

ENHANCING ENERGY STORAGE PERFORMANCE OF BaTiO₃/PDMS COMPOSITES WITH CNT FILLERS

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Received: July 07, 2024; Revised: December 17, 2024; Accepted: December 29, 2024

Abstract

This study explores the dielectric, energy storage, and piezoelectric properties of BaTiO₃ (BT)/polydimethylsiloxane (PDMS) composites with varying BT concentrations (10-25 vol%) and an additional 1.5 vol% carbon nanotube (CNT) filler. Microstructural analysis confirmed the uniform distribution of BT nanoparticles, while phase characterization using XRD and FT-IR verified the presence of BT with no evidence of new bond formation. Dielectric measurements revealed a significant increase in the dielectric constant with higher BT content, reaching its peak in the 25 vol% BT/PDMS composite with CNTs. However, energy storage analysis showed that the 20 vol% BT/PDMS composite exhibited the highest energy density, indicating an optimal balance between dielectric constant and loss. The addition of CNTs did not significantly enhance energy storage performance due to their increased electrical conductivity, leakage currents, and agglomeration effects. Furthermore, piezoelectric measurements confirmed the piezoelectric behavior of the composites through an alternating voltage response under mechanical force. The CNT-filled composite exhibited a slightly lower voltage amplitude, likely due to charge dissipation and leakage. These results underscore the potential of BT/PDMS composites for flexible energy storage and piezoelectric applications, while also highlighting the limitations of CNT incorporation.

Keywords: Barium Titanate; Dielectric Properties; Energy Storage; Polydimethylsiloxane

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DOI: <https://doi.org/10.55766/sujst9705>

Suranaree J. Sci. Technol. 32(2):030291(1-7)

Introduction

Energy storage technologies are crucial for powering modern devices, ranging from portable electronics to renewable energy systems. Among the various materials explored for enhancing energy storage capabilities, composites of barium titanate (BaTiO₃) and polydimethylsiloxane (PDMS) have attracted significant attention due to their unique combination of properties. BaTiO₃ is a well-known ferroelectric ceramic material celebrated for its high dielectric constant and excellent piezoelectric properties. These attributes make it an ideal candidate for applications in capacitors, actuators, and other electronic devices that require efficient energy storage and conversion. However, despite its impressive dielectric performance, the brittleness and rigidity of BaTiO₃ pose challenges for its use in flexible and lightweight devices. In contrast, PDMS is a flexible, durable, and biocompatible polymer known for its excellent mechanical properties and thermal stability. It is widely used in fields such as microelectronics, sensors, and biomedical devices. Nevertheless, PDMS alone has a low dielectric constant, which limits its direct application in energy storage devices where high dielectric performance is essential. The combination of BaTiO₃ with PDMS offers a promising solution by forming a composite material that harnesses the high dielectric properties of BaTiO₃ and the flexibility and mechanical resilience of PDMS. This composite not only improves energy storage capabilities but also introduces mechanical flexibility, making it suitable for modern applications such as wearable electronics, flexible capacitors, and energy harvesting systems.

In recent years, extensive research has been dedicated to enhancing the energy storage performance of BaTiO₃/PDMS composites. Key challenges, such as limited mass loading of BaTiO₃ and inefficient stress transfer, have been addressed by creating interconnected networks within the polymer matrix. These innovations have significantly improved the dielectric and piezoelectric properties of the composites. Meng *et al.* (2021) explored the dielectric properties and output performance of nanogenerators based on BaTiO₃/PDMS composite films. These composites were fabricated using the spin-coating method with varying concentrations of nanosized BaTiO₃ particles (5-40 wt%) and micro-sized BaTiO₃ flakes. The results demonstrated that the dielectric constant and loss increased significantly with higher concentrations of nanoparticles but decreased with increasing frequency. Specifically, the dielectric constant rose from 6.5 to 8 as BaTiO₃ nanoparticle concentration increased,

while frequencies from 10² to 10⁶ Hz reduced this value. Their study concluded that the electrical output of nanogenerators was enhanced by the synergistic effects of piezoelectricity and triboelectricity. This included (1) triboelectric friction generating positive and negative charges across the composite's surface and (2) piezoelectric potential arising from compression-induced electric dipole moments in the composite. In 2023, Meisak *et al.* (2023) developed flexible, environmentally friendly BaTiO₃/PDMS composites using a simple dispersion method. These composites, containing 200 nm ferroelectric BaTiO₃ particles (7-23 vol%), were studied for their dielectric, piezoelectric, and ultrasonic properties. Similar to Meng's findings, Meisak *et al.* (2023) reported that BaTiO₃ significantly influenced the dielectric properties of the composite. At high temperatures, the dielectric permittivity spectra remained frequency-independent over a wide range, while at low temperatures, an anomaly associated with the dynamic glass transition of PDMS was observed. Under longitudinal stress from a 10 MHz ultrasonic wave, a piezoelectric voltage signal was only detectable in samples with the highest BaTiO₃ concentration (23 vol%) due to low ultrasonic excitation power and the decrease in the piezoelectric coefficient with higher frequencies. Ayadi *et al.* (2024) further investigated the electromechanical coupling factors and piezoelectric figure of merit (FoM) in piezoelectric energy generators (PEGs) with varying BaTiO₃ concentrations. Using a casting process, BaTiO₃-PDMS composite thin films were fabricated, and tensile tests determined their mechanical properties. Experimental results indicated a maximum FoM ($\text{FoM}_{31} = 13.1 \times 10^{-12} \text{ m}^2 \text{ N}^{-1}$) and a power output of approximately 35 nW at a BaTiO₃ concentration of 15 wt%. The study concluded that increasing BaTiO₃ concentrations in PEGs enhances their effectiveness and productivity for diverse applications.

Therefore, based on this literature, the quantity and distribution of BaTiO₃, along with key preparation parameters such as dispersants and fillers, are critical factors in enhancing its efficiency as an energy storage material. Researchers have also investigated the incorporation of various fillers, such as graphene oxide (GO), one-dimensional (1D) and two-dimensional (2D) carbon structures, and TiO₂, to improve energy density and permittivity (Dang *et al.*, 2016; Zhu *et al.*, 2018; Zhu *et al.*, 2021; Bera *et al.*, 2023). Nawani *et al.* (2019) studied the preparation and electrical properties of BaTiO₃/PDMS nanocomposites, demonstrating that

the addition of carbon nanotubes (CNTs) can significantly enhance dielectric permittivity by improving the dispersion and distribution of BaTiO₃ nanoparticles within the PDMS matrix. Similarly, research by Guan *et al.* (2021) and Tang *et al.* (2024) revealed that CNT fillers not only enhance dielectric permittivity but also play a pivotal role in reducing dielectric loss. Building on these findings, this work aims to investigate the effects of CNT fillers in improving the energy storage performance of BaTiO₃/PDMS composites. The study examines the physical properties, phase formation, and chemical characteristics of these composites. The frequency dependence of dielectric properties for all samples is measured using an LCR meter, while hysteresis (P-E) loops are analyzed to calculate the energy density and energy loss density of the materials. Additionally, the piezoelectric properties of the composites are assessed by measuring the output voltage and current generated when mechanical force is applied to the samples.

Experimental Procedure

Polydimethylsiloxane (PDMS) and its curing agent, supplied as SYLGARD™ 184 silicone elastomer from Dow Chemical Company, were used in this study with a mixing ratio of 10:1 (w/w). Barium titanate (BaTiO₃ or BT) nanopowder, with 99.95% purity and an average particle size of 50 nm, purchased from Inframat® Advanced Materials™, was selected as the piezoelectric filler. Initially, BT nanopowder was added to the PDMS matrix at varying concentrations, ranging from 10% to 25% by volume, to investigate the effect of filler content. The mixtures were thoroughly stirred, cast into rectangular molds (3 cm × 3 cm), vacuum degassed at a pressure of 700 mmHg, and cured at room temperature overnight. The physical, chemical, and dielectric properties, including the dielectric constant and dielectric loss, were analyzed for all samples. Physical properties were examined using an optical camera, while chemical properties were studied through Fourier transform infrared (FT-IR) spectroscopy (Spectrum GX spectrometer, PerkinElmer). Phase formation of the composites was characterized by room-temperature X-ray diffraction (XRD; Rigaku SmartLab®) using Cu K α radiation. For electrical measurements, silver paste was applied to both sides of the samples. Dielectric properties were evaluated as a function of frequency at room temperature, with capacitance measured using an LCR meter (HP-4284, Hewlett-Packard Inc.) controlled by a computer system. The dielectric constant (ϵ_r) for all samples was calculated based on their geometric dimensions and thickness.

Polarization hysteresis (P-E) loops were analyzed to determine the energy density and energy loss density of the materials. The piezoelectric properties of the composites were further assessed by measuring the output voltage generated under mechanical force.

To enhance the dielectric performance of these composites, multi-walled carbon nanotubes (MWCNTs) with 99.95% purity and an average diameter of 12 nm, purchased from Nano Generation Co., Ltd., were selected as an additional filler. The MWCNT content was fixed at 1.5 vol% and incorporated into the BT nanopowder mixed with the PDMS matrix under optimal conditions, following the same preparation procedure described earlier. The physical, chemical, and electrical properties of the composites were re-evaluated for comparison. The energy storage performance was assessed through analyses of dielectric properties, energy density, and energy loss density.

Results and Discussion

Polydimethylsiloxane (PDMS) was used as a matrix and combined with 10, 15, 20, and 25 percent by volume of BaTiO₃ (BT) nanopowder. Microstructure and phase investigation for BaTiO₃/PDMS composites are revealed in Figure 1. The cross-sectional morphological SEM images of the 10 and 25 vol% BT composites are shown in Figure 1(a) and 1(b), respectively. SEM imaging was conducted to capture these images and assess the uniformity of particle distribution. For 25 vol% BT/PDMS composite, the image reveals distinct spherical particles, ranging from 5 to 55 μm in size, with an average diameter of $\sim 20 \mu\text{m}$, dispersed within the matrix. These particles, likely barium titanate nanoparticles, appear to be uniformly distributed to some extent, confirming a homogeneous microstructure. The phase composition of all composites, along with their starting components, was analyzed using XRD, as shown in Figure 1(c). The results indicate that only the BaTiO₃ phase is detectable in all composites, as the PDMS phase is non-crystalline and therefore not observable using the XRD technique. FT-IR analysis was performed to further investigate the phase formation of these composites, as shown in Figure 1(d). The FT-IR spectrum of pure PDMS, represented by the red line, displays key transmittance bands at 790 cm^{-1} , 1,012 cm^{-1} , 1,259 cm^{-1} , and 2,964 cm^{-1} . These peaks correspond to CH₃ rocking and Si-C stretching in Si-CH₃, asymmetric stretching of Si-O-Si, CH₃ deformation in Si-CH₃, and asymmetric CH₃ stretching in Si-CH₃, respectively (Johnson *et al.*, 2013; Silva *et al.*, 2016). These bands are

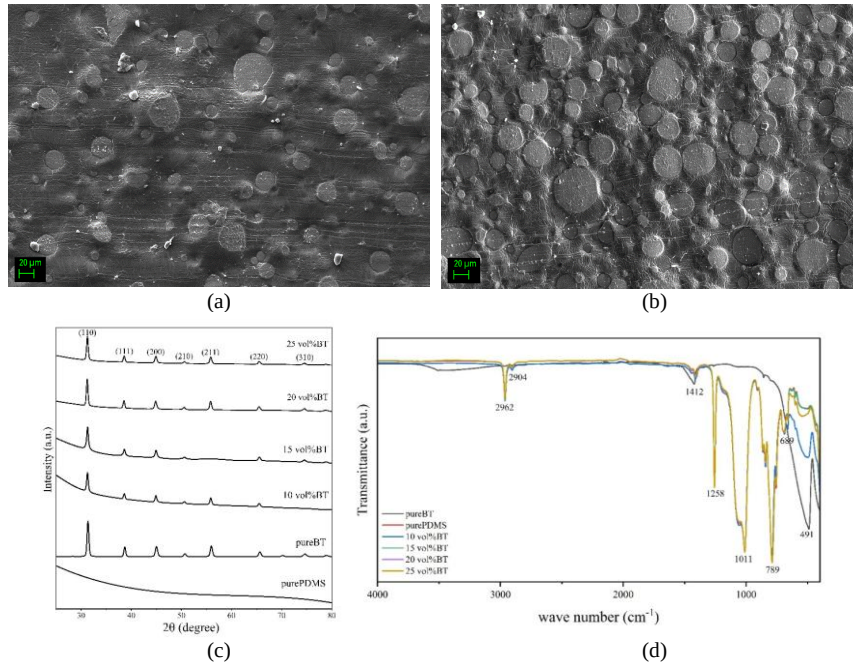


Figure 1. Scanning electron microscopy (SEM) images of (a) 10 vol% BT/PDMS and (b) 25 vol% BT/PDMS composites, (c) XRD patterns and (d) FTIR spectra for all BaTiO₃/PDMS composites

characteristic of the PDMS polymeric network. For the BT/PDMS composites, the spectra are similar to that of pure PDMS, with additional peaks around 512 cm⁻¹ and 1,418 cm⁻¹. The broad peak at approximately 450-500 cm⁻¹ is significant for barium titanate, where the wave number at 500 cm⁻¹ corresponds to vibrations of the titanium-oxygen (Ti-O) bond, and the wave number at 450 cm⁻¹ corresponds to vibrations involving metal-oxygen bonds (Ti-O and Ba-O) (Asimakopoulos *et al.*, 2014). The peak at 1,418 cm⁻¹ is attributed to a carbonate compound (C-O bond), likely originating from precursors used during BaTiO₃ synthesis (Pant *et al.*, 2006). The FT-IR spectra confirm the presence of both BaTiO₃ and PDMS phases in all composites, with no evidence of new bond formation or foreign bands.

Figure 2 illustrates the frequency dependence of the dielectric constant and dielectric loss at room temperature for BT/PDMS composites with varying compositions. The graph shows that both the dielectric constant and dielectric loss of these composites remain largely independent of frequency. Upon incorporating BT into the PDMS matrix, the dielectric constant (ϵ_r) increases significantly with higher BT content, while the dielectric loss ($\tan\delta$) remains unchanged. This behavior can be attributed to the increased presence of the barium titanate phase, which exhibits higher dielectric properties than pure PDMS. Subsequently,

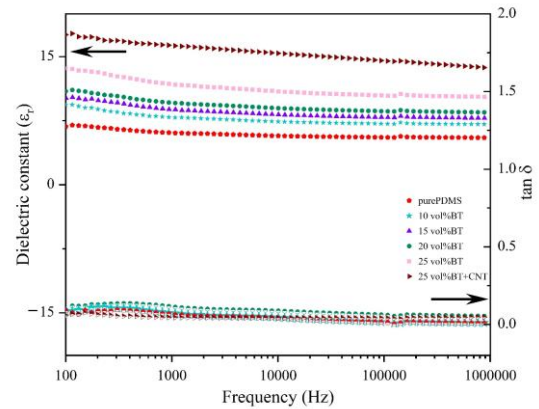
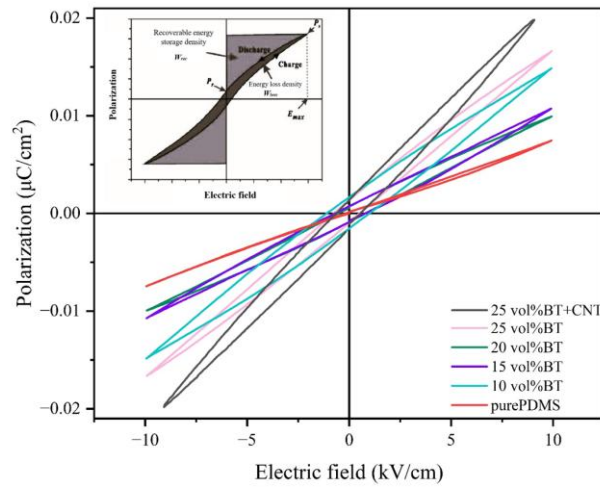


Figure 2. Frequency dependence of dielectric constant and loss for BaTiO₃/PDMS composites

the 25 vol% BT/PDMS composite, which exhibited the highest ϵ_r value, was selected for the addition of 1.5 vol% CNT to investigate the effect of CNT fillers on the dielectric properties and energy density of these composites. The results are also presented in Figure 2, and the ϵ_r and $\tan\delta$ values are tabulated in Table 1. It can be observed that the dielectric constant significantly increases after adding CNT to the system. This is because CNTs act as conductive functional materials, forming percolation networks

Table 1. Dielectric and energy storage properties of BaTiO₃/PDMS composites

Samples	Dielectric constant (ϵ_r) at 10 kHz	Dielectric loss ($\tan\delta$) at 10 kHz	Energy density ($\mu\text{J}/\text{cm}^3$)	Energy loss density ($\mu\text{J}/\text{cm}^3$)	Energy efficiency η (%)
PDMS	5.663	0.0384	0.0368	0.0020	105.87
10 vol% BT/PDMS composite	7.331	0.0417	0.0633	0.0198	145.44
15 vol% BT/PDMS composite	8.164	0.0439	0.0493	0.0108	128.07
20 vol% BT/PDMS composite	8.941	0.0439	0.1314	0.0091	107.46
25 vol% BT/PDMS composite	10.935	0.0483	0.0724	0.0146	125.23
25 vol% BT/PDMS composite filled with 1.5 vol% CNT	15.334	0.0404	0.0819	0.0184	128.90

**Figure 3. Hysteresis loops for BaTiO₃/PDMS composites**

(conducting pathways) even at low volume fractions. These conduction paths reduce the internal resistance of the composite material, thereby generating a high dielectric constant (Nawani *et al.*, 2019).

The typical P-E hysteresis loops of all BT/PDMS composites were measured at room temperature and are shown in Figure 3. The recoverable energy storage density (W_{rec}) and energy loss density (W_{loss}) of all samples were calculated from these P-E loops, as illustrated in the inset of Figure 3. From the hysteresis loop, as the electric field decreases from E_{max} to zero during the discharge process, P_s decreases to P_r . The recoverable energy storage density (W_{rec}) is released upon discharge from E_{max} to zero, as demonstrated by the light grey area in the inset of Figure 3. W_{rec} can be determined using the following equation: $W_{rec} = \int_{P_r}^{P_s} E dP$. However, the stored energy is not completely recovered due to hysteresis loss, which is represented by the dark grey area in Figure 3. This lost energy is referred to as the energy loss density (W_{loss}). Additionally, the energy efficiency (η) can be calculated using the following equation: $\eta = \frac{W_{rec}}{W_{rec} - W_{loss}} \times 100\%$. The calculated

values are tabulated in Table 1. The results indicate that pure PDMS exhibits the lowest values for all dielectric and energy storage properties. However, incorporating BT into the PDMS matrix significantly enhances these properties. The data shows that increasing the BT content in PDMS composites improves the dielectric constant, with the highest value observed in the 25 vol% BT/PDMS composite filled with 1.5 vol% CNT. In terms of stored energy, the 20 vol% BT/PDMS composite achieves the highest energy density, suggesting an optimal balance between dielectric constant and loss. While other composites display moderate energy density values, pure PDMS remains the lowest. Additionally, the 20 vol% BT/PDMS composite also exhibits the lowest energy loss density. The decline in energy density and increase in energy loss density when BT content reaches 25 vol% may be attributed to poor dispersion and a higher degree of agglomeration of BT nanoparticles. Furthermore, the addition of CNT (1.5 vol%) does not significantly improve energy density, as the 25% BT/PDMS composite with CNT has a slightly lower energy density than the 20% BT/PDMS composite. The addition of CNTs does not significantly improve the energy storage

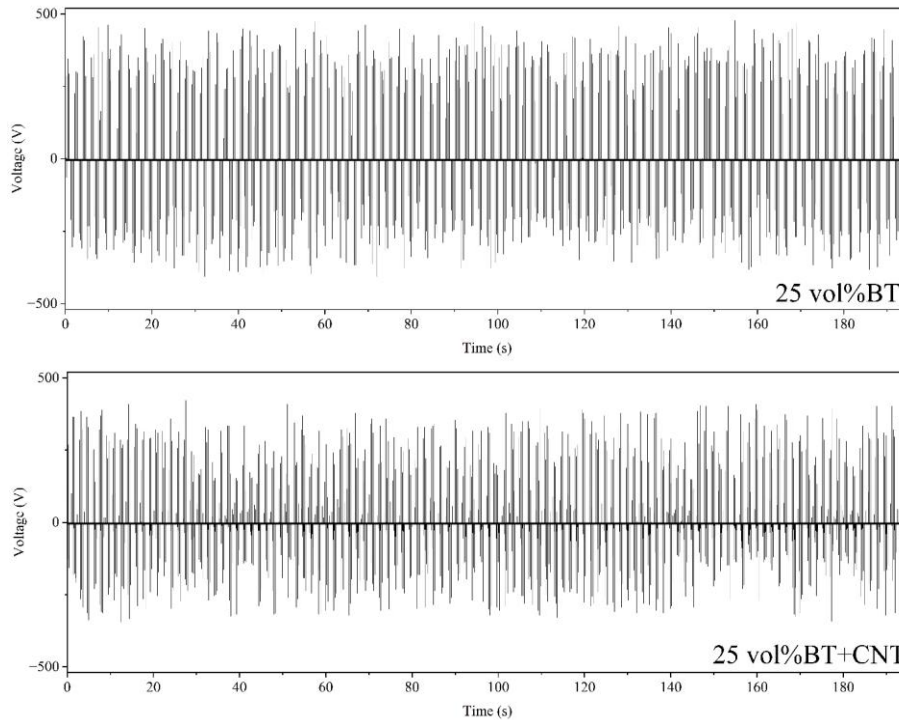


Figure 4. Output voltage of 25 vol% BT/PDMS composite with no filled and filled with 1.5 vol% CNT measured with respect to time under an applied force at 300 N

properties of BT/PDMS composites due to several factors. While CNTs can enhance the dielectric constant by improving polarization, they also introduce high electrical conductivity, leading to increased leakage currents and dielectric loss, which negatively impact energy storage efficiency. Furthermore, CNTs have a strong tendency to agglomerate due to van der Waals interactions, resulting in non-uniform dispersion within the polymer matrix. This can create localized conductive paths and increase loss mechanisms, reducing the overall energy storage capability. Additionally, while BT nanoparticles are high-permittivity dielectric materials, CNTs exhibit semiconductive to conductive behavior depending on their type and structure. As a result, the incorporation of CNTs may disrupt the dielectric synergy between BT and PDMS, preventing optimal energy storage (Rubel *et al.*, 2019; Tamayo-Vegas *et al.*, 2022; Choi *et al.*, 2023).

Furthermore, the piezoelectric properties were demonstrated by measuring the output voltage generated under mechanical force. Figure 4 presents the output voltage over time for two different composite materials: the 25 vol% BT/PDMS composite (top graph) and the 25 vol% BT/PDMS composite filled with 1.5 vol% CNT (bottom graph). Both samples exhibit an alternating voltage

response under an applied force of 300 N, confirming their piezoelectric nature. However, the amplitude of the output voltage is slightly lower in the CNT-filled composite compared to the pure BT/PDMS composite. This reduction in voltage amplitude may be attributed to the increased electrical conductivity introduced by CNTs, leading to higher charge dissipation and leakage. Additionally, CNT aggregation and uneven dispersion may have further reduced charge trapping efficiency, diminishing the voltage output. Despite this, the overall trend and frequency of voltage fluctuations remain similar in both cases, suggesting that while the addition of CNTs does not significantly alter the response pattern, it primarily affects the output magnitude.

Conclusions

This study successfully developed and analyzed BT/PDMS composites with varying BT loadings (10-25 vol%) and an additional 1.5 vol% CNT filler to assess their dielectric, energy storage, and piezoelectric properties. Microstructural analysis confirmed the uniform dispersion of BT nanoparticles within the PDMS matrix, while XRD and FT-IR phase characterization verified

the presence of BT without any additional bond formation. Dielectric measurements showed a significant increase in the dielectric constant with higher BT content, attributed to BT's high permittivity. The inclusion of CNTs further enhanced the dielectric constant due to their conductive nature and percolation effects. However, energy storage analysis indicated that the 20 vol% BT/PDMS composite achieved the highest energy density, suggesting an optimal balance between dielectric constant and loss. In contrast, CNT incorporation did not significantly improve energy density, likely due to increased electrical conductivity, leakage currents, and agglomeration, which disrupted charge trapping and storage efficiency. Piezoelectric measurements confirmed an alternating voltage response under mechanical force, demonstrating the piezoelectric nature of the composites. However, CNT addition slightly reduced the output voltage amplitude, possibly due to charge dissipation and leakage, while the frequency and overall trend of voltage fluctuations remained unchanged.

Acknowledgement

This work was supported by King Mongkut's Institute of Technology Ladkrabang (KMUTL) under Grant No. KREF046706. The work of Apinya Sasipongpan was financially supported under Grant No. KREF016726 from KMUTL. The work of Nakulkarn Rerngroen was financially supported under Grant No. KREF016724 from KMUTL. The authors acknowledge the facilities and technical assistance provided by the Nanotechnology and Material Analytical Instrument Service Unit (NMIS), School of Integrated Innovative Technology, King Mongkut's Institute of Technology Ladkrabang.

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