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Anode Performance of Sustainable, Hemp-derived, Flexible, Binder-free, Carbon Fabrics in Lithium-Ion Batteries

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Abstract

Fabrication of sustainable products are of significance from many aspects recently. Industrial hemp as one of the most sustainable, environment friendly plant can be used for many applications. In this study, various sustainable, hemp-derived, binder free, flexible anode materials were prepared by the two-step carbonization method. Plain woven hemp fabric was used as a starting material. Fabrication of hemp-derived anode materials were carried out in two steps known as stabilization and carbonization. While the stabilization step was performed at 220 °C for all samples, carbonization was carried out at 600, 700, 800 and 900 °C in order to optimize the carbonization process. Morphological, electrical and electrochemical characterization of the hemp-based carbon fabric anodes were carried out. Electrical resistance of the hemp-based carbon fabric anodes showed differences depending on the carbonization temperature. Electrochemical results showed that 800 °C is the optimum condition in terms of carbon yield and cell performance if the reversible capacity, cycling stability and rate capability values are considered.

Keywords: Hemp fabric, Anode, Li-ion battery, Energy storage

Introduction

The importance of sustainable engineering applications has increased recently because of serious environmental regulations. Renewable, sustainable, cheap and environmentally friendly raw materials have been preferred in industry. Hemp, as one of the most sustainable plants, attracts great deal of attention for many reasons. Firstly, hemp agriculture is easy and almost no pesticide is required. Secondly, production yield is high, and it can be harvested more than once every year in the proper climate conditions. Thirdly, it has relatively low carbon footprint when compared with other natural polymers. Because of above mentioned advantages, hemp can be used in many industries. Cellulose, hemicellulose, lignin and pectin are the main components of the hemp plant. Since cellulose is mostly found in the fiber form, for many years hemp has been used in the textile and paper industry (Brosius, 2006; Horne, 2020). In addition to these, hemp has been used as a carbon source recently for many systems. In these studies various forms of hemp was used including hemp dust (Rosas, Bedia, Rodríguez-Mirasol, Cordero, research, 2008), hemp hurd (Liu et al., 2017), hemp stem (Yang et al., 2014), hemp canes (Rosas et al., 2008) and hemp fibers (Hossain et al., 2018; Mijailović et al., 2017). The hemp-derived carbon materials obtained from these studies generally showed morphologies such as activated carbon (Liu et al., 2017), activated carbon monoliths (Rosas et al., 2008) and activated carbon fibers (Mijailović et al., 2017), interconnected carbon nanosheets (Wang et al., 2013) As known, activated carbon is generally used to increase the adsorption

performance of any system including gas (Liu et al., 2017), water vapor (Rosas et al., 2008), metal (Yang et al., 2014), pesticide (Vukcevic et al., 2012) etc. In addition to such applications hemp-derived carbon was investigated for its energy storage properties (Guan et al., 2019; Mijailović et al., 2017; Shi et al., 2019; Sun et al., 2016; Tan et al., 2020; Wang et al., 2013; Xiong et al., 2015; Yang et al., 2017).

In one of these studies carbon fibers were obtained from hemp fibers and activators were used in order to obtain activated carbon fibers. Process conditions esp. carbonization temperature was found significant in terms of specific capacitance (Mijailović et al., 2017). In another study various parts from the hemp plant were used in order to understand the effect of precursor morphology. Hemp hurd and hemp bast were used in order to obtain 2-D activated carbon structures. Hemp hurd was determined as a better precursor. Porosity and surface area were reported to be directly related with the performance of the capacitors (Sun et al., 2016). Wang *et al.*, synthesized hemp-derived interconnected carbon nanosheets and investigated the capacitor performance. As reported in the study that hemp-derived unique morphology was found efficient as a supercapacitor material (Wang et al., 2013).

As known for the energy storage not only capacitors but also batteries can be used. When recent technology, costs and requirements are taken into account secondary batteries are of vital importance for the energy storage. Although there are many studies carried out in the area of hemp-derived capacitors as given above; the number

of studies for hemp-derived Li-ion batteries are limited (Guan et al., 2019; Um et al., 2018). As far as known, there are two studies in the literature. The first study for the development of hemp-based anodes for lithium-ion batteries (LIBs) was carried out by Um *et al.* in 2018 (Um et al., 2018). In the study hemp stem was grounded into small particles (< 1.5 mm). Hemp was carbonized at 800 °C for 2h under N_2 . For the activation process stem was used under N_2 flow at 800 °C for 2h. Then one part of the material was ball-milled and other part kept as fabricated (natural hemp). Anode performance of both samples in LIBs were investigated under 300 mA g^{-1} current density and samples were reported to show lower capacity values until 20 cycles and stable capacity between 20-100 cycles. Ball-milled sample showed higher capacity values throughout the 100 cycles. Charge capacity values were determined as 190 and 300 mAh g^{-1} for natural hemp and activated hemp, respectively. Second study was carried out by Guan *et al.* in 2019. In this study, pulverized hemp stems were thermally treated at 300 °C for 3 h under argon atmosphere. Following that, this precursor was mixed with $ZnCl_2$ and processed at 500 - 800 °C for 3 h. Samples were grounded and treated with HCl and washed with water and activated carbon was obtained. In order to compare the performance of hemp-derived activated carbon, hemp-derived carbon was synthesized. Amorphous activated carbon materials were synthesized with sheet-like morphology and showed a high reversible capacity of 495 mAh g^{-1} after 100 cycles (Guan et al., 2019).

As obvious from the literature hemp fabric was not used as an anode source for the LIBs. Until the fabric form is obtained, many different processes are applied to the hemp plant. Although there are many steps, the most important steps are peeling off and/or mechanical separation of the fibers, spinning of the fibers and weaving. In all these steps fibers are separated from some other components (hurd etc.) and they are aligned. When fabric and other forms of hemp (hurd, stem, outer layer fibers, fiber ribbons etc.) are compared some significant differences become prominent. Firstly, fiber ratio is higher, secondly fibers and yarns are aligned. In addition to these, after the carbonization, flexible and binder free anode can be obtained. No extra step is required for the preparation of negative electrode. The most important novelty of the study stems from the fact that it is suitable for industrial production. Since fabric can be produced as desired, anode morphology and performance can be controlled. On the other hand, for other forms of hemp such as hurds, ribbons and stem repeatability of the system in terms of morphology (particle geometry, particle size distribution, porosity etc.) is difficult that is probably due to structural variations and contaminations.

In this study, hemp fabric was used for the preparation of flexible, binder-free anode materials for LIBs for the first time in the literature. Fabric samples were carbonized at different temperatures in order to obtain the best anode material. Following that flexible, binder-free hemp-derived fabric anodes were used to fabricate cells. Morphological, structural, electrical and

electrochemical performance of the hemp-derived anodes were investigated.

Materials and Methods

Plain fabric made out of hemp yarn was kindly supplied from CC Textiles (Istanbul, Turkey). Hemp fabrics were purified order to remove surface finish. For the purification, hemp fabric was cut into small pieces (25 mm x 50 mm) and they were soaked into acetone at 40 °C for 24 hr. After washing with distilled water, they were soaked into distilled water at 80 °C for 24 hr. Then, samples were washed with distilled water 3 times and vacuum dried at 80 °C for 24 hr. At the end of this process, weight loss was $\approx 2.67\%$.

Thermogravimetric Analysis (TGA)

Before the synthesis of the anode material, thermal gravimetric analysis (TGA) of the hemp fabric performed for the analysis of the thermal behavior. TGA was carried out by Seiko, TG/DTA 6300 between 25 and 950 °C at a rate of 10 °C min^{-1} under nitrogen atmosphere with a gas flow of 200 ml min^{-1} . TGA and derivative thermogravimetric (DTG) curves can be seen from Fig. 1. Thermal degradation of hemp fabric started 250 °C and ends around 380 - 400 °C.

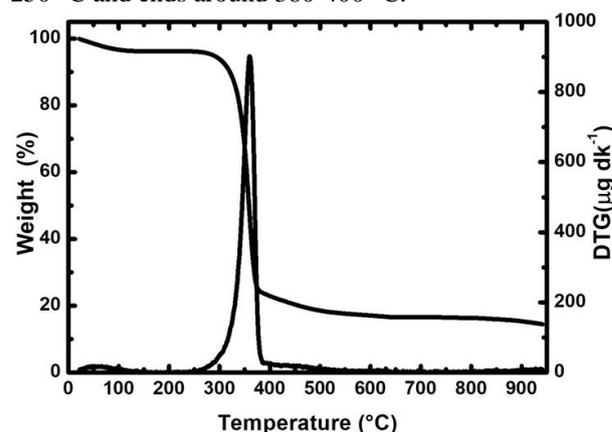


Figure 1. TGA and DTG curves of the hemp fabric

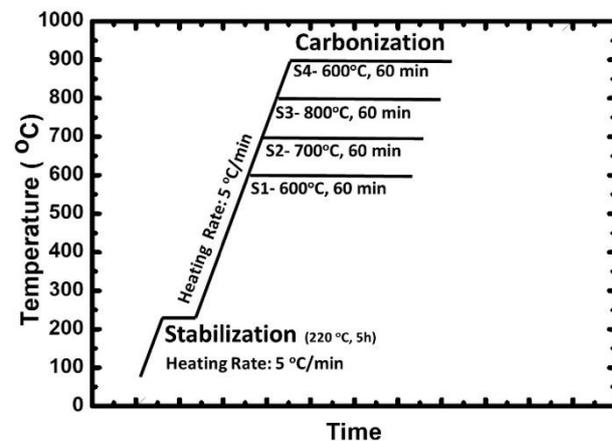


Figure 2. Thermal treatment conditions of hemp fabric

By considering the TGA data, hemp fabric samples were heat-treated in 2 steps in order to get conductive negative electrode for LIBs as shown in Fig. 2. The process was carried out by using a tube furnace (OTF-1200X, MTI). In the first step, samples were stabilized at 230 °C for 5

h under air atmosphere. After this process weight loss was around 5%. Then, samples were carbonized at four different temperatures (600, 700, 800 and 900 °C) for 1 h under N₂. Heating and cooling rates were kept constant (5 °C min⁻¹) during heat-treatment.

Sample codes, carbonization temperature and carbon yield of the hemp-derived anodes can be seen from Table 1. During carbonization, hemp fibers turned into electrically conducting carbon fibers. At the end of this process 4 different hemp-derived carbon anodes were obtained.

Morphology

The morphology of the hemp fabric and hemp-derived carbon fabrics was analyzed by an optical microscope instrument (BX51M, Olympus) at 5, 10 and 20 x.

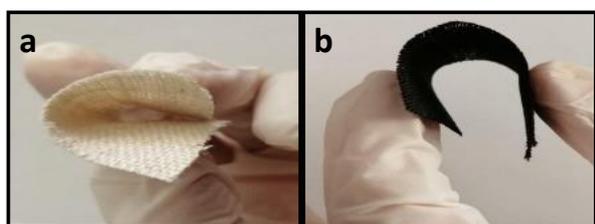


Figure 3. a) Hemp fabric and b) carbonized hemp fabric

Table 1. Sample codes and corresponding carbonization conditions, carbon yield, 4-probe resistance and thickness values of the samples

Sample Code	Carbonization Temperature (°C)	Carbon Yield (%)	Resistance (Ω)	Thickness (mm)
H-600	600	16.25	8.14*10 ⁴	0.45
H-700	700	15.62	6.72*10 ²	0.45
H-800	800	14.96	1.67*10 ¹	0.48
H-900	900	14.85	6.33*10 ⁰	0.48

Resistance

The resistance of the samples was measured by a system consists of 4-probes that were connected to a current source (Keithley 6221) and a nanovoltmeter (Keithley 2182A) under an input current of 1 μA. 4 measurements were taken, and average values were calculated. Before the resistance measurements thickness of the samples were determined by a digital multimeter

Electrochemical Characterization

Electrochemical characterization was performed using CR2032-type cells. Half-cells were prepared in atmosphere-controlled glove-box. Half-cell consisted of an anode (binder-free and free-standing carbon fabric on copper-foil), a lithium metal reference electrode, electrolyte and a Celgard 2400 separator. The electrolyte was 1M LiPF₆ in EC:DMC:DEC (1:1:1 in volume ratio). Cyclic tests were galvanostatically performed between 0.01-2 V at 50 mA g⁻¹.

Results

In order to examine the morphology, samples were analyzed by an optical microscope. Fabric morphology was analyzed at different magnifications (5, 10, 20 x). While low magnification is useful in terms of

observation of wider area and observing the fabric morphology, higher magnifications are useful for observing the fiber morphology. As shown in Fig. 4, untreated hemp fabric is a plain fabric and consisted of weft and wrap yarns. As obvious from Fig. 4, anode samples were carbonized without losing the plain fabric morphology regardless of the process conditions. However, weft and warp yarns shifted from the fabric axis (x, y). This is probably caused by the soft and flexible nature of the carbon fabric (Fig. 3 b).

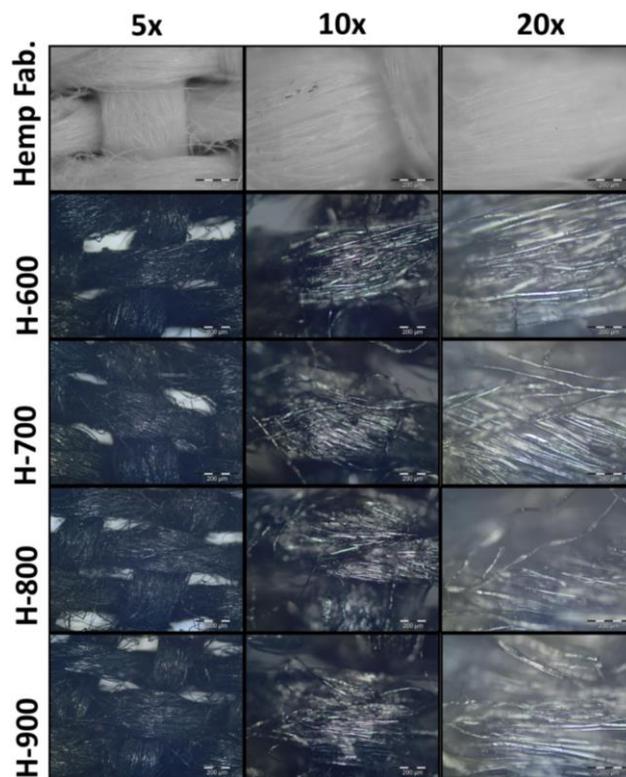


Figure 4. Hemp fabric and carbonized hemp fabrics at 5, 10 and 20x magnifications

In addition to that weft and wrap yarns showed some level of shrinkage. As can be seen, the fabric porosity increased with the increment in carbonization temperature. This is parallel with the carbon yield of the samples. As seen from Table 1, carbon yield values were 16.25, 15.62, 14.96 and 14.85 % for H-600, H-700, H-800 and H-900, respectively. That means around 85% of the raw material was lost. As reported in the literature this value is between 10-30 % for cellulose based carbon fibers and This is probably caused by depolymerisation of the macromolecular chains and removal of carbon in the form of CO, CO₂ and tar etc. (Dumanlı Windle, 2012; Huang, 2009) Although samples showed different carbonization yields, this was not observed in optical microscope images.

4-probe electrical resistance values of samples can be seen from Table-1 and Fig. 5. As obvious from these results, carbonization temperature directly affected the electrical resistance values (ERV) of the samples. While H-600 had ERV of 8.14*10⁴ Ω, H-700 showed ERV around 6.72*10² Ω. Two orders of magnitude drop were observed for 100 °C increase in carbonization temperature. Similar decrease was observed for H-800

and H-900 those showed ERV of 1.67×10^1 and $6.33 \times 10^0 \Omega$, respectively. Especially H-800 and H-900 can be used as a conductive filler for many applications. As known, cellulose based fibers do not melt during thermal treatments and good source of carbon fibers. However, these non-melting polymers lead to formation of non-graphitizable carbon fibers (Dumanlı Windle, 2012; Ishida et al., 2004; Watt Sciences, 1970). As mentioned before by increasing the carbonization temperature carbon yield decreased. That probably led to higher level of collapsing between fibers and decrease in the fiber – fiber distance. All these facilitated the current flow and ER values decreased (Kwon et al., 2013).

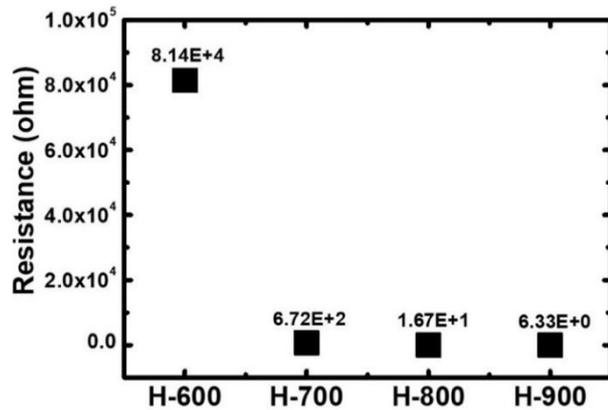


Figure 5. 4-probe resistance values of samples

The initial charge/discharge behavior of half cells made out of carbonized hemp fabric anodes were shown in Fig. 6a. Samples were charged and discharged in the voltage range of 0.001 – 2 V at 50 mA g^{-1} . The initial charge capacities of all the samples decreased in the following order: H-800 > H-700 > H-600 > H-900 and the initial discharge capacities of all samples decreased in the following order: H-800 > H-700 > H-900 > H-600. Among the all samples, H-800 exhibited the best performance with discharge and charge capacities of 1603 and 538 mA h g^{-1} , respectively. The coulombic efficiency of this electrode was 33.56%. The first cycle coulombic efficiencies of H-600, H-700, H-800 and H-900 were 33.71, 33.72, 33.56, and 47.24%, respectively. The irreversible capacity loss can be connected with the formation of an SEI layer at the surface of the carbonized hemp fabric anodes due to reduction of the electrolyte (around 0.5 – 0.7 V). Another reason for this capacity loss might be irreversible lithium insertion nearby residual H atoms in the carbon material (Asenbauer et al., 2020).

In Fig. 6b, the charge and discharge capacities of carbonized hemp fabric anodes at different carbonization temperatures were compared for 50 cycles. Accordingly, H-900 showed the most stable cyclic performance. Charge capacity of H-900 changed from 382 to 368 mA h g^{-1} between 2nd and 50th cycle. Capacity values decreased slightly during first 10 cycles for H-600, H-700 and H-800. Charge capacities of H-600, H-700 and H-800 changed from 291, 481, 531 to 312, 343, 438 mA h g^{-1} between 2nd and 50th cycle, respectively.

General specific capacity values of H-700, H-800 and H-900 were higher than theoretical capacity of graphite (372 mA h g^{-1}). This might be due to the existence of internal porosity, structural defects and low crystallinity of carbonized hemp fabric. These can create much more space for lithium insertion (Wang et al., 1995). After 10 cycles, capacity values did not change crucially. Among the samples, H-600 showed the worst cyclic performance in terms of capacity. Its low capacity values might be attributed to its high resistance ($\sim 8.14 \times 10^4 \Omega$) (Striebel et al., 2004).

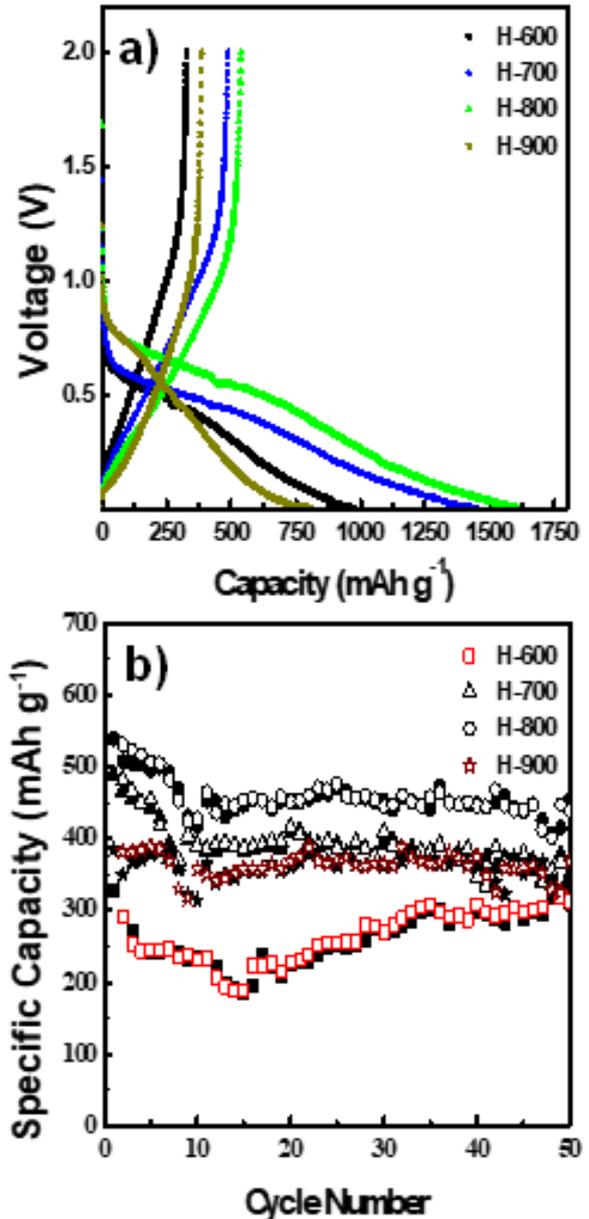


Figure 6. a) The initial charge/discharge curves and b) the cyclic performance of carbonized hemp fabric anodes at different temperatures

Discussion and Conclusions

In this study, anode performance of hemp-based, binder-free, flexible carbon fabric has been evaluated first time in the literature. The effects of carbonization temperature on physical and electrochemical properties of hemp-derived carbon fabrics have been studied. Among

different samples, H-800 gave the best performance in terms of capacity, stability and carbon yield. The better performance might be attributed to high electronic conductivity, aligned Li-ion pathway due to aligned nature of fibers in the fabric, more active sites and amorphous nature of carbon compared with graphite.

The strategy presented in this study could be extended to other cellulose-based fabrics or materials for the next generation green energy storage applications or environmentally friendly flexible electronics.

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