

Preparation And Application of Carbon Aerogel/Cobalt Phosphate Composite for Desalination by Hybrid Capacitive Deionization Method

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Abstract

The availability of freshwater remains a global challenge. But the abundant saline water from the sea and estuaries can be harnessed for the production of freshwater by desalination. Hence there is a pressing need to develop a cost-effective and dependable desalination technology. In this study, we investigate the application of carbon aerogel cobalt phosphate composite for desalination of saline water by the hybrid capacitive deionization method. Aerogel was prepared by the sol-gel method, the aerogel was then heated to 1000°C under nitrogen gas flow to give carbon aerogel (CA). The CA was impregnated with cobalt and phosphate to give the composite [CA/Co₃(PO₄)₂]. From the cyclic voltammetry analysis, the specific capacitance of the CA is 191F/g while that of the composite is 750F/g. The galvanostatic charge/discharge test of the composite shows a capacitance retention of 99% after 2000 cycles of charge/discharge. The composite was applied as electrode material for desalination of 1000 mg/L, 2000 mg/L and 3000mg/L saline solutions separately by hybrid capacitive deionization at an applied potential of 2.0V. The desalination results shows that the salt adsorption of the electrode at 1000 mg/L, was 76 mg/g, at 2000 mg/L it was 112 mg/g, and at 3000 mg/L it was 157 mg/g. From the results, it is clear that the composite material will perform very well when used as electrode in building a HCDI equipment for commercial use.

Key words: Carbon aerogel, Cobalt Phosphate, Hybrid Capacitive Deionization, Desalination

1. Introduction

We live in a world where the demand for freshwater is always on the increase[1]. Hence new and innovative methods of freshwater production is essential to meet up with the demand for freshwater[2,3]. Desalination of the abundant saline water in the ocean and estuaries is one of such innovative methods for freshwater production. Developing a cost-effective and dependable desalination technology is key towards achieving this feat. Hence there has been much research lately in this field. Different desalination technologies have been developed over the decades[4–6], these are: thermal methods, osmotic methods, Electrodialysis, and Capacitive Deionization method. Among these methods, capacitive deionization (CDI) has gained much attention due to its low operational cost and dependability. CDI technology works by removing charged particles from a medium by electrically charge electrodes. The success of CDI for desalination depends on the nature of the electrode used[7,8]. When carbon materials are used as electrodes, it is called CDI technology, but when the carbon electrodes are electrochemically enhanced or completely replaced with other materials, it is called hybrid capacitive deionization (HCDI). In this study, carbon aerogel is used as the base electrode, it was then modified with cobalt phosphate. The resultant composite material (carbon aerogel cobalt phosphate) was then applied as electrode material for HCDI desalination study. The choice of these materials is based on previously reported performance of carbon aerogel (CA), Co, and Phosphates in HCDI desalination. M. Beke et al.[9], Applied a carbon aerogel polypyrrole (CA-PPy) composite for HCDI desalination, and a salt adsorption of 15.7 mg/g

was reported for the desalination of 800 mg/L saline solution. Also, the specific capacitance of the CA reported was 115F/g while that of the CA-PPy was 360F/g. Also, H. Zhou et al.[10], used a cellulose CA-PPy composite for energy storage material (supercapacitor) study. The composite was reported to have an electrical capacitance of 387.6 F/g and after a galvanostatic charge/discharge test of 10,000 cycles the composite was reported to have a capacitance retention of 92.7%. The result shows the composite has potentials as electrode material for HCDI desalination. Cobalt and phosphates are widely applied as battery materials[11]. They are reported to have Faradaic behaviour which enhance energy storage and charge transfer[12–14]. Cao. et al [11] applied $\text{Na}_3\text{V}_2(\text{PO}_4)_3@C$ as electrode for HCDI desalination. An impressive ion adsorption value of 137.2 mg/g at an applied potential of 1.0V was reported. Hence the application of the adopted electrode material should perform very well in HCDI desalination.

2. Materials and Methods

Materials:

Resorcinol, Formaldehyde, Cobalt nitrate hexahydrate [$\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$], Phosphoric acid (H_3PO_4), Sodium chloride, Ethanol, Deionized water, Hydrochloric acid.

All the materials are used as purchased from the vendors.

2.1 Synthesis of Carbon Aerogel (CA).

The synthesis of CA started with the preparation of aerogel by the sol–gel condensation of formaldehyde and resorcinol. 0.45g of resorcinol was dissolved in 20mL of acetonitrile, 0.63mL of formaldehyde was added, followed by 0.06mL of concentrated hydrochloric acid as catalyst; the mixture was stirred for 5min. The solution was then transferred into 50mL centrifuge tubes and left to age for three days. The sol–gel was then frozen to -80°C , after which it was freeze-dried to form the aerogel. The aerogel was then carbonised in a tube furnace at 1000°C for 4hrs. under nitrogen gas flow to give CA.

2.2 Synthesis of Carbon Aerogel/Cobalt Phosphate Composite [CA/ $\text{Co}_3(\text{PO}_4)_2$].

$\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and H_3PO_4 were dissolved in ethanol solution in a 1:2 molar ratio. A 1.0 molar portion of the CA was measured and added into the precursor solution; this was then sonicated for 10 hours. The resultant composite was filtered off, washed with ethanol and dried. The composite was then heated to 300°C under nitrogen gas. This converts the cobalt phosphate in the composite into nano size particles.

3. Characterization

3.1 FTIR SPECTROSCOPY

FTIR spectroscopy was employed to investigate the functional groups in the materials prepared. The spectrum gave a broad absorption band at $\sim 3400\text{ cm}^{-1}$ this indicate the presence of $-\text{OH}$ on the surface of the material[15,16], while the band observed at $\sim 1630\text{ cm}^{-1}$ is associated with aromatic ($-\text{C}=\text{C}-$) specie[17,18], and the cobalt and phosphate groups band are observed at ~ 1040 and 560 cm^{-1} [19,20]. These observations shows that the composite material was successfully synthesized. Figure 1, is the FTIR spectrum of the composite.

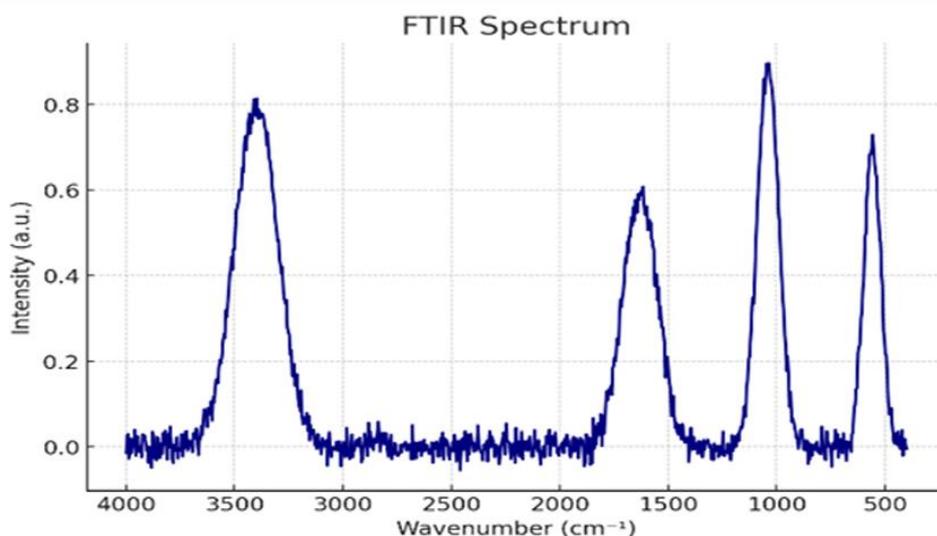


Figure 1. FTIR spectrum of composite.

3.2 X-RAY DIFFRACTION (XRD) Analysis

The crystalline nature of the material was analysed by XRD. The peaks observed at (16.5°, 25.2°, 31.3°, 36.8°) are crystalline peaks of Co₃(PO₄)₂[9,21], while the peak at ~24° indicates the presence of amorphous carbon [21,22]. Figure 2, shows the XRD pattern of the composite.

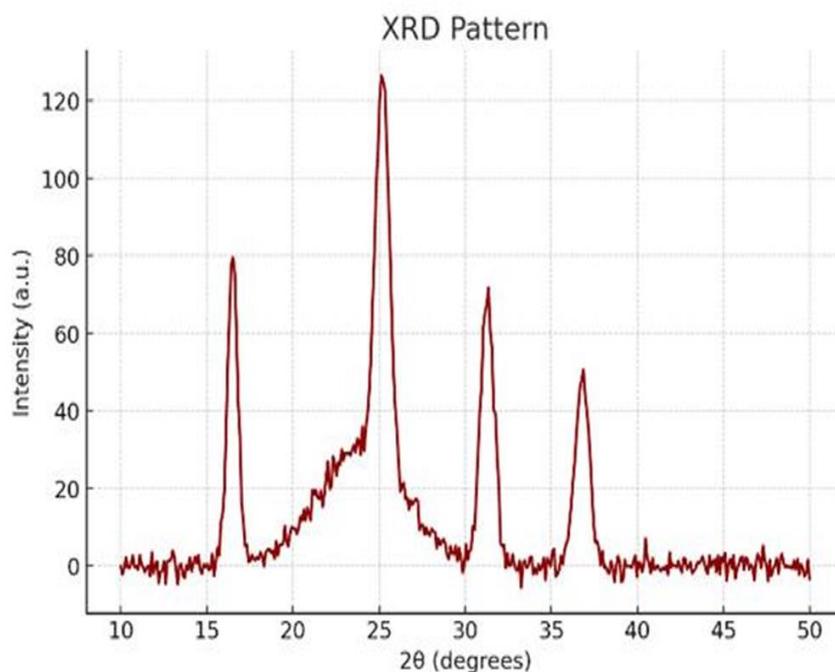


Figure 2. XRD of Composite.

3.3 Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) Analysis

SEM and TEM were used to analyse the microstructure of the materials. The SEM image shows that the CA have a porous sponge-like structure which indicates the presence of a well-developed CA structure that can support the cobalt phosphate[23,24]. This

morphology allows ion accessibility. Figure 3(A), is the SEM image of the composite. The TEM image is shown in Figure 3(B). It reveals the composite exist as nano particles and they are dispersed within the CA matrix. The bright and dark patterns observed suggest a crystalline region that is consistent with cobalt phosphate materials[18,25–27].

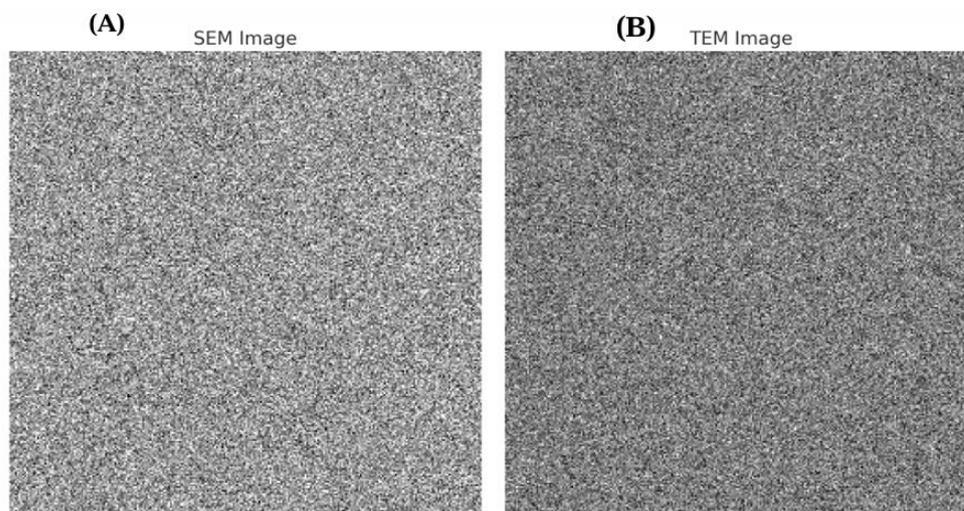


Figure 3. SEM and TEM images of Composite.

3.4 Brunauer-Emmett-Teller (BET) Analysis

The surface area and porosity of the material was analysed by N₂ adsorption /desorption measurements. The composite shows an IUPAC Type IV isotherm with a H3 hysteresis loop. The data obtained from the analysis confirms that the composite is mesoporous, which will facilitate rapid ion diffusion, ideal for supercapacitor application[9,28,29]. The values of the surface area, pore volume and average pore diameter are given in Table 1. The isotherm of the composite is shown in Figure 4.

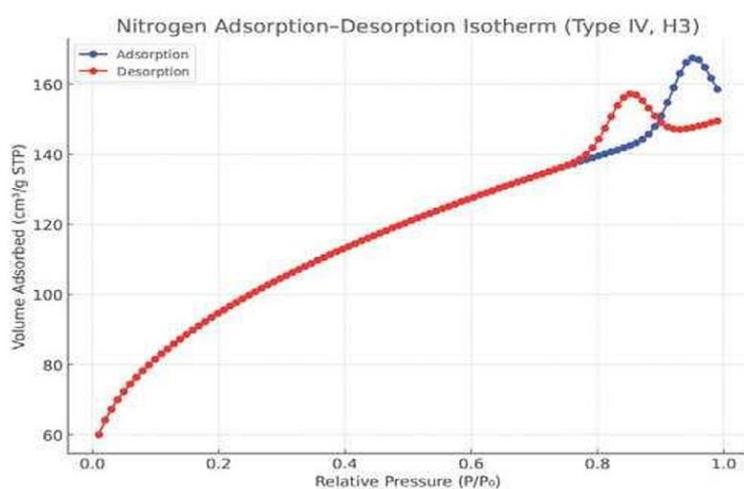


Figure 4. BET Isotherm of composite

Table 1. BET Surface Area Analysis Summary

Parameter	Value
Surface Area	520 m ² /g
Pore Volume	0.45 cm ³ /g
Average Pore Diameter	4.8 nm

4 Electrochemical Analysis

The electrochemical properties of the materials were analysed by Cyclic voltammetry (CV) and Electrochemical Impedance spectroscopy (EIS).

4.1 Cyclic Voltammetry (CV)

The electrochemical tests were done using a Gamry electrochemical potentiostat work-station. A 1.0M KCl electrolyte was used and the applied potential window was (-0.3 to+0.3) V. The CV curve of CA shows a quasi-duck shape which is common with electric double layer capacitance (EDLC) behaviour. The slope and sinusoidal pattern suggest the presence of graphite in the CA. This will support some pseudocapacitance through REDOX reactions[25,26,30].

The duck shaped cyclogram of the composite is associated with Faradaic behaviour, which will enhance the power and energy density of the material[28,31–33]. The specific capacitance of the CA is 191 F/g while that of the composite material is 750 F/g. The values of the specific capacitance were calculated from the data of the CV analysis. Importantly, the addition of the cobalt phosphate to the CA enhanced the electrochemical properties of the composite as expected. It also means that the material will performance excellently when applied as electrode material for HCDI desalination[34–37].

The cyclogram of the CA and composite are shown in Figure 5.

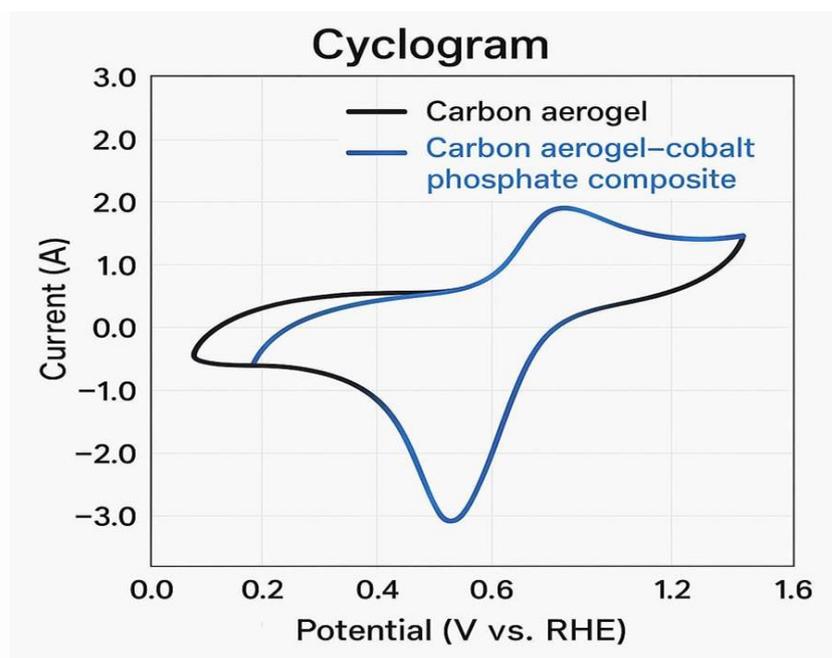


Figure 5, Cyclogram of CA and composite

4.2 Electrochemical Impedance Spectroscopy (EIS)

EIS was employed to study the charge transfer of the materials. The EIS was conducted using a three-electrode electrochemical work station. A 1.0 M KCl electrolyte was used, the applied potential was 0.22V and the frequency range is 10^5 Hz to 0.1 Hz. The EIS result shows that the charge resistance of CA is 199Ω while that of the composite is 100Ω . The Nyquist plot shows that the composite has a rapid charge transfer, which is demonstrated by the smaller semi-circle of the composite[38–40]. This supports the fact that the addition of the cobalt phosphate enhanced the electrochemical properties of the CA.

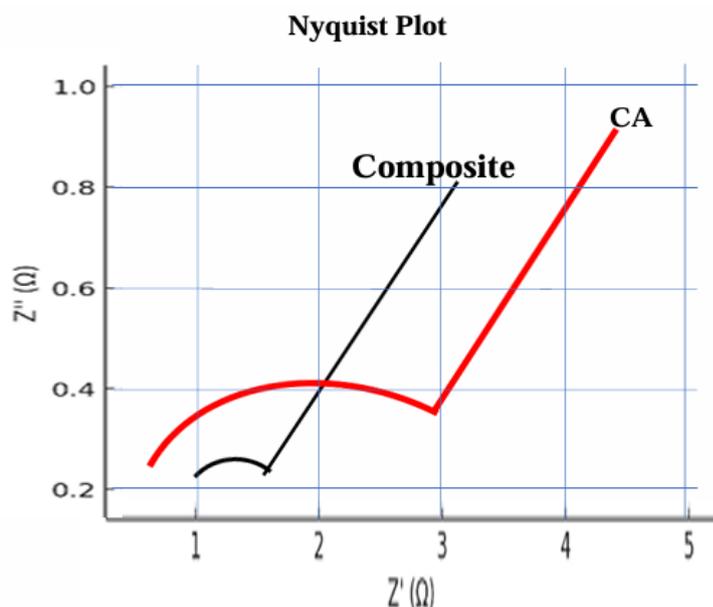


Figure 6, Nyquist plot of CA and Composite

5. Galvanostatic Charge/Discharge

Galvanostatic charge/discharge was used to investigate the electrochemical stability of the composite. The composite was stable over 2000 cycles of charge/discharge. The capacitance retention over the 2000 cycles was 99%. This means that the material can withstand 2000 cycles of usage and still maintain its electrochemical properties. Figure 7 shows a complete three cycles of charge/ discharge curve.

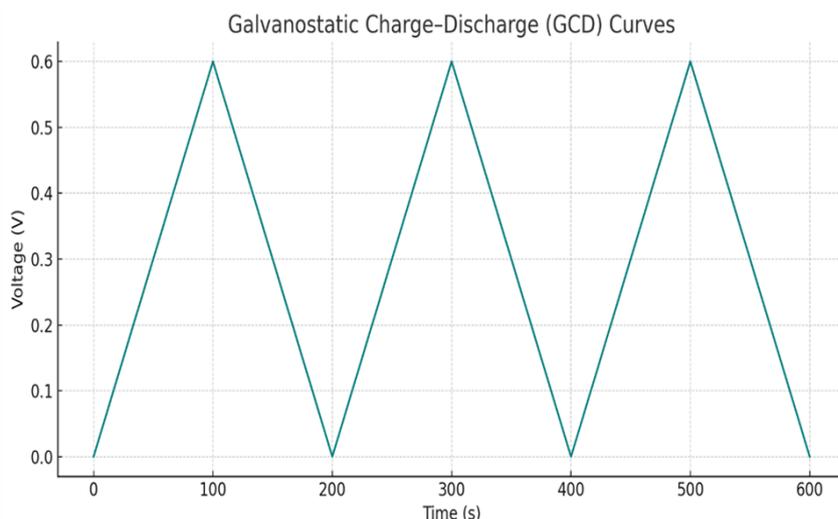


Figure 7, Galvanostatic charge/discharge curve.

6 DESALINATION EXPERIMENT

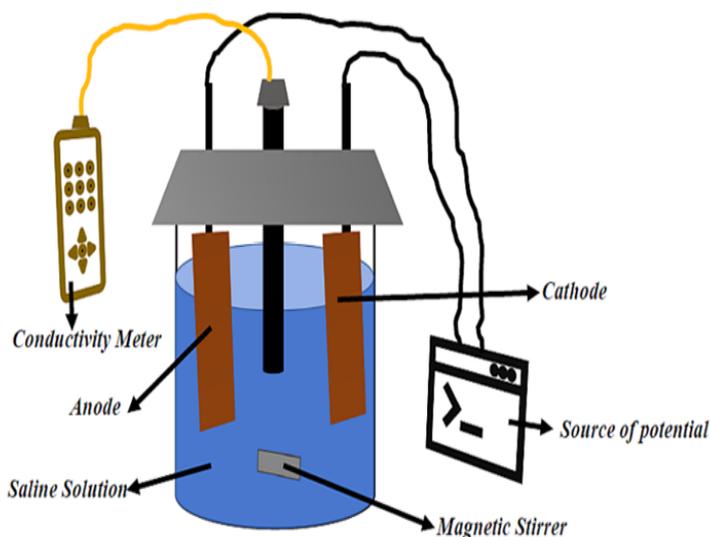
The desalination experiment was carried out in a batch mode using a CDI cell, as shown in Scheme 2. The cathode is activated carbon modified with the composite, while the anode is activated carbon. The electrodes are separated with a parafilm material, 1.5 cm apart in a 100 mL beaker containing 50 mL of NaCl solution. An electrical conductivity meter (abs) was used to measure the conductivity of the solution throughout the experiment. 1000 mg/L, 2000 mg/L, and 3000 mg/L concentrations of NaCl were used for this experiment. A direct current (DC) of 2.0 V was then applied to the CDI cell through the electrodes. The formula in equation 1. was used to determine the ion adsorption capacity (SAC) of the electrode.

$$SAC = \frac{\Delta C \times \Delta V(L)}{m} \quad (1) [41]$$

SAC = salt ion adsorption capacity, ΔC = Change in concentration of the saline solution, V = volume of saline solution, and m = mass of the composite on the electrode.

6.1 HCDI Desalination

Hybrid capacitive desalination experiment was done in a batch mode. 1000 mg/L, 2000 mg/L, and 3000 mg/L concentrations of NaCl solutions were used. At the start of the experiment, ion adsorption increases sharply from the conductivity meter reading, it gradually slowed until it stopped. The reduction in conductivity in the desalination experiment is indicative of ion adsorption. This demonstrates that the composite electrode material is electrochemically active and effective for HCDI desalination. It also proves that the desalination was successful. The desalination proceeds as the positive ions migrates to the negative electrode and negative ions to the positive electrode. The adsorbed ions were stored on and within the electric double layer (EDL) of the CA while it is stored by Faradaic intercalation in the cobalt phosphate within the composite electrode material until saturation is reached. The saturated electrodes were transferred into a beaker containing deionized water and the polarity of the applied potential reversed for desorption of the ions loaded on the electrodes. The desorbed electrodes are rinsed with deionized water and the desalination is repeated until the desired concentration of dissolved salt is reached. For freshwater, the concentration of total dissolved salt is about 500mg/L For deionized water all the dissolved ions need to be removed. The results of the desalination shows that the SAC at 1000mg/L is 76mg/g, at 2000mg/L the SAC is 112mg/g, and at 3000mg/L the SAC is 157mg/g. Table 2 shows the results and relevant data of the desalination.



Scheme 1. Schematic of HCDI Desalination[41]

Table 2, HCDI Desalination Data.

Conc. of NaCl (mg/L)	Desalination time (min)	Initial Conc. mg/L	Final Conc. mg/L	Mass of Composite (m)	Volume of NaCl (L)	Salt Adsorp. Cap. (mg/g)
1000	350	995	159	0.55	0.05	76
2000	310	1991	714.2	0.57	0.05	112
3000	260	2988	1198.2	0.57	0.05	157

Comparing Results

The results of this study shows that the electrode material adopted performed better than most of the previous electrode materials used for HCDI desalination study as could be seen in Table 3.

Table 3, Results compared with that previously reported.

s/n	Electrode Mat.	Specific Capac(F/g)	Applied Volt(V)	SAC (mg/g)	Reference
1	CA	141	1.2	5.27	[21]
2	A-NiO	46.53	1.2	7.46	[42]
3	CA	156	1.2	29.7	[43]
4	CA-PPy	360	1.2	15.7	[9]
5	Na ₃ V ₂ (PO ₄) ₃ @C	----	1.0	137.2	[11]
6	CA/Co ₃ (PO ₄) ₂	750	1.2	157	Current Study

7 Conclusion

The composite material CA/Co₃(PO₄)₂ was successfully prepared and its properties analysed. The results show that the composite has both electric double layer capacitance (because of the CA) and pseudocapacitance due to the presence of the cobalt phosphate. The specific capacitance of the composite from the CV analysis is 750 F/g. The electrochemical stability analysis shows that the material is stable over 2000 cycles of charge/discharge. The results of the HCDI desalination shows that the salt adsorption capacity of the composite at 1000 mg/L of saline solution is 76 mg/g, at 2000 mg/L is 112 mg/g, and at 3000 mg/L is 157 mg/g. The

results show that the composite performed better than previously reported carbon aerogel composite materials. This research further consolidates on the claim by Cao J. et al (2019) and other researcher with similar remarks that the application of Faradaic electrode materials as electrodes in HCDI desalination will overcome the limitations observed with carbon-based electrodes by improving their electrochemical properties and ion adsorption capacity.

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Conflict of interest: The author declares that there is no conflict of interest

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