Afyon Kocatepe Üniversitesi Fen ve Mühendislik Bilimleri Dergisi
Afyon Kocatepe University – Journal of Science and Engineering
https://dergipark.org.tr/tr/pub/akufemubid

Araştırma Makalesi / Research Article
e-ISSN: 2149-3367 DOI: https://doi.org/10.35414/akufemubid.1744934
CITE: Afyon Kocatepe University Journal of Science and Engineering 26(3) (2026) 727-734

Evaluation of Supercapacitive Performance of Ni Nanoparticle-Decorated Flexible Graphene Paper Electrodes

*Makale Bilgisi / Article Info
Alındı/Received: 17.07.2025
Kabul/Accepted: 07.02.2026
Yayımlandı/Published: 03.06.2026

Ni Nanopartikül-Dekore Esnek Grafen Kağıt Elektrotların Süperkapasitif Performansının Değerlendirilmesi

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Abstract

Supercapacitors (SCs) are one of the most extensively investigated energy storage applications in recent years, especially since their working mechanism is based on eco-friendly electrochemical methods. In this study, graphene papers (GP) decorated with nickel nanoparticles (NiNPs) for the development of flexible SCs electrodes were developed as flexible electrodes and the SCs performance of this electrodes (NiNPs/GP) were evaluated. For this purpose, the GP, fabricated with the practical mold-casting method, was decorated with NiNPs via electrochemical method. Thus, the NiNPs/GP flexible electrodes were obtained with a practical approach. The morphological and structural characterization of the obtained electrodes were carried out using scanning electron microscope, X-ray photoelectron spectrometer, and X-ray diffraction techniques. The supercapacitive performances of the prepared flexible and free-standing NiNPs/GP electrodes were investigated with cyclic voltammetry and galvanostatic charge/discharge methods. The specific capacitance (Cs) value of the NiNPs/GP electrode was measured to be 414 F/g at a current density of 1.0 A/g. Furthermore, the long-term usage studies such as flexibility and cycle stabilities of the NiNPs/GP electrode were also performed. As a result of these studies, it was determined that the Cs value of flexible and free-standing electrode decreased by only 19.7% and 16.1% after 500 bending cycles and 2000 charge/discharge cycles, respectively. The excellent supercapacitive performance of the flexible, free-standing electrode could be attributed to the flexibility of the graphene paper structure and the synergistic interaction between the graphene and the NiNPs.

Keywords: *Graphene paper; Flexible electrode; Nickel nanoparticles; Supercapacitors; Flexible supercapacitors.*

Öz

Süperkapasitörler (SC), özellikle çalışma mekanizmalarının çevre dostu elektrokimyasal yöntemlere dayanması nedeniyle son yıllarda en çok araştırılan enerji depolama uygulamalarından biridir. Bu çalışmada, esnek SCs elektrot geliştirmeye yönelik nikel nanopartiküller (NiNPs) ile dekore edilmiş grafen kağıtlar (GP) esnek elektrot olarak üretilmiş ve bu elektrotların (NiNPs/GP) SCs performansları ortaya konulmuştur. Bu amaçla uygulaması pratik kalıp döküm yöntemi ile hazırlanan GP, elektrokimyasal yöntemle NiNP'lerle dekore edilmiştir. Böylece pratik bir şekilde NiNPs/GP esnek elektrotları elde edilmiştir. Elde edilen elektrotların morfolojik ve yapısal karakterizasyonu, taramalı elektron mikroskobu, X-ışını fotoelektron spektroskopisi ve X-ışını kırınımı teknikleri kullanılarak gerçekleştirilmiştir. Hazırlanan esnek ve serbest duran NiNPs/GP elektrotların süperkapasitif performansları dönüşümlü voltametri ve galvanostatik şarj/deşarj yöntemleri ile araştırılmıştır. NiNPs/GP elektrotun spesifik kapasitans (Cs) değeri 1,0 A/g akım yoğunluğunda 414 F/g olarak belirlenmiştir. Ayrıca, NiNPs/GP elektrotun esneklik ve dönüşüm stabilitesi gibi uzun süreli kullanım çalışmaları da gerçekleştirilmiştir. Bu çalışmalar sonucunda esnek ve serbest duran elektrotun 500 kez eğip-bükme sonucunda ve 2000 şarj/deşarj döngüsü sonucunda Cs değerlerinde sırasıyla yalnızca %19,7 ve %16,1 azalma olduğu belirlenmiştir. Esnek, serbest duran elektrotun mükemmel süperkapasitif performansı, GP'ın esnek yapısına ve grafen ile NiNPs arasındaki sinerjik etkileşime atfedilebilir.

Anahtar Kelimeler: *Grafen kağıt; Esnek elektrot; Nikel nanopartiküller; Süperkapasitörler; Esnek süperkapasitörler.*

1. Introduction

The advancement of flexible energy storage systems for devices such as flexible displays, wearable devices, portable electronic devices, electronic sensors, and mobile phones has been actively explored particularly in recent years (Liu *et al.* 2019, Erçarıkcı *et al.* 2023). Flexible energy storage systems are required to possess high energy storage capacity and sustain this performance under bending conditions.

Supercapacitors (SCs), also known as electrochemical capacitors, are promising energy storage systems in energy storage applications due to features including high stability, superior electrochemical performance, and environmental compatibility (Banerjee *et al.* 2025,

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Erçarıkcı *et al.* 2022). The electrode material is crucial in the supercapacitive performance of SCs (Çepni and Öznülüer 2022). At this stage, flexible SC electrodes can be produced by adapting new and superior capacitive materials for use in flexible structures (Yan *et al.* 2022).

Graphene papers (GPs), which are graphene-based materials that are both flexible and exhibit high capacitive performance, can be customized to specific sizes, and can be free-standing and rolled, have been extensively explored for diverse applications in recent years (Hayat *et al.* 2025, Wang *et al.* 2025, Shafiyev *et al.* 2024, Zhou *et al.* 2024, Erçarıkcı and Alanyalıoğlu 2021). Owing to these favorable characteristics, the use of GP as flexible SC electrode material has been investigated with increasing interest in recent years (Mollamehmetoğlu and Alanyalıoğlu 2024, Dağcı Kıranşan and Topçu 2020, Lv *et al.* 2021, Qu *et al.* 2020).

The design of hybrid electrodes exhibiting dual capacitive characteristics (both electrical double layer capacitor (EDLC) and pseudocapacitor (PC) or battery-like capacitor (BC)) properties is regarded as a promising strategy to improve the capacitive performance of SCs (Jiang *et al.* 2018). In this regard, it was focused on enhancing the capacitive performance by decorating GP with structures such as metal sulfides (Dağcı Kıranşan and Topçu 2020), metal oxides (Mollamehmetoğlu and Alanyalıoğlu 2024), and metal nanoparticles (Erçarıkcı vd. 2021) and the capacitive performances of these electrodes were systematically examined.

Metal nanoparticles are considered as a promising candidate for electrochemical applications due to their excellent electroactive properties and chemical stability (Lavanya *et al.* 2023). Nickel nanoparticles (NiNPs) have been investigated as electrode materials in electrochemical studies owing to their high electrical conductivity, high stability, fast electron transfer, economic feasibility, and substantial surface area (Bai *et al.* 2020). It is widely considered that NiNPs enhance the performance of SCs through increasing electrical conductivity and specific surface area, increasing structural stability, facilitating ion diffusion and leveraging synergistic interactions between the materials in graphene-based produced composite electrodes (Pour et al. 2023). A review of the literature reveals, SC performance has been investigated by decorating NiNPs on non-free-standing materials such as nickel foam (Cheng *et al.* 2019, Youssry *et al.* 2023) and flexible substrates like carbon cloth (Valipour *et al.* 2015). However, no report has been identified examining the SC performance of NiNPs decorated GP.

NiNPs can be synthesized by multiple synthesis routes such as hydrothermal method (El-Hout *et al.* 2017), microwave-assisted method (Xu *et al.* 2008), and electrochemical method (Badarulzama *et al.* 2010). Among these methods, electrochemical method is a particularly practical technique that is considered as an eco-friendly method. Electrochemical method provides precisely tailored morphologies and highly crystalline structure in a practical way for the synthesis of NiNPs (Ali Ahmadi Diva *et al.* 2019, Çepni 2024).

This study aims to develop a flexible SC based on a NiNPs/GP electrode and assess its capacitive properties. To this end, GP was fabricated via the mold-casting technique, and then decorated with NiNPs in an electrochemically practical way to obtain a flexible NiNPs/GP electrode. The supercapacitive properties of this flexible electrode were then investigated. Characterization of the produced NiNPs/GP was carried out by various methods such as X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), and field emission scanning electron microscopy (FESEM). The SC performance of the characterized flexible and free-standing NiNPs/GP electrode was investigated using cyclic voltammetry (CV) and galvanostatic charge/discharge (GCD) methods.

2. Materials and Methods

2.1 Chemicals and Materials

All reagents utilized in the study were purchased from Sigma-Aldrich and were of analytical reagent grade purity. Distilled water was employed throughout all experimental procedures requiring aqueous media.

2.2 The fabrication of GP

The modified Hummers method was used to synthesize graphene oxide (GO), the fundamental precursor of GP (Hummers and Offeman 1958, Erçarıkcı *et al.* 2023). A schematic illustration of the production of GP is presented in Figure 1. 40 mL of GO (5.0 mg/mL) dispersion obtained after synthesis was cast into an appropriate mold and dried at room temperature (Erçarıkcı *et al.* 2022). The obtained paper was soaked in 57% (w/w) hydrogen iodide (HI) solution for 1 hour to provide a chemical reduction of GO, and GP was obtained following the washing steps. GP was sectioned into strips (15 mm x 6 mm) each time and used directly as an electrode material in electrochemical studies.

2.3 The preparation of NiNPs/GP

A potential window of 0 to –1.05 V was employed for the electrodeposition of NiNPs onto GP in a Watts-type

electrolyte containing Ni^{2+} ions in a three-electrode electrochemical cell where GP served as the working electrode (Shafiyev *et al.* 2024, Badarulzaman *et al.* 2010) (Figure 1). Through this process, NiNPs/GP was successfully fabricated.

2.4 Instruments and Electrochemical Techniques

The morphological characteristics of the synthesized materials were examined using a ZEISS SIGMA 300 FESEM instrument. XRD patterns were recorded with a Rigaku TTR III X-ray diffractometer with monochromatized Cu Ka radiation (λ = 1.5406 Å), and XPS analyses were conducted with a Spect-Flex spectrometer equipped with a standard Al Kα X-ray source.

Electrochemical measurements were conducted using a Gamry Reference 600+ potentiostat/galvanostat system in a three-electrode electrochemical configuration. CV and GCD experiments were performed to evaluate the supercapacitive performance. In addition, Ag/AgCl (saturated 3.0 M KCl) electrode was used as a reference electrode and a platinum (Pt) wire served as the counter electrode in the electrochemical cell.

The capacitive performance of the fabricated flexible electrodes was assessed in 1.0 M KOH aqueous electrolyte. The specific capacitances of the electrodes were calculated according to the equations given below: (Erçarıkcı *et al.* 2023; Rawat *et al.* 2026):

$$C_{s,CV} = \int \frac{I(V)\Delta V}{m \times \Delta V \times \nu} \quad (1)$$

$$C_{s,GCD} = \int \frac{I \times \Delta t}{m \times \Delta V} \quad (2)$$

In this equation, Cs represents the specific capacitance (F/g); I is the the applied current (A); ΔV is the potential window formed during charging and discharging (V); ν is the scan rate (V/s); Δt is the discharge time (s); and m is the total mass of the active material (g).

3. Results and Discussions

3.1 Structural and Morphological Characterization

A schematic illustration of the fabrication process of NiNPs/GP and its subsequent capacitive performance evaluation is provided in Figure 1. It was demonstrated that the GP prepared practically by the mold-casting method was remarkably flexible. It was observed that the NiNPs/GP electrode produced via electrodeposition retained the intrinsic flexibility of the GP as a result of this process. Subsequently, the supercapacitive performance of the fabricated flexible NiNPs/GP electrode was investigated.

To analyze the morphological properties of the produced paper electrodes, FESEM images were captured, as shown in Figure 2. Upon examining the FESEM image of GP in Figure 2a, the characteristic wrinkled structure of the GP is clearly visible. The decoration of NiNPs on the GP surface resulted in a homogeneous distribution of NiNP structures across the wrinkled surface. This uniform distribution is expected to enhance the surface area of the paper and ultimately improve its capacitive performance.

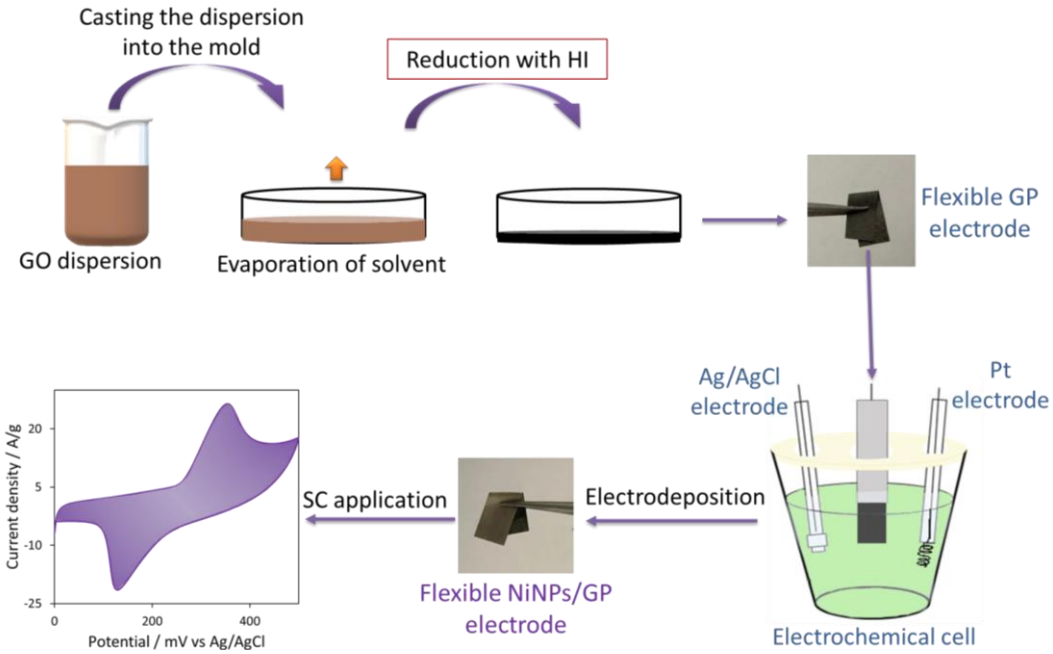


Figure 1. A schematic illustration of the fabrication and the capacitive performance evaluation of flexible and free-standing NiNPs/GP.

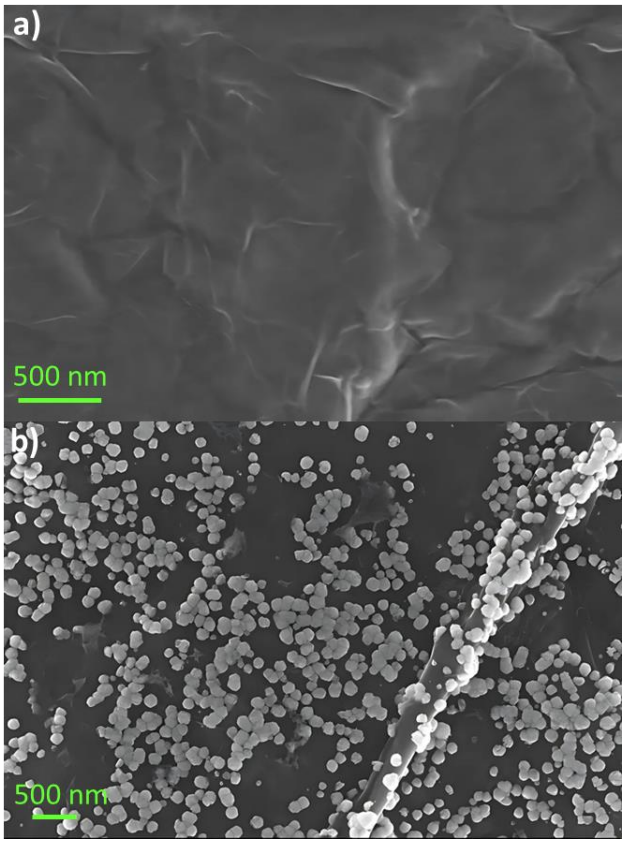


Figure 2. FESEM images of (a) GP and (b) NiNPs/GP.

XRD technique was used to analyze the crystal structure of GP and NiNPs decorated on graphene paper (NiNPs/GP), as shown in Figure 3. Upon examining the XRD spectrum of GP, the characteristic diffraction peak of graphene was observed at 24.5°, corresponding to the (002) plane of the graphitic structure (Jiao *et al.* 2017). For the NiNPs/GP composite, in addition to the graphene (002) peak, diffraction peaks appearing at approximately 44.6°, 52.2°, and 76.8° can be indexed to the (111), (200), and (220) crystal planes of face-centered cubic (fcc) metallic NiNPs (JCPDS card No. 04-0850), respectively (Ganeshchandra Prabhu *et al.* 2015). These findings confirm the successful decoration of NiNPs onto GP.

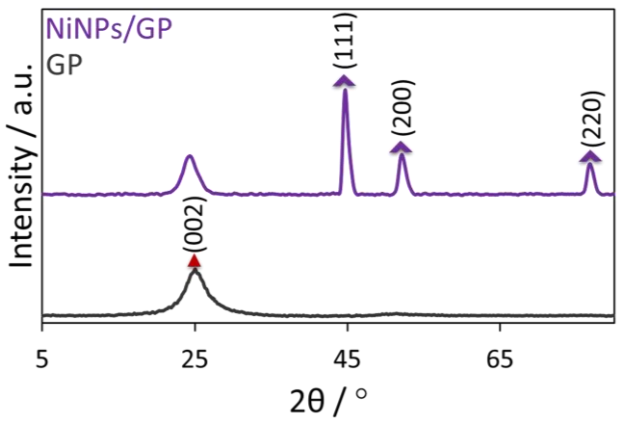


Figure 3. XRD patterns of GP and NiNPs/GP.

The structural characterization of the prepared NiNPs/GP was conducted using XPS analysis, as shown in Figure 4.

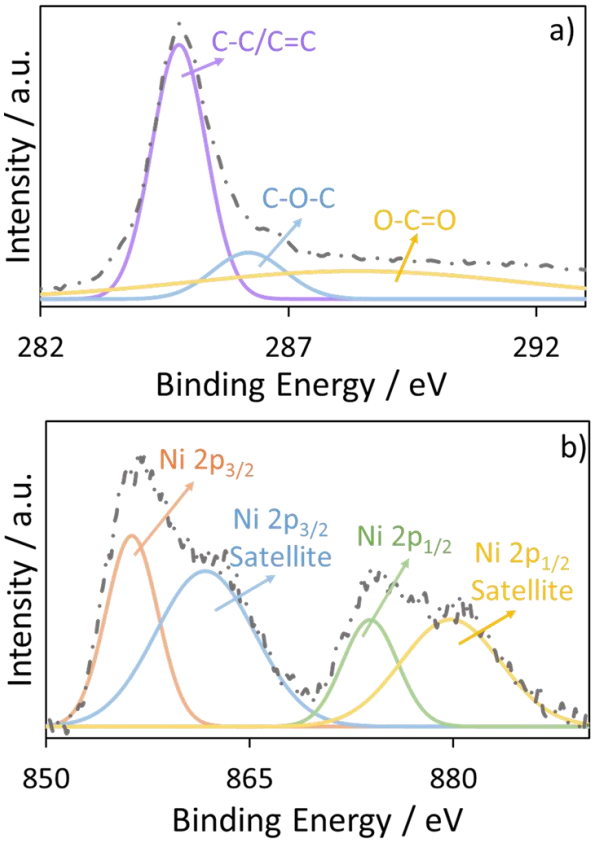


Figure 4. Core level XPS spectra of NiNPs/GP: (a) C 1s (b) Ni 2p.

The C 1s and Ni 2p core level XPS spectra of NiNPs/GP are shown in Figure 4. The fitting of the C 1s peak in the NiNPs/GP spectrum revealed the presence of peaks corresponding to the chemical bond structures C=C, C-O, and O-C=O at binding energies of 284.8 eV, 286.8 eV, and 288.7 eV, respectively (see Figure 4a). Additionally, the core level XPS spectrum of the nickel (Ni) atom in the NiNPs/GP structure is depicted in Figure 4b. This spectrum displays the Ni $2p_{3/2}$ and Ni $2p_{1/2}$ orbitals, along with two satellite peaks at binding energies of 856.8 eV, 874.5 eV, 863.5 eV, and 880.5 eV. It can therefore be concluded that the NiNPs/GP were successfully prepared with the intended chemical composition.

3.2 Capacitive performance of NiNPs/GP

To achieve the SC performance of NiNPs/GP, CV graphs were taken of the paper electrodes that had undergone the electrodeposition process at -1.05 V applied potential for 4, 5, and 6 min (Erçarıkcı *et al.* 2022). The electrochemical performances of these electrodes were compared (Figure 5). Upon examining the CV results, it was found that the highest peak current density was achieved with NiNPs/GP prepared in 5 min. Based on the results, the optimal electrodeposition time was determined to be 5 min.

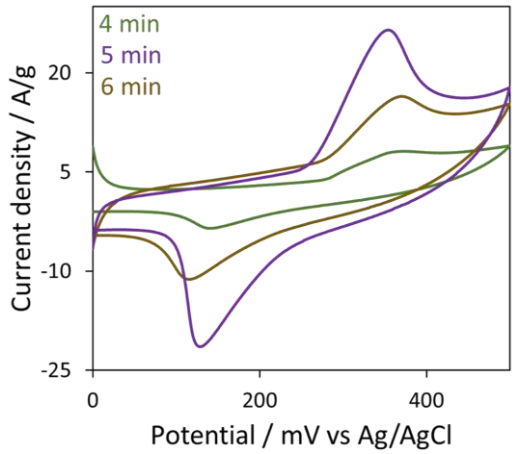


Figure 5. CV graphs of NiNPs/GP prepared using different electrodeposition time.

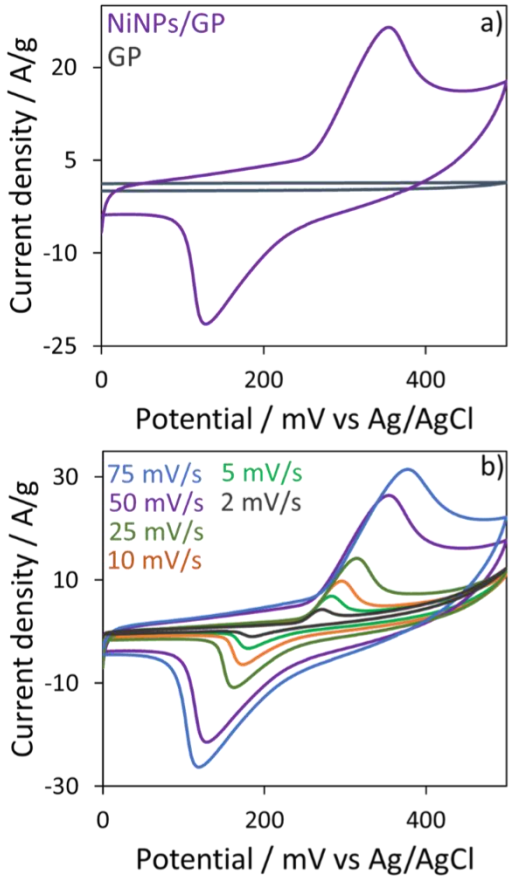


Figure 6. CV graphs of GP and NiNPs/GP in 1.0 M KOH solution at a scan rate of 50 mV/s. (b) CV graphs of NiNPs/GP at different scan rates.

To compare the electrochemical performances of GP and NiNPs decorated GP, CV analyses were performed, and the obtained CVs are presented in Figure 6.a. When the CVs were examined, no peak formation was observed in GP, CV was semi rectangular in shape, and thus it was thought to exhibit EDLC properties (Erçarıkcı *et al.* 2022). When NiNPs were decorated on the GP structure, the electrode was considered to be a hybrid electrode material resulting from a well-defined redox peak pair and the semi rectangular shape provided by the GP (Erçarıkcı *et al.* 2022). These redox peaks were attributed to the reversible transition of Ni^{2+} and Ni^{3+} (Akiyama *et al.* 2022). In Figure 6.b, CVs of NiNPs/GP recorded at different scan rates are presented. Upon examining the CVs, it was observed that the peak current densities of a well-defined pair of redox peaks increased proportionally with the increase in scan rate. The specific capacitance values for the NiNPs/GP electrode at different scan rates were calculated using Equation 1 and are presented in Table 1. Furthermore, the specific capacitance for the NiNPs/GP electrode against different scan rate were plotted and presented in Figure 7. These findings suggest that the capacitive current enhanced due to the faster charge accumulation in the double layer.

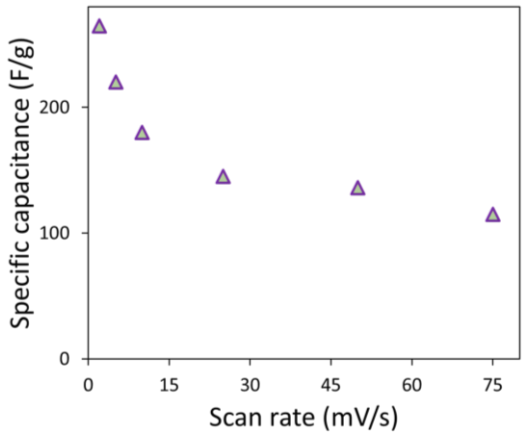


Figure 7. The specific capacitance of NiNPs/GP against scan rate.

Table 1. The specific capacitances (Cs) of the NiNPs/GP is estimated based on the results of CV and GCD measurements.

	Cs (CV) (mV s^{-1})					
Electrode	2	5	10	25	50	75
NiNPs/GP	265	220	180	145	136	115
	Cs (GCD) (A g^{-1})					
Electrode	1	2	4	6	8	
NiNPs/GP	414	255	124	61	36	

The GCD curves of the flexible and free-standing NiNPs/GP electrode at 1, 2, 4, 6, and 8 A/g current density values are presented in Figure 8. It is seen that the charge-discharge time decreases as the current density values increase. This shows that the electrolyte ions intercalate into the pores of the electrode more easily at lower current density values (Xu *et al.* 2019, Erçarıkcı *et al.* 2023). The specific capacitances of the NiNPs/GP electrode at 1, 2, 4, 6, and 8 A/g current density values were calculated as 414, 255, 124, 61, and 36 F/g, respectively (Table 1).

The cycles stability of NiNPs/GP electrode were also investigated. The GCDs of 1st and 2000th cycles for NiNPs/GP were presented in the inset of Figure 8. It was observed that the variation in specific capacitance was minimal. These findings demonstrate the excellent

stability and suitability of NiNPs/GP for prolonged charge/discharge cycles.

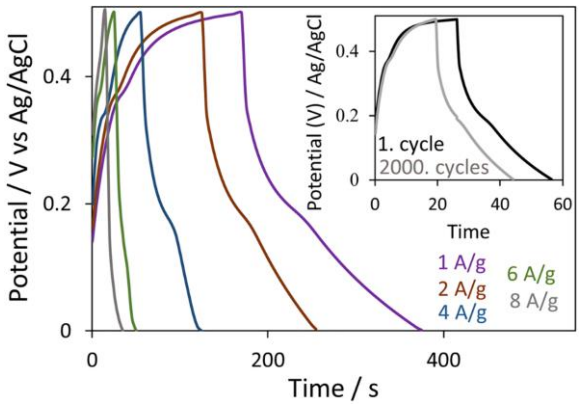


Figure 8. GCD graphs of NiNPs/GP at different current densities. Inset: GCD graphs of NiNPs/GP after 1st and 2000th cycles.

To evaluate the flexibility of the NiNPs/GP electrode in SC applications, the electrode was folded at 180° prior to each measurement. Specific capacitance values were determined using CV graphs at a scan rate of 50 mV/s in 1.0 M KOH solution. Figure 9 presents the percentage retention of the specific capacitance as a function of the number of folding cycles. It was determined that the initial specific capacitance value of the electrode decreased by 19.7% after 500 folding. This finding suggests that flexible NiNPs/GP electrode is suitable for use as flexible electrode material in SC applications.

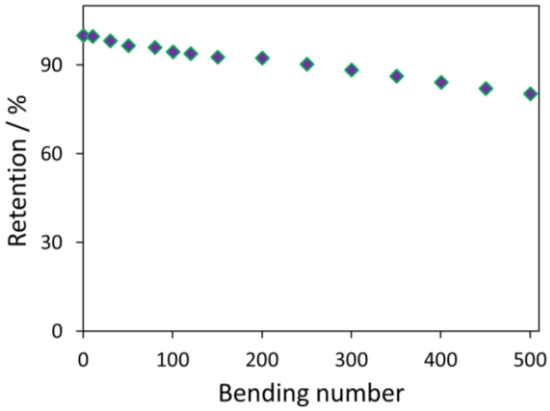


Figure 9. The specific capacitance retention of NiNPs/GP against bending number.

The capacitive performance of GP electrodes prepared with diverse configurations in the literature is compared with the present study in Table 2. To our knowledge, there has been no prior report reporting the SC performance of a GP composite electrode decorated with NiNPs. Table 2 shows that the NiNPs/GP electrode has higher capacitance values and cycling performance, which is attributed to the high electrochemical activity of the Ni atoms in the composite structure and the electrode's enlarged effective surface area.

Table 2. Comparison of capacitance performances of as-prepared NiNPs/GP with various GP electrode materials.

Electrodes	Specific capacitance (F/g)	Cycle number /Change in capacitance	Ref.
GP	174 at 1 A/g	1000 / 86%	Yan *et al.* 2019
GP/MnO_2	410 at 1 A/g	3000 / 68%	Liu *et al.* 2021
NiNPs/GP	414 at 1 A/g	2000 / 83.9%	This work

4. Conclusions

A flexible and free-standing nickel nanoparticles decorated GP (NiNPs/GP) electrode was fabricated using highly practical methods for flexible SC electrode applications. The surface of the GP electrode, prepared via mold-casting, was coated with NiNPs using an electrochemical method to prepare the NiNPs/GP electrode. Structural and morphological characterizations of the NiNPs/GP electrode was performed using various techniques. The SC performance of the produced NiNPs/GP electrode was systematically evaluated. It was determined that NiNPs/GP exhibited higher capacitive performance compared to GP in SC applications. After undergoing 500 cycles of 180° folding, the specific capacitance of the electrode showed only a 19.7% decrease. In light of these results, NiNPs/GP electrode can be considered a promising candidate as a flexible electrode material for SC applications.

Declaration of Ethical Standards

The authors declare that they comply with all ethical standards.

Credit Authorship Contribution Statement

Author-1: Methodology/study design, validation, and investigation.

Author-2: Conceptualization, investigation, formal analysis, data curation, supervision.

Author-3: Conceptualization, investigation, formal analysis, data curation, visualization, supervision, writing – original draft, and writing – review and editing.

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.

Data Availability Statement

All data generated or analyzed during this study are included in this published article.

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