ISSN: 1410-8917
e-ISSN: 2597-9914

Jurnal Kimia Sains dan Aplikasi 26 (8) (2023): 293-299

Jurnal Kimia Sains dan Aplikasi
Journal of Scientific and Applied Chemistry

Journal homepage: http://ejournal.undip.ac.id/index.php/ksa

Synthesis and Electrochemical Properties of SnO_2 Composited Activated Carbon from Coffee Ground Waste for Supercapacitor Applications

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https://doi.org/10.14710/jksa.26.8.293-299

Article Info

Article history:

Received: 21st August 2023
Revised: 06th November 2023
Accepted: 07th November 2023
Online: 15th November 2023

Keywords:
waste coffee ground; tin oxide; composite; electrochemistry; activated carbon

Abstract

Biomass has been considered an alternative source of electrode materials. Converting biomass into activated carbon is one of the possible approaches. Coffee ground waste is abundant as the world's coffee-drinking culture grows. This paper describes a study that converted coffee grounds into activated carbon and tested its feasibility for electrode materials. We use a simple pyrolysis technique (800°C) to synthesize activated carbon from waste coffee grounds, with potassium hydroxide (KOH) as an activator. Tin oxide (SnO_2), which has a high theoretical capacity, was impregnated into the carbon framework using a hydrothermal method operating at 180°C for 2 hours. The X-ray diffraction (XRD) pattern and Fourier transform infrared spectroscopy (FTIR) results show that SnO_2 was successfully impregnated into the carbon structure. Raman analysis also shows that the carbon structure of the activated carbon still retains despite the presence of the metal oxide during the hydrothermal synthesis. Furthermore, electrochemical measurements utilizing the galvanostatic method using a three-electrode system demonstrated that the specific capacitance of the material increased by approximately 106% at 5 A/g following SnO_2 impregnation. Long cycle testing further demonstrates that including SnO_2 in the carbon, structure may sustain outstanding performance even at high current densities of 5 A/g for 100 cycles with 99% capacity retention. The results demonstrate the possibility of activated carbon from coffee ground waste composited with tin oxide as supercapacitor electrodes.

1. Introduction

Today, the world still relies on limited fossil energy as its primary energy source, which has already become a severe problem due to global warming. As a solution, alternative and renewable energy development is intensively carried out to explore new energy storage and conversion technologies. One of the energy storage devices that are attractive is the supercapacitor. Supercapacitors can provide higher power density than batteries and energy density than dielectric capacitors [1, 2]. The working principle of this supercapacitor itself is that when charging, the existing electrons will be deposited, resulting in a potential difference between the electrodes. When a circuit causes these two electrodes to be connected, electrons will flow through the circuit, and the existing electrical energy can be utilized according to the user's needs. Electrode materials are one of the main factors determining the performance of supercapacitors and have become the focus of ongoing research [3].

Supercapacitors work via two mechanisms: the Electric Double-Layer Capacitor (EDLC) and the pseudocapacitor. The EDLC process stores energy using electrostatic interactions between electrolyte ions and the electrode surface. This mechanism is incredibly fast and responds quickly to voltage changes. The second is pseudocapacitors, which store energy through electrode reactions that involve charge storage. They conduct redox reactions during the charging and discharging operations, allowing quick and reversible charge transfer at the electrode-electrolyte interface. Pseudocapacitors have a higher capacitance but a lower electrical conductivity and cycle stability [4].

Activated carbon (AC) is one of the most widely studied materials used as a supercapacitor electrode material, especially in developing environmentally friendly activated carbon materials, one of which is biomass-based [2, 5]. Many research groups have developed biomass-based activated carbon, which shows good potential to be used as supercapacitor electrodes [2, 6, 7, 8, 9, 10, 11, 12]. Coffee ground waste is abundant as the world's coffee-drinking culture grows. Coffee ground waste is one of the biomasses that can be converted to activated carbon due to its high cellulose (12.4%), hemicellulose (23.9%), and lignin (39.1%) contents. Recent research has noted the potential for activated carbon based on coffee ground waste to produce activated carbon with a specific surface area of 399.05 m^2/g [13]. Rufford *et al.* [14] reported using activated coffee ground waste with a high specific capacitance of 368 F/g at 0.05 A/g. However, the cyclability at long cycles and specific capacitance at high current density have limited the application of activated carbon to the electrode of supercapacitors. Adding metal oxide as a composite to the AC is one way to increase these performances.

In recent years, extensive research has been conducted investigating metal oxides for their potential in supercapacitors due to their high theoretical specific capacitance, which is critical in increasing energy density. RuO_2 has received much interest because of its excellent capacitance, impressive voltage cut-off, and remarkable cycle stability [15]. However, the widespread usage of RuO_2 is hindered by its high cost despite its remarkable performance. Researchers are currently investigating alternate materials to address these issues. NiO and MnO_2 have emerged as attractive possibilities among them [16, 17]. These materials offer good performance while being more cost-effective than RuO_2.

Another metal oxide that has drawn the interest of researchers is SnO_2. This is attributed to its high theoretical capacitance value of 2815.2 F/g, low cost, environmental friendliness, and ease of access [18]. Therefore, SnO_2 has a competitive advantage since it provides higher performance despite low cost compared to RuO_2. For example, the synthesis of mosaic-structured SnO_2-carbon porous microspheres from triphenyltin acetate at a current density of 1 A/g provided an amazing specific capacitance of 420 F/g with a high energy density of 34.2 Wh/kg [15]. Furthermore, when SnO_2 is combined with activated carbon (AC) derived from gasified rice husk, the capacitance increases to 177 F/g at 1.5 A/g, a substantial improvement above the 141 F/g achieved without SnO_2 [18]. Other previous research has also proved the cyclic stability of SnO_2-based materials. For instance, Zhang *et al.* [19] obtained outstanding cyclic stability of 96% over 6000 cycles utilizing the same Sn precursor integrated into a reduced graphene oxide structure.

In this study, the use of coffee ground waste as a supercapacitor electrode with tin oxide composite was carried out. The as-synthesized activated carbon was characterized using X-ray diffraction (XRD), Raman, and Fourier transform infrared (FTIR) spectroscopy. At the same time, electrochemical properties were analyzed using cyclic voltammetry (CV) and galvanostatic charge-discharge (GCD). Thus, this study is expected to produce materials with high specific capacitance to become an alternative to supercapacitor electrode materials.

2. Experimental

2.1. Materials

Materials used were potassium hydroxide (KOH, Merck), Tin (IV) Chloride ($SnCl_2$, Merck), Sodium hydroxide (NaOH, Merck), Sulfuric acid (H_2SO_4, Mallinckrodt), and Nafion (Merck). All used reagents were of analytical grade. Coffee ground waste was obtained from coffee shops in Jakarta. The solution was prepared using double-distilled water.

2.2. Synthesis of Activated Carbon

First, coffee ground waste was cleaned with water to remove impurities and dried in the oven at 110°C for 24 h. Dried coffee ground waste was ground with a ball mill and filtered to 100 mesh. The powdered material was pre- carbonized in a muffle furnace at 400°C for 1 h. The pre-carbonized powder was mixed with KOH at a mass ratio of C/KOH 1:3. The mixture was further activated at 800°C for 2 h in N_2 to obtain the activated carbon labeled CGW-AC.

2.3. Synthesis of SnO_2/Activated Carbon

The SnO_2/AC composite was synthesized with a hydrothermal step to do SnO_2 impregnation into a carbon structure. 0.1 g $SnCl_4$ powder was mixed with 120 mL distilled water. Next, 0.1 g of activated carbon powder was dispersed in the tin precursor solution and stirred for 30 min. The suspension was then transferred into a 200 mL Teflon-lined autoclave and was heated at 150°C for 6 h. After multiple washing with distilled water, SnO_2/AC composite was dried in the oven at 70°C for 8 h. The SnO_2/AC sample was labeled as CGW-AC-SnO_2. The yield was calculated by dividing the weight of the final activated carbon obtained after activation by the dry mass of the original raw materials (coffee ground waste). The yield of AC and AC-SnO_2 from coffee ground waste was calculated using Equation (1).

$$\%\ Yield = \frac{\text{Weight of AC Obtained}}{\text{Weight of Raw Material}} x\ 100\% \quad (1)$$

where, the weight of raw material is the weight of coffee ground waste powder. In the case of the SnO_2/AC sample, the weight in the numerator would be the weight of the SnO_2/AC powder.

2.4. Characterization

The sample's functional groups were analyzed using Thermo Scientific iS5 in 600-4000 cm^{-1} wavenumber. The crystal structure of the samples was characterized by X-ray Diffraction Olympus BTX II Benchtop using Co-Kα radiation (λ = 0.17902 nm) with a scanning speed of 0.5°/min. Raman spectra were analyzed by a Horiba spectrometry (LabRAM HR Evolution Raman Microscope) at an excitation wavelength of 532 nm and 1800 g/mm.

2.5. Electrochemical Measurement

The working electrode was made by mixing 1.6 mg of activated carbon sample (CGW-AC or CGW-AC-SnO_2) and 0.5 mL of isopropanol and then sonicated for 15 min to make the ink. A 0.6 µL ink was dropped to a glassy carbon electrode and coated with 0.3 µL nafion. Using 1 M Na_2SO_4 aqueous electrolyte, the three-electrode system was arranged with the working electrode, counter electrode (Pt wire), and reference electrode (Ag/AgCl). The complete procedure was similar to the previous study for the three-electrode measurement set-up [20, 21]. Cyclic Voltammetry was measured within 5 to 100 mV/s, while galvanostatic charge/discharge was measured within 0.5 to 10 A/g.

These various measurements were designed to evaluate the material's performance under high scan speeds and current densities. Typically, high current density was assessed to assess how the material performs in shorter charge-discharge cycles, which is helpful for fast-charging applications. The specific capacitance (Cs) can be calculated from CV and GCD. As for Cs calculated from the CV curve, it can be done by using Equation (2).

$$Cs_{CV} = \frac{\int I\,dV}{V \times m \times \Delta V} \quad (2)$$

where ∫ I dV represents the area under the CV curve, while m is the mass of the material in the working electrode (g). The term I/m corresponds to the current density (A/g). V denotes the scan rate (V/s), and ΔV is the potential window (V). On the other hand, Equation (3) is used for Cs determined using the GCD curve.

$$Cs_{GCD} = \frac{I\Delta t}{m\Delta V} \quad (3)$$

where the Δt is the discharge time (s).

3. Results and Discussion

3.1. Characterization

In the FTIR test results (Figure 1a), it can be seen from the results of the FTIR test that there are peaks at 3445 cm^{-1}, 2918 cm^{-1}, 1634 cm^{-1}, and 625 cm^{-1}, respectively. Each peak indicates the presence of O-H stretching, C-H stretching, C-C stretching, and Sn-O stretching functional groups successively [22]. The yield of the activated carbon synthesized using this process is 44.2%. In the XRD test (Figure 1b), the CGW-AC shows some peaks at 26.54 and 43.3°. The peak indicates the presence of porous carbon material due to the commonly found in activated carbon [23]. The XRD pattern of CGW-AC-SnO_2 samples contains peaks from SnO_2 crystal at 2-theta 26.9, 34.3, and 38°. This agrees with JCPDS no 41-1445 for the SnO_2 crystal structure [18].

The yield of the CGW-AC-SnO_2 is 20.5%, calculated from the starting materials using Equation (1). The results of the Raman test show (Figure 1c) that there are bands at 1340 and 1588 cm^{-1} representing D-Band and G-Band. The D-Band represents the degree of amorphous and lattice distortion in the sample. Usually, these defects can occur due to various factors, including impurities, vacancies, edge defects, and functional groups. In contrast, the G-Band represents the degree of graphitization [24]. The lower disorder degree (I_D/I_G = 0.96) of the CGW-AC-SnO_2 compared to CGW-AC (I_D/I_G = 1.05) corresponds to the low degree of carbon disorder attributed to the successful integration of SnO_2 nanoparticles into the activated carbon structure.

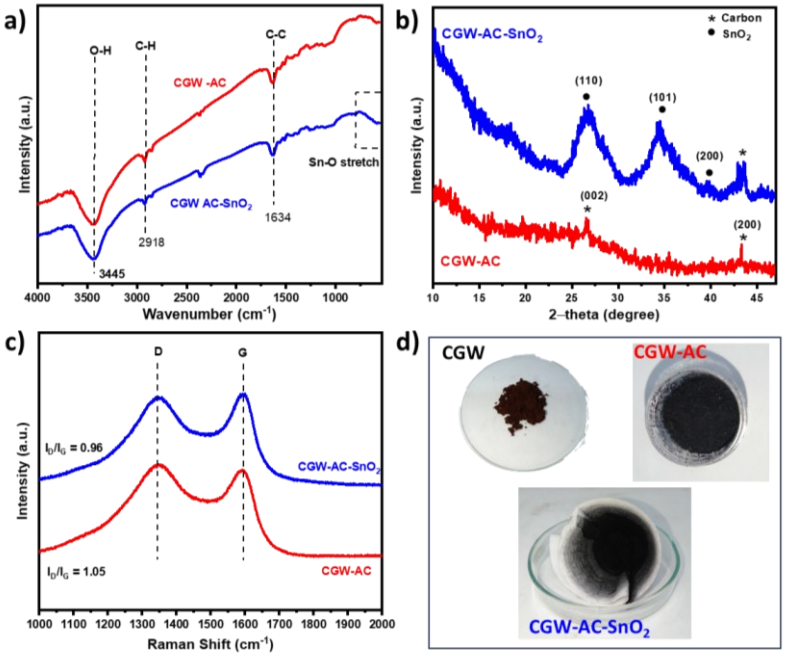

Figure 1. (a) FTIR spectra, (b) XRD pattern, (c) Raman result, and (d) samples of CGW-AC and CGW-AC-SnO_2

In the previous research, metal or metal oxide can act as a catalyst for more graphitic structure on the carbon materials [25, 26]. The SnO_2 formation during hydrothermal synthesis led to a more graphitic structure in the sample. Raman analysis result suggested that the carbon structure is still retained even with the addition of SnO_2 by hydrothermal reaction. The decreasing yield and I_D/I_G ratio show the catalytic effect of the metal oxide, which aligns with the previous study on the high-temperature graphitization reaction of carbon materials [25, 26].

3.2. Electrochemical Measurement

Electrochemical testing is carried out with CV tests for all materials. The result is shown in Figure 2. CV testing was carried out in a potential window of -0.4 to 0.6 V, which was determined by the trial method until obtaining a rectangular-shaped curve that showed the occurrence of an EDLC mechanism for supercapacitors [27]. CV test results for CGW-AC and CGW-AC-SnO_2 samples showed an area increase in the increasing scan rate, as seen in Table 1. The increase in specific capacitance with higher scan rates can be attributed to the complex interaction of these components, which includes better double-layer formation, greater faradaic reactions, and ohmic drop effects.

However, it is important to note that this gain may only be detected up to a certain degree since extremely high scan rates may result in reduced specific capacitance due to limited ion transport and other kinetic restrictions. The CV curve shows good symmetry, which indicates that the composite material has good reversibility as an electrode. With the increase in scanning rates, the area shows an uptrend, while the basic shape of the curve has not changed, which indicates that the electrochemical properties of both samples in this potential range are stable [19].

The GCD analysis result is shown in Figure 3 for both samples. The CGW-AC samples indicate that the curve forms a triangle, indicating the occurrence of the EDLC mechanism [17]. In the CGW-AC-SnO_2 samples, it can be seen that in the discharge position, there is a slight arc indicating a redox reaction of SnO_2. As the sample is composited with SnO_2, it shows the combined mechanism of EDLC and pseudocapacitance. The occurrence of pseudocapacitance causes the area of the triangle to be larger with a longer discharge time [28]. The calculation of specific capacitance is based on Equation (3), presented in Table 2. The specific capacitance of CGW-AC samples resulted in 263.5 F/g, comparable with similar material as presented by Rufford *et al.* [14] with a specific capacitance of 368 F/g at 0.05 A/g. The composited SnO_2 samples show a higher specific capacitance of 451.5 F/g.

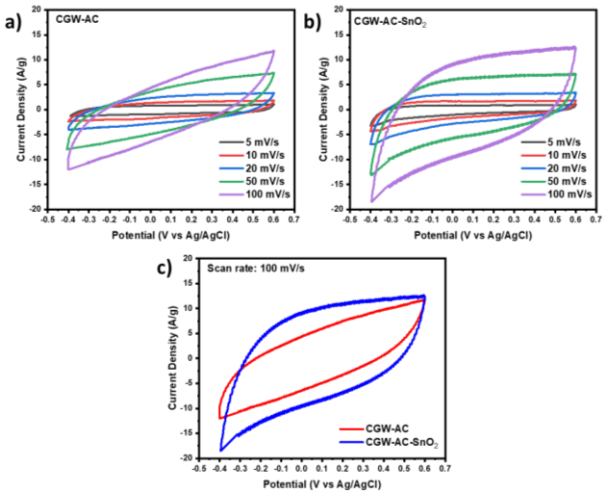

Figure 2. CV Test result for (a) CGW-AC, (b) CGW-AC-SnO_2, and (c) Comparison at scan rate 10 mV/s

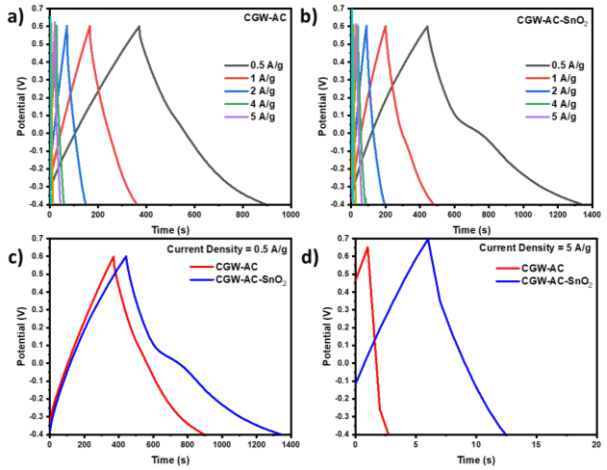

Figure 3. GCD test result for (a) CGW-AC, (b) CGW-AC-SnO_2; Comparison at current density (c) 0.5 A/g and (d) 5 A/g

Table 1. Specific Capacitance for CGW-AC and CGW-AC-SnO_2 based on CV measurement

Sample	Scan rate (mV/s)	Specific Capacitance (F/g)	Specific Capacitance Increase (%)
CGW AC	5	290.74	-
	10	248.46	-
	20	207.65	-
	50	140.10	-
	100	90.45	-
CGW AC SnO_2	5	333.34	14.65
	10	297.29	19.65
	20	254.00	22.32
	50	195.31	39.41
	100	148.74	64.45

Table 2. Specific Capacitance for CGW-AC and CGW-AC-SnO_2 based on GCD measurement

Sample	Current Density (A/g)	Specific Capacitance (F/g)	Specific CapacitanceIncrease
CGW-AC	0.5	263.5	-
	1	194	-
	2	154	-
	4	116	
	5	110	-
CGW-AC-SnO_2	0.5	451.5	71.35 %
	1	279	43.81 %
	2	210	36.36 %
	4	172	48.28 %
	5	165	50.00 %

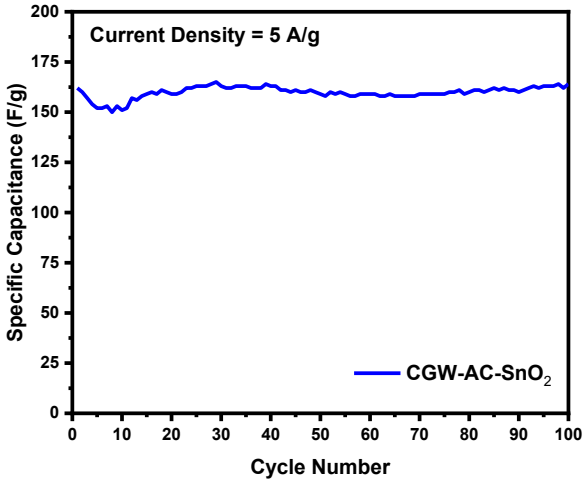

Figure 4. Cycle Performance CGW-AC SnO_2

The increased activated carbon capacitance is thought to be due to a synergistic effect between metal oxides and carbon materials. Metal oxides with a high theoretical capacitance value may improve the capacitance of activated carbon by simultaneously generating EDLC and pseudocapacitive mechanisms [29]. Furthermore, specific capacitance estimates employing CV and GCD data show a similar tendency. This synergy between metal oxides and carbon materials not only increases capacitance but also allows unique charge storage processes to coexist, resulting in better energy storage capabilities in supercapacitors. The extended cycle test of the CGW-AC-SnO_2 shown in Figure 4 presented that the specific capacitance is relatively stable from 123 F/g to 122 F/g in the 100th cycle at a current density of 5 A/g. The result indicates that metal composite does not affect cycle stability even at the high current rate.

4. Conclusion

Coffee grounds from the waste of the coffee-making can be used as a precursor to synthesize activated carbon by pyrolysis and hydrothermal methods using KOH as an activator. The characterization test results showed that the impregnation of tin oxide to the activated carbon material has been successfully carried out. This is evidenced by the Sn-O functional group from the FTIR test, the increasing degree of irregularity or defect from the Raman Spectroscopy test, and a tin oxide peak from the XRD test. The addition of tin oxide has improved the electrochemical performance of the synthesized activated carbon material. This is evidenced by an increase in the specific capacitance value of the CV and GCD tests for samples that SnO_2 has composited.

Acknowledgment

This project is funded by the Directorate of Research, Technology and Community Service, Directorate General of Higher Education, Research, and Technology, Ministry of Education, Culture, Research and Technology Republic of Indonesia under the PKDN research scheme 2023. The authors are grateful for the funds given.

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