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Research Article

The Effect of Metal Oxide Choice on the Pseudocapacitive Performance of Polyaniline Composites

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ABSTRACT

In recent years, there is a dire need for energy, and energy storage appears to be a cost-effective solution to meet these needs. Supercapacitors are the most widely used for this purpose. Materials based on polyaniline (PANI) are commonly used in supercapacitors due to their impressive properties, such as significant specific capacitance, excellent electrochemical performance, cost–effective of synthesis. However, a significant drawback of PANI is its limited durability. To address this issue, PANI is often combined PANI with various materials. In this study, we investigate supercapacitor's electrode materials by combining PANI with various metal oxides such as ZrO_2, ZnO_2, TiO_2 and MnO_2. Herein we used X-Ray Diffraction (XRD) and Fourier Transform Infrared spectroscopy (FTIR) to confirm PANI/Metal oxides composite synthesis. The electrochemical performance was conducted by cyclic voltammetry. The PANI/ZrO_2 exhibited a specific capacity of 563.5 F/g the highest comparing to the other composites, which make it more suitable for supercapacitors application. On the other hand, PANI/TiO_2 demonstrated lower conductivity, therefore it could not be selected to this application.

KEYWORDS

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Cyclic voltammetry

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1 Introduction

The In this last decade, clean and renewable energy demand is constantly increasing. To fulfill human energy needs, the development of energy conversion and storage became more than an imperative to address this demand. Indeed, supercapacitors have been emerged as a promising category of energy storage solution, due to their outstanding electrochemical performances [1]. According to energy storage mechanisms, supercapacitors could be divided into electric double-layer capacitance (EDLC), pseudo-capacitance, and hybrid capacitance. The supercapacitors electrode materials are crucial component in overall performance of these devices. The electrode active material largely determines its capacitance and energy storage density [2]. Conducting polymers have exhibited a promising property as electrode active materials. Among them, polyaniline (PANI) is widely studied, due to its high capacitive, low cost and facile synthesis [3]. Furthermore, the heterojunction with other elements can enhance significantly their electroactivity. Transition metal oxides and electrically conducting polymers are extensively studied as pseudocapacitive active materials. It has been proved that hybrid electrode can make full use of both components conducting polymers and metal oxides, and thus improve the electrochemical performance of the assembled supercapacitors [4].

Deshmukh *et al.*, has prepared PANI/TiO_2 composite electrode material using chemical route at room temperature. According to their finding, the electrode exhibited a specific capacitance (SC) of 783 F/g at 5 mV/s in 1 M H_2SO_4 electrolyte solution [5]. Similarly, Shuang Xi *et al.*, synthesized PANI/MnO_2 composite on carbon cloth sheet via one step electrochemical deposition method. The electrochemical performances displayed high SC of 1694.25 mF/cm at 1 mA/cm which offered significant potential for energy storage devices [4]. Abirami *et al.*, obtained a maximum SC of 1903 F/g at a scan rate of 10 mV/s for PANI/$CeVO_4$ composite [6]. By using a chemical oxidation technique, Priyanka *et al.*, have synthesized polyaniline-nickel oxide (PANI-NiO). The obtained outcomes of SC was 522.45 F/g at a current density of 1 A/g. The stability study of the electrodes shows a high capacitance retention of 79.5 % after 3000 GCD cycles [7].

In the light of these researches, reporting different experimental parameters and testing conditions, which may result difficulties to directly compare among metal oxides paired with PANI yielded the best electrochemical performance.

Despite the extensive research conducted on PANI/metal oxide composites, identifying the most appropriate remains difficult due to the diverse synthesis methods, electrode preparation procedures, electrolytes, and test conditions used across studies. This variability obscures the intrinsic contribution of each metal oxide to the electrochemical

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behavior of PANI-based electrodes. Consequently, maintain the experimental parameters consistent is advisable to pinpoint the optimal combination that delivers enhanced electrochemical performance for supercapacitor applications.

The present study focuses on electrode elaboration from PANI and metal oxide such as MnO_2, TiO_2, ZnO and ZrO_2, by using unified in situ oxidative polymerization method, according to the general procedure described by previous references [8, 9]. The synthetized composites were comparatively evaluated by examining their structural and electrochemical properties and determine which composite exhibits the best electrochemical performance for supercapacitor application.

2 Materials and Methods

The electrode composite material was synthesized via in situ oxidative polymerization method as indicated elsewhere [10]. First 4.5 ml of aniline was added to 100 ml HCl (1N) in beaker containing 2 % of metal oxide (MnO_2, TiO_2, ZnO or ZrO_2), under stirring at 0 ° C. This ratio is relatively expressed to the aniline weight. After 15 hours 4.5 g ammonium persulfate dissolved in 20 mL deionized water was added dropwise. The mixture was kept at constant stirring during 14h. Then the polymerization solution was filtered and the precipitate was washed and dried at 40 °C all night to obtain a fine dark

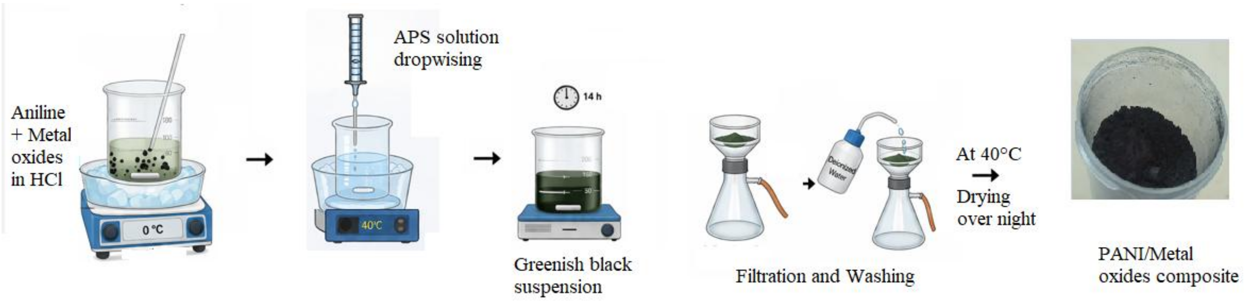

Fig. 1. Composites synthesis method.

powder. The pristine PANI was obtained with the same route without adding metal oxides. The overall steps are illustrated in Figure 1.

XRD and FTIR were used to confirm the integration of metal oxides into polymer matrix. Otherwise, the electrodes composites were evaluated by cyclic voltammetry (CV) in a 3-electrode setup electrochemical cell, using Autolab 302N, connected to NOVA 1.7 software. Ag/AgCl saturated KCl electrode and platinum sheet (1 cm^2) were used as reference and counter electrodes respectively. The CV was conducted at scan rate of 50 mV/s with voltage window from -0.4 V to 1.2 V. 1M H_2SO_4 was used as electrolyte solution and the SC was calculated using the following Eq (1) [11]:

$$C_s = \frac{\int i\, dV}{2m\upsilon\Delta V} \quad (1)$$

To ensure reliability and reproducibility, the composites synthesis was carried out 3 times and the electrochemical tests were all performed under identical conditions.

3 Results and discussion

3.1 Structural characterization

The structural and compositional properties of the composites were analyzed using X-ray diffraction (XRD) and FTIR spectroscopy. The recorded XRD patterns are depicted in Figure 2. Pure PANI demonstrates amorphous behavior described by a board diffuse band locate around ~22 - 25°. This is typical of polyaniline, whose low crystallinity is often associated with limited order between polymer chains [12].

The addition of oxides significantly alters the diffraction pattern: the broad polymer halo gives way to narrower peaks, indicating a greater crystalline contribution from the inorganic fillers. The entire composites reveal peaks around 20-23° corresponding to PANI. The identification of different crystalline phases in the samples was performed by comparing the data with the Joint Committee for Powder Diffraction Standards (JCPDS) files.

PANI/ZrO_2 composite exhibits several peaks located at 2θ values of 25.57, 26.2, 32.8, 36.98 and 42.79°, which is consistent with crystallographic planes of (020), (200), (210), (220) and (031) respectively, and correspond to the tetragonal zirconia JCPDS#83-809.

Similarly, the PANI/TiO_2 showed a dominant peak at 25.55° accompanied by weak reflections around 37.93, 48.30, 53.90, 55.15 and 62.82° corresponding to the lattice planes of (111), (311), (022), (230), (421) and (331) respectively, which are attributed to the orthorhombic structure of TiO_2 (JCPDS #72-100). It is worthy to note that the PANI diffuse background remains visible, while the anatase appears as a dispersed phase in the composite.

For PANI/ZnO, which demonstrated more crystallinity, the patterns provided diffraction peaks located at 2θ of 31.80°, 34.51, 36.38, 47.58, 56.71, 62.93 and 68.12°, which could be assigned to the (100), (002), (101), (102), (110), (102) and (201) planes of hexagonal wurtzite structure of ZnO (JCPDS#36-1451).

The XRD patterns of PANI/MnO_2 composite exhibited broad diffuse halo and several sharp diffraction peaks, indicating the coexistence of amorphous and crystalline phases. The main peak appears around 25.30° followed by other distinct and visible at 37.75, 42.94, 50.20 and 62.65° corresponding to the diffraction from (110), (101), (111), (201) and (300) crystal planes respectively of tetragonal structure of MnO_2 (JCPDS#71-71). These outcomes confirm the well incorporation of metals oxides in the polymer matrix and thereby validate the composite preparation method.

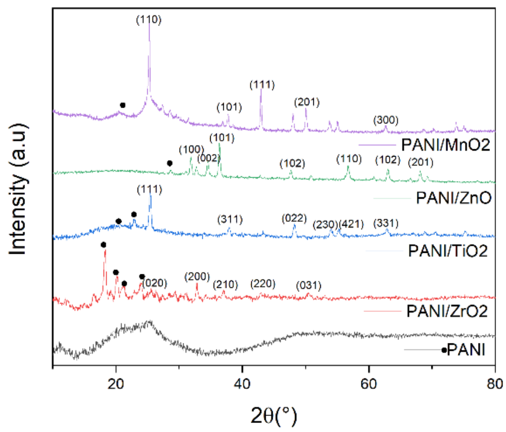

Fig. 2. XRD patterns of composites.

Moreover, by meaning of the characteristics peak for each composite, the interaction between the metal oxides and PANI backbone was evaluated by measuring the particle size using the Debye–Scherrer formula:

$$D = \frac{K\lambda}{\beta cos\theta} \quad (2)$$

where K is the Scherer constant=0.89, "D" is the crystallites size, λ=1.5406 Å (wavelength of X-rays), β (in radians) is the FWHM and θ is the peak position. The average crystallite size were found to be 0.15 nm, 0.35 nm, 0.32 nm, 0.52 nm and 0.27 nm for PANI, PANI/ZrO_2, PANI/TiO_2, PANI/ZnO and PANI/MnO_2 respectively. Crystallite size was significantly increased after metal oxides incorporation comparing to pristine PANI.

The revealed peaks from the FTIR curve as shown in Figure 3 indicate

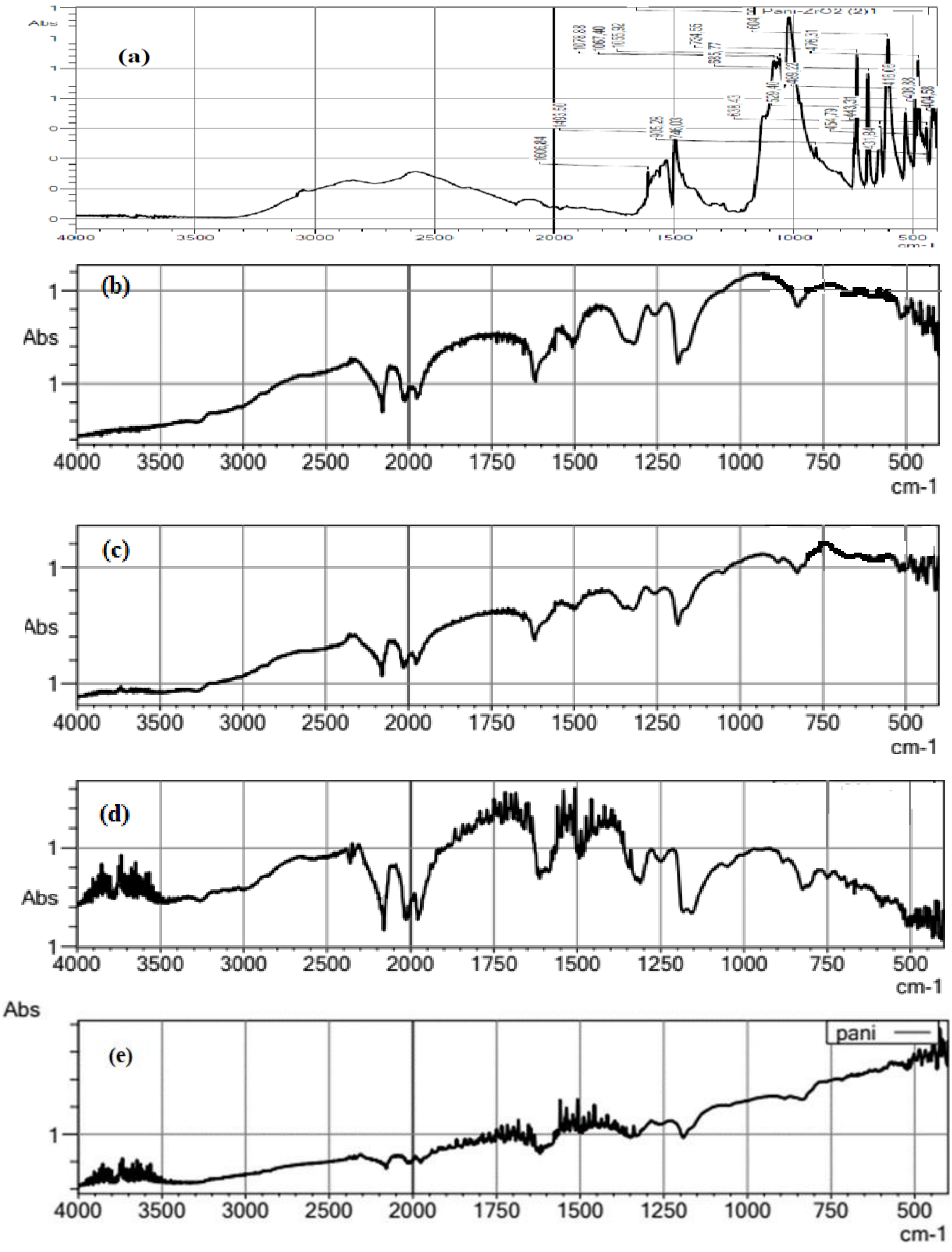

Fig. 3. FTIR spectrum of composites electrodes (a) PANI/ZrO_2, (b) PANI/ZnO, (c) PANI/TiO_2, (d) PANI/MnO_2 and (e) PANI.

characteristic vibrational mode, which confirm the composites' structure. The composites curves presented similarities in bands owing to the presence of PANI (Figure 3(e)). Its characteristics bands appeared at 1600 cm^{-1} and 1500 cm-1 attributed to C=C/C=N stretching vibrations of quinonoid and benzenoid units and to N-H vibration of Amin group, respectively. The band at 1300 and 1350 cm-1 is further assigned to C-H liaisons. These outcomes are in line with previous research [7, 13]. For the metal oxide as observed in Figure.3 (a, b, c) and (d) the presence of ZnO, TiO_2, MnO_2 and ZrO_2 is verified by vibrational bands in the 400-850 cm-1 region, which are characteristic of metal oxides [14-16]. These outcomes confirmed the incorporation of metal oxides into polymer matrix, which consisted the results from XRD analysis and allowed to validate the synthesis process.

3.2 Electrochemical performance

Cyclic voltammetry is essential for assessing reaction rates and the electrochemical behavior of energy storage systems under various operating conditions. The composites were investigated by CV over 80 cycles. The recorded curves are illustrated in Figure 4.

As depicted from the Figure 4, all the curves exhibited a pseudo-rectangular shape meaningful the pseudocapacitive behaviour of the electrodes arising from faradaic charge transfer process. A clear pair redox peak demonstrates the reversibility of charge and discharge of the material. PANI has showed a pair of narrow redox peaks corresponding to leucoemaraldine/emeraldine and emeraldine/pernigraniline transition [17]. The peak separation ΔEp = 0.127 V, which is above the theoretical minimum (59 mV) indicates,

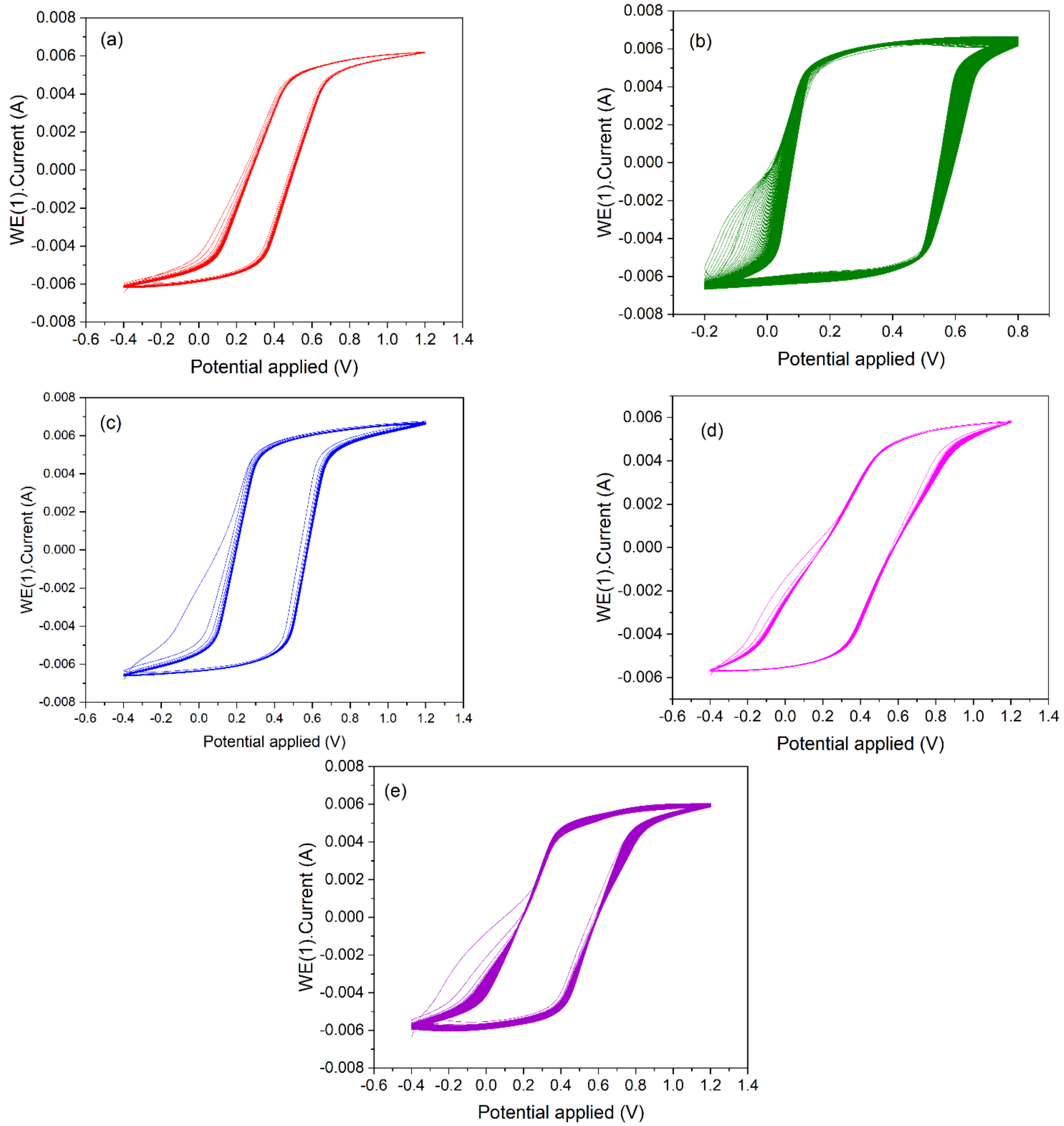

Fig. 4. CV curves of composites electrodes, (a) PANI, (b) PANI/ZrO_2, (c) PANI/ZnO, (d) PANI/TiO_2 and (e) PANI/MnO_2.

according to Nicholson [18], fast electron transfer kinetics and excellent reversibility, consistent with PANI's excellent pseudocapacitive nature [17].

The incorporation of metal oxide such as ZrO_2, ZnO and MnO_2 has generated an excellent performance that can be assigned in increasing of the current and significant larger enclosed area. The potential peak separation for composites measured from curves are 0.357, 0.149, 0.205 and 0.220 V for respectively, PANI/ZrO_2, PANI/ZnO, PANI/TiO_2 and PANI/MnO_2, which highlight enhancement in capacitive performance. This enhancement can be attributed to metal oxides particles that acted as spacer to prevent PANI chain aggregation, exposing thus more electoactive surface area. Moreover, the synergy is raised from the complementary redox reactions. In the composites, PANI undergoes Faradaic transitions between its leucoemeraldine, emeraldine, and pernigraniline states. While in PANI/MnO_2 composite, MnO_2 with the Mn^{4+}/Mn^{3+} redox pair, was involved in protonation/deprotonation at the surface [19]. Whereas the enhancement of PANI/ZnO composite is attributed to the large surface area and electronic donor nature of ZnO, which facilitate charge transfer at the interface [20].

On the other hand, ZrO_2 oxide in PANI/ZrO_2 composite acted as structural spacers that prevent PANI chain aggregation, leading to an increase in the electroactive surface area. Thus, it contributed significantly in obtained SC, even with its bandgap that does not participate in the pseudocapacitive faradaic reactions [21, 22].

The shape retains the clear redox peak of PANI with enhanced current, which indicate increasing in charge storage by creating more active sites for reaction to occur. Exception for PANI/TiO_2, unlike expectation but agrees with such previous study [23], its performance was affected. The current and even the enclosed area is reduced and the redox peaks are less pronounced. These could be explained that TiO_2 possess a wide bandgap (≈ 3.2 eV), which could act as electron trap at the composite/electrolyte interface, hindering the charge transfer across the electrode surface. This phenomenon is intensified when TiO_2 particles aggregate [17].

From these curves and according to the Eq (1) the specific capacities of composites electrodes were determined and are summarized in Table 1.

As can see from the table, among the composite's electrodes, the PANI/ZrO_2 exhibited the enhanced capacity, which make it more suitable for supercapacitor application. However, it is being a bit hindered because of its cost comparing to the raw materials MnO_2, TiO_2 and ZnO, which their pricing falls within a comparable bracket [24, 25]. Thus, a compromise emerges, for applications requiring a small footprint, particularly portable electronic devices such as sensors, smartwatches, and medical implants [26], PANI/ZrO_2 composite's superior specific capacity makes it a worthwhile choice. Conversely, for larger scale applications without masse restrictions, PANI/ZnO or PANI/MnO_2 composites offer better cost efficiency [27, 28].

Table 1. Specific capacitance of PANI/Metal oxides electrodes.

Electrodes	PANI	PANI/ZrO_2	PANI/ZnO	PANI/TiO_2	PANI/MnO_2
Specific capacity (F/g)	248.43	563.5	448.27	336.56	377.81

4 Conclusion

In summary, PANI/metal oxides composites were prepared by in situ polymerization cost effective route. Based on XRD patterns, the metals oxides were well integrated to PANI matrix. Notably, the crystallite size was increased, suggesting enhanced structural ordering within the composite. The FTIR confirmed the integration of metal oxide into polymer matrix. From CV analysis, the incorporation of ZrO_2, ZnO, and MnO_2 significantly enhances capacitive performance, reflected in increased current response, indicating the creation of additional active sites for redox reactions, thereby boosting charge storage capacity.

PANI/ZrO_2, PANI/ZnO and PANI/MnO_2 have significantly enhancing the charge storage capacity of pure PANI. They are excellent candidates for high-performance supercapacitor electrodes. However, PANI/TiO_2 has demonstrated a significant degradation in performance and should be discarded. Among them PANI/ZrO_2 has exhibited the best performance by means of specific capacity of 563.5 F/g which make it suitable for supercapacitor application. Additionally, investigations should explore the material's long-term cycling durability, as well as the construction of functional symmetric or asymmetric supercapacitor cells to evaluate their practical performance.

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Declaration of Competing Interest

The authors declare no conflict of interest.

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