



Preparation and characterization of novel nano-structured porous nickel alloy composite induced by electroless deposition and its performance in the hydrogen separation



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ABSTRACT

The current research work focuses on the synthesis of a novel nano-structured porous nickel alloy based composite material with a high catalytic activity for the hydrogen separation application. The revealed composite through this study is a metallic nickel–boron based catalyst supported by nylon fiber. The preparation procedures of this composite had started by loading the nickel alloy particles on the fabrics of the template. This was executed via a special technique of the electroless plating (ELP). This technique differs fundamentally from the ELP impregnation systems since the porosity of the obtained composite is reduced as a result of the compaction during the procedures of the metal particles deposition. Furthermore, the mechanism of depositing the metal particles within the pores of the template, as provided in this technique, is novel. The nano-sized metallic structure of the catalyst was subsequently obtained via a high temperature thermal treatment step. The produced nickel-alloy composite was then applied in the separation of hydrogen from binary mixtures composed of different ratios of hydrogen to nitrogen. A non-typical separation technique is introduced through this research study. Specifically, a combination between the concepts of the cross flow membrane separation and pressure swing adsorption (PSA) techniques is presented. The performance of the prepared composite was evaluated, in terms of the acquired hydrogen recovery. Promising results were demonstrated at the end of this investigation since a significant catalytic separation activity was detected. The structural and morphological properties of the nickel based material were characterized by both scanning electron microscopy (SEM) and X-ray diffraction (XRD).

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1. Introduction

Although catalysis accelerates reaction rates and chemical conversions, the accessibility of reactants and the removal of products from the catalytic sites are important to realize the full potential of catalysts. Supported catalyst systems are intended for these provisions. Catalysts are either deposited as a film/or skin layer on a support or they are used as pellets [1,2]. The catalysts which are prepared via these two techniques have certain drawbacks. In coated systems, catalyst adhesion can be non-uniform and weak while the accessibility of the active sites within the interior of the catalyst is hindered due to low porosity; small pore size and connectivity. Therefore, in the transport limited reactions such sys-

tems cannot provide adequate accessibility to the catalytic sites since the support materials themselves restrict transport processes [3–6].

In recent years, the so called ‘structured catalysts’ became available in order to address these problems. In such catalysts, supports are often introduced in the form of porous structures. Structured catalysts can be obtained via the coating of the supporting templates through different techniques. In these catalytic systems, the thickness of the catalyst coating can be controlled in order to obtain well defined catalytic particles in a packed bed configuration [7,8]. Nevertheless, these catalysts might suffer of some troubles such as cost of fabrication, low catalyst loading and the integrity of the catalyst coating on the structure of the support. This in turn might only provide a partial accessibility of the catalytic sites. The accessibility of the nano-sized catalytic sites is achieved most effectively by porous network structures which are ubiquitous in nature [9–11].

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Structured catalysts can provide an optimum architecture via incorporating both the nano-scale concept with the chemically functionalized features at a high surface area suitable for catalytic applications. Such catalysts can be more efficient and may robust the chemical processes if the catalysts contain well-distributed pores at different sizes. This porous structure will in turn provide an enhanced surface area and easy accessibility to active sites [12–15]. In the design of novel porous catalysts/catalyst supported systems, the first step is to obtain structures with controlled pores size and porosity. These structures can be then used as templates for the catalysts preparation. Templates may include spherical latexes or carbon nanoparticles/carbon nano-tubes, macro- and micro-emulsions [16,17], two-dimensional micro-molding and micelle [16,18]. Slightly similar structures can be also obtained by using different raw components where dissimilar mechanical properties are provided to the acquired material at the end [18–21].

Hydrogen (as a key element of energy matrix) production/or separation either from certain structures or mixed gases has been extensively studied over the last two decades. Hydrogen-energy based technology has certain attractions in combating the environmental crises; namely pollution and global warming [22–27]. A number of studies have been directed toward the manufacturing of defect free and highly active catalysts for the hydrogen production. Several techniques have been used during these studies in order to achieve this goal. Of these methods, chemical vapor deposition (CVD) and electroless plating (ELP) have been considered as the most promising. However the ELP is more preferred since it exhibits many advantages. ELP can provide thin metal and metal alloy films that have excellent adhesion properties, high deposition and easily controlled rate and does not require expensive setup [28,29].

Over the last few decades, different templates have been used as supports for the hydrogen selective catalysts/membranes such as alumina, iron oxides and zeolite. However, certain problems with regard to the hydrogen flux and chemical stability were addressed [10,12,19,30]. In order to overcome these problems, novel composite catalyst based on nickel alloy nano-particles is introduced through the presented research work. The application of this nickel-based catalyst for the hydrogen separation and purification is also reported. The Ni alloy particles were used (supported on a new template) as a catalytic adsorbent of which its structural and morphological properties were studied.

2. Experimental work

In the current research work, intensified hydrogen selective nickel–boron based catalyst supported on alumina-silicate (nylon) fiber was synthesized using the electroless plating technique. Maintaining the simplicity of catalyst fabrication and retaining of its chemical stability while producing high level of hydrogen yield are the main aims of this work.

2.1. Metal deposition process

The metal deposition setup as well as the preparation and composition of the plating solution which were used through this study are previously described in details by El Naggar and his co-workers [31–34]. In this setup, the deposition cell had housed the catalyst template (nylon fiber) in the form of a disk; 6 mm thick and 25 mm in diameter. In order to attain the metal deposition within the pores and the inner structure of the template, the temperature of the plating cell was raised to 50 ± 3 °C. The selection of this particular operating temperature was previously illustrated by El Naggar [35]. The metal solution bath was kept at room temper-

ature and was pumped into the deposition cell using a syringe driver at a rate of 2 mL/min. After pumping 50 mL of the plating solution, the deposition process was stopped and a hot water washing was executed. The direction of flow was then reversed and the same cycle was done. The cycles were repeated several times for both sides of the plating cell until sufficient amount of the metal alloy was deposited; particularly 300 mL of the metal solution was used for each side. Reversing the flow direction after pumping each 50 mL of the deposition solution was meant in order to obtain uniform deposition, hence a better structure for the produced catalyst at the end. After the completion of the metal deposition, a final hot water washing was conducted at 90 °C.

2.2. Heat treatment

After the completion of both the metal deposition and the final washing, the samples were charged into a high temperature oven. The temperature of the oven was next increased from the room temperature to a pre-determined level (600 °C) at a suitable ramping rate. The total heating time was 60 min; to reach the targeted temperature. Dwell time was then continued for another 60 min at the same set temperature.

2.3. Analytical techniques (catalyst characterization)

2.3.1. Scanning Electron Microscopy (SEM)

The utilized scanning electron microscope in the present work was an Environmental SEM model (XL30 ESEM-FEG). The ESEM system consisted of a control computer and analysis software. It offers high resolution secondary electron imaging at pressures as high as 10 Torr and sample temperature up to 1000 °C. No extra coating was needed for the tested sample since that the metallic samples are conductive and are examined in their natural state without any further modification or preparation.

2.3.2. X-ray Diffraction (XRD)

The X-ray Diffraction (XRD) was recorded via a diffractometer model PAN analytical X'Pert Pro fitted with an X'Celerator. The X'Celerator has the effect of giving a good quality pattern in a fraction of the time, compared to the traditional diffractometer.

2.3.3. BET surface area analysis

The surface analyses of the prepared nano-catalyst was measured via a nitrogen adsorption–desorption isotherms at -196 °C. The samples were pre-treated under vacuum (10^{-6} Torr) at 200 °C for a time of 2 h. Surface areas (S_{BET}) were calculated by applying the BET equation.

2.4. Catalytic hydrogen separation

The production of hydrogen from binary gas mixtures via a catalytic separation process using the synthesized nickel alloy based catalyst was carried out at atmospheric pressure in a stainless steel horizontal fixed-bed reactor. The length and diameter of the reactor were 50 and 2 cm respectively. However, the diameter of the reactor at one end (the exit side) was reduced to 0.5 cm. The reduction in the diameter of reactor was meant in order to provide flow turbulence during the departure of the remains of the feeding gas from the reactor. This subsequently could help in generating an extra gas pressure inside the reactor which might work as a driving force during the separation step. The configuration of the gas separator setup is shown schematically in Fig. 1.

All experiments were performed in the following manner. Before connecting the reactor with the heating cartridge, 2 g of the adsorptive catalyst (Ni–B based catalyst) was charged in the middle part of the tubular reactor. The temperature was then set

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