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SYNTHESIS OF MESOPOROUS NICKEL USING ELECTRODEPOSITION METHOD

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Abstract

Mesoporous material technology has recently become increasingly popular among scientists and industry professionals. Electrodeposition is a metal deposition process that employs an electric current. The most typical applications of electrodeposition are metal coating (electroplating) and the creation of porous materials. This study aimed to analyze the effects of current and nickel synthesis time on the formation of mesoporous nickel morphology using corn starch. The method for producing mesoporous nickel deposits uses an electrodeposition process using corn starch additives. The process of making mesoporous nickel varies at currents of 4, 5, and 6 A, holding times of 3, 4, and 5 hours, and additional weights of corn starch of 1, 2, and 3 g, with the weight of nickel deposits produced being 1.19, 1.3, and 1.9 g, with an increase in nickel deposits from currents of 4 to 6 A (holding time of 3 to 5 hours) of 0.71 g (59.66%). The smallest pore diameter of 112 nm was obtained using a current of 6 A, a holding time of 5 hours, with 3 g of corn starch. Characterization was carried out on the electrodeposition layer using XRD (x-ray diffraction) and SEM-EDS (scanning electron microscope-energy dispersive spectroscopy), where the results of the XRD analysis showed the presence of α*-Ni,* γ*-Fe, and* Fe_1Ni_3 *compounds, and the results of SEM-EDS showed porous Ni deposits with the highest Ni concentration above 80%. The bigger the current employed and the holding period in the electrodeposition process, the heavier the Ni deposit formed.*

Keywords: *Mesoporous, electrodeposition, nickel,* α*-Ni,* Fe_1Ni_3 *compounds*

1. INTRODUCTION

The study of porous materials has attracted the curiosity of scientists and engineers alike. Compared with solid metals, porous metals have low density, large specific surface area (large specific surface area), good energy absorption, and greater specific strength and stiffness. The open-cell porous metal has good heat exchange, radiation properties**,** sound absorption, and permeation and infiltration properties [1].

Mesoporous nickel is an excellent sound-absorbing material, especially at high frequencies. Designing a sound absorption structure can increase sound absorption performance in low frequencies. Nickel foam is also one of the best electrode materials for making cadmium-nickel batteries, hydrogen-nickel batteries, and filtration materials. In addition, nickel is widely used for high-temperature materials, so it is commonly used in industrial applications.

Development of monoatomic Ni metal electrocatalysts supported by triazine-based controllable microporous carbon nanospheres (Ni SAcs/McNPs) and the catalysis was prepared using a self-assembly strategy of aqueous lotion polymerization. The catalyst can function effectively in strong alkaline electrolyte solutions [2]-[3]. Furthermore, in the latest research in the present work, ordered mesoporous silica containing Ni atoms as active sites was synthesized by a new low-temperature

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condensation of silica precursors on a micellar template from aqueous solutions in the presence of nickel salt [4]. This method is advantageous because it avoids high temperatures or hazardous organic solvents, making it more environmentally friendly [4]. Further research on the formation of 2D mesoporous has shown additional structural functions with great potential in electrocatalysis that enhance electrocatalytic activity and stability and optimize electrocatalytic selectivity [5]. A popular new approach involves using ordered mesoporous silica as a pattern, where nickel is incorporated into the silica framework [6]. And then researchers also explored the nanocasting technique, where mesoporous nickel is made by infiltrating nickel precursors into the pattern, which are then removed, leaving a porous nickel structure. This technique allows high control over the porosity and surface area of the material [6].

Recent research explores different methods of synthesizing mesoporous nickel, such as silica templates, which allow precise control over pore size and structure. This ensures a uniform distribution of active nickel sites, improving the material's filtration and catalysis efficiency.

Porous materials can be made by mixing metal powder with a blowing agent and compacting the mixture (compaction), and one of them is EDP (electrodeposition) [6].

Electrochemical deposition, or electrodeposition, is explained for the fabrication and mass production of functional and nanostructured device materials. As basic information for understanding the concepts used to nanostructure electrodeposited materials, reviewing electrodeposition constituents, thermodynamics and kinetics of electrodeposition, electrochemical and instrumental assessment techniques, as well as other physical factors influencing electrodeposition [7]. Electrodeposition is often fabricated with wire electrolytes because the resulting pore structure is bi-continuous and has a very high surface area-to-volume ratio [8]. Electrodeposition not only uses ferrous or non-ferrous matrices, but also composites, which are renowned as an excellent approach for creating metal matrix composite layers. It contains finely scattered inert particles [9]. However, electrodeposition does not only use sulfate and chloride solutions; it also uses a thin layer of silicon (Si) to replace other Si wafer formation techniques known as silicon electrodeposition predecessors [10].

Electrodeposition (EDP) has been the main method of coating metals for various applications for nearly 100 years. Its popularity is due to its straightforward design, low cost, uniform yield, and successful application of various metals and substrates [6]. Many factors have been shown to influence the composition, texture, and chemical properties of the resulting precipitate, such as current density, nature and concentration of metal ions, solution temperature, composition, the waveform of the applied current, substrate surface, and agitation. In particular, additives play a complex role in metal deposition due to their ability to greatly alter the growth mechanism and structure of the resulting precipitate[2],[9],[11].

Meanwhile, the novelty in this study is the Ni electrodeposition process, which is given a catalyst in the form of corn starch to form Ni pores on the surface with uniform and homogeneous mesopore sizes. However, additives must be used to open the porous layer. Corn starch as an additive in the electrodeposition process is usually associated with its use as a binder or particle binder in the manufacture of electrodes or coating certain materials on the surface of the substrate. This method is generally known as porous electrodeposition or template electrodeposition. Corn starch is a mold material that will provide a porous structure to the deposited material. Therefore, this study aimed to analyze the effects of nickel synthesis current and time on the formation of mesoporous nickel morphology using corn starch additives or catalysts.

2. MATERIALS AND METHODS

The materials used for the electrodeposition process are ST 37 steel as the cathode, and 99% purity nickel plate as the anode. Electrodeposition solution materials use 32% technical HCl solution from Kimiamart products, technical NaOH crystal solids from Kimiamart products, technical H_2SO_4 solution with 94% purity from Kimiamart products, technical $NiSO_46H_2O$ powder from Bratachem products, and distilled water (*aqueous*) and the cornstarch used is a commercial product with 100% purity from the Maizenaku brand as an additive Cornstarch is primarily composed of carbohydrates, which consist of carbon, hydrogen, and oxygen. Nitrogen content in pure cornstarch is typically very low or negligible due to Carbon (C): Approximately 44-45%, Hydrogen (H): Approximately 6-7%, Nitrogen (N): Typically very low, often below 0.5% with elements are Na, K, Ca, Mg, P, S, Fe, Zn and Cu [10]. The tools used for the process of making Ni shafts in this research are an analytical balance, chemical beaker, measuring cup, measuring pipette, spatula, measuring flask, stir bar, spray bottle, dryer, and ampere meter [12].

Before the electrodeposition process, a 1 M $NiSO_4$ electrolyte solution was made, and an additive (*corn starch*) was added. A mixing process followed this until the solution was homogeneous, which was then vibrated for 30 minutes using an ultrasonic device. These additives are intended to form a medium for porous materials, and the ultrasonic process aims to prevent oligomerization (clumping) from forming.

Electrodeposition layer thickness is achieved by depositing a material (usually metal) onto an electrode using an electric current. The amount of material deposited on an electrode can be calculated using a combination of Faraday's first and second laws of electrolysis [13].

$$m = \frac{M \, x \, I \, x \, t}{n \, x \, F} \qquad (1)$$

Where: m is the mass of the substance deposited (g), M is the molar mass of the substance (g/mol), I is the current in amperes (A), t is the time in seconds (s), n is the number of electrons transferred (valency), F is Faraday's constant = 96,485 C/mol [13].

The EDP (electrodeposition procedure) process was carried out following the ultrasonic process by injecting 1 M $NiSO_4$ electrolyte solution into a beaker, followed by a succession of electrolysis operations as shown in Fig. 1 [2],[9]. The EDP experiment used process parameters, namely the concentration of electrolyte solution, cathode (workpiece), anode Ni plate (pure Ni), current, and time.

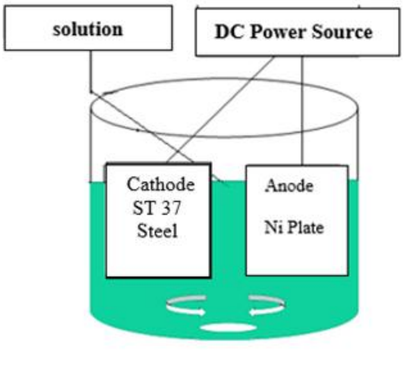

Figure 1. The series of electrodeposition processes

The EDP procedure began by applying an electric current of 4, 5, and 6 A for 3, 4, and 5 hours, respectively, and then adding 1, 2, and 3 g of additives to achieve the best porous sediment outcomes. This electrolysis procedure was carried out by providing an external current, and a direct current source connected in series with an ammeter, and the electrodeposition process took place at a temperature of 25 °C. After electrodeposition, the cathode was removed and rinsed with distilled water (aquades) and then dried in the open air using a hairdryer.

To create a porous material, the cathode was heated in a tube furnace for approximately 30 minutes at room temperature to 275 °C. This caused the nickel layer to peel off and the corn starch to evaporate.

In this study, testing and characterization of nickel mesoporous deposits from the electrodeposition process were carried out using visual inspection, XRD (x-ray diffraction), and SEM (scanning electron microscope) equipped with EDS (energy dispersive x-ray spectroscopy) characterization [8]-[10]. The visual inspection method uses sunlight and the human eye. The machine tools used for XRD characterization were SmartLab, the Rigaku Ultima IV (Cu-Source) brand, where XRD testing was carried out on mesoporous nickel, which underwent an electrodeposition process, with a current of 4A and a time of 3 hours. The SEM-EDS characterization was a Hitachi SU 3500.

3. RESULTS AND DISCUSSION

3.1 Electrodeposition Process

Figure 2 depicts the effects of the electrodeposition process on the weight of the deposit formed during EDP (electrodeposition) as time and current adjustments were applied. Figure 2 illustrates a graph of the effect of time on the weight of realistic deposits created using the electrodeposition procedure. Nickel deposits were produced using electrodeposition at holding times of 3, 4, and 5 hours and currents of 4, 5, and 6 A, respectively. The nickel deposits were 1.19, 1.3, and 1.9 g.

This research showed an increase in the weight of nickel deposits from a current of 4A to 5A (holding time 3 to 4 hours), weighing 0.11 g (9.24%). And the increase in nickel deposits also occurred from a current of 4A to 6 A (with a standing time of 3 to 5 hours) with a weight of 0.71 g (59.66%), for an additional current of 5 to 6 A with a holding time of 4 to 5 hours which having a difference of one hour, the addition of deposits increased significantly by 46.15% (0.6 g).

In the Ni synthesis process, it can be concluded that the greater the current and the holding time of the EDP process, the greater the weight of the Ni precipitation obtained, as shown by the graph of current flow and the duration of the EDP process against the weight of the metal precipitation (actual weight) obtained.

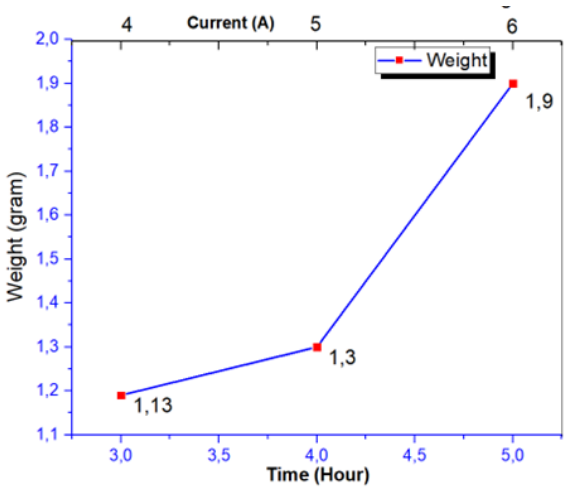

Figure 2. The effect of time on the processed weight of the deposit (actual weight)

Meanwhile, based on equation (1), the theoretical weight indicates that nickel with a process time variation of 3, 4, and 5 hours yields nickel weights of 13.14, 21.90, and 32.85 g, respectively. This is based on Faraday's first law of electrolysis. This law states that the magnitude of chemical changes (mass of substances deposited or dissolved) is directly proportional to the magnitude of electricity (charge) flowing through the electrolyte [16].

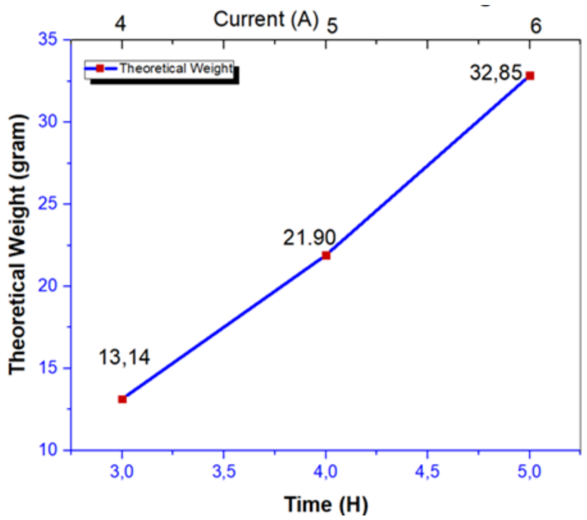

Figure 3. The effect of time and current on the theoretical weight of the deposit

Based on equation 1 for theoretical calculation of Ni electrodeposition process with time (t) = 3 hours (10800 s), current (I) = 4 A, molar (M) Ni = 58.69 g/mol, n is Ni's valence = 2, and F is the Faraday constant = 96.485 C/mol. Accordingly, the nickel (Ni) mass deposited during the three-hour EDP operation at 4 A is approximately 13.14 g. The weight of Ni obtained at times (t) 4 and 5 and current (5 and 6 A) is 21.90 and 32.85 g, respectively. Figure 3 shows the results of theoretical calculations. The results of theoretical calculations show that the addition of deposits increased by 66.67% (8.76 g) at a holding time of 3 to 4 hours, while at a holding time of 3 to 5 hours, it reached 150% (19.71 g).

The weight of Ni deposits produced by the EDP process for the 3 samples is smaller than their theoretical weight. The actual weight of the EDP process of 3 samples only reaches <3 g of nickel. Based on the theoretical weight, it reaches > 8 g, as shown in Fig. 4. In an electrolysis system using Nickel sulfate hexahydrate ($NiSO_4 \cdot 6H_2O$) in hydrochloric acid (HCl) as the electrolyte, the deposition of nickel (Ni) at the electrode follows Faraday's laws of electrolysis (equation 1), meaning that more current and longer time will lead to more nickel being deposited.

The solubility limit and other practical constraints inherent in this electrolyte system refer to the maximum concentration of $NiSO_4 \cdot 6H_2O$ that can be dissolved in solution. Once the solution becomes saturated with nickel sulfate, no additional nickel sulfate can be dissolved unless the temperature is raised or the solubility of the solution is changed. Suppose the concentration of Ni^{2+} ions drops below a certain level (due to electrolysis or limited solubility). In that case, the deposition rate will slow and eventually stop when insufficient ions are in the solution to precipitate on the electrodes [16]-[17].

The difference can be caused by a variety of factors, including the fact that the electrodeposition results do not adhere perfectly; this is also caused when removing (grinding) the nickel deposition; sanding must be done so that some of the nickel deposition is eroded, reducing the weight of the nickel deposition attached to the cathode. The pretreatment of the substrate surface before electroplating can have an impact on the adherence and homogeneity of the deposited nickel layer. Surface cleanliness, roughness, and the activation process. Furthermore, the electrochemical reactions involved in nickel electrodeposition are complex and controlled by variables such as overpotential, ion concentration gradients near the surface, and competition between hydrogen evolution and nickel deposition events. This will result in non-uniformity or divergence from the desired weight of nickel deposition as determined by theoretical data. The distribution of current density on the substrate surface might vary due to geometric factors such as substrate shape, proximity to the anode, and so on, which can cause the thickness or deposition of the deposited nickel layer to be non-uniform.

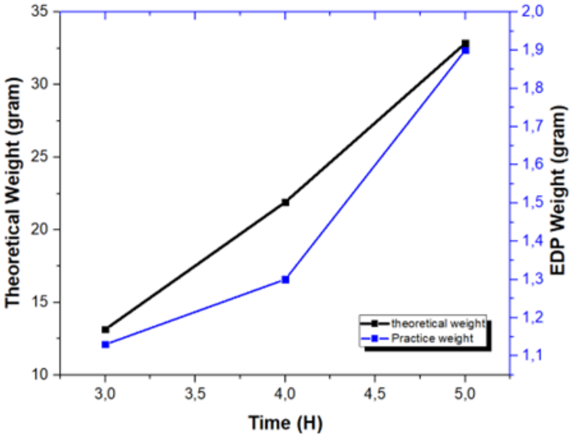

Figure 4. Weight comparison of Ni-EDP results processed with the theoretical weight

Because the electrodeposition process consumes current, the length of the process time influences the current efficiency. For example, sample 1, with a current of 4 A and a time of 3 hours, has a current efficiency of 9.01%, while sample 2, with a current of 5 A and a time of 4 hours, has a current efficiency of 5.9%. Sample 3, with a current of 6 A and a processing time of 5 hours, has a current efficiency of 6.9%, as shown in Fig. 5.

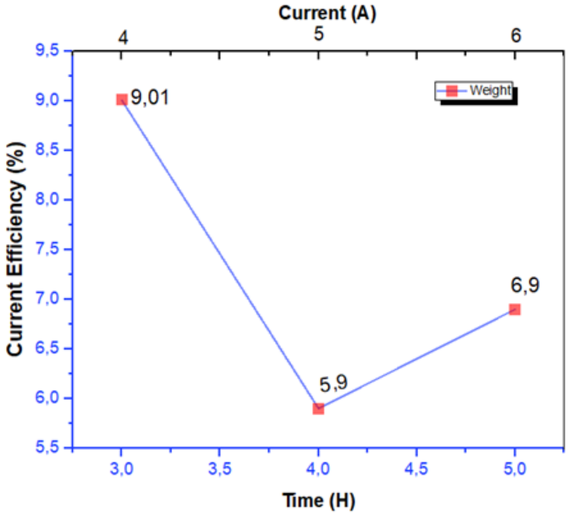

Figure 5. The effect of time on current efficiency

Current efficiency is related to the ratio of theoretical weight to actual weight. If the real weight approaches the theoretical weight, it generates a high percentage of current efficiency, and vice versa. If the actual weight deviates from the theoretical weight, the percentage of efficiency will decrease. The Optimal EDP process can cause variation of current efficiency during nickel EDP with different holding times of 3 hours. The conditions (e.g., nickel ion concentration, temperature, pH, current) are likely optimal, resulting in higher efficiency due to balanced ion availability and minimal side reactions [17]. During the 4-hour EDP process, Depletion and Increased Side Reactions occur; after 4 hours, the electrolyte may be depleted of nickel ions, and side reactions such as hydrogen evolution may increase, reducing the current efficiency. While at 5 hours of re-stabilization here, a new equilibrium may be reached, with some replenishment or redistribution of ions and reduced side reactions compared to 4 hours, leading to a slight increase in efficiency. The changes in efficiency are due to dynamic changes in electrolyte composition, surface conditions, and side reactions over time.

3.1 Visual Inspection

The visual inspection aimed to identify the color and appearance of nickel deposits formed during the electrodeposition process.

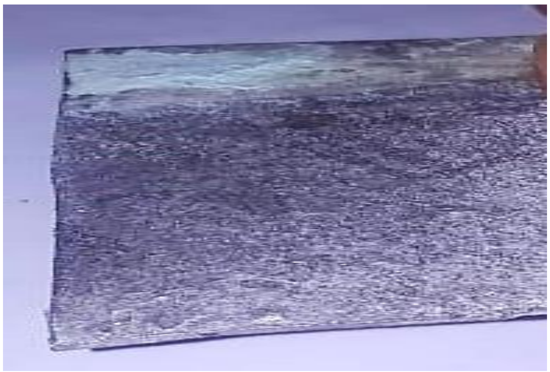

Figure 6. Visual appearance of nickel deposits attached to the cathode

The results of the Ni electrodeposition method with cornstarch additives yield porous nickel deposits linked to the cathode (ST-37 steel), as illustrated in Fig. 6.

Figure 7. Visual appearance of nickel deposits after being removed from the cathode

The electrodeposition method produces a thin layer. This layer has been peeled off the cathode and cured; it appears polished and has no color change, as shown in Fig. 7. However, the peeling thin layer only comprised a small portion of the existing cross-sectional area because the nickel deposits adhering to the cathode were not precisely even. Because the electrodeposition process time was less than ideal, the thickness of each surface of the nickel deposit was not uniform. The strength of the resulting nickel deposit layer varied depending on its thickness.

3.3 XRD Results

This XRD (x-ray diffraction) test detects the phase and compound generated during the Ni electrodeposition process, which uses $NiSO_4.6H_2O$ electrolyte solution [6]. Figure 8 shows the XRD test results, which identified phases such as α-Ni (81%), Ɣ-Fe (0.6%), and the compound produced Fe_1Ni_3 (18.5%).

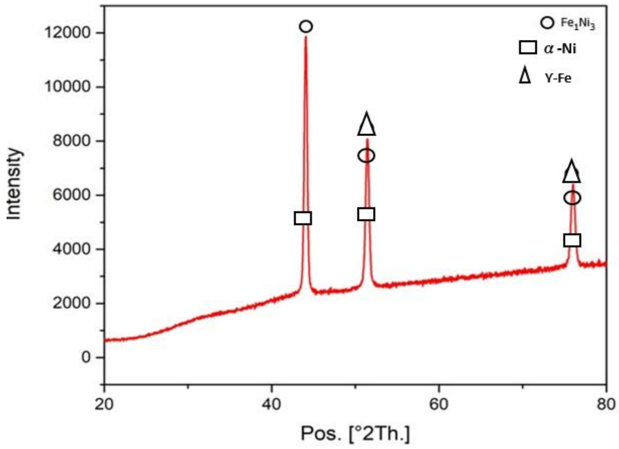

Figure 8. XRD results of nickel (electrodeposition)

3.4 SEM Results

The goal of SEM testing is to observe the electrodeposition process's results as well as the shape of the pores created by the operation. SEM testing findings are utilized to determine the size of pores generated during the electrodeposition process.

Based on the results of SEM (scanning electron microscopy) data characterization with a magnification of 500x on the test sample of the EDP process for 3 hours and a current of 4 A using 1 g of additive (corn starch), the surface morphology shows that there were two pores formed with a fairly large distance between them and their distribution was uneven, indicating that the pores formed did not reach the mesoporous size, as shown in Fig. 9. To demonstrate this, magnification was increased to 3000x, resulting in pores estimated to be ±10μm in diameter, as seen in Fig. 10.

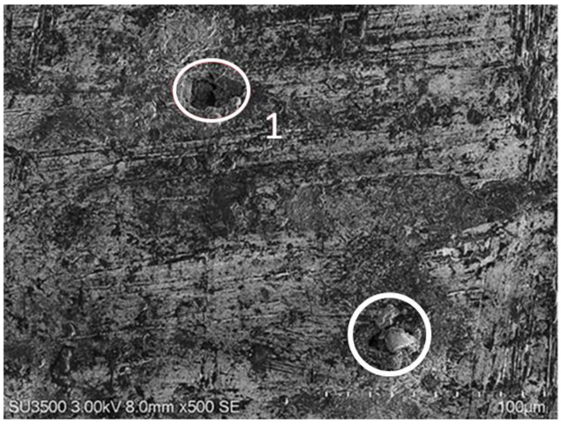

Figure 9. SEM results for mesoporous Ni for 3 hours, 4 A current, and 1 g of corn starch

SEM characterization results of EDP process samples using current I = 5 A, time t = 4 hours, and cornstarch weight of 2 g with a magnification of 2000x identified the presence of pores that began to spread evenly on each surface.

Figure 10. SEM results show that the porous size is estimated at 10 micrometers

Pore measurements were carried out at three points to obtain the diameters 1.09 μm, 689 nm, and 527 nm, as shown in Fig. 11.

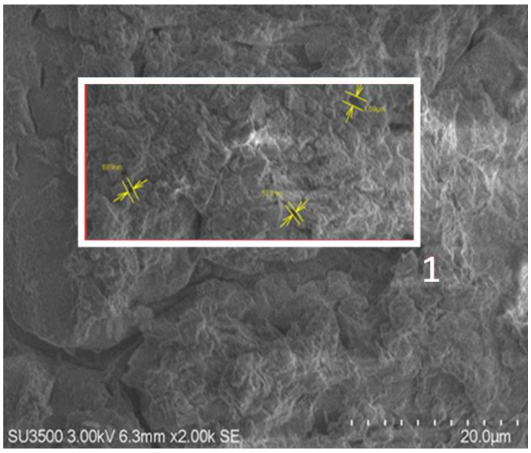

Figure 11. SEM test results of Mesoporous Ni for 4 hours, 5 A current, and 2 g of corn starch

The smallest pore size (527 nm) was found from three sites in these samples, indicating that the pore size created by this EDP technique is less than 600 nm. After performing a 3000x magnification, the surface morphology of the shaft size revealed several smaller shafts than before. Three points of the shaft size were taken, approximately in diameter 1.05 µm, 725 nm, and 516 nm. The smallest shaft size taken from the three different points was 516 nm in diameter. All the porous sizes obtained were almost evenly distributed between the two magnifications.

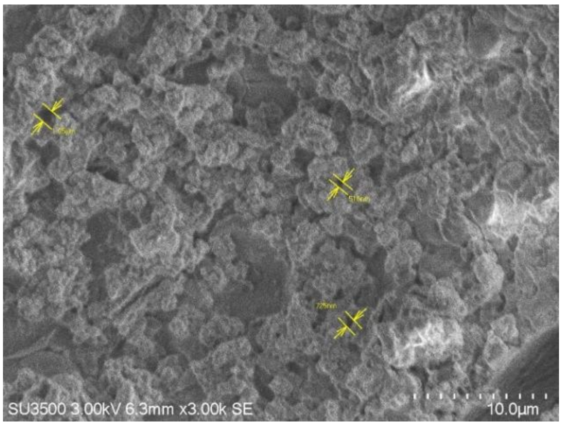

Figure 12. SEM test results on EDP process samples for 4 hours, 5 A current, and 2 g of corn starch, porous size is almost even

Figures 11 and 12 show the results of SEM testing, which used porous measurements to evaluate data distribution and how close the data was to the mean value, and the standard deviation or data deviation. The porosity generated at 2000x magnification was 1090 nm, 689 nm, and 527 nm, with a standard deviation of 289.83 nm and a mean of 768.67 nm. The standard deviation value is smaller than the average value (mean) of the results of measurements with 2000x and 3000x magnification, which indicates that the data is less varied using the data sufficiency test where the data confidence level is 90% with an error rate of 10% (0.1) requiring data from 16 test points. Due to testing limitations, the data confidence level is reduced to 80-90%, with an error rate of 20% (0.2). Consequently, the data sufficiency test requires 5.33 or 6 test points, given the almost homogeneous pore sizes.

At 3000x magnification, the pore sizes of 1050 nm, 725 nm, and 516 nm have a standard deviation of 269.09 nm and an average of 763.67 nm. The standard deviation value is smaller than the average value (mean) of the measurement results at 3000x magnification, this indicates that the data is less varied and the data confidence level ranges from 80-90% with an error rate of 20% (0.2) where the data adequacy test becomes 4.64 or at least there are 5 test points, with almost even and homogeneous pore sizes.

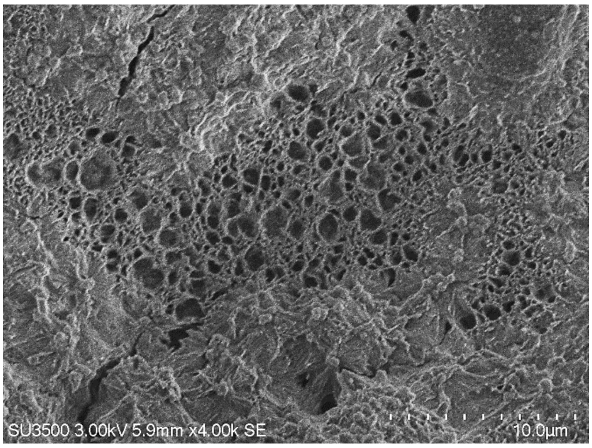

Figure 13. SEM test results of Mesoporous Ni for 5 hours, current of 6 A, and corn-starch weight of 3 g

SEM characterization results of samples with an EDP process duration of 5 hours, a current of 6 A, and a corn starch weight of 3 g at a magnification of 4000x revealed the presence of pores on all surfaces. The porous diameter was around 200 nm, and the space between pores was extremely tight, as illustrated in Fig. 13. A larger magnification was applied to the test sample, specifically 5000x. It was determined by the surface morphology of the porous size obtained at three sites along the porous diameter size, namely 195 nm, 170 nm, and 112 nm. According to the comparison of the three spots, the smallest porous size was 112 nm in diameter, as illustrated in Fig. 14.

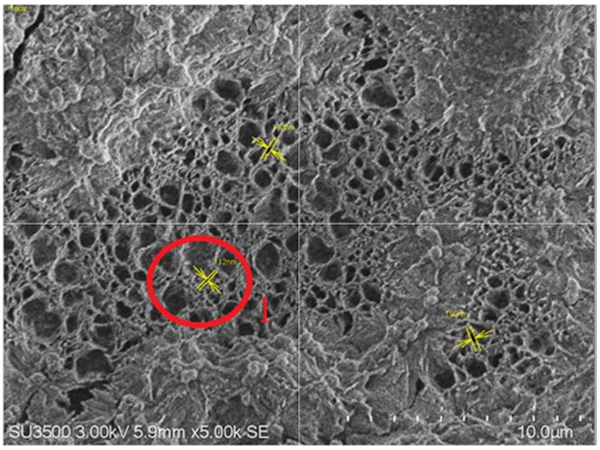

Figure 14. SEM test results for the sample with an EDP process time of 5 hours, current of 6 A, and cornstarch weight of 3 g show the shape of the shaft with a mesoporous size with a diameter of 112 nm

Based on SEM data, it can be observed that adding a substance, specifically corn starch, to a 1 M $NiSO_4{\cdot}6H_2O$ solution can produce pores in a thin layer of nickel deposits. The higher the content of corn starch given, the more pores are formed and distributed. However, this study is currently being conducted to determine the limits of corn starch addition, as excessive amounts of corn starch can increase oligomerization (clumping), rendering the electrodeposition process less than ideal.

3.5 EDS Results

Based on the data obtained from the EDS (energy dispersive spectroscopy) characterization of samples with variations of I = 4 A, t = 3 hours, with the addition of corn starch additives = 1 gram, testing in selected area 1, the composition of the identified elements was Ni, Fe, P, and Cl, with levels of 81.96 wt.%-Ni and 6.52 wt.%-Fe, the remaining of 0.83 wt.%-Cl and 1.86 wt.%-P, shown in Fig. 15 and Table 1.

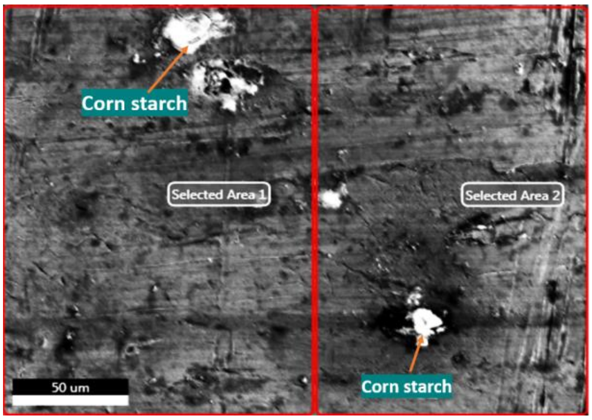

Figure 15. Selected area SEM EDS selected area 1 & selected area 2

While in selected area 2, the composition of the elements was 85.35 wt.% Ni, the second position is Cl of 6.86 wt.%, At the same time, Fe ranked third at 6.52 wt.%, with P at 0.79 wt.%, as shown in Fig. 15 and Table 2.

Table 1. EDS test results selected area 1

Element	**EDS Test Selected Area 1**	
	Weigth%	**Atomic%**
FeL	7.63	6.51
NiL	81.96	66.53
ClK	1.86	2.86
PK	0.83	1.12

The EDS measurements reveal that the nickel EDP procedure was successful, as the Ni deposit on the workpiece's surface exceeded 80%.

Table 2. EDS test results selected area 2

Element	**EDS Test Selected Area 2**	
	Weigth%	**Atomic%**
FeL	6.52	5.73
NiL	85.35	71.32
ClK	6.86	21.04
PK	0.79	1.25

Ni concentration on the surface of ST 37 steel exceeds 80 wt%. The EDS test results show a low P content (<1% wt%) in areas 1 and 2. This is due to the cornstarch additive that was left behind/attached to the Ni deposit, as evidenced by the SEM test results in Fig. 15. The presence of Cl (Chloride) comes from the HCl electrolyte solution used in the EDS process, which was left behind and attached to the Ni deposit. The BCPS, ESC, MMBPs, LLC, and EDP are examples of current approaches used in mesopore research [12]-[18]. The EDP process carried out by previous researchers used ethanol, alkali, and polymer catalysts [12]-[19]. In contrast, the new thing about this EDP process is cornstarch as a catalyst/additive that creates porous materials.

4. CONCLUSION

The electrodeposition process showed that the amount of nickel (Ni) deposited increased with higher electric current and longer processing time, with the lowest deposition of 1.19 g occurring at 4 A and 3 hours, increasing to 1.3 g at 5 A and 4 hours, and reaching the highest at 1.9 g with 6 A and 5 hours. SEM (scanning electron microscope) analysis revealed that the largest pore diameter, 1090 nm, was found in sample 1, which used a current of 4 A, a duration of 3 hours, and an addition of 1 gram of corn starch, while the smallest pore diameter of 112 nm was observed in sample 3 (6 A, 5 hours), with an average pore size of approximately 200 nm and closely packed structures. These findings suggest that increasing the concentration of corn starch leads to more porous and evenly distributed deposits. Furthermore, XRD (x-ray diffraction) analysis identified the presence of α-Ni, Ƴ-Fe, and Fe_1Ni_3 phases, and combined XRD and SEM-EDS results confirmed the formation of porous nickel deposits with a nickel content exceeding 80%.

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