

EFFECT OF TORREFACTION AND PYROLYSIS ON THE ELECTROCHEMICAL REACTION IN DIRECT CARBON FUEL CELLS

THONG, W. H.¹, VEENA, D.^{1,*}, WONG, W.Y.²

¹School of Engineering, Taylor's University, Taylor's Lakeside Campus,
No. 1 Jalan Taylor's, 47500, Subang Jaya, Selangor DE, Malaysia

²Fuel Cell Institute, Universiti Kebangsaan Malaysia,
43600 UKM Bangi, Selangor DE, Malaysia

*Corresponding Author: veena_doshi@yahoo.com

Abstract

Direct carbon fuel cells (DCFC) is a device capable of producing high electrical efficiency by converting chemical energy stored in biomass at high temperatures. This research will attempt the thermal treatments of biomass to convert it into the carbon fuel source of DCFC. Thermal treatment methods applied such as pyrolysis and torrefaction can enhance carbon compositions and energy densities of biomass by conversion into biochar. This study will investigate the performance of DCFC fuelled by torrefied oil palm mesocarp fibre at 230, 260 and 290°C and samples undergoing pyrolysis at 650 °C. Treated biochar will be analysed on the physicochemical characteristic by thermogravimetric analysis, fourier-transform (FTIR) analysis and x-ray diffraction (XRD) analysis. The performance of the operational fuel cell will be evaluated via peak power density and impedance demonstrations using electrochemical impedance spectroscopy and open circuit testing. DCFC fuelled by biochar obtained from direct pyrolysis exhibited higher peak power density compared to that obtained via torrefaction; while biochar derived from torrefied biomass at 260 °C can highly be suggested for storage purposes while preserving fuel properties in a DCFC.

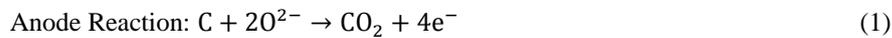
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1. Introduction

Consumption of electrical energy from recent history has shown an increase in demand for electrical energy with the incremental human population. However, energy generation has begun to see gradual depletion of non-renewable fuels with developing countries such as Malaysia, which draw 93% of its energy consumption from non-renewable methods. These methods are non-sustainable with the depletion of the natural gas reserves in addition to emission of greenhouse gases from the power

generation [1]. Leveraging the energy requirement by new technologies and innovations with a sustainable mind-set will in turn provide a stable economic, socialistic and environmental platform in the future. Fuel cell technology is among the more beneficiary in terms of carbon emission when commissioning modern sustainable energy generation. The technology itself has pioneered energy generation with the credibility to have electrical efficiencies of above 80% in addition to be powered by waste compounds [2]. Key business drivers are keen on the chemical reactivity potential of fuel cells to sustain low emission methods of generating energy with high fuel efficiency. Inadvertently this seeks potential of using a form of renewable waste source as fuel to power the fuel cells.

Direct carbon fuel cells (DCFC) are a form of newly improvised fuel cells that is fuelled by carbon compounds. This shows high potential in applying waste forms of organic material from industrial processing and commercial bio-degradable waste [3]. The high fuel efficiency of the technology is sometimes applied with non-renewable carbon compounds such as coal which is often set as a benchmark to be followed by renewable bio-energy fuels such as biomass. DCFC do come with a compromise of operating at temperatures between 600 °C to 900 °C in order to successfully perform the carbon oxidation and reduction respectively shown in Eqs. (1) and (2) [4]. However, commercial applications of fuel cells are reliant on none renewable sources such as coal.



Henceforth, one of the more abundantly generated forms of biomass in the South East Asian region is palm oil. Malaysia which leads the production and exportation of palm oil has conducted several sustainable implementations for the pro-longed supply of palm oil production. This includes the formation of the Roundtable of Sustainable Palm Oil assigned in monitoring the developing 4.3 million hectares committed to palm oil in Malaysia [5]. Thus, palm oil waste derived from commercial processing can then be credible for use as fuels for DCFC. Palm oil waste are mainly constituent of lignocellulosic biomass that shows high energy densities required for fuel cell operations. Palm oil mesocarp fibre (PMF) is the likeliest candidate for fuelling DCFC chemical operations as it shows high calorific values and is the least utilised palm oil derived waste due to high moisture issues [6].

Prior to being utilised as the fuel source for DCFC, the lignocellulosic biomass would have to be subjected to thermal treatment via torrefaction and later converted into biochar with pyrolysis. Torrefaction was introduced as a thermal treatment originally to reduce moisture for logistic and pelletisation purposes [7]. However, the lower held oxygen to carbon ratios from torrefaction can improve gasification to potentially increase the performance in DCFC [8]. Pyrolysis is proven to remove volatile matter composition and ash metal content of the biomass while improving surface areas of biomass [9]. No previous publications attempting the use of thermally treated palm oil mesocarp fibre biochar as fuel for DCFC. This was the investigated criteria of the research.

2. Methodology

This research composes the effect of torrefaction and pyrolysis on palm mesocarp fibre to be used as fuel for DCFC. The fuel is characterised by the torrefaction

temperatures pre-pyrolysis (#A), direct torrefaction of sample (#B) and direct pyrolysis of sample (#C).

2.1. Thermal treatment

The palm mesocarp fibre was collected from post production of palm oil mill processing from Seri Ulu Langat plant located in Dengkil. 2kg of the raw fibre was sundried for 6 hours to remove lingering odours. The biomass was then subjected to oven drying at 105 °C for 24 hours to evaporate unbound moisture. The biomass was then mechanically grinded and sieved to particle size below 5 mm.

The mesocarp fibre was then torrefied at 230, 260 and 290 °C for 1 hour under inert conditions whereby the fibre was heated in a chamber purged of nitrogen. The torrefaction was performed in a sealed cylindrical muffle furnace with 15 g of mesocarp fibre loaded. Nitrogen gas (99%) was transported into the furnace at 100 ml/min throughout the period of ramping and torrefaction. The furnace was set to ramp at 10 °C/min until desired temperatures were reached. Torrefied samples were then stored under standard room conditions of 25 °C with a relative humidity of 84% for a duration of 7 days.

Biochar preparation

After the storage duration, one sample of raw mesocarp fibre of 15 g and the remaining torrefied biomass of their respective temperatures will undergo pyrolysis at 650 °C with a ramping rate of 10 °C/min to produce the biochar. Similarly, pyrolysis was performed with inert conditions with high purity nitrogen gas (99%) channelled into the furnace volume at 100 ml/min.

2.2. Fuel characterisation and analysis

2.2.1. Thermogravimetric analysis

Thermogravimetric analysis was performed to analyse the mass loss of the respective samples as the temperature was increased. This analysis was carried out with the Perkin Elmer TGA 8000 model. The biomass samples of 10 mg were heated from 30 °C to 950 °C with a ramping rate of 10 °C/min. High purity nitrogen (99%) was channelled into the analysis chamber at 20 ml/min during this period. The sample was then allowed to dwell for 7 minutes at 950 °C with a 20 ml/min flowrate of oxygen.

2.2.2. Fourier-transform-infra-red

FTIR analysis was conducted using Perkin Elmer FTIR Spectrum 100 model. The samples were scanned over the wave number range of 400 cm^{-1} - 4000 cm^{-1} using KBr pellet method. A ratio of 4 to 1 was used for KBr to sample and relatively 20 mg of KBr was used.

2.2.3. X-ray diffraction

X-ray diffraction is performed to show amorphous structural differences for PMF after being subjected to torrefaction and pyrolysis. The analysis was done externally via D8 Discover (Bruker) diffractometer. Scans were collected for 2θ from 10° to 90° with a step size of 0.05 and 15 seconds per step.

2.3. Direct carbon fuel cell performance

Compressed button cells were used as the reaction medium for the DCFC operations with the high temperature conditions. The composition and configuration of the button cell was comprised of Nickel-yttria-stabilised zirconia (Ni-YSZ), lanthanum strontium manganite (LSM), and Yttria-stabilised zirconia (YSZ) for the anode, electrolyte and cathode respectively. The performance of the DCFC was analysed with a potentiostat (Gamry Interface 1000 model) with two electrode configurations.

The button cell was supported on a perforated ceramic plate sandwiched between two identical ceramic cylinders to house the cell in a sealed volume. This can be seen in the computer aided model of the set-up shown in Fig 1. PMF derived biochar of 0.1 mg was loaded onto the anode side of the button cell with exposed area of 1.743 cm² as the fuel. The furnace was then programmed to ramp at 10 °C/min until the desired temperature of 800 °C before remaining at that temperature for 2 hours. While the temperature is increasing, nitrogen was flowing into the anode and cathode ends of the DCFC at 200 and 600 ml/min respectively. The gas supply to the cathode end was switched with oxygenated air before 800 °C was reached with a flowrate of 600 ml/min. Peak power densities are determined by linear sweep voltammetry method. To analyse the anodic resistance of the DCFC, an electrochemical impedance spectroscopy would be used. This method of analysis enables one to obtain the voltage-current-power curves in addition to the anode polarisation resistance. The process will have the electrochemical impedance spectroscopy (EIS) obtained at the open circuit voltage with frequency range of 100 kHz to 0.1 Hz at current perturbation of 10 mA.

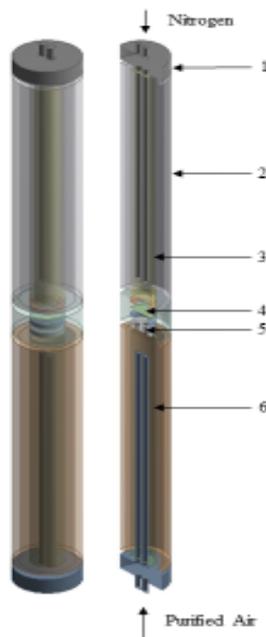


Fig. 1. Isometric and cross section view of DCFC.1: flange, 2: ceramic cylinder, 3: anode wiring, 4: button cell, 5: button cell platform, 6: cathode wiring.

3. Results and Discussions

3.1. Fuel analysis

3.1.1. Mass analysis

The overall mass characterisations of the biomass through stages of torrefaction and pyrolysis is shown in Table 1. The mass yield of mesocarp fibre samples demonstrated a detrimental trend with higher torrefaction temperatures. The decrease of mass is credited from the release of moisture and thermal decomposition to form volatile matter. The higher mass loss seen from temperatures of 260 and 290 °C showed more devolatilisation of hemi-cellulose compared to mass losses at 230 °C attributed mainly from moisture. The decomposition rate of the PMF was also seen to have increased at the temperature of 290 °C attributing to complete devolatilisation of cellulose composition. This is in agreement with findings from other research works [10,11]. The overall mass yields between the ranges of 28 wt. % to 30 wt. % are representative of rapid decomposition of cellulose. This indicates the high cellulose composition nature of PMF [10]. Therefore, torrefaction can improve the logistic and storage capabilities of PMF before being converted into biochar.

Table 1. Mass characterisation for thermally treated PMF.

Biomass Characteristic	Initial Mass (g)	Final Mass (g)	Mass Balance (wt %)	Pyrolysis at 650 °C			Overall Yield (wt %)
				Initial Mass (g)	Final Mass (g)	Mass Yield (wt %)	
Torrefied at 230 °C	15.00	12.74	84.88	13.03	4.26	32.69	28.39
Torrefied at 260 °C	15.00	10.71	71.37	11.59	4.48	38.69	29.89
Torrefied at 290 °C	15.00	8.39	56.19	9.02	4.41	48.88	29.40
Raw				15.00	4.34	28.93	28.93

3.1.2. Thermogravimetric analysis

The TGA curves of thermally treated PMF via torrefaction and pyrolysis are shown in Fig. 2. It can be seen that the pyrolysis process heavily influences the mass loss trend of the biomass. All curves show an initial weight lost step contributing to the moisture content of the sample. This is compared further in detail in Table 2. The moisture content for samples that have undergone torrefaction demonstrates a moisture content that is above 9.00% with raw mesocarp fibre having a moisture content of 7.95%. The directly torrefied sample at 260 °C shows the highest moisture content at 9.949%. This demonstrates that torrefaction is a susceptible method for storage and pelletization of mesocarp fibre similar to other works [7].

The shallow degradation of the samples 230A & 290A post-torrefaction curves after 115 °C demonstrates the lower volatile matter composition and would suggest a higher percentage of lignin in the biomass. This can be supported by the tendency of hemicellulose to deconstruct at a lower temperature due to a short chain polymer structure [12]. Whereas lignin is credited with a more complex chemical composition that enables thermal stability at higher temperatures. In accordance

with other works, biomass samples rich in lignin generically demonstrates gradual mass loss past 300 °C followed by an observable mass loss at 750 °C representing the decomposition of lignin [13]. In addition, the yields of char from the analysis is evidence of high lignin biomass [14].

The rapid devolatilisation can be analysed as the fast decomposition and liberation of hemicellulose and cellulose evident by the heavy mass drop of both samples. This relates to a respective mass drop of 70.01% and 52.91% for raw and torrefied mesocarp fibre at 260 °C. Observably, this temperature is where the biomass sample is reduced to below 50% of original weight. This is supported by typical biomass composing of 80 wt. % volatile fraction with the remaining 20 wt. % representing solid carbonaceous residue [15]. This corresponds to a lower fixed carbon weight composition for torrefied and raw mesocarp fibre.

Table 2. Moisture and volatile matter content of mesocarp fibre torrefied at 230 °C (230A), 260 °C (260A) and 290 °C (290A) before pyrolysis at 650 °C.

Biomass Sample	Moisture Content (%)	Volatile Matter Content (%)
Raw	7.102	57.845
230A	9.804	2.757
260A	9.949	3.213
290A	8.897	3.132
650C	8.612	3.244

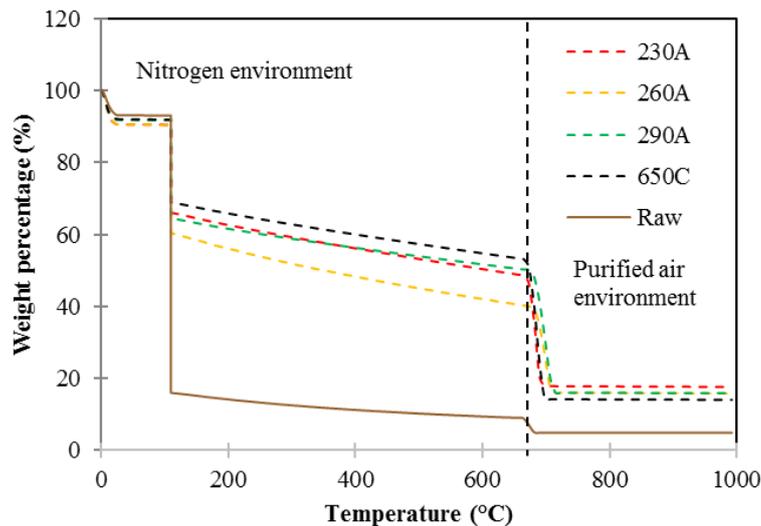


Fig. 2. TGA curve against time for PMF; Raw (brown), direct torrefaction at 650 °C (black), direct pyrolysis (black), torrefied at 230 °C (red), 260 °C (orange) and 290 °C (green) before pyrolysis.

Comparatively, this ranks biomass that is directly pyrolysed to show a lesser fixed carbon percentage followed by the sample thermally treated at 290 °C and 260 °C. This is similarly observed in research works whereby higher torrefaction temperatures can prematurely decompose lignin compositions in biomass [10]. Hence, torrefied biomass exhibit good thermal stability which can support their performance in DCFC.

3.1.3. Fourier-transform-infra-red

The IR spectra for the mesocarp fibre exposed to thermal treatment via torrefaction and pyrolysis is shown in Fig. 3. The spectra of the biomass samples show distinctive peaks that corresponds to cellulose, hemi-cellulose and lignin compositions in biomass.

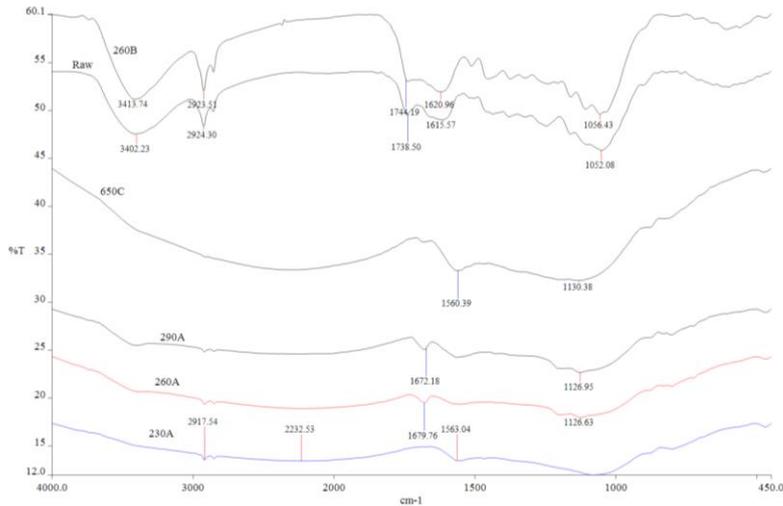


Fig. 3. FTIR for PMF; raw, direct torrefaction (260B), direct pyrolysis (650 °C), torrefied at 260 °C (260A) and 290 °C (290A) before pyrolysis.

As indicated in Figure 3, mesocarp fibre that has not undergone pyrolysis exhibit peaks around the 3400 cm^{-1} which demonstrates O-H stretching common in phenolic compounds. This is trait commonly associated with lignin rich biomass [13]. Peaks around the band of 2900 cm^{-1} indicate C-H stretching in the compound. These peaks attributes to the alkane and alkene functional groups associated with presence of cellulose. Additionally, this shows successfully decomposition of lignin and cellulose via pyrolysis of the biomass.

Peaks shown between the wavelengths of 1560 cm^{-1} and 1620 corresponds to C=O axial deformation that indicates the presence of various acids, aldehydes, esters and ketones related to the presence of aromatic ring models. This observation is normally associated with presence of hemicellulose and cellulose. Peaks observed in the range of 1130 cm^{-1} and 1500 cm^{-1} are assigned as O-H deformation and C-O stretching of oxygenated compounds such as alcohols, phenols and ethers. The aromatic C-O stretching of methoxyl and phenyl propane structure and aromatic ring vibration in lignin correspond to the peaks that occur between 1500 cm^{-1} and 1000 cm^{-1} indicating degradation of lignin. This supports the decomposition of lignin during pyrolysis of the mesocarp fibre.

3.1.4. X- ray diffraction

The XRD patterns for the thermally treated PMF are presented in Fig. 4. High intensity peaks detected at 15° for the biomass torrefied at 260°C . This peak phenomenon is remained after the sample is subjected to pyrolysis at 650°C which is representative of cellulosic material such as rubber wood sawdust [9].

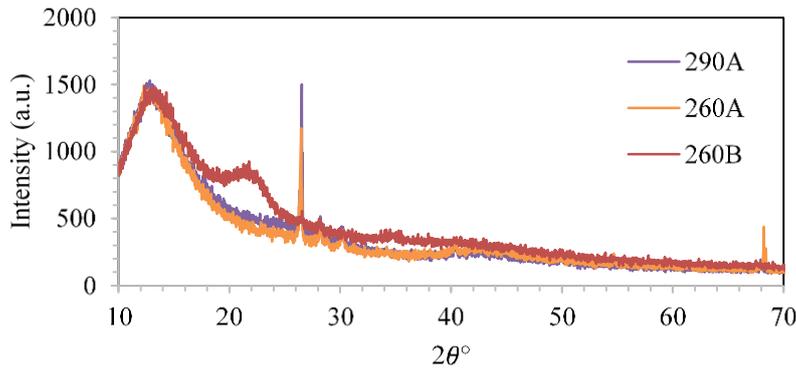


Fig. 4. XRD analysis of PMF torrefied at 260 °C (orange), biochar torrefied at 260 °C (green) and 290 °C (yellow) before pyrolysis at 650 °C.

PMF torrefied at 260 °C also demonstrate composition of cellulose from broad peak patterns at 21°. This peak is shifted to the right to 2° after pyrolysis indicating a loss of crystal structure in cellulose attributed to a devolatilisation of cellulose composition. This is in accord with research of pinewood heat treatment [16]. The peak transformed becoming narrower indicating possible formation and evolution of turbostratic crystallites [17].

Non-discriminative peaks detected at 68° show possibility of ash formation from the pyrolysis process. However, the better degree of graphitisation shown for treating PMF with pyrolysis can support performances in DCFC [18].

3.2. Fuel performance in DCFC system

The performance of the PMF subjected to different methods of thermal treating can be seen in Fig. 3. Thermal treatment via torrefaction and pyrolysis show significance from one another based on their Open circuit voltage (OCV) and peak power density. The highest OCV of 0.82 obtained was from the biochar torrefied at 230 °C. Lowest observed OCV of 0.74 was detected from the biomass subjected to torrefaction at 260 °C before being pyrolysed at 650 °C. The range of generated OCV values are in accord with research concerning activated carbon and waste coffee grounds [3]. This supports credibility for the PMF to be used as a fuel when compared to other forms of biomass.

The high OCV value is possibly credited to the presence of CO originated from decomposition of ash components containing oxygen or alternatively reaction with CO₂ subsequently released from the fuel cell reaction. CO has a higher tendency to form in lignocellulosic biomass where substances of impurities in the form of Ca, K and Fe could catalyse the formation [19].

This can be observed from works of torrefied PMF shown in Table 3. The lowest shown OCV was demonstrated by PMF torrefied at 260 °C which could be credited to the targeted decomposition of biomass constituents for impurity and moisture reductions. The more rapid linear decrease of the voltage for the PMF is likely credited to the ash formation degrading the performance of the DCFC for temperatures of 230 °C.

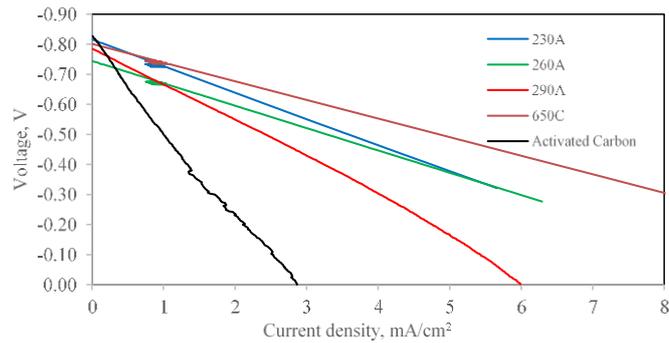


Fig. 5. Linear Swept Voltammetry for PMF biochar torrefied at 230 °C (red), 260 °C (green) and 290 °C (blue) before pyrolysis, activated carbon (black).

Table 3. Ultimate analysis of PMF thermally treated via torrefaction.

Temperature	Ultimate Analysis (Weight %)			Source
	C	H	O	
260	50.50	4.27	43.59	[10]
260	57.08	4.85	14.63	[7]
250	47.70	5.20	40.18	[20]

The highest peak power densities obtained was 2.5 mW/cm² from the directly pyrolysed PMF at 650 °C. This can be credited to a more amorphous carbon structure, better thermal reactivity and higher compositions of impurities [21]. Torrefaction temperatures of 230 °C and 260 °C showed similar peak power densities. This follows the original intent to reduce hemi-cellulose and cellulose components. Targeted devolatilisation temperatures from torrefaction thermal treating ultimately provided more thermal stability that demote DCFC performance. The highest torrefaction temperature of 290 °C targeted to reduce lignin components in the biomass ultimately provided the thermal stability which demonstrates its low peak power density. This shows similarity for low current density operations of DCFC with woody biomass crediting low current density performance to surface functional groups [22].

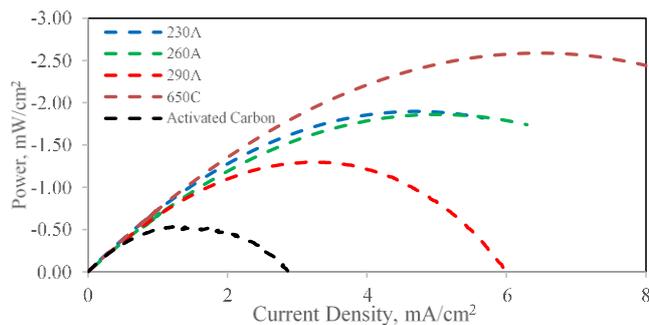


Fig. 6. Peak Power Density for PMF biochar torrefied at 230 °C (red), 260 °C (green) and 290 °C (blue) before pyrolysis, activated carbon (black).

3.3. Potentiostat electrochemical impedance spectroscopy

The cumulative impedance spectra of all biochar and activated carbon are shown in Fig. 7. However, the evident difference in total resistance magnitudes for the total resistance is too large when comparing samples of activated carbon and 290A. Therefore, a minor graph is represented in the inset of Fig. 7. Biochar samples used for the DCFC exhibits two distinctive arcs that represents the ohmic and polarisation resistance of the biochars. These arcs resemble similar works that demonstrates two similar arcs [23]. The numerical tabulation for the results is shown in Table 4 after fitted with the simplex model.

Table Error! No text of specified style in document.. Tabulated resistances for biochars and activated carbon from potentiostat EIS.

Sample	Ohmic resistance, $\Omega \text{ cm}^2$	Polarisation resistance, $\Omega \text{ cm}^2$	Total resistance, $\Omega \text{ cm}^2$
650C	1.05	2.27	3.32
230A	1.53	2.14	3.67
260A	2.65	5.41	8.06
290A	43.50	94.40	137.90
Activated Carbon	14.16	10.45	24.61

In terms of comparison, biochars derived from PMF demonstrated an overall lesser impedance compared to that of commercial fuels. This relates to the generated peak power densities shown in Table 4. The least resistance is evident from the biochar that underwent pyrolysis only, producing a total resistance of $3.32 \Omega \text{ cm}^2$. As the torrefaction temperatures increased for the biochar samples, the resistance progressively increases. Hence a more thorough form of torrefaction at higher temperatures increases the overall resistance of the biochar.

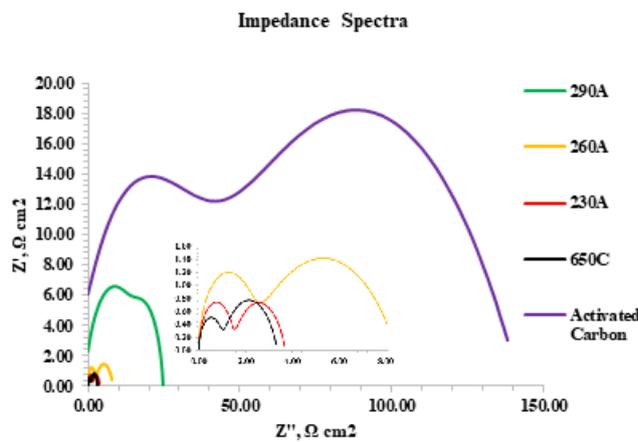


Fig. 7. Impedance Spectra of all tested biochars and activated carbon.

4. Conclusions

Thermally treated mesocarp fibre was utilised as a fuel in DCFC operations in this work. Thermal treatments were done using selective temperatures targeted for volatile matter decomposition. The findings of this research show that torrefaction

of mesocarp fibre that show release of volatiles can improve the carbon composition of the biomass from fixed carbon. Torrefaction of PMF with a temperature of 290 °C can cause lignin degradation that ultimately increase thermal stability of the biomass.

The capability of the fuel cell performance for low current density regions favoured direct pyrolysis of mesocarp fibre. Subjecting biomass to torrefaction temperatures that are too high ultimately jeopardises its performance in a DCFC credited from the loss of surface functional groups and increased ash. If the biomass were to be torrefied for logistic and storage purposes, a temperature of 260 °C would be recommended as it shows a lesser potential for ash generation in DCFC. In addition, the peak power density and OCV of the biochar previously torrefied at 260 °C shows similar performances to those performed at minor torrefaction temperatures.

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