cáncer de pulmón (Künzli et al 2000, Pope III, 2000, Šrám et al 2005 y Schwela, 2000). Hasta 2007, el relleno sanitario de la ciudad de Morelia se mantenía como un tiradero a cielo abierto, a partir de diciembre de ese año entró en operación el Relleno Sanitario de Morelia, en el cual se contienen hasta el momento cerca de once millones de toneladas, sólo en una primera celda de doce que se planean realizar en el lugar. El relleno sanitario de Morelia es uno de los cinco que a nivel nacional cuentan con la certificación Internacional ISO 14001-

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[SCD-120] Celda de combustible como sistema de energía renovable y sustentable

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Las celdas de combustible convierten permanentemente sustancias químicas en electricidad a través de una serie de reacciones electroquímicas. Son similares a las baterías de los automóviles que producen corriente eléctrica. El problema con las baterías, es que su tiempo de vida útil es limitado y terminan disfuncionales. La celda de combustible, es un sistema muy sencillo, típicamente está compuesto de un ánodo y un cátodo, donde se introduce principalmente hidrógeno y oxígeno respectivamente. En ellos se presentan dos reacciones químicas; oxidación del combustible (H_2) en el ánodo y reducción del oxígeno en el cátodo. Entre estos dos componentes se encuentra un tercero, el electrolito, encargado de conducir los iones de oxígeno al electrodo anódico para su reacción y producción de electrones que pasan por un circuito externo conductor. Asociado a la electricidad producida, también se genera agua 100% pura y calor, que se producen como resultado de las reacciones químicas en el ánodo y cátodo al utilizar hidrógeno. Las celdas de combustible tienen una eficiencia de hasta el 60% y, con algunas modificaciones tecnológicas al utilizar sistemas de co-generación, pueden alcanzar hasta un 80%. En comparación, las turbinas tienen una eficiencia de entre el 40 y el 50%, y todavía es más baja en los motores de combustión interna de entre el 10 y el 25%.

El uso de celdas de combustible reducirá la dependencia del petróleo en el mundo. La contaminación, que principalmente esta compuesta de CO_2 y produce el efecto invernadero, se vería reducida drásticamente, debido a que el combustible utilizado (hidrógeno), no generaría emisiones contaminantes, y se obtendría muy probablemente de gases como butano, propano y metano. Cabe resaltar el uso del metano, que, de entre otras fuentes, puede obtenerse de la descomposición orgánica de los rellenos sanitarios, pirólisis de biomasa o residuos agrícolas, y utilizarse de manera directa en una celda de combustible. Actualmente, en el mercado existen diversos tipos de celdas de combustibles, sin embargo, las celdas de óxidos sólidos (SOFC), son los sistemas de generación eléctrica más atractivos por su alta eficiencia y durabilidad en operación. Además, son mejoradas día a día y podrán ser introducidas en muy poco tiempo para reducir el costo de la producción de energía eléctrica libre de contaminantes. Se pueden fabricar desde pequeños dispositivos de 0.2 W hasta centrales de 50 MW. Las investigaciones actuales, pretenden desarrollar componentes para este tipo de celdas que funcionen a temperaturas menores a los 1000°C.

Physicochemical Characterization of Water Hyacinth (*Eichhornia crassipes* (Mart.) Solms)

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Water hyacinth (*Eichhornia crassipes*) is an aquatic flowering plant that belongs to the Pontederiaceae family. The plant is a freshwater hydrophyte that grows in subtropical and tropical regions of the world. The objective of this study was to determine the physicochemical characterization of roots, stems, and leaves of *E. crassipes*. The pH, ash, 1% alkali solubility, extractives, lignin, holocellulose, tannins, and calorific value were determined. Our results showed that the mineral content is relatively high, whereas that for lignin and tannins is low. The pH is moderately acid, and the soluble substances easily dissolved in alkali or organic solvents. Potassium, calcium, and silicon are the major constituents present in the ash of this plant. The determined calorific value was approximately 14.4 MJ/kg.

Keywords: pH; Ash; Extractives; Lignocellulosic material; Calorific value

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INTRODUCTION

The water hyacinth (*Eichhornia crassipes* (Mart.) Solms) is an aquatic flowering plant that belongs to the Pontederiaceae family. The plant is a freshwater hydrophyte that grows in subtropical and tropical regions of the world. Sometimes the water hyacinth is considered an undesirable weed; but various studies have reported its uses, such as in the production of ethanol (Manivannan *et al.* 2012; Awasthi *et al.* 2013; Fileto-Pérez *et al.* 2013; Manivannan and Narendhirakannan 2014); as an adsorbent for heavy metals present in polluted water (Murithi *et al.* 2014); for phytoremediation (Vijetha *et al.* 2014); as a raw