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Fabrication and characterization of Self-Supported NiCo-LDH Arrays for High-performance Supercapacitor

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ABSTRACT

Electrochemical capacitors have emerged as promising complementary energy storage systems to conventional batteries due to their high-power density, rapid charge–discharge capability, and long cycle life. Extensive research has focused on carbon-based electrode materials, which offer high surface area and good electrical conductivity, enabling enhanced charge storage through electric double-layer mechanisms. However, despite these advantages, the widespread replacement of batteries by electrochemical capacitors remains constrained by limitations associated with electrode materials and fabrication methodologies. In particular, complex synthesis routes, material costs, and scalability challenges continue to impede large-scale industrial deployment. Consequently, the development of cost-effective materials and simplified fabrication strategies remains a critical objective in advancing next-generation supercapacitor technologies. *In this paper* Nickel–cobalt layered double hydroxide (NiCo-LDH) self-supported electrodes were fabricated via a cost-effective electrodeposition route on hydrophilically treated indium tin oxide substrates for high-performance supercapacitor applications. Surface roughening using potassium permanganate treatment enhanced wettability and effective surface area, while subsequent nickel electroplating reduced substrate resistance from 34.4 Ω to 1.8 Ω, significantly improving charge transport. Field-emission scanning electron microscopy revealed a hierarchical nanosheet morphology with pronounced peaks and valleys, providing abundant electroactive sites for Faradaic reactions.

Electrochemical performance was evaluated in 2 M KOH using cyclic voltammetry, galvanostatic charge–discharge, and electrochemical impedance spectroscopy. The electrode exhibited well-defined redox peaks within a potential window of 0–0.55 V and delivered a high areal capacitance of 475 mF cm^{-2} at 1 mA cm^{-2}, retaining 275 mF cm^{-2} at 30 mA cm^{-2} (42% retention). The small semicircle in Nyquist plots indicates low charge-transfer resistance and efficient ion diffusion. Compared with previously reported carbon-based electrodes and conventional NiCo-LDH systems, the present electrode exhibits competitive areal capacitance and improved conductivity through direct current collector integration. The combination of simple fabrication, reduced internal resistance, and stable electrochemical behavior highlights its potential for scalable industrial energy storage applications such as backup power systems and hybrid capacitive devices.

Keywords*:* Nickel-cobalt layered double hydroxide; Self-supported electrodes; Electrodeposition; Electrochemical capacitor, Supercapacitors, cyclic stability.

INTRODUCTION

Electrochemical capacitors have been achieved due to the ever-increasing need for energy by the population. The research and development of an electrochemical capacitor began with the invention of a Leiden Jar which yielded a capacitor which is a device that stores electrical charge in small quantities i.e. microfarads [1]. Capacitors play a crucial role in electrical and electronic appliances since they are used in rectification, backup circuits of the microcomputers, and timer circuits making use of periods to either charge or discharge electricity. Capacitors are also applied to block the flow of direct current in filters responsible for extracting or eliminating specific frequencies. These circuits are applied on circuits where excellent frequency characteristic is a requirement [2] [3] [4] [5].

The advancement of technology in 21st century has given rise to the application of supercapacitor due to high demand of portable and wearable devices. Electrochemical capacitor is made up of an electrolyte material sandwiched between two identical electrodes. It stores electrical energy in the form of an electrochemical double layer which is created at the interface of the solid electrode with either liquid or solid electrolyte. Both positive and negative ionic charges present in the electrolyte materials accumulate at the surface of the electrode and compensate for the electronic charges present at the surface of the electrode [6] [7]. The charging process of this capacitor results in the alignment of opposite polarity charges in their respective opposite charge electrodes, ions present in the electrolyte diffuse over the separator and onto the pores of the electrode with opposite charge or polarity. Ions are prevented from recombining at the electrodes using a double layer of charge that is created in the process. Combining both double layers results in an increase in the specific surface area, and the distance between electrodes results in EDLCs attaining high energy density [2] [8].

The mechanism used by EDLCs to store electrical charges makes it possess a feature such as fast energy uptake, delivery, and better power performance compared to other types of electrochemical capacitors. Because of the non-faradaic process, swelling that is common in an active material demonstrated by a battery during charging and discharging is eliminated. EDLCs and batteries vary from each other in few ways, for instance, EDLC can undergo millions of charge-discharge cycles compared to batteries which can withstand few thousands of such charge-discharge cycles before it is replaced. Also, the charge storage mechanism in EDLC does not involve the solvent of the electrolyte while in battery storage mechanism contributes to solid electrolyte inter-phase when high potential cathodes are used or graphite anodes [9] [10]. Even though EDLCs have better properties than batteries, they experience a limited energy density thus explaining the reason why various recent research on EDLC is focused on increasing energy performance and improving the temperature range where batteries cannot operate. The performance of an EDLC is adjusted based on the type of electrolyte used.

Since the commercialization of ECs in the 1960s, there has been a lot of research work aimed at improving their performances. Materials used in fabricating ECs play a vital role in the amount of capacitance it can store. Materials of an EC are substrate-supported, implying that more than one material with different properties can be combined using a specific method [11]. For this reason, most of the research has focused on the best materials that can produce an EC or supercapacitor with better performance and the method in which the same materials are brought together to make up a supercapacitor or EC. In this section, therefore, the previous research work will be discussed. It will be categorized into two parts, that is.; the research focused on materials.

In the research done by [12] and [13], they found that the energy density of an EC can be increased by making use of its specific surface area through wettability improvement [14] [15]. A highly flexible supercapacitor electrode synthesized using the electro-deposition method was done. In their paper, manganese (IV) oxide was electrodeposited onto carbon cloth designed using the facile in-situ electrodeposition method. From the successfully synthesized electrode, it showed high flexibility with multiple layers structures which possessed very high specific capacitance $325Fg^{-1}$ at a current density of $0.2Ag^{-1}$and an excellent rate capability with capacitance retention of 70% at a high current density of $5.0Ag^{-1}$ [16],

In a work published in [17], an attempt was made to synthesize a flexible supercapacitor using nano-carbons, manganese (IV) oxide, and PEDOT: PSS fibers. The team synthesized the flexible fiber supercapacitor by wet-spinning using carbon nanotube. The surface treatment was deposited manganese (IV) oxide and PEDOT: PSS onto the substrates to make the ternary composite fibers. Their research found out that by coating the fibers after the wet spinning step, a simple solution-based continuous process results in forming a fiber-based energy storage device.

Xing et al., 2017 [18] investigated the potential use of nickel-cobalt layered double hydroxide as a battery-type hybrid supercapacitor. This material has a unique spatial structure, excellent electrochemical activity and good electrical conductivity. However, this material is associated with challenges such as low electronic conductivity resulting in low capacitance. [18]. An investigation aimed at optimizing the electrode preparation methodologies to realize the superior performance of supercapacitor through a rigorous understanding of underlying physical parameters [19].

Zhang et al., 2014 [20] team, conducted a study on Nitrogen-doped hierarchically porous carbon Nano-whisker ensembles on carbon nano-fiber for high-performance supercapacitors.

They combined surfactants and template less wet chemical and post-high-temperature carbonization strategies for obtaining a new class of nitrogen-doped hierarchical porous carbon nano-whisker ensembles supported on carbon Nano-fibers (NHCNs) with tunable micro-pores and a nitrogen-doping level for high-performance supercapacitors [20].

In their work, Patrice et al. (2009) investigated the materials suitable for supercapacitor applications. They noted in their research that supercapacitors have the potential of replacing batteries as energy storage devices.

A notable performance improvement could be achieved through recent advances in understanding charge storage mechanisms and the development of advanced nanostructured materials [21].

Kim et al., 2015 [22] in their research on the electrochemical capacitor for energy storage and conversion, acknowledge the fact that EC has the potential of replacing a lithium-ion battery in the future. Their work was focused on analyzing the current trends in the research and development of EC and the challenges that are currently preventing ECs from replacing the batteries fully entirely.

Their findings acknowledged that supercapacitors have better properties such as instant charging and discharging with high power and retaining their performance for many million cycles compared to the batteries [23].

With the good properties of N-doped carbons, including their high electronic conductivity, improved hydrophilic properties and easy syntheses and functionalization, Deng et al., 2016 [24] reviewed the potentiality of N-doped carbons in energy storage and conversion applications.

Their work focused on the methods of preparations and their applications in supercapacitors in the past six years. Their review study, found out that N-doped carbons have been prepared in the lab using various strategies ranging from simple heat-treatment of carbons with nitrogen-containing complexes to carbonization of nitrogen-containing complexes under an inert atmosphere or hydrothermal treatment.

During the preparation and heat treatment, it was clear that the strategies that result in high nitrogen content tend to result in a low surface area, while the strategies that lead to an increase in the surface area resulted in a reduction in nitrogen content.

The barrier responsible for the same challenges were attributed to the lack of effective methods to be used in precisely controlling and obtaining ordered porous structures of N-doped carbons Efforts should be focused on the control of the nature of nitrogen content in N-doped carbons for tunable electronic or chemical properties and lack of scalable and inexpensive N-doped carbons preparation method [24].

Wang et al. (2016) [25] reviewed the latest progress in supercapacitors in charge storage, electrode materials, electrolyte materials, systems, characterization methods, and applications.

They mainly focused on the newly developed charge storage mechanism for inter-collative pseudocapacitive behavior which connects the gap between battery behavior and conventional pseudocapacitive behavior and their comparisons. They also touched on the prospects and challenges of supercapacitors in real-world applications.

They found that porous carbon materials are still widely used for electrode materials for ECs or supercapacitors. Most of the previous research focused on optimizing the pore structure through the preparation of ordered and hierarchical pores to improve the utilization of the pores.

The diping process has been used to improve the specific Pseudo-capacitance. They also found that carbon nanotubes and reduced graphene oxides show high electronic conductivity making it a suitable candidate for flexible and printable supercapacitors [25].

From the above reviewed literature of recent publications there is still no simple and robust method and material for use a capacitor electrode, therefor this work intends to describe a novel cost-effective electrodeposition method for sizing NICOLDH based EC electrode.

Materials and equipment

The chemicals used in this research were sourced from Sinopharm Chemical Reagent (SCR) and they were of analytical grade; thus, they were used without further purification.

METHODOLOGY

The fabrication involved two main steps, the preparation of current collectors and active materials. The current collectors' preparation was done by first conducting hydrophilic treatment on the substrate. After this process, the preparation for the electrodeposition plating solutions was done.

The solutions were categorized as solutions A, B, C, and D. Solution B comprised palladium (II) chloride and hydrochloric acid. Solution C and D comprised of nickel (II) sulfate hexahydrate, citric acid monohydrate, nickel (II) sulfate, and ammonium solution. Solution A was the last to be prepared because it is highly deliquescent. It comprises stannous chloride and hydrochloric.

The indium tin oxide substrate was then prepared for electrodeposition by placing solutions A and B in their respective beakers. Inside a water bath. After which, the indium tin oxide substrate was placed inside a beaker with solution A for 10minutes, later removed and cleaned using deionized water, then dried.

The dried substrate was then placed inside a beaker with solution B for 15minutes, after which it was removed and cleaned using deionized water and dried. The water bath temperature was raised to 70°C then a beaker containing solution C and D was placed inside the water bath.

After this, the substrate was placed inside the beaker, and a vigorous reaction was allowed to take place for 3minutes then, the substrate was cleaned using deionized water then dried.

Finally, The current collectors' electrodeposition was prepared using nickel foam as a positive electrode (anode), the substrate being the negative electrode and nickel sulfate as a plating solution.

The parameters were set accordingly on the ‘Kick Start’ software, The electrodeposition of current collectors was done for 600 seconds, then the electrode substrate was removed and cleaned using deionized water then dried. The characterization was done on the NiCo-LDH specimen using field emission scanning electron microscopy.

RESULTS AND DISCUSSIONS

Solution Emersion using Potassium permanganate

Surface roughness plays a critical role in supercapcitor. For that reason, the surface area of the substrate was increased by immersing it in potassium permanagnate solution which reduces the surface energy.

The process resulted in the increase in substrate’s surface roughness which directly contribute to an increase in the specific surface area where the capacitance would be stored. Figure 3.1 shows a microscopic image of the substrate surface after immesion in the potassium permanganate.To make the induced roughness on the substrate’s surface permananent, the substrate was cleaned using plasma cleanr.

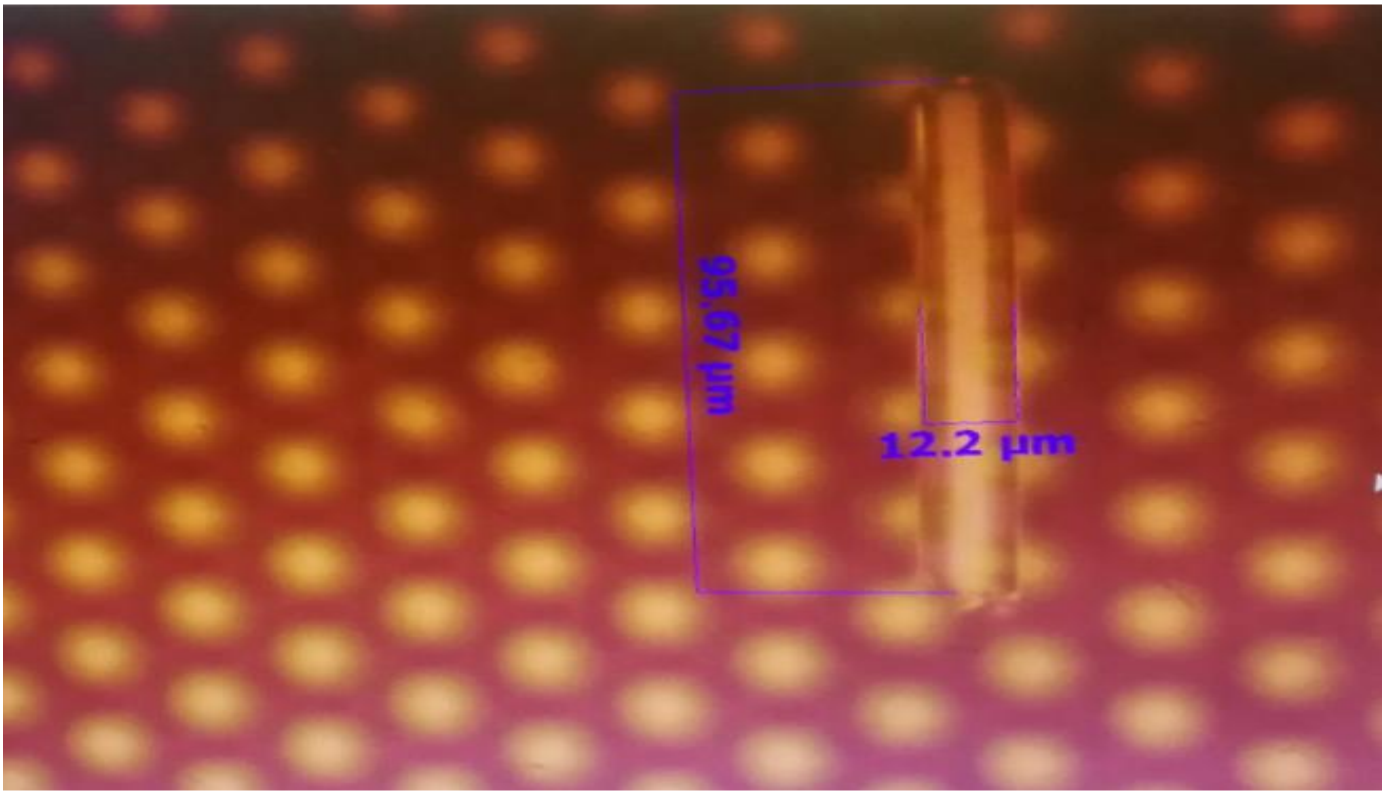

Figure 3. 1: Microscopic image of the surface of the substrate after inserting it in potassium permanganate.

Electroplating.

Theoretically, a supercapacitor has small or negligible internal resistance. This was achieved during the experiment through electro-depositing nickel on the hydrophilic treated electrode substrate using source meter equipment. After inserting the electrode substrate in solutions A, B, C, and D, the resistance of the substrate electrode was still high as shown by figure 3.2(a) i.e. 34.4Ω . The problem was solved by depositing nickel metal onto the substrate ; the resistance decreased by 94.77%, as shown in Figure 3.2(b) i.e. 1.8Ω. As it is evident in figure 3.2(c), the chemical deposited and electroplated nickels substrate is dull-grey while the electrode substrate without nickel was shiny grey.

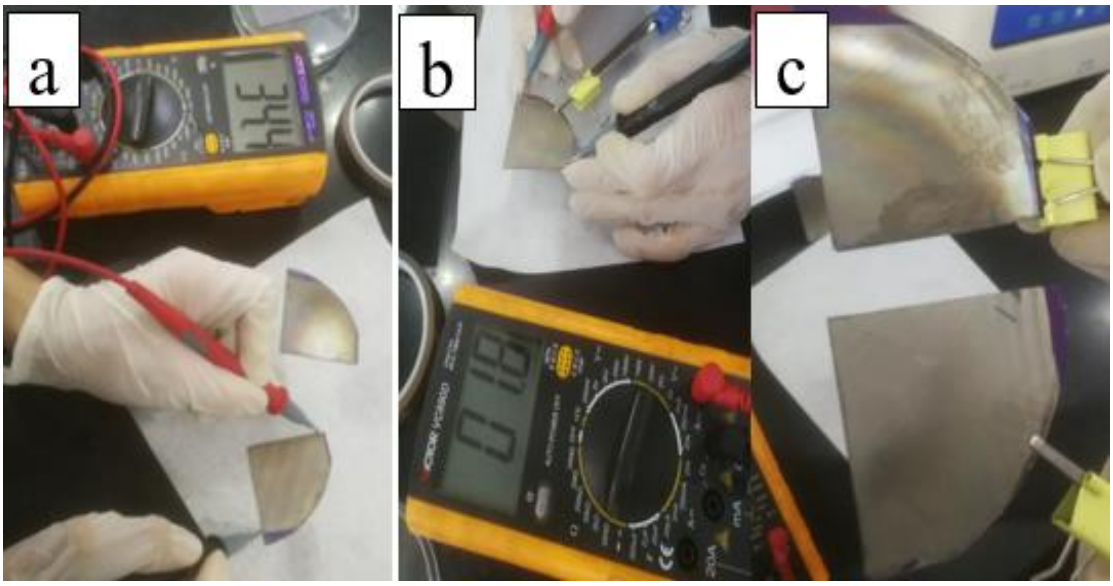

Figure 3. 2(a) Resistance before nickel deposition, figure 3.2(b) Resistance after nickel deposition, and figure 3.2(c) Comparison between the surface of the substrate electrode with and without nickel deposited .

Nickel Cobalt Double-layered Electrode Hydroxide SEM Structure.

Figure 3.3(a), 3.3(b), 3.3(c), 3.3(d), and 3.3(e) show the microstructure of NiCo-LDH scanned at different magnification factors. On the other hand, Figure 3.3(f) shows the NiCo-LDH optical microscope image.

As can be seen, the surface has peaks and valleys which contributes to an increase in the functional surface area of the electrode which directly correspond to more capacitance being retained.

The rough surface of the NiCo-LDH structure indicates an improved contact angle within the microstructures. This corresponds to an improved specific surface area utilization of the electrode.

An improved specific surface area utilization of the electrode, will result in a corresponding increase in the amount of positive and negative ions that can accumulate on the surface of the electrode and thus improve capacitance.

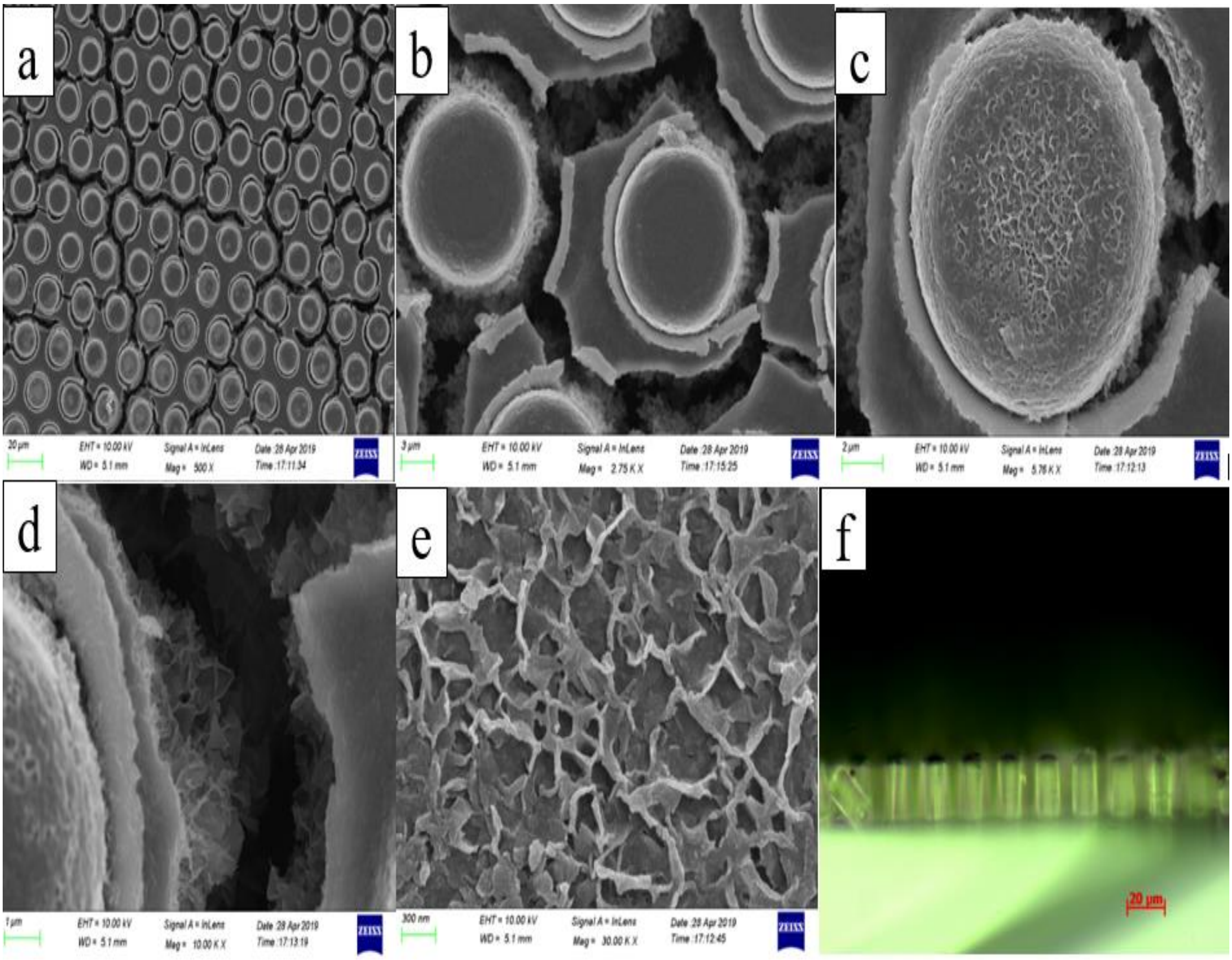

Figure 3. 3: Scanning Electron Microscopy images of NiCo-LDH.

Electrochemical characterization.

The characterization of the synthesized NiCo-LDH was done using electrochemical workstation using cyclic voltammetry, chronopotentiometry, open-circuit potential, and specific capacitance as shown in Figure 3.4(a), 3.4(b), 3.4(c), and 3.4(d).

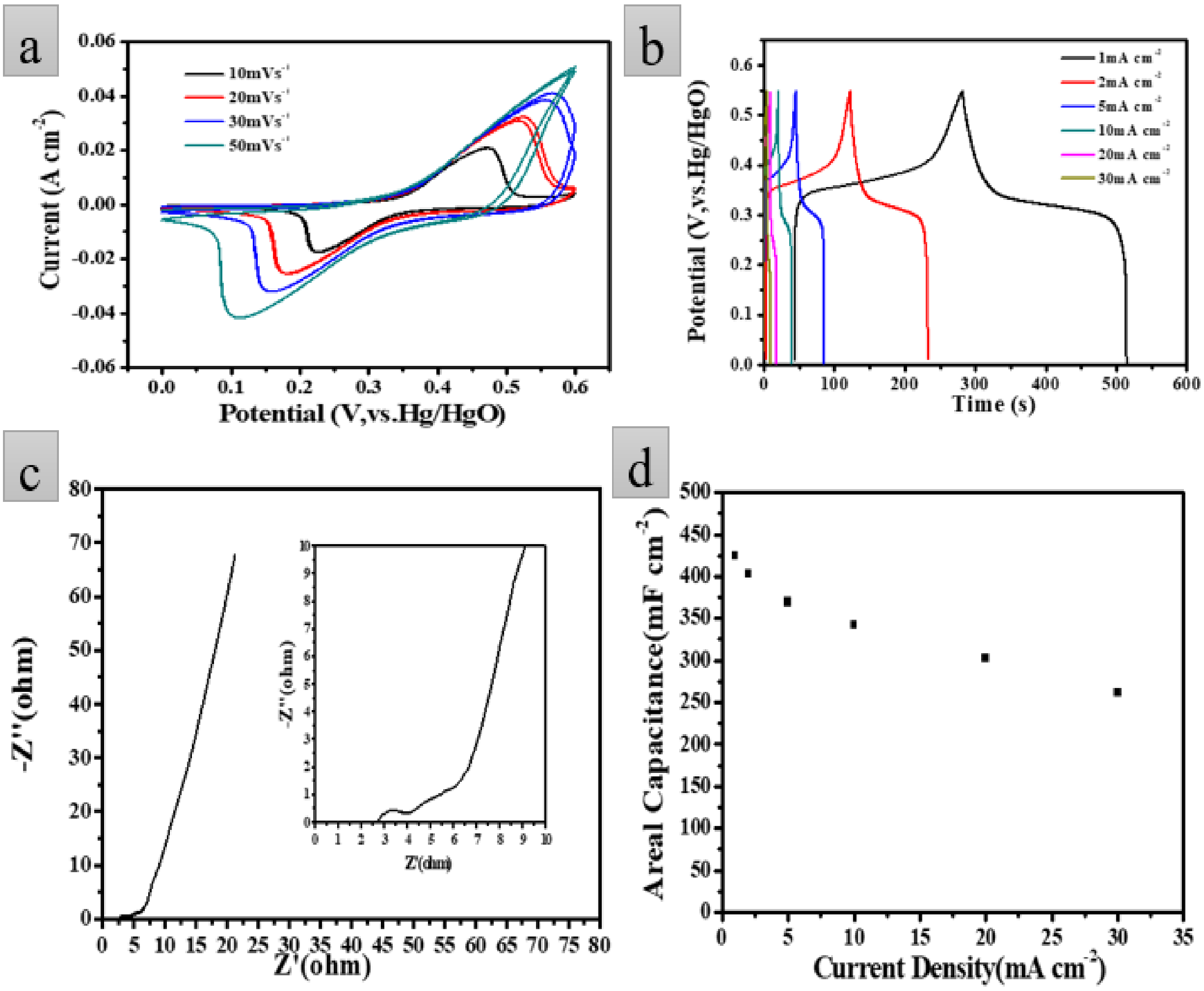

Figure 3. 4 (a) CV curves at various scan rates inside 2M KOH electrolyte, figure 3.4(b) Charge-discharge curves for NiCo-LDH in 2M KOH electrolyte at different current densities, figure 3.4(c) electrochemical impedance spectroscopy in 2M KOH electrolyte and figure 3.4(c) Areal capacitance of NiCo-LDH in 2M KOH electrolyte at different current densities.

Figures 3.4 shows the results processed using the chemistry workstation. The graphs play an important role in interpreting the performance of the synthesized NiCo-LDH electrode.

The synthesized NiCo-LDH electrochemical supercapacitor electrode was characterized using electrochemical workstation at various scan rates. As seen in Figure 3.4(a), the NiCo-LDH electrode specimen showed good reversible redox reaction peaks falling in the range of -0.04Acm^{-2} to 0.05Acm^{-2}. This is attributed to the faradaic redox reaction between nickel and cobalt ions as illustrated by chemical equations below.

$$Ni(OH)_2 + OH^- \leftrightarrow NiOOH + H_2O + e^-$$
$$CO(OH)_2 + OH^- \leftrightarrow CoOOH + H_2O + e^-$$
$$CoOOH + OH^- \leftrightarrow CoO_2 + e^-$$

It can also be seen in Figure 3.4(a) that the NiCo-LDH electrode specimen had very high current peaks implying that the charge transfer on the electrode specimen improved. The good redox reaction symmetry of the curves at various scan rates implies that the NiCo-LDH electrode specimen has good electrochemical reversibility. It is also clear from the same Figure 3.4(a) that the current peaks increase with the scan rates due to more diffusion rates compared to reaction rate.

Figure 3.4(b) shows charge-discharge curve at different current densities;. 1mAcm^{-2}, 2mAcm^{-2},5mAcm^{-2}, 10mAcm^{-2}, 20mAcm^{-2} and 30mAcm^{-2}. From the curve, the potential range between charging and discharging falls within 0V to 0.55V and is attributed to improved specific surface area utilization of the electrode substrate, which in turn increases specific surface area utilization responsible for more capacitance. Also from the same curve, the higher the current density, the lower the charging-discharging time and vice versa as evidenced by the longer charging and discharging time for a current density of 1mAcm^{-2} than a charging and discharging time for a 30mAcm^{-2} current density.

Electrode electrochemical impedance spectroscopy test was also carried out to investigate the impedance of the resulting electrode. As shown in figure 3.4(c), the curve has a region of high frequency represented by half arc, a region of intermediate frequency as represented by the transition section, and low frequency region represented by the sizeable straight slope. The diameter of the half arch region shows the electrode and electrolyte surface charge transfer resistance, while the small arc diameter represents the small or negligible electrochemical impedance. On the other hand, the low-frequency region represented resistance of ion diffusion within the material with larger straight slopes, a performance with similarities with an ideal capacitor.

Figure 3.4(d), illustrates that the amount of capacitance reduces as the current density increases. This is evident by the falling curve with a reduction of 5.89%, 11.76%, 17.65%, 29.41%, 41.18% of capacitance for the current densities of 1mAcm^{-2}, 2mAcm^{-2}, 5mAcm^{-2}, 10mAcm^{-2}, 20mAcm^{-2} and 30mAcm^{-2} respectively. The poor capacitance retention of NiCo-LDH as the current density increases is attributed to its poor intrinsic conducting nature otherwise, good capacitance retention at small current densities makes it good for industrial applications such as energy power backups in computers, among others.

As discussed in the previous paragraphs, the synthesized supercapacitor based on NiCo-LDH shows an excellent response to the application of various parameters. The results make the NiCo-LDH supercapacitor electrode a suitable candidate for supercapacitor electrode applications. For instance, carbon-based electrodes have been in applications for a long time. Now that there is a new material candidate, the reality of constructing a supercapacitor that can store more electrical energy can be realized.

CONCLUSIONS AND RECOMMENDATIONS

The NiCo-LDH electrochemical capacitor electrode synthesized using the electrodeposition method was found to show good reversible redox reactions, which in turn improved its faradaic reaction. The results exhibited an improved charge transfer as evidenced by high current peaks and good electrochemical reversibility thus having battery-like features.

The electrode was also found to retain more capacitance at lower current density.For instance, it could retain 425mFcm^{-2} and 225mFcm^{-2} at current densities of 1mAcm^{-2} and 30mAcm^{-2} , respectively. The charge-discharge potential range of the synthesized electrode was 0.55V and this was contributed by improved wettability. All the research objectives were achieved and thus, it was successfully done. The study recommend the use of NiCo-LDH as a material for supercapacitor electrode applications.

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