TITLE: Electrochemistry testing on biochar from Juncus rushes Reference:

SCOPE OF THIS REPORT

BIOCHAR from Juncus rushes was analysed for supercapacitance properties

Surface Analysis Report:

Report Date: 26-02-2023
Authors Name: Dr Anne Beaucamp Mc Loughlin Professor Maurice Collins

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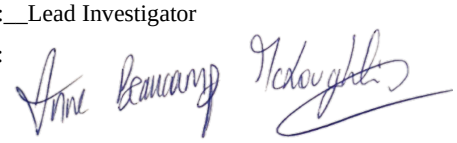
SIGNED

By:

Name: Dr Anne Beaucamp

Title:__Lead Investigator

Date: 

27-02-2023

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SAMPLE DESCRIPTION

A bag on biochar (black, fine particles) was received.

ANALYSIS TYPE

The objective of this study was to determine the electrochemical behaviour of the constituent materials. Accordingly, the samples were prepared in a slurry and mounted for 2 and 3 electrodes testing.

METHODOLOGY

SEM (Hitaichi SU70) was performed at 5 and 10KeV.

A slurry of 80% Biochar was formed in N-methyl-2-pyrrolidone using 10% PVDF as a binder and 10% Carbon black (CB45) as the conductive material. The three powders were fully mixed in an agate mortar for 30 min and then mixed with NMP solvent until it became viscous. The resulting slurry was coated on the nickel foam current collectors with a square area of 1 cm2 (1 × 1 cm) and dried in a vacuum drier at 100 °C overnight.
The electrodes were mounted in a 2 and 3 electrodes set-up, with a platinum counter electrode and a saturated calomel electrode as reference and tested using an IVIUM n-stat potentiostat (Eindhoven, Netherlands) in a solution of KOHat 6 mol.L^{-1}.
The values of the specific capacitance C_p were calculated using the following equation:

$$C_p = \frac{I.\Delta t}{\Delta V.m'} \quad (1)$$

Where *I* is the constant charge current, Δ t / Δ V is the inverse of the slope of the galvanostatic curves and *m* is the total mass of the two hydrogels.

RESULTS

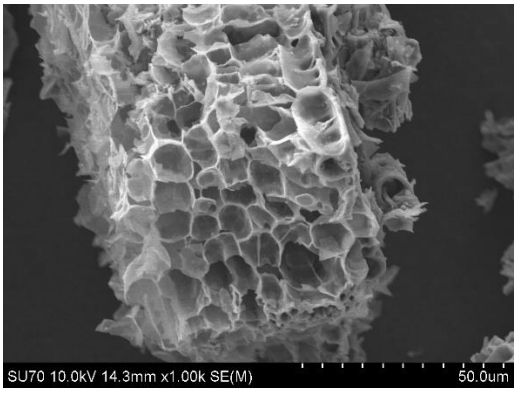

Figure 1 : Micrograph of the as received biochar

Table 1 : EDX results for the as-received biochar

Spectrum	In stats.	C	O	Na	Mg	P	Cl	K	Ca	Mn	Total
Spectrum 1	Yes	68.92	21.65	1.43	0.78	0.52	1.39	3.31	1.49	0.52	100.00
Spectrum 2	Yes	70.34	18.65	1.17	0.77	0.47	2.52	4.57	1.07	0.44	100.00
Mean		69.63	20.15	1.30	0.78	0.50	1.95	3.94	1.28	0.48	100.00
Std. deviation		1.00	2.12	0.18	0.01	0.03	0.80	0.89	0.30	0.06	
Max.		70.34	21.65	1.43	0.78	0.52	2.52	4.57	1.49	0.52	
Min.		68.92	18.65	1.17	0.77	0.47	1.39	3.31	1.07	0.44	

Figure 1 shows the micrograph of the as-received sample. The microstructure of the biochar shows a honey-comb, alveolar structure that is typical of juncus rushes plant stems. The charring process fully preserved the structure. The material is composed of 68% carbon, 21% oxygen and traces of minerals (table 1). Consequently, the as-received biochar couldn't be tested for supercapacitance.

Subsequently, the material was subjected to a carbonisation cycle to 900°C in inert atmosphere.

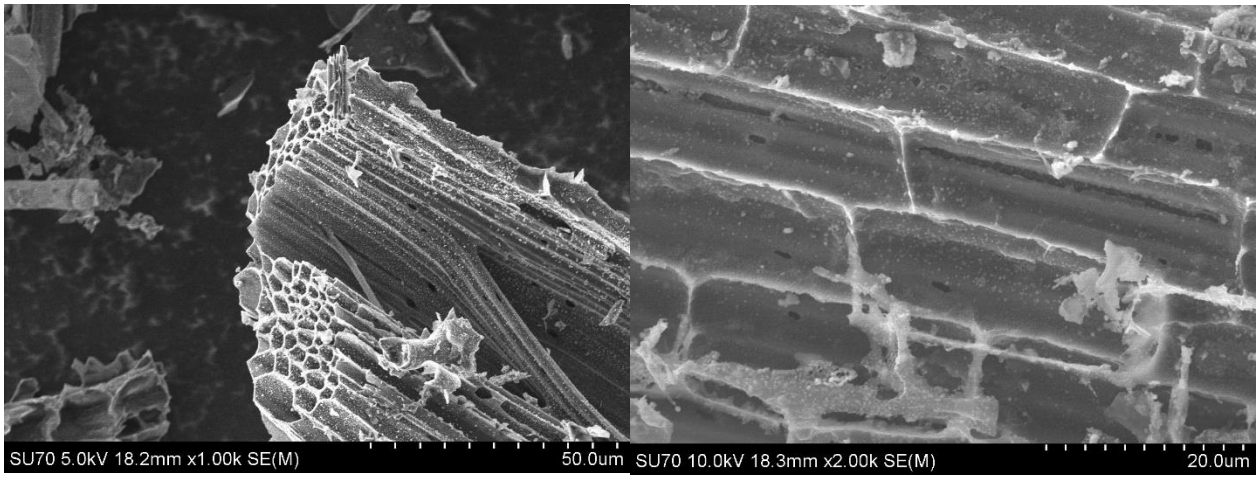

Figure 2: SEM micrograph of the carbonised biochar

After carbonisation, the material presents full shape retention, with long tubular channel structure. Electrochemistry testing in the 3-electrodes configuration presented a distorted boat shape cyclovoltammetry plot, indicating possible capacitance effect. Further testing in the 2-electrode, charge discharge configuration showed a non-symetrical triangular shape, typical of a pseudocapacitor behaviour.

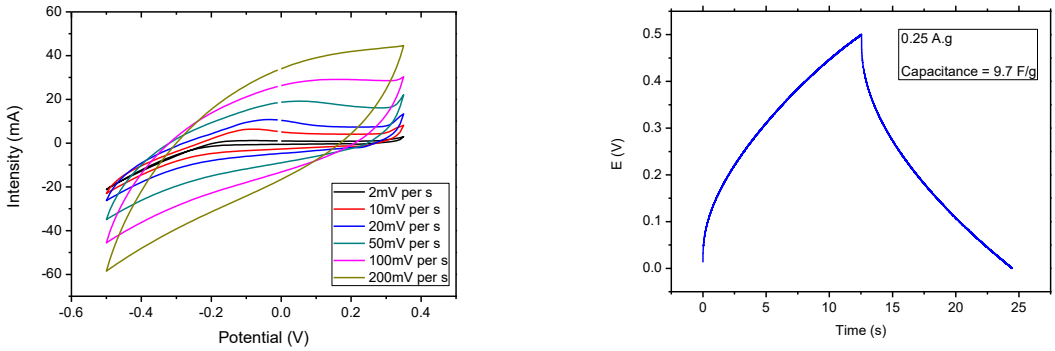

Figure 3 : Electrochemistry results, in 3-electrode configuration (left) and in charge-discharge configuration (right)

The capacitance of the material was measured at Cp =9.7 F/g, which is close to state-of-the art for non activated carbon [(1)].

Conclusion

Based on the analysis carried out on the biochar received, the following can be concluded:

- The as-received biochar presents a honeycombed structure with no capacitance properties ;
- The carbonisation allows for retention of the structure and capacitance effect with a Cp close to 10F/g ;

This material presents strong potential for energy, supercapacitor application.

Recommendation:

Further testing after activation of the biochar to open mesopores and micropores would increase Cp.

REFERENCES

(1) Beaucamp, A., Muddasar, M., Crawford, T., Collins, M. N., & Culebras, M. (2022). Sustainable lignin precursors for tailored porous carbon-based supercapacitor electrodes. *International Journal of Biological Macromolecules*, *221*, 1142-1149.