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From: Materials Science for Energy Technologies (em@editorialmanager.com)

To: andrifis@ymail.com

Date: Saturday, July 23, 2022, 08:57 PM GMT+7

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Date: Tuesday, August 9, 2022, 01:14 PM GMT+7

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Journal: Materials Science for Energy Technologies
Title: Effect of surface area on electrical properties of NiCo2O4-reduced graphene oxide nanocomposites for supercapacitor electrodes applications
Corresponding Author: Mr Syahrul Humaidi
Co-Authors: Andriono Manalu, M.Pd; Kerista Tarigan, Dr.; Masno Ginting, Prof.; Istas Pratomo Manalu, M.Sc.; Ikhwanuddin Ikhwanuddin
Manuscript Number:

Dear Mr. Andriono Manalu,

Mr Syahrul Humaidi submitted this manuscript via Elsevier's online submission system, Editorial Manager, and you have been listed as a Co-Author of this submission.
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To: "andrifis@ymail.com" <andrifis@ymail.com>
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Dari: **Materials Science for Energy Technologies** <em@editorialmanager.com>
Date: Jum, 16 Sep 2022 pukul 23.57
Subject: Decision on submission to Materials Science for Energy Technologies
To: Syahrul Humaidi <syahrul1@usu.ac.id>

CC: ejaz@iitism.ac.in, ejaz.alchemy@gmail.com, ejaz.iitd@gmail.com

Manuscript Number: MSET-D-22-00029

Effect of surface area on electrical properties of NiCo2O4-reduced graphene oxide nanocomposites for supercapacitor electrodes applications

Dear Mr Humaidi,

Thank you for submitting your manuscript to Materials Science for Energy Technologies.

I have completed my evaluation of your manuscript. The reviewers recommend reconsideration of your manuscript following major revision. I invite you to resubmit your manuscript after addressing the comments below. Please resubmit your revised manuscript by Oct 16, 2022.

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Editor-in-Chief

Materials Science for Energy Technologies

Editor and Reviewer comments:

Reviewer #1: File attached

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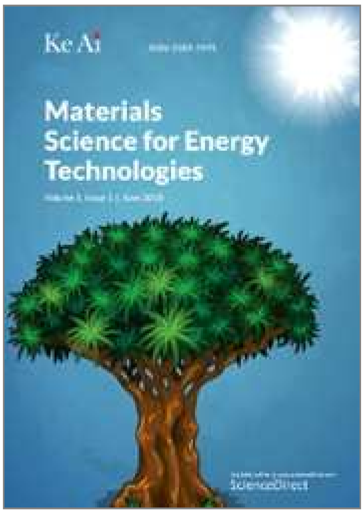

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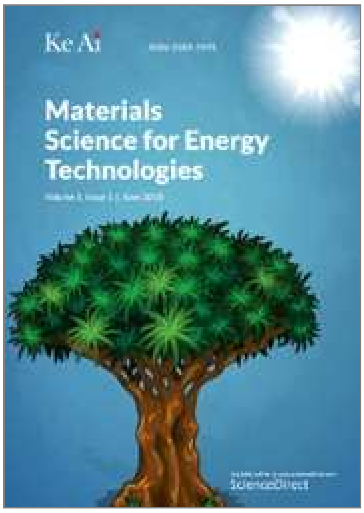

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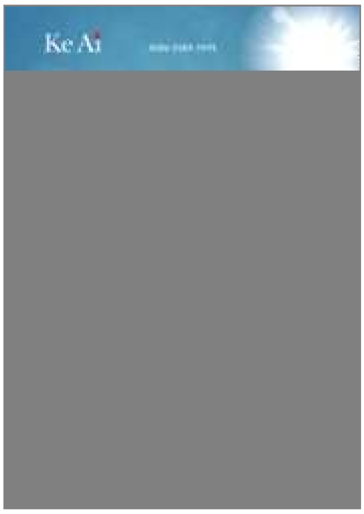

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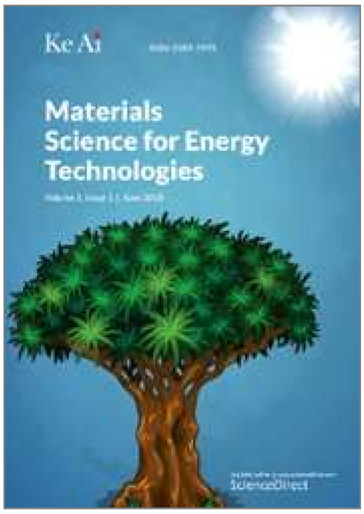
> 
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To: arielgibran91@gmail.com

Date: Friday, November 4, 2022, 09:37 AM GMT+7

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Kepada: "andrifis@ymail.com" <andrifis@ymail.com>
Cc:
Terkirim: Jum, 4 Nov 2022 pada 7:56
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Our reference: MSET 303
Article reference: MSET_MSET-D-22-00029
Article title: Effect of surface area on electrical properties of NiCo2O4-reduced graphene oxide nanocomposites for supercapacitor electrodes applications
To be published in: Materials Science for Energy Technologies
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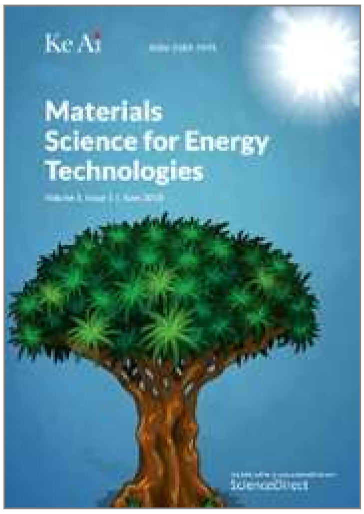

As co-author of the article *Effect of surface area on electrical properties of NiCo2O4-reduced graphene oxide nanocomposites for supercapacitor electrodes applications*, we are pleased to let you know that the final open access version – containing full bibliographic details – is now available online.

The URL below is a quick and easy way to share your work with colleagues, other co-authors and friends. Anyone clicking on the link will be taken directly to the final version of your article on ScienceDirect.

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Submissions with an Editorial Office Decision for Author

Page: 1 of 1 (1 total completed submissions)

Action	Manuscript Number	Title	Authorship	Initial Date Submitted	Status Date	Current Status	Date F Dispo Set
View Submission	MSET-D-22-00029	Effect of surface area on electrical properties of NiCo2O4-reduced graphene oxide nanocomposites for supercapacitor electrodes applications	Other Author	Aug 09, 2022	Oct 19, 2022	Completed - Accept	Oct 19 2022

Page: 1 of 1 (1 total completed submissions)

1

Materials Science for Energy Technologies

Effect of surface area on electrical properties of NiCo2O4-reduced graphene oxide nanocomposites for supercapacitor electrodes applications

--Manuscript Draft--

Manuscript Number:	MSET-D-22-00029R1
Article Type:	Research Paper
Keywords:	Keywords: NiCo2O4/rGO nanocomposite, Electric charge storage, Pseudocapacitor electrode, Specific capacitance, Specific surface area
Corresponding Author:	Syahrul Humaidi, Dr. University of Sumatera Utara Medan, North Sumatera INDONESIA
First Author:	Andriono Manalu, M.Pd
Order of Authors:	Andriono Manalu, M.Pd
	Kerista Tarigan, Dr.
	Syahrul Humaidi, Dr.
	Masno Ginting, Prof.
	Istas Pratomo Manalu, M.Sc.
	Ikhwanuddin Ikhwanuddin
Abstract:	Based on the method of electrical charge storage, supercapacitors are divided into two categories, double-layer electrical capacitor (EDLC) and pseudocapacitors. Utilizing three processes—reversible adsorption, redox reactions on metal oxides, and reversible electrochemical—pseudocapacitors are utilized for high power applications involving metal oxide electrodes and the transfer of electric charge based on a reversible faradaic. In the fabrication of supercapacitors, a high specific surface area with a relatively narrow pore size distribution is essential. Therefore, it is required to increase the capacitance of the material. In this work, nickel cobaltite (NiCo2O4) synthesized from nanocomposite NiS.5H2O and Co2SO4.7H2O precursors were mixed with reduced graphene oxide (rGO). Coprecipitation and calcination were used to create the nanocomposites. The produced NiCo2O4/rGO nanocomposite was used as a pseudocapacitive supercapacitor electrode. The results showed that sample code S2 with mass variations of NiO, Co3O4, and rGO at a ratio of 2:3:2 had the best performance. The sample had a hexahedron-shaped surface morphology, an average particle size of about 0.005 m2, a specific surface area of 12.75 m2/g, an average pore radius of 9.534nΩ.m, and a pore volume of 0.06404 cm3/g.It also performed exceptionally well in terms of electrical conductivity of 6.078 S/m, electrical resistivity of 0.16 nΩ.m, and capacitance of 289.93 F/g.
Suggested Reviewers:	Jiujun Zhang Fuzhou University wgpcd@yahoo.com.cn His research regarding capacitor gives high impact in many works by other researcher.
	Francois Beguin Poznan University of Technology francois.beguin@put.poznan.pl His published works mostly about material sciences, including electrochemical capacitor. His research gains vast amount of attention by many other publications.
	Jayan Thomas University of Central Florida Jayan.Thomas@ucf.edu We found that his work is vital in the development of supercapacitor. Besides, his contribution in the material sciences must be beneficial for broad studies.
Opposed Reviewers:	
Response to Reviewers:	Dear reviewer, hereby, I attach the final revised file.

COVER LETTER

Andriono Manalu
Universitas Sumatera Utara
Medan 20155, North Sumatra, Indonesia

Dear Editor of the Materials Science for Energy Technologies,

We wish to submit an original research article entitled "**Effect of surface area on electrical properties of $NiCo_2O_4$-reduced graphene oxide nanocomposites for supercapacitor electrodes applications**" for consideration by the Materials Science for Energy Technologies.

We confirm that neither the manuscript nor any parts of its content are currently under consideration or published in another journal.

Our research investigates the electrical properties of modified graphene oxide nanocomposites for supercapacitor application. This study used the $NiCo_2O_4$/rGO nanocomposite as a pseudocapacitive supercapacitor electrode to increase the capacitance of material. The results of this study show that This material's performance as a supercapacitor is better to that of Ni/NCO/RGO. We see that this research fits in the scope of Materials Science for Energy Technologies because the topics we researched and discussed were related to material chemistry. In addition, we expect that the results of this study may be useful for other researchers focused in the development of supercapacitors.

All authors have approved the manuscript and agree with its submission to Materials Science for Energy Technologies. We have no conflicts of interest to disclose.

Please address all correspondence concerning this manuscript to me at andrifis@ymail.com.

Your consideration is very much appreciated. We are looking forward to your favorable reply.

Sincerely,

Andriono Manalu

Responses to Reviewer

Manuscript Number: MSET-D-22-00029

Title: Effect of surface area on electrical properties of $NiCo_2O_4$-reduced graphene oxide nanocomposites for supercapacitor electrodes applications

Responses to Reviewer #1

Reviewer's Comments	Authors' Responses
Abstract: Authors have mentioned about two categories of supercapacitors However, the second type has not been discussed	We have added another type of supercapacitor in the abstract.
Abstract: There are some repeated lines such as "The $NiCo_2O_4$/rGO nanocomposite was employed as a pseudocapacitive supercapacitor"	We have revised this sentence.
Abstract: Before using any abbreviation, the full form should be mentioned. The term rGO is directly used.	We have revised this as suggested.
Why authors have selected Nickel cobaltite ($NiCo_2O_4$) as an electrode substance? Proper justification needs to be given citing reported literature.	We have added an explanation regarding this supporting by some references.
In section 2.2.2, the Authors have mentioned about the preparation of $NiCo_2O_4$ nanoparticles. However, in the characterization section, It is not clear that the particles are in the nano range. HR-TEM of the samples would be the more appropriate technique to confirm the size of nanoparticles.	We are aware of this. Our study and experiments were conducted when laboratory access was still restricted due to the covid pandemic. However, for now, HR-TEM test takes longer to complete (estimated 2-3 months). This is due to the limited supply of testing equipment in our laboratory and the large number of samples awaiting testing. If it is allowed, we will provide the additional data after the test results are obtained.
Fig 4 shows the BJH adsorption and desorption isotherm, However, the pore size distribution of the samples should be shown in BJH pore size distribution.	We have added the pore size distribution plot.

Figure 6 shows that the specific surface area is almost the same for both sample 1 and sample 2 but the specific capacitance of S2 is significantly high, why?	We have added an explanation regarding this.
The authors have prepared 7 samples of nanocomposites with various compositions. However, the comparison of their surface area is missing as per the title of the paper. Only sample 1 and sample 2 have been characterized.	We have corrected the data we presentin the manuscript. So, we removed the data from samples 4 to 7 and left samples 1, 2, and 3. We have focused on the discussion only on samples 1, 2, and 3, in the next subsection.
Author should rewrite the conclusion where both results of S1 and S2 should be compared	We have revised the conclusion as suggested. However, we only state the values for the optimum sample (S2).
Line number in the manuscript is missing.	We have inserted line number in the revised manuscript.
The grammar needs to be checked carefully throughout the manuscript. Some sentences need rewriting.	We have revised the manuscript and corrected some error in writing.

Responses to Reviewer #2

Reviewer's Comments	Authors' Responses
This study doesn't give any new insights for readers. The manuscript should highlight the novelty and innovation of work. Similar work has already been done by researchers before. There is a need to rewrite the manuscript, as it fails to convey the theme of work.	We have added some explanations regarding this. We have pointed out the novelty of our study. In addition, we have rewrite the manuscript thoroughly and corrected some mistakes.
Why does the manuscript contain two "Results" sub sections.	We have revised the section. The second section should be "Discussion".
There are many ambiguities, grammatical errors, and mistakes in the paper.	We have rechecked the manuscript writing thoroughly and corrected many errors in sentences, including grammar and typo.
Mention BET analysis and N2 adsorption and desorption isotherm in the revised manuscript.	We have added a subsection about this anlaysis.
In section 4.3.1, typo need corrections "The S2 sample has an average pore radius of 95.34 nm, or 9.534 nm,"	We have corrected this typo.
In manuscript, Section 4.3.2. what does "quantity of holes" refers to.	We have revised the sentence to clear the statement.

Graphical Abstract

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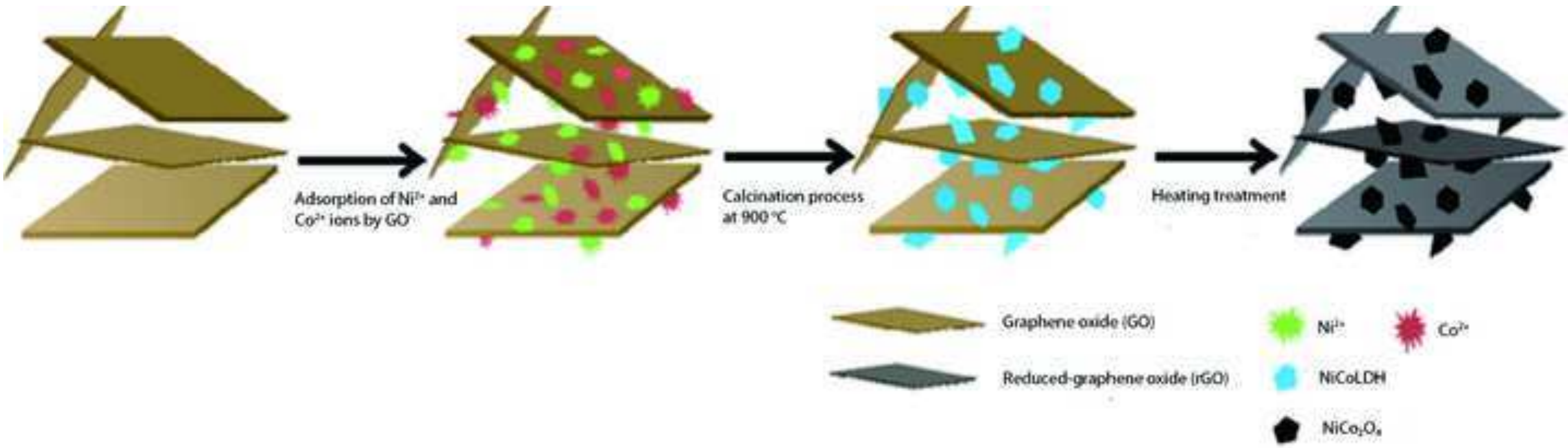

Effect of surface area on electrical properties of $NiCo_2O_4$-reduced graphene oxide nanocomposites for supercapacitor electrodes applications

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ABSTRACT

Based on the method of electrical charge storage, supercapacitors are divided into two categories, double-layer electrical capacitor (EDLC) and pseudocapacitors. Utilizing three processes—reversible adsorption, redox reactions on metal oxides, and reversible electrochemical—pseudocapacitors are utilized for high power applications involving metal oxide electrodes and the transfer of electric charge based on a reversible faradaic. In the fabrication of supercapacitors, a high specific surface area with a relatively narrow pore size distribution is essential. Therefore, it is required to increase the capacitance of the material. In this work, nickel cobaltite ($NiCo_2O_4$) synthesized from nanocomposite $NiS.5H_2O$ and $Co_2SO_4.7H_2O$ precursors were mixed with reduced graphene oxide (rGO). Coprecipitation and calcination were used to create the nanocomposites. The produced $NiCo_2O_4$/rGO nanocomposite was used as a pseudocapacitive supercapacitor electrode. The results showed that sample code S2 with mass variations of NiO, Co_3O_4, and rGO at a ratio of 2:3:2 had the best performance. The sample had a hexahedron-shaped surface morphology, an average particle size of about 0.005 m^2, a specific surface area of 12.75 m^2/g, an average pore radius of 9.534nΩ.m, and a pore volume of 0.06404 cm^3/g.It also performed exceptionally well in terms of electrical conductivity of 6.078 S/m, electrical resistivity of 0.16 nΩ.m, and capacitance of 289.93 F/g.

Keywords: $NiCo_2O_4$/rGO nanocomposite, Electric charge storage, Pseudocapacitor electrode, Specific capacitance, Specific surface area

1. Introduction

As a developing country, Indonesia has a significant need for a variety of resources to advance. This also contributes to the expanding use of electronic components in the energy, transportation, technology, and information technology industries. The increasing use of electronic component materials each year will increase the electricity demand, including electrical energy storage components, causing the development of electrical energy storage component devices to garner a great deal of attention [1]. A supercapacitor is one of the frequently utilized electrical energy storage materials. A supercapacitor or electrochemical capacitor is an electrical double layer that functions as an electrical energy storage device based on charging and discharging at the electrode-dielectric interface [2].Currently, the supercapacitor electrode material is growing as an energy storage materialas it has various advantages. For example, it is maintenance-free, has a longer lifespan, rapid chargeand discharge cycles, and can operate effectively in various environmental conditions[3]. In addition, it has > 100,000cycles, high energy density, extensive energy storage capability, simple principles, and easy construction[4].

Based on the technique of electrical charge storage, supercapacitors are often split into two groups: electrical double-layer capacitors (EDLC) and pseudocapacitors [5]. EDLC capacitors are often utilized in low-power applications [6] using activated carbon electrodes which have a wide surface area for storing electric charge at the electrode/electrolyte interface, such as carbon fiber, carbon aerogel, and carbon paper. The power density and stability of these capacitors are remarkable, but their specific capacitance is low [7]. In contrast, pseudocapacitors are used for high power applicationswith metal oxide electrodes whose electric charge transfer is based on a reversible Faradaic process[8], including reversible adsorption, redox processes on metal oxides, and reversible electrochemical doping on conductive polymers for electrodes [9]. Pseudocapacitors can store electric charge more effectively than EDLC, but their stability is still inferior to that of EDLC. Therefore, this capacitor needs further improvement using redox-active electrode materials and increasing the specific surface area for energy storage applications in hybrid electric cars and household electric devices [10].

Supercapacitors must be designed with a high specific surface area and a narrow distribution of pore size to maximize their capacitance performance [11]. Nickel cobaltite ($NiCo_2O_4$), the electrode substance used in this study, was created by coprecipitating and calcining precursors of $NiS.5H_2O$ and $Co_2SO_4.7H_2O$ with rGO. The spinel $NiCo_2O_4$ possesses superior electrical conductivity and redox activity than single-metal oxides like NiO and Co_3O_4. In addition, $NiCo_2O_4$ is a promising option for high-performance supercapacitors due to its high theoretical capacitance, ease of manufacture, abundant materials, low cost, and eco-friendliness.

NiO has a bandgap of 3.37 eV, a conductivity of 1.4 x 107 S/m, and a resistivity of 69.3 nΩ.m[12]. In addition, NiO is a cheap member of the transition metal oxides whose composites exhibit a mesoporous structure in the form of nanoflake crystals with a large surface area [13] as a result of the nano-dimensional electrochemical double layer mechanism with a confirmed faradic redox reaction [14].This mechanism produces a maximum specific capacitance of 401 F/g at a current density of 0.5 mA/cm^2[15].Meanwhile, Co_3O_4 has an energy gap between 2.8 and 2.2 eV, a conductivity of 1.6 x 10^7 S/m, and a resistivity of 62.4 nm[16], with an electron mobility of 200,000 cm^2/Vs, a specific surface area of 26,300 m^2/g, an intrinsic electrochemical capacitance of 21 mF/cm^2, a specific capacitance of 220 F/g, a hexagonal structure with a strength of 42 N/m, and a thermal conductivity of 5,000 W/mK. Furthermore,in addition to these characteristic, the presence of other materials, such asrGO with chromophoric properties may enable Co_3O_4 to quickly absorb free electrons [17].

In order to understand how $NiCo_2O_4$ and rGO affect surface morphology, microstructure, pore size distribution, and the link between the specific surface area and the electrical properties of the supercapacitors generated, this study synthesizes and evaluates $NiCo_2O_4$/rGO nanocomposites.In addition, this study reported thermal, chemical, and electrical properties in order to explore suitability of the synthesized materual for use as supercapacitor electrodes.

2. Materials and methods

4.1. Materials

Natural graphite powder, nickel sulfide pentahydrate ($NiS.5H_2O$), cobalt sulfate hydrate ($Co_2SO_4.7H_2O$), sulfuric acid (H_2SO_4), zinc powder (Zn),nickel nitrate ($Ni(NO_3)_2$),hydrogen peroxide (H_2O_2), aquabidest (H_2O), urea (CH_4 N_2 O), cobalt nitrate ($Co(NO_3)_2$), hydrochloric acid (HCl), deionized (DI) water,sodium nitrate ($NaNO_3$), citric acid ($C_6H_8O_7$),potassium permanganate ($KMnO_4$), and sodium hydroxide (NaOH)were obtained from Merck. All chemicals are of analytical grade.

4.2. Experimental

2.2.1. Synthesis of rGO

The modified Hummers' methodwas used to synthesis rGO. As many as 2 g of graphite powder were dissolved in 98 mL of H_2SO_4 and 4 g of $NaNO_3$ while being stirred for an hour. After stirring for two hours, 8 g of $KMnO_4$ was gradually added to the mixture. Four hours were spent gently stirring the mixture in an ice container at a temperature between 0 and 20 °Cuntil it turned greenish black. At 35 °C, the mixture was agitated for 20 hours until a light brown tint emerged. After beingstirred for an hour, the mixture was then washed with 200 mL of aquabidest. Once pH 7 was achieved, the mixture was centrifuged and repeatedly rinsed with 80 mL HCl and deionized water. Following that, the mixture was dried for 12 hours at 110 °C to produce sheets of GO. The next phase involved adding 40 mg of GO to 40 ml of DI water, stirring it for an hour, and then ultrasonifying it for 1.5 hours at 50/60 Hz. After that, GO was lowered by stirring 0.8 g of zinc powder with 10 mL of strong HCl for an hour. The mixture was added to 10 mL of concentrated HCl and stirred for an additional 30 minutes before being repeatedly rinsed with DI water and 5% HCl until the pH was 7. A dry precipitate of rGOwas created by heating the precipitate from the washing process for 18 hours at 160 °C in a tiny stainless steel and Teflon tube.

2.2.2. Preparation of $NiCo_2O_4$nanoparticles

NiS.5H_2O and Co_2SO_4.7H_2O were combined to form $NiCo_2O_4$ nanoparticles via coprecipitation, with a mole ratio of 1:2 (Ni^{2+}:Co^{2+}).NiS.5H_2O (1.188 g) and Co_2SO_4.7H_2O (2.701 g) were dissolved in separate 20 mL of aquabidest. The solution was added dropwise to 50 mL of NaOH solution (precipitation agent) and stirred with a magnetic stirrer at 1000 rpm for one hour. The fluid was then put on a permanent magnet to speed up the deposition process.In order to get rid of any leftover salts from earlier operations, the precipitate was then rinsed seven times with DI water. After being heated in an oven at around 90 °C, the precipitate produced a black powder. The whole process is characterized by the following chemical reactions:

$$NiS.5H_2O + 3Co_2SO_4.7H_2O \rightarrow NiO + 2Co_3O_4 + 4H_2SO_3 + 22H_2O \quad (1)$$

$$2Co(NO_3) + Ni(NO_2)_2 + GO + OH^- \rightarrow NiCo_2O_4 + rGO + 7NO_2 + ½H_2O \quad (2)$$

2.2.3. Fabrication of $NiCo_2O_4$/rGO nanocomposites

This nanocomposite is fabricated using rGO, $Co(NO_3)_2$, and $Ni(NO_3)_2$ as precursors. As many as 40 mg of rGO was added to 40 mL of DI water, and the mixture was stirred for an hour. $Ni(NO_3)_2$ and $Co(NO_3)_2$ powder were doped with rGO powder in various amounts (see Table 1) after two hours of agitation at 180 °C [18]. The mixture was stirred while ureaand citric acid were added. The samples were then rinsed with DI water at a pH between 6.8 and 7. The washing procedure precipitate was placed in a tiny Teflon tube within a stainless-steel tube and burnt for 18 hours at 160°C. Following a two-hour calcination at 900 °C, the resulting powder was stored. Fig. 1 shows a schematic depiction of the production process of $NiCo_2O_4$/rGOnanocomposite.

Table 1

Variations in the composition of $NiCo_2O_4$/rGO nanocomposites.

Sample code	Composition of NiO:Co_3O_4:rGO

	(g:g:g)
S1	2:2:2
S2	2:3:2
S3	3:2:2

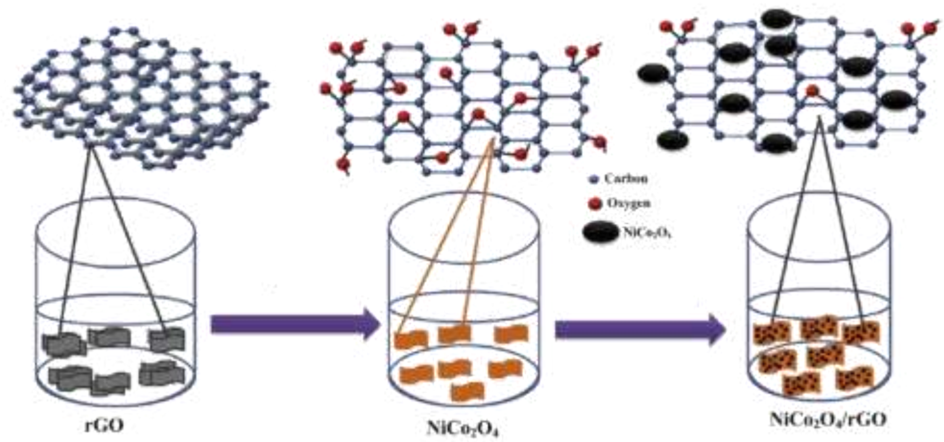

Fig. 1. The synthesis processof $NiCo_2O_4$/rGOnanocomposite.

2.2.4. Characterizations of the$NiCo_2O_4$/rGO nanocomposites

$NiCo_2O_4$/rGO nanocomposites were analyzed microscopically using SEM (Scanning Electron Microscope, JEOL JSM-5310) by shooting electrons at 15 kV, at 50,000× magnification. Based on nitrogen adsorption-desorption measurements, the BJH (Barrett-Joyner-Halenda) and BET (Brunauer-Emmet-Teller) characterization were performed using the Sorption Analyzer NOVA 1000. Based on the N_2 adsorption-desorption isotherm study, this was performed at 77 K and a relative pressure of 0.05-0.30 P/P_0. Lastly, the electrical characteristics of graphene samples were evaluated using a CV meter, GW-Instek LCR 816, at a frequency of 1-300 kHz and an RC circuit with current propagation at four places probes.

3. Results

Analyses of the surface morphology and microstructural properties of the synthesized $NiCo_2O_4$/rGO, Co_3O_4/rGO, NiO/rGO, pure Co_3O_4, and pure NiOwere conducted using SEM at an increasing voltage of 15 kV and a magnification of 50,000×, as presented in Fig. 2.

Based on Fig.2a, 2b, and 2c, the surface morphology of $NiCo_2O_4$/rGO nanocomposites in samples S1, S2, and S3, shows $NiCo_2O_4$ nanoparticles with a diamond-like hexahedron morphology on a thin transparent sheet (nanosheet) with a multilayer structure. There is folding of the rGO layer in the form of nanoflakes so that it appears thicker like large granules or clumps on the surface [19]. In addition, the presence of crystal growth such as the formation of small nano-sized needles (nanoneedles) that accumulate on the nanoplate resulting from the hydrothermal process or calcination [20].

The illustration also depicts thecomparison of Co_3O_4/rGO nanocomposite (Fig. 2d) and pure Co_3O_4 (Fig. 2f) without the addition of NiO, which resemble tiny, nearly spherical lumps that are homogenous and grouped in a random manner. Meanwhile, NiO/rGO nanocomposite (Fig. 2e) and pure NiO (Fig. 2g) generated a nanoflower-like crystal structure from agglomerated NiO nanoparticles on the surface of the nickel foam substrate.

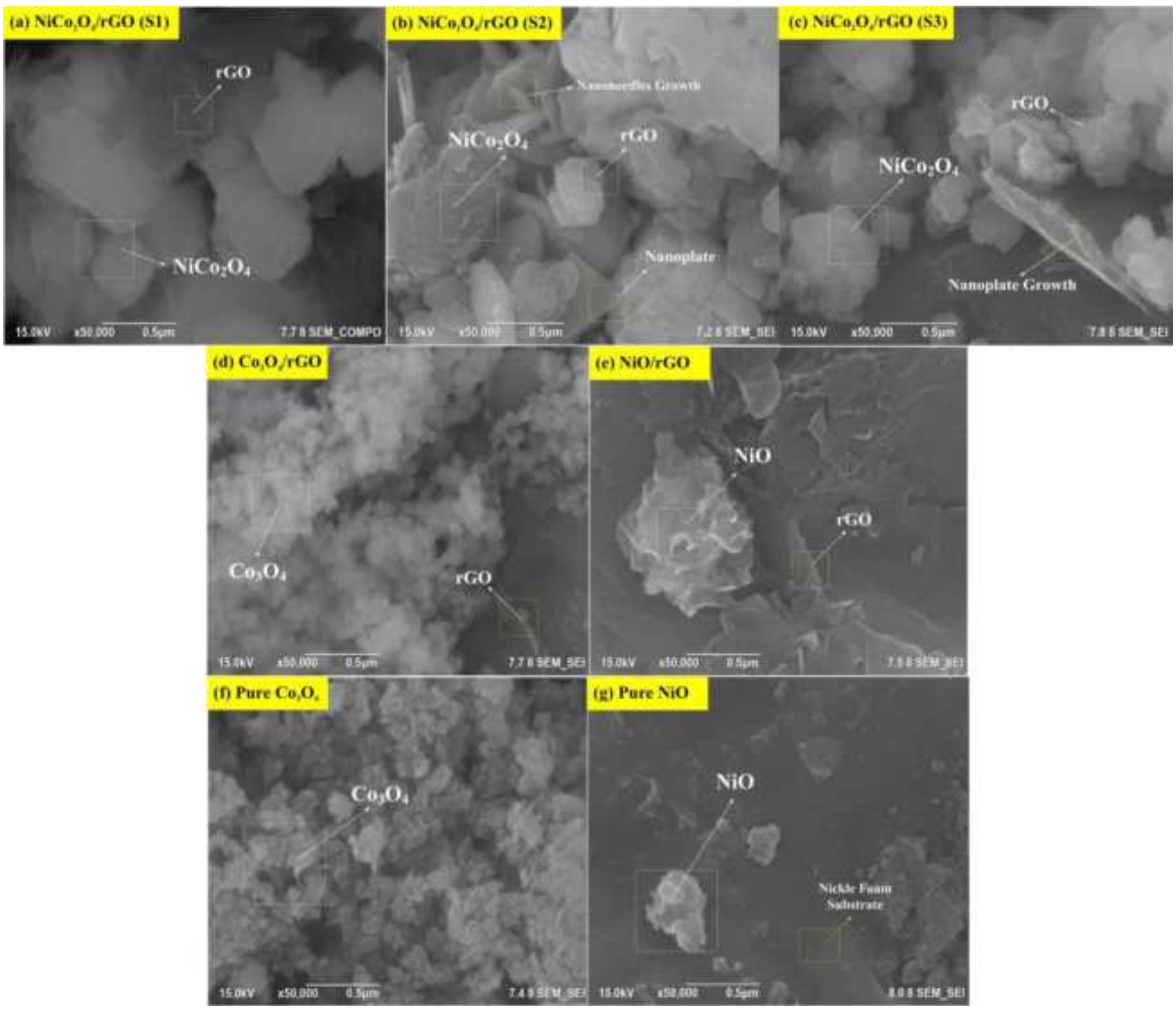

Fig. 2.SEM micrograph analysis of $NiCo_2O_4$/rGO nanocomposites with varying NiO:Co_3O_4:rGO compositions: (a) S1 (2:3:2), (b) S2 (3:2:2), and (c) S3 (2:2:2) % wt, as well as SEM analysis of (d) Co_3O_4/rGO, (e) NiO/rGO, (f) pure Co_3O_4, and (g) pure NiO.

Table 2 summarizes the results of quantitative testing of the nanocomposite surface area using *Image-J Software* analysis based on the crystal structure size distribution on the material surface.

Table 2

Average surface area of nanocrystal structure in nanocomposite.

Types of nanocrystal form	Nanocrystal size in nanocomposites		
	Average length (μm)	Average width (μm)	Average surface area (μm^2)
Hexahedron on the structure of $NiCo_2O_4$	1.112	1.135	0.005
Elongated cylinders like tiny needlesinserted in nanoplate on $NiCo_2O_4$/rGO	0.089	0.089	3.166×10^{-4}
Nanoflower crystal structure of NiOnanoparticles and NiO/rGO	1.022	0.785	0.004
Uniform rounded lumps of Co_3O_4/rGO and Co_3O_4	0.424	0.322	0.001

The BET test was performed to evaluate the surface area of active absorption in collecting and binding free electrons during the surface contact interaction process [21] of the $NiCo_2O_4$/rGO nanocomposite when it was charged to produce a type IV isothermal curve according to the IUPAC classification. Fig.3 presents the results of BET test for S1 and S2.Meanwhile, the BJH characterization of the average pore size of $NiCo_2O_4$/rGO nanocomposite as a pseudocapacitive supercapacitor electrode material is presented in Table 3.In the study of BET and BJH, we only conducted tests on S1 and S2 because the objective of this characterization was to determine whether the material synthesized was mesoporous. In addition, based on the results of the specific capacitance test, S2 exhibits the best results among the other samples, which, according to the analysis, is influenced by the abundance of Co_3O_4. Therefore, as a comparison, S1 was choosen since it had difference composition on Co_3O_4compared to that in S2.

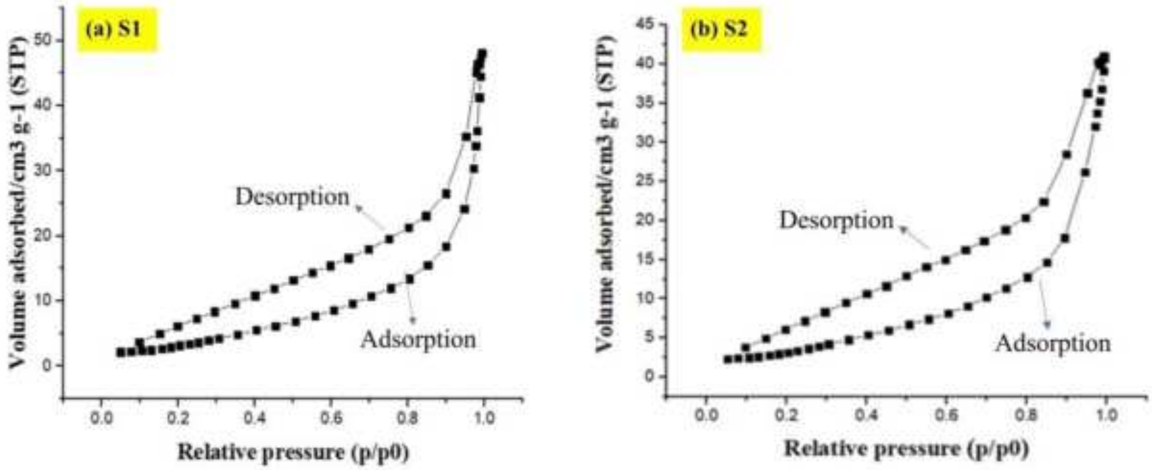

Fig.3. BET analysis on $NiCo_2O_4$/rGO nanocomposite (S1 and S2) with N_2adsorption-desorptionisotherm reaction.

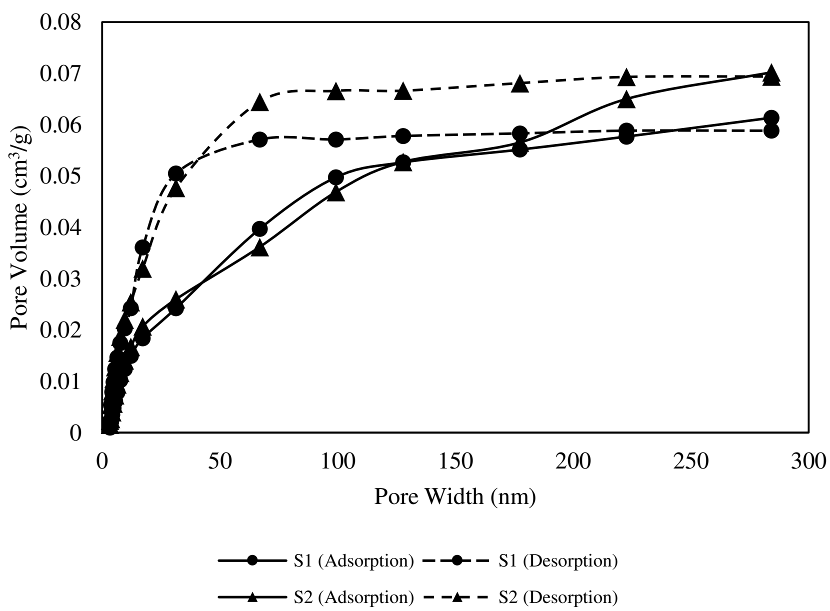

Fig. 4. BJH pore size distribution plot of S1 and S2

Table 3

Average specific surface area, average pore radius, and total volume of $NiCo_2O_4$/rGO nanocomposite samples

Sample code	Average specific surface area (m^2/g)	Average pore radius (Å)	Average pore volume (cm^3/g)
S1	12.90	109.77	0.07455

S2	12.75	95.34	0.06304

Electrical conductivity was analyzed since it is an important factor in storing and distributing electric charge on a polarized pseudocapacitor, which produces a potential difference between the electrodes and the solid electrolyte [22].Fig.5depicts the results of electrical conductivity analysis. In addition, the average electrical conductivity and resistivity of the nanocomposites are shown in Table 4.

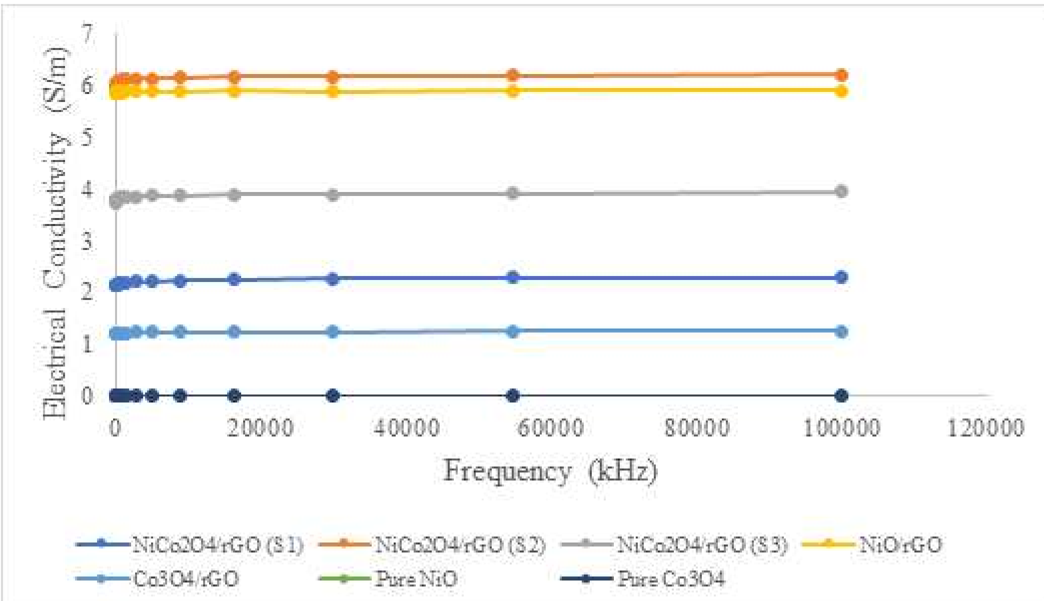

Fig. 5. Analysis of electrical conductivity from the samples in many frequencies.

Table 4

Average electrical conductivity and electrical resistivity of samples.

Sample	Average electrical conductivity (S/m)	Average electrical resistivity (nΩ.m)
S1	2.190	0.46
S2	6.078	0.16
S3	3.825	0.26
NiO/rGO	5.871	0.17
Co_3O_4/rGO	1.232	0.81
Co_3O_4	2.85×10^{-3}	350.88
NiO	1.44×10^{-3}	694.44

Specific capacitance affects the number of electrons that can be stored under pressure exerted by an electric current through a redox reaction or the ratio of the number of polarized charges per potential change[23]. The capacitance measurement was conducted utilizing a three-electrode impedance technique setup in an RC circuit. Table5 displays the results of specific capacitance testing performed using the RC circuit-based impedance method. In addition, the following impedance equationswere be used to obtain the capacitance value[24]:

$$Z = \frac{R}{1+\left(R^2 \omega^2 {C_{RC}}^2\right)} \quad (1)$$

$$C = \frac{I.t}{\Delta V} \quad (2)$$

$$C_{total\ pseudocapacitor} = C + C_{RLC} \quad (3)$$

$$J = \frac{I}{A} \quad (4)$$

$$E\ (\frac{Wh}{kg}) = \frac{Csp\ x\ \Delta V^2}{7.2} \quad (5)$$

$$P\ (\frac{W}{kg}) = \frac{E\ x\ 3600}{t} \quad (6)$$

where Z denotes the $NiCo_2O_4$/rGO nanocomposite's impedance(Ω), R denotes the RC circuit's resistance, ωis the wave propagating angular frequency (rad/s)through the sample equal to 2πf, and C is the capacitance value of the sample (Farad).

Table 5

Specific capacitance, current density, energy density, and power density of samples, as determined by characterization.

Sample	Specific capacitance (F/g)	Current density (A/m^2)	Energy density (Wh/kg)	Power density (W/m^3)
$NiCo_2O_4$/rGO (S1)	106.69	91.84	0.0760	273.5467
Ni Co_2O_4/rGO (S2)	289.93	106.32	0.1573	566.3885
Ni Co_2O_4/rGO (S3)	182.59	96.51	0.1094	393.6968
NiO/rGO	277.02	105.87	0.0726	261.4843
Co_3O_4/rGO	39.77	68.89	0.0021	7.624335
Co_3O_4	0.11	0.44	0.0018	6.4440
NiO	0.06	0.21	0.0036	12.8010

According to faradaic theory, the specific surface area of the $NiCo_2O_4$/rGO nanocomposite is inversely proportional to its specific capacitance[25]. Numerous variables affect the specific surface area of $NiCo_2O_4$/rGO nanocomposites, including pore size distribution, volume, and particle diameter. By modifying the surface area of the pore structure, specific capacitance was increased. The relationship between specific capacitance and specific surface area is presented in Fig.6.

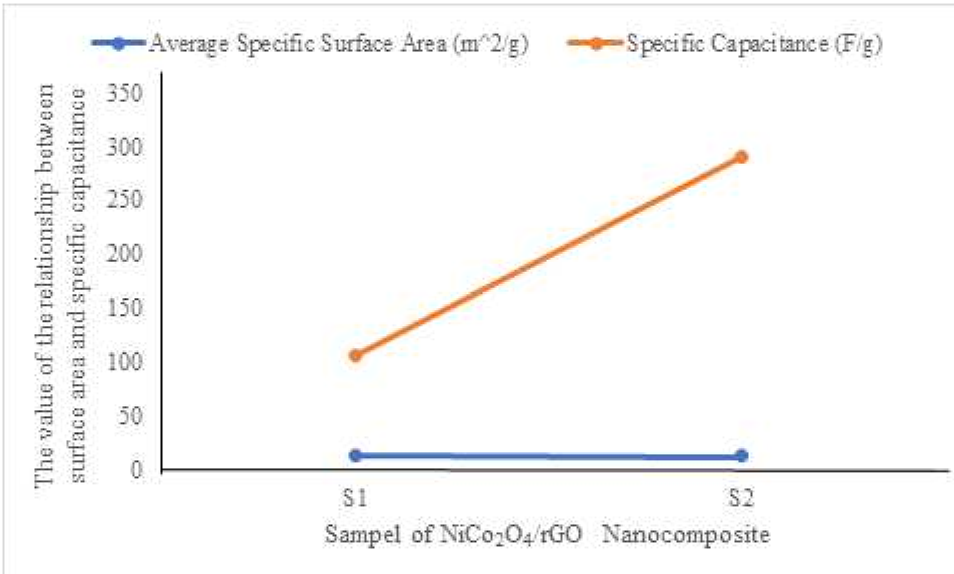

Fig. 6. Effect of specific surface area on the capacitance of $NiCo_2O_4$/rGO nanocomposites.

4. Discussion

4.1. Surface morphology analysis

Fig. 2 shows the micrographs of $NiCo_2O_4$/rGO nanocomposites produced by coprecipitation and hydrothermally at a calcination temperature of 900ºC. They indicate that the rGO layer, which is present in the bulk, is folded and that the $NiCo_2O_4$ nanoparticles have a diamond-like hexahedron morphology. Crystals are formed during the hydrothermal process of calcination, as shown in the aggregation of nanoneedles on the nanoplate.

4.2. Specific surface area and pore size distribution based on BET and BJH analyses

The isotherm reaction process demonstrates the unique behavior of the pores[26]in absorbing and releasing dinitrogen gas molecules (N_2)with a smaller relative pressure range (P/P_0) for samples S1 and S2 of 0.1-0.95 P/P_0 and 0.12-0.99 P/P_0, respectively. This shows that the existence of mesopores (2nm $< d <$50nm) may be the result of detached or loose $NiCo_2O_4$/rGO nanocomposite sheets on the nanoparticle stack, leading to pore gaps[19].

The $NiCo_2O_4$/rGO nanocomposites of S1 and S2 have average pore radii of 10.977nm and 9.534nm, respectively. These pore size distribution ranges, namely between 9 and 11 nm, are best in mesoporous structure since the diffusion of active species (DOS) reaction on the supercapacitor electrode materialcan increase the electrical charge storedwith these pore size distribution [27].In addition, the pore volumes of BJH desorption for S1 and S2 are 0.00745 and 0.6304 cm^3/g, respectively, and their specific surface areas are 12.90 and 12.75 m^2/g, respectively.The specific surface area is crucial since it may enhance the contact interaction between the electrode and electrolyte as well as the electroactive properties by reversibly increasing the redox reaction between the electrolyte and the surface of the electroactive electrode.

4.3. Electrical properties

4.3.1 Electrical conductivity and resistivity

The results indicated that S2 has the highest electrical conductivity (6.078 S/m) and the lowest electrical resistivity (0.16 nΩ.m) compared to others. The degree of the resistivity of the compounds that make up the $NiCo_2O_4$/rGO nanocomposite, which is produced by the formation of crystal defects during manufacturing, affects the material's electrical conductivity. This leads awider distribution of holes on the surface of the nanocomposite particles, which prevents free electrons from being excited [28].

Based on the results (see Table 4 for S1, S2, and S3(, electrical conductivity of the samples rises along with Co_3O_4bulk. This is due to the presence of cobalt ions in cobalt oxide, which have electrical resistivities of 62.4nΩ.m, conductivities of 1.6 x 10^7 S/m, and bandgap energies of 2.8-2.2 eV. As a result, these conductor materials increase the electrical conductivity and electrocatalytic activity of nanocomposites during the redox reaction process [29]. The $NiCo_2O_4$/rGO nanocomposites causes Co_3O_4 nanoparticles to entirely dissolve, resulting in nanoparticles with perfect segmentation, low electrical resistance, high electrical conductivity, and fewer holes that might speed up the movement of free electrons.

4.3.2 Specific capacitance

The S2 sample have the highest specific capacitance, 289.93 F/g, as shown in Table 5. This suggests that increasing the bulk concentration of Co_3O_4, which carries the charge of the Co^{2+} ion, may result that increasing the specific surface area, and finally the quantity of holes to store free electrons also increase. The mobility and oscillation of free electrons are improved when an electric field with bandgap energy is applied to the supercapacitor electrode. Free electron adsorption takes place above the bandgap of the $NiCo_2O_4$/rGO nanocomposite, producing electron-hole pairs that may augment the electric current with a density of 106.32 A/m^2, an energy density of 0.1573 Wh/kg, and a power density of 566.3885 W/m^3.

The specific capacitance of the $NiCo_2O_4$/rGO nanocomposite supercapacitor rose with an increase in current density, energy density, and power density due to the supercapacitor electrode particles' capacity to disseminate electrolyte ions in the electrode microspores [30]. It also results from the performance behavior of the $NiCo_2O_4$/rGO nanocomposite type supercapacitor electrodes in alkaline electrolytes during exposure to electric charge. This is influenced by an electrochemical process involving redox reactions that lead to changes in the valence electrons of Co^{3+}/Co^{4+} and M^{2+}/M^{3+} (M = Co or Ni) on the surface of the nanocomposite electrode, making the Faradaic reaction more reversible[31]. This phenomenon can be illustrated in Fig. 6.Fig. 6 depicts the reaction to generate current density when cations (H^+) in a solid electrolyte produce a single layer that is adsorbed on the surface of a nanocomposite electrode with a higher redox potential, such as Co^{3+}, which forms a reduced ion diffusion bridge (OH^-) and results in ion transfer due to the Faradaic process and oxidation of Co^{4+} elements in $NiCo_2O_4$/rGO.

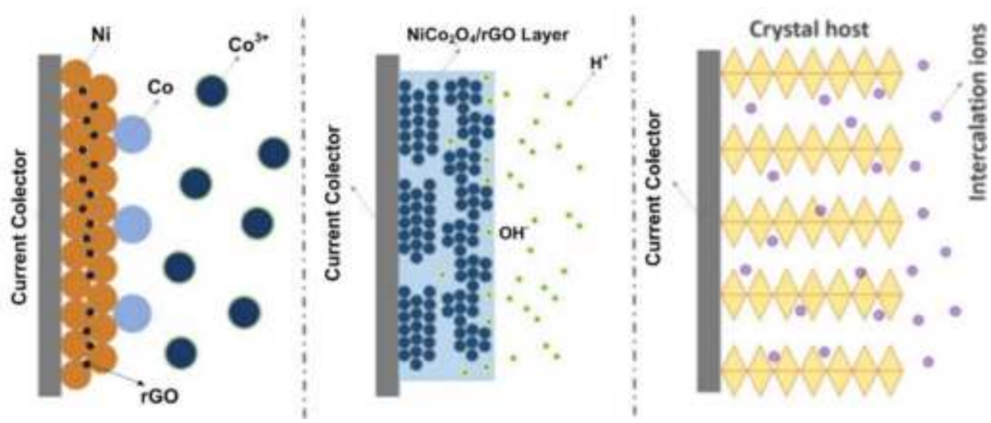

Fig.7. Faradaic reaction process on reversible pseudocapacitance in storing electric charges.

Based on the Faradaic reaction, the chemical reaction equation that happens in $NiCo_2O_4$/rGO nanocomposite cells with ideal composition separated by electrolyte-soaked electron configuration can be described with the following reaction [32]:

Cell A: $NiCo_2O_4 \rightarrow$ rGO || H_2SO_4 (1 M) || $NiCo_2O_4 \rightarrow$ rGO (3)

Cell B: $NiCo_2O_4 \rightarrow$ rGO || KOH (6M) || $NiCo_2O_4 \rightarrow$ rGO (4)

In the RC circuit containing the electrolyte solution, the redox reaction process denoted by reaction3 and 4 is a flow of excited free electrons from the positive electrode to the negative electrode, which produces a stored electric current.

4.4. Relationship between specific surface area and specific capacitance

$NiCo_2O_4$/rGO nanocomposites exhibit a nonlinear relationship between capacitance and surface area at all current, energy, and power densities. Due to the comparable material characteristics and pore size distribution of $NiCo_2O_4$/rGO nanocomposite, in which the effective adsorption surface area and ion transport channel grow linearly with increasing specific surface area, the capacitance of the electric pseudocapacitor increases. Based the correlation between specific surface area and specific capacitance, the little change in specific surface area significantly affect the specific capacitance. This is in accordance with a study by Chmiola et al.[33], which indicated that the little increase in volume pores smaller than 2 nm will significantly increase specific capacitance.

5. Conclusion

$NiCo_2O_4$/rGO nanocomposite, which was synthesized through coprecipitation and hydrothermal methods and used as a pseudocapacitive type supercapacitor electrode, resulted in the optimal composition for sample S2 (NiO:Co_3O_4:rGO = 2:3:2)compared to other samples (S1 and S3). This nanocomposite (S2) produces a hexahedron surface morphology with an average particle size of approximately 0.005 μm^2, a specific surface area of 12.75 m^2/gr, an average pore radius of 9.534 nm, and a pore volume of 0.06304 cm^3/g. In adition, S2 shows the best performance based on the analysis of electrical properties with high electrical conductivity value of 6.078 S/m, while commonly, standard supercapacitor electrode type pseudocapacitor is between 0.1-1 S/m. Furthermore, the electrical resistivity of S2 is 0.16 nΩ.m, which is the lowest (best) among others (S1 and S3). The capacitancevalue ofS2 is the highest, whic is 289.93 F/g, while generally,the standard pseudocapacitor type for $NiCo_2O_4$ is 120 F/g.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT author statement

Effect of surface area on electrical properties of $NiCo_2O_4$-reduced graphene oxide nanocomposites for supercapacitor electrodes applications

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