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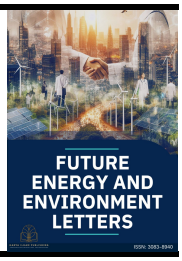

Molarity-Controlled KOH Activation of Sugarcane Bagasse-Derived Carbon for Energy Storage Applications

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ABSTRACT

As world shift to a world with green and high technology, it is a challenge to find alternative carbon source for the energy storage electrode. Activated carbon are known to have high surface area and porosity which significant to various applications including energy storage electrode materials. It was developed from a sustainable non-toxic and low-cost precursor source. In this work, activated carbon was prepared from sugarcane bagasse using single-step chemical activation using various molarities of potassium hydroxide (KOH) as an activating agent followed by pyrolysis at 650 °C. This study aims to investigate the influence of different KOH molarities on structural and electrochemical properties of activated carbon. This parameter will also affect the properties of the activated carbon produced. The characterization of sugarcane bagasse activated carbon using specific techniques such as scanning electron microscopy (SEM), thermo-gravimetric analysis (TGA), X-ray diffraction (XRD), Linear Sweep Voltammetry (LSV) and Fourier transform infrared (FT-IR) spectroscopy revealed that increasing in KOH molarities will also enhance the properties of activated carbon such as pore development and structural disorder, resulting in predominantly amorphous carbon with enlarged interlayer spacing, reaching ~0.3 nm for the 1.2 M sample. Linear sweep voltammetry analysis conducted within a voltage window of −1.5 V to 2.3 V demonstrated that the activated carbon produced using 1.2 M KOH indicates the pseudocapacitor trend compared to lower molarity samples. These findings emphasize the role of chemical activation parameters in enhancing the microstructure and electrochemical performance of biomass-derived activated carbon and highlight sugarcane bagasse as a promising, low-cost, and sustainable electrode material for energy storage applications.

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1. Introduction

The rising need for efficient and sustainable energy storage systems has driven extensive research into alternative electrode materials that are both low-cost and environmentally friendly. Conventional electrode materials often rely on non-renewable resources and involve complex or energy-intensive fabrication processes, which raise concerns related to cost, sustainability, and long-term availability [1,2]. Due to their abundance, tunable structure, and favourable electrochemical properties of electrode materials derived from agricultural waste, it becomes a favourable candidates to encounter the problems of conventional electrode materials in energy storage applications [3].

Owing to its high surface area, porous structure, and chemical stability, activated carbon becomes one of the most widely used carbon materials in energy storage devices, particularly in supercapacitors [4]. The microstructure of activated carbon, including pore size distribution, surface functional groups, and degree of structural disorder are strongly influencing its electrochemical performance [3]. Through careful selection of precursor materials and optimization of processing parameters during activation and carbonization, the characteristics and performance of activated carbon can be modified and improved. Among various biomass precursors, sugarcane bagasse has attracted considerable attention because it is an abundant agricultural by-product with high carbon content and is readily available in many sugar-producing regions.

A commonly employed method to produce activated carbon with well-developed porosity and high surface area is chemical activation using acid or base solution. Known as an effective microporous and mesoporous carbon structures generator, potassium hydroxide (KOH) is frequently used as an activating agent at relatively lower activation temperatures compared to physical activation methods [5]. During KOH activation, complex reactions between the carbon precursor and the activating agent promote pore formation and structural rearrangement, which can significantly affect the resulting electrochemical behavior of the activated carbon [6]. However, the extent of activation and the final carbon structure are highly dependent on parameters such as activating agent concentration, activation temperature, and impregnation conditions [7].

Although numerous studies have reported the use of KOH-activated biomass-derived carbon for energy storage applications, systematic investigations focusing on the effect of KOH molarity at a fixed pyrolysis temperature remain limited. Understanding how variations in activating agent concentration influence the structural and electrochemical properties of activated carbon is essential for optimizing material performance. Therefore, this study aims to investigate the effect of KOH molarity on the microstructure and electrochemical behavior of activated carbon derived from sugarcane bagasse, prepared via chemical activation followed by pyrolysis at 650 °C. The findings of this work are expected to contribute to the development of sustainable and cost-effective carbon-based electrode materials for energy storage applications.

Despite the growing interest in biomass-derived activated carbon for energy storage applications, the influence of chemical activation intensity, particularly potassium hydroxide molarity, has not been sufficiently examined under controlled carbonization conditions. In many reported studies, multiple activation parameters are varied simultaneously, which limits clear understanding of how activating agent concentration alone affects pore development, structural disorder, and electrochemical behaviour [8,9]. Clarifying this relationship is important for improving control over the microstructure of activated carbon and, consequently, its charge storage performance. Therefore, this study aims to investigate the effect of potassium hydroxide activation molarity on the structural and electrochemical properties of activated carbon derived from sugarcane bagasse, prepared at a fixed pyrolysis temperature of 650 °C.

2. Methodology

2.1 Materials Preparation

The sugarcane bagasse was collected directly from local juice stall. The bagasse was then air-dried and crushed to obtain smaller-sized biomass. It was then thoroughly washed with tap water to remove the impurities and residual. The cleaned bagasse was then air-dried again for 72 hours. Then it was oven-dried for another 24 hours at 100 °C to ensure no excess moisture was trapped in the bagasse.

2.1 Chemical Activation and Pyrolysis

To synthesis sugarcane bagasse activated carbon, single-route chemical activation using potassium hydroxide (KOH) at different molarities (0.4M, 0.8M and 1.2M) were carried out. The dried sugarcane bagasse is impregnated with a chemical activating agent (KOH) and then subjected to simultaneous carbonization and activation at elevated temperatures in a single operation. Firstly, sugarcane bagasse was immersed in four different molarities of chemical activating agent solution for 24 hours at room temperature, separately. After completion, the sample is filtered and oven dried at 100 °C for 24 hours to remove excess moisture prior to pyrolysis.

Activated carbon was then thermally decomposed and activated at a fixed temperature of 650 °C in an inert atmosphere to prevent oxidation. After cooling to room temperature, the samples were washed repeatedly with hydrochloric acid (HCl) and distilled water to remove residual impurities and unreacted KOH until a neutral pH was achieved. Then, the activated carbon was dried in the oven at 100 °C for 48 hours and stored in a closed container before further characterizations.

2.3 Structural and Morphological Characterization

The surface morphology and pore structure of the raw sugarcane bagasse and the activated carbon samples were examined using scanning electron microscopy (SEM). Fourier transform infrared spectroscopy (FTIR) was employed to identify surface functional groups and chemical bonding characteristics present before and after activation. The crystalline structure and degree of structural order of the activated carbon were analyzed using X-ray diffraction (XRD), with particular attention given to peak broadening associated with amorphous carbon structures. Interlayer spacing, d values were estimated from the XRD patterns using Bragg's law as Eq.1.

$$d = \frac{\mathrm{n}\lambda}{2\sin\theta} \quad (1)$$

where n is the order of reflection at constant 1, λ is the wavelength at constant 0.15418 nm, and θ

2.4 Electrochemical Characterization

The electrochemical performance of the activated carbon samples was evaluated using linear sweep voltammetry (LSV). Electrodes were prepared by mixing sugarcane bagasse activated carbon with polyvinylidene fluoride (PVDF) and carbon black as conductive additive at ratio of 8:1:1. The sugarcane bagasse activated carbon and carbon black were dry-mixed and later being added to the mixture of PVDF and N-Methyl-2-Pyrrolidone (NMP) solvent and stirred until homogeneous. The mixtures were then mixed in sonicator to avoid agglomeration of the carbon particles. the doctor blade was used to spread the mixture onto copper sheet as current collector. After drying in oven at 100 °C for 24 hours, the copper sheet coated with sugarcane bagasse activated carbon was punch in

a circle shape. Fibre glass sheet was used as separator and being assembled in a CR3202 coin cell with lithium metal acts as counter and reference electrodes. Electrochemical measurements were conducted within a voltage window of −1.5 V to 2.3 V at a fixed scan rate 0.01 V/s using a standard electrochemical analyzer.

3. Result

3.1 Structural and Morphological Characterization of Sugarcane Bagasse

Thermogravimetric analysis (TGA) was first carried out on raw sugarcane bagasse to determine its thermal decomposition behaviour and to support the selection of an appropriate pyrolysis temperature. The thermal analysis of SCB was performed on a dry basis revealed that there are three distinct mass loss profiles (figure 1a). The first profile shows demoisturization of SCB at temperature less than 200°C. As the raw SCB particles were exposed to 230°C, pyrolytic devolatization starts where decomposition of hemicellulose took place. The subsequent reaction which occurred between 340°C - 375°C was decomposition of cellulose. At higher temperature range, 380°C - 450°C, a small weight loss was observed and it represented delignification. Lignin decomposes more slowly and over a broader temperature range (200–900 °C), with significant decomposition continuing up to 500 °C and beyond [8,9]. Thus, the best temperature range in order to produce activated carbon shall fall between 500 to 700°C. Beyond 700°C, the gasification takes place which will produce gases as its major product. There is no significant mass loss in sugarcane bagasse, suggesting that the main thermal decomposition reactions are complete by this point [9].

SEM images of the raw bagasse show a fibrous structure with relatively smooth surfaces and limited visible porosity (figure 1b), which shows the fibrous strands and pith. The fibre surfaces are characterized by parallel stripes, while the pith exhibits a more porous structure with micropores [10].

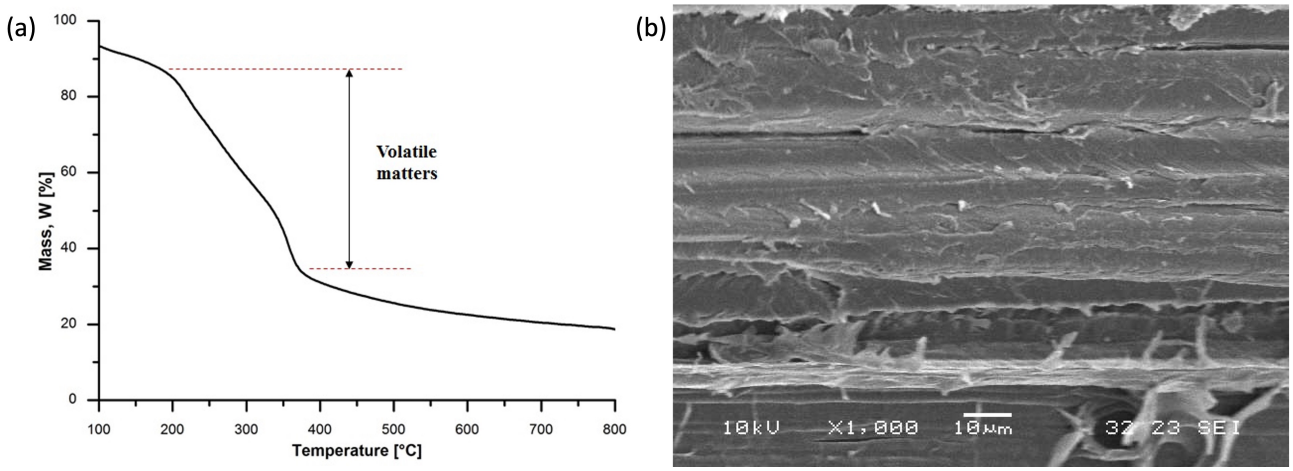

Fig. 1. (a) TGA Analysis of Sugarcane Bagasse, (b) Morphological View of Sugarcane Bagasse

3.2 Effect of KOH Molarity on Activated Carbon Morphology

The surface morphology of activated carbon samples prepared using different KOH molarities was examined using SEM (figure 2). The activated carbon produced with 0.4 M KOH exhibits limited pore development, with relatively compact surface features and fewer visible pores. An increase in pore formation is observed as the KOH molarity increases, indicates that the chemical etching occurred during activation and made the surface rougher and looser [11,12]. The sample activated

with 1.2 M KOH displays the most developed porous structure, with a higher density of interconnected pores and thinner pore walls.

This progressive increase in porosity with increasing KOH molarity can be attributed to the more aggressive chemical interaction between KOH and the carbon matrix at higher concentrations, which promotes pore formation which facilitate the development of micro- and mesopores in the carbon matrix [13]. However, excessive KOH molarity may also lead to partial pore collapse or structural weakening, indicating the importance of optimizing activation conditions rather than simply increasing activating agent concentration [14,15].

The porosity have different in size and increase in amount of porous due to continue heating for activation. The result of this deformation lead to completely fallen of wall and thus closed the pores. Based on the result, it shows that activated carbon with 1.2M produced more porous structures than 0.8M and 0.4M and this is due to the surface area that increases in size during the pyrolysis.

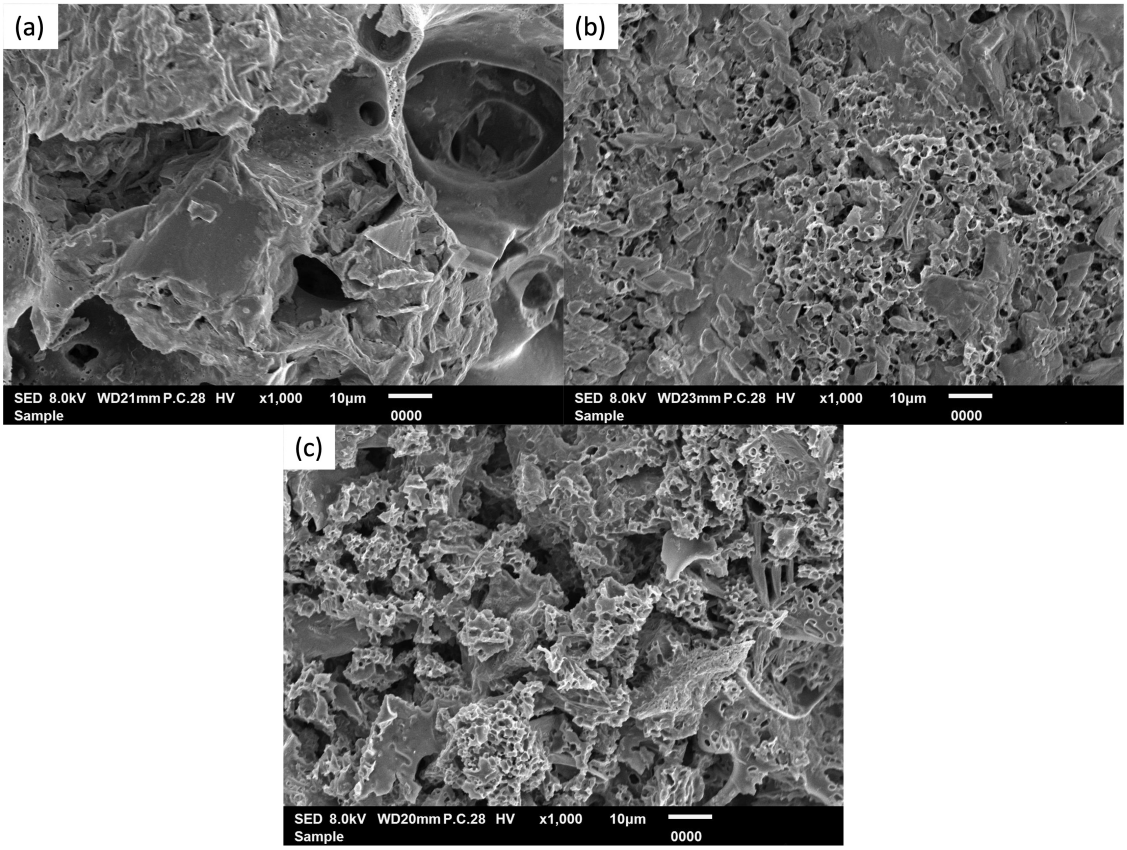

Fig. 2. Morphology of sugarcane bagasse activated carbon with (a) 0.4M KOH, (b) 0.8M KOH, and (c) 1.2M KOH

3.3 Chemical Structure and Crystallinity

FTIR results was obtained from produced activated carbon upon activation with various molarity of KOH reveals significant information about functional groups present in the samples. Several peaks were observed in the FTIR spectre indicating the presence of specific functional groups resulting from the activation process with different molarity of activating agent (KOH) [16]. The asymmetric absorption band were observed in all three samples at 3130 to 3200 cm^{-1} which indicates the presence of O–H stretching of hydroxyl groups [17]. As the molarity of KOH was increased, the peak was observed to be shifted to the right due to stronger hydrogen bonding occurs [18]. The surface

functionalization was also expected to increase as the molarity of KOH is increased which will be beneficial for ion accessibility [19].

There were weak peaks also observed at region 2000 to 2160 cm^{-1}, which indicate the presence of triple bond carbon [20]. The presence of those peaks indicate the progressive carbonization and extended π-electron systems within the carbon framework due to chemical activation which can be significant for charge transport [21,22]. Strong absorption peaks were observed between 1655 to 1660 cm^{-1} which indicates the C=O / C=C stretching. These functional groups are expected to contribute to pseudocapacitive behaviour and enhanced the redox activity [23]. The spectra also shows the peak around 1450 cm^{-1} which shows a methyl group which is C-H symmetric bending that suggest partial retention of disordered carbon structures [22,24]. The peaks around 840 cm^{-1} indicates the aromatic C–H bending and its presence is typically significant for aromatic C–H out-of-plane bending and possibly ester groups in turbostratic or disordered carbon and commonly related to indicates the retention of aromatic structures and oxygen-containing functionalities in the interlayer carbon frameworks [25,26].

Overall, FTIR analysis confirms that increasing molarity of KOH during activation process enhance the formation of oxygen-containing functional groups on the surface of sugarcane bagasse activated carbon, which improves its electrochemical properties.

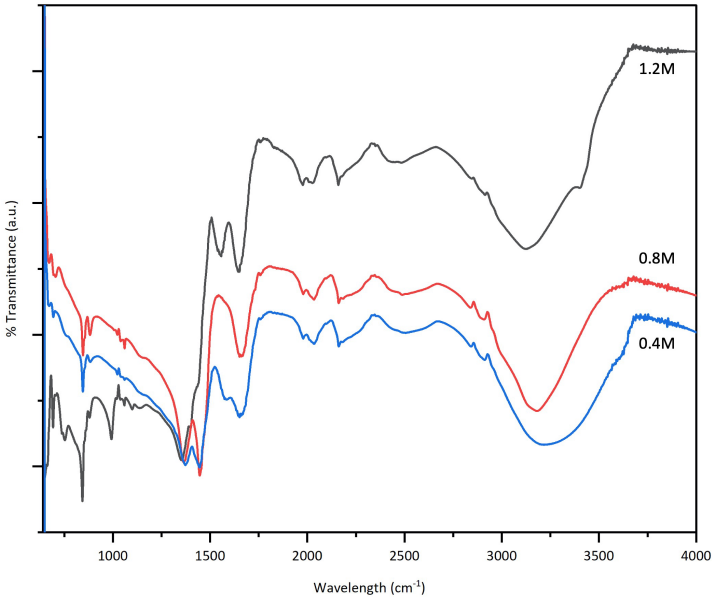

Fig. 3. FTIR spectra for the prepared sugarcane bagasse activated carbon with different molarity of KOH

XRD patterns for sugarcane bagasse activated carbon prepared with different KOH molarities in figure 4 exhibit a broad diffraction peak in the range of 30–35° corresponding to the (002) planes which indicates a predominant amorphous or turbostratic carbon [27,28]. Another weak broad peaks were also observed around 40–43° which commonly associated with the (100) plane of disordered graphitic carbon [26]. As the molarity of KOH increases, the peak intensity also increased. The reduction of peak intensity indicating that there is a reduction in graphitic order and increase in disorder within the carbon structure which caused by the disruption of graphitic domains in the sugarcane bagasse activated carbon [29,30].

Interlayer spacing values calculated using Bragg's law show a gradual increase with increasing KOH molarity, reaching approximately 0.3 nm for the 1.2 M sample (table 1). Potassium ions can penetrate the carbon structure and enlarge the interlayer spacing of graphite-like domains, favouring

the reversible intercalation of ions such as Li^+ and K^+, and maintaining structural stability during charge–discharge cycles. The enhancement of interlayer spacing allows for better electrolyte infiltration and faster ion diffusion [31].

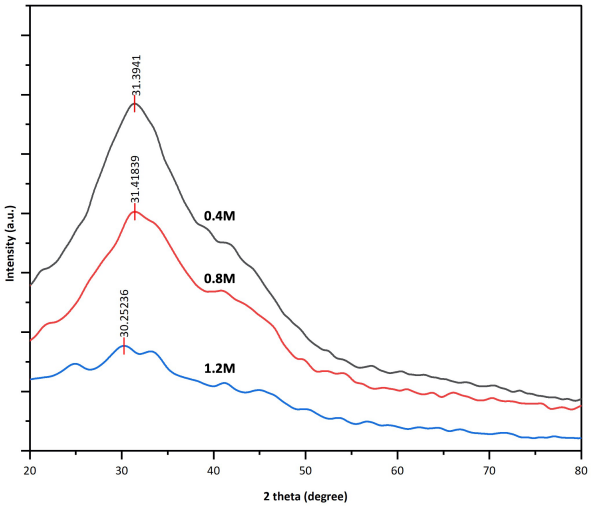

Fig. 4. XRD of sugarcane bagasse activated carbon at different molarity of KOH

Table 1
Interplanar spacing calculated interplanar spacing of sugarcane bagasse activated carbon at different molarity of KOH (0.4M, 0.8M, And 1.2M)

Molarity of KOH	Peak position, 2θ (°)	d (Å)	d (nm)
0.4M	31.3941	2.8540	0.2854
0.8M	31.4184	2.8530	0.2853
1.2M	30.2524	2.9600	0.2960

3.4 Electrochemical Performance

The electrochemical behaviour of the activated carbon samples was evaluated using linear sweep voltammetry (LSV) within a voltage window of −1.5 V to 2.2 V (figure 5). The plot revealed that the increase of KOH molarity lead to the increase in current. Activated carbon with 1.2M KOH shows the highest absolute current which indicates the higher ionic conductivity presents that give better charge transport at higher molarity of KOH. This enhanced performance is consistent with the structural observations, where increased porosity and enlarged interlayer spacing facilitate improved electrolyte access and ion transport within the carbon structure [13].

At negative potential region, activated carbon with 1.2M exhibits strong cathodic current with sharp current rise. The sharp rise suggested most likely due to the electrolyte decomposition. For activated carbon with 0.4M and 0.8M, smaller current response were observed which indicates more resistive behaviour. At near-zero potential region, all samples were observed to cross near the origin and no clear redox peak pair were observed. This situation occurs due to weak reversible redox behaviour in the cell and it indicates that the response is dominated by capacitive or resistive components, in line with the pattern in positive potential region that shows the gradual increase in current with potential and no sharp anodic peak was observed.

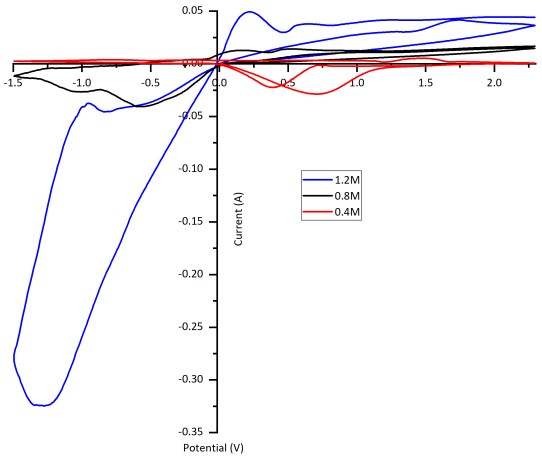

Fig. 5. Linear Sweep Voltammetry (LSV) of sugarcane bagasse activated carbon

4. Conclusion

In conclusion, activated carbon has been successfully prepared from sugarcane bagasse. The produced sugarcane bagasse activated carbon imposed a potential properties to be used as electrode materials as an alternative to conventional electrode materials. The chemical activation has been done using potassium hydroxide (KOH) at different molarities (0.4M, 0.8M, and 1.2M). The structural characterization of the produced activated carbon revealed that the properties has been enhanced. From the SEM analysis, the pore development has been observed upon increasing the KOH molarity. The XRD analysis has proven that the activated carbon has an amorphous structure and the increase in KOH molarity also promoted structural disorder, and increased interlayer spacing, which resulting in predominantly amorphous carbon structures. Through the electrochemical analysis study, the activated carbon produced with 1.2M KOH has shown a pseudo-capacitive response. However, during the cyclic voltammetry, 0.4M KOH has the highest capacitance. Among the studied samples, the activated carbon prepared using 1.2 M KOH exhibited the most developed porous morphology and the largest interlayer spacing, indicating improved accessibility for electrolyte ions.

Linear sweep voltammetry demonstrated that the 1.2 M KOH-activated carbon showed a pseudo-capacitive behaviour. The observed electrochemical performance is closely related to the enhanced microstructural features induced by higher activation intensity. Overall, the findings highlight the importance of controlling chemical activation parameters in tailoring the properties of biomass-derived activated carbon. This work confirms the potential of sugarcane bagasse as a low-cost and sustainable precursor for producing activated carbon electrodes in future energy storage material development.

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