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# The Enhanced Electrosorption Capacity of Activated Charcoal (AC) and WC@AC Composite for Water Desalination through Capacitive Deionization

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Capacitive deionization (CDI) is a promising electrochemical water desalination technology. It is intended to be a low-energy-consumption technology. Because of its low cost and easy availability, activated carbon (AC) has been extensively tested as a CDI electrode material. It does, however, have hydrophobic properties and a low specific capacitance. In this study, tungsten carbide (WC) was incorporated with AC and used in the hydrothermal treatment process. WC plays an important role on enhancing the electrochemical and electrosorption activity of WC and overcoming the aforementioned issues. A single-batch mode CDI cell was used to test the desalination performance. In comparison to pristine AC (118.3 Fg<sup>-1</sup>) and WC (11.4 Fg<sup>-1</sup>), the addition of 10% WC to AC resulted in a high specific capacitance (1471 Fg<sup>-1</sup>) at 5 mVs<sup>-1</sup>. As a result, in CDI technology, this material is regarded as a promising candidate for water desalination.

Keywords: Sodium chloride; Activated carbon; tungsten carbide; Capacitive deionization

# **1. INTRODUCTION**

Rapid population growth, accompanied by technological progress (particularly those innovations requiring clean water) have increased demand for fresh water, but its scarcity is one of the most serious global issues confronting humans today [1,2]. Desalination of sea or brackish water is the best solution to this problem as it reduces fresh water shortage [3]. Some widely-used techniques for obtaining clean water from the desalination process include reverse osmosis, multi-stage water distillation, ion exchange,

and large-scale electrolysis. However, these technologies have a number of drawbacks, including high maintenance costs and high energy consumption [4]. As global awareness of the importance of environmental protection and energy conservation has grown, so has the interest in and focus on research and development to find low-cost, clean, and environmentally safe water desalination technologies. Capacitive deionization (CDI) is a comparatively newer desalination technology with low energy consumption, simple operation and maintenance, and eco-friendly properties [5,6]. It is distinct in that it has a compact and tunable structure that allows for the selective removal of soluble ions in water [5,7]. The CDI process involves the flow of saline water between two electrodes connected to a power supply. When the power source is turned on, an electrostatic field is formed between the electrodes and the ions in the solution, and the cations in the solution are transferred to the negative electrode, while the anions in the solution are transferred to the positive electrode, forming the electrode double layer (EDL)[6,8,9]. This step is known as ion removal, charging, or adsorption. The adsorbed ions are desorbed and transferred to the bulk solution as the electrodes are reversed or the power supply is turned off. This step is known as discharging or desorption. The electrode material and its electrochemical properties are regarded as the primary key parameters influencing CDI performance [6,10]. As a result, much effort is required for the electrode material. Furthermore, in order to be easily commercialized, the electrode material should have some characteristics such as large surface area, high specific capacitance, high adsorption capacity, low polarization effect, high chemical stability, good wettability, high conductivity, rapid response to ion insertion/de-interference, easy manufacturing, and low cost [9,11–13]. Therefore, carbonaceous materials are the best candidates for use in CDI as they possess the aforementioned properties. Carbon materials (including porous carbon, activated carbon, carbon nanotubes, and graphene) have been widely used as CDI electrode materials [6,10,11,14-19]. Among all the carbon compounds, activated carbon (AC) is the best candidate for use as an electrode in CDI due to its distinct features, such as high surface area (1000–2000  $m^2g^{-1}$ ), high electrical conductivity, well pore-size distribution, availability, and low cost [20]. Furthermore, crevices, cracks, and holes on the outer surface of AC increase the specific surface area and provide tunnels for ion entry during the CDI process [21]. However, it has low wettability and specific capacitance, as well as poor physicochemical stability [5]. Nanocomposites have sparked a lot of interest due to their ability to improve the properties of each individual component [22]. These issues could be addressed by forming carbonaceous composites with metal oxide, because metal oxide has the ability to improve the wettability, surface chemistry, charge nature, and electrochemical properties of carbon materials due to its synergistic effects with carbon [23,24]. However, metal oxides also have low electrical conductivity [25]. Transition metal carbides (TMCs) have unique properties such as high electronic conductivity, chemical stability, and melting point, as well as excellent mechanical, electrochemical, and magnetic properties, which have led to their use in a variety of applications [25]. Among them, tungsten carbide (WC) has received little attention despite its excellent physical and chemical properties such as chemical stability, thermal (110 W $\cdot$ m<sup>-1</sup> $\cdot$ K<sup>-</sup> <sup>1</sup>) [26], electrical conductivity ( $10^5$  S cm<sup>-1</sup> at 20°C) [1], and catalytic activity. It has been used in a variety of applications (eg. microelectronic and aerospace material industries, fuel cells, dye-sensitized solar cells, and sensors) [27]. MXene materials, which are two-dimensional transition metal carbides, carbonitrides, and nitrides, have recently been used as efficient electrodes for CDI cells [28,29]. They have distinct physical and chemical characteristics, as well as exceptional conductivity and

electrochemical behavior [30]. Srimuk and his group reported that Ti3C2-MXene (13 mg/g) has a high ion adsorption capacity at 1.2 V in the presence of 5 mmol NaCl aqueous solutions [28]. Recently, they prepared a Mo<sub>1.33</sub>C-MXene electrode for CDI, where the adsorption capacity of the ions was 15 mg/g in 600 mmol/L of NaCl at 1.2 V [29]. Bharath et al. [2], Ni/(Ti3AlC2) (anode)/porous reduced graphene oxide (cathode) electrode system prepared In a CDI system, electrodes were used to remove multivalent ions (F-1, Pb++, and AS(III)). Due to its high electronegativity, the anode material was used to remove F-1 and AS(III), while the cathode material was used to remove Pb++. The specific capacitance of the electrode materials was 760 Fg-1 and 385 Fg-1, respectively. For monovalent F-, divalent Pb++, and trivalent AS(III) ions, electrode materials on a CDI setup operated at 1.4 V for 160 minutes and had electrosorption capacities of 68, 76, and 51 mg g-1, respectively. Yong Liu et al. created a MoC NPs@CNFs aerogel as the pseudocapacitive electrode material for a CDI cell. MOCs@CNFs and CNFs electrodes are used to create the asymmetric cell. With a solution feed concentration of 3000 mg, the cell has a high desalination capacity (37.03 mg.g-1). At 1.2V, L-1 [31]. Ayman et al. enhanced the electrosorption capacity of carbon nanofibers used chromium carbide and titanium carbide. The electrode materials have demonstrated a good electrosorption capacity of 20.40 mg g<sup>-1</sup> and 33.10 mg g<sup>-1</sup> at 1.2 for chromium carbide@ carbon nanofibers and titanium carbide@carbon nanofibers, respectively. Thus, the combinations of WC and AC could effectively increase its specific capacitance [32]. Furthermore, it has the potential to improve the final prepared material's electrical conductivity, ion diffusion, and electrochemical properties, thereby enhancing the ion electrosorption of the CDI process [32]. When compared to other carbon materials, AC is easily produced in large quantities at a low cost. In this study, we introduced WC NPs modified AC as a novel nanocomposite material to be used in CDI technology for the first time. The fabricated composite was prepared using a hydrothermal process in an alkaline solution to improve the formation of hydrophilic groups on the surface and, as a result, the electrosorption and desorption performance. The nanocomposite that was created demonstrated high specific capacitance, low charge transfer resistance, and exceptional performance in water desalination.

### 2. MATERIALS AND METHODS

#### 2.1 Materials

In this experiment, pharma-grade activated charcoal (acid wash, LOBACheme) and WC (Manografi, 99.9%, 1 m, Germany) were used. Sigma–Aldrich provided polyvinylidene fluoride (PVDF, Mw = 35000 kg mol1), potassium hydroxide (KOH, 99.99 percent), sodium chloride (NaCl, 99.99 percent), and dimethylformamide (DMF). The chemicals were used without being treated first.

# 2.2 Preparation of WC@AC composite

Typically, AC and 10 wt.% WC-based AC are immersed in 100 mL of 5 M KOH. The mixtures were magnetically stirred for 1 h before being ultrasonically sonicated for 1 h. The sonicated mixtures were microwave-irradiated for 50 seconds. The mixtures were then transferred to a Teflon crucible inside

a stainless-steel autoclave hydrothermal reactor. The reactors were heated in a muffle furnace (Thermo Fisher Scientific, 1400 °C, USA) for 24 h at 120 °C. They were then allowed to cool down naturally. The filter papers' black residues were washed with distillated water until the filtrate's pH reached 7. The finished products were dried overnight at 80 °C. Figure 1 shows the scheme for the preparation procedure.



Figure 1. Schematic depiction of the preparation procedure of WC@AC.

### 2.3 Characterization

A scanning electron microscope was used to examine the surface morphology of the WC@AC composite electrodes (JEOL, JSM-5900, Japan). An X-ray diffractometer (Bruker Co., D8 Discover, USA) was used to verify the crystallinity phases of the WC@AC composite electrode from 10° to 90°. Furthermore, the Raman spectra of AC and WC@AC composite electrodes were examined using a Raman microscope (Senterra–Brucker, Germany) with a wavelength of 532 nm. Using a potentiostat, the electrochemical properties of the WC@AC composite electrodes were investigated using cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) (Metrohm Autolab, Netherlands).

# 2.4 Electrochemical characteristics of AC and WC@AC composite electrode materials

A three-electrode system was used for the electrochemical CV analyses where a Pt rode, Ag/AgCl/(3 M KCl), and WC@AC composite were attached to glassy carbon electrode (GCE) as

counter, reference, and working electrodes, respectively. Two mg of WC@AC composite were sonicated for 30 minutes in 420  $\mu$ l of isopropanol and 20  $\mu$ lof Nafion solution. Then, 15  $\mu$ l of the sonicated solution was deposited on the GCE and allowed to dry for 10 minutes at 80 °C. Using 1 M NaCl aqueous solutions, different scan rates, and a potential window ranging from 0.4 to 0.6 V, the CV for each electrode (AC and 10% WC@AC composite) was determined. In the same cell, EIS was performed using a frequency response analyzer (FRA). The alternating voltage had a 5 mV amplitude and a frequency range of 10<sup>-2</sup>–10 KHz. At various applied potentials, the chrono potentiostatic charge-discharge (PCDs) for AC and 10% WC@AC composite electrodes were obtained. At 1, 2, 3, 4, and 5 Ag<sup>-1</sup>, a chrono galvanostatic charge–discharge (GCD) test for a 10% WC@AC composite electrode was performed.

#### 2.5 Electrosorptive capacity measurement

Overnight, a few drops of DMF, 85 weight percent of each electrode material (AC and WC@AC composite), and 15 weight percent PVDF were magnetically mixed. The formed pastes were sonicated for 1 hour to achieve homogeneous pastes before being coated on carbon sheets (10 10 cm2). The coated carbon sheets were placed in a drying oven and left to dry overnight at 80 °C. A peristaltic pump delivered an aqueous NaCl solution with an initial conductivity of 100  $\mu$ S cm<sup>2</sup> to the CDI cell at a flow rate of 10 mlmin<sup>-1</sup>. Using a DC power supply, the electrosorption capacity of the prepared electrode materials was determined at various applied potentials (1, 1.2, and 1.4 V). An ion-conductive meter was used to measure the change in the effluent's solution conductivity (Hanna Instruments, HI-2300). The electrode electrosorption capacity (q, mg g1) was calculated using the following equation for the three studied potential values:

 $q = \frac{(C_0 - C)V}{m} \tag{1}$ 

where V(L) is the total volume of NaCl solution, and m(g) is the total mass of the working electrodes.

# **3. RESULTS AND DISCUSSIONS**

#### 3.1 Morphology and architecture

The pristine AC SEM image (Fig. 2A) revealed a rock-like shape with an irregular and heterogeneous surface morphology. The morphology of the 10% WC@AC is shown in Fig. 2B. To avoid blocking the pores of the AC, WC NPs were deposited on the surface and dispersed in the surface without aggregation. The XRD analysis of AC, WC, and 10% WC@AC is shown in Fig. 3. As shown in Fig. 3A, the AC revealed two broad peaks at 2 values of 24.3° and 43.1°, which correspond to the graphite (002) and (100) crystal planes respectively (JCPDS card no. 41-1487).



Figure 2. SEM images of pristine AC (A) and WC@AC (B).



Figure 3. XRD patterns of AC, WC, and WC@AC



Figure 4. Raman spectra of AC and WC@AC.

The broad diffraction peaks revealed the AC's disordered, amorphous structure. The XRD spectra (Fig.3B) showed a high intensity peak of pristine WC (Hexoganal, COD 2100654, CP-6 m2 (187)) at 2θ values 31.544°, 35.701°, 48. 371°, 64.134°, 65.860°, 73.230°, 75.621°, 77.229°, and 84.233° agree well with crystal planes (00-1), (-100), (-10-1), (-210), (00-2), (-21-1), (-200), (-10-2), and (-20-1), respectively. The XRD of 10% WC@AC appeared the AC peaks and the first three main peaks of WC (Fig.3C).

Raman spectroscopy is a technique used to obtain structural information about the molecular vibration or rotational energy of a particular material (Fig.4). Furthermore, it is commonly used to describe the structural characteristics of carbonaceous and oxide materials that exhibit lattice disorder [33]. The Raman spectra of pristine AC and WC@AC. Two spectrum peaks appeared at 1350 and 1595 cm1, denoting the D and G bands respectively [33].



### 3.2. Electrochemical characterizations

**Figure 5.** (A) Cyclic voltammograms of AC, WC, and WC@AC in 1.0 M NaCl at 5 mVs<sup>-1</sup>. (B) Specific capacitance plots as a function of scan rate at AC, WC, and WC@AC.

CV, EIS, GCD, and PCD were used to assess the electrochemical performance of AC and WC@AC electrode materials. The CV profiles of AC and WC@AC electrode materials are shown in Fig. 5A at a sweep rate of 5 mV/s in a 1 M NaCl aqueous solution with a potential window ranging from 0.4 to 0.6 V. As a result of the formation of EDL, all samples displayed a rectangular CV profile with no faradic reactions in the applied potential range, indicating the ideal capacitive behavior of the introduced electrode materials [34]. The EDL was formed due to a Coulombic interaction instead of redox reactions [35]. Furthermore, the symmetrical rectangular CV shapes of the electrode materials indicated that the ions in the solution could be electro-adsorbed on the electrode materials efficiently and rapidly [36]. The formation of EDL, combined with an increase in the current density of the as-prepared composite electrode materials, demonstrated ideal and fast reversible capacitor behavior. [37]. In general, the addition of WC to AC improved the electrochemical properties.



**Figure 6.** (A) Cyclic voltammograms of WC@AC in 1.0 M NaCl solution at various scan rates. The estimated specific capacitance values of pristine AC, WC and WC@AC were plotted versus the scan rate in section (B).

As shown, the WC@AC composite electrode outperformed the pristine AC and WC electrode materials in terms of largest integrated area and current density, demonstrating the high contact area, low interfacial resistance, high capacitance, and excellent electrochemical performance of WC@AC [38]. The CV performance of the WC@AC composite electrode material is shown in Fig. 6A at various scan rates ((5, 10, 25, 50, 75, and 100) mV s1) in a 1 M NaCl aqueous solution with a potential window ranging from 0.4 to 0.6 V. The CV plots demonstrated distortion from rectangular to oval shapes as the scan rate increased from 5 mV s-1 to 100 mV s-1 in a 1 M NaCl solution. This finding can be attributed to the salt solutions' inherent resistivity to ion movement at high scan rates, resulting in a negative effect on the EDL formation [39]. A low scan rate promotes ion adsorption into the electrode and increases electrode reversibility [40,41]. At a low scan rate, ions have enough time to move from the bulk solution to the electrode surface, whereas at a high scan rate, ions do not have enough time to move to the electrode. Specific capacitance (Cs, Fg1) is regarded as an important factor in determining the CDI behavior of electrode materials. The Cs values of the fabricated electrode materials can be estimated by

integrating the entire CVs at different scan rates to obtain an average value using the equation below [6,10,11]:

$$Cs = \frac{\int idV}{2\nu\Delta Vm} \tag{2}$$

where Cs is the specific capacitance (Fg–1), I is the current (A), V is the potential (V), and m is the mass of the electro-active material in the fabricated electrode (g). Figure 6B depicts a relationship between Cs values and scan rate. The Cs values generally decreased as scan rate values increased. As previously discussed, ions could easily access the electrode surface at low scan rates, resulting in a significant increase in electrosorption capacitance. The Cs of the WC@AC composite electrode material were higher than those of the pristine AC and WC electrode materials. At 5 mVs-1, the WC@AC composite electrode material had a higher specific capacitance (1471 Fg<sup>-1</sup>) than the pristine AC (118.3 Fg<sup>-1</sup>) and WC (11.4 Fg<sup>-1</sup>) (Fig. 5B). Thus, the WC@AC composite electrode material demonstrated the best EDL capacitance behavior among the other electrodes towards salt ion electrosorption, thereby improving charge transfer and contact area [21]. EIS measurements are a tool to determine the performance of electrode materials with respect to the electrolyte solution and the electrode material charge transfer behavior at the electrode/electrolyte interface.



**Figure 7.** (A) WC@AC regeneration profiles in 1.0 M NaCl solution at various potentials. The cyclic stability experiment with desalination retention of WC@AC for 20 cycles under the same electrochemical conditions was also presented in section (B).

The Nyquist plots of AC (Fig. 7A) and WC@AC (Fig. 7B) composite electrode materials in a 1 M NaCl aqueous solution in the frequency range of 0.01–10 Hz with an AC perturbation of 5 mV. In general, for electrode materials, a defined semicircle should appear in the high frequency region. This semicircle, as previously stated, is intimately related to the electron-transfer process, the electrical conductivity of the electrodes, and the solution interface. The Nyquist plots revealed that all electrodes had an inclined line in the high-frequency section alternate of semicircle shape, indicating a significant reduction in polarization resistance [42–44]. Furthermore, because of their typical double-layered

capacitive behavior, the vertical line was continuous in the low-frequency section, implying the great capacitive behavior of the electrode materials, thus demonstrating the fast electrolyte ion diffusion into the pores of electrode materials [44,45]. The main factors influencing the commercialization of CDI electrodes are lifetime and cost. As a result, the regeneration of the introduced WC@AC composite electrode material was investigated using a charge–discharge test at 1, 1.2, 1.4, and 1.6 V in 1 M NaCl aqueous solution (Fig. 8A).



**Figure 8.** Nyquist plots of AC (A) and WC@AC (B) in 1.0 M NaCl solution in the frequency range of 10000 – 0.01 Hz at a potential value of 0 mV with an ac perturbation of 5 mV.



**Figure 9.** GCD of WC@AC electrode at different currents (D) and the long-term stability of WC@AC nanocomposites electrode at 5 Ag<sup>-1</sup>.

The plots demonstrated excellent reversibility and electrochemical stability. During the desorption process, the conductivity values returned to their initial values, indicating that the adsorbed

ions had displayed complete desorption to the electrolyte after the voltage was released. These findings demonstrated that the fabricated materials have good regeneration properties. During the discharge process, the WC@AC composite electrode material had a long time. This discharge behavior demonstrated the WC@AC composite electrode material's ability to store more ions. Furthermore, the cycling stability of the fabricated WC@AC composite electrode material was investigated (Fig. 8B) for 20 cycles at 1.4 V in a 1 M NaCl aqueous solution, and it demonstrated high cycling stability. GCD tests were carried out in the WC@AC composite electrode material at potential windows ranging from 0.4 to 0.6 V and current densities ranging from 1 A g<sup>-1</sup> to 5 Ag<sup>-1</sup> (Fig. 9A). At all current densities, the profile had nearly triangular shapes, and the discharged time increased with decreasing current density, demonstrating excellent capacitance behavior and electrochemical reversibility. After 20 cycles of GCD at 5 A/g, the long-term stability of the WC@AC composite electrode material was maintained (Fig. 9B). After 20 cycles, a similar pattern was obtained, confirming the high electrochemical reversibility and stability. The WC@AC composite electrode material was an excellent candidate for CDI electrode due to its excellent electrochemical cycling stability.

# 3.5 CDI cell performance

The electrosorption performance of the CDI cell is shown in Fig. 10A using AC and 10% WC@AC electrode materials in the presence of NaCl aqueous solution (100 S cm1) at a flow rate of 10 ml min1 and a potential between the electrodes of 1.6 V.



Figure 10. (A) CDI cell performance of AC and WC@AC in  $100 \square S \text{ cm}^{-1}$  NaCl solution<sup>1</sup> at 1.6 V. (B) The electrosorptive capacity of AC and WC@AC.

The initial conductivity of the saline solution rapidly decreased as the potential was applied between the CDI electrodes, indicating that the NaCl ions were adsorbed by the charged electrodes. The electrosorption process slowed until the electrode surface was saturated with salt ions and the electrosorption equilibrium was attained. The electrosorption capacity of the WC@AC electrode material was greater than that of the AC electrode material. The electrosorption capacity of AC and WC@Ac was 3 and 7.9 mgg-1, respectively, as shown in Fig. 10B. Table 1 shows the electrosorption capacity of different carbon-based electrode materials. The high electrosorption capacity could be attributed to the addition of WC to AC, which increased the electrical conductivity, electrochemical behavior, and decreased the internal resistance of the prepared electrode material, resulting in increased ion transport towards the electrodes. As a result, the salt-ion adsorption capacity increased significantly.

Electrodes	Applied voltage	Initial	Electroadsorption	Ref.
	(V)	concentration	capacity (mg/g)	
		(mg/L)		
AC	1.5	200	3.68	[46]
TiO <sub>2</sub> /AC	1.2	100	8.05	[47]
ZnO/AC	1.2	100	8.5	[48]
MnO <sub>2</sub> AC	1.2	25	8	[49]
ZnO/AC	1.2	100	8.5	[50]
CeO <sub>2</sub> /AC	1.4	49	8.9	[51]
WC/AC	1.6	49	7.9	This study

**Table 1.** Electroadsorption capacity of different carbon-based electrode materials.

# **4. CONCLUSION**

Sonication and hydrothermal processes in aqueous alkaline media were used to prepare the WC@AC composite. At 5 mV s<sup>-1</sup>, the specific capacitance of the WC@AC composite electrode material was 1471.6 Fg<sup>-1</sup>, which was greater than that of the pristine AC (118.3 Fg<sup>-1</sup>) and WC (11.4 Fg<sup>-1</sup>) electrode materials. For 20 cycles of GCD at 5 Ag<sup>-1</sup>, the WC@AC composite electrode material demonstrated long-term stability. At 100 S cm1 of NaCl solution and 1.6 V, the electrosorption capacity of WC@AC (7.9 mgg<sup>-1</sup>) is greater than that of pristine AC (3 mgg<sup>-1</sup>).

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