

International Journal of Engineering

Journal Homepage: www.ije.ir

NiO-Ni-Al₂O₃(γ) Nanocatalyst by Pulse Electrocodeposition Over Ni Open-cell Foam for Methane Reforming

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PAPER INFO

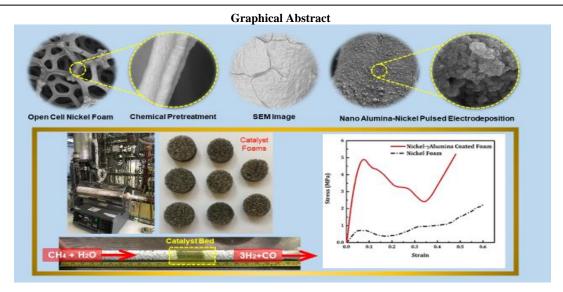
Paper history: Received 16 June 2023 Received in revised form 24 July 2023 Accepted 26 July 2023

Keywords: Nickel Foam y-alumina Methane Reforming Nanocatalyst Pulse Electrocodeposition

ABSTRACT

Global warming persuades researchers to improve the effectiveness of renewable energy technologies, such as H_2 production by methane steam reforming (MSR) an endothermic process. Herein, a nanocatalyst based on open-cell nickel foam 40 (pore per inch) with high thermal conductivity was prepared. The nanocatalyst was synthesized with a chemical stepwise synthesis approach, chemical pretreatment, pulsed electrocodeposition of Ni-Al₂O₃(γ) nanoparticles, and calcination. Measurements of thermal diffusivity(α) with flash xenon technique gained 4.41×10^{-6} m²s⁻¹ and values of specific heat capacity, Cp, by differential scanning calorimetry (DSC) and thermal conductivity(λ) enhanced by 65% in temperature range of 150 to 550°C in Ni-alumina(γ) foam nanocatalyst. Furthermore, characterization and tests for comparing nickel foam and Ni-alumina(γ) foam indicated that the hardness improved from 145 Vickers hardness (HV) to 547 HV and compression strength increased from 1.1 MPa to 5MPa and specific surface area (S_{BET}) from 1.48 m²g⁻¹ to 48 m²g⁻¹. XRD (x-ray diffraction) analysis showed NiO and NiAl₂O₄ in the structure. The interface between the catalytic component (NiAl₂O₄), and nickel affected the catalytic ability for MSR, and the efficiency gained at low tempreture 500 °C was the same as reported at 720°C by other investigations.

doi: 10.5829/ije.2023.36.10a.15



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Please cite this article as: M. Zafardoagoo, S. K. Sadrnezhaad, J. Mostaghimi, NiO-Ni-Al₂O₃(γ) Nanocatalyst by Pulse Electrocodeposition Over Ni Open-cell Foam for Methane Reforming, *International Journal of Engineering, Transactions A: Basics*, Vol. 36, No. 10, (2023), 1908-1918

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Cp	Specific thermal capacity(Jg ⁻¹ °C- ¹)	Greek Symbol	ls
HV	Vickers hardness	ρ	Density (kg/m³)
MPa	Mega Pascal	α	Thermal diffusivity(m ² S ⁻¹)
MSR	Methane Steam Reforming	λ	Thermal conductivity (Wm ⁻¹ °C ⁻¹)
PPI	Pore per Inch	heta	Duty cycle (%)
_f	Frequency (Hertz)		

1. INTRODUCTION

Environmental concerns of fossil fuels have motivated scientists to develop sustainable energies like wind, solar and hydrogen. Hydrogen is a clean energy with no exhaust contaminants during combustion [1]. The availability of immense natural gas reservoirs in the globe justifies reforming natural gas methane, to produce hydrogen by steam, carbon dioxide, and oxygen. The endothermic methane steam reforming, MSR, reaction Equation (1) required extensive energy at a temperature range of 700-720 °C on the catalytic bed.

$$CH_4 + H_2O \rightarrow CO + 3H_2$$
 $\Delta H_{298}^{\circ} = +206 \frac{kJ}{mol}$ (1)

Mostly water gas shift reaction (WGS) Equation (2) goes along MSR.

$$CO + H_2O \rightarrow CO_2 + H_2 \quad \Delta H_{298}^{\circ} = -41.1 \frac{kJ}{mol}$$
 (2)

Moreover, during MSR some side reactions occur at high temperatures (T > 750 °C) via Equation (3), and Boudouard reaction at low temperatures (300–450 °C) as Equation (4) [2].

$$CH_4 \rightarrow C_{(s)} + 2H_2$$
 $\Delta H_{298}^{\circ} = +75.6 \frac{kJ}{mol}$ (3)

$$2CO \rightarrow C_{(s)} + CO_2$$
 $\Delta H_{298}^{\circ} = -172 \frac{kJ}{mol}$ (4)

Usually, combining transition and/or noble metals catalysts on stabilized supports (alumina, ceria, ceria promoted alumina, zeolite, etc. in catalysts have been frequently used [3-5]. However, nickel was the most common catalyst for being cheap, active, and selective in MSR [6, 7]. Nevertheless, considering some drawbacks like Ni-sintering and carbon deposition maybe by hotspots and poor uniform temperature profile of pelletized catalysts bed, persuade researchers to modify catalysts structure [8-12]. Therefore, any attempt to fabricate novel catalysts for lowering carbon deposition, good heat transfer, decrease energy consumption, high thermal stability, high mechanical strength and increase lifetime are valuable [13-15]. Open-cell metal foams have a special set of materials properties such as low density, high thermal conductivity, ductility, resilience to thermal shock resistance, fluid permeability, and high surface area [16, 17]. Recently, application of nickel foams in MSR have been investigated for good heat distribution which increased [18] the CH₄ conversion inside the reformers by high interface area between the

gaseous reactants and the microporous catalysts [19-21]. In fact, higher thermal conductivity limits the thermal gradient among reactants to gain higher catalytic performance in methane reforming [9, 10, 15, 22-24]. Moreover, metal foams are attractive for compression loading applications such as in catalysts bed for their progressive crushing whilst tensile loading caused a fast, brittle fracture [16]. In the compression stress-strain curve of metallic foams three deformation stages seen as 1. Linear short elastic, 2. Long plastic plateau and 3. Densification [25-30]. Thus, this study place a focus on developing nickel foam structured catalyst reinforced by alumina (y) nanoparticles to increase the plastic plateau region for mechanical strengthening and prevent catalyst brittle crushing [23, 31].

Furthermore, recently it is studied to find that catalytic performance of the Ni foam based catalyst is highly dependent on the pre-treatment and reaction temperature [18, 32, 33]. Pegios et al. [34] reported a Nifoam catalyst aluminium oxide precursors by dip coating method over Ni-foams with MgO and SiO₂ as promoters for methane reforming. Results indicated that chemical pretretment conditions increase the active metal surface and active sites probably at the nickel-alumina interface.

Chai et al.[33] indicated among Ni/MO_x(M=Al, Zr or Y) binary catalysts, Ni foam/ Al₂O₃ possesses the largest specific surface area and the highest amount of NiO species (Ni active site), and as a result, exhibits the best catalytic performance. Lajevardi et al. [35] indicated that Ni-nano Al₂O₃ with applied pulse electrcodeposition had higher hardness than direct current. Applied nanoparticles locked dislocation at the grain boundaries and recrystallization temperatures. Therefore, microhardness and thermal stability increased [36-38]. In the pulse current state, less nano particles agglomerated, and caused more uniform dispersion of nano particles [39]. The metal foam structure does not in itself have a high surface area; thus, in catalyst applications, deposition of a high surface area coat e.g., Al₂O₃(γ), SiO₂, or zeolite by wash-coating, solelectrochemical deposition, electrophoretic, chemical vapor deposition (CVD) or physical vapor deposition (PVD) is necessary [9, 40, 41].

The utilization of appropriate ceramic nanoparticles with high specific surface area such as $Al_2O_3(\gamma)$ reinforcement in a porous metal matrix in catalysis would be interesting candidate for enhancing thermal conductivity, hardness and compression strength [17, 42-

441. Cimino et al. [18] applied Re/Ce via electroprecipitation and pulse deposition methods on nickel foam catalyst in methane reforming to increase heat transfer by support metal foam strucure. To date, only a limited number of studies have been examined the effect of alumina(γ) nanoparticles in Ni-foam as nanocatalyst by pulse codeposition for compression strengthening, more thermal conductivity and MSR operating at low temperature as a way of energy saving. By applying this concept, we aim at increased thermal conductivity, a lower thermal gradient through the reaction chamber for more heat transfer and better interaction between the gas-solid reactantants'active Ni phase and alumina(γ) nanoparticles. The MSR pilot test of this nanocatlyst indicated the same efficiency at 500 °C compared to MSR efficiency of common catalysts at 720 °C that reported by other works [9, 22, 23].

2. MATERIALS METHOD

2. 1. Experimental The procedure consists of several steps for nanocatalyst fabrication of nickelalumina(y) foam, briefly, such as (a) open-cell nickel foam fabrication over polyurethane (PU) as substrate, (b) chemical pre-treatment or etching, and (c) Ni-alumina(y) nanoparticles pulse electro co-deposition. Firstly, the 40 pores per inch (ppi) open cell PU with the density of 0.021 gcm⁻³ became conductive by nickel electroless deposition. We used the electroless bath containing nickel sulfate and sodium hypo-phosphide (NaPO₂H₂) at 82-85°C with a pH of 4. Afterward, the foam was nickel electrodeposited by complex nickel salt solution (including nickel(II) sulfate hexahydrate (NiSO_{4.6}H₂O), Merck 180 gL⁻¹, ammonium chloride (NH₄Cl), Merck 25 gL⁻¹ and boric acid (H₃BO₃), Merck, 30gL⁻¹) at 55 °C, pH 5.5, and direct surface current density 20 Adm⁻² for 60 min. All the chemicals used with the grade for analysis. Then foam was put into a tube furnace flowing 99.99% purity argon gas to eliminate the PU substrate by pyrolysis as per the procedure explained in another work [16]. In continue, chemical pre-treatments were applied to increase the roughness of the surface of the foam's strands in 6 Molar (M) hydrochloric acid (HCl) at three different temperatures (i.e., 60, 70, and 80 °C) for 10 and 15 minutes. Then the foam was rinsed thoroughly with deionized water, dipped into acetone and dried. Each specimen with dimensions 10×40×40 mm³ was pulse electro codeposited by two solutions to gain nickel-yalumina nanocomposite over the etched nickel foam. Analytical reagents and deionized water were used to prepare the plating solutions. Firstly, a suspension of nano Al₂O₃(γ) powder (99.99%, particle size 20-50 nm, BET surface area >150 m²g⁻¹) (5 gL⁻¹) and sodium dodecyl sulfate (SDS) (1.5 gL⁻¹) as surfactant dispersed, stirred for 21 hours with 200 rpm and agitated by

ultrasonic waves for 20 min to break the weak-bonded agglomerations. Secondly, the nickel watt bath type (including nickel(II) sulfate hexahydrate (NiSO_{4.6}H₂O), gL^{-1} , Merck. 250 Nickel(II) chloride hexahydrate (NiCl₂.6H₂O) 40 gL⁻¹, boric acid (H₃BO₃), 35 gL⁻¹) was prepared. Then the nano-suspension was added to it and well stirred. Afterwards, pulsed electro co-deposition was performed with frequency 5 Hz, duty cycle 50%, current density 27.5 Adm⁻², stirring rate 200 rpm, the electrolyte pH 4.2, temperature 50±2°C exposed time 90 minutes for nickel foam as the cathode. Finally, the pulse electrodeposited nickel-nano yalumina foam was rinsed with deionized water, dried, and calcined at 720 °C.

2. 2. Characterization and Tests We performed the thermogravimetric analysis (TG, TA Instrument BAHR STA 503) to determine the thermal stability of the PU foam before plating. In this study, X-ray diffraction (XRD) patterns for identifying crystalline phases of the specimens were collected with a PANalytical X-ray diffractometer using Cu K_{α} radiation (λ =0.15406 nm, 40 kV, 40 mA). Scanning electron microscopy (SEM) and field emission electron microscopy (FESEM) were used to investigate the specimen morphology and elemental analysis. The compression strength test of the specimens with dimensions 10×20×20 mm³ was conducted at room temperature by HOUNSFIELD (H10KS-UK) test machine. The surface hardness (on the Vickers scale) of nanocomposite coatings was measured by a microhardness measurement device that applied a 50 g load for 15 seconds. The nickel foam specimens were etched for 15 s with a mixture of 3 mL HF and 80 mL HNO₃, and the thickness of struts was determined by an optical microscope. Specific heat capacity Cp was measured by the Mettler Toledo thermal analysis with N₂ gas flow and thermal diffusivity a by Xenon Flash model XFA 500 was quantified. The H₂-TPR (temperature-programmed reduction) experiments were carried out in the NanoSord instrument (Sens Iran Co.), under a mixture of 5% H₂-95% argon gas with 10 sccm flow rate to determine the reducibility of nickel-alumina catalyst. Ni-alumina(γ) foam specimen was heated with a rate of 10 ° Cmin⁻¹ up to 950 ° C.

2. 3. Experimental Set-Up for Catalytic Reaction The performance of the foam catalysts was studied in a pilot system under atmospheric pressure, as shown in Figure 1. A quartz tube reactor with a 28 mm inner diameter was loaded with a 7.64 g synthesized foam catalyst, 50 mL, and the remained volume was filled with inactive alumina(α). First, the Ni-alumina(γ) foam was reduced in situ before injecting methane/steam mixture by pure hydrogen at 430 °C for 120 min with a 5 °Cmin⁻¹ heating rate. The methane-water mixture (Steam/CH₄; v/v:3) was injected into the quartz tube through a dosing

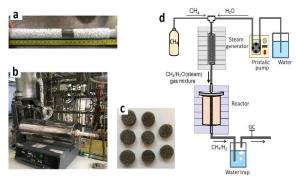


Figure 1. (a) Quartz tube filled with Ni-alumina foam catalysts, (b) catalysts of test pilot, (a) Ni-alumina (γ) foams, and (d) schematic picture for catalyst test setup

pump (LATEK- P700), which controlled the feed rate with gas hourly space velocity (GHSV) of 2000 h⁻¹. The tube reactor temperature was set at 500 °C. The product gasstream passed through a condenser at the outlet of the reactor, and the unreacted water was stored in a tank. The dry gas outlet (H₂, CH₄) was analyzed by the online gas chromatography (GC) (model Agilent Technologies 6890N) equipped with a thermal conductive detector (TCD) and flame ionization detector (FID). The catalytic performances of the Ni-alumina(γ) coated layer on nickel foam prepared in situ were measured.

3. RESULTS AND DISCUSSION

Thermogravimetric analysis was performed to determine the thermal stability of polyurethane foam by using a TA Instrument BAHR STA 503. Samples of 0.22g were heated up to 650 °C at a heating rate of 10 °Cmin⁻¹ in a nitrogen atmosphere. As shown in graph Figure 2, a very slight rate change seen at 100-120 °C may be for water evaporation while PU foam was decomposing with a rapid rate at 290-400 °C that was associated with the polyol main chain and urethane bond breaking as reported by other researches [45, 46]. As mentioned pyrolysis was done to remove PU substrate with the least probable crack formation by a controlled procedure [16]. Moreover, the alumina must penetrate uniformly inside the tortuous nickel foam without blocking the foam pores. In continue chemical pre-treatment by 6 M HCl was applied to the pure nickel foam to increment the adhesion of the strands' surface for pulsed electrodeposition of Ni-yalumina [34]. As per SEM images, in Figure 3(a), pure nickel foam showed sound struts with no cracks. Moreover, in Figure 3(b), (c) and (d) grain boundaries of nickel foams pre-treated in HCl at 70 and 80 °C for 10 and 15 minutes were obviously seen. Here, the foam at 70 °C in HCl was observed more stable than that of the specimen heated at 80 °C, however, etching the foam at severe conditions, heated at 80 °C for 15 min, caused the foam losing integrity. Therefore, the optimized condition for chemical pretreatment by 6 M HCl at 80 $^{\circ}$ C for 10 min by firm struts in foam structure.

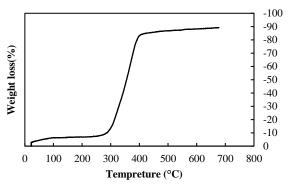


Figure 2. Thermal stability analysis of PU foam

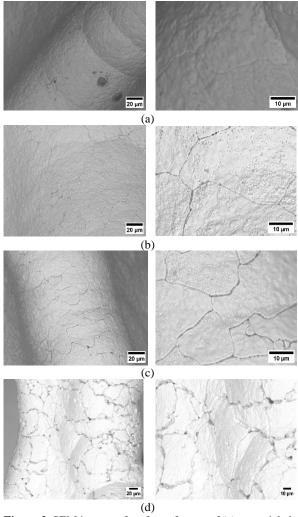


Figure 3. SEM images of surface of struts of (a) pure nickel foam, (b) pretreated nickel foam at 70 °C for 10 min, (c) pretreated nickel foam at 80 °C for 10 min, and (d) pretreated nickel foam at 80 °C for 15 min

According to XRD patterns illustrated in Figure 4, three primary reflections at 20 of 44.64°, 51.92°, and 76.46° designated to the (111), (200), and (220) planes of the face-centered-cubic (FCC) nickel, confirmed that Ni was the dominant constituent of the obtained foam (JCPDS no. 04-0850). Meanwhile, there were small peaks of Ni-P in the XRD pattern, due to the sodium hypophosphite reducing agent in the plating solution. However, by reducing the concentration of the reducing agents and plating solution with a higher pH, the amount of phosphorus and sodium to form the Ni-P layer can be minimized [16]. For nickel foam coated by Ni-γ-alumina in this study displayed spinel NiAl₂O₄ at 2θ =31.4°, 45°, 68.9° , 80° (JCPDS no. 10-0339). However, some other detected peaks 2θ = 25.1°, 37.7°, 57.5°, 68°, and 70° corresponding to deposited alumina (JCPDS no. 46-1212) and rhombohedral phase NiO at 2θ = 43°, 62.8° (JCPDS no. 22-118) was observed and via the following reaction and calcination at temperatures above 700 °C, the NiAl₂O₄ formed as Equation (5). Calcination causes formation of NiAl₂O₄ spinel phase, especially in the case of Al_2O_3 (y) presence [47].

$$NiO + Al_2O_3 = NiAl_2O_4 \tag{5}$$

Here, surface area of Ni-alumina(γ) foam measured by BET method and reported in Table 1 for comparison. Figure 5 indicated cross-sectional micrographs of nickel- γ -alumina nanocomposite foams. In fact, the wall thickness of 2-5 μ m gained after electroless then increased to 50-80 μ m by electrodeposition. In continue, by pulse electro- codeposition coating of nickel- γ -alumina nanocomposite over the former foam gave a three-dimensional structure with 230 μ m thick and hollow struts. The white sections indicated foam cell struts and the grey section for the empty space between

TABLE 1. Specific surface area (BET) of investigated nickel foam catalysts

Sample	S.S.A (m ² g ⁻¹)	PPI
Nickel foam	1.48	45
Ni- alumina (γ) foam	48	45
Ru/Ce/Ni foam (Ni foam via CeO ₂ electroprecipitation and impregnation with Ru [18]	39	75
AlSB/Ni foam aluminium tri-sec butylate (AlSB) [34]	5.6	-
AlIp/Ni foam ,aluminium isopropoxide (AlIP) [34]	6.9	-
Pre-treated Ni foam in catalytic methane decomposition (CMD) [32]	5.4	110
Pre-treated Ni foam with grown carbon nanofibers in catalytic methane decomposition(CMD) [32]	8.7	110

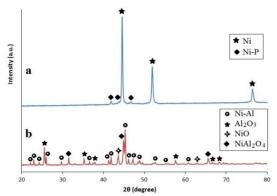


Figure 1. XRD patterns for (a) pure nickel foam and (b) nickel foam coated with nickel- γ -alumina

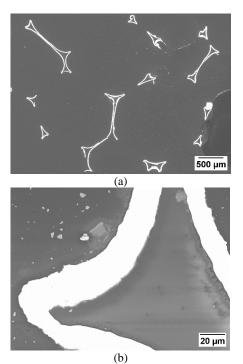
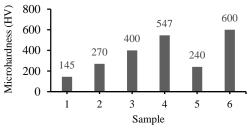


Figure 2. Optic microscopic images of electrodeposited Nialumina (γ) nanocomposite open cell foam specimen mounted in epoxy resin and polished, a) x40, b) x1k

the cells and the separate islands seen because of three-dimensional porous skeleton of foam appear in the surface by polishing. In the current work, using pulse codeposition of Ni/alumina(γ) reinforced nanoparticles on nickel foam increased hardness from 145 to 547 (HV). The average of five different measurements determined the final hardness shown in Figure 6.

The compressive stress-strain curves of nickel foams and Ni-alumina(γ) coated foam indicated in Figure 7. In these curves, three distinct regions seen as: (i) the linear elastic deformation (cell edges yield in bending area), (ii) collapse plateau (sudden decrease in stress after the first



- Nickel foam (electrodeposited Ni foam) 1 2
 - Ni coating over steel substrate [37]
- Nickel foam (electrolyt jet electrodeposited Ni 3
- 4 Ni-nano yalumina codeposition over Ni foam
- Open cell Ni-35Cr-22Fe foam(gas-phase 5 codeposition of Cr and Fe over Ni foam) [48]
- Ni-8.5% nano Al₂O₃ coating over steel substrate 6

Figure 6. Results of the micro-hardness tests conducted at room temperature

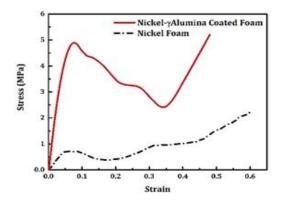


Figure 7. Compressive stress-strain curves of nickel foams and Ni-alumina(y) coated foam

peak of stress and plateau area), and (iii) densification region (slight increase in strain, rapid growth of stress, and more significant line slope). The mechanism of linear elasticity depends on whether the cells are open or closed. At low relative densities, open-cell foams deform primarily by cell-wall bending [49, 50]. Therefore, in this study we tried to increase the strength by struts thickness growth from 50 µm to 230 µm. As shown in Figure 7, nickel foam does not oscillate from beginning to end in stage II with good compressive stability; in contrast, in the Ni-alumina(γ) nanocomposite, noticeable oscillation and a sudden decrease in stress were seen in stage II. Compressive strength (stress peak after linear elastic phase) of foams for nickel foam obtained 1.1 MPa. However, compression strength of Ni-alumina(y) nanocomposite foam increased to 5MPa by adding reinforcement alumina nanoparticles. Ni-Al₂O₃(y) foam compression strength increased drastically by pulse

electro codeposition of the Ni-alumina and its elastic modulus improved. Reasons for increasing the strength and elastic modulus of nickel foam with the electrochemical coating are alumina nano particles. Higher compression strength could mean high mechanical stability of bottom-bed catalysts against upper-bed catalysts in the reformer where a suitable gas permeability is required. Thermal conductivity of Ni-Al₂O₃(γ) foam with density (ρ) of 0.20 gcm⁻³ calculated by measurements of thermal diffusivity (α) and specific heat capacity Cp. Thermal diffusivity (α) gained 4.41×10⁻ ⁶ m²s⁻¹ with flash xenon technique and values of specific heat capacity, Cp, measured by differential scanning calorimetry (DSC). Moreover, the changes of α by temperature rise was negligible.

The thermal conductivity calculated by Equation (6) [51, 52].

$$\lambda = \alpha \times \rho \times C_{p} \tag{6}$$

As stated in Table 2 by temperature rise, both Cp and thermal conductivity (\(\lambda\)) of nanocomposite Nialumina(y) foam increased as well. Besides, the distribution of alumina coat over nickel foam and all of the other elements dispersed uniformly as seen in Figure 8. Pure nickel crystalline coatings have a truncated pyramidal structure, i.e., a typical morphology for pure nickel electrodeposits with (111) preferred texture [53, 54]. The modifications in the surface morphologies are attributed to the change from a preferred orientation to a random-oriented electrodeposit, and the addition of Al₂O₃ particulates deteriorates the pyramidal structure [54]. Considering FESEM images in Figure 9, a large number of irregularly indicated spherical nanoparticles with sizes 45 to 89 nm of Al₂O₃ and NiO.Al₂O₃. In fact, the layer of nanocomposite coating was less uniform and rougher with cauliflower morphology and the surface morphology could be attributed to the change from a preferred orientation to a random-oriented electrodeposition, and the embedded alumina nano particles in nickel matrix deteriorates the pyramidal structure [54, 55]. As known the reduced metal present on the surface of the catalyst was considered active in the partial oxidation of methane. The temperatureprogrammed reduction (TPR) can suggest the number of species and a relative classification of energy bonds between the reducing element and its environment. Nialumina foam specimen with a rate of 10 °C.min⁻¹ heated up to 950 ° C. The result, the H₂-TPR graph in Figure 10, exhibited two reduction peaks a sharp one at 400 °C and a broader peak at 700-720 °C. Ni was reduced at a relatively low temperature (lower than 450 °C). The sharp peak at 400 °C was related to nickel oxide reduction having weak interaction with alumina support and this reduction leads to the deposition of nickel metal on γ-Al₂O₃. In contrast, the second broader peak at 700-720 °C

relates to the reduction of the spinel NiAl₂O₄ occurred at high temperature in a large range of temperatures [56, 57].

As depicted in Figure 11, the conversion of methane to products was calculated via the difference between

TABLE 1. Specific heat capacity (Cp) measurement by temperature

T (°C)	Cp (Jg ⁻¹ ∘C-¹)	Thermal Conductivity (Wm ⁻¹ °C ⁻¹)
150	0.92	0.81
250	1.00	0.88
300	1.08	0.95
350	1.17	1.03
400	1.21	1.06
450	1.31	1.15
500	1.43	1.26
550	1.53	1.35

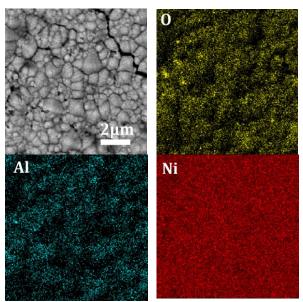


Figure 3. Map of the uniform dispersion of alumina on the Ni foam

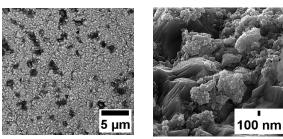


Figure 9. FESEM images of synthesized nickel foam with pulsed deposition of Ni-Al₂O₃ (γ) at f=5 Hz, θ =50%, time=60 min

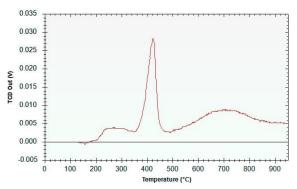


Figure 4. H2-TPR graph of calcined nickel-alumina foam

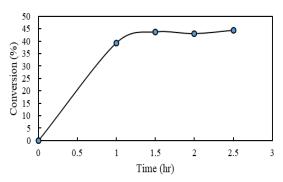


Figure 5. Methane conversion(%)-time of MSR

inlet and outlet flowrates (F_{CH4-in} , $F_{CH4-out}$) measured by online GC. After about two hours, the conversion of CH₄ reached 44% at 500 °C. The conversion of MSR reaction calculated by Equation (5). However, with the Boudouard reaction as Equation (4) occurrence at low temperatures (300–450 °C) carbon deposited on catalyst's surface during MSR and caused it to deactivate some parts of the nickel foam catalyst and declined its efficiency.

For the selectivity, the below equation was used. The selectivity toward the hydrogen production calculated by Equation (6) that F_{H2.out} was outlet H₂ flowrate in online GC, then, the selectivity H₂ gained 2.4 upon reaching steady state phase as shown in Figure 12. Figure 13 depicted compositions of methane and hydrogen in the outlet at different time intervals for nickel foam coated with nickel-alumina(γ). Hydrogen synchronized with methane conversion and did not change significantly after 60 min. Methane exhibited a slight decreasing behavior with time. After 90 min stream time at 500 °C, H₂ and CH₄ reached 64.2% and 18%, respectively. The flowrate of hydrogen in the outlet did not match with stoichiometry of the reaction and there is an approximately 10 – 20 % error. This difference could be attributed to the purge of hydrogen at the beginning of the process and reduction of free NiO at lower temperature gave small metallic nickel particles.

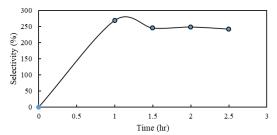


Figure 6. H₂ selectivity-time produced in MSR

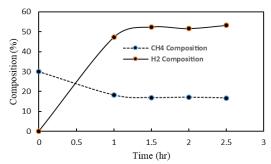


Figure 7. Composition of hydrogen and methane in the outlet of a MSR reactor

The free nickel foam without deposited alumina (metal oxide) showed no catalytic activity because the interface between nickel and alumina assumed as active sites necessary for catalytic reactions and the nickelalumina interface by the presence of NiAl₂O₄ nanoparticles spinel improved catalytic properties. In nickel-valumina foam catalysts, Al₂O₃(y)nanoparticles with high specific surface area over metal foam with high thermal conductivity improve heat transfer in MSR reformers. Then, at lower temperatures, the required heat flux provided with the appropriate efficiency in MSR [9, 22]. Furthermore, the use of metal foam increases CH4 conversion due to improved heat distribution inside the reformer with better contact between the gas mixture and the open-cell foam catalyst.

Conversion
$$CH_4(\%) = \frac{(F_{CH_4in} - F_{CH_4out})}{F_{CH_4in}} \times 100$$
 (7)

$$SH_2 = \frac{(F_{H \, 2out})}{F_{CH_4, out} - F_{CH \, 4.in}} \times 0.5 \tag{8}$$

To produce hydrogen, H₂O reacts with nickel surface atoms to absorb oxygen and produce gaseous hydrogen. Methane was also adsorbed on nickel surface atoms. The adsorbed methane either reacts with the adsorbed oxygen or decomposes to form chemical radicals [58].

Considering the catalytic test in this study, it resulted that the favorable temperature for the MSR decreased to 500 °C compared to the common MSR reaction

temperature at 700 °C to 720 °C. It could be concluded that here open-cell nickel foam coated with Ni-alumina utilization as nanocatalyst caused better heat transfer through reaction chambers and furthermore somehow the purged H_2 into the reactor for the catalysts reduction, the unreacted hydrogen can be burned to supply heat.

4. CONCLUSIONS

In this work, coating Ni-Al₂O₃(γ) nanocatalyst by pulse electrocodeposition over nickel foam is a way to enhance the thermal properties of it for MSR. Results indicated that the catalysts prepared by the nickel foam yielded higher thermal conductivity and compression strength compared to the nickel based pellet catalyst. By measuring thermal diffusivity(α) gained 4.41×10-6 m²s⁻¹ and values of specific heat capacity, Cp, and thermal conductivity (λ) increased by 65% with temperature increment between 150 to 550°C in Ni-alumina(γ) foam nanocatalyst. XRD analysis indicated NiO, NiAl₂O₄ in the synthesized nickelalumina(γ) foam. The nickel-alumina(γ) nanocomposite plus NiAl₂O₄ and alumina(γ) nanoparticles increased the hardness to 547 (HV) and the compressive strength of the fabricated nanocatalyst from 1.1 MPa to 5 MPa with a progressive crushing instead of a brittle failure. In addition, S_{BET} of the pure nickel foam was 1.48 m²g⁻¹, which increased to 48 $m^2g^{\text{--}1}$ by adding alumina($\!\gamma\!$) nano particles. The Ni-alumina(γ) nanocomposite had active sites at the nickel-alumina interface and the deposited NiAl₂O₄ spinel, which increased the catalytic activity of the product. The reason for the low-temperature MSR in this study could be stated as the metallic nickel foam with high thermal conductivity made it a potential attraction for the novel design in this study by decreasing the favorable temperature for the MSR to 500 °C due to highly heat transfer compared to the traditional steam reforming reaction temperature at 700 °C.

Despite the above advances, further improvement should be focused on effective parameters such as micro and mesopores supports and utilization of different promoters like rare earth elements such as lanthanum and cerium or their oxides in synthesizing new alloyed foam nanocatalysts for hydrogen production. However, few investigations have been done to the challenges of Ni-Alumina foam nanocatalysts regeneration, as well as the kinetics study of the catalytically active sites in methane reforming reactions.

5. ACKNOWLEDGEMENT

The authors are grateful Sharif University of technology, University of Toronto for general support and Research Institute of Petroleum Industry (RIPI) of assistance catalytic pilot tests.

6. DATA AVAILABILITY

The raw/processed data required to reproduce these findings cannot be shared at this time due to technical or time limitations.

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Persian Abstract

چکیده

گرمایش بی رویه کره زمین محققین را برای ارتقاء تکنولوژی تولید انرژیهای پایدار اعم از تولید هیدروژن توسط ریفرمینگ متان-بخارآب، فرایندی اندوتررم واداشته است. در این تحقیق نانوکاتالیستی بر پایه فوم سلول باز نیکل ۶۰ (حفره در اینچ) با هدایت حرارتی بالا ساخته شد. این نانوکاتالیست با یک فرایند شیمیایی چندمرحله ای، عملیات پیش آماده سازی، تجزیه حرارتی، رسوب همنشینی الکتروشیمیایی پالسی نیکل -نانوذرات آلومینا (گاما) و کلسینه سنتز شد. ضریب دیفیوزن حرارتی (۵) با تکنیک فلش زنون ا ۶۰ مترمربع بر ثانیه و ظرفیت حرارتی، رسوب همنشینی الکتروشیمیایی پالسی نیکل –نانوذرات آلومینا (DSC) اندازه گیری شدند و محاسبه هدایت حرارتی (۸) نشان می دهد این خواص با افزایش سختی از دما از ۱۰۵ به ۵۰۰ درجه سلسیوس حدود ۲۰٪فزونی می یابند. بعلاوه توسط روشهای شناسایی و آزمایشات مواد، با مقایسه فوم نیکل با فوم نیکل –آلومینا افزایش سختی از ۱۶۵ به ۷۵۰ و یکرز و استحکام فشاری از ۱/۱ به ۵ مگاپاسکال و سطح ویژه (SBET)از ۱/۲۸ به ۸۸ مترمربع بر گرم بدست می آید. آنالیز پراش اشعه ایکس نشاندهنده اکسید نیکل و ۱۸۵۱ در ساختار می باشد. فصل مشترک بین جزء کاتالیستی ۱۸۱۹ و نیکل روی قابلیت کاتالیتی اثرگذاشته و کارایی این نانوکاتالیست در تست پایلوت در درجه سلسیوس نسبت به نتایج گزارش شده توسط تحقیقات دیگر در ۷۲۰ درجه سلسیوس یکسان بدست آمد.