

Chicken feather-derived carbon electrodes for capacitive deionization using poly(vinyl alcohol)-glutaraldehyde as the binder

Bakhtiar Ali Samejo^a, Naveed Qasim Abro^a, Najma Memon^a, Nusrat Jahan Upoma^b and Ahsan Habib ^{b,*}

^a National Centre of Excellence in Analytical Chemistry, University of Sindh, Jamshoro, Pakistan

^b Department of Chemistry, University of Dhaka, Dhaka 1000, Bangladesh

*Corresponding author. E-mail: habibchem@du.ac.bd

 AH, 0000-0001-6378-5915

ABSTRACT

Capacitive deionization (CDI) is an emerging technology which is being developed as a promising desalination alternative for charged species from salt water, being electrode materials as key drivers for highly efficient process. Here, we describe synthesis of highly porous activated carbons from chicken feathers using pyrolysis followed by chemical activation with potassium hydroxide in a 1:4 ratio to produce sustainable, scalable, and sustainable carbon electrodes for CDI. Poly (vinyl alcohol) (PVA) was used as binder to modify chicken feather activated carbons (CF-AC), which were subsequently crosslinked with glutaraldehyde (GA) to produce CF-AC-PVA-GA polymers through acetylation reaction. This resulted from improving hydrophilicity of CF-AC-PVA-GA polymers to raise the electrodes' resistance. Prepared materials were characterized using scanning electron microscopy, Fourier transform infrared, X-ray diffraction, Brunauer-Emmett-Teller and cyclic voltammetry. Fabricated CDI electrodes were used to investigate their performance for desalination, and exhibited different electrosorption capacity at different applied potentials. Modified electrodes possessed good stability in shear conditions and CDI process was stable and reproducible around 16 electrosorption cycles. Salt removal capacity of the modified electrodes (CF-AC-PVA-GA) was found to be 3.89 mg g⁻¹. The obtained outcomes offer important considerations of ions electrosorption and help advancing CDI system for water treatment and desalination.

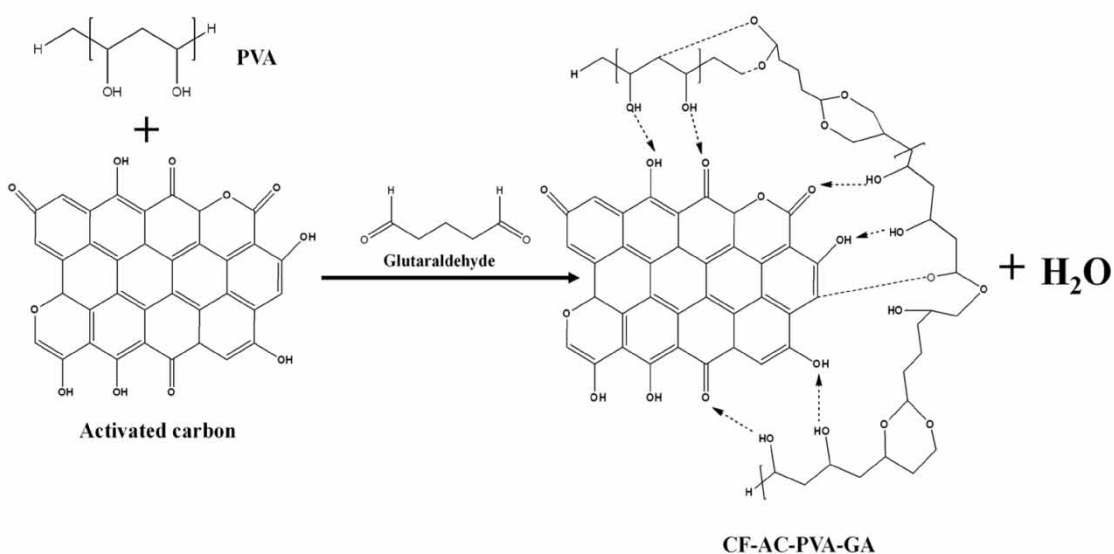
Key words: activated carbon electrodes, capacitive deionization, desalination, electrosorption, waste chicken feathers

HIGHLIGHTS

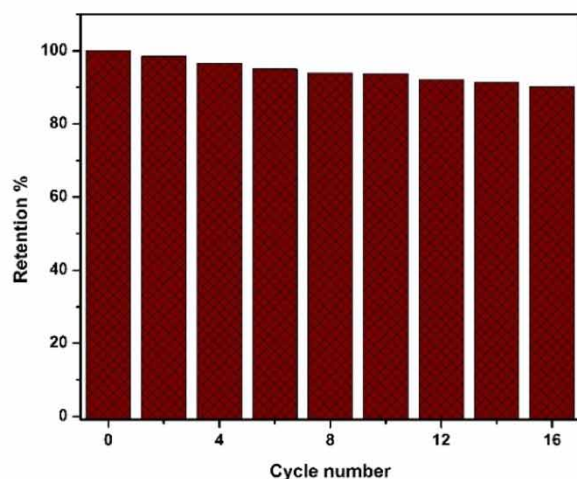
- Activated carbons (ACs) were prepared from chicken feathers via pyrolysis using KOH.
- Poly (vinyl alcohol) and glutaraldehyde were used for modification of ACs.
- Modified carbon materials were used for capacitive deionization (CDI) electrodes.
- The CDI electrodes retained good stability around 16 electrosorption cycles.
- Salt removal capacity of the fabricated CDI electrodes was found to be 3.89 mg g⁻¹.

GRAPHICAL ABSTRACT

Functionalization of the prepared activated carbon (AC) materials from chicken feathers (CF)



Electrosorption performance of the functionalized CF-AC electrodes



1. INTRODUCTION

Freshwater scarcity has emerged as a great concern globally due to the world's booming population and rising water demands (Elimelech & Phillip 2011). To overcome this situation, there are multiple options for water purification, the feasible and effective way to acquire freshwater from saline or brackish water has become the demand of the hour due to their abundant reserves on the earth, thermal-based (Ng *et al.* 2015), chemically activated systems (Youssef *et al.* 2014), membrane-based and electrical processes (Demirer *et al.* 2013) are used. These conventional techniques, for example, reverse osmosis (Kaschemekat *et al.* 1983) and multi-stage flash distillation consume high energy, have relatively poor efficiency, are environmentally damaging and take up massive infrastructure. As a result, an alternative is immediately needed. In this regard, capacitive deionization (CDI) has been introduced as an emerging technique for excellent desalination of low or moderate salt concentrations where salt ions are removed electrostatically from water. An electric field of low direct current voltage

(usually 1.2 V) is given to the porous carbon electrodes which attract the counter-ions toward their respective electrodes (Welgemoed & Schutte 2005; Xu *et al.* 2008, 2014; Li *et al.* 2009). CDI has a facile operation, high energy efficiency and is eco-friendly.

Electrosorption reaction takes place at the electrodes surface, hence it is significant to enhance the ions adsorption capacity of electrodes for better desalination. Various carbon materials, such as carbon fiber, carbon cloth, carbon aerogels and carbon nanotubes have widely been used as electrodes for CDI applications to improve the capacitance of carbon electrodes (Oren 2008). Also, electrodes should be mechanically stable and low cost which are crucial for commercially achievable CDI process. Instead of having fine electrical and physical properties these carbon materials have disadvantages due to their complicated synthesis procedures and are expensive for CDI applications. Whereas activated carbons are inexpensive and are excellent materials with high surface area. Therefore, for the CDI process AC is extensively used as carbon electrode materials. Also, for the synthesis of carbon electrodes polymer binders are used which exhibit thermal and chemical stabilities. However, polymer binders enhance internal resistance and may block some carbon electrode pores (Kinoshita 1988).

Therefore, with achieving applications, the feasibility of production is required in the industries and we need the prior factors in which the environment should be friendly and cost-effective. The fabrication of electrodes takes place by brushing or printing the electrode paste or applying a slurry on a substrate on a metal element such as nickel foam, a mixture of active materials, binders and conductive agents when stirred ultrasonically, the electrode slurry or paste is formed. To avoid the falling active materials during the performance of the electrode, binders play a vital role which attaches the conductive agent and active material to the substrate and it strengthens the electrode and the pore size of it. Nevertheless, the surface area and pores on the material are covered by the binders or cohesive agents. So, the properties of binders and their components direct influence the electrochemical performances of supercapacitors (Zhu *et al.* 2016).

In the CDI process, to attain better desalination performance, it is important to increase the adsorption capacity (capacitance) of carbon electrodes. The carbon electrodes capacitance depends on different factors such as oxygenated functional groups, pore size distribution, surface area and pore structure. Similarly, the wettability of the electrode surface area is crucial for better adsorption capacity and if there is no contact between the surface area and the aqueous solution that is not usable for salt removal. Hence, for high CDI performance, it is essential to enhance the wetted surface area of carbon electrodes, surface structure and functionalities that have a critical role in electrode wettability. Carbon electrodes prepared by using hydrophobic polymer binders such as polyvinylidene fluoride or polytetrafluoroethylene have low wettability of electrodes, which accounts for the decrease in adsorption capacity.

Poly(vinyl alcohol) (PVA) and poly(vinyl acetate) are water-soluble polymers, these can be used for enhancing the wetted surface area of polymer-bonded carbon electrodes. Because of innate hydrophilicity, thermal and chemical stability, good flexibility (Gao & Yang 2017) and high antifouling potential, PVA membranes possess wide applicability and are also utilized as a water treatment application. Though, PVA exhibits a large number of hydroxyl functionalities, but possesses poor water repellence and a high swelling degree in water, which cause limitation of PVA. The water repellence can be enhanced by acetalized groups compared to unmodified PVA which can destroy the film's reliability and reduce its performance. However, the water resistance of membranes can be improved through numerous cross-linking, methods for instance, heat treatment, freezing, irradiation and chemical cross-linking. Herein, the chemical cross-linking procedure exhibits the advantages to decrease the energy expenditure and time utilization. The most widely used chemical cross-linking agents of PVA include carboxylic acid, acid anhydride, acid chlorides, boric acid, epichlorohydrin, glycidyl methacrylate, and aldehydes. Aldehyde cross-linking is the most common route utilized because of the high rate of cross-linking. The formation of a three-dimensional network is achieved through the acetylation reaction of aldehyde compounds with PVA's hydroxyl groups (Zhang *et al.* 2010). Normally, acetalized PVA membranes synthesis is facile and settled: an aldehyde such as formaldehyde (Liao *et al.* 2018) or glycidyl acrylate mixed with hydrochloric acid, is combined with PVA aqueous solution and later pasted into membranes (Holland & Hay 2002) and the prepared membranes are thermosetting.

In this study, poultry chicken feathers waste was carbonized to produce carbon materials, which were then chemically activated at high temperatures by KOH. The slurry of carbon material and PVA solution was prepared and GA was added as a cross-linking agent and stirred to achieve a homogenous slurry. Scanning electron microscopy (SEM), X-ray diffraction (XRD), Fourier transform infrared (FTIR), and Brunauer-Emmett-Teller (BET) and cyclic voltammetry (CV) were used to evaluate the physical and chemical properties of the AC materials. The electrosorption performance of the synthesized CF-AC-PVA-GA electrodes was also investigated.

2. EXPERIMENTAL SECTION

2.1. Materials

Poultry chicken feathers obtained from a farmers' market were washed with distilled water to remove impurities. PVA was purchased from Daejung (Korea), glutaraldehyde and potassium hydroxide were supplied from Sigma Aldrich (Germany).

2.2. Synthesis of materials

The collected CFs were washed with distilled water in order to remove impurities. After drying at 80 °C for 24 h, the entire feathers were crushed using a high-speed pulverizer into small particles that had a fluffy appearance. Afterward, CFs were carbonized at 450 °C for 4 h. The KOH was used for the chemical activation of the synthesized material at a ratio of 1:4. KOH-impregnated CFs were carbonized at 600 °C for 2 h in an inert atmosphere (N₂ gas) to obtain CF-AC material (Evans *et al.* 2019) as depicted in Figure 1. Finally, the acquired activated materials, for instance, carbonized CFs, were dried overnight in a conventional oven at 101 °C after being washed with distilled water until they reached a pH of neutral.

2.3. Fabrication of carbon electrodes

CF-AC electrodes were fabricated by mixing CFs and polymer (PVA and GA). The mass ratios of AC, PVA and GA were taken 90:10, where the binders (PVA-GA) ratio was 1:1. Carbon slurry was prepared through mixing CF-AC with PVA solution and GA was added in the slurry dropwise as a cross-linking agent. PVA solution was dissolved in water (dissolved in DI water at 90 °C for 4 h), and later was crosslinked with GA because of its hydrophilic nature. The mixture was magnetically stirred overnight at room temperature (Park *et al.* 2011). The slurry was pasted on the copper sheet (2 cm × 4 cm) and dried at 70 °C for 1 h and affixed by heating in a thermostat oven at 120 °C for 3 h and pressed under 5 tons for 10 min to get a modified CF-AC-PVA-GA electrode as shown in Figure 5. Electrodes were washed with distilled water to remove loosely bound carbon and the amount of carbon was measured on electrodes by subtracting the weight of the same size copper sheet.

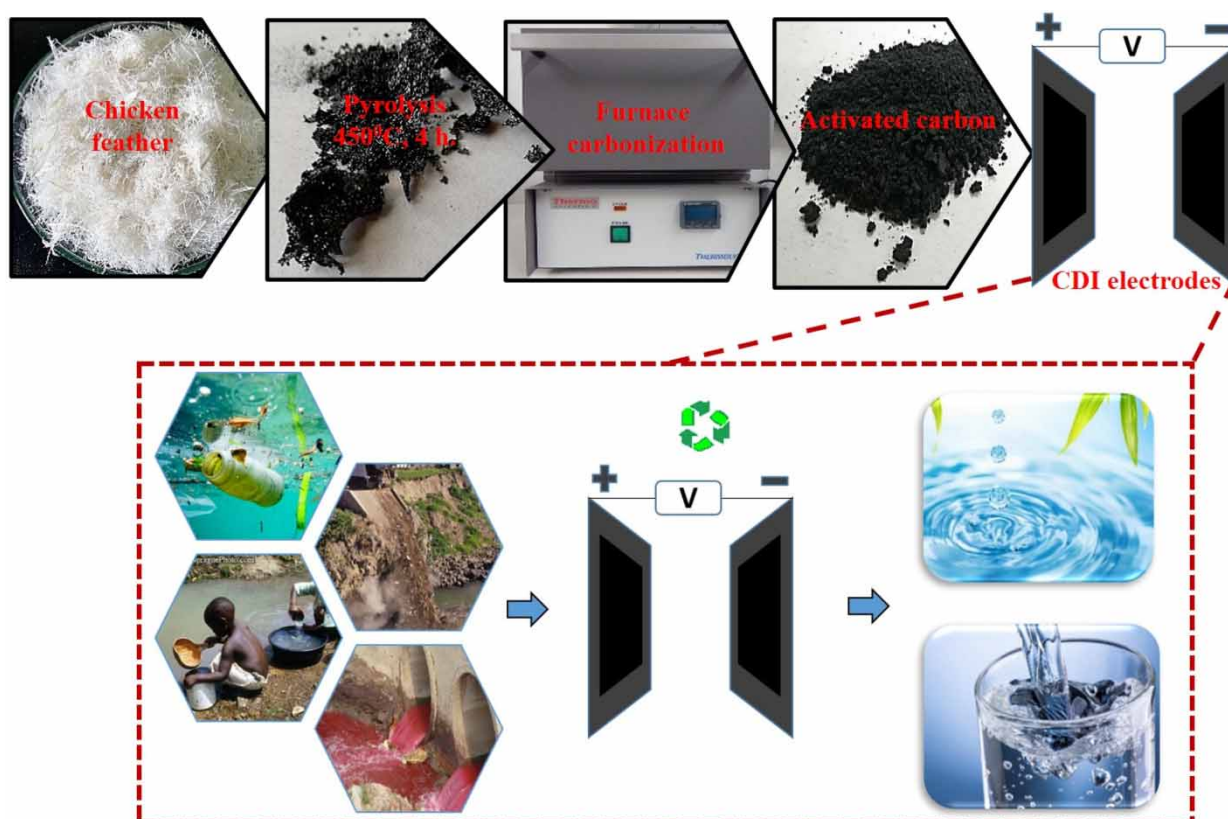


Figure 1 | Synthesis route of activated carbon from chicken feathers.

2.4. Characterization

2.4.1. Physical characterization of carbon material

XRD pattern of CF-AC was recorded on D8 Advance Bruker powder diffractometer with a Cu-K α radiation (Beijing Scientific Technology Co., Ltd). The Scanning electron microscopy (SEM) using Jeol LV-6490 was used to study the physical structure of carbons at an accelerating power 15 kV. Furthermore, the FTIR spectra were recorded using Thermo Scientific Nicolet TM iS10, samples were mixed with KBr and ground with mortar and pestle, and pressed into a transparent disk for analysis. Whereas, surface information and pore structure of CF-AC were characterized by nitrogen adsorption at 77 K using an Auto-sorb-iQ-MP. Samples were degassed at 300 °C for 6 h before adsorption experiments. CF-AC pores distribution was determined by employing Barrett-Joyner-Halenda (BJH) model for pores and Brunauer-Emmett-Teller (BET) for surface area.

2.4.2. Electrochemical analysis

CV (PalmSens4) was used to investigate the electrochemical performance of the prepared CF-AC electrode materials. A three-electrode configuration was applied with an Ag/AgCl as a reference electrode, the working counter electrode of Pt and Cu foil attached with active carbon material utilized as a working electrode (1 cm \times 1 cm). CV was used at different scan rates such as 30, 40 and 50 mV/s in a potential window of +1.0–0.0 V in a 1.0 M NaCl solution. Specific capacitance was measured using the following equation.

$$C = \frac{i}{v.m} \quad (1)$$

Here, C shows the specific capacitance (F/g), i is current density (A), v represents scan rate (V/s) and m is the activated carbon electrode mass (g).

2.5. Electrosorption experiment

Desalination experiments were conducted through a CDI setup to calculate the modified electrode ions removal performance and pairs of electrodes were arranged in a parallel manner. We prepared CF-AC-PVA-GA electrodes having 2×4 cm² area. Sodium chloride (SIGMA) solutions of 800 mg/L were used and the flow rate was set to 25 mL/min as depicted in Figure 2. The conductivity changes were measured with a conductive meter (EUTECH Pc510).

$$SAC = \frac{(C_o - C)V}{m} \quad (2)$$

Later, the salt adsorption capacity (SAC) was calculated using Equation (2). Here, C_o and C are initial and final concentrations (mg/L), V is the volume of salt water (mL) and m is the mass (g) of the electrode.

3. RESULTS AND DISCUSSION

3.1. Characterization of chicken feather-activated carbon (CF-AC)

Using the XRD technique, the crystalline structure of the synthesized CF-AC material was characterized and is depicted in Figure 3(a). Due to the lack of sharp bands, it was possible to detect two broad peaks in the CF-AC XRD patterns that occurred at 25° and 46°, corresponding to the 0 0 2 and 1 0 1 planes of graphite, respectively, as well as the minimal residual ash of structures (Isahak *et al.* 2013; Nunthaprechachan *et al.* 2013).

As can be seen in the SEM image in Figure 3(b), the SEM analysis of the CF-AC material revealed that it had flakes with a rough shape. The high rough surface of CF was visible after chemical activation. FTIR analysis revealed that the surface structure of the synthesized CF-AC materials included a variety of oxygenated functional groups (Figure 3(c)). The several distinctive peaks of the synthesized AC materials between 3,380 and 3,490 cm⁻¹ are related to -OH stretching vibration. The asymmetric stretching of the oxygenated functional group in the O-N molecule and the C=O molecule, respectively, may be responsible for the bands at 1,640 and 1,435 cm⁻¹ (Sugumaran *et al.* 2012; Xu *et al.* 2014).

Additionally, as shown in Figure 3(c), distinctive bands of the carbonized feather were identified as C=C stretching vibrations or C=O (stretching) around 1,700–1,600 cm⁻¹ and approximately 1,050 cm⁻¹ (Alfredy *et al.* 2019). While C-N stretching is shown by the band at 1,240 cm⁻¹ (Tuna *et al.* 2015). This shows that the corresponding amide peak was reduced

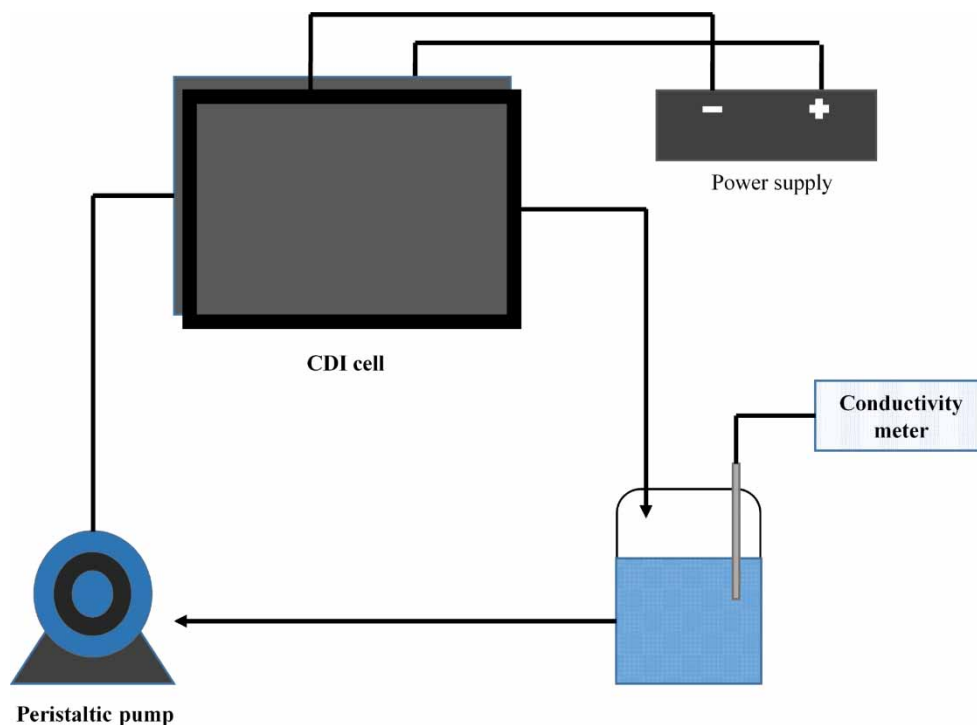


Figure 2 | Schematic diagram of CDI electrosorption setup.

by becoming a broad band between 1,240 and 1,200 cm^{-1} . Another amide peak that can be seen at 1,570 cm^{-1} also seemed to be lessened by converting into a wider band at 1,600 cm^{-1} , indicating that the N-H bond was probably broken (Brebun & Spiridon 2011; Tuna *et al.* 2015; Partuti *et al.* 2019; Chen *et al.* 2021).

3.2. Pore structure of activated carbon

As shown in Figure 4, nitrogen adsorption/desorption isotherms were used to investigate the pore structure of the prepared CF-AC materials. Nitrogen adsorption/desorption measurements at $-196\text{ }^{\circ}\text{C}$ were used to determine the surface area and diameter of the pore size. Before the adsorption experiments, the CF-AC material underwent a 6 h degassing at $300\text{ }^{\circ}\text{C}$ (Chandra *et al.* 2009). The surface area and pore diameter of the produced CF-AC material was calculated using the BET method, and the surface area was found to be $181.029\text{ m}^2/\text{g}$ and the pore diameter to be 3.709 nm. According to IUPAC classification, the AC-B and AC-J showed a typical type-IV isotherm indicating the mesoporous nature and adsorption proceeds via multilayer adsorption followed by capillary condensation. H4 loops are often found with micro-mesoporous carbons (Thommes *et al.* 2015). The pore volume of the activated carbon material was determined to be 0.046 cc/g using the BJH method. It should be noted that such pore distributions could result in the formation of a powerful electrical double layer during the deionization process (Hou & Huang 2013).

3.3. Fabrication of CF-AC electrode

The produced activated carbon electrode must be reliable in order to be used in desalination processes. Carbon electrodes should specifically be able to withstand the shear stresses seen in a water stream (Zhang *et al.* 2012). To explore their stability, the AC electrodes were made with various binder ratios, as shown in Figure 5. Table 1 lists the various ratios of activated carbon with binder ratios of PVA and GA (PVA/GA: 1:1), such as 85:15, 90:10, and 95:5, along with their electrosorption capacities. No active material was lost when rubbing a carbon electrode with an 85:15 ratio because of its relatively smooth and uniform surface. In comparison to the 95:5 fabricated electrode, which had significant surface cracks, the prepared electrode with a 90:10 ratio revealed only tiny cracks and lost a modest amount of active material after rubbing (Fang *et al.* 2016). Thus, the prepared electrode with 90:10 ratio was utilized for further studies.

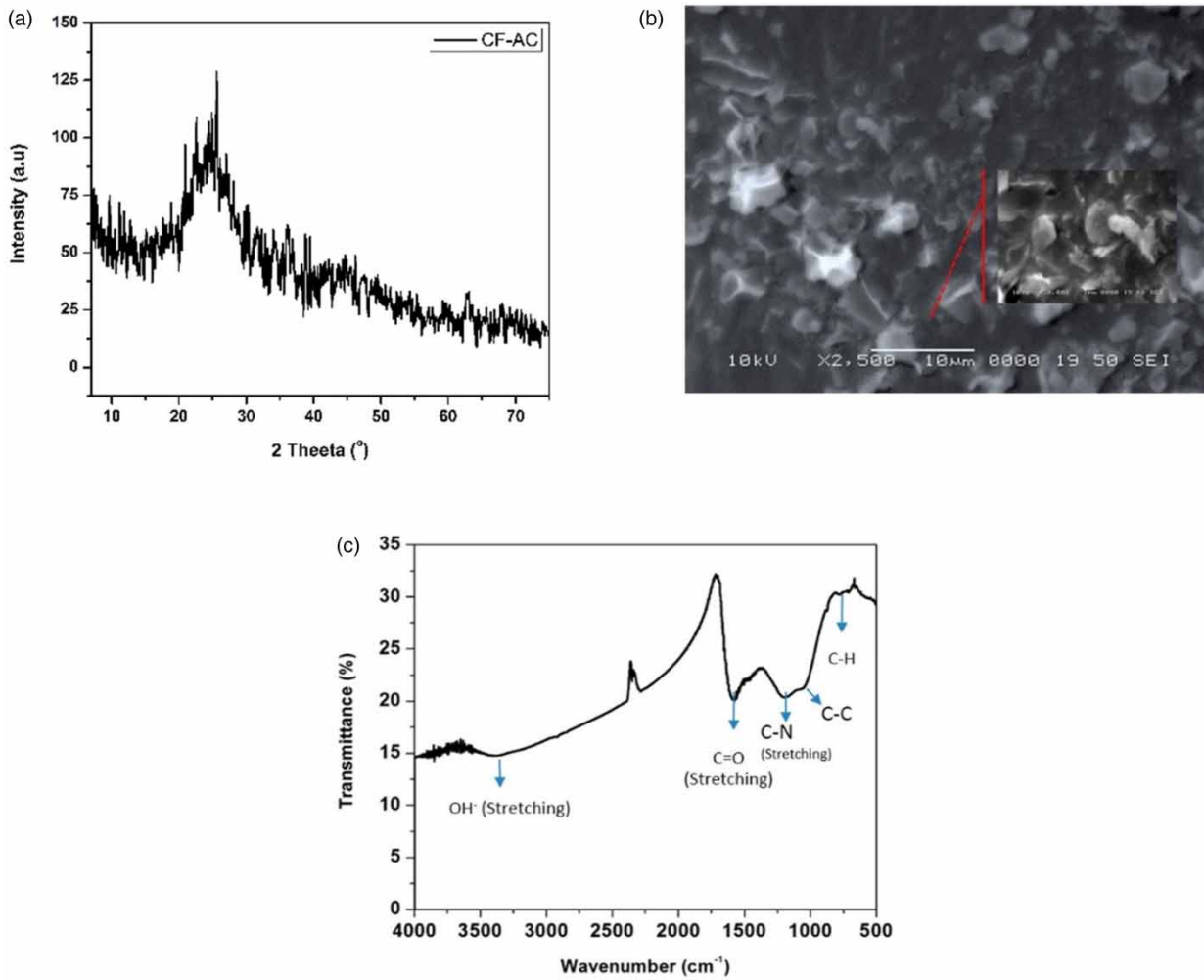


Figure 3 | (a) XRD pattern, (b) SEM image and (c) FTIR spectra of CF-AC material.

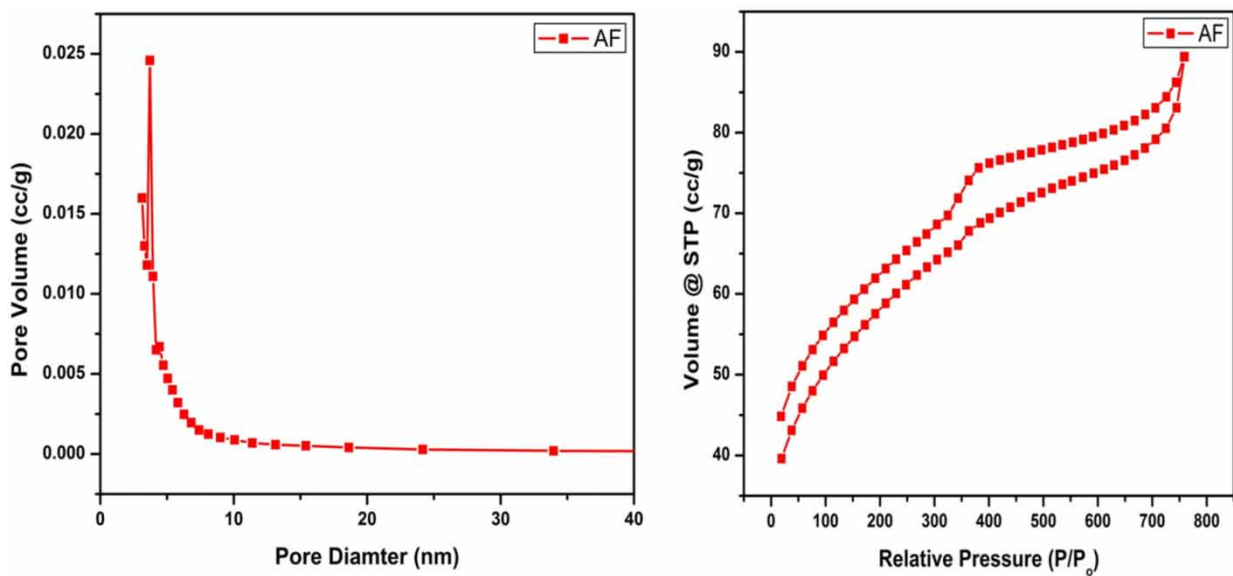


Figure 4 | N₂ adsorption/desorption isotherm and BJH pore size distribution of CF-AC material.

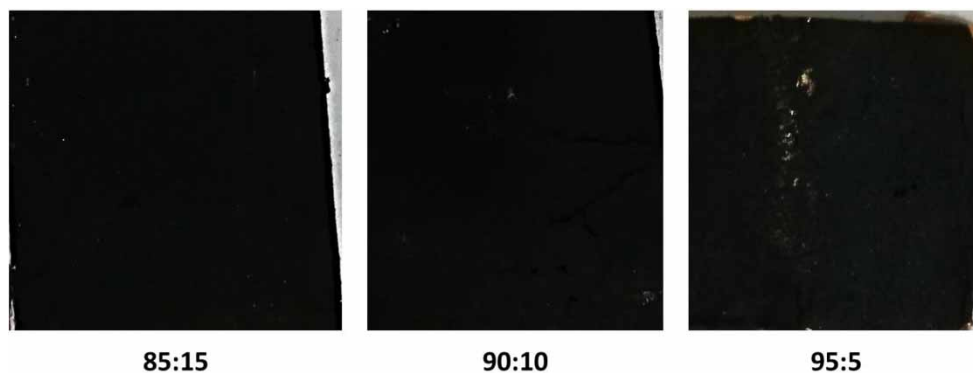


Figure 5 | Carbon electrode fabrication at different ratios.

Table 1 | Carbon electrode fabrication at different ratios

Active material	Active material + binder (PVA-GA) ^a ratio	Applied voltage (V)	SAC (mg/g)
CF-AC	85:15	1.2	2.06
CF-AC	90:10	1.2	3.89
CF-AC	95:5	1.2	2.47

^aBinder (PVA-GA) ratio is 1:1.

3.4. Morphology of PVA-GA-activated carbon electrode

A uniform thin layer electrode was fabricated using the modified activated carbon materials made from chicken feathers (CF-AC-PVA-GA). As shown in Figure 6(a), the intermolecular crosslink chains between the activated carbon and PVA and between individual PVA chains are formed during hydrothermal treatment. The majority of these crosslinks were formed through acetalization reactions between glutaraldehyde and the hydroxyl functional groups of PVA, or either of the activated carbon (Podsiadlo *et al.* 2007; Figueiredo *et al.* 2009; Tan *et al.* 2013). As the hydroxyl groups are more prevalent in PVA molecules, it is important to note that cross-linking largely took place between the discrete PVA series as opposed to on the activated carbon. The prepared CF-AC-PVA-GA electrodes also demonstrated good durability and appeared repeatable for more than 16 cycles (as shown in Figure 6(b)), in contrast to other PVA-based CDI electrodes that showed stability around 5 cycles. Furthermore, the minimal active material loss was seen, and electrode stability of 90.2% was obtained after 16 electroadsorption cycles. This may be because the hydroxyl functionalities of the PVA and GA showed increased stability due to intermolecular cross-linking.

3.5. Cyclic voltammetry

The electrode capacitance of the prepared CDI materials was calculated using CV, demonstrating the ability of charged species accumulation to produce the EDLs across porous carbon electrodes (Cai *et al.* 2017). However, ions adsorb more readily with higher capacitances, which improves desalination efficiency. Figure 7 shows the capacitance of a CF-AC-PVA-GA electrode at various scan rates. Although no apparent oxidation/reduction peak could be seen on the graph, this indicates that charged species are adsorbed through electrostatic forces rather than redox reactions (Yin *et al.* 2013; Moronshing & Subramaniam 2017). The symmetry of the CF-AC-PVA-GA electrode curve shows that ions adsorption may be highly reversible. According to Figure 7, the curve area was used to calculate the capacitance of the CF-AC-PVA-GA electrode. The CF-AC-PVA-GA electrode material showed a specific capacitance of 81.08 F/g in the CDI system.

3.6. PVA-GA carbon electrodes electroadsorption performance

Using a CDI cell with an initial NaCl concentration of 800 mg/L, the desalination performance of the modified electrodes was evaluated. The CDI cell received a 5 min application of cell voltage to reach maximum saturation before being promptly reduced to 0.0 V to regenerate the electrode surface for the following cycle. The concentration of NaCl was reduced to its

(a)

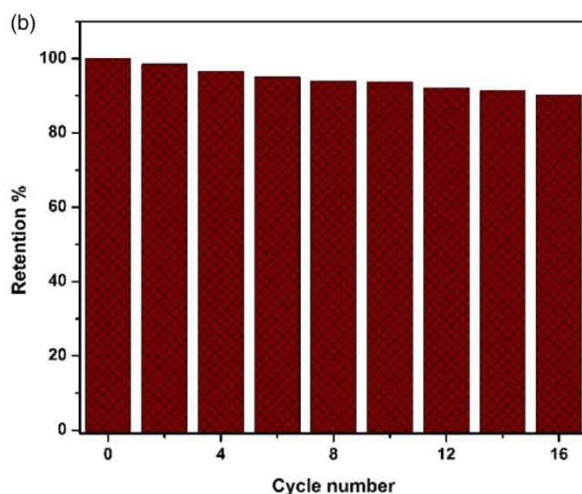
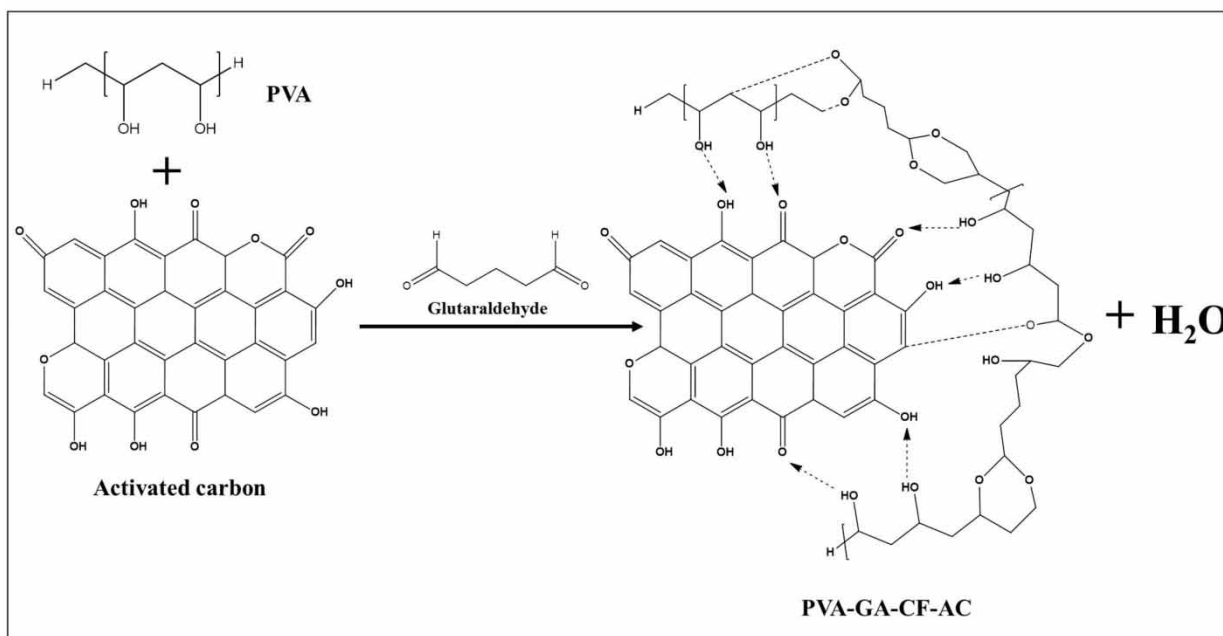


Figure 6 | (a) Acetalization reaction between PVA and GA and (b) electrode stability after the electrosorption process of CF-AC-PVA-GA electrode.

lowest level as the adsorption took place after applying the potential difference across two electrodes, and it then steadily increased as the CF-AC-PVA-GA electrodes were fully saturated (Park *et al.* 2011). Since the applied voltage and concentration have a substantial impact on the deionization efficiency of the CDI electrodes, different potentials of 0.6, 0.8, 1.0, and 1.2 V were applied to the electrodes, as shown in Figure 8 (Farmer *et al.* 1995; Oren 2008). At a cell potential of 1.2 V, the CF-AC-PVA-GA electrode's maximal capacity for NaCl adsorption was measured to be 3.89 mg g^{-1} . The fabricated electrodes also showed excellent endurance and repeatability for a period of 16 cycles and had good stability against feed water shear stress (Table 2). However, the effectiveness of modified electrodes needs to be increased more in order to make the desalination process economical. By adjusting the level of the cross-linking agents, activated carbon materials, temperature, and applied voltage, the desalination efficiency of the modified electrode can be improved.

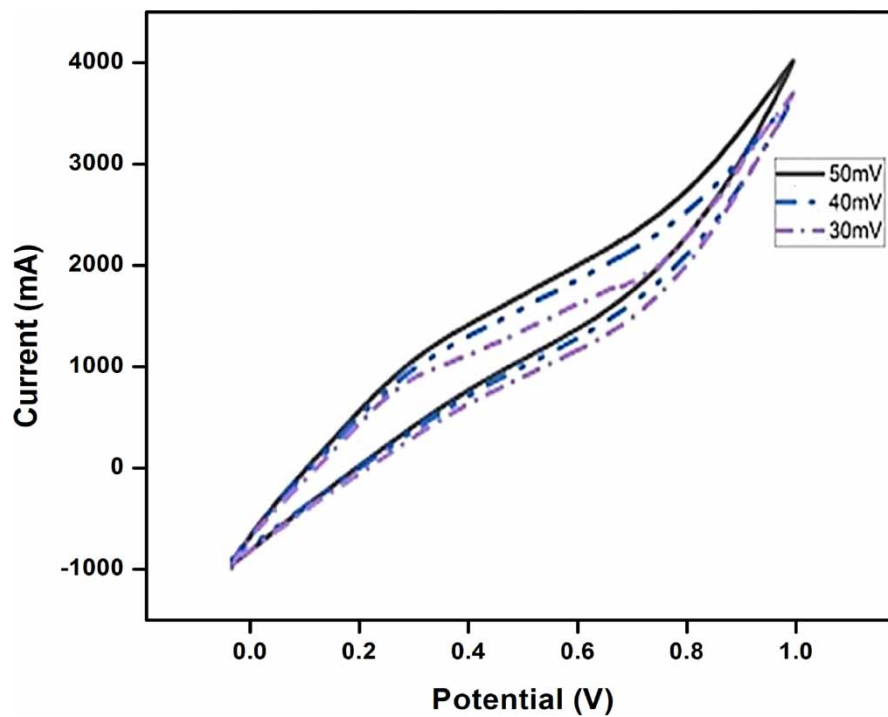


Figure 7 | Cyclic voltammograms of CF-AC-PVA-GA at different scan rates.

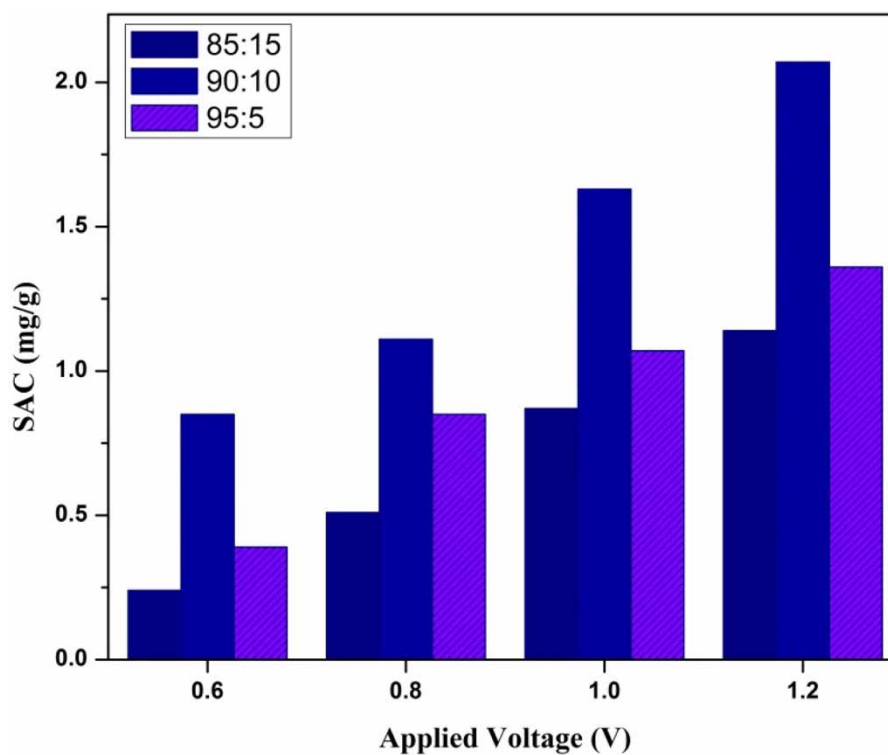


Figure 8 | Electrosorption performance of CF-AC-PVA-GA electrodes.

Table 2 | Summary of electrode stability performance of various CDI electrodes

S. No	Electrode material	Capacitance (F/g)	No. of cycles confirming stability	Application	References
1	MAX (Ti ₃ AlC ₂)	347	10	Removal of Cr _(IV) ion	Bharath <i>et al.</i> (2020)
2	FePO ₄ @rGO	–	10	Removal of NaCl from water	Ma <i>et al.</i> (2019)
3	α -MnO ₂ /G	375	4	Separation of sodium and chloride ions from saline water	Jaoude <i>et al.</i> (2020)
4	GA10_T120	–	5	Removal of NaCl solution	Park <i>et al.</i> (2011)
5	CSG	207.4	4	Removal of brackish water	Chang & Hu (2019)
6	Commercial AC	–	10	Removal of paraquat (PQ) herbicide	Alfredy <i>et al.</i> (2021)
7	NaOH-Ti ₃ C ₂ T _x //AC	–	20	Removal of brackish water	Chen <i>et al.</i> (2020)
8	CF-AC-PVA-GA	81.08	16	Removal of NaCl solution	This work

4. CONCLUSION

To improve the wettability of the CDI electrode, the activated carbon synthesized from chicken feathers was chemically treated with KOH and subsequently functionalized using PVA binder. The fabricated activated carbon was used as an electrode material for the desalination process. However, as PVA is a water-soluble polymer binder, in order to make it insoluble, PVA was crosslinked with glutaraldehyde through an acetalization reaction. The PVA-GA carbon electrodes were prepared at different binder's ratios and heated at 120 °C overnight in order to make it a homogenous slurry. Through nitrogen adsorption–desorption isotherms, it was observed that most of the synthesized possessed mesopores which contribute to higher electrosorption performance. CV exhibited good conductivity properties of the functionalized activated carbon (CF-AC-PVA-GA) and the value was found to be 81.08 F/g. The functionalized CF-PVA-GA-activated carbon electrodes were used for the removal of salt where the SAC was found to be 3.89 mg g⁻¹ at 1.2 V. Furthermore, CF-AC-PVA-GA electrodes exhibited good stability in the shear conditions and, it was observed that the adsorption/desorption cycles of the fabricated CDI electrodes were more reproducible and stable more than 16 cycles with minimum loss of the active material. In conclusion, PVA binder fabricated carbon electrodes can be efficiently utilized for CDI performances with considerable certainty of better results.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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