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Original Research

Investigating the Reliability of Zinc Oxalate Based Porous Polymeric Crystals on Gossypiumherbaceum in Battery Application as Separators

Naveen Kumar Saravanakumar [1], Prithika Saravanakumar [2], Kavitha Nagarasampatti Palani [3], Jothi Ramalingam Rajabathar [4,*], Saleh M. H. Aljamhoor [4], Selvaraj Arokiyaraj [5]

[1] Department of Chemical Engineering, AC Tech Campus, Anna University, Chennai, India;

[2] Department of Physics, The Gandhigram Rural Institute, Chinnalapatti, Dindigul District, India;

[3] Department of Chemical Engineering, Sri Venkateswara College of Engineering, Sriperumbudur, Kancheepuram, India;

[4] Chemistry Department, College of Science, King Saud University, P.O. Box 2455, Riyadh 11451, Kingdom of Saudi Arabia;

[5] Department of Food Science and Biotechnology, Sejong University, Seoul 05006, South Korea.

* Correspondence: jrajabathar@ksu.edu.sa

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Abstract: With the concept of Waste-to-Energy our team has developed a more-promising composite separator material for battery applications. Zinc-based porous co-ordination polymeric crystals (CALF-20) were made to grow on the surface of cellulose-based substrate, residual cotton (Gossypium herbaceum). These CALF-20 crystals were produced in a greener approach utilizing the aqueous solvent caustic potash (i.e. mother liquor). The compatibility behaviour between CALF-20 crystals and cotton was enhanced by the addition of surface modifier dopamine. The results of the scanning electron microscopy (SEM) were excellent, and it revealed the good adherence characteristics developed between CALF-20 crystals and the cotton. The sharp crystalline peak at 2θ = 22.36° in 002 plane of X-ray diffraction (XRD) spectra proved the strong bonding of hydroxyl molecules onto the cotton surface. The Brunauer-Emmett-Teller (BET) surface area was calculated to be 23.98 m2/g and the isotherms obtained demonstrated the existence of both microporous and mesoporous structural characteristics. The charge transfer resistance (Rct) is found to be low with high ionic conductivity. From the above-mentioned results, it is highly positive that our composite could serve its purpose as separator in batteries.

Keywords: aqueous solvent; porous co-ordination polymeric crystals; cotton; microporous; mesoporous; separator

1. Introduction

The metallic substances ligated to organic compounds by means of strong chemical bonding results in the formation of Metal-organic frameworks (MOF). They possess another name asporous co-ordination polymers (PCPs). These materials owe alarming properties such as increased crystallinity and microporous nature with large specific surface area (SSA) of around 70,000 cm^2/mg make these frameworks suit into the domains like gas adsorption, photo catalysis, drug delivery, clean energy and effluent treatment [1]. When looking into its molecular structure it gets tuned easily depending on the application for which it is being employed [2]. Another major advantageous

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property of these materials are they could withstand elevated temperatures [3].These MOFs are formed by bridging the inorganic metal ions with organic complexes [1]. Although, they

are advantageous in many aspects, there are some limitations associated with it like poor electrical conductivity and extremely narrowed microporous structure which hinders the successful implementation in the energy storage devices [4]. Irrespective of its pros and cons it could not be employed effectively in numerous applications as they result in powdered forms upon synthesis. To make the crammedutilization of the MOFs without any wastage it could be processed into thin films or membrane-like structure [5].Another emerging method is growing crystals of MOF on the surface of various fibers which are formed because of electrospinning technique [6]. The growth of MOF crystals onto a substrate which is commercially viable, easy to access at low-cost without causing hazard to the environment gives aproductive means to extend its applications in potential ways.The cellulose-based materials are found to be the best substrate and it has met out all the requirements mentioned above for the growth of MOF crystals onto it [7].

Cellulose is one of the major abundantly occurring biopolymer in nature and it has got various forms upon processing like cellulose nanofibers, cellulose nanocrystals, and whiskers with wide range of applications such as filter cloth, membrane filter in filtration process, fabrics in textile industry, fillers for adhesives and many more applications in the field of medicine, pollution control and so on. When looking into the properties of cellulose it has got good tensile strength, it is cheap and found in plenty in environment with excellent process stability towards the processes with ready recyclable ability and all these properties makes cellulose a suitable material for making composites. Although it possesses many advantages, it could not compete with the synthetic polymers due to its hydrophilicity and polar nature and it does not compatiblyget along with hydrophobic and nonpolar substances. The adherence characteristics of the substrates made of cellulose could be improvised by doing the surface modification to grow the crystals of MOF [8].

On analyzing the surface modification agents of different substrates, dopamine is found to be the best surface modifier and an inspiration is made by the protein chemistry of the mussels living in marine eco-system. The mussels possess the ability to attract foreign objects which finds its origin from proteins and that is a rich source of catechol groups which is extracted from lysine (amine) and 3, 4-dihydroxy-L-phenylalanine groups [9]. The similar functional groups shall be observed in tiny dopamine molecules which could be polymerized under moderate circumstances with the application in nearly all types of superficial layers through catechol and amine based functional groups. The polymerization mechanism of dopamine to polydopamine is hard to understand as it is a self-polymerization reaction, but a modified surface could be obtained employing dopamine as a surface modifier on substrates through easy way of treatment even at room temperature. Upon treating the surface of the substrates with dopamine we could explicitly notice the formation of thin polymeric film andthere exists a direct relation between the cake layer thickness and the immersion time. Hemmatpour et al. used dopamine as a surface modifier and experimented on different substrates for growing thin films of MOF-5 on polydopamine where polydopamine acts a nucleation site [10]. The crystal growth of MOF-5 surrounding the cellulose fibers were investigated by Mirkovic et al. and it resulted in the necklace like structural morphology. On addition to it, there is afirm bonding of crystals on to the superficial layer of cotton [11]. Kusgensandhis co-workers testified the HKUST-1 crystals growth on fibers of pulp and these MOF crystals were grown by employing in-situ technique on the pulp fiber substrate. From the works of Kusgens and his team we could observe that coverage of surface area gets varied with the ratio of chemicals used in the fibers [12]. The immobilized crystal of copper-basedMOF on substrates of cotton employing the surface modification of cotton with linkers of carboxylate ions upon dipping the surface modified cotton into mother liquor solution of MOF [13]. The results obtained by his team showed the crucial importance of surface coverage making surface modification of the substrates [14]. On making the analysis of all the literature works, we could observe that crystals of MOF grow well on the substrates of cellulose-based materials. The variables such as displacement and pore pressure present in the poroelastodynamic model can be readily used as an alternative to analyze membranes in batteries [30]. And also, all these literatures used organic solvents for preparing MOF seed solution which could be poisonous and vulnerable to the globe.

On considering the environmental impacts caused by the organic solvents to the earth, the main aim our research team is to use the caustic potash for the first time instead of organic solvents toprepare MOF seed solution and thereby generating MOF crystals to alleviate toxicity. These resulted MOF crystals are made to seat on the cotton, acellulose-based substrate and this work also demonstrates how the surface area increases due to substrate modification along with the growth ofzinc-oxalate based MOF, Calgary framework (CALF-20). The performance of MOF crystals is analyzed through various instrumental analysis such as FTIR, XRD, SEM, TGA, BET, Cyclic Voltammetric studies and EIS. This work suggests that this prepared material could be employed as separators in batteries and it could be primarily inferred through the CV and EIS results.

2. Essential resources and implemented methodology

2.1. Necessary supplies

The employedchemicals list consists ofzinc oxalate dihydrate ($ZnC_2O_4.2H_2O$), 1,2,4- Triazole ($C_2H_3N_3$), dopamine hydrochloride ($C_8H_{11}ClNO_2$), terephthalic acid ($C_6H_4(CO_2H)_2$), potassium hydroxide(KOH), methanol (CH_3OH), tris(hydroxymethyl)aminomethane (Tris) ($C_4H_{11}NO_3.C_2H_4O_2$) and deionised water. The supply of chemicals and reagentsfrom Sigma were found to possess high purity, and hence, they were used directly without any purification. The remains of *Gossypiumherbaceum*after cultivationcommercially referred to as cotton (a cellulose-based substrate) employed in this work is acquired from the local farms located at Dindigul district of Tamil Nadu, India.

2.2. Modification of substrate

Cotton was made into small bulbs. It was rinsed with acetone and made to dry at 40°C. 10 mM of Tris was taken, and it was made into a solution by adding 200 ml deionized water. Then the 1 g of dopamine hydrochloride was made to dissolve in Tris solution. Our substrates earlier treated with acetone and dried was made to immerse completely in dopamine hydrochloride solution for 24 hours and then washed with ethanol several times to get rid of excessive dopamine. After washing, drying of substrate was done in the oven at 65°C for a span of 1 day.

2.3. Preparation of CALF-20 framework

The dissolution of 8 g of zinc oxalate dihydrate and 5 g of 1,2,4- Triazole were done in 70 ml carbinol, and it was stirred continuously for 1 hour. Then the Teflon autoclave with makeup solution was placed in the hot air oven for the duration of 48 hours with temperature maintained at 200℃. Then the gradual cooling of the resultant white precipitate solution was carried out until the solution reaches 35°C. The washing of the obtained solution with methanol for 3 times followed by drying at 50°C at 1 h duration yields the expected MOF, CALF-20 frameworks.

2.4. Preparation of CALF-20 seed solution

The CALF-20 mother liquor is prepared by adding 4.2 g of CALF-20 and 970mg of $C_6H_4(CO_2H)_2$ to 200 ml of 3 M KOH solution. The colorless mother liquor was then prone to heating at 65°C for 2days. Upon heating we could observe the formation of CALF-20 crystals at the bottom of the beaker. The formed CALF-20 crystals were then brought toatmospheric temperature by natural cooling phenomena. The cooled crystals were then cleaned with the deionized water. The cooling process was followed by drying in the air circulating oven with 65°C for a day to yield the CALF-20 crystals.

2.5. Preparation of CALF-20 @ cotton

The modified substrate was made to immerse in the CALF-20 mother liquor and it was allowed to stay in the solution for 48 hours at 65 °C. After immersion, the formed CALF-20 @ cotton were subjected to washing in the presence of deionized water following by drying in the oven with circulation of air for 24 hours.

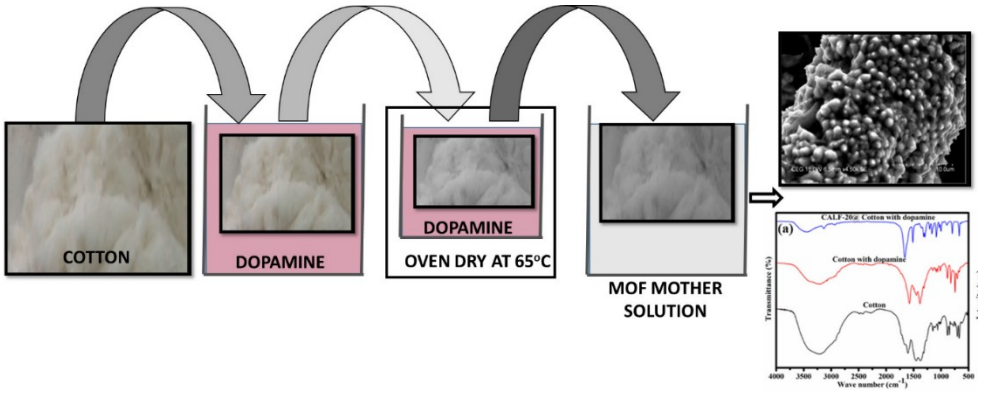

Figure 1. The synthesis route for the yield of CALF-20 crystals @ Cotton with dopamine.

2.5.1 Characterization

The structural appearance of the cotton and the modified substrates were analyzed employing the technique of scanning electron microscopy(SEM) applying 20 kV and 20 mA by employing the device manufactured by Hitachi (Model No. S-3400 N). The chemical bonding between the molecules were examined with the help of Fourier Transform infrared (FTIR) spectroscopy adopting spectrophotometer (THERMO iS50) at an intensity varying between 4000 cm^{-1} to 500 cm^{-1}. The diffractograms were obtained employing X-ray diffractometry (XRD) studies for our samples. The samples were mounted on the pure Si wafers and the data acquisition was done employing XRD (Bruker D8 advance) instrument. The sealed tube made up of **copper** acted as a source material and it produced a required wavelength λ of 1.5406nmat 35 kV voltage,0.045 acurrent with the micro-slit distancedat 0.5 mm. The 2Θ values were ranged from 0 to 80°. Thermogravimetric analysis (TGA) was done employing TGA analyzer (Q500 Hi-Res, TGA)with a temperature ranging from 0 to 800 °Cunder the influence of atmospheric airby employing the rate of heatingwith 10 °Cper minute.The examination of the pore size distribution and the specific surface area (SSA) of our composite wascarried out by the application of Bruner–Emmett–Teller (BET) analytical method and on the other hand, the nitrogen adsorption-desorption isotherms curve data was given out by the Micrometrics ASAP 2020 analyser.

2.5.2 Electrochemical characterizations

The synthesized materials were subjected to electrochemical stability with the help of cyclic voltammetry (CV) curves and impedance spectroscopic (EIS) curves obtained from electrochemical workstation Autolab PGSTAT 204. These studies were done under the conditions of ambient temperature with 1 M sulfuric acid (H_2SO_4) electrolyte and the samples were sandwiched between zinc and stainless steel (SS) electrodes.The CV curves were obtained applying progressively increasing scanning rates over a range of 10 to 500 mV $s^{-1}$with voltage window ranging from -0.5 to 2V. The EIS curves were obtained by employing a frequency of range 0.01 – 100 kHz. The computation of ionic conductivity, σ (mS cm^{-1}) was done by employing Equation 1 [15].

$$\sigma = \frac{L}{R_b s} \qquad (1)$$

In this equation L represents thickness of separator (cm), R_b represents bulk resistance (Ω) and S represents the composite material's area (cm^2).

3. Results obtained & detailed discussion

3.1. Scanning electron microscopy

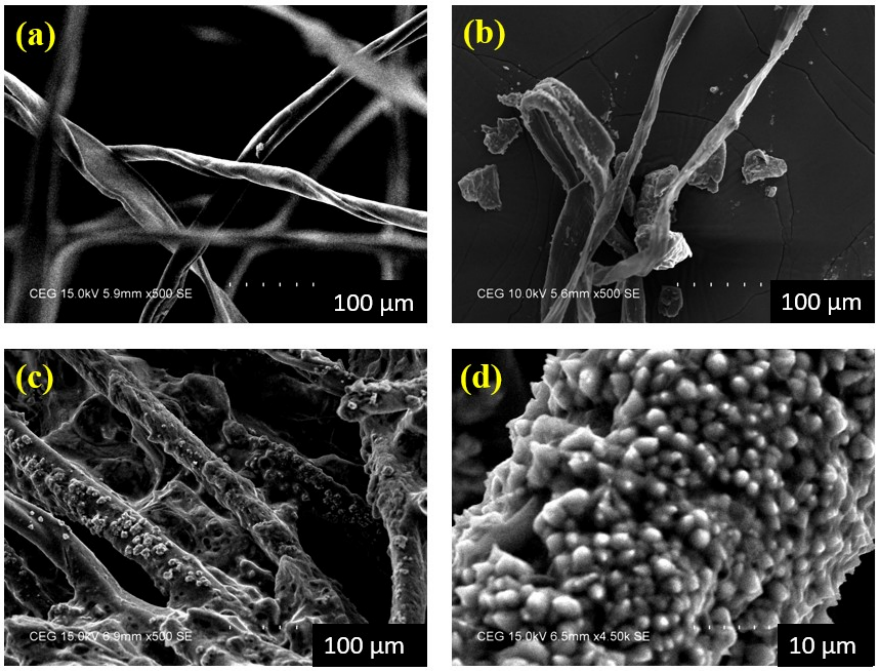

Figure 2. Scanning electron microscopy (SEM) photosdepicting (a) Cotton (b) Cotton with dopamine (c) CALF-20 @ Cotton with dopamine and (d) Magnified view of CALF-20 crystals @ Cotton with dopamine (10 µm).

The growth of CALF-20 crystal and its adherence to the cotton, a cellulose-based substrate on influence of the surface modifier, dopamine was clearly analyzed employing the obtained SEM photographs shown in Figure 2. On seeing the SEM image of cotton (Figure 2a) there is an existence of uneven twisted cylindrical structures, and these structures demonstrate that cotton is made of cellulose fibers [16]. The resulted SEM image (Figure 2b) of the surface modified cotton employing dopamine shows the presence of some uneven particles sticking to the cotton surface and it may be attributed to the vigorous polymerization reaction of dopamine to polydopamine.

The cellulose hydrophilicity is found to reduce because of this modification, and it is due to the reacting nature of hydroxyl groups found on the exterior of the substrate with catechol groups associated with dopamine [17]. It is evident from the Figure 2c & 2d that the growth of CALF-20 on modified cotton substrates were comparatively high, and this might be due to the loosely bound fibrillated filamentous structure associated with the cotton bulbs. Additionally, the effective coating of polydopamine established on the bounded surfaces of the cotton provide adequate sites of nucleation for CALF-20 crystals. Upon reviewing the SEM images, we could infer that cotton holds the superior place to grow CALF- 20 crystals [18, 19].

3.2. Analysis of fourier transform infrared spectra and X-ray diffracted patterns

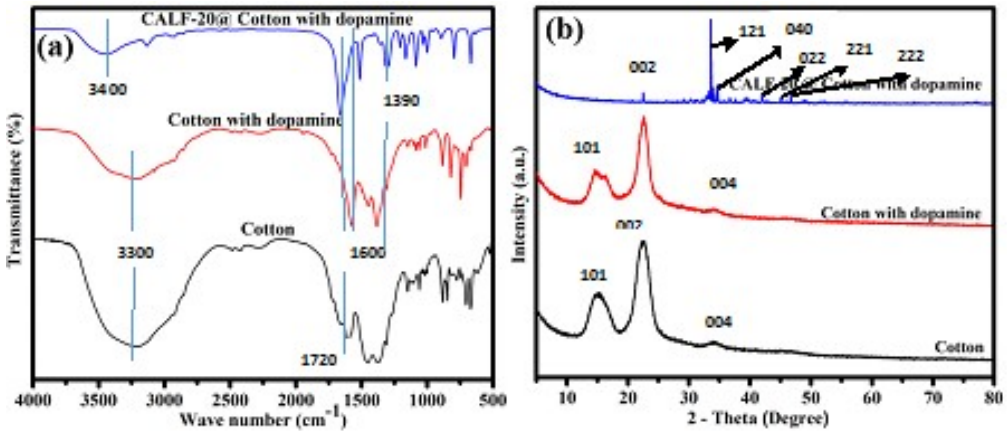

Figure 3. a) FTIR spectra illustrating Cotton, Cotton with dopamine and CALF-20 @ Cotton with dopamine, b) X-Ray diffractions showing Cotton, Cotton with dopamine and CALF-20 Cotton with dopamine.

The FTIR spectra shown in Figure 3a of the pure cotton, surface modified cotton employing dopamine and CALF-20 @ Cotton with dopamine show a similar resemblance with respect to structures. The spectra from 3300 and 3400 cm^{-1}could be because oftwo reasons: 1.) −OH group stretching and 2.) −NH from dopamine [20]. The manifestation of the quinone group in polydopamine is provedthrough the spectrum at 1720 cm^{-1} that could be seen in both cases 1.) cotton modified with dopamine and 2.) Surface modified cotton with dopamine along with crystals of CALF-20 [21]. Additionally, the FTIR spectral lines ofsurface modified cotton with cotton exhibited a broadened and weakened signal about 1600 cm^{-1}, and specifically it shows a existence of −C=C stretching vibrations. Theprevailingrobustspikesat 1390 and 1600 cm^{-1}in the composite material is mainly because of the action of organic linker, (i.e.) terephthalic acid [11].

The X-ray diffractograms illustrated in Figure 3b for the materials demonstrates the crystallinity nature of materials.When we observe the pattern of cotton there exist wide XRD spectra usually referring to Cellulose I (JCDPS. No. 03-0226). The existence of peak at 2θ = 14.5° in (101) plane tend to reduce in surface modified cotton with dopamine and there is no evidence of that peak when looking into the XRD curves of composite. This may be attributed to the intense thermal treatment on the substrate cotton [22]. The peak at 2θ = 22.36°at (002) plane gets improved in crystallinity, and it changes from amorphous to crystalline and may be because of the high alkali concentration acting on cotton [15]. The XRD spectra at 2θ = 33.6°in (004) plane of cotton and cotton with dopamine may be a weak amorphous peak due to loosely bound substances found in traces but in composite there is existence of sharp crystalline peak,and this is due to the reaction of hydroxyl molecules on cellulose structures [23]. The presence of other minor peaks may correspond to the chemical treatment of cotton, and it confirms the additive nature of employed experimental technique.

3.3. Thermal stability and porosity measurements

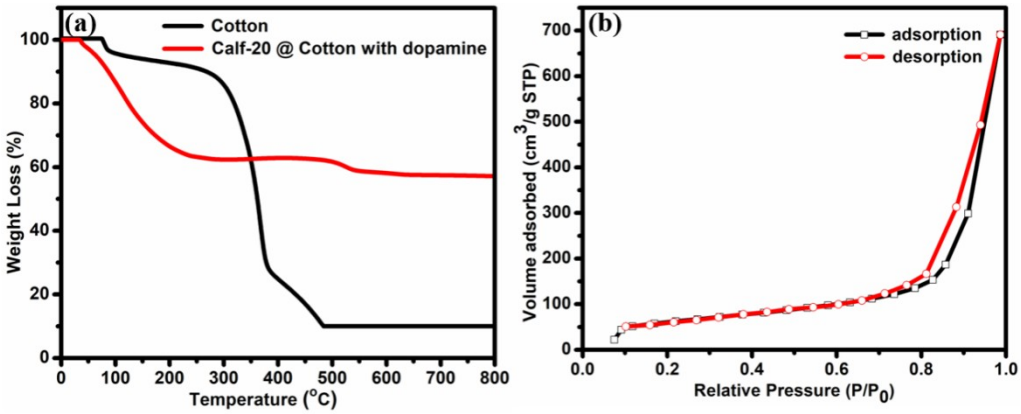

Figure 4. TGA curves of (a) Cotton and CALF-20 @ Cotton with dopamine (b) Nitrogen adsorption-desorption isotherm of CALF-20 @ Cotton with dopamine.

The thermal stability of cotton and the composite are analyzed by the application of Thermogravimetric analyser, and they are illustrated in Figure 4a. The initial weight loss, fewer than 10% prior to 100℃ is attributed to the vaporization of water content in cotton and volatile compounds in the case of composite. A major weight loss of around 65 to 70% could be observed from 100 to 380℃ in TGA curve of cotton, and this is because of the conversion of cellulose to levoglucosan residue upon pyrolysis and the last part of weight loss (nearly 20%) between 380 to 485℃ represents the formation of char due to oxidative degradation reaction in the presence of air [24]. The TGA curve of composite shows a significant weight loss of around 90% between 50 to 540℃. The main reason for the weight loss in composite is attributed to the breakage of alkyl bonds and it subsequently results in the removal of hydroxyl radicals [24].

The resulted isotherm for our composite is illustrated in Figure 4b. It is found that our composite exhibits dual characteristicsof bothType IV and Type I isothermal curves. This significantly emphasizes there is an existence of weak Vanderwall'sinteractions between CALF-20 and substrate and the estimated BET surface area yielded a value of 23.98 m^2/g. The point of inflection lies below the P/P_0 value of 0.1 and it demonstrates the existence of mesoporous nature and it corresponds to the fact that cellulose based materials have high mesoporous nature [15]. The microporous nature was also observed in combination and which is attributed the characteristic nature of Zn-based MOF [11].

3.4. Electrochemical analysis

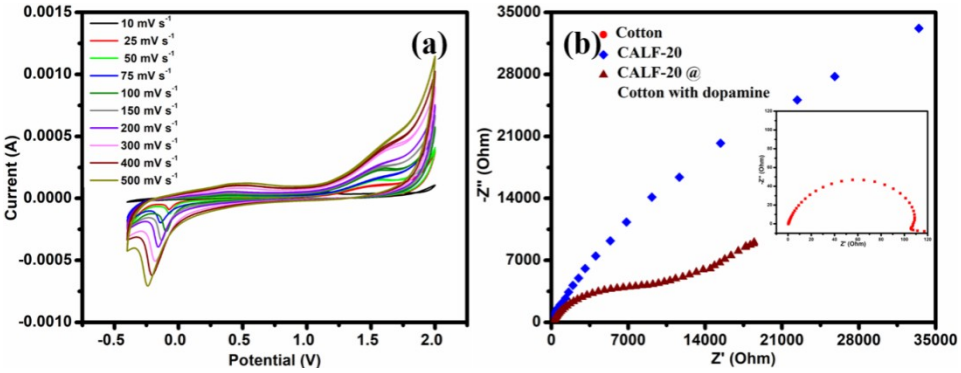

Figure 5. (a) CV curves with varying scanning rates for CALF-20 @ Cotton with dopamine (b) EIS curves of Cotton, CALF-20, and CALF-20 @ Cotton with dopamine.

The occurrence of both cathodic and anodic peaks in the Cyclic Voltammetric (CV) curves of CALF-20 @ Cotton (Figure 5a) in the potential region of −0.5 and 1.7 V with asymmetric electrodes of Zinc and stainless steel separated by our material explicitly proves our material is suitable for battery applications. The well-defined redox peaks indicates that the electrochemical processes are diffusion controlled. In addition to it, the shift of peak potentials to the sides of positivity and negativity due to various scanning rates evidences the material is quasi-reversible. The lower ionic diffusivity or the lower ohmic resistance properties of the redox-type active materials supports the quasi-reversibility phenomena and when considering charge storage processes, large charge storage performance could be obtained in diffusion-controlled process [25].

The EIS spectra for Cotton, CALF-20, and CALF-20 @ Cotton with dopamine is shown in Figure 5b. The EIS curve of cotton is in the shape of a semicircle indicating the material is found to possess high resistivity. On considering CALF-20,the gradient measured in the low-frequency region is expected to result in the larger value, indicating the furious movement of ions during the process of charging and discharging, thus resulting in fast ion diffusion rate. Another observation which we shall infer from nearly a straightened line at the section of low frequency confronts, our material can outperform as it holds the superior capacitance in the absence of limitations happening during the diffusion of ions [26]. The rapid motion of electrons will favorably enhance the mechanism of conduction between electrodes and separator/electrolyte interface which results in the minimum charge transfer resistance (R_{ct}).All these extensive findings mentioned above gives a clear picture that zinc based framework composite materials as separator in electrolytic medium has sufficient rate capability [27]. The high frequency section with no half circle represents a negligible faradic charge transfer resistance and the vertically erected stroke at low range region of frequency confirmations the characteristics of ideal capacitor.The vertically erect stroke at low ranged region of frequency observed in CALF-20 seem to approach towards the unreal scale of axis, thus representing the characteristic virtue of pseudo capacitor [28]. When looking into the composite, CALF-20 @ Cotton with dopamine the curve is found to have a half semicircle with a spike which indicates that the ionic conductivity increases along with the increased electron transfer, and so the large specific capacitance and huge energy density for the energy storage device [29].

4. Conclusion

A zinc-based porous co-ordination polymeric crystal was synthesized employing aqueous solvent caustic potash and the developed crystals were grown successfully on the surface of waste cotton. The adherence of zinc-based porous co-ordination polymericcrystals onto the surface of cotton was established. The compatibility between synthesizedporous co-ordination polymeric crystals and cotton was improved greatly with the help of surface modifier dopamine and it showed an excellent structural appearance which is evident through the obtained SEM images.Our composites possessed a combinational characteristic of both microporous and mesoporous assemblies with the estimated BET surface area value of 23.98 m^2 per gram and this demonstrates that our material is sufficiently porous with uniformity in pore distribution. The charge transfer resistance (R_{ct}) was analyzed to be

decreased with high ionic conductivity. Upon analyzing these results,we suggest you that the prepared Zn based composite could be used as separator in batteries and it is being primarily supported by the results of EIS and CV curves. Further work on this prepared composite is being carried out by our team in order to examine its full potential in energy storage applications.

Availability of Data and Materials: The datasets used and analyzed during the current study are available from the corresponding author on reasonable request.

Author Contributions: All authors contributed to the study conception and design, material preparation, data collection and analysis, and manuscript writing. All authors have read and approved the final manuscript. All authors contributed to editorial changes in the manuscript. All authors have participated sufficiently in the work and agreed to be accountable for all aspects of the work.

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Conflict of Interest: On behalf of all authors, the corresponding author states that there is no conflict of interest.

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