



A NEW GENERATION HYBRID SUPERCAPACITOR: GO/ZNO

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ABSTRACT

Supercapacitors are energy storage materials used to store higher and larger amounts of charge than known capacitors. Energy density can be increased. Capacity and energy amount can be increased by increasing the amount of surface area used. In this study, graphene oxide (GO) synthesised by Modified Hummers method was reinforced into ZnO synthesised by sol-gel method and hybrid material with supercapacitive properties was produced. XRD, SEM, FTIR, DTA-TGA, UV- Vis-NIR and CV analyses were performed on the produced hybrid nanosupercapacitive composites. It was observed that ZnO particles were located between GO layers and were nano-sized. XRD diffractograms showed hexagonal ZnO crystallographic planes (103), (200), (112), (201), (004) and (202). DTA analyses revealed endothermic peaks due to water loss and organic decomposition. FT-IR analyses confirmed the presence of ZnO and GO in the structure. CV measurements indicate rapid charge transfer at the electrode-electrolyte interface.

1. INTRODUCTION

As energy storage systems, supercapacitors bridge the performance gap between traditional capacitors and batteries. They have higher energy density than traditional capacitors and higher power density than batteries. The energy storage mechanisms of supercapacitors are entirely electrostatic and do not involve slow electron transfer reactions as in batteries.

Supercapacitors are energy storage materials that can store higher and more charge than known capacitors. Other names for supercapacitors are electrochemical capacitors or ultracapacitors. Supercapacitors, which are improved versions of classical capacitors, have similar main features to classical capacitors. Classical capacitors consist of two conductive electrodes kept apart by insulating material. When voltage is applied to the capacitor, an oppositely charged charge accumulates on the surface of both electrodes. An electric field is created by the charges kept apart by the insulating material, enabling the capacitor to store energy. The ability of capacitors to store electric charge is equivalent to the ratio of stored positive charge to voltage. [1-7]

Supercapacitors usually consist of an electrolyte, two electrodes and a separator surface. In supercapacitors; unlike classical capacitors, the surface areas of the electrodes used are very large and the distance between the electrodes is very limited. As a result of these changes, the capacitance and energy absorption of supercapacitors exhibit superior qualities compared to classical capacitors. Supercapacitors have a number of distinctive characteristics that make them more accepted than other energy storage devices and are open to improvement. Electromagnetic field energy amounts are high. Thus, they can realise high electricity transfer rate at a time. Charge and discharge speed is quite high. It has the ability to charge in a short time. Operating ranges are quite high. They can be charged and discharged approximately 600,000-2,000,000 times. Thus, they can have a service life of 10-20 years depending on the place of use. Their internal strength is low. Therefore, cycle capacity is around 90-95%. The rate of discharging its own energy is quite low. It can store the stored energy for a long time after charging. Operating temperature range is quite wide. It performs between temperatures such as -45°C and +75°C. Environmentally friendly. It does not contain toxic and harmful substances. Energy

density can be increased. Capacity and energy amount can be increased by increasing the amount of surface area used [8-12].

Hybrid capacitors are supercapacitors produced to achieve higher performances by utilising the unique properties of electrical double layer capacitors and pseudocapacitors. In hybrid supercapacitors, the electrodes determined as cathode and anode consist of various combinations. In hybrid supercapacitor configuration; electrodes with carbon as the main material are generally preferred as the cathode and non-faraday electrostatic charge storage is performed with the cathode. As the anode, metal oxide material is generally preferred and reduction-oxidation reactions are carried out via the anode. Thus, hybrid supercapacitors have the properties of pseudocapacitor and electrical double layer capacitor. Due to the fact that hybrid supercapacitors work with both Faradaic and non-Faradaic systems, their energy and power densities are higher than those of electric double layer capacitors. On the other hand, the higher stability of hybrid supercapacitors compared to pseudocapacitors allows for a higher cycle life. Therefore, hybrid capacitors with higher cycle life than pseudocapacitors and higher capacitance and energy density than electrical double-layer capacitors have been obtained [8,11-14]. There are three types of hybrid capacitors: asymmetric, battery-type and composite:

As is well known, supercapacitors consist of a suitable electrolyte, two electrodes and a separator surface. However, when the internal resistance of the electrode material is high, collectors can be added to the supercapacitor system to reduce this resistance. To achieve high power and energy density, electrode selection and surface area determination are critical parameters. The preferred electrode's high surface area and the material's high specific capacitance directly affect the energy storage mechanisms of supercapacitors [8,11,15]. Three main electrode materials are preferred in the production of supercapacitors: conductive polymer, metal oxide and carbon-based. In addition, composite electrode materials are obtained by combining one or more of these main electrode materials and applied in supercapacitors [8].

In this study, hybrid materials with supercapacitive properties were produced by reinforcing graphene oxide (GO) synthesised by Modified Hummers method with ZnO synthesised by sol-gel method. Various characterisation techniques were applied to the fabricated hybrid nanosupercapacitive composites to investigate their supercapacitance properties and the effects of different solvents on these properties.

2. MATERIAL AND METHOD

2.1. Graphene Oxide Production

Graphene oxide was synthesized from graphite powder using the Modified Hummers method, which is a widely used chemical approach. Oxidation of the graphite layers introduces oxide groups that expand the interlayer spacing, allowing sonication to separate the layers effectively. As a result, graphene oxide sheets are formed. In this study, graphite powder was initially mixed with 1,5 gr sodium nitrate (NaNO_3) and 69 ml sulfuric acid (H_2SO_4) in an ice bath. During the second stage, 9 gr potassium permanganate (KMnO_4), a strong oxidizing agent, was gradually added to the mixture, which was then stirred at 35 °C. In the third stage, 300 ml deionised water was introduced into the solution while stirring continued. Finally, hydrogen peroxide (H_2O_2) at a 30% concentration was added, and the mixture was stirred further. Upon completion of these steps, the resulting mixture was washed repeatedly with deionised water until reaching a neutral pH of 7 and then filtered. The final product was dried in an oven at 50 °C for 24 hours, yielding graphene oxide in powder form.

2.2. Preparation of metal oxide solution without additives

Following the washing with deionised water and drying steps, 10 ml of solvent (deionised water, acetone, ethyl alcohol, and 2-methoxyethanol) and zinc acetate dehydrate ($(\text{ZnCH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$) at a concentration of 2 M were introduced into bottles and stirred using a magnetic stirrer at 500 rpm and 60 °C for approximately 15-20 minutes. After mixing, the resulting solution appeared white in color. Then, 0.3 ml of Monoethanolamine as a stabilising agent was added to the mixture, which was subsequently stirred again at 500 rpm and 60 °C for 2 hours, followed by ultrasonic mixing for an additional hour. As a result of this process, the previously white solution turned transparent. These

procedures were repeated separately for each solvent (deionised water, acetone, ethyl alcohol, and 2-methoxyethanol) to prepare the ZnO-based metal oxide solution.

2.3. Preparation of Nanocomposites

Graphene oxide powders synthesized via the modified Hummers method were individually dispersed in various solvents including deionised water, 2-methoxyethanol, acetone, and ethyl alcohol, followed by sonication. In each case, 1 gr of graphene oxide was dissolved in 10 ml of the selected solvent. These solutions were then added to the metal oxide solutions prepared through the sol-gel technique and stirred magnetically for 24 hours. After mixing, the resulting composite suspensions were dried at 175 °C for 6 hours. Once dried and solidified, the composites were ground into powder. The ground powders were subsequently pressed into pellets of the desired shape, yielding GO/ZnO nanocomposite electrodes with supercapacitor characteristics.

3. RESULTS AND DISCUSSION

3.1. Surface Morphology of Prepared Samples

Scanning electron microscope (SEM) consists of three parts: optical column, sample cell and imaging system. With a scanning electron microscope (SEM), after electrons accelerated by high voltage are focused on the sample, when this electron beam is effects caused by interference between electrons and atoms, the image is displayed on the screen of a cathode ray tube. It is scanned on the sample surface, collected by the relevant sensors and transmitted to signal amplifiers. received by bank transfer. In modern systems, the signals from these sensors are converted into digital signals and transmitted to the computer monitor [16].

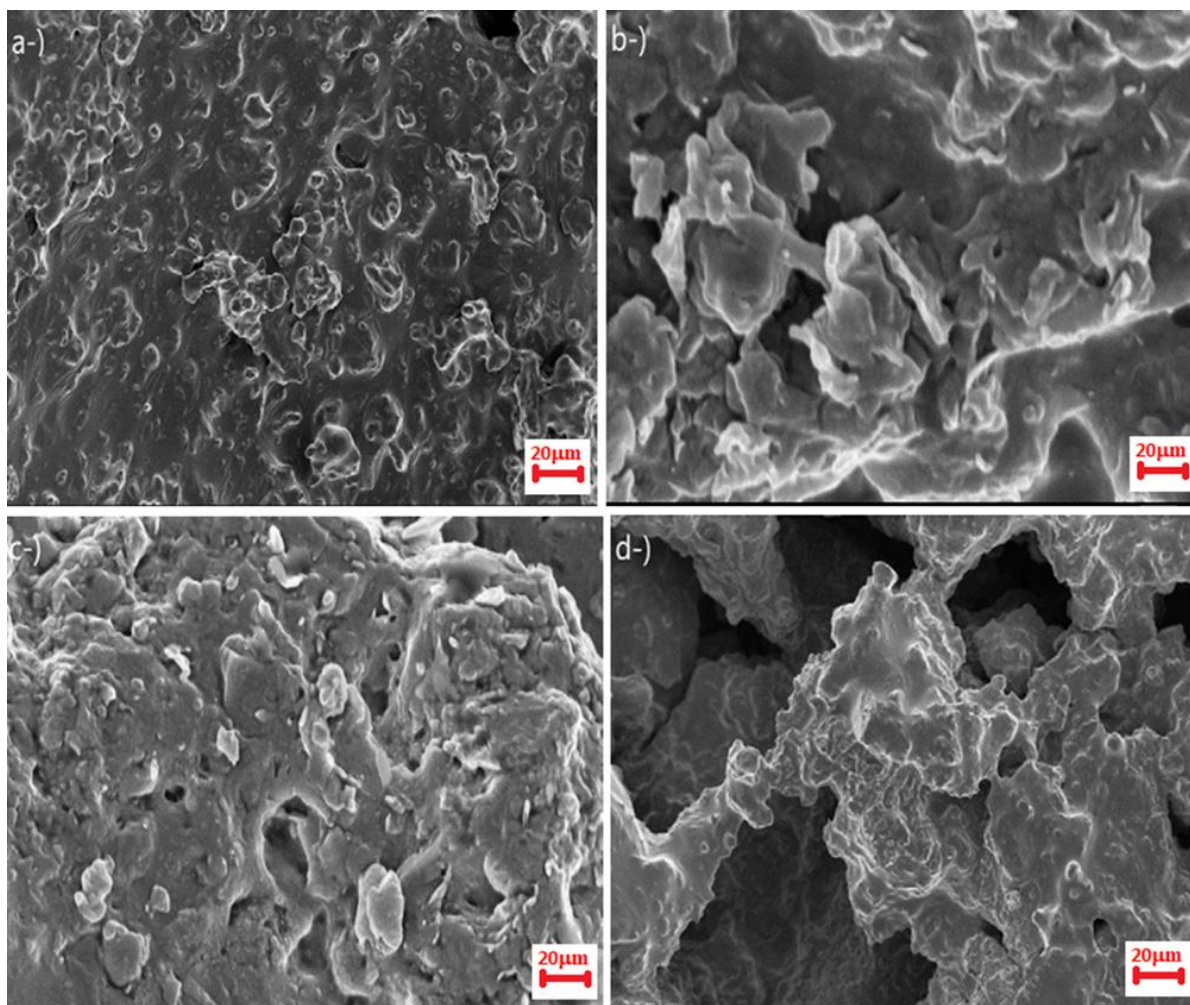


Figure 1. SEM micrographs of the produced samples (a-) ZnO-Acetone, b-) ZnO-2-Methoxyethanol, c-) ZnO-Ethyl Alcohol, d-) ZnO-Pure Water)

Scanning electron microscopy (SEM) is used to examine the structural properties of materials and to obtain information about their structural properties. In order to obtain SEM images of the Graphene Oxide doped nanostructures to be examined, the surfaces of the samples were first coated with gold by plasma technique by vacuuming for 15 seconds in the coating device. Microscope images of the powders with different solvent, power and time parameters were analysed in ZEISS SIGMA 300 scanning electron microscope.

SEM images of GO/ZnO composite materials produced using ZnO particles obtained by sol-gel method and GO produced by Hummers method are shown in Figure 1. When the SEM images of GO/ZnO composite materials were examined, it was observed that ZnO particles were placed between GO layers and were nano-sized. Thus, clustering of the layers was prevented.

3.2. X-Ray Diffractometer (XRD) Analysis

When an intense X-ray beam strikes a crystalline structure consisting of ordered groups of atoms, general scattering occurs and these scattered rays interfere with, interfere with and cancel each other. However, at certain angles these waves merge into a single phase, increasing the intensity of the wave. This phenomenon is known as X-ray diffraction. These diffraction directions vary depending on the size and shape of the crystal unit cell. The diffraction intensity is determined by some structural features of the crystal [15].

X-ray diffraction spectroscopy, known as X-ray diffraction spectroscopy (XRD), is an analytical technique that uses a beam with more energy than ultraviolet radiation, called X-rays, but weaker than gamma radiation. This characterisation by X-ray diffractometer system has different applications depending on the type of sample. The working principle is based on obtaining refraction and scattering data by sending X-rays to the sample [17]. XRD analyses of the samples, whose scan angle and various parameters were determined, were characterised in PANalytical Empyrean model.

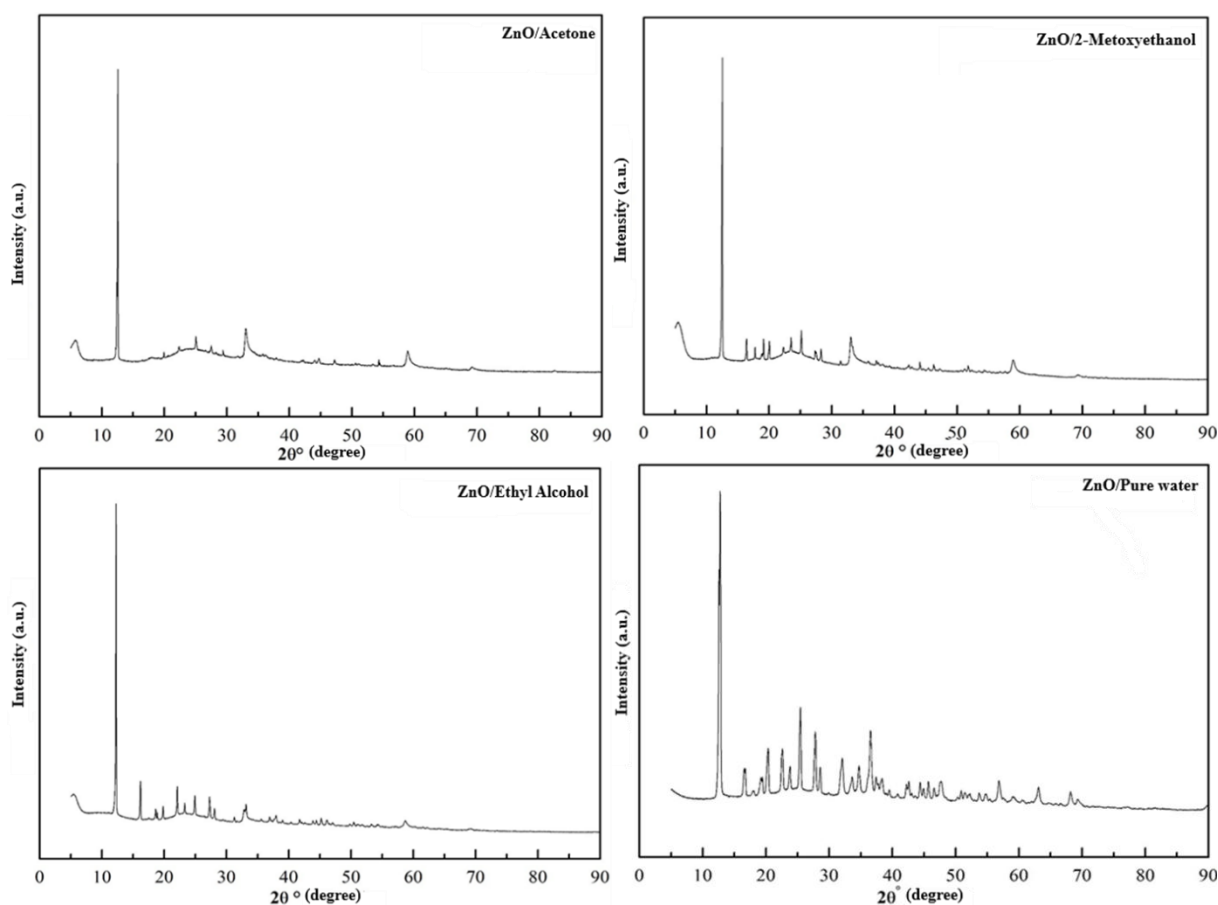


Figure 2. XRD diffraction patterns of ZnO/GO sample dissolved with different solvents

XRD analyses of ZnO particles obtained by sol-gel method using different solvents from $Zn(CH_3COO)_2 \cdot 2H_2O$ salts and GO/ZnO composites produced using GO produced by modified Hummers method are shown in Figure 2. Figure 2 shows the peaks of ZnO and GO. After the mixture of GO and zinc solutions obtained with different solvents, the obtained GO - ZnO samples provided data with well resolved peaks. For the hexagonal ZnO crystallographic planes (101), (100), (002), (110), (103), (102), (112) as 2θ degree 36.24, 31.76, 34.41, 56.60, 62.86, 47.53 and 67.96 were observed, respectively.

3.3. Thermogravimetric Analysis (TGA)

The TGA method (thermogravimetric analysis) records the mass of a sample as a function of temperature or time, while the temperature of the sample to be analysed increases continuously (usually linearly over time) in a controlled atmosphere. The diagram of mass or mass percentage versus time is called a thermogram or thermal decomposition curve [18].

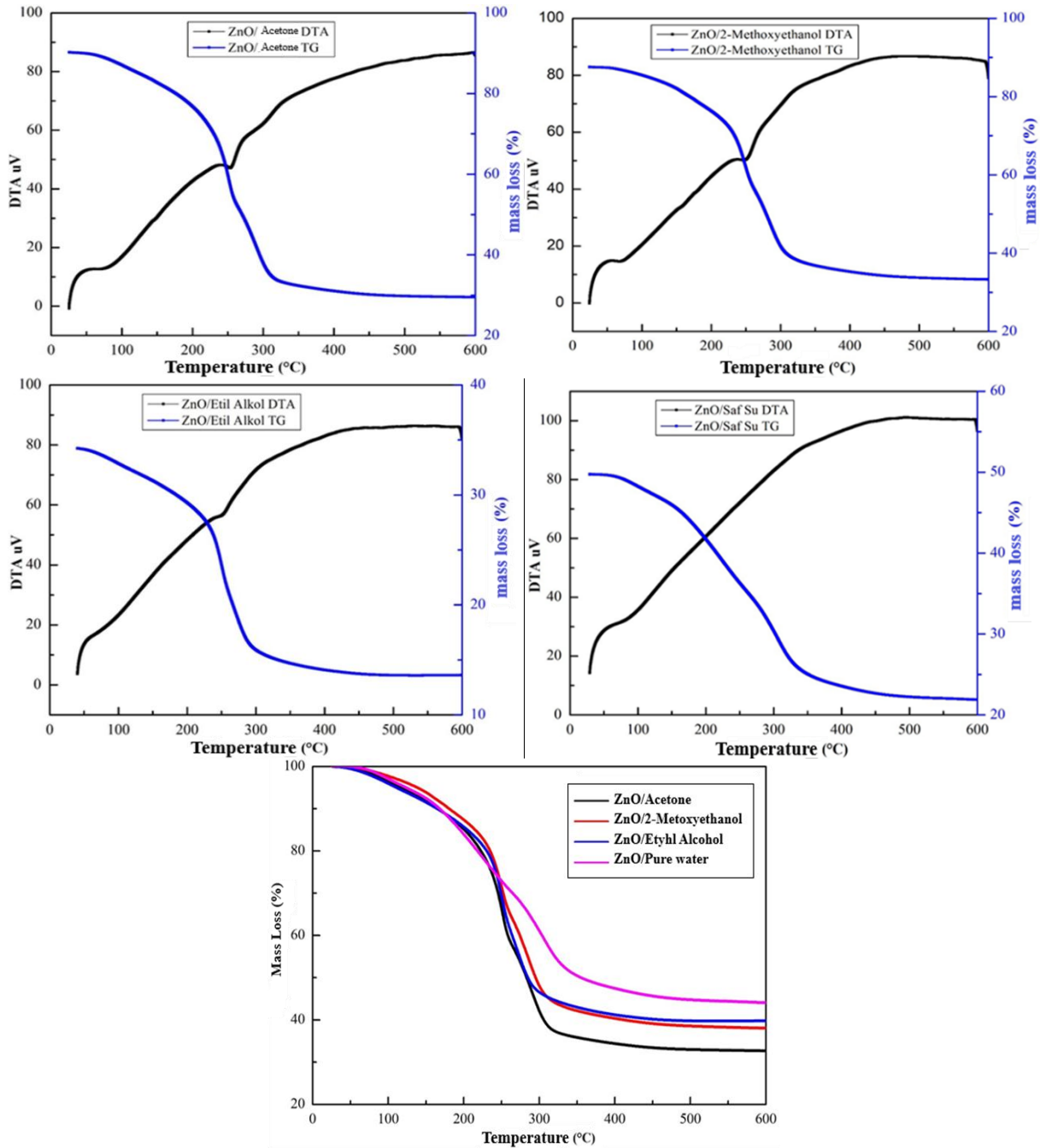


Figure 3. DTA-TG graphs of ZnO/GO samples dissolved with different solvents.

The TGA instrument consists of a precision microbalance called a thermo balance, an oven, sometimes providing an inert gas atmosphere and sometimes a reactive clean gas system, a computer system to control the instrument, data acquisition and processing. Thermal balances with sample holding chambers from 1 mg to 100 g are available. Usually 1 to 100 mg is used. The balances can record mass changes of 0.1 μg . The ovens can reach temperatures of up to 1000 $^{\circ}\text{C}$. Heating options range from 0.1 $^{\circ}\text{C}/\text{min}$ to 100 $^{\circ}\text{C}/\text{min}$. The balance of insulation and internal cooling zone ensures no heat exchange. Ar and N₂ gases are purge gases used to prevent oxidation of the sample.

Thermogravimetry/differential thermal analysis (TGA/DTA) was performed to confirm the formation of a stable ZnO phase and to determine its thermal stability. Figure 3 shows the graphs of graphene oxide doped ZnO samples obtained using different solvents. The samples were analysed under nitrogen (N₂) atmosphere between 50 $^{\circ}\text{C}$ and 600 $^{\circ}\text{C}$ at a heating rate of 10 $^{\circ}\text{C}/\text{min}$. Thermal decomposition of the samples dissolved using acetone, 2-Methoxyethanol, ethyl alcohol and distilled water takes place in several steps and the residue remains up to 600 $^{\circ}\text{C}$ after thermal analysis. The initial weight loss from room temperature to 250 $^{\circ}\text{C}$ is due to thermal dehydration of the samples. After further heating up to 320 $^{\circ}\text{C}$, a second weight loss was observed in the samples, followed by the final weight losses. When heated up to 600 $^{\circ}\text{C}$, no further weight loss was observed. The endothermic peaks in the DTA analyses are probably due to water loss and zinc organic decomposition, respectively. ZnO/Acetone/GO sample was the sample that lost the most mass with 67.40% mass loss.

3.4. Fourier Transform Infrared (FT-IR) Analysis

Fourier transform spectrometers operating in the infrared spectral range are called FT-IR spectrometers. These spectrometers divide the infrared (IR) signal, which consists of a range of frequencies, into frequencies using the mathematical method of the Fourier transform and reproduce the spectrum indicated by the intensity of each individual. In this type of spectrometer, no monochromator is used and all frequencies of the light source interact with the sample at the same time and the change in time of this information containing all frequencies is monitored. This spectrometer is based on the Michelson interferometer [19].

FT-IR is an analytical technique in chemistry that evaluates the intensity of infrared light as a function of wave number, employing the mathematical principles of Fourier transformation. [20].

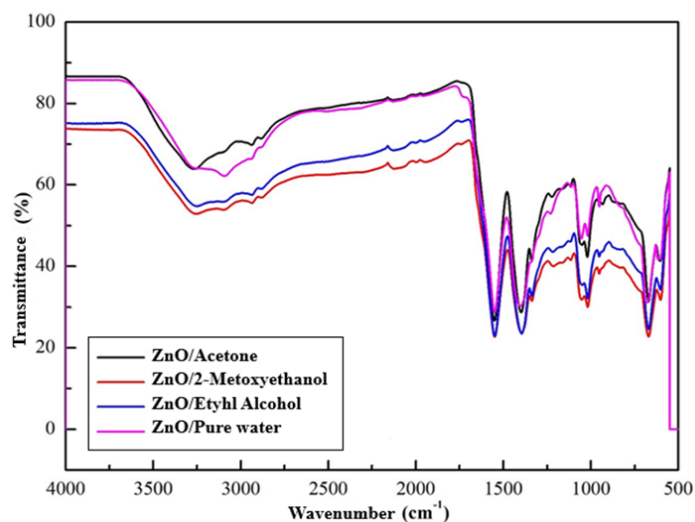


Figure 4. FT-IR spectra of ZnO/GO nanocomposites

In the FTIR spectrum of GO (Figure 4), the broad absorption band centered around 3190 cm^{-1} is attributed to O-H stretching vibrations. The peaks located at 1731, 1625, 1183, and 1040 cm^{-1} correspond to the stretching of C=O bonds, vibrations of sp^2 -hybridized C=C groups, O-H bending, C-OH stretching, and C-O stretching, respectively. However, in comparison, the peaks at 1731, 1183, and 1040 cm^{-1} are absent. In another FTIR spectrum of GO, a broader peak at 3431 cm^{-1} is linked to O-H stretching, while 1717 cm^{-1} arises from C-O stretching vibrations. The peak at 1621 cm^{-1} is associated

with skeletal vibrations of unoxidized graphite domains, and the signal at 1184 cm^{-1} is also observed. In the FTIR spectrum of the GO/ZnO nanocomposite, the broad peak appearing at 3437 cm^{-1} is attributed to O–H stretching of water molecules adsorbed on the surface. These spectral features suggest that the composite structure remains largely similar to that of pure GO.

3.5. UV Spectrophotometer

Spectroscopy is a branch of science that studies the interaction of matter with light or electromagnetic radiation in general terms. Spectroscopy is the measurement and interpretation of the electromagnetic radiation absorbed or emitted by atoms, molecules or ions in a sample during their transition from one energy level to another energy level. Ultraviolet and visible region spectroscopy is based on transitions between electronic energy levels. These transitions occur between bonding orbitals or orbitals containing electron pairs and non-bonding (counter-bonding) or empty orbitals.

The wavelength of absorption is a measure of the difference between the energy levels at which the transitions occur. The highest energy difference is observed when the electrons forming the sigma bond are excited and absorbed. This corresponds to a wavelength of about 120 - 200 nm. This is called vacuumised ultraviolet. Because in order to be able to measure in this range, the ambient air must be removed. Otherwise an illuminating spectrum cannot be obtained. However, above 200 nm, the transitions of electrons in p, d, π orbitals and conjugated π -systems can be easily monitored and illuminating spectra can be obtained.

Since rays or electromagnetic waves move through space at very high speeds, they generate energy in the form of continuous particles. They can exist in various energy forms such as light, heat, radio waves and X-rays. Among these forms of energy, only light energy is visible to the eye. Absorption or emission spectroscopies of light are spectroscopies of the absorption or emission of radiation by atoms or molecules. In order to identify substances and elucidate their chemical structure, rays in the range of 110 - 3000 nm are sent to the substance.

In order to send the beam in this range to the substance and to determine which wavelengths are absorbed, devices that can operate between certain wavelengths have been developed. Spectrochemical methods are the most frequently used methods for the qualitative and quantitative determination of inorganic and organic compounds and molecular structure elucidation.

Figure 5 shows the Reflectance (R), Absorbance (A) and transmittance (T) spectra in the range of 100-900 nm for the sample dissolved in different solvents, respectively. The average transmittance in the visible region was observed about 35 % for the ZnO/Pure water samples. Whereas, ZnO/Ethyl Alcohol sample shows a lower transmittance than all other films in whole investigated wavelength range. As seen clearly in Fig. 5, the ZnO/Ethyl Alcohol sample shows the higher absorbance than all other samples in whole investigated especially in the strong absorption edge region. Fig. 5 presents the plots of the Reflectance for dissolved in different solvents ZnO nanopowders. It is clear that the reflectance for samples increased with increasing wavelengths. As seen in Figure 5, the absorption edge changes strongly with changing solvent, indicating a strong interaction between the solvent molecules with Zn and GO molecules.

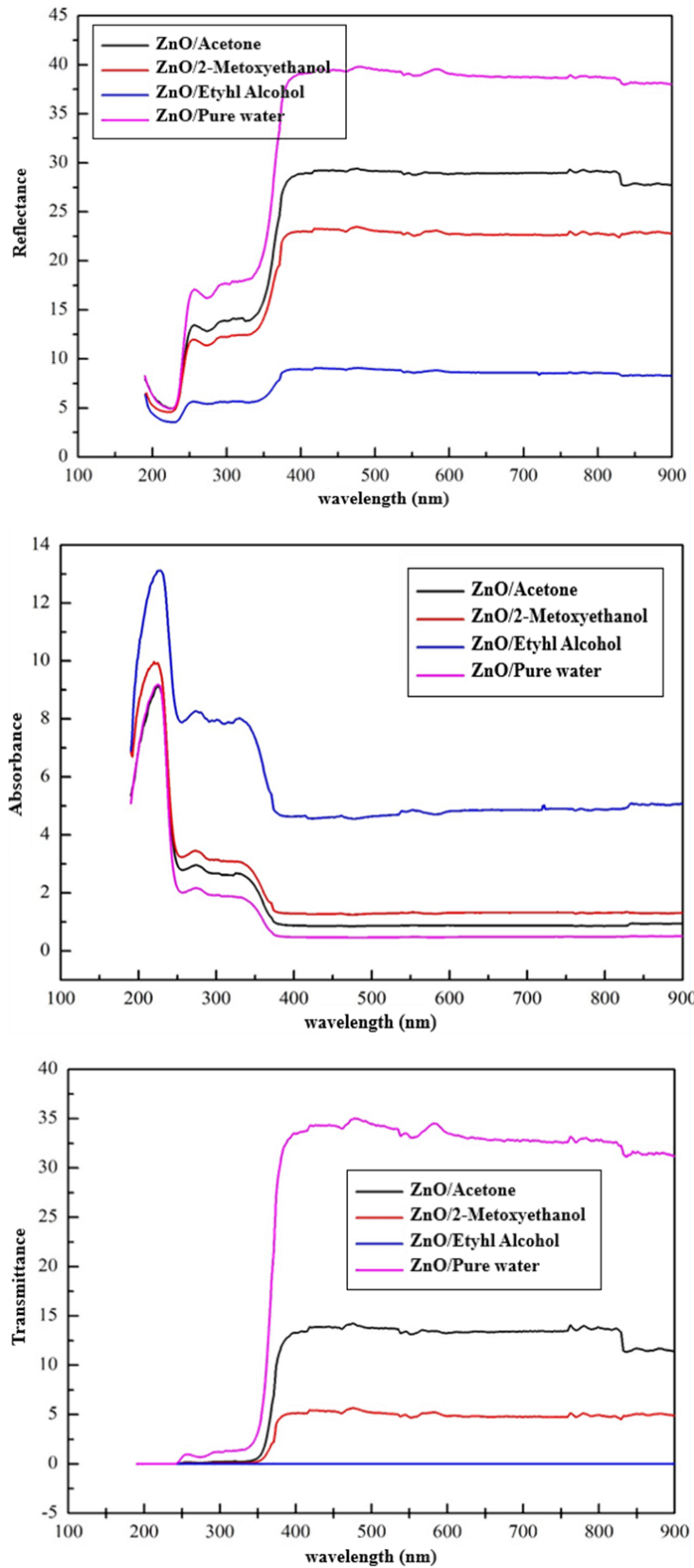
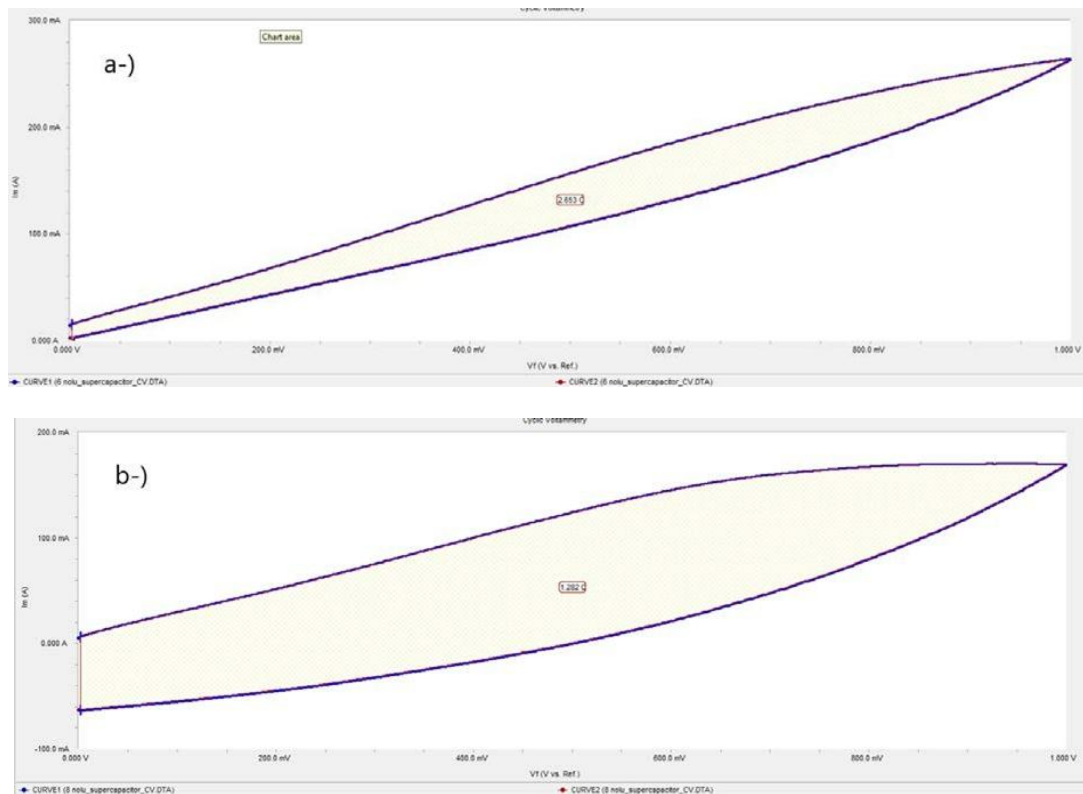


Figure 5. Reflectance, absorbance, transmittance graphs of ZnO/GO sample dissolved in different solvents.

3.6. Cyclic Voltammetry (CV) Analysis

It is the most widely used method to determine the electrochemical properties of manufactured materials. It is an analytical method used to understand at which potentials a system is reduced and oxidised, whether the electrode material is capacitive, whether it is reversible, and whether reagents adhere to the surface [21]. CV is a system that operates with a constant potential scan rate over a given potential range. It is a method that measures the current against the potential applied to the electrode. A triangular wave-shaped potential scan is applied to the working electrode. When the rising potential reaches its extreme point, it returns to the starting point at the same speed [22]. The obtained curves can determine the capacitance values and the service life of the electrodes. If you consider the fully capacitive state of the ideal reversible supercapacitor, a rectangular curve is formed and there is no redox reaction on the surface. But in the case of pseudocapacitors, a capacitance behaviour with redox reaction is observed. In other words, we can obtain information about the thermodynamics and kinetics of redox reactions by CV [23,24].

Figure 6 shows the CV plots of the produced ZnO/GO particles. CV measurements were taken at frequencies between 0.01 Hz and 10 kHz. In the graph, the x-axis (real axis) gives the equivalent series resistance (ESR) of the electrode. In the CV, the semicircular curve, which is observed at low frequencies and expresses the charge transfer resistance at the electrode interface, was not observed. This indicates a rapid charge transfer to the electrode-electrolyte interface. The capacitance value of the sample dissolved with acetone reached the highest value [25-26].



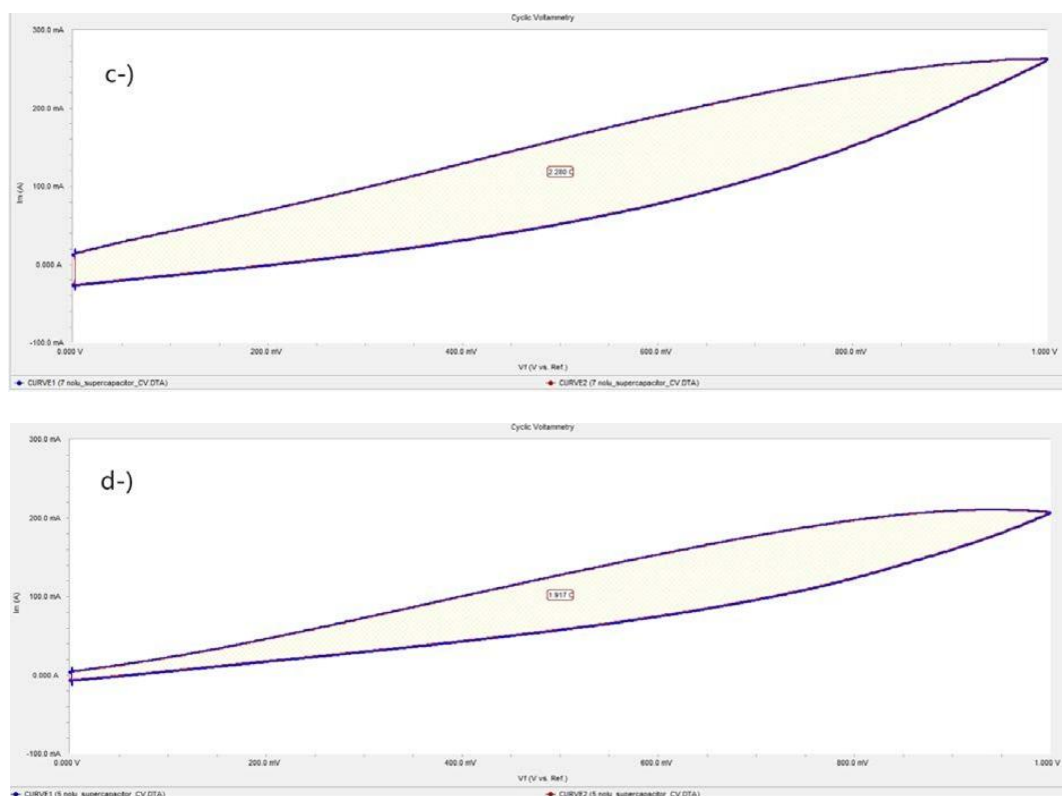


Figure 6. CV plots of ZnO/GO samples dissolved in a-) acetone, b-) 2-Methoxyethanol, c-) ethyl alcohol, d-) pure water solvents.

4. CONCLUSION AND SUGGESTIONS

In this study, ZnO/GO based supercapacitive nanocomposites were obtained by sol-gel method in different solvents using different salts as starting materials. These metal oxides were then doped with GOs produced by Hummers method. XRD, SEM, FTIR, DTA-TGA, UV-Vis-NIR and CV analyses of the samples were performed. Structural, morphological, thermal and optical changes of the obtained composite samples depending on different solvents were investigated. Electrodes for supercapacitor applications were fabricated using the obtained GO/NiO composites. The cyclic voltammogram and impedance spectra of these electrodes were determined by CV analysis. The results obtained were evaluated. When the SEM analysis images of the samples were examined, it was observed that they were nano-sized. It is clearly seen from the images that graphene oxide structures are homogeneously distributed among the obtained metal oxide powders and nanocomposite structures are obtained in accordance with the desired purpose. XRD results showed the presence of metal oxide and graphene oxide phases. It was determined that there were differences in the intensity and width of the peaks obtained with the differentiation of the solvent. In the DTA-TG analysis results, weight loss was observed in all of the samples as a result of the analyses performed in the presence of nitrogen gas for 10 minutes. When FT-IR analysis results were examined, bonds belonging to graphene oxide structure were observed in the peaks. The presence of solvents, metal oxides and hydroxyl groups are also clearly seen in the spectra. UV analyses were applied to examine the optical properties of the samples. The results showed that the samples reached high absorbance values. The results obtained from CV measurements show that the charge storage and discharge stages occur rapidly and reversibly with near-paralogue behaviour at low scan rates and that the double layer formation on the electrode surface is rapidly reorganised against potential changes. The highest capacitance value among the samples was observed in the ZnO/GO sample dissolved with acetone.

In this study, the role, importance and effect of different solvents were investigated. The effects of four different solvents on supercapacitor efficiency are compared and characterised together with the analyses performed.

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Conflict of Interest Statement

There is no conflict of interest between the authors.

Statement of Research and Publication Ethics

The study is complied with research and publication ethics.

Artificial Intelligence (AI) Contribution Statement

This manuscript was entirely written, edited, analyzed, and prepared without the assistance of any artificial intelligence (AI) tools. All content, including text, data analysis, and figures, was solely generated by the authors.

Contributions of the Authors

All authors contributed to the study's conception and design. Cihat AYDIN conducted the analysis, interpretation, writing, and literature review. Material preparation, data collection, and characterization were performed by Oktay Emre YILDIZ. All authors read and approved the final manuscript.

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