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Production of $ZnFe_2O_4$ Doped Carbon Cloth-Based Flexible Composite Electrodes for Supercapacitors

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Keywords
Flexible supercapacitor, Carbon cloth, $ZnFe_2O_4$, Hydrothermal method

Abstract: In this study, it is aimed to develop carbon cloth-based (CC) $ZnFe_2O_4$ doped super capacitor electrode. For this purpose, cotton fabric was first carbonized in a nitrogen atmosphere at 800 °C and turned into a conductive substrate. Then, metal oxide with ZnFe2O4 spinel structure was synthesized from the chlorinated compounds of Zn and Fe elements by hydrothermal method on carbon fabric surfaces. In the results of the XRD analysis of the produced electrodes, it was determined that the $ZnFe_2O_4$ structure was successfully synthesized, but some Fe3O4 and ZnO structures were formed. In the SEM analysis, it was observed that the synthesized structures were formed to completely cover the CC surfaces. Three-electrode system and 3 M KOH were used for the electrochemical performance of the electrodes. Specific capacitance measurements were performed starting from 5 mV/s scanning speed to 100 mV/s scanning speed. According to the results obtained, it was determined that the highest capacitance value was 66 F/g at 5 mV/s speed, the energy density was 2.95 Wh/kg, and the amount of stored charge was 159 C. As a result, it can be said that flexible supercapacitors have been successfully developed, but higher capacitance values can be achieved by optimizing the production conditions.

Süper Kapasitörler için $ZnFe_2O_4$ Katkılı Karbon Kumaş Bazlı Esnek Yapılı Kompozit Elektrotların Üretimi

Anahtar Kelimeler
Esnek süperkapasitör, Karbon kumaş, $ZnFe_2O_4$, Hidrotermal metod

Özet: Bu çalışmada karbon kumaş (CC) bazlı $ZnFe_2O_4$ katkılı süper kapasitör elektrotu geliştirilmesi amaçlanmıştır. Bu amaçla ilk olarak pamuklu kumaş 800 °C'de azot atmosferinde karbonize edilerek iletken substrat haline getirilmiştir. Daha sonra Zn ve Fe elementlerinin klorlu bileşiklerinden hidrotermal yöntemle karbon kumaş yüzeylerinde $ZnFe_2O_4$ spinel yapılı metal oksit sentezlenmiştir. Üretilen elektrotların XRD analizi sonuçlarında $ZnFe_2O_4$ yapısının başarılı bir şekilde sentezlendiği ancak bir miktar Fe_3O_4 ve ZnO yapılarının meydana geldiği tespit edilmiştir. SEM analizinde ise sentezlenen yapıların CC yüzeylerini tamamen kaplayacak şekilde meydana geldiği gözlenmiştir. Elektrotların elektrokimyasal performansları için üç elektrotlu sistemi ve 3 M KOH kullanılmıştır. Spesifik kapasitans ölçümleri 5 mV/s tarama hızından başlanmış 100 mV/s tarama hızına kadar gerçekleştirilmiştir. Elde edilen sonuçlara göre en yüksek kapasitans değerinin 5 mV/s hızda 66 F/g, enerji yoğunluğunun 2.95 Wh/kg ve depolanan yük miktarı ise 159 C olarak tespit edilmiştir. Sonuç olarak başarılı bir şekilde esnek yapılı süper kapasitörlerin geliştirildiği ancak üretim koşullarında optimizasyon yapılarak daha yüksek kapasitans değerlerine ulaşılabileceği söylenebilir.

1. INTRODUCTION

The increase in the global population and the decrease in natural resources have kept human beings in search of alternative energy sources. In this regard, many studies have been carried out to develop clean energy sources such as wind, solar, biomass, hydrogen, geothermal and hydro-thermal energy. One of these studies is triboelectric nanogenerators (TENG) [1], which has been proposed by Wang and his team in recent years. They are designed to convert activities such as walking, running and hand-arm movement into electrical energy, based on the principle of electrification by friction. Being able to benefit from waste movements in daily life thanks to such a development has been accepted as a

very important step in terms of clean energy production and many scientific studies have been carried out in this field since the first day it was proposed [2]. In most of the studies, it has been stated that very high-performance voltage outputs can be obtained depending on the type of material used and the geometric design, but it also brings with it a significant disadvantage such as not being able to provide a constant voltage output in daily use because it is directly dependent on the movement [3]. For this reason, it has been stated by many researchers that there is a need for flexible, lightweight, and high-performance devices where the electricity produced by TENGs can be stored and used with a constant voltage output.

On the other hand, flexible electrical energy storage devices are needed not only for TENGs, but also to meet the needs of consumer electronics such as flexible robots, bio-sensors and new generation mobile phones with flexible screens developed in recent years [4,5]. In this context, Lithium-ion batteries (LIB) and supercapacitors are two commercial power sources that support consumer electronics devices. Supercapacitors play an important role especially in flexible devices due to their higher power densities (>10 $kWkg^{-1}$), fast charge-discharge characteristics and longer operating life than LIBs [6–8]. However, the energy density of supercapacitors (~5 $Whkg^{-1}$) is significantly lower than that of LIBs (~150 $Whkg^{-1}$), and this parameter needs improvement to meet the performance demand for next generation flexible devices [8,9]. In addition to power and energy densities, areal performance parameters such as capacitance per unit area are considered important performance indexes for flexible supercapacitors. Therefore, higher power and energy densities per unit area are demanded from flexible supercapacitors compared to existing energy storage devices [10]

One of the basic components that gives flexibility to an energy storage device is a flexible and conductive substrate that can be used as a current collector [11]. In this context, textile products are considered as ideal substrates due to their low cost, flexibility and highly porous structure that can absorb active electrode materials [12]. However, since such products are insulators, they do not act as current collectors. Therefore, it has been stated in the literature that such flexible fabrics can be easily converted into a conductive substrate without losing their flexibility by carbonizing with a short heat treatment of one hour in an inert gas environment at 800 °C [13]. Such carbon fabrics, which can be obtained with a very simple and inexpensive method, have become one of the best substrate materials developed in recent years for flexible supercapacitors [14]. However, it has been stated that such fabrics exhibit a very low capacitance value when used without being modified with another active material [15].

Surface modification or materials development processes have been carried out by considering the charge storage mechanism of supercapacitors. In this regard, it has been stated that a supercapacitor has two types of charge storage mechanisms, electrochemical double-layer capacitance (EDLCs) and pseudo capacitance (PC) [16]. EDLCs have a longer cycle life by storing charge by adsorption-desorption of ions at the electrode-electrolyte interface, while PCs have a higher capacitance value by storing energy through a fast surface redox reaction. However, in recent studies, it has been revealed that hybrid materials containing both PC and EDLC are more effective in terms of both cycle life and specific capacitance [17–19]. Therefore, it is thought that the development of hybrid electrode materials is important in terms of increasing the charge storage performance of supercapacitors. In this context, it is known that materials with large surface areas such as activated carbon, carbon nanotube, graphene and graphene oxide have been widely used to increase the EDLC property of a current collector electrode [20]. However, it has been stated that nanomaterials such as carbon nanotubes and graphene also have significant disadvantages such as high production costs and easy agglomeration due to electrostatic interaction during doping on the electrode surfaces [21]. Therefore, the synthesis of metal oxides on the metal substrate surface has been directed. Thus, it is aimed to expand the surface area and to benefit from the reduction and oxidation properties of oxides. In this context, oxides of many transition metals such as RuO_2, MnO_2, Co_3O_4, NiO have been used for pseudocapacitive purposes [22–24]. However, spinel transition metal oxide (AB_2O_4) emerges due to its unique electronic structure and the use of two metal elements. Among them, $ZnFe_2O_4$ structure is one of the most promising spinel transition metal oxides due to its high electrical conductivity and electrochemical activity [25–27].

In this study, it is aimed to first carbonize cotton fabric and use it as a conductive substrate in order to develop a cotton flexible supercapacitor. Then, $ZnFe_2O_4$ metal oxide was synthesized on this fabric surface by hydrothermal method. XRD and SEM analyses were performed for characterization. The electrochemical performance of the produced electrode was determined by determining the specific capacitance, energy density and the amount of stored charge.

2. MATERIALS AND METHOD

2.1. Materials

$ZnCl_2$ and $FeCl_3$ used in this study were purchased from Aromel kimya medical A.Ş. Urea and ammonium bifluoride were from Labshop41 company. Cotton fabric was met from unused home textile products. Pure water, acetone, ethanol, other laboratory materials and heat treatment furnace were provided from Karabuk University Materials Research and Development (MARGEM) laboratories.

2.2. Production of Carbon Cloth from Cotton Fabric

Carbon cloth (CC) planned to be produced to be used as electrode material were obtained from 100% cotton fabric according to the procedure in the literature [28]. For this purpose, a 1:1:1 mixture of ethanol, pure water and acetone was first mixed in a beaker, and a piece of cloth cut in 5x5 cm dimensions was immersed in this

mixture for 10 minutes and then dried in an oven at 60 °C for 48 hours. Thus, organic residues on the fabric were removed. Then, the piece of fabric was taken to the atmosphere-controlled tube furnace and heated up to 800 °C with argon gas with a temperature increase of 5 °C per minute and kept at this temperature for 60 minutes. Then, when the temperature of the oven reached room condition, the cover was opened, and the carbonized fabric piece was taken. The electrical conductivity of each fabric to be obtained was checked with a multimeter. In this context, the images of the carbon fabric (approximately 5.6 mg/cm^2) produced as an example are given in figure 1.

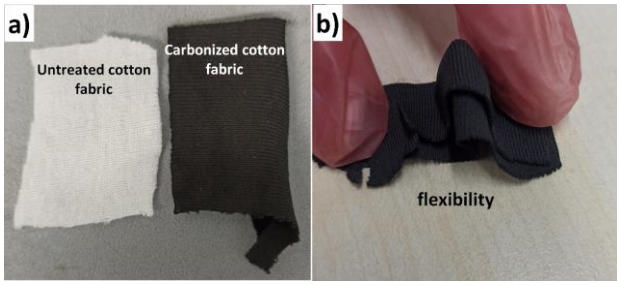

Figure 1. Digital images of a) heat treated and untreated fabrics and b) flexibility of carbon cloth

2.3. $ZnFe_2O_4$ Synthesis on CC Surfaces

Metal oxide synthesis on CC surfaces was carried out by hydrothermal method. $ZnFe_2O_4$ synthesis on CC surfaces was carried out by hydrothermal method. For this purpose, firstly, 100 mg of $ZnCl_2$ and 200 mg of FeCl3 metal salts were dissolved in 20 ml of distilled water. These amounts were adjusted to be 1:2 according to the stoichiometry in the formula. Then, 130 mg ammonium fluoride (NH_4F) and 300 mg urea (CH_4N_2O) were added to the solution as surfactant and mixed with a magnetic stirrer until a clear solution was obtained. These additions, on the other hand, have been applied for the ion mobility in the solution and the easy attachment of oxide crystals to the CC surfaces, as stated in the literature [29]. After these processes, the solution was transferred to an autoclave with a capacity of 30 ml, made of Teflon inside and stainless-steel outside, together with the previously prepared carbon fabric. It was then placed in the oven with its mouth closed and left at 100 °C for 10 hours. At the end of 10 hours, it was taken from the autoclave oven and cooled naturally to room conditions. When cooling was achieved, metal-added carbon fabrics taken from the autoclave were dried on the watch glass in an oven at 60 °C for 12 hours. Then, it was heated up to 200 °C with a heating rate of 10 °C per minute in another furnace and calcined was achieved by keeping it at this temperature for 2 more hours. Finally, the weight of the obtained sample was weighed, and the amount of metal oxide and total active substance coated on the surface were determined. A brief schematic representation of this process was given in Fig. 2.

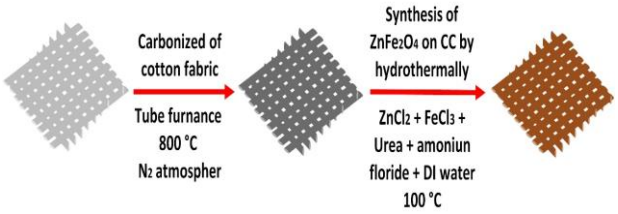

Figure 2. Schematic representation of production setup

2.4. Characterization and Electrochemical Measurement

XRD analysis was performed by using a Cu-based X-ray diffraction instrument (Rigaku Ultima IV) with fixed monochromator in the range 15–90° at 40 kV and 40 mA to characterization of crystal structure of synthesized $ZnFe_2O_4$. Microstructure examination of ZnFe2O4 crystals synthesized on carbon fabric surfaces was carried out at different magnifications with scanning electron microscope (Carl Zeiss Ultra Plus Gemin) equipped with secondary electron detector after coated with platinum. Electrochemical cyclic voltammetry measurements were performed with Parstat 4000 at room condition. For this purpose, a three-electrode cell was first established. Graphite rod was used as counter electrode, Ag/AgCl as reference electrode and $ZnFe_2O_4$ doped carbon cloth as working electrode. 3 M KOH solution was used as the electrolyte. The measurements were carried out at different scan rates in the range of -0.5 to +0.5 V and 0.1 and 0.5V. The specific capacitance C_s was calculated according to Eq. (1):

$$C_s = \frac{I}{m \times \frac{dV}{dT}} \quad (1)$$

where I is average current, m is amount of doped $ZnFe_2O_4$ (about 3-5 mg) and $\frac{dV}{dT}$ is scanning rate (mV/s). Energy density of electrodes E were calculated using Eq. (2) [30]:

$$E = \frac{1}{2} C_s V^2 \quad (2)$$

where C_s specific capacitance (F/g) and V potential difference. Amount of stored charge of electrodes were calculated using Eq. (3) [30]:

$$q = C_s \times m \times \Delta V \quad (3)$$

where m is amount of doped $ZnFe_2O_4$, q is stored charge and ΔV is change of potential difference.

3. RESULT AND DISCUSSION

For the characterization of the produced electrodes, firstly, XRD analysis were carried out. For this purpose, the results of the analysis after the pure and hydrothermal treatment of the carbonized fabric are given in Fig. 3. According to these results, the broad peak observed between 18° and 30° indicates the amorphousness in the fabric and the carbon crystals in the inner structure of the cloth [31]. It is observed that many sharp peaks appear after the hydrothermal

treatment. These indicate the presence of crystal structures on the fabric surface. XRD analysis of the post-synthesis fabric shows that the peaks at approximately 27°, 35°, 43°, 53°, 56°, and 68° indicate (220), (311), (400), (422), (511) and (440) planes of cubic ZnFe2O4, respectively [32]. At the same time, it is thought that the sharp peak observed at about 27° may be due to the crystallization of the carbon fabric resulting from the heat treatments [31]. Apart from this, the peaks observed at approximately 34°, 47°, 62° and 66° originate from the (002), (102), (103) and (200) planes of the hexagonal ZnO phase, respectively [33]. There is also a peak observed at about 39°, which originates from the (006) plane of the alpha-structured hematite (Fe_2O_4) phase [34]. Based on these results, it can be said that $ZnFe_2O_4$ crystal structures were successfully synthesized on carbon fabric surfaces after hydrothermal treatment, but at the same time, small amount of impurities such as Fe_2O_3 and ZnO phases occurred within these crystal structures.

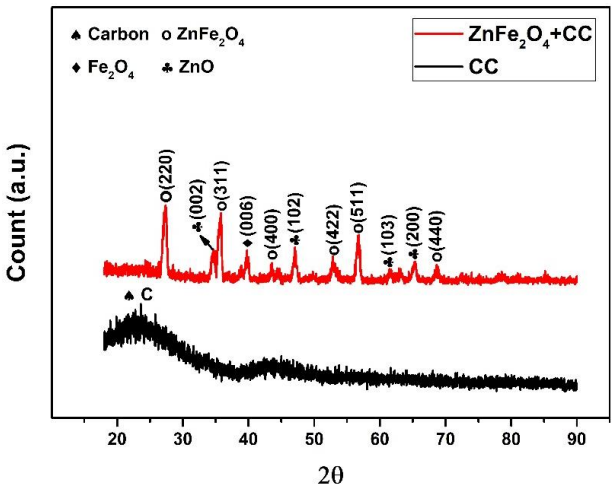

Figure 3. XRD results of pure and $ZnFe_2O_4$ doped CC electrode

The formation of $ZnFe_2O_4$ structures on the fibre surfaces was investigated by SEM device. Figures 4a and b show the surfaces of the untreated carbon fabric. In these images, the fibre surfaces were observed quite smooth fibre and diameters of its were measured to be 7-8 microns on average. After the hydrothermal treatment, all fibre surfaces were surrounded by a new spherical-like structure, as can be seen in figures 4c and d. Based on the XRD results, these structures were thought to be mostly composed of $ZnFe_2O_4$ crystals. The strong adhesion of these structures, which are formed on the surface of the fibre in an interlocked form, strengthens the fibre-metal oxide interface interaction. For this reason, it can be said that the hydrothermal process has been carried out successfully, making the fibre surfaces highly functional. For this reason, it can be said that the hydrothermal process has been carried out successfully, making the fibre surfaces highly functional. However, there is a disadvantage observed in this case, which is that the metal oxides formed are larger in size than expected. The fact that these structures, which have reached the size of about 1-2 microns, actually occur in submicron sizes, could have made the fibres have a larger surface area. It is thought that this problem can be solved by reducing the 10 hours specified during production or by adding a smaller amount of reactants.

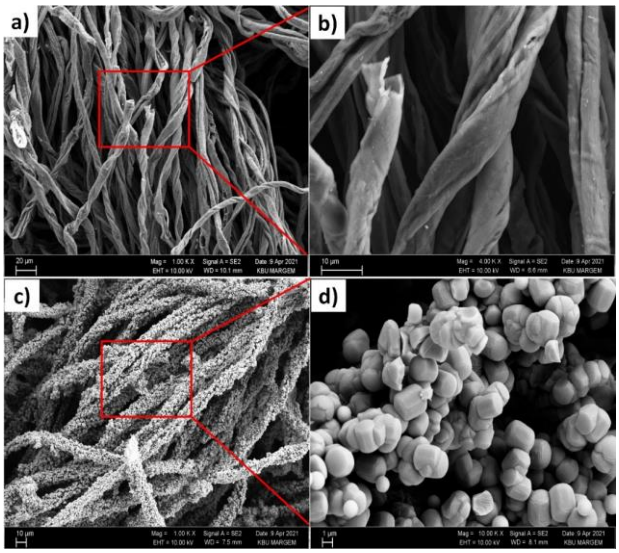

Figure 4. SEM images of a-b) pure CC, c-d) ZnFe2O4 doped CC electrode

In order to evaluate the energy storage amount of the produced electrodes, cyclic voltammetry measurements were carried out. These measurements were first performed on pure CC and $ZnFe_2O_4$ doped CC at scanning rate of 50 mV/s between 0.1V–0.5 V potential. Obtained results were given in the graph in figure 5. According to these results, pure CC did not show any faradic current, only EDLC feature was present. In other words, there was no reduction oxidation reaction between the surface and the electrolyte in terms of ion storage. This shows that it does not have a pseudocapacitive feature. Since the area in the middle of the current-voltage graph is very small, the load storage capacity is also very low. As stated in the literature, pure CC is a good substrate, but the necessity of increasing the charge storage capacity by functionalizing the surface was confirmed by this result. On the other hand, when looking at the $ZnFe_2O_4$ doped CC, there were two different peaks. These peaks were due to the oxidation and reduction properties of iron in $ZnFe_2O_4$ to +3 and +2.

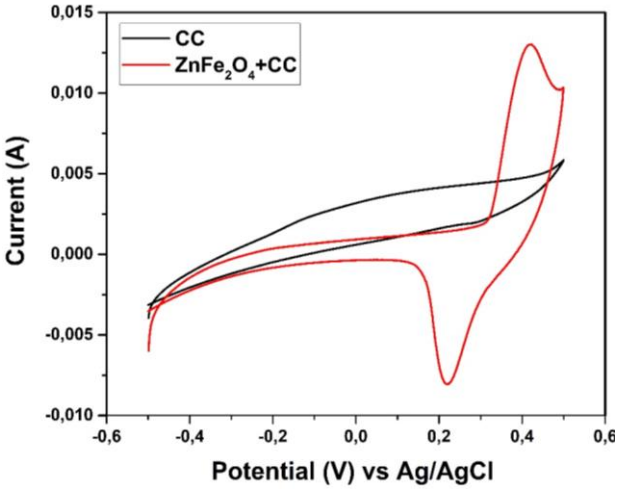

Figure 5. Cyclic voltammetry of pure and $ZnFe_2O_4$ doped CC electrode at 50 mV/s scan rate

Since the $ZnFe_2O_4$ doping process was successful compared to pure CC, cyclic voltammetry measurements were also performed on the doped CC at different scanning rates. In these measurements, the scanning rate was started from 5 mV/s to 100 mV/s and the results were given in figure 6 between 0.1 V and 0.5V. In addition, the specific capacitance, energy density and power density calculations at each scanning speed of the produced electrode were also made according to these results and all the results were given in detail in Table 1. According to these results, the highest capacitance value was determined as 66 F/g at a scanning speed of 5 mV/s. It was observed that this value decreased by approximately 50% when the scanning rate increased to 10 mV/s and regressed to 34 F/g. However, when the scanning rate was increased up to 100 mV/s with a scanning speed of 10 mV/s, it was observed that the decrease did not continue at the same rate and the lowest was 19 F/g. Similarly, it was calculated that the energy density decreased from 2.95 to 1.54 before then to the lowest 0.863 Wh/kg. It was calculated that the amount of stored cargo decreased from 159 C to 46 C. According to these results, there is a significant change in all values with the increase in scanning speed. The reason for this is directly related to the electrode-electrolyte interface interaction. Namely, $ZnFe_2O_4$ coated on the CC surface consists of a highly indented porous structure. It is believed that at low scanning rates, this structure interacts with the electrolyte longer, and therefore the charges in the electrolyte stick to the surface more. For this reason, it can be said that higher capacitance value is reached at low scanning speeds. On the other hand, it is believed that in higher speed scans the electrolyte does not have enough time to penetrate the surface of the active substance and therefore stores a lower charge [35].

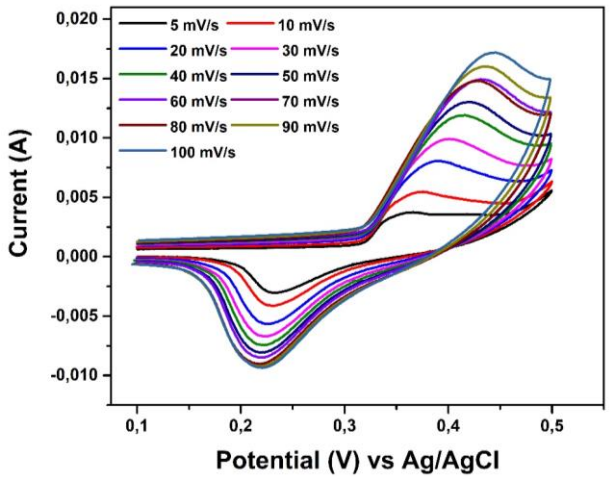

Figure 6. Cyclic voltammetry of $ZnFe_2O_4$ doped CC electrode at different scan rate

Table 1. Specific capacitance, energy density and power density of $ZnFe_2O_4$ doped CC electrode

Scan rate (mV/s)	Cs (F/g)	E (Wh/kg)	q (C)
5	66.27	2.95	159.05
10	34.69	1.54	83.27
20	33.89	1.51	81.34
30	31.19	1.39	74.86
40	28.93	1.29	69.45
50	26.5	1.18	63.62
60	24.69	1.10	59.26
70	22.98	1.02	55.18
80	20.44	0.908	49.05
90	19.01	0.845	45.63
100	19.4	0.863	46.59

4. CONCLUSION

In this study, it is aimed to develop a flexible supercapacitor to keep up with the new developing technology. For this purpose, cotton fabric as a substrate has been successfully carbonized to make it conductive and flexible. Then, $ZnFe_2O_4$ structure was successfully synthesized on the surfaces by hydrothermal method. In XRD and SEM analyzes, it was determined that ZnO and Fe_3O_4 structures were formed to some extent together with $ZnFe_2O_4$. In the electrochemical measurement results, it was determined that the highest specific capacitance was 66 F/g at a scanning speed of 5 mV/s, the energy density was 2.59 Wh/kg, and the amount of stored charge was 159 C. It is thought that these results can be used in supercapacitors, but higher performances can be obtained if the production parameters are optimized.

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