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# Fabrication and Performance of Fuel Cell Catalyst Layer Made with Graphitic and Carbon Black Catalyst Supports

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Fabrication and Performance of Fuel Cell Catalyst Layer Made with  
Graphitic and Carbon Black Catalyst Supports

by

Sushmit Sadanand Poojary

A THESIS

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# Abstract

Hydrogen fed polymer electrolyte membrane fuel cells (PEMFCs) are at the forefront of clean energy technologies. Their application in transportation sector is now considered to be primarily in heavy duty vehicles (HDVs). Currently, the technological challenges for PEMFC development for HDV application is to increase the fuel cell stack durability. One of the components considered weak link for PEMFC durability is the carbon support for platinum catalyst used in the cathode and anode catalyst layers. Conventionally used carbon black type catalyst support have poor corrosion resistance as they are prone to both cathodic and anodic corrosion. Graphitization of the carbon black or use of graphene as catalyst support has been reported in the literature. However, the challenges associated with the fabrication and performance of graphene-based catalyst layer need to be addressed.

In this thesis, base line data for catalyst layer made with commercially available carbon-black type catalyst support is first established. As a part of this study, the influence of ionomer side chain length or equivalent weight (Aquivion-825 and Nafion-1100) on catalyst layer properties and cell performance was quantified for the commercial catalysts. The observation of higher ORR kinetic activity ( $A/cm^2_{Pt}$ ) for the Aquivion-825 CL than the Nafion-1100 CL is an interesting finding and is hypothesized to different interfacial protonic concentrations between the two CLs at the Pt/ionomer interface. Aquivion-825 CL had a higher local oxygen transport resistance than the Nafion-1100 CL, which is also indicative of changes in the Pt/ionomer interface and is consistent with a stronger contact between Pt and ionomer in the case of more acidic ionomers. Similar dependence on Pt utilisation as a function of relative humidity (RH) is seen for the two CLs (ratio of electrochemically active area at any RH to that at 100% RH). As anticipated, a significant impact of humidity on proton conduction is seen. The CL with the larger equivalent weight ionomer (Nafion-1100) demonstrates lower conduction.

A graphene-based catalyst was fabricated, first by using one-step electrochemically exfoliated graphene co-doped with nitrogen and phosphorus, upon which platinum catalyst was subsequently deposited. The in-situ electrochemical characterization showed that the carbon black or Vulcan carbon-based CL outperformed the graphene-based CL. Even though the CV demonstrated that there was electrochemically active Pt present in the CL, the poor performance, absence of a limiting current, and high CL protonic resistance suggest that the reactant gas is unable to reach the active sites due to poor CL porosity, possibly as a result of the stacking of the graphene layers. These results highlight a significant problem in the development of graphene-based catalyst layers for PEM fuel cells.

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Lastly, always remember:

**“ A trail of red in the snow leads to the gates of iron in the sky”**

**-Sushmit Poojary**

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## List of acronyms

ACL - Anode catalyst layer

AST - Accelerated stress test

BET - Brunauer-Emmett-Teller method

CA - Cathode

CL - Catalyst layer

CCL- Cathode catalyst layer

CCM - Catalyst coated membrane

CDL - Double layer capacitance

CE - Counter electrode

CNT - Carbon nanotube

CNC – Carbon nanocage

CNF – Carbon NanoFibres

CV - Cyclic voltammetry

DC - Direct current

DI – Deionized

DL - Double layer

DLS – Dynamic Light Scattering

DOE - Department of energy

ECSA - Electrochemical active surface area



EDS or EDX - Energy dispersive x-ray spectroscopy

EG - Ethylene glycol

EIS - Electrochemical impedance spectroscopy

EW - Equivalent weight

FC - Fuel cell

FCEV - Fuel cell electric vehicle

FCV - Fuel cell vehicle

FF - Flow field

GDE - Gas diffusion electrode

GDL - Gas diffusion layer

GO – Graphene Oxide

HER - Hydrogen evolution reaction

HFR - High frequency resistance

HOR - Hydrogen oxidation reaction

HUPD - Hydrogen underpotential deposition

ICE - Internal combustion engine

IPA - Isopropyl alcohol

IPCC - Intergovernmental panel on climate change

LDV - Light duty vehicle

LSV - Linear sweep voltammetry

MEA - Membrane electrode assembly

MPL - Microporous layer

NLPM - Normal liter per minute

OCV - Open circuit voltage

ORR - Oxygen reduction reaction

PEFC - Polymer electrolyte fuel cell

PEM - Polymer electrolyte membrane

PEMFC - Polymer electrolyte membrane fuel cell

PFSA - Perfluorosulfonic acid

PGM - Precious group metal

PI - Pressure independent

PD - Pressure dependent

PSD - Particle size distribution

PTFE - Polytetrafluoroethylene

RDE - Rotating disc electrode

RE - Reference electrode

RF - Roughness factor

rGO- Reduced graphene oxide

RH - Relative humidity

RHE - Reversible hydrogen electrode

RT - Room temperature

SA - Surface area

SEM - Scanning electron microscopy

SLPM - Standard liter per minute

SOA -State of the art

TEM - Transmission electron microscopy

TGA - Thermogravimetric analysis

TKK - Tanaka Kikinzoku Kogyo K.K.

UHP - Ultra-high purity

UHV - Ultra-high vacuum

UK - United Kingdom

USA - United States of America

UV - Ultra-violet

VC - Vulcan carbon

WE - Working electrode

XPS - X-ray photoelectron spectroscopy

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# Chapter 1: General introduction

## 1.1 Global energy challenge

There continues to be a steady rise in world energy consumption due to rapid socio-economic growth on a global scale. Currently, majority of the world's energy supply comes from carbon-based energy sources including coal, petroleum, and natural gas [1]. These conventional sources of energy are not sustainable for two reasons. First, they are non-renewable energy sources. Second, they cause the build up of CO<sub>2</sub>, greenhouse gases and other criteria pollutants in the atmosphere leading to health problems, climate change in addition to other dire environmental impacts[2]. This has led to increased efforts towards the development of renewable sources of energy such as solar, wind, nuclear and biomass energy. The energy produced by these sources require an efficient, low emission method of energy conversion for practical purposes such as stationary power, portable power, and transportation.

## 1.2 PEM fuel cells – Overview

Fuel cells are electrochemical devices that convert chemical energy stored in fuels to electricity. They are widely regarded as the next generation energy conversion systems due to their high efficiency and low emissions. Polymer electrolyte membrane fuel cells (PEMFCs) use hydrogen gas as fuel. Low temperature PEMFCs (LT-PEMFCs) typically operate at temperatures below 100°C. PEMFCs are currently being developed for portable power, transportation, and small stationary power requirements [3]. PEM fuel cells generate current by oxidizing hydrogen on the anode catalyst layer and simultaneously reducing oxygen at the cathode where the electrons are forced to move through external circuit while the protons are transported through the PEM separating the anode and the cathode. PEM fuel cells require the use of expensive platinum metal catalysts to catalyze the oxygen reduction reaction (ORR) at low temperatures and this is a major contributor to the high cost of a PEMFC.

Also, PEMFCs are prone to durability issues due to the poor corrosion resistance of conventionally used catalyst supports (For example, carbon black). This adds to the operational and maintenance cost of a PEMFC. In addition to low temperature operation, PEMFCs offer quick start up, high power density and are easy to scale up making it a worthy successor to IC engines. In addition, PEMFCs can provide small scale power (1 – 250 kW) through distributed power systems [4]. PEMFCs offer several benefits over IC engine powered vehicles, including higher efficiencies (>60%), similar driving range (>300 miles) and refuelling time (<5 mins) [5].



### 1.3 Technological hurdles

PEMFCs have potential applications ranging from stationary power applications (e.g. primary power source for homes and buildings), portable power applications for consumer electronics to transportation applications like buses, trucks, and cars.

In the last decade, the interest for PEMFCs in the transportation sector has shifted from light duty vehicles (LDVs) to heavy duty vehicles (HDVs). This paradigm shift was due to the unique scalability of PEMFCs in terms of power and energy as compared to lithium-ion batteries [6]. Also, commercialization of PEMFC powered HDVs requires lesser infrastructure investment in terms of required number of H<sub>2</sub> refuelling stations due to predictable routes. However, the different operation conditions, different drive cycles and longer required lifetimes means that more focus is required on improving durability and fuel efficiency as compared to LDVs [7]. The focus on HDV market is also critical in terms of reducing emissions and energy consumption. As shown in Figure 1.1, the U. S. Department of Energy (DOE) recently released targets for hydrogen class 8 long haul trucks which further emphasizes the need to focus on longer lifetimes and increased efficiency demands of HDV applications.

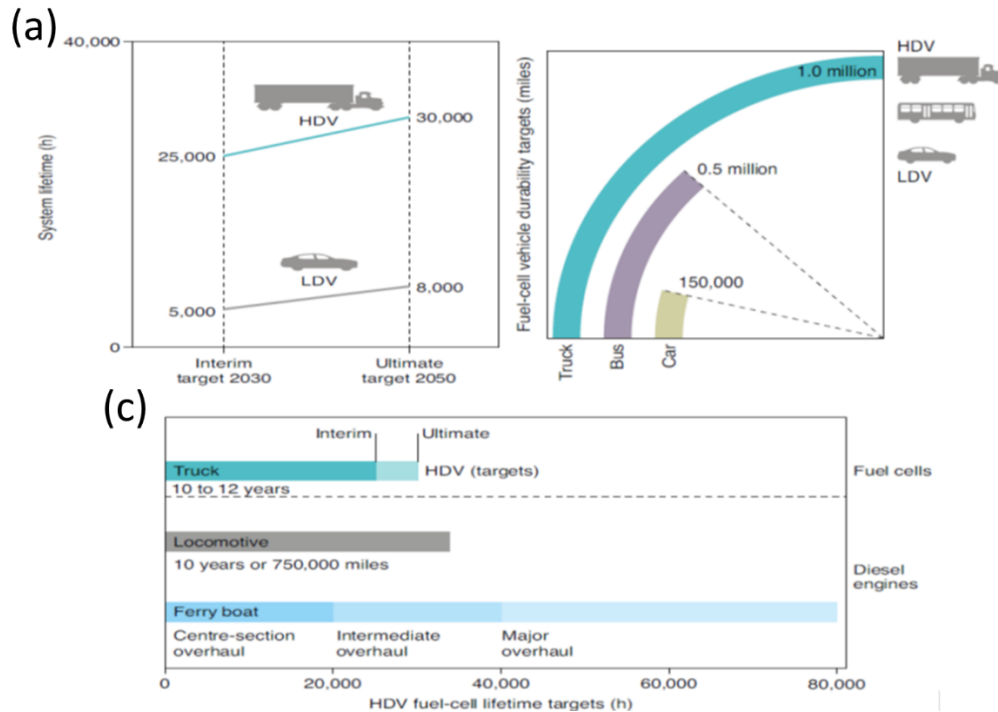


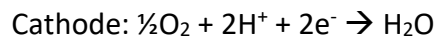
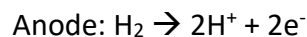
Figure 1.1: a,b. Interim and ultimate DOE fuel-cell targets for automotive and trucks, c. Comparison of HDV fuel-cell lifetime targets with the useful service lifetime of current diesel engines for rail and marine applications (Reproduced with permission from [8]).

PEMFCs have demonstrated long term operating ability but only with high loading of expensive platinum metal catalyst and under ideal operating conditions. There is a demonstrated need to better understand the relationship between the mechanisms of PEMFC performance degradation, factors affecting durability, operating conditions, and changes in material properties during PEMFC operation. This has motivated several recent studies on the effect of PEMFC material properties on PEMFC durability and degradation[8].

Since HDVs require extended lifetime (25,000 hours), the issue of durability is critical. Thus, some of the key material development related areas of research are (1) catalysts and ionomers stable at temperatures above 90–100 °C, (2) highly stable ionomers and membranes under dry conditions, and (3) high durability catalysts, catalyst supports and membranes. The above-mentioned components will be explained in detail in the succeeding section.

#### **1.4 Working principle and components of PEMFC**

In a PEMFC, the chemical energy stored in hydrogen is converted to electrical energy through an electrochemical reaction. The key most component of a PEMFC is the membrane electrode assembly (MEA). The structure of an MEA is similar to that of a sandwich. It consists of a solid polymer electrolyte membrane that is sandwiched between anode and cathode side catalyst layers on either side of it (as shown in Figure 1.2). This membrane is made of an ionomer which can conduct  $H^+$  ions or protons [9]. Externally stored  $H_2$  gas is delivered to the anode catalyst layer through the anode flow field and the air/oxygen is delivered to the cathode catalyst layer through a similar flow field. At the anode,  $H_2$  is oxidized into protons (via hydrogen oxidation reaction or HOR) and electrons. The protons migrate to the cathode side of the PEMFC through the proton conducting membrane and the electrons flow to the cathode side through the external circuit where it combines with the protons and oxygen to form the only product water. The latter reaction is also known as oxygen reduction reaction or ORR, and it dictates the kinetics of the overall fuel cell reaction as shown below:



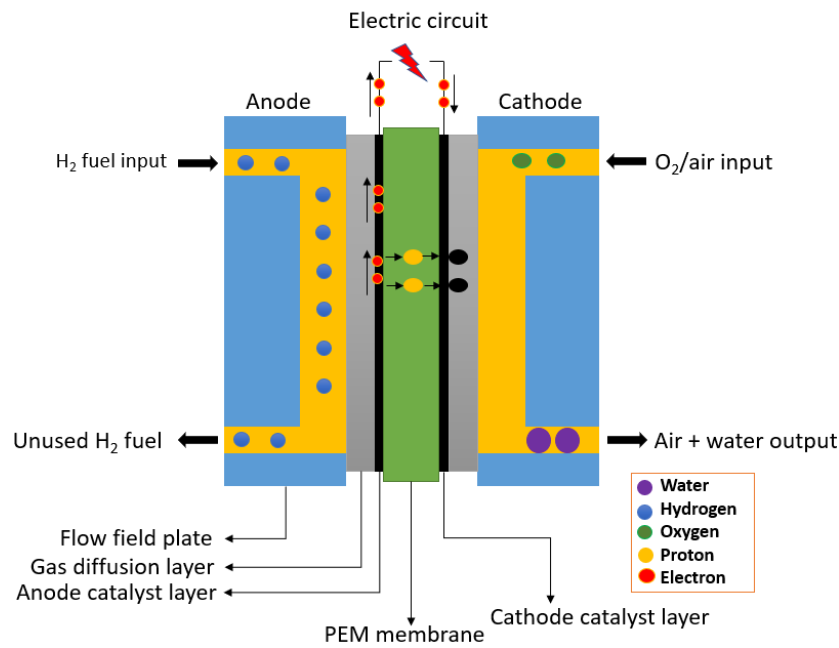


Figure 1.2: A schematic diagram of PEM fuel cell showing the key components.

The respective roles of each component have been explained in Table 1.1.

Component	Constituents	Dimensions	Role
Membrane	Ionomer	25-100 $\mu\text{m}$	<ul style="list-style-type: none"> <li>Proton transport from anode to cathode [11]</li> </ul>
Gas diffusion layer (GDL)	Porous carbon paper coated with PTFE	240-400 $\mu\text{m}$	<ul style="list-style-type: none"> <li>Ensures even diffusion of gaseous reactant to the CL</li> <li>Removes excess water from the CL-membrane interface [12]</li> </ul>
Catalyst layer (CL)	Ionomer, carbon, Platinum nanoparticles	10-20 $\mu\text{m}$	<ul style="list-style-type: none"> <li><b>Platinum:</b> Acts as a catalyst and provides surface area for electrochemical reaction to take place.</li> <li><b>Ionomer:</b> Transports proton from CL to membrane and vice versa.</li> </ul>

			<ul style="list-style-type: none"> <li>▪ <b>Carbon:</b> Provides support to the Pt catalyst [13]</li> </ul>
Bipolar plates	Metal, graphite, carbon		<ul style="list-style-type: none"> <li>▪ Facilitates electron transport from anode to cathode.</li> <li>▪ Contains the gas flow field</li> <li>▪ Provides mechanical strength [14]</li> </ul>
Gaskets	PTFE	175 $\mu\text{m}$	<ul style="list-style-type: none"> <li>▪ Enables a gas tight seal [14]</li> </ul>

Table 1.1: Roles of each PEMFC component

## 1.5 Voltage breakdown for a PEMFC

The performance of PEMFC is most commonly described by current-voltage characteristics. The i-V curve of a fuel cells is also known as the polarization curve. A typical polarization curve is shown below. It is to be noted that typically the current is normalized with the active area of the electrode and represented as current density, i.e., amperes per  $\text{cm}^2$  of catalyst layer or electrode. With an increase in current being generated in the fuel cell, the cell voltage decreases due to various losses [15]-(i)  $\text{H}_2$  cross over loss, (ii) Activation loss, (iii) ohmic loss and (iv) Mass transport loss. The regions of polarization curves “dominated” by different losses are identified and have been illustrated in the Figure 1.3.

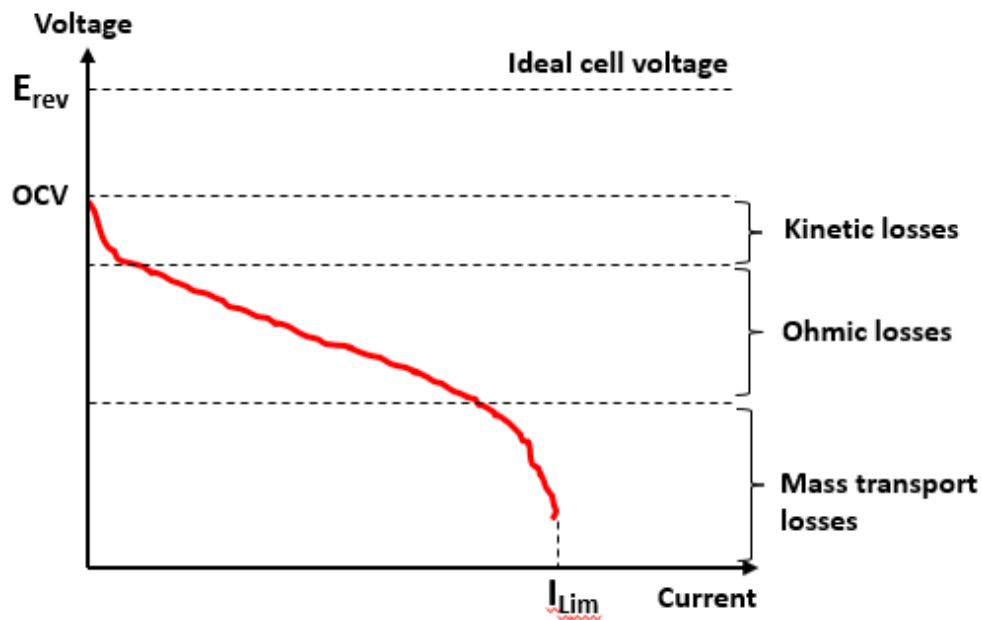


Figure 1.3: PEMFC polarization curve highlighting voltage losses

The different types of voltages losses are explained below:

(i) **Hydrogen cross-over loss:** H<sub>2</sub> cross-over loss happens due to the undesirable diffusion of H<sub>2</sub> gas molecules from anode to cathode through the membrane and it reduces the open circuit voltage (OCV) of the cell.

(ii) **Activation loss ( $E_{act}$ ):** This voltage loss occurs at low current densities and is caused by low activation energy of the electrochemical reactions at the electrodes. Slow ORR kinetics at the cathode, which is 5 orders of magnitude slower than HOR kinetics at the anode, is mainly responsible for activation losses [17].

(iii) **Ohmic loss ( $E_{ohmic}$ ):** At intermediate current densities, ohmic losses are caused by resistance to proton and electron flow in the polymer electrolyte and electrodes respectively. In this region, the cell potential decreases linearly with current density.

(iv) **Mass transport loss ( $E_{con}$ ):** At higher current densities, mass transport losses dominate due to transport limitations of reactant gasses through the GDLs and catalyst layers (CL) and this causes a drastic drop in cell performance.

The output voltage of a single cell ( $E_{cell}$ ) is given by:

$$E_{cell} = E_{OCV} - E_{act} - E_{ohmic} - E_{con}$$

## 1.6 PEMFC Catalyst layer – Structure and composition

A PEMFC's performance, durability, and cost are strongly linked to the catalyst layer, which conventionally is a nanoporous, nanocomposite of platinum nanoparticles (2 –5 nm) supported on a carbon support made of carbon black nanoparticles (20 –30 nm) covered with a thin film of ionomer [18]. The Pt catalyst accounts for nearly 50% of the stack cost, kinetic and transport losses contribute to over 65% of voltage losses that dominate the overall cell performance [19] and the corrosion of carbon support as well as Pt aggregation and dissolution are of major concerns for the CL durability [20]. Moreover, the electrochemical performance and the effective utilization of Pt catalysts are controlled by the complex nanostructure of the CLs. The local CL nanostructure has been depicted in Figure 1.4.

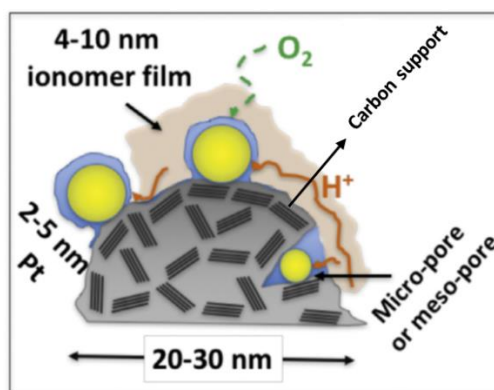


Figure 1.4: CL structure and components (Reproduced with permission from [21])

The CL consists of the following four components:

**Carbon support:** The most commonly used catalyst support is 20-30 nm Vulcan carbon. The primary role of carbon support is to conduct electrons and support the catalyst nanoparticles. An ideal carbon support must possess high electrical conductivity, high surface area and a highly porous structure to facilitate even coverage of ionomer thin film for optimum proton conductivity in the CL [22]. Also, low combustion rate at low operating temperature range ( $<150^{\circ}\text{C}$ ) and high stability under PEMFC operating conditions are favourable qualities in a CL [23]. Evidently, conventional carbon supports like carbon black (CB) are prone to corrosion which negatively impact the durability and performance of the PEMFC [24]. The use of non-conventional PEMFC carbon supports like activated carbons and graphitized materials have also been reviewed [25]. There also has been a considerable amount of effort towards modifying the surface chemistry of carbon supports by functionalization. The catalytic properties of carbon supports can be favourably altered by tuning surface chemistry [26]. Furthermore, multiple studies have reported that the introduction of functional groups (like nitrogen, phosphorus, sulphur, etc) on the surface of carbon supports facilitated activity improvements in addition to the deposition of well-distributed, highly dispersed, and smaller platinum nanoparticles [27,28,29].

**Ionomer thin film:** The ionomer thin film is what brings the CL to life! The ionomer thin film in the catalyst layer is approximately 4-7 nm and should ideally cover the entire surface area of the catalyst particles (Pt/C) [R]. It is responsible for conducting protons ( $\text{H}^+$ ) to and from the surface of the Pt catalyst and it also controls the static and dynamic microstructure of the CL which is sensitive to the relative humidity, temperature, ionomer concentration in the CL and catalyst ink composition. In addition, local transport of reactant gases ( $\text{H}_2$  or  $\text{O}_2$ ) to the active catalyst sites are dictated by the ionomer.

Nafion®, a perfluoro-sulphonated ionomer developed by DuPont in the 1960s [31,32,33], has most commonly been used as ionomer in PEMFC CL owing to its high proton conductivity ( $\sim 0.1$  S/cm at 100% RH, 80°C) [34] and chemical resistance [35]. It has been reported that proton transport is hindered at low Nafion® loading, while electron conductivity and O<sub>2</sub> diffusivity, on the other hand, is challenged at high loading. High Nafion loading is also held accountable for flooding in CL due to lack of water management, which restricts the transport of gasses to active site contributing to further mass transport losses. Thus, optimization of ionomer loading in CL is the key to CL performance. An ionomer loading of approximately 2 mg/cm<sup>2</sup> has been reported as ideal in CL [36].

**Catalyst:** In conventional catalyst layer, the 2-5 nm platinum catalyst is dispersed on the carbon support and provides sites for electrochemical reactions. An ideal PEMFC catalyst must have (i) high activity for the targeted reaction (ii) exhibit stability during prolonged operation (iii) possess high selectivity for the desired reaction over any side reactions and (iv) resistance to CO poisoning. Pt nanoparticles are ideally used as a catalyst for the PEMFC electrochemical reactions, and they must be evenly distributed on the carbon support for optimum catalyst utilization [37]. Presently, Pt is the only element among all available active metal catalysts which can fulfill these criteria while avoiding slow reaction kinetics. However, the high cost and dearth of Pt are currently the major obstacles holding back large scale PEMFC commercialization.

**CL nanostructure:** An ideal CL must have a uniform pore size distribution to allow the diffusion of reactant and product gases through the CL. Poor CL porosity results in high O<sub>2</sub> gas transport resistance to the catalyst surface. Depending on pore size, these pores can be classified as micropores (<20nm), mesopores (>20nm), and macropores [38]. Also, the ionomer must be evenly distributed as a thin film (4-7 nm) over the catalyst-carbon-support interface to allow optimal protonic conductivity. If this ionomer thin film is too thick, the mass transport resistance of reactant gases will be too high. Ideally, the Pt nanoparticles needs to have interfacial contact with both - carbon support and ionomer thin film to allow efficient conduction of protons and electrons [39].

### 1.6.1 Importance of ionomer in the PEMFC catalyst layer

It is well known that catalyst layer (CL) is a multi-scale composite structure made up of carbon supports, ionomer materials, PGM electrocatalysts and pores. Therefore, enhancing cell performance requires a catalyst-ionomer-carbon support design structure that works. However, research has revealed that the ionomer coating on the catalyst particles reduces the oxygen reduction reaction (ORR) activity and makes oxygen diffusion to the Pt surface more challenging, which has a significant impact on cell performance [40]. This behaviour becomes more pronounced as the Pt loading is lowered.

To bind the catalyst layer (CL) and increase its proton conductivity, perfluoro sulphonic acid (PFSA) ionomers have been chosen as the preferred ionomers. PEMFC proton conductivity is enhanced by the extensive intrinsic chemical stability and mechanical strength of PFSA ionomers [41]. Until recently, Nafion<sup>®</sup>, which has a long side chain (LSC), has been the most widely used ionomer for PEMFC membrane and catalyst support application [42]. Recent years have seen numerous innovations in the design of different alternative ionomers, one of which is the short-side chain (SSC) ionomer produced by Solvay Solexis, commonly known as Aquivion<sup>®</sup> [43].

The shorter PFSA side chain enhanced the ionomer's mechanical stability[43]. To improve proton conduction, the PFSA ionomers are added to the catalyst's surface area. The level of a PEMFC's proton and electron conductivity, as well as the degree of hydration, all affect how well it performs [42]. The ionomer in the CL must facilitate effective gas and water transport. Ionomers improve PEMFC performance when used appropriately, but if used excessively, they run the risk of limiting PEMFC efficacy by blocking the Pt sites. Nafion<sup>®</sup> and Aquivion<sup>®</sup> PFSA membranes have been compared in several experiments, but there is limited information in the literature comparing how well they work when employed as ionomers in the catalyst layers. The impact of ionomer type on fuel cell performance has been covered only in a few studies in the literature [43,44,45]. Even fewer studies [47,48,49,50] cover the ionomer-dependent electrochemical characteristics of catalyst layers, including ECSA, double-layer capacitance, kinetics, and local oxygen transport resistance. No single study covers all of these characteristics for a single catalyst layer. In Addition, the impact of RH on the aforementioned properties—ECSA, ORR kinetics, double-layer capacitance, and oxygen transport resistance of the catalyst layer has also only been investigated in a small number of studies. Also, the combined impact of ionomer structure and RH on the catalyst layer electrochemical characteristics is not adequately described in the literature. These are the main motivations for the work described in Chapter 3.

Furthermore, the nature of ionomer - carbon support interaction has a major impact on PEM fuel cell performance, durability, and ionomer coverage [51]. This phenomenon has been examined in chapter 3 and 4 of this thesis by comparing the interaction of Nafion<sup>®</sup> with a Vulcan carbon based catalyst against a graphene based catalyst. This was done by first establishing a baseline with a Vulcan carbon based CL and then comparing its performance and other CL properties with a graphene based CL. One of the motivations for chapter 4 was to understand how graphitization of the catalyst support influences PEMFC performance and other CL properties, which is why it was important to establish a baseline with a non-graphitic carbon support like Vulcan carbon in chapter 3.



## 1.7 Durability issues with PEMFC

The components of the PEMFC that are most vulnerable to degradation are – membrane, catalyst and carbon-based catalyst support [52].

**Membrane durability:** Perfluorinated sulfonic acid (PFSA) membranes are expensive and are the most used type of PEMFC membrane. The failure of these membranes can happen by chemical degradation, mechanical degradation, and thermal degradation [53]. PFSA-based polymer membranes are expected to continue to play a dominant role for the next 5-10 years [54].

**Catalyst durability:** Degradation of Pt and Pt-alloy catalyst nanoparticles and the cathode electrode structure continues to be major concerns for hindering the commercialization of PEMFCs for transportation applications. Currently used Pt-Co catalysts have stability issues arising from ECSA loss due to particle growth and transition metal leaching [55]. Methods to alleviate these issues have been identified and include BOL (Beginning of Life) catalysts with lower Co content (~10% Co), mono-disperse nanoparticle distributions, and larger BOL catalyst nanoparticle sizes of 4-5 nm [56]. However, such methods limit the catalyst's ORR mass activity due to the low mass-specific surface area ( $\text{m}^2/\text{g}_{\text{Pt}}$ ) of larger particles, thereby necessitating higher Pt loadings to achieve performance targets. Many of the primary degradation mechanisms can be mitigated by implementing system operating strategies, as is the case for systems in current commercialized FCEVs. Although system mitigation strategies have provided a near-term pathway for commercialization, material-based solutions would help to reduce cost and improve operability and efficiency [57]. Future material R&D should focus on stabilization of the electrode structure and minimization of the susceptibility of catalyst nanoparticles to dissolution and agglomeration plus minimization of support corrosion [58].

**Catalyst support durability:** The corrosion of carbon support caused by the electrochemical oxidation of carbon is inevitable during long-term PEMFC operation. Novel materials such as graphitized carbon, CNTs, CNFs, metal oxides, etc have been developed to improve the catalytic activity and stability. Moreover, there has been some significant progress made in the research on Pt alloy catalysts have also made exciting progress, which can provide an effective strategy to improve the lifetime of PEMFCs [59].

The two major barriers to widespread fuel cell commercialization and use – Cost and durability, are closely linked because Pt catalyst is expensive and is also a major source of PEMFC degradation. The PEMFC components in fuel cell electric vehicles (FCEVs) like Toyota Mirai, do not meet the long-term cost and durability targets set by

the USDOE. Table 1.2 highlights the current status and future electrocatalyst durability specific targets set by USDOE.

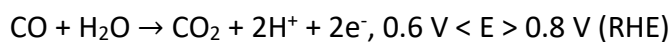
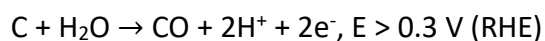
Characteristics	Units	Status	2025 target
Durability with cycling	Hours	4100	8000
Loss in catalytic mass activity	%	40	≤40
Electrocatalyst support stability	% Mass activity loss	Not tested	≤40

Table 1.2: Electrocatalyst durability targets set by USDOE [60]

The focus of the proposed research in chapter 4 is on the incorporation of durable catalyst support in catalyst layers. Hence, a review of key durability issues related to catalyst support is presented in the next section.

## 1.8 Carbon support corrosion and ECSA loss mechanism

We need to understand carbon support corrosion behavior to understand the degradation behaviour of PEMFC electrocatalysts. It has been found that, in the presence of Pt, at  $E > 0.3$  V (RHE), the carbon in the catalyst support is oxidized to CO followed by further oxidation to CO<sub>2</sub> at potentials between 0.6 V and 0.8 V (RHE) [61].



Due to the formation of CO or CO<sub>2</sub>, the carbon in the catalyst support gets consumed, reducing the amount of support left to load the Pt catalyst particles. This forces the Pt to detach from the carbon support surface

thereby reducing the ECSA. Also, the formation of oxygen functional groups on the carbon support surface reduces the conductivity of the catalyst layer. This corrosion of the carbon support increases the hydrophilicity of the CL and the GDL which negatively impacts the transport of reactant gases and water through the CL [62].

ECSA loss can happen via two mechanisms as illustrated in Figure 1.5. As shown in the figure on the left, the Pt particles oxidize to  $\text{Pt}^{2+}$  ions by dissolution into the surrounding ionomer medium. These Pt ions are then redeposited on the larger Pt particle, while the electrons move through carbon support. This mechanism is known as Ostwald's ripening [63,64]. In the second mechanism (Figure on the right), the corrosion of the carbon support causes the Pt particles to detach and migrate to a nearby Pt particle thus causing agglomeration. In both cases, transport of Pt occurs from the smaller particle to the larger particle [65]. Both Ostwald ripening and particle agglomeration lead to a continual increase of mean particle size of Pt in the CL, causing a reduction in ECSA [66].

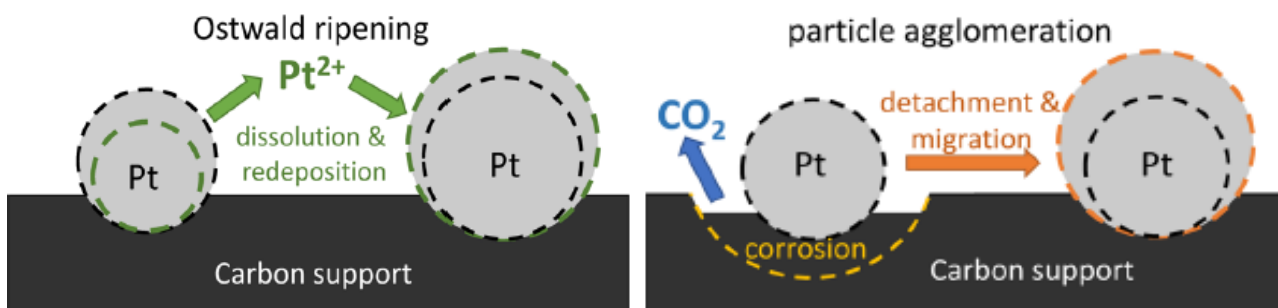


Fig 1.5: ECSA loss mechanisms- Ostwald ripening vs particle agglomeration (Reproduced with permission from [67])

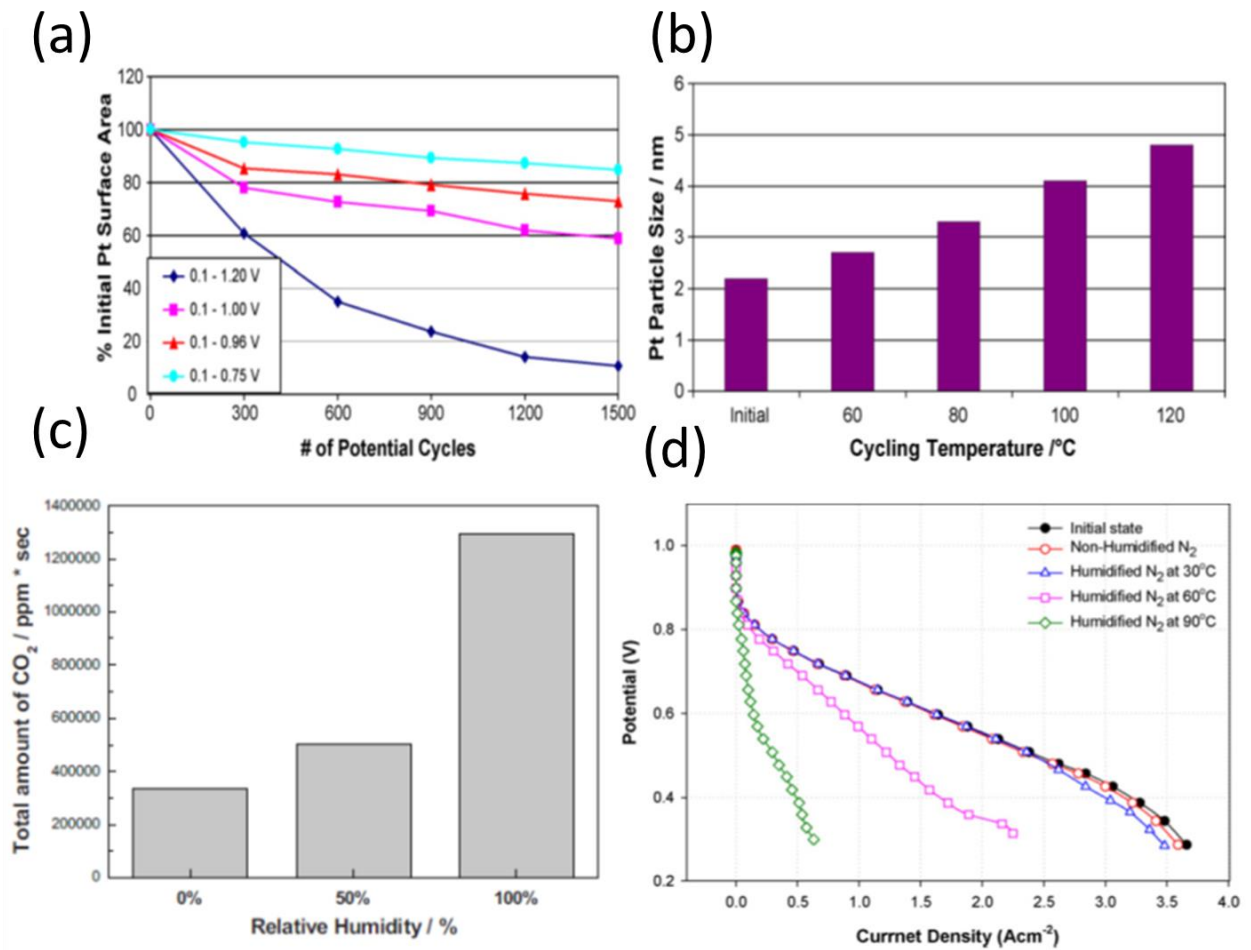
### 1.8.1 Factors influencing carbon corrosion

The mechanism of carbon corrosion has been introduced in previous section. Electrode potential and humidity are two key factors influencing carbon corrosion.

- (i) **Electrode potential:** The CL normally operates at high electrode potentials ( $>0.6\text{V}$  vs Reversible Hydrogen Electrode or RHE) and sometimes even higher potentials ( $\sim 1.4\text{ V}$ ) are required during start-up and shutdown [68]. Furthermore, PEMFC CLs are required to operate in highly acidic ( $\text{pH} < 1$ ), high temperature ( $80\text{ }^{\circ}\text{C}$  or above) and humid conditions that accelerates its corrosion rate [69]. Additionally, carbon support corrosion studies under simulated PEMFC operating conditions help us further understand the degradation mechanism of the CL. This mechanism has

been explained in further detail in the next sections. Furthermore, studies have shown that carbon monoxide (CO) formed on the surface of carbon support at  $E > 0.3$  V (RHE) is oxidized to  $\text{CO}_2$  at potentials between  $0.6 < V < 0.8$  (RHE) [70]. This formation of CO and  $\text{CO}_2$  causes the Pt nanoparticles to detach from the carbon support causing a reduction in the electrochemically active surface area (ECSA) available for HOR and ORR. Also, the formation of oxygen functional groups on the carbon support reduces the conductivity of the CL [71]. A study by Borup et. al. showed the correlation of carbon support corrosion with cell potential and temperature as shown in Figures 1.6a and 1.6b [72]. Note that the increase in Pt particle size is a measure of increase in carbon support corrosion. Moreover, carbon support corrosion results in changes in the surface chemistry of the support which negatively impacts the transport behavior of reactant gases and water due to an increase in hydrophilicity of the support surface. Carbon corrosion also reduces the CL thickness which causes an increase in CL resistance [73].

- (ii) **Humidity:** In addition to electrode potential, carbon support corrosion has been found to be influenced by relative humidity and temperature too. A corrosion study [74] done by measuring  $\text{CO}_2$  emission at a constant potential of 1.4V for 30 min using on-line mass spectrometry showed that electrochemical carbon corrosion increases with increasing humidity and cell temperature (as shown in Fig 1.6d). This phenomenon was also observed in another study done by Ofstad et. al. [75]. In addition, another study revealed that pronounced Pt agglomeration and corrosion of the carbon support were observed at higher cathode inlet relative humidity (as shown in Fig 1.6c).



Figures 1.6: a) Effect of cell potential on catalyst surface area loss, b) Pt particle size after cycling from 0.1 to 0.96V as a function of operating cell temperature, c) Dependence of carbon support corrosion on RH, d) Polarization curve showing performance loss affected by temperature and humidity (Reproduced with permission from [76,77])

It would be noteworthy to add that, most of the above studies were conducted under potential static conditions. There are very few studies that have conducted carbon support corrosion studies under potential dynamic conditions which closely resembles the actual drive cycle operation of a PEMFC stack in a vehicle.

## 1.9 Durability of currently used catalyst supports

Catalyst support plays critical roles in PEM fuel cell performance and durability. The corrosion of supports in electrocatalysts leads to the degradation of PEM fuel cell electrocatalysts. It also causes the changes of electrode structure, including porosity and surface chemistry, which affects mass transfer and water management.

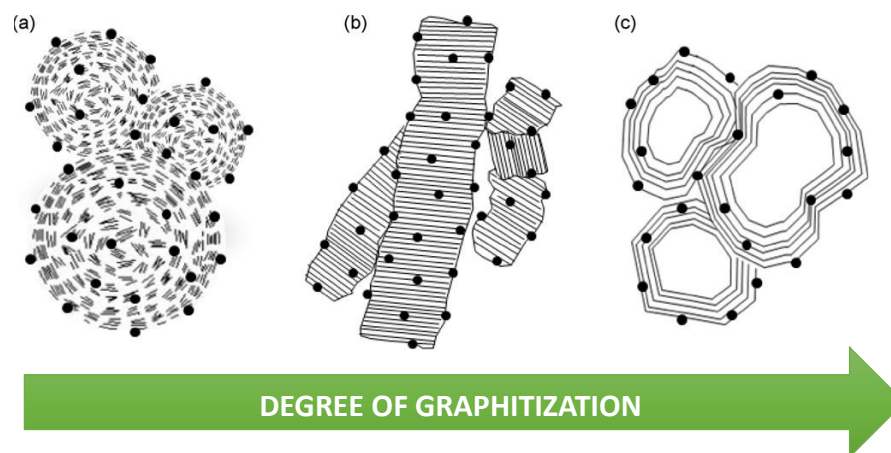
### 1.9.1 Non-carbon supports

Good progress has also been made on non-carbon support (metal oxides) and metal oxide–carbon composites [78]. Several types of non-carbon supports have been investigated, including titanium oxide, cerium oxide, niobium oxide, tungsten oxide, carbides, nitrides, borides, conductive polymers, etc [79,80,81]. General properties of non-carbon support (mainly ceramic materials) include corrosion-resistance, strong metal catalyst - support interaction, and stable porous structure, but these materials suffer from low electrical conductivity and low-porosity [82]. Furthermore, these novel support materials need to be tested in a working PEMFC setup.

### 1.9.2 Carbon based supports

Carbon is the most widely used support material for PEM fuel cell catalysts because of its high electrical conductivity, accessibility and affordability, relatively high chemical and electrochemical stability [83]. Because of the poor corrosion resistance for common carbon black, some novel carbon materials have been investigated, which include carbon nanotube (CNT, 1D), graphene (2D), and 3D structural carbon materials such as porous carbon, carbon nanocomposites, 3D graphene and highly ordered porous carbon [84,85,86].

Graphitized carbon materials particularly show improved stability and offer an alternative solution under the demanding operating conditions faced by automotive PEMFCs. In a study by H.-S. Oh et al [87], the corrosion resistance of carbon black (CB), carbon nanofiber (CNF) and carbon nanocage (CNC) used as catalyst supports in PEMFCs was examined by monitoring CO<sub>2</sub> emissions using on-line mass spectrometry at 1.4V of applied voltage (As shown in Fig 1.7). They found that the amorphous CB support was highly vulnerable to carbon corrosion compared to the other graphitized carbon supports (CNF and CNC).



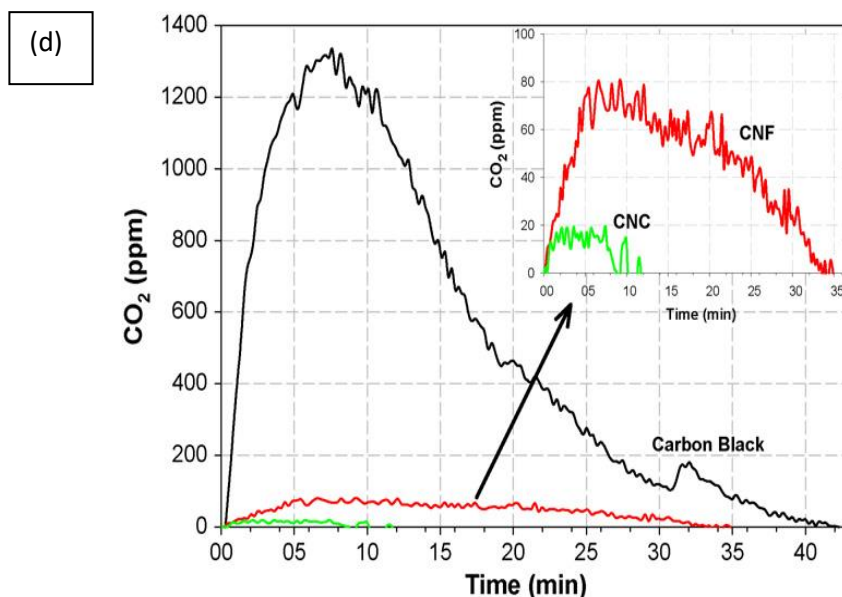


Figure: 1.7: Three different types of carbon supported Pt particles, (a) Pt/CB, (b) Pt/CNF, (c) Pt/CNC, d)  $\text{CO}_2$  mass-spectra profiles for MEAs using Pt/CB, Pt/CNF, and Pt/CNC, during the corrosion test at 1.4 V for 30 min. (Reproduced with permission from [87])

Furthermore, a study by Que et. al. showed that the carbon support with the most complete graphite lattice (Pt/EC-G1800) was more resistant to electrochemical corrosion [88] than catalysts with a defective graphite lattice support (Pt/EC-G1600) or structurally disordered carbon supports (Pt/EC and Pt/C-JM) during stress testing protocols (Figure 1.8). The graphitized catalyst support displayed the lowest amount of ECSA loss while maintaining the highest performance. Therefore, using a graphitized carbon support can effectively improve the antioxidation capabilities, alleviating the coarsening and agglomeration of Pt nanoparticles. Porous carbon black remains the most widely used catalyst support in today's technology. New carbon materials such as CNTs, graphene, mesoporous carbon, graphite nanopallates, etc and their composites are under investigation for PEM fuel cells. However, there are still gaps and challenges between the properties of today's materials and practical application requirements.

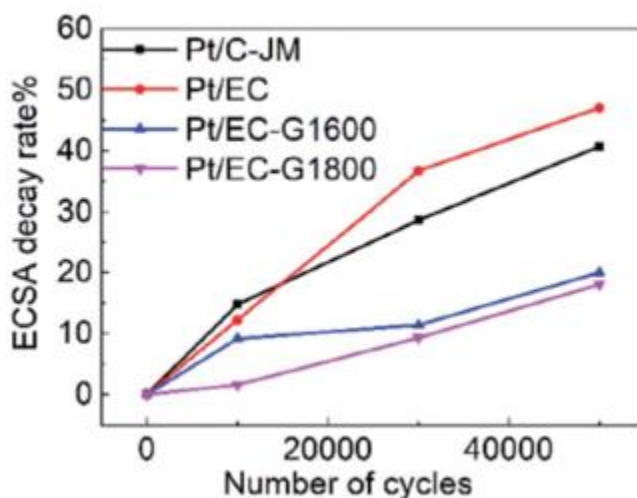


Figure 1.8: The ECSA decay rates of Pt/EC, Pt/EC-G1600, Pt/EC-G1800, and Pt/C from the CV data  
(Reproduced with permission from [88])

### 1.9.3 Graphene based PEMFC catalyst supports

Several studies have highlighted that conventional PEMFC catalyst supports suffer from poor durability due to carbon corrosion and poor Pt anchoring ability [89]. Graphene as a catalyst support has gained attention due to enhanced electrochemical activity and durability in comparison with conventional carbon black supported Pt [90]. In addition, Graphene based catalyst supports offer several benefits including high surface areas, electronic conductivity, electrochemical stability, and the ability to homogeneously distribute uniformly sized catalyst particles [91]. Also, Graphene based carbon supports can reduce Pt loadings in PEMFC catalyst layer by inducing favourable catalyst–support interactions to provide stability and activity enhancements on a platinum-mass basis [92]. However, reduced graphene oxide (rGO) flakes restack together during the Pt deposition step, through  $\pi$ - $\pi$  interaction, blocking fuel (especially  $O_2$ ) diffusion to the Pt catalyst active sites [93].

In another study [94], urea was added to the graphene-based catalyst ink to improve the porosity of the CL. As seen in Figure 1.9, the improved porosity in the CL resulted in improved performance. However, the performance was still subpar compared to conventional Pt/C CL, possibly due to mass transport issues caused by the stacking of graphene.



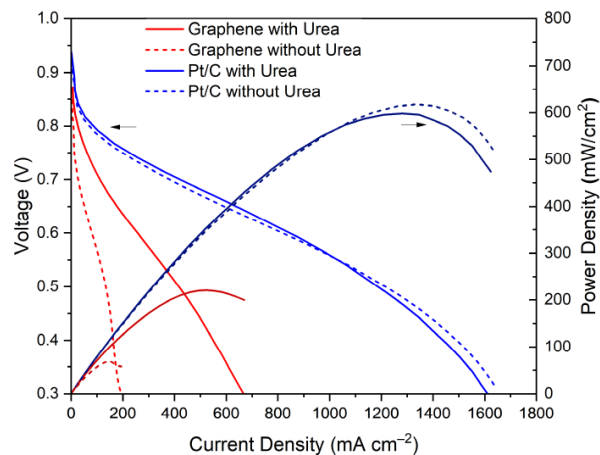


Figure 1.9: Polarisation and power density curves of graphene-based (in red) and commercial carbon black (in blue), tested in H<sub>2</sub>/air (Reproduced with permission from [94]).

To resolve the stacking issue of graphene flakes, a study [95] created a 3-D support structure by mixing different ratios of electrochemically exfoliated graphene oxide (EGO) with CB as Pt catalyst support. As shown in Figure 1.10 (a,b&c), the CL that was made with a rGO:CB ratio of 2:3 (Pt/rEGO<sub>2</sub>-CB<sub>3</sub>) showed 1.8 times performance improvement in a fuel cell setup, activity retention, 71% ECSA retention and excellent durability at 0.60 V after long term operation compared with Pt/CB. This excellent performance was attributed to the 3D structure of the hybrid support mitigating the rGO stacking issue, thereby reducing the CL agglomeration and coarsening.

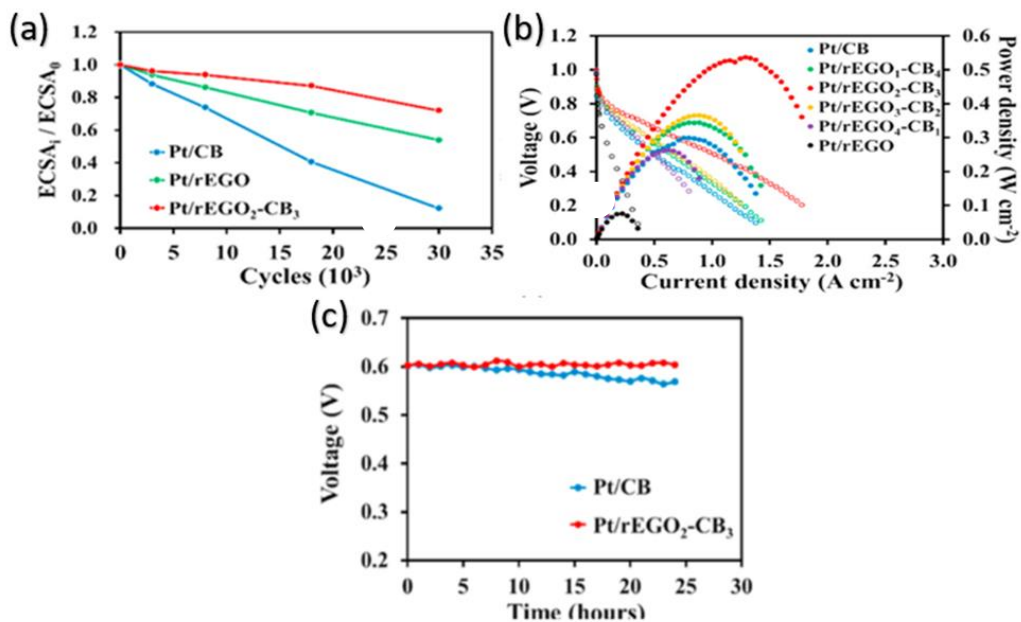


Figure 1.10: a) ECSA comparison after 30000 cycles of AST, b) Performance comparison of CLs made with rGO and CB in different ratios, c) Durability after 24h (Reproduced with permission from [95])

## 1.10 Doped graphene – Why?

Pristine or non-doped graphene does not make a good catalyst support due to the lack of functional groups that anchors the Pt particles. These heteroatom species (S, N, P, etc.) serve as anchors and nucleation sites, facilitating the deposition of uniformly sized and well dispersed Pt particles. Also, the dopant atoms can favourably modify the electronic structure of the Pt particles leading to ORR activity improvements. In addition, strengthened interactions between the doped carbon support and catalyst particles can lead to a strong anchoring effect that can minimize dissolution and Ostwald ripening, which are the two main causes of ECSA loss [96]. In one study [97], the ORR activity and stability of 3 electrocatalysts – Pt/G (Pt supported on graphene), Pt/SG (Pt supported on sulphur doped graphene) and the conventional Pt/C (Pt supported on carbon black), were compared using a liquid electrolyte test. As shown in Figure 1.11, Pt/SG displayed the highest ECSA retention, which is an indicator of durability. Pt/SG also showed the highest specific activity and ORR activity retention.

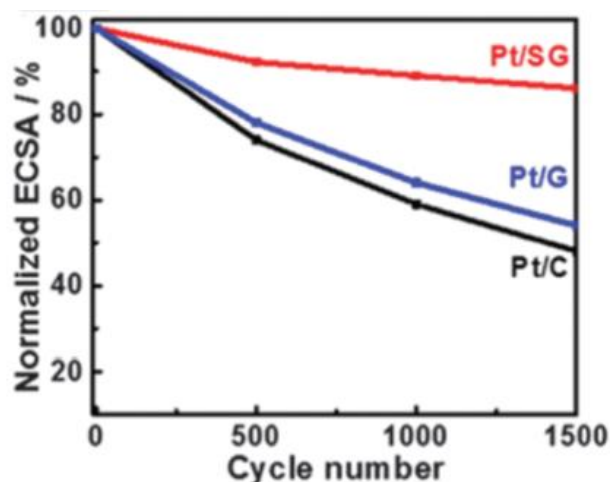


Figure: 1.11: ECSA retained after durability testing (Reproduced with permission from [97])

Similar results were observed in another liquid electrolyte study [98], where the durability and ORR activity retention of Pt/C was compared with Pt NTs (Standalone Pt nanotubes), and Pt NTs/NG (Pt nanotubes supported on nitrogen doped graphene). As shown in Figure 1.12, Pt NTs/NG showed the highest mass activity and ECSA retention and durability testing. However, the reason why the doping of graphene leads to improvements in Pt dispersion, ionomer coverage and durability has only been theorized and is yet to be confirmed empirically. Also, the durability of graphene based CLs needs to be examined using the protocol established by the USDOE.

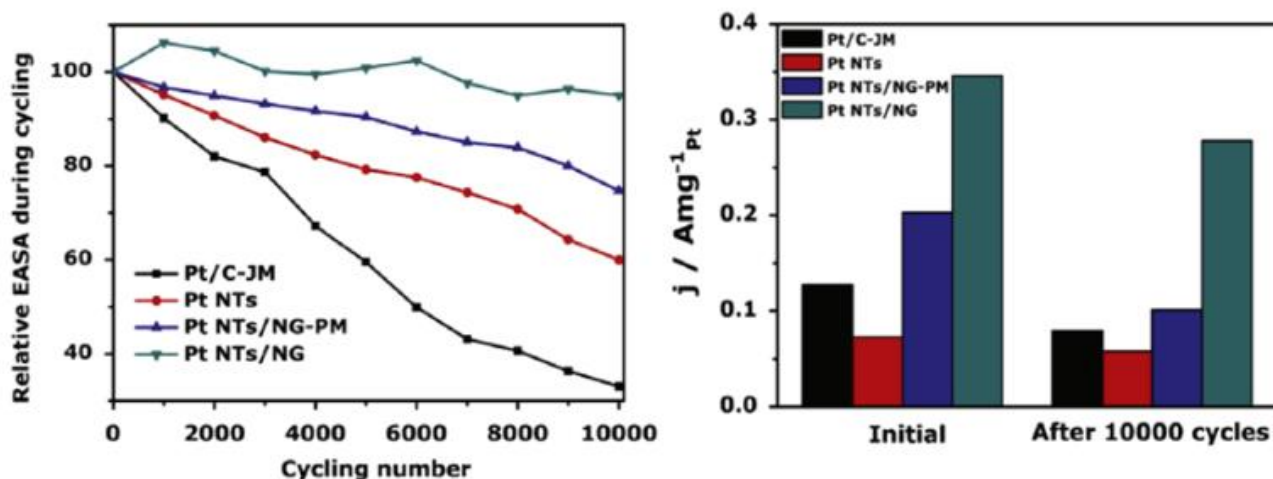


Figure: 1.12: ECSA and mass activity retained after durability testing (Reproduced with permission from [98])

### 1.11 One step electrochemically exfoliated graphene – Why?

Graphene oxide (GO) produced by the Hummers' method, which is costly, has environmental and safety issues and has a long preparation time [99]. Recently, electrochemical exfoliation of graphite, which is fast, environmentally friendly, has a high yield and is easy to scale-up, has attracted intensive research. The lifespan of graphene-based materials can be significantly reduced when they are utilized in high-temperature applications, as carbon may corrode and combust when exposed to such an oxidative environment [100]. Thus, improving the stability of graphene-based materials against oxidation is important to maximize their potential applications. Electrochemically exfoliated graphene has shown to have a higher concentration of functional groups than GO prepared by hummer's method and also a higher thermal stability [101].

This method is a facile, simple, one step, environmentally friendly and scalable method to produce high quality electrochemically exfoliated doped graphene with excellent high-temperature stability [102]. This method involves exfoliation of graphite in inorganic electrolytes (e.g.  $(\text{NH}_4)_2\text{HPO}_4$  and  $(\text{NH}_4)_2\text{SO}_4$  and different combinations of these salts). The electrolyte type and composition were found to have a significant impact on the yield, morphology, structure, and high-temperature stability of the graphene sheets. The nitrogen and phosphorous doped graphene (N, P-G) used in this work was prepared using  $(\text{NH}_4)_2\text{HPO}_4$  electrolyte as it showed the lowest level of defects in comparison to the other electrochemically exfoliated graphene and an exceptionally high-temperature stability in air, at temperatures of up to 750 °C; much higher than typical high

surface area carbon materials which are unstable around 400 °C to 500 °C in air. This improved resistance to corrosion makes it a promising material suitable for PEMFC electrodes.

Electrochemically exfoliated graphene has been synthesized in different types of electrolytes such as ionic liquids, acidic media, and inorganic salts [103]. Among these, inorganic salt has been found to produce graphene with large lateral size and lower amount of oxygen functional groups compared to other types of electrolytes [101].

Finally, the graphene exfoliated in di-ammonium phosphate electrolyte was also found to exhibit a higher degree of crystallinity and fewer defects, due to the phosphorous functional groups binding with diols during the exfoliation process and avoiding further oxidation of the surface.

### **1.12 Research gaps**

There are limited number of studies on graphene-based catalyst layers for fuel cells. Graphene flakes stack due to strong van Der Waals interaction expected to result in a low porosity CL. However, reported work lack in the characterization of catalyst layer properties such as oxygen limiting current which can provide an in-situ evidence of the poor porosity. Similarly, it is not understood how graphene affects the interaction with ionomer in the catalyst ink formulation stage and thereby affecting the catalyst layer ionic resistance. Thus, there is limited understanding of challenges imposed using graphene as catalyst support for PEMFC catalyst layer. Furthermore, there are no prior reports on the effect of co-doping of graphene on Pt catalyst dispersion, ionomer coverage, and durability of support/catalyst has not been reported.

### **1.13 Thesis objective**

The objectives of this thesis are to:

- Establish a baseline of PEMFC properties such as ECSA, CL ionomer protonic resistance, performance, and O<sub>2</sub> transport resistance with a conventionally used Vulcan based CL.
- Study the effect of ionomer equivalent weight, ionomer side chain length and relative humidity (RH) on PEMFC CL properties.
- Use co-doped electrochemically exfoliated graphene as a PEMFC cathode CL and develop a catalyst ink recipe for it.

- Examine the effect of graphitization on the properties of the PEMFC catalyst layers such as ECSA, CL ionomer protonic resistance, performance, durability and O<sub>2</sub> transport resistance.
- Investigate the performance and properties of an alternative cathode catalyst layer architecture and compare it to the conventionally used vulcan carbon based CL.
- Develop a new cathode CL structure that offers enhanced durability, performance, catalyst activity for oxygen reduction reaction and improved local oxygen mass transport in the CL.

## 1.14 Thesis layout

This thesis consists of 4 chapters which have been explained briefly below.

**Chapter 1. General introduction:** The purpose of this chapter is to provide the reader with a detailed literature review and relevant background information of the research in this work. It also outlines the main motivations, overall goals of this research, and the research gaps in the literature. This chapter begins with a detailed review of PEMFC technology followed by a review of the durability issues of the PEMFC. It also explains the mechanism of carbon corrosion and factors affecting ECSA loss in addition to the motivation behind using co-doped electrochemically exfoliated graphene as a PEMFC CL.

**Chapter 2. Experimental methodology:** This chapter explains the experimental methodology followed in Chapters 3 and 4.

**Chapter 3. Effect of ionomer equivalent weight and relative humidity on the PEMFC catalyst layer's interfacial and transport properties:** This section is a published work and it examines the effect of relative humidity and the effect of using two different ionomers of different equivalent weights and side chain length on PEMFC CL properties.

**Chapter 4. Graphene based PEMFC catalyst layer:** This chapter details the experimental methodology of fabricating and testing a graphene based PEMFC CL. It includes the physicochemical and electrochemical characterization of graphene based CL followed by the results. The chapter ends with concluding remarks.

**Chapter 5. Final conclusions and recommendations for future work:** This chapter summarizes the conclusions of chapters 3 and 4 including the knowledge that has been added and the problems that have been identified. This is followed by recommendations for future work.

## Chapter 2: Experimental methodology

### 2.1 Preparation of samples

#### 2.1.1 Synthesis of graphene nanoparticles

Graphene nanoparticles doped with N, P and O functional groups were synthesized by electrochemical exfoliation of graphite foil as described in Sharif et al.[102]. Electrochemical exfoliation of graphite was performed in a two-electrode setup using a stainless-steel plate as the counter electrode (cathode) and a flexible graphite foil (Alfa Aesar) as the working electrode (anode) as shown in Figure 2.1.

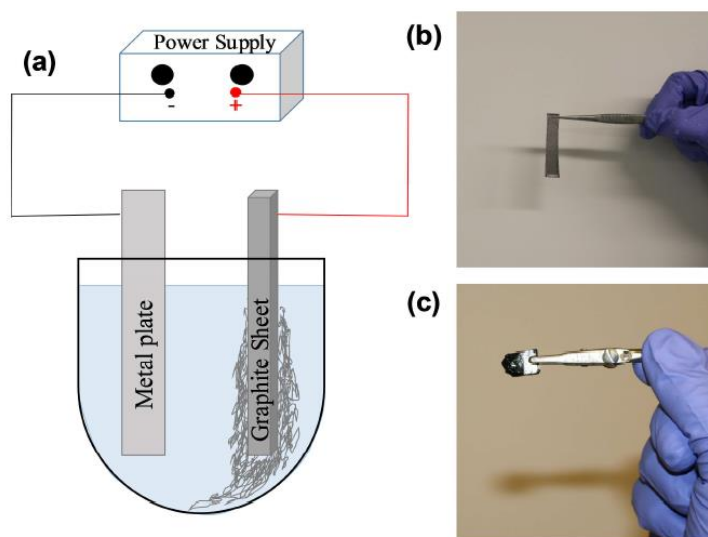


Figure 2.1: (a) Schematic diagram of the synthesis set-up, photographs of graphite electrodes (b) before and (c) after electrochemical exfoliation (Reproduced with permission from [102])

The stainless-steel plate and graphite foil (16 cm<sup>2</sup>) was placed 4 cm apart in 300 ml of electrolyte containing 0.1M diammonium phosphate solution. A constant voltage of 10 V was applied to the cell until the exfoliation was complete and current dropped to 0 A (1.5 hrs). After the electrochemical exfoliation, the resultant product was filtered and washed with DI water using a vacuum filtration setup containing a cellulose membrane. The obtained material was sonicated and dispersed in DI water using a bath sonicator for 15 mins. The graphene solution was then centrifuged at 10000 RPM to separate the unexfoliated graphite. The graphene solution was frozen for 2 days and then freeze dried for another 2-3 days. The result was nanoflakes of graphene oxide co-doped with nitrogen and phosphorus (N, P-G).

### **2.1.2 Pt catalyst deposition on the catalyst support**

200 mg of catalyst support was sonicated for 10 mins with 150 ml of ethylene glycol solution (60% v/v) in a triple-neck round bottom flask. A calculated amount of dihydrogen hexachloroplatinate (V) hydrate (99.99% chloroplatinic acid, Alfa Aesar) was added to the mix and stirred using a magnetic stirrer. The amount of chloroplatinic acid added depends on the Pt loading target. The pH of the dispersion was adjusted to 11 using 0.2 M NaOH solution. The dispersion was then heated to 140 °C using an oil bath-hotplate setup and then stirred for 24h. The dispersion was cooled and then filtered and washed with DI water using a vacuum filtration setup using a cellulose membrane. The obtained material was sonicated and dispersed in DI water using a bath sonicator for 5 mins. The Pt/catalyst support solution was frozen for 2 days and then freeze dried for another 2-3 days resulting in catalyst powder.

## **2.2 Membrane Electrode Assembly (MEA) preparation**

### **2.2.1 Catalyst ink preparation**

The catalyst ink preparation is unique to the type of Pt/catalyst support used. The catalyst ink was prepared using Pt/catalyst support and ionomer. The ionomer to carbon (I/C) and solid to liquid (S/L) ratio was adjusted to the required value. The ionomer stock dispersion was first diluted using a solvent, and the resulting mixture was sonicated for 20-30 mins to break up the ionomer aggregates. Then, 200 mg of Pt/catalyst support was added into the diluted ionomer dispersion using an ice jacket. Then, the mixture was subjected to three hours of magnetic stirring and 48 h of ball milling. The specifics of the catalyst ink preparation protocol are unique to the type of catalyst support used and have been explained in further detail in chapters 3 and 4.

### **3.2.2 Catalyst layer preparation**

Using a doctor blade-equipped automatic film coater (MSK AFA-II), the catalyst ink was coated onto a 75 µm thick ETFE substrate. Alcohol was used to clean the ETFE sheet on both sides, then IPA was used to attach it to the coater's glass plate. It was made sure that the ETFE sheet was free of any dust or air bubbles. Getting a uniform coating is crucial. The wet film thickness of the doctor blade was adjusted to get the right catalyst layer loading. The coating machine's doctor blade was set at a height of 100 µm. The ink was then applied next to the doctor blade using a disposable plastic pipette (touching its surface). No air bubbles were allowed to enter the ink during coating as this could lead to subpar coating. The viscosity of the ink, which is determined by the ratio

of solid to liquid and ionomer to carbon, is one of the most crucial factors for a proper coating. The machine's (MSK AFA-II) coating speed was set to ten. The catalyst-coated decal was then air-dried for 1 hour (shown in Fig 2.2). An excellent coating has an even surface that completely prevents light from going through. Then, using a manual punch, the required decal size was carved out.

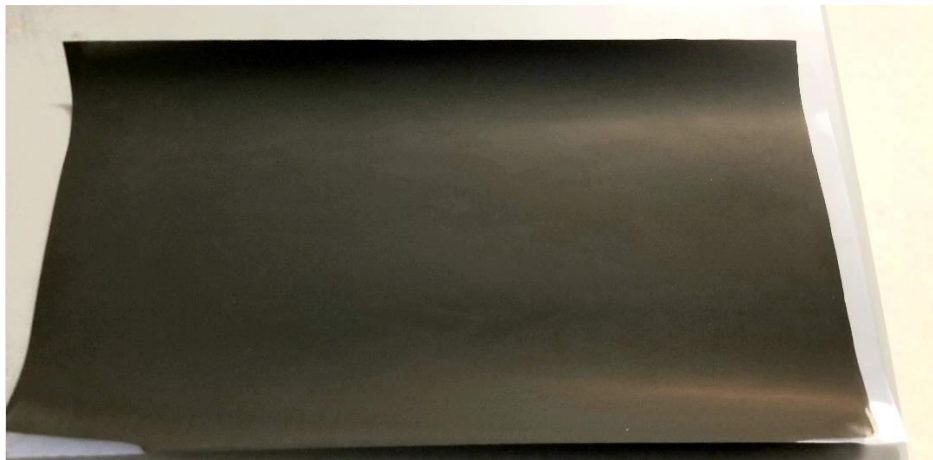


Fig 2.2: Air dried catalyst coated decal prepared using a doctor blade.

### **2.2.3 Membrane Electrode Assembly (MEA) fabrication and cell assembly**

The MEA was fabricated using decal transfer method. Firstly, the catalyst-coated decal was hot-pressed against the Nafion 211 (25  $\mu\text{m}$ , NRE-211) membrane at 150  $^{\circ}\text{C}$  and 2 MPa pressure for 3 minutes to create the catalyst coated membrane (CCM) with the required geometrical active area. A thermostat and digital temperature gauge were used to keep track of the temperature. A polytetrafluoroethylene (PTFE; Teflon) guiding layer with a thickness equivalent to the decal kept the catalyst-coated decals in place. The CCM materials were placed on both sides of a sheet of 50  $\mu\text{m}$  thick PTFE and sandwiched between two metal plates along with a sheet of Pacopad (Pacothane Technologies) that was only around 2 mm thick. The pressing pressure was applied to the CCM using this multilayer system of materials. The layered materials were promptly removed after 3 minutes of pressing and allowed to cool for at least 10 minutes at room temperature. When the assembly had sufficiently cooled, the support materials were carefully removed by hand. Figure 2.3 depicts a schematic of the MEA hot-pressing process. By weighing the decal before and after being hot-pressed, Pt loading in the produced MEA was estimated gravimetrically.



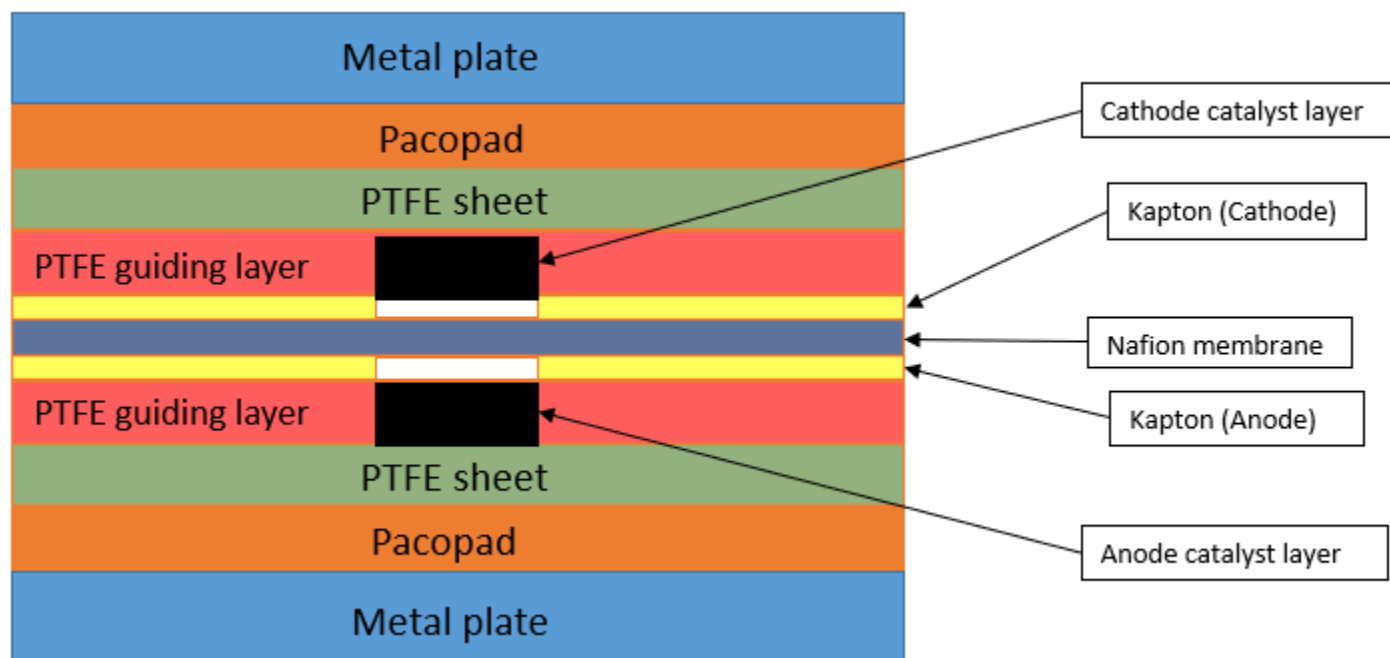


Figure 2.3: Schematic illustration of the hot-pressing process for preparing the catalyst coated membranes (CCMs)

In this work, CCMs with cathode active area of  $1 \text{ cm}^2$  was used. A list of the materials and their sizes that were used to create the CCMs can be found in Table 2.1. Using an electronic cutting tool with a rotary cutting blade, the Silhouette Cameo 3 was used to cut all the materials needed for CCM preparation and cell assembly. A cell was constructed by sandwiching a hot-pressed CCM between two square-shaped gas diffusion layers (GDLs) and a microporous layer (SIGRACET 29BC or TGP H-060, Fuel cell store). The PEMFC hardware had gas flow channels with serpentine pattern. During cell assembly, a torque of 25 in-lb was applied to the hardware. After being put together, the cell was linked to the fuel cell test station (G20, Greenlight Innovation, Canada) along with gas lines, thermocouples, load bank or potentiostat connections, and heating elements to conduct testing. The cells were tested using a potentiostat (Biologic SP-200 or Ivium Vertex) and a fuel cell test station. The protocols for each test as well as cell conditioning are listed in the next chapters.

Active area ( $\text{cm}^2$ )		Membrane (cm)	GDL (cm)	Kapton (cm)	Decal/CL (Circular, $\text{cm}^2$ )		Guiding layer (cm)	GDL gasket (cm)
Cathode	Anode				Ca	An		
1	1.44	7.6 x 7.6	1.5 x 1.5	7.6 x 7.6	1.6	2	10 x 10	10 x 10

Table 2.1: Sizes of materials required for the preparation of CCMs.

## **2.3 Physical and chemical characterizations**

### **2.3.1 Dynamic light scattering (DLS)**

The particle size distribution of graphene samples was analyzed by dynamic light scattering (DLS) using a Malvern Zetasizer Nano Zs. Graphene dispersion was prepared by dispersing graphene in DI water (0.005 wt%). The dispersion was then filled into a disposable sizing cuvette (DTS0012). The refractive index (RI) of the material and dispersant was set at 1.96 and 1.33 respectively and the measurements were taken at 25 °C.

### **2.3.2 Zeta potential measurement**

The zeta potential of graphene samples was analysed using Malvern Zetasizer Nano Zs to study the stability of graphene particles during the platinum deposition step (modified polyol method). Multiple graphene dispersions of different pH values were prepared by dispersing graphene in DI water (0.005 wt%). The pH was varied by adding 0.1M HCl or 0.1M NaOH. The dispersion was then filled into a disposable folded capillary cell or a zeta cell (DTS1070). The Smoluchowski model was chosen for the measurements.

### **2.3.3 Thermo-gravimetric analysis (TGA)**

The weight changes of the materials were measured during thermogravimetric analysis (TGA) in air using a Mettler Toledo TGA instrument at temperatures ranging from room temperature (RT) to 600 °C at a heating rate of 5 °C/min. To remove any remaining moisture, the sample was heated from room temperature to 120 °C and then brought back down to RT before the actual test.

### **2.3.4 Scanning electron microscopy (SEM)**

The surface morphology and Pt distribution of the graphene samples was examined using scanning electron microscopy (SEM) on a Phenom ProX tabletop SEM machine. The mode was set to analysis mode at 15 kV. Intensity was set to 'high' on 'map mode' and resolution was adjusted to 1024 pixels at 4.8 Hz.

### **2.3.5 Energy-dispersive X-ray spectroscopy (EDX)**

The chemical composition of the graphene samples was examined using Energy-dispersive X-ray spectroscopy (EDX) on a Phenom ProX system. The initial settings were same as the SEM measurements. In addition, the quantification settings were set to show oxides and atomic %. The elements chosen were C, O, N, P and Pt.

## 2.4 Electrochemical characterizations

### 2.4.1 MEA conditioning

Prior to testing, all cells were conditioned to hydrate the ionic network, activate the MEAs, and get rid of any potential contamination. The cell was first hydrated in  $\text{H}_2/\text{N}_2$  (0.05/0.1 NLPM) for 8 to 12 hours under an over humidified condition ( $T_{\text{cell}} - 70\text{ }^\circ\text{C}$ ,  $T_{\text{hum}} - 80\text{ }^\circ\text{C}$ ). In order to ensure the quality of the built cell, a set of main diagnostic tests (CV, LSV, and EIS) were conducted beforehand to measure the electrochemical surface area,  $\text{H}_2$  crossover, and series resistance of the cell. After that, the cell was purged with high flow rate  $\text{N}_2$  for 20 minutes to release any remaining surplus moisture. Until the current reached steady state, the cell was maintained at a constant voltage of 0.6 V in  $\text{H}_2/\text{O}_2$  (0.2/0.5 NLPM) at  $70\text{ }^\circ\text{C}$ , 100% RH, and 200 kPag pressure. A typical MEA conditioning profile is shown in Figure 2.4. The primary diagnostic tests were repeated after conditioning the MEA and the cell was purged for another 20 minutes.

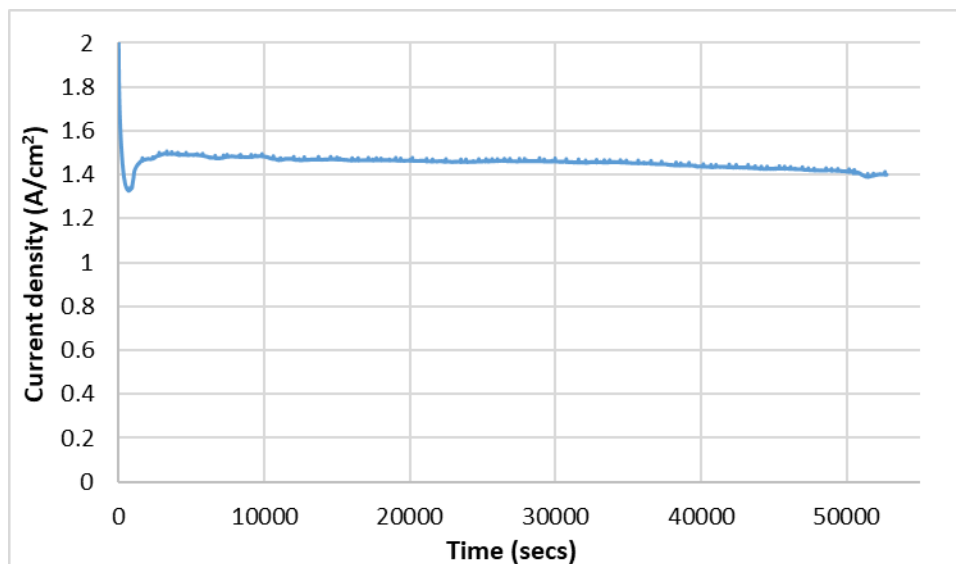


Figure 2.4: Typical conditioning profile of an MEA conditioned at 0.6 V constant voltage hold in  $\text{H}_2/\text{O}_2$  (0.2/0.5 NLPM) at  $70\text{ }^\circ\text{C}$ , 100% RH and 200 kPag pressure.

### 2.4.2 Cyclic voltammetry (CV) and linear sweep voltammetry (LSV)

The  $\text{H}_{\text{desorption}}$  charge (HUPD) and double layer current ( $i_{\text{DL}}$ ) of a CV were used to calculate the electrochemical active surface area (ECSA) and double layer capacitance ( $C_{\text{DL}}$ ) of the catalysts or CL in WE. An example voltammogram is shown in Figure 2.5. CVs were run at a scan rate of 200 mV/s between 0.05 and 1.2 V to assess ECSA. For the ECSA determination of the cathode in MEA, measurements were made in  $\text{H}_2/\text{N}_2$  (0.1/0.2 NLPM) at  $70\text{ }^\circ\text{C}$ , 100% RH, and 50 kPag pressure. ECSA was calculated by integrating the highlighted area of the CV and

taking the capacitive contribution out (Figure 2.5a) (using the same scan rates as above). The integration limitations were set to the double layer capacitance at the high potential limit and the beginning of hydrogen development at the low potential limit (see Figure 2.5a). Equations 2.1 and 2.2 were used to determine the ECSA or roughness factor (RF).

$$RF (cm^2_{Pt}/cm^2_{geo}) = \text{Integrated Area (V. mA cm}^{-2}) / [210 (\mu\text{C cm}^{-2}_{Pt}) \times \text{scan rate (mV s}^{-1}) \times 10^{-3} \times \text{geometrical area (cm}^2_{geo})] \quad (2.1)$$

$$ECSA = RF (cm^2_{Pt} cm^{-2}_{geo}) / \text{Loading (mg}_{Pt} cm^{-2}_{geo}) \quad (2.2)$$

The commonly accepted charge to remove a monolayer of protons on Pt in this instance is 0.210 mC cm<sup>-2</sup><sub>Pt</sub>. The charging and discharging currents in the range of 0.40 - 0.5 V were averaged to get the double layer current.

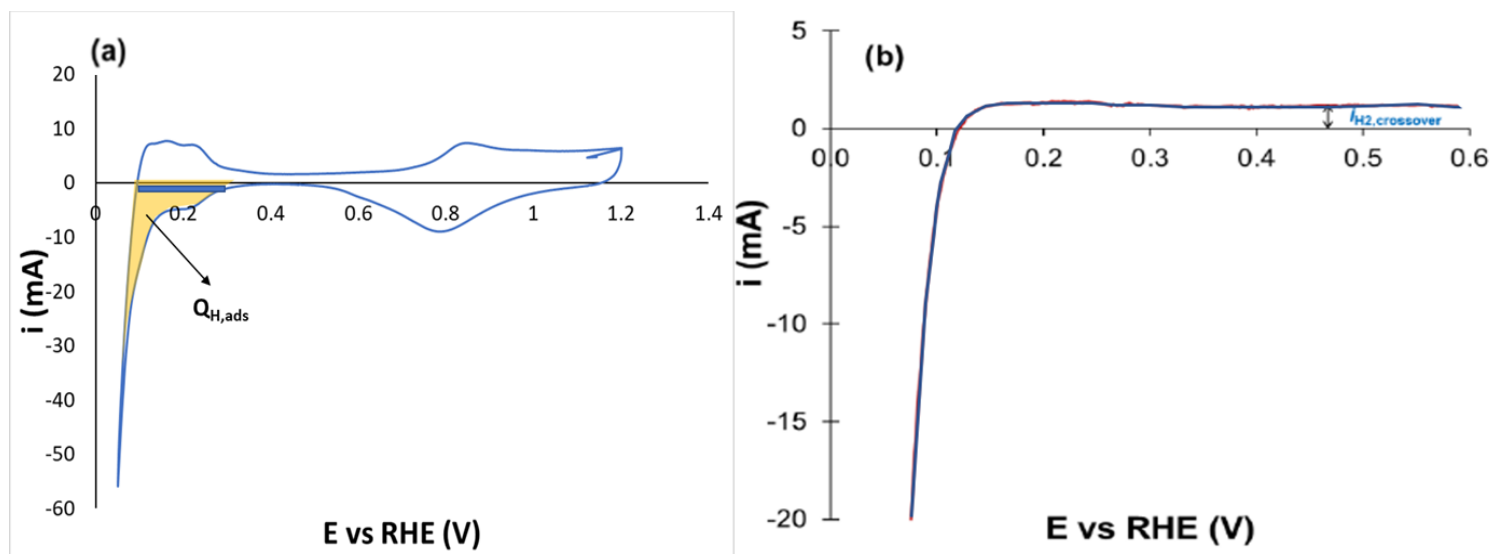


Fig 2.5: a) A standard CV plot used to calculate ECSA using HUPD and double layer capacitance. ECSA and double layer capacitance calculations both used the marked integrated area (stripes) and shaded area (blue). CV was performed in H<sub>2</sub>/N<sub>2</sub> (0.1/0.2 NLPM) at 70 °C, 100% RH, and 50 kPag pressure at a scan rate of 10 mV s<sup>-1</sup>. b) LSV measurements for the same MEA is used to calculate the H<sub>2</sub> gas cross-over. At the same conditions as the CV, LSV was done in the 0.05–0.6 V voltage range at a 5 mV s<sup>-1</sup> scan rate.

The gas phase H<sub>2</sub> crossover current is determined by performing LSV in the voltage range of 0.05 – 0.6 V at the same condition as the CV but at a scan rate of 5 mV/s. The current marked in Figure 2.4b is denoted as crossover current ( $i_{H_2 \text{ x-over}}$ ).

### 2.4.3 Electrochemical impedance spectroscopy (EIS)

During the performance analysis, electrochemical impedance spectroscopy (EIS) measurements were made to estimate the in-situ high frequency resistance (HFR) and the cathode CL ionic resistance ( $R_{H^+,CL}$ ). During the performance measurements, potentiostatic electrochemical impedance spectroscopy (PEIS) was recorded at each voltage point with an AC amplitude of 10 mV in the frequency range of 1 Hz to 1 MHz. Later, iR loss correction was performed using the derived HFR from these data. A previously described method was used to conduct EIS in order to calculate  $R_{H^+,CL}$  [106]. With an AC amplitude of 10 mV at 0.2 V in  $H_2$  (anode)/ $N_2$  (cathode) (0.1/0.2 NLPM) at 70 °C and 50 kPag pressure, EIS for  $R_{H^+,CL}$  determination was carried out in the frequency range of 1 Hz to 1 MHz. To explore the humidity-dependent CL ionic conductivity and account for  $iR_{H^+,CL}$  losses,  $R_{H^+,CL}$  was determined at each RH.

### 2.4.4 PEM fuel cell performance analysis

To reduce gradients in gas concentration and relative humidity, performance measurements were carried out in highly differential flow cells, i.e., at high reactant stoichiometries ( $> 20$ ), as the change in reactant concentration is negligible at such high flows and it allows more precise measurement conditions. During measurements, the pressures were kept under control at the exit with a tolerance of  $\pm 2$  kPa<sub>abs</sub>. Prior to each performance measurement, the operating parameters (cell and humidifier temperatures, pressures, and flows) were maintained for at least 10 mins with a tolerance of  $\pm 1$  °C. With in-situ EIS in voltage-control mode and steady flows of 0.2 NLPM of  $H_2$  on the anode and 0.5 NLPM of  $O_2$  or air on the cathode for 3 minutes at 70 °C, polarisation curves were collected for HFR measurements. The obtained current typically achieves steady state after 3 minutes. The cell was purged with a strong flow of  $N_2$  gas for 10 minutes following each performance measurement to eliminate the produced water, and an HFR monitoring test (by EIS) was then carried out to ensure equilibration.

### 2.4.5 Determination of kinetic parameters

To test oxygen reduction reaction (ORR) performance in  $H_2/O_2$ , current was measured at intervals of 30 mV for 2 minutes in order to determine ORR kinetic characteristics (mass activity, specific activity, and Tafel slopes). The voltage was then normalised to the RF or mass of Pt after being adjusted for iR (HFR) and  $H_2$  crossover losses. After that, the mass activity ( $i_m$ , 0.9 V) and specific activity ( $i_s$ , 0.9 V) were calculated.

### 2.4.6 Limiting current study

The limiting current method created by Baker et al [107] was used to calculate the total gas phase O<sub>2</sub> transport resistance ( $R_{O_2,T}$ ). At 70 °C and 80% RH, limiting current ( $i_{lim}$ ) measurements were made. Differential flow conditions (0.2 NLPM of H<sub>2</sub> and 0.5 NLPM of O<sub>2</sub>:N<sub>2</sub> mixes) were utilised. Two external mass flow controllers are used to change the dry mole fraction of oxygen in the cathode from 2 to 24% O<sub>2</sub> in N<sub>2</sub>. The cell voltage was reduced from 0.4 V to 0.1 V at each test point, and the polarisation curve was examined for a vertical trend close to 0.1 V, which would suggest that a limiting current had been attained. A sample plot for determining limiting current is shown in Figure 2.6 (a). The term " $i_{lim}$ " refers to the current density in the zone of limiting current (depicted in Figure 2.6a). With rising O<sub>2</sub> mole fraction, a typical cell's current density originally increased practically linearly before plateauing. The plateau often denotes the moist area where a sharp rise in  $R_{O_2,T}$  was noted. For the limiting currents with an O<sub>2</sub> mole fraction of 2–6%,  $R_{O_2,T}$  typically remains nearly constant; however, it can vary from cell to cell depending on the operating conditions, electrode materials, and structure. However, at four different total pressures (50, 100, 150 and 200 kPag), limiting currents with O<sub>2</sub> mole fractions of 2 to 6% were recorded. At lower current density and low O<sub>2</sub> concentrations, very little water is generated. Then, using equation 2.3 and the presumption that the oxygen concentration at the Pt active sites is zero,  $R_{O_2,T}$  was calculated from the slopes of the  $i_{lim}$  vs. O<sub>2</sub> concentration plot (see Figure 2.6b). Then, as indicated in the example,  $R_{O_2,T}$  was plotted versus total pressure, and pressure independent  $R_{O_2}$  ( $R_{O_2}^{PI}$ ) was calculated from the Y intercept of this plot (Figure 2.6c).

$$R_{O_2,T} = \frac{4FC_{O_2,ch}}{i_{lim}} = \frac{4F}{slope} = R_{O_2,ch} + R_{O_2,GDL} + R_{O_2,MPL} + R_{O_2,CL} + R_{O_2,ionomer} \quad (2.3)$$

$$C_{O_2,ch} = \frac{x_{O_2,dry}(p_{total} - p_{H_2O})}{RT} \quad (2.4)$$

Here,  $x_{O_2,dry}$  is the dry molar fraction of oxygen in nitrogen,  $p_{total}$  and  $p_{H_2O}$  are the total gas pressure and the partial pressure of water, respectively.  $C_{O_2,ch}$  is the oxygen concentration in the channel.  $F$  is the Faraday constant.  $i_{lim}$  is the limiting current density (A cm<sup>-2</sup>). In equation 2.3,  $R_{O_2,T}$  is made up of the local ionomer resistance ( $R_{O_2,ionomer}$ ), the channel's diffusion resistances ( $R_{O_2,ch}$ , pressure-dependent molecular diffusion), the GDL ( $R_{O_2,GDL}$ , pressure-dependent molecular diffusion), the MPL ( $R_{O_2,MPL}$ , combined molecular and Knudsen diffusion, pressure independent), and the CL ( $R_{O_2,CL}$ , mostly Knudsen diffusion). As per equation 2.5, the interfacial effects at or near the Pt surface are assigned to local ionomer resistance ( $R_{O_2,ionomer}$ ), which is supposed to scale inversely with the normalised Pt area (roughness factor, RF).

$$R_{O_2,ionomer} = \frac{R_{O_2,Pt}}{RF} \quad (2.5)$$

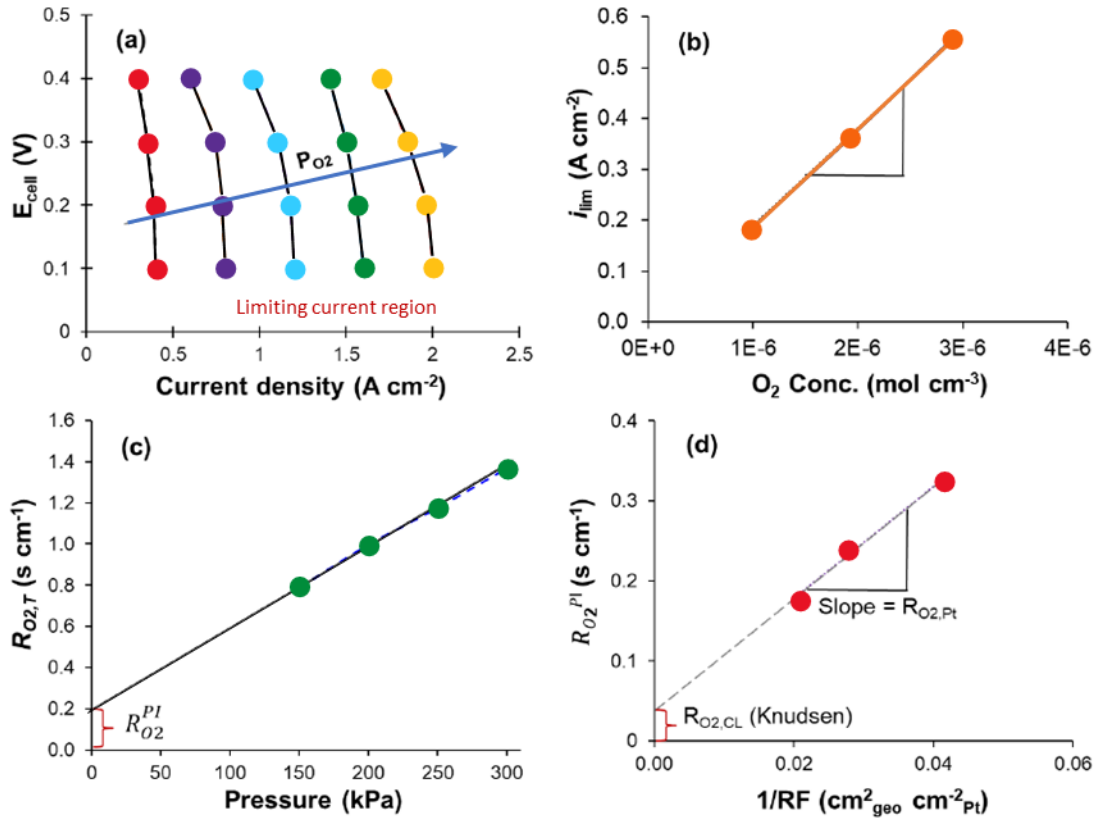


Fig 2.6: Examples of plots related to  $i_{lim}$  and various types of  $R_{O_2}$  determination: (a) Polarization curves at various  $O_2$  concentrations for  $i_{lim}$  determination; (b)  $i_{lim}$  as a function of  $C_{O_2,ch}$  at 50 kPa pressure (plotted by combining  $i_{lim}$  from Figure 2.6a); (c)  $R_{O_2,T}$  (calculated using the slopes obtained from Figure 2.6b at each pressure) as a function of total pressure; and (d)  $R_{O_2}^{Pt}$  as a function of inverse of Pt RF ( $R_{O_2}^{Pt}$  was estimated from the intercept in Figure 2.6c), each data points in this Figure were obtained by performing the analyses shown in Figure 2.6a, b and c for electrodes with varying RF.

It is possible to estimate local Pt resistance ( $R_{O_2,Pt}$ ) and CL Knudsen diffusion resistance ( $R_{O_2,CL}$ ) by altering the Pt roughness factor (RF) while maintaining the same electrode configurations. An example plot of the  $R_{O_2}^{Pt}$  against  $1/RF$  obtained from electrodes with various RF is presented in Figure 2.6(d). The  $R_{O_2,ionomer}$  and  $R_{O_2,CL}$  (Knudsen) are responsible for the slope and Y intercept of this plot, respectively.

# Chapter 3: Effect of ionomer equivalent weight and relative humidity on the PEMFC catalyst layer's interfacial and transport properties

*This chapter has been reproduced from “Transport and Electrochemical Interface Properties of Ionomers in Low-Pt Loading Catalyst Layers: Effect of Ionomer Equivalent Weight and Relative Humidity, Molecules 2020, 25, 3387 (<https://doi.org/10.3390/molecules25153387>)” with open access license from Molecules, MDPI.*

*Note: The references for this chapter in particular have been included separately at the end of this chapter.*

Dr. Muhammad Naoshad Islam (PhD graduate), Dr. Udit Shrivastava (former postdoctoral fellow), Dr. Edward P. L Roberts (Professor, University of Calgary) and Dr. Kunal Karan (Professor, University of Calgary) are the co-authors in the manuscript presented in this Chapter. Apart from training me on MEA fabrication and testing, Dr. Islam and Dr. Shrivastava supervised and assisted me with the electrode fabrication and fuel cell testing mentioned this chapter. they also assisted me in the analyses, interpretation and presentation of the collected data. Furthermore, Dr. Islam, Dr. Shrivastava and Dr. Karan significantly contributed to writing, editing and revising the manuscript mentioned in this Chapter. Furthermore, Dr. Roberts helped me with the editing, revision and also guided me in the work for this manuscript.

The primary goal of the work in this chapter is the study the effect of ionomer equivalent weight, ionomer side chain length and relative humidity on PEMFC catalyst layer interfacial and transport properties like roughness factor, Pt utilization, double layer capacitance, CL protonic conductivity, CL activity for oxygen reduction reaction, and CL oxygen transport resistance.

The secondary goal was to establish a baseline for the aforementioned properties of the conventionally used vulcan carbon based CL which will later be compared to the properties of graphene based CL as described in Chapter 4.



### 3.1 Introduction

Ion-containing polymer or ionomer is a key material in polymer electrolyte fuel cells (PEFCs). Until a decade ago, the focus of studies on ionomeric materials and their fuel cell properties were largely limited to their application as the polymer electrolyte membrane separating the anode and the cathode. However, ionomer is also one of the critical material constituents of the cathode and the anode catalyst layers. Catalyst layers of the polymer electrolyte fuel cells are complex, nanoporous, nanocomposite of ionomer, and Pt/C catalyst with co-continuous phases [1–3]; see Figure 3.1 [3]. In a typical Pt/C-based catalyst layer, a 4–10 nm thin film of acid ionomer covers the aggregates of Pt/C catalyst [4]. The catalyst layer ionomer is often described as a binder [5], which does not capture the multiple crucial functions it serves: (i) as an ion-conducting material phase that ensures transport of protons over 10–20 micron thick catalyst layers, (ii) as an acidic medium that together with Pt catalyst forms the electrochemically active interface where the oxygen reduction reaction (cathode) or hydrogen oxidation reactions (anode) occur, and (iii) as a material phase that controls the transport of reactants ( $O_2$  or  $H_2$ ) and products to/from the active Pt sites.

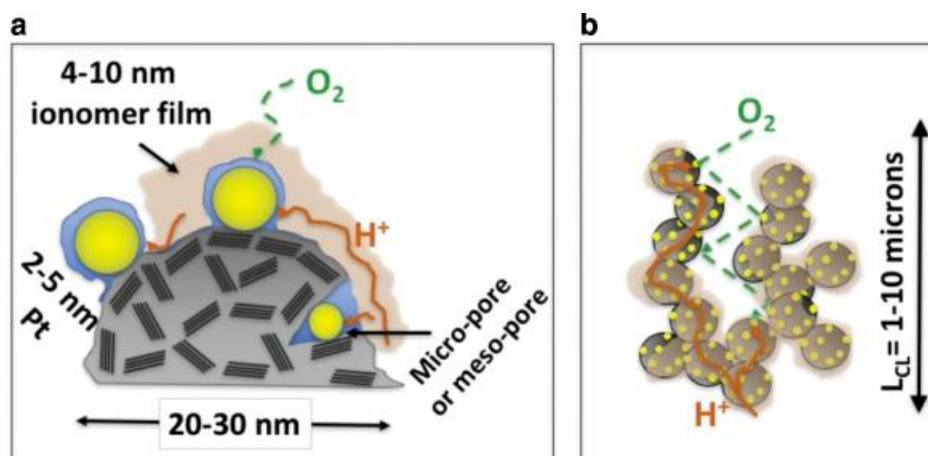


Fig 3.1: Depiction of microstructure of and key processes in a polymer electrolyte fuel cell (PEFC) catalyst layer. (a) Local transport of oxygen and protons to the surface of Pt catalyst particles (yellow) on the surface and in the micropores of carbon support coated with ionomer thin film (b) Long-range transport of proton and oxygen transport through the thickness of the catalyst layer. Reproduced with permission from Karan [3], Current Opinions in Electrochemistry; published by Elsevier, 2017.

The ionomer in a catalyst layer is essential for facilitating proton transport to achieve high in-operando electrochemical surface area (ECSA) utilization [6]. Pt catalysts not in direct contact with ionomer (see Figure 3.1a) may not be accessible to protons under low relative humidity (RH) conditions. Recently, it has been identified that the chemical structure of ionomer dictates the chemical environment of the electrochemically

active interface including poisoning by sulfonic group and ultimately affecting the oxygen reduction reaction (ORR) activity [7].

Oxygen transport through the ionomer thin film covering the catalyst is known to be a limiting factor for low-Pt loading catalysts [8–11]. It is now well established that confinement effects and interactions with substrates (e.g., Pt/C) strongly influence the structure and properties of the ionomer such that it differs significantly from that of the free-standing membrane [12–20]. The proton conduction, the nature of the electrochemical interface, and the local transport of reactants in the catalyst layer ionomer all depend on its hydration state (water content). In addition to bulk water content, the interfacial water content also becomes important for various transport processes and electrochemical phenomena in catalyst layer ionomers. Both bulk and interfacial water content depend on the relative humidity (RH). Exposure to humid environment stimulates the evolution of hydrophilic domains in the bulk ionomer and water sorption at the Pt-ionomer interface [15]. Hydrophilic domains are necessary for proton transport [21] and also assist in oxygen permeation through the thin film [11,22]. Hydration can lead to reorganization of surface, bulk, and buried interfacial structure of ionomers [23,24]. Although discussed sparingly in the literature, it can be expected that interfacial water will influence (a) the extent of acid dissociation, i.e., local pH, and thereby the electrochemical reaction kinetics, (b) the conduction of protons on the catalyst surface, and (c) the transport resistance of oxygen to the Pt catalyst. Moreover, increase in RH enhances the accessibility of Pt catalyst residing in the internal pores of the support and thus affects ECSA [25]. The chemical structure of ionomer and the chemical nature of substrate surface affect the size of the hydrophilic domains in the bulk and at the interface [14,26]. A handful of studies in the literature reports the effect of ionomer type on fuel cell performance [5,27,28]. Even fewer studies discuss the ionomer-dependent relevant catalyst layer electrochemical properties such as ECSA, double-layer capacitance, kinetics, and the local oxygen transport resistance [29–32] and no single study discusses all of these properties for the same catalyst layer. Incorporation of higher ion exchange capacity (IEC) ionomers improve ECSA and specific activity [29] but at the expense of higher local oxygen transport resistance [31]. However, only a few studies have examined the effect of RH on the aforementioned characteristics—ECSA, ORR kinetics, double-layer capacitance, oxygen transport resistance—of the catalyst layer. The literature lacks a systematic account of the combined effect of ionomer structure and RH on the catalyst layer electrochemical properties. In addition, the discussion of the catalyst layer electrochemistry in the context of Pt/ionomer interface is absent. Overall, humidity-dependent probing of catalyst layer properties can provide significant insight into how ionomer molecular structure influence both transport properties and Pt/ionomer interfacial characteristics.

Here, the RH-dependent bulk and interfacial properties of catalyst layer ionomer are reported for long side chain (LSC) Nafion ionomer with an equivalent weight (EW) of ca. 1100 (Naf-1100) and short side chain (SSC) Aquivion ionomer, EW ca. 825 (Aq-825) as a function of relative humidity. Figure 3.2 presents the molecular structure of both ionomers. The method of catalyst layer fabrication and details of experimental measurements are provided in the Materials and Method section.

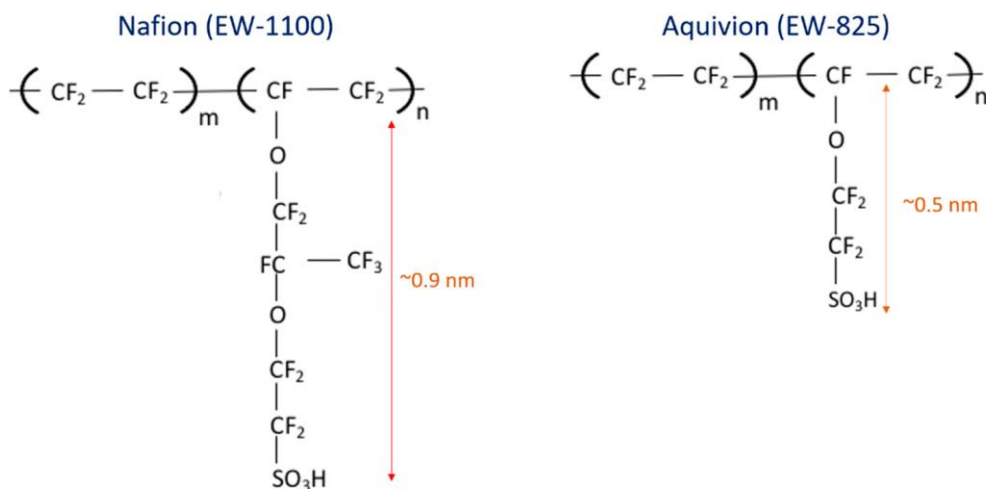


Fig 3.2: Chemical structure of Nafion and Aquivion ionomers.

## 3.2 Results

The RH-dependent electrochemical and transport properties for low-Pt loading catalyst layers (nominal Pt loading of 0.04 mg/cm<sup>2</sup>) prepared with 10 wt% Pt/C catalyst and ionomer:carbon (I:C) ratio of 0.8 for the two different ionomers are reported. I:C was kept at 0.8 to minimise reactant gas transport issues and proton conductivity issues due to poor ionomer coverage in the CL. Electrochemical properties studied include the double-layer capacitance, electrochemically active surface area, and ORR kinetics, while the transport properties studied are the proton conduction and oxygen transport resistance. Double-layer capacitance, electrochemically active surface area, and protonic conductivity: The electrochemically active surface area obtained from H-adsorption peak integration from cyclic voltammetry (CV) scan was normalized with respect to the electrode area and denoted as the roughness factor (RF). Figure 3.3a compares the RH-dependent RF and double-layer capacitance ( $C_{dl}$ ) for the catalyst layers Aq-825 and Naf-1100. RF for both ionomers increases with RH, similar to that reported in other studies [8,25], whereas  $C_{dl}$  almost remains constant for Aq-825 but

increases with RH for Naf-1100.  $C_{dl}$  mainly arises from the interfacial charges at the ionomer/carbon and ionomer/Pt interface. Differences in the catalyst layer microstructure or interfacial characteristics can result in the differences in  $C_{dl}$  for two catalyst layers. If the ionomer coverage on the carbon for the two catalyst layers is different, it would result in different  $C_{dl}$ . If the charge concentration at the ionomer/carbon and ionomer/Pt interface is different, it could also result in different  $C_{dl}$ . The higher magnitude of  $C_{dl}$  for Aq-825 catalyst layer than Naf-1100 catalyst layer points toward either a higher coverage of ionomer in Aq-825 catalyst layer or high interfacial charge concentration than that in Naf-1100 catalyst layer. Although the ionomer to carbon ratio was kept the same for the two catalyst layers and similar coverages are expected, we do not have direct evidence of the microstructural similarity. Thus, the origin of differences in magnitude of  $C_{dl}$  as well as its RH-dependence remains unresolved. Only one previous study that has reported  $C_{dl}$  of catalyst layers with different ionomers [31] could be found. In that study, for catalyst layers made with Nafion and Aquivion ionomers with similar ionomer loading (30 wt%), the  $C_{dl}$  was reported to be 19 mF/cm<sup>2</sup>. However, it was also reported that a catalyst layer with lower ionomer content (10 wt% Aquivion ionomer) had higher  $C_{dl}$  than that for high ionomer content catalyst layer, which is counterintuitive. To the best of our knowledge, no studies have reported a comparison of RH-dependent  $C_{dl}$  of CLs with different ionomers. Thus, the differences in RH-dependent  $C_{dl}$  observed for two ionomers in this study cannot be cross-checked with results from other studies. Moreover, it is not so straightforward to delineate the effect of RH on double-layer capacitance. The double-layer capacitance is known to be pH-dependent and complicated by the contributions from charges in the Helmholtz and diffuse double layer, even in simpler liquid electrolyte/bare metal electrode systems [33]. Double-layer capacitance in such systems usually increases with decreasing pH. However, the system studied here has a solid electrolyte (ionomer), wherein the water content is controlled by RH. The interfacial water will control the interfacial pH, while the bulk water (in the ionomer phase) will control the connectivity of the proton-conducting water channel. Thus, while the interfacial water controls the charge distribution across the solid/polymer interface, the bulk water controls the accessibility of the protons to the interface during potential scanning. Interestingly, for Aq-825, in dry conditions, the CL double-layer capacitance was 1.33 times greater than that of the Naf-1100 CL, which is equal to the ratio of their ion exchange capacities (IECs). At a given RH, the RF for Aq-825 catalyst layer is greater than that of the Naf-1100 catalyst layer. The electrochemical surface area (ECSA) at 100% RH for Aq-825 catalyst layer is 78 m<sup>2</sup>/g<sub>pt</sub>, while that for Naf-1100 catalyst layer is 56 m<sup>2</sup>/g<sub>pt</sub>, indicating higher Pt accessibility in Aq-825 catalyst layer. A previous study also reported a higher Pt utilization for catalyst layer prepared with high IEC (EW 980) Aquivion ionomer than that prepared from low IEC (EW 1100) Nafion ionomer [29]. Both higher IEC and, possibly, higher coverage of Pt/C by ionomer in Aq-825 catalyst layer than in Naf-1100

catalyst layer must contribute to the higher  $C_{dl}$  as well as RF of Aq-825 catalyst layer. The RH-dependent Pt utilization was calculated by dividing ECSA at any RH by the ECSA at 100%RH. The Pt utilization (see inset of Figure 3.3a) of the two catalyst layers at any given RH was very similar, indicating that the type of ionomer does not affect the RH-dependent Pt utilization. For 10 wt% Pt/Vulcan catalyst, Padgett et al. [25] reported that Pt utilization was dependent on RH. From tomography performed on 10 wt% Pt particles, they found that Pt particles located in the internal pores contributed about 20–30% to the total surface area. However, they did not report the double-layer capacitance, and as such a correlation between ECSA accessibility and double-layer capacitance is not available from their study. The observed increase in RF with RH could be due to the increased accessibility of protons to the Pt in the internal pores.

Figure 3.3b shows the variation of the protonic conductivity of Aq-825 and Naf-1100 catalyst layers as a function of humidity. For comparison, the protonic conductivity of Nafion membrane has also been included. The protonic resistances for the catalyst layers and the membrane were determined from analyses of the electrochemical impedance spectra (EIS) at each RH. The inset of Figure 3.3b shows an example of the impedance spectra at 30% RH in  $H_2/N_2$ . Similar impedance spectra were used at each RH to estimate the high-frequency resistance or HFR (marked by point A in inset of Figure 2b) and the catalyst layer protonic resistance  $R_{CL}$ . The high-frequency resistance is the sum of the electrical and membrane protonic resistances, and Equation (3.1) below was used to calculate the membrane protonic conductivity.

$$\sigma_{mem} = \frac{t_{mem}}{HFR - R_e}$$

Equation 3.1

where  $\sigma_{mem}$  is the membrane conductivity,  $R_e$  is area-specific electrical resistance ( $10 \text{ m}\Omega \text{ cm}^2$ ) determined from an ex-situ resistance measurement, and  $t_{mem}$  is the thickness of the membrane. In a typical EIS of a fuel cell catalyst layer, the point along the  $45^\circ$  line at which spectra transition to a completely capacitive behavior, for example, point B in the inset, the real component of spectra at this point is summation of HFR and a third of  $R_{CL}$  [34]. Using equation 3.2 (see below), the catalyst layer conductivity is obtained [35].

$$\sigma_{CL} = \frac{t_{CL}}{R_{CL}\epsilon^n}$$

Equation 3.2

where  $\sigma_{CL}$  is catalyst layer conductivity,  $\epsilon$  is ionomer volume fraction, the Bruggeman exponent  $n$  is 1.5 [34], and  $t_{CL}$  is thickness of the catalyst layer.

Expectedly, protonic conductivity of catalyst layer and membrane are strong functions of RH. At 30% RH, both Aq-825 and Naf-1100 catalyst layer conductivities are almost an order of magnitude smaller than the Nafion membrane conductivity, and at any given RH, Naf-1100 catalyst layer conductivity is smaller than the Aq-825 conductivity. In a catalyst layer, ionomer exists in thin film form (depiction shown in Figure 1) and it is known that at comparable RH, ionomer thin film exhibits much lower protonic conductivity than the bulk membrane [13]. The IEC of Aq-825 ionomer (IEC  $\sim 1.2$ ) is higher than that for Naf-1100 ionomer (IEC  $\sim 0.9$ ). The difference in conductivity can be attributed to the intrinsic effect of difference in acid content of the ionomers and to the extrinsic effect of how ionomer is spatially distributed in the catalyst layer. The former effect is well known for bulk membranes, while the latter effect (differences in microstructure) is complicated. For example, lower coverage of ionomer would imply thicker ionomer films that have higher conductivity, but the connectivity and tortuosity may also be higher.

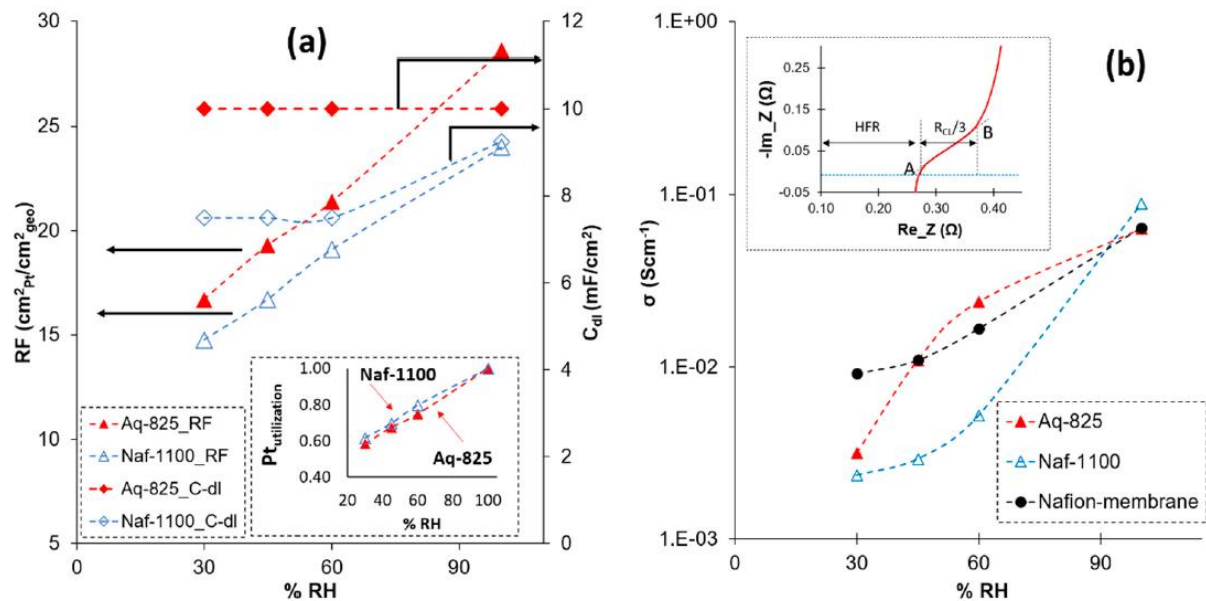


Figure 3.3: Comparison among relative humidity (RH)-dependent properties of Naf-1100 and Aq-825 derived from cyclic voltammetry (CV) and electrochemical impedance spectra (EIS) in  $\text{H}_2/\text{N}_2$  at  $70^\circ\text{C}$  and 100% RH **a)** roughness factor and double-layer capacitance **b)** protonic conductivity.

### 3.2.1. Oxygen Reduction Reaction (ORR) Kinetics

Little is understood about the influence of ionomer EW and side chain on the ORR kinetics. Ionomer EW or IEC is a measure of its acidic strength. From early studies of ORR on Pt electrodes in liquid electrolyte, Damjanovic and Brusic proposed the following kinetic expression highlighting the dependency of ORR kinetic current ( $i_{\text{ORR}}$ ) on proton activity/concentration [36]:

$$i_{ORR} = k P_{O_2}^n [H^+]^m \exp\left(-\frac{\alpha F}{RT} \eta_{ORR}\right)$$

Equation 3.3

where  $k$  is the electrochemical rate constant akin to exchange current density,  $n$  and  $m$  are reaction order stated to be 1 and 1.5 [36],  $\alpha$  is transfer coefficient,  $p_{O_2}$  is oxygen partial pressure,  $\eta_{ORR}$  is ORR overpotential,  $F$  is Faraday constant,  $R$  is universal gas constant, and  $T$  is temperature. It must be noted that in the original work [33], the formal potential rather than overpotential was used. The interfacial proton concentration in the catalyst layer, i.e., proton concentration at the Pt/ionomer interface, would depend on the abundance of sulfonic acid groups at or near the Pt/ionomer interface as well as the interfacial water content, both of which are not directly accessible in fuel cell experiments. From our neutron reflectometry (NR) study of different ionomers on planar Pt, it is known that the amounts of water present at the Pt/ionomer interface varies with RH and at 100% RH the water content correlates to the ionomer side-chain length [12,14]. However, the abundance of sulfonic group is not known. CO desorption electrochemistry applied to estimate Pt-sulfonic group interactions could offer insight into this but was not applied in the current study [37]. Regardless, it can be expected that the proton concentration at the Pt/ionomer interface would vary with RH and could be different for different ionomers. Accordingly, proton concentration-dependent ORR kinetics (Equation 3.3) can be expected to occur. A comparison of RH-dependent ORR kinetic behavior of Aq-825 and of Naf-1100 catalyst layers is presented in Figure 3.4. By neglecting oxygen transport resistance and hydrogen oxidation reaction overpotential, the  $\eta_{ORR}$  was estimated using Equation (3.4) below and plotted against the log of specific current density ( $i_s$ , A cm<sup>-2</sup> Pt), see Figure 3.4(a1–a4), clearly following the Tafel behavior.

$$\eta_{ORR} = OCV - E_{cell} - i (HFR + R_{CL}/3)$$

Equation 3.4

where OCV is open-circuit voltage,  $i$  is current density, and  $E_{cell}$  is cell voltage.

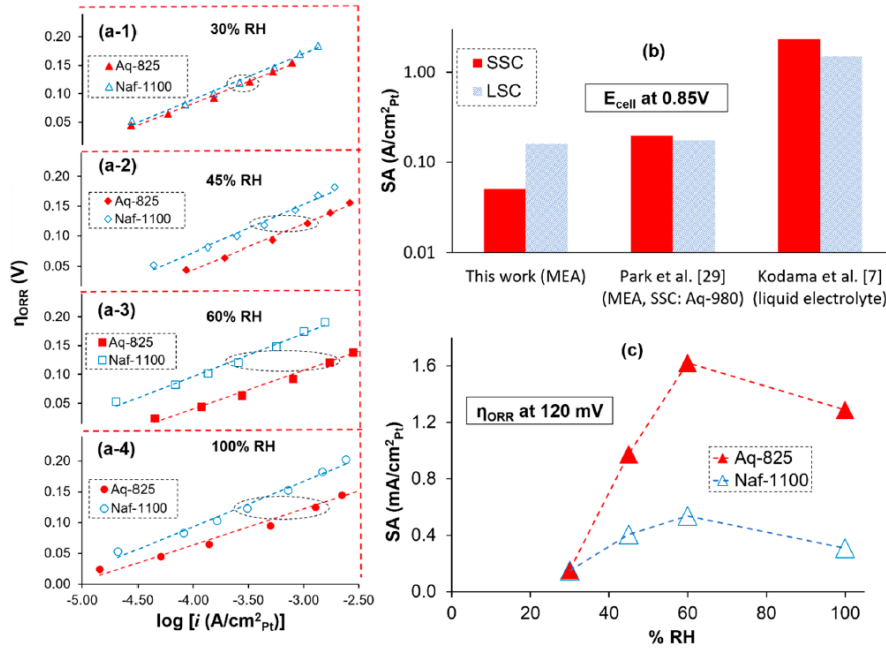


Figure 3.4: Comparison among kinetic properties of Naf-1100 and Aq-825 in H<sub>2</sub>/air **a)** activation overpotential against current density in Tafel region at different RHs **b)** specific activity at 0.85 V from this work, park et al. [29], Kodama et al. [7] in liquid electrolyte **c)** specific activity at activation overpotential of 120 mV and at different RHs.

For most electrochemical reactions, two kinetic parameters are often considered—the Tafel slope, which is related to the transfer coefficient ( $\alpha$ ), and the exchange current density. From Figure 3.4a, it can be noted that the slopes of the plot, which is the Tafel slope, are similar for both catalyst layers at each RH. The Tafel slopes were found to be 66–70 mV/decade. It is also obvious from the data in Figure 3.4a that at each RH, ORR current in the Aq-825 catalyst layer is superior to that in the Naf-1100 catalyst layer. Since Tafel slope (or  $\alpha$ ) are similar, considering the Damjanovic and Brusic formulation of ORR kinetics [36], it can be deduced that higher current density (expressed on a per  $\text{cm}^2$  of Pt basis) for Aq-825 catalyst layer compared to that for Naf-1100 catalyst layer would be a result of higher interfacial concentration of protons at Pt/ionomer interface for Aq-825 catalyst layer. The higher IEC of Aq-825 and its shorter side chain can be expected to create a higher abundance of sulfonic groups at the interface and, thereby, in a higher interfacial proton concentration. To the best of our knowledge, there has been no prior study comparing RH-dependent ORR kinetics for catalyst layers with different ionomers. In literature, the kinetic performance of a catalyst layer is generally evaluated by defining specific activity or mass activity at a voltage in kinetic or Tafel region of the polarization curve. In Figure 3.4b, the specific current density, i.e., specific activities (SA) for ORR at 0.85 V for catalyst layers made with short and long side chain (SSC and LSC) ionomers in this work are compared along with results from other studies for SA determined in membrane electrode assembly (MEA) [29] and in liquid electrolyte [7]. In MEAs, regardless of the



type of ionomer used in fabricating the catalyst layer, SA at 0.85 V in MEA can be as much as ten times lower than that in liquid electrolyte (rotating disc electrode or RDE setup) [7]. In the present work, SA at 0.85 V in 100% RH for Aq-825 catalyst layer is lower than that for Naf-1100 catalyst layer. Since OCV for the Aq-825 catalyst layer at 100% RH is 40 mV lower than the Naf-1100 catalyst layer, as per Equation (4),  $\eta_{\text{ORR}}$  for both cells are different at same  $E_{\text{cell}}$  and, hence, comparison of SA at same voltage may not be valid. Therefore, SA in Figure 3.4c is compared at a similar  $\eta_{\text{ORR}} = 120$  mV marked by the dotted ellipses in Figure 3.4a. Except at 30% RH, at each RH, SA for Aq-825 catalyst layer is 2–5 times greater than Naf-1100 catalyst layer. For both catalyst layers, SA increased with RH. From 30% RH to 60% RH, SA for Aq-825 SA catalyst layer increased ten folds whereas for Naf-1100 SA increased by three times. Then, for both catalyst layers between 60% and 100%, RH SA decreases—trend is similar to a previous study [29].

### 3.2.2. Oxygen Transport Resistance

Limiting current technique explained elsewhere [38] was employed to determine the oxygen transport resistance ( $R_{\text{O}_2,T}$ , s/cm) via Equation 3.5 below.

$$R_{\text{O}_2,T} = \frac{4FC_{\text{O}_2, \text{ch}}}{i_{\text{lim}}} = \underbrace{[R_{\text{O}_2, \text{GDL}} + R_{\text{O}_2, \text{MPL}}^{\text{M}}]}_{\text{Pressure dependent}} + \underbrace{[R_{\text{O}_2, \text{MPL}}^{\text{Kn}} + R_{\text{O}_2, \text{CL}}]}_{\text{Pressure independent}}$$

Equation 3.5

where  $C_{\text{O}_2, \text{ch}}$  is gas concentration at the channel,  $i_{\text{lim}}$  is limiting current density,  $F$  is Faraday constant.  $R_{\text{O}_2,T}$  is a combination of the pressure-dependent and -independent terms. In gas diffusion layer (GDL), pore size is in order of 1–10  $\mu\text{m}$  [39] in microporous layer (MPL) pore size varies from ~50 nm to 1  $\mu\text{m}$  [39,40], and in catalyst layer pore sizes are below 100 nm [41]. Therefore, oxygen transport through the gas diffusion layer and some of the pores of MPL occurs via molecular diffusion and the associated transport resistances are denoted as  $R_{\text{O}_2, \text{GDL}}$  and  $R_{\text{O}_2, \text{MPL}}^{\text{M}}$ , respectively. Transport through smaller pores in MPL occurs via pressure-independent Knudsen diffusion. The associated oxygen transport resistance is denoted as  $R_{\text{O}_2, \text{MPL}}^{\text{Kn}}$ . In addition to the GDL and MPL, catalyst layer offers additional pressure-independent resistance  $R_{\text{O}_2, \text{CL}}$ , which includes Knudsen oxygen diffusion resistance as well as oxygen permeation resistance through the thin ionomer film coating of the Pt particles. The inset in Figure 3.5 shows an example of the linear increase in  $R_{\text{O}_2,T}$  with the pressure. The slope of this line is inversely proportional to the molecular diffusion resistance in GDL and MPL, the pressure-

dependent terms, and intercept represents the pressure-independent terms. From our internal study, we estimated  $R^{Kn}O_{2,MPL}$  is 0.1 s/cm, which is very small compared to the intercept of  $R_{O_2,T} \sim 1.5$  s/cm. The catalyst layer thickness is determined to be in the 10–12  $\mu\text{m}$  range. Considering the Vulcan carbon support, most of the Pt catalysts are expected to be on the surface of the carbon particle. Thus, Knudsen diffusion within the micropores of carbon support as expected for high surface area carbon can be neglected for the present study wherein Vulcan carbon support has been used. A mix of Knudsen and molecular diffusion through the macropores of catalyst layer is expected. In our analyses, the gas phase  $O_2$  transport resistance is considered to be significantly smaller than local transport resistance. Hence, the magnitude of intercept in the inset mainly corresponds to the  $R_{O_2,CL}$ . Accordingly, the average local transport resistance to Pt/ionomer interface ( $R_{O_2,Pt}$ ) can be approximated via Equation 3.6 as [9]:

$$R_{O_2,Pt} = R_{O_2,CL} \cdot RF$$

Equation 3.6

$R_{O_2,Pt}$  in both catalyst layers decrease significantly with RH. Between 30% and 80% RH, Aq-825 exhibits 30% reduction in  $R_{O_2,Pt}$  whereas Naf-1100 shows 50% reduction in  $R_{O_2,Pt}$ . The magnitude of  $R_{O_2,Pt}$  for Naf-1100 catalyst layer is similar to the reported values in the literature [9,22]. At each RH,  $R_{O_2,Pt}$  for Naf-1100 catalyst layer is lower than that for Aq-825 catalyst layer, e.g., at 80% RH  $R_{O_2,Pt}$  of Aq-825 catalyst layer is close to 40% higher than  $R_{O_2,Pt}$  for Naf-1100 catalyst layer. Ono et al. also reported higher local  $R_{O_2,Pt}$  for higher IEC ionomers than lower IEC ionomers [31]. RH dependency of  $R_{O_2,Pt}$  follows the trend reported by the Toyota group in a study that is the only known direct measurement of oxygen transport resistance of ionomer on Pt [22]. Hydrated ionomer promotes oxygen transport through the ionomer, while the side-chain interactions with Pt likely influences the ionomer thin film morphology, especially the interfacial structure.

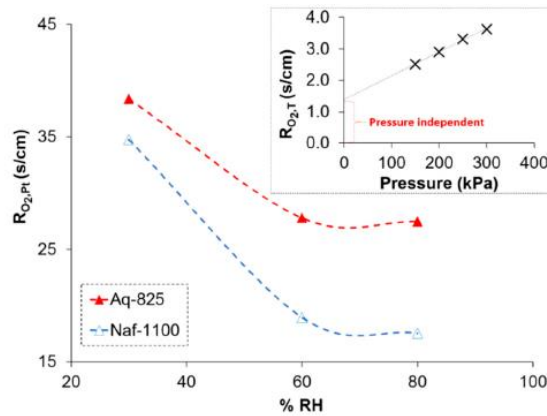


Figure 3.5: RH-dependent local ionomer transport resistance for both Naf-1100 and Aq-825 catalyst layers estimated at 70 °C. Inset shows total transport resistance as a function of pressure.

### 3.3 Discussion

In a majority of the prior studies, a key stated motivation of preparing fuel cell catalyst layers with higher IEC ionomer such as Aq-825 is to achieve higher protonic conductance within the catalyst layer (see e.g., Park et al. [29]). However, interfacial processes, whether ORR kinetics or local  $R_{O_2, Pt}$ , can also be affected by the ionomer. The higher conductivity of catalyst layer prepared with high IEC ionomer (Aq-825) than that of catalyst layer prepared with low IEC ionomer (Naf-1100) is evident in our study (Figure 3.3b) and other works [31]. Additionally, our work indicates that the ionomer in a catalyst layer also affects the ORR electrochemical kinetics, an aspect previously not investigated in depth in other studies. Consistent with previous findings, a significant influence of CL ionomer on microstructure-dependent properties (i) Pt utilization (Figure 3.3a) and (ii) local oxygen transport (Figure 3.5) is also noted. Our study elucidates the RH dependency of these properties, which has been investigated to a limited extent. A key point we would like to emphasize is that the interfacial properties are significantly affected by the nature of Pt-ionomer interface. In a previous neutron reflectometry (NR) study from our group [12,14], we examined the temperature- and RH-dependent bulk and interfacial water distribution in 15 nm ionomer films on planar Pt substrates for different ionomers including 3M EW-725 (SSC) and Nafion-1100 (LSC). Based on these findings, Figure 3.6 depicts the Pt/Aq-825 and Pt/Naf-1100 interfaces [12,14,42]. Although 3M-725 is a different ionomer from Aq-825, both of them have same backbone and similar side chain length, the only difference is spacing between the ionic groups. At 97% RH, Pt/Aq-825 interface may have only a monolayer of water (3Å) separating Pt surface from the polytetrafluoroethylene (PTFE) backbone. In same condition, two monolayers thick water (6Å) is formed at Pt/ionomer interface. In both cases, sulfonic groups are shown to interact with Pt surface (based on IR [43]), and hydrophobic back is separated by hydrophilic domains (based on GISAXS [15]).

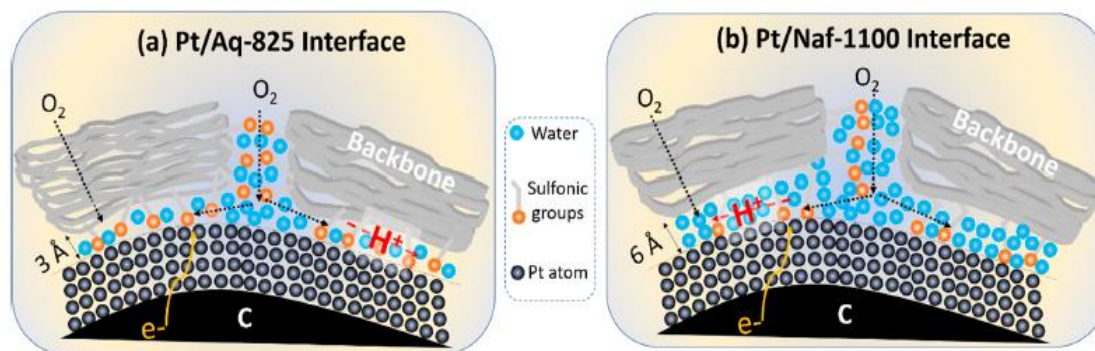


Figure 3.6: Depiction of Pt/ionomer interface based on neutron reflectometry (NR) at 97%RH (a) Pt/Aq-825 interface (b) Pt/Naf-1100 interface.

### 3.3.1. Pt Utilization

At each RH investigated in our study, Aq-825 catalyst layer exhibited 1.4 times higher ECSA than Naf-1100 catalyst layer. Pt utilization is essentially a quantification of accessibility of protons to the Pt catalysts. Ionomer coverage and poisoning effect are two factors that can affect the Pt utilization. Naf-1100 has a longer side chain with two ether groups, while Aquivion-825 has a shorter side chain with only one ether group. The combination of RDE and surface-enhanced infrared absorption spectroscopy provides evidence of absorption of the oxygen atom of flexible LSC ether group on Pt atom. Such absorption of oxygen atom of SSC ether group on Pt atom is absent. Thus, a higher blockage of Pt sites is observed in catalyst layer made with LSC ionomer (Nafion) compared to that made with SSC ionomer (Aq-825), resulting in a lower ECSA for Naf-1100 catalyst layer [7]. In absence of any microstructural characterization, we cannot ascertain to what extent ionomer coverage differences contribute to the Pt utilization differences.

### 3.3.2. ORR Kinetics

As discussed earlier, the differences in the Pt-area normalized kinetic current for the two catalyst layers at a given overpotential is attributed to the interfacial proton concentration. As discussed above, the interfacial water and the sulfonic acid abundance at the interface both will affect the interfacial proton concentration. At low RH (30% RH), the ORR kinetic currents for Aq-825 and Naf-1100 catalyst layers are similar. At this RH, the interfacial water content is expected to be similar for both CLs. It would then appear that there is very little difference in sulfonic acid abundance at the Pt/ionomer interface for the two CLs. At higher RH, significant differences are observed. As depicted in Figure 3.5 above, there will be higher amount of water at the Pt/ionomer interface in the Naf-1100 CLs. This would be tantamount to diluting the sulfonic acid by different amounts of water, effectively lowering the interfacial proton concentration. Using Equation (3.7) below, ratio of proton concentration is estimated to be ~3, and the kinetic current of Aq-825 is nearly 4 times greater than Naf-1100.

$$C_{H^+, interface} \propto \frac{IEC}{\phi_{w, interface}}$$

Equation 3.7

where  $C_{H^+, interface}$  is the interfacial proton concentration and  $\phi_{w, interface}$  is the interfacial water volume fraction.

### 3.3.3. Oxygen transport resistance

The oxygen transport to the Pt in a catalyst layer comprises of gas-phase transport (mix of Knudsen and molecular diffusion depending on the local pore dimension), transfer from gas-phase to the ionomer phase, diffusion through the ionomer film, and then additional interfacial resistance. For high surface area carbon and agglomerated Pt/C structure, diffusion through the micropores within carbon and pores inside the agglomerate structure, respectively, would also have to be considered. At low Pt loadings, ionomer thin films in the catalyst layer thought to be large contributors to the local oxygen transport resistance [9,11]. The local ionomer thin film resistance for both catalyst layers significantly decreases with RH similar to reported by an ex-situ oxygen transport study on Nafion thin film on Pt [22]. Within the ionomer thin films, oxygen has two transport pathways: (a) through the free volume within the hydrophobic matrix, and (b) via the water-filled hydrophilic domains. Oxygen has relatively high solubility in the hydrophobic Teflon-like matrix but restrictive diffusion. On the other hand, oxygen can be solvated in the water phase of hydrophilic domains and be diffused with ease. As RH increases, both the bulk and interfacial water content increases, thereby making the oxygen transport more facile [11,22]. At same RH, water volume fraction in bulk of Aq-825 ionomer film is expected to be higher than that Naf-1100 ionomer film. However, as depicted in Figure 3.6, the interfacial water layer thickness for Naf-1100 is almost two times the thickness of Aq-825 interfacial layer. The local oxygen transport resistance ( $R_{O_2,Pt}$ ) at 80% RH for the Aq-825 catalyst layer is 1.6 times higher than that for the Naf-1100 catalyst layer. If the oxygen transport through the bulk portion of the ionomer film rather than near interface region was the dominant resistance, Aq-825 catalyst layer would not exhibit higher  $R_{O_2}$  than Naf-1100 catalyst layer. It is hypothesized that the higher interfacial water content at the Pt/ionomer interface in the Naf-1100 catalyst layer promotes faster oxygen transport than that in the Pt/Aq-825 catalyst layer with low interfacial water content.

## 3.4 Materials and methods

### 3.4.1 Catalyst ink preparation

The catalyst inks were prepared using commercially available 10.2 wt% Pt/C (Tanaka) electrocatalyst and Nafion (EW 1100) purchased from Ion power Inc. (New Castle, DE, USA) and Aquivion (EW 825) purchased from Sigma Aldrich (St. Louis, MO, USA). The ionomer to carbon (I/C) and solid to liquid (S/L) ratio was maintained at 0.8 and 0.1, respectively. The ionomer stock dispersion was first diluted using a mixture of deionized (DI) water

and isopropanol, and the resulting mixture was probe-sonicated for 2 min using an ice jacket to break up the ionomer aggregates. Then, 285 mg of Pt/C catalyst was added into the diluted ionomer dispersion. Then, the mixture was bath sonicated for 15 min followed by three hours of magnetic stirring and 24 h of ball milling.

### 3.4.2 Membrane Electrode Assembly (MEA) Preparation

The catalyst layer decals were prepared by coating the catalyst inks on ethylene tetrafluoroethylene (ETFE) sheet using a micrometer adjustable film casting doctor blade (EQ-Se-KTQ-250, MTI corporation, Richmond, CA, USA) and air dried for 24 h. The doctor blade was adjusted to a thickness of 100  $\mu\text{m}$  for coating of both catalyst layer decals. Decal transfer method was used to prepare an MEA. Nafion membrane (25  $\mu\text{m}$ , NRE-211, DuPont, Wilmington, DE, USA) was hot-pressed in between the anode (0.2  $\text{mg}_{\text{Pt}}/\text{cm}^2$ ) and the cathode decals at 150°C and 2 MPa pressure for 3 min [44,45]. The MEAs had an anode and cathode active area of 1.44 and 1  $\text{cm}^2$ , respectively. The larger anode area was maintained to ensure redundant supply of reactant (protons) from the anode. The thickness, platinum loading, and ionomer loading of the cathode CLs are listed in Table 1. The changes in local and interfacial transport properties become very insignificant and remain obscured at high cathode Pt loading as it scales inversely with Pt ECSA, thus CLs with ultra-low Pt loading were prepared in this study.

**Table 1.** Summary of the cathode catalyst layer properties.

Catalyst Layer Type	CL Thickness ( $\mu\text{m}$ )	Pt Loading ( $\text{mg}_{\text{Pt}}/\text{cm}^2_{\text{geo}}$ )	Ionomer Loading (wt%)	Ionomer Loading ( $\text{mg}/\text{cm}^2_{\text{geo}}$ )
Nafion	11 $\pm$ 1	0.043	42.07	0.34
Aquivion	10 $\pm$ 1	0.036	42.04	0.28

### 3.4.3 Fuel Cell Assembly and Testing

A small scale and differential flow field cell was assembled by sandwiching the hot-pressed MEA between 160  $\mu\text{m}$  thick Toray gas diffusion layers coated with microporous layer (MPL) (TGP H-60, Toray, Japan). The differential cell was used to avoid any discrepancies arising from the gradient in RH and reactant gas concentrations along the active area. The single cell was tested using a fuel cell test station (100W, Greenlight Innovation, Burnaby, Canada) coupled with two potentiostats (Biologic SP-200, Seyssinet-pariset, France and Ivium Vertex, Eindhoven, The Netherlands). Electrochemical impedance spectroscopy (EIS), cyclic voltammetry

(CV), and linear sweep voltammetry (LSV) tests were performed under H<sub>2</sub>/N<sub>2</sub> for determining high-frequency resistance (HFR) for the electrodes, ECSA, and H<sub>2</sub> crossover, respectively. The CV and LSV tests were performed at 200 and 5 mV/s, respectively. The protonic resistance of ionomer in the cathode CL was determined by performing EIS at 0.4 V under H<sub>2</sub>/N<sub>2</sub> by sweeping frequencies in the range of 1 Hz to 1 MHz with an amplitude of 10 mV. Pt electrochemical surface area (ECSA) was determined by integrating the hydrogen underpotential deposition (H<sub>upd,desorption</sub>) region while subtracting capacitive currents. The cell was initially conditioned (see Table 2 for the conditioning protocol details).

**Table 2.** Summary of the conditioning protocol used in this study.

Step #	Test	T <sub>cell</sub> (°C)	T <sub>hum</sub> (°C)	Flow Rate An/Ca (NLPM)	Reactant Gas An/Ca	Back Pressure (kPag)	Time (hrs)
1	H <sub>2</sub> pumping	30	45	0.5/0.5	H <sub>2</sub> /H <sub>2</sub>	0	0.5
2	Flooding	60	70	0.05/0.1	H <sub>2</sub> /N <sub>2</sub>	50	8–12
3	0.6 V hold	70	70	0.3/0.5	H <sub>2</sub> /O <sub>2</sub>	200	12
4	Potential cycling (0.6–0.8 V)	70	70	0.3/0.5	H <sub>2</sub> /O <sub>2</sub>	50	Holding 5 min at each potential

The cell performance was characterized by collecting potentiostatic polarization curves with in-situ high-frequency resistance (HFR) using an Ivium Vertex potentiostat (Ivium, Eindhoven, Netherlands). The potentiostatic polarization curve was obtained by holding the voltage for 3 min at each voltage at a resolution of 0.1 V. The limiting current study was performed by varying O<sub>2</sub> concentrations (1–24% O<sub>2</sub>:N<sub>2</sub>) [38,46]. The detail of all the testing conditions is outlined in Table 3.

**Table 3.** Summary of membrane electrode assembly (MEA) testing conditions for all the tests performed in this study.

Test	T <sub>cell</sub> (°C)	RH (%)	Flow Rate An/Ca (NLPM)	Reactant Gas An/Ca	Back Pressure (kPag)
Diagnostics (CV, LSV, EIS)	70	30, 45, 60, and 100	0.1/0.2	H <sub>2</sub> /N <sub>2</sub>	50
Performance			0.3/0.5	H <sub>2</sub> /Air	
Limiting current		30, 60, and 80	0.2/0.5	H <sub>2</sub> /O <sub>2</sub> (1–24%):N <sub>2</sub>	50, 100, 150, and 200

### 3.5 Conclusions

In this work, the effect of ionomer side chain length (or EW) on the electrochemical interfacial properties and mass transport properties including long-range proton transport, local O<sub>2</sub>-transport, Pt utilization, double-layer capacitance, and ORR reaction kinetics was investigated at varying RH. In summary, the CL prepared with shorter side chain (Aq-825) exhibited higher ECSA, higher CL ionic conductivity, higher CL double-layer capacitance, and higher CL local O<sub>2</sub>-transport resistance compared to the CL prepared with longer side chain (Naf-1100). The differences in these properties can be explained on the basis of differences in the EW and side chain. However, the differences in catalyst layer microstructure such the ionomer coverage and connectivity or the pore size can also be responsible. A systematic study combining microstructural characterization and catalyst layer properties is needed to ascertain the origin of the observed differences in catalyst layer properties. At 120 mV ORR overpotential, the Aq-825 CL showed 2-5 times higher ORR activity compared to the Naf-1100 CL at any given RH, except at 30% RH, which can be ascribed to the higher interfacial concentration of protons at the Pt/ionomer interface for Aq-825 CL. Our findings also indicate that at each RH, Naf-1100 CL illustrated lower local oxygen transport resistance ( $R_{O_2,Pt}$ ) compared to Aq-825 CL, for instance, Aq-825 CL showed 40% higher  $R_{O_2,Pt}$  at 80% RH. For both CLs,  $R_{O_2,Pt}$  decreased with increasing RH as higher interfacial water content at the Pt/ionomer interface in the CL promotes faster oxygen transport.



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# Chapter 4: Graphene based PEMFC catalyst layer

## 4.1 Introduction

PEMFC catalyst support plays a crucial role in the dispersion of catalyst particles and also ionomer distribution. Recent work in our group has demonstrated that uniformly distributed functional groups can ensure uniform distribution of Pt catalyst on the support, which then controls Pt-Pt particle distance and control of particle size [104]. The works by Orfanidi et al [108] and Ott et al [109] have shown that amine functionalization of carbon support affects its interaction with ionomer resulting in uniform distribution of ionomer on catalyst as well as in improving ionomer/catalyst interfacial structure. Morphologically different carbon supports with high microporosity has also gathered interest because Pt lodged within the micropores avoid direct contact with ionomer and are not prone to sulphonic acid poisoning, resulting in higher activity.

However, much of the R&D interest on catalyst support has been on improving the durability of carbon support, which upon corrosion can impact dispersion of supported catalyst as well as collapse of CL microstructure [110]. This is why PEMFC researchers have been exploring the benefits of graphene based PEMFC CLs. The potential benefits of graphene based CLs have been explained in sections 1.9-1.11. The following sections detail the process and results of fabricating and characterizing a PEMFC catalyst layer made with electrochemically exfoliated co-doped graphene.

## 4.2 Experimental work

### 4.2.1 Synthesis of co-doped graphene

Electrochemical exfoliation of graphite was performed in a two-electrode setup using a stainless-steel plate as the counter electrode (cathode) and a flexible graphite foil (Alfa Aesar) as the working electrode (anode) as mentioned in section 2.1.

### 4.2.2 Platinum deposition by modified polyol method

Pt catalyst was deposited on graphene nanoparticles using the method described in 2.1.2. 200 mg of graphene was sonicated for 10 mins with 150 ml of ethylene glycol solution (60% v/v) in a triple-neck round bottom flask. A calculated amount of dihydrogen hexachloroplatinate (V) hydrate (99.99% chloroplatinic acid, Alfa Aesar) was added to the mix and stirred using a magnetic stirrer. The amount of chloroplatinic acid added depends on the Pt loading target. The pH of the dispersion was adjusted to 11 using 0.2 M NaOH solution. The dispersion was then heated to 140 °C using an oil bath-hotplate setup and then stirred for 24h. The dispersion

was cooled and then filtered and washed with DI water using a vacuum filtration setup using a cellulose membrane. The obtained material was sonicated and dispersed in DI water using a bath sonicator for 5 mins. The Pt/G solution was frozen for 2 days and then freeze dried for another 2-3 days resulting in catalyst powder. The target was 10 % Pt/G.

#### 4.2.3 Catalyst ink formulation

The catalyst ink was prepared using 12.2% Pt/G catalyst powder and Nafion (EW 1100) purchased from Ion power Inc. (New Castle, DE, USA). The ionomer to carbon (I/C) and solid to liquid (S/L) ratio was maintained at 0.8 and 0.03, respectively. The ionomer stock dispersion was first diluted using a mixture of dimethylformamide (DMF) and isopropanol in a ratio of 1:4 (v/v), and the resulting mixture was sonicated for 20-30 mins to break up the ionomer aggregates. Then, 200 mg of Pt/G catalyst was added into the diluted ionomer dispersion using an ice jacket. Then, the mixture was subjected to three hours of magnetic stirring and 48 h of ball milling.

#### 4.2.4 Membrane electrode assembly (MEA) preparation

The catalyst layer decals will be prepared by coating the catalyst ink on ethylene tetrafluoroethylene (ETFE) sheet using a micrometer adjustable film casting doctor blade (MSK AFA-II, MTI corporation) and air dried for 24 h. The doctor blade will be adjusted to a thickness of 100  $\mu\text{m}$  for coating of the catalyst layer decal. Decal transfer method will be used to prepare an MEA. Nafion membrane (25  $\mu\text{m}$ , NRE-211, DuPont, Wilmington, DE, USA) will be hot-pressed in between the anode (0.2  $\text{mg}_{\text{Pt}}/\text{cm}^2$ , commercial Vulcan carbon-based CL) and the cathode decal (Pt/G) at 150  $^{\circ}\text{C}$  and 2 MPa pressure for 3 min. The MEAs will have an anode and cathode active area of 1.44 and 1  $\text{cm}^2$ , respectively. The larger anode area will be maintained to ensure surplus supply of reactant (protons) from the anode. The thickness, platinum loading, and ionomer loading of the cathode CLs are listed in Table 4.1. The changes in local and interfacial transport properties become very insignificant and remain obscured at high cathode Pt loading as it scales inversely with Pt ECSA, thus CLs with ultra-low Pt loading will be prepared in this study.

**Table 4.1:** Summary of the cathode catalyst layer properties.

Catalyst Layer Type	CL Thickness ( $\mu\text{m}$ )	Pt Loading ( $\text{mg}_{\text{Pt}}/\text{cm}^2_{\text{geo}}$ )	Ionomer Loading (wt%)
Pt/G	$8 \pm 1$	0.06	39
Pt/C	$11 \pm 1$	0.043	42.07

#### 4.2.5 Fuel cell assembly and testing

A small scale and differential flow field cell will be assembled by sandwiching the hot-pressed MEA between 160  $\mu\text{m}$  thick Toray gas diffusion layers coated with microporous layer (MPL) (TGP H-60, Toray, Japan). A differential cell will be used to avoid any discrepancies arising from the gradient in RH and reactant gas concentrations along the active area. The single cell will be tested using a fuel cell test station (100W, Greenlight Innovation, Burnaby, BC, Canada) coupled with a potentiostat (Biologic SP-200, Seyssinet-pariset, France)

Electrochemical impedance spectroscopy (EIS), cyclic voltammetry (CV), and linear sweep voltammetry (LSV) tests will be performed under  $\text{H}_2/\text{N}_2$  for determining high-frequency resistance (HFR) for the electrodes, ECSA, and  $\text{H}_2$  crossover, respectively. The CV and LSV tests will be performed at 200 and 5 mV/s, respectively. The protonic resistance of ionomer in the cathode CL will be determined by performing EIS at 0.4 V under  $\text{H}_2/\text{N}_2$  by sweeping frequencies in the range of 1 Hz to 1 MHz with an amplitude of 10 mV. Pt electrochemical surface area (ECSA) will be determined by integrating the hydrogen underpotential deposition ( $\text{H}_{\text{upd, desorption}}$ ) region while subtracting capacitive currents. The cell will be initially conditioned as per details mentioned in Table 4.2.

**Table 4.2:** Summary of the conditioning protocol used in this study.

Step #	Test	$T_{\text{cell}}$ (°C)	$T_{\text{hum}}$ (°C)	Flow rate An/Ca (NLPM)	Reactant gas An/Ca	Back Pressure (kPag)	Time (hrs)
1	$\text{H}_2$ pumping	30	45	0.5/0.5	$\text{H}_2/\text{H}_2$	0	0.5
2	Flooding	60	70	0.05/0.1	$\text{H}_2/\text{N}_2$	50	8–12
3	0.6 V hold	70	70	0.3/0.5	$\text{H}_2/\text{O}_2$	200	12
4	Potential cycling (0.6 – 0.8 V)	70	70	0.3/0.5	$\text{H}_2/\text{O}_2$	50	Holding 5 min at each potential

The cell performance will be characterized by collecting potentiostatic polarization curves with in-situ high-frequency resistance (HFR) using a Biologic SP-200 potentiostat. The potentiostatic polarization curve will be obtained by holding the voltage for 3 min at each voltage at a resolution of 0.1 V. The limiting current study will be performed by varying  $\text{O}_2$  concentrations (1–24%  $\text{O}_2:\text{N}_2$ ). The details of all the testing conditions is outlined in Table 4.3.

**Table 4.3:** Summary of membrane electrode assembly (MEA) testing conditions for all the tests performed in this study.

Test	T <sub>cell</sub> (°C)	RH (%)	Flow rate An/Ca (NLPM)	Reactant Gas An/Ca	Back Pressure (kPag)
Diagnostics (CV, LSV, EIS)	70	30, 45, 60, and 100	0.1/0.2	H <sub>2</sub> /N <sub>2</sub>	50
Performance			0.3/0.5	H <sub>2</sub> /Air	
Limiting current		30, 60, and 80	0.2/0.5	H <sub>2</sub> /O <sub>2</sub> (1-24%):N <sub>2</sub>	50,100, 150, and 200

## 4.3 Results and discussion

### 4.3.1 Graphene synthesis and characterization

Graphene oxide doped with nitrogen and phosphorus heteroatoms were synthesized using one step electrochemical exfoliation. It was then characterized by DLS and zetasizer to determine the particle size distribution and polar solvent stability respectively.

#### 4.3.1.1 Particle size distribution measurement by dynamic light scattering (DLS)

The graphene was characterized by DLS to determine its particle size distribution. The particle size distribution of co-doped graphene in DI water showed a peak diameter of 605 nm (as shown in Figure 4.2). However, DLS is not considered a reliable method of characterizing the size of non-spherical particles. The high peak diameter could also be a result of agglomeration and re-stacking of graphene layers due to Van der Waal's forces. This characterization was done using Malvern Zetasizer Nano ZS.



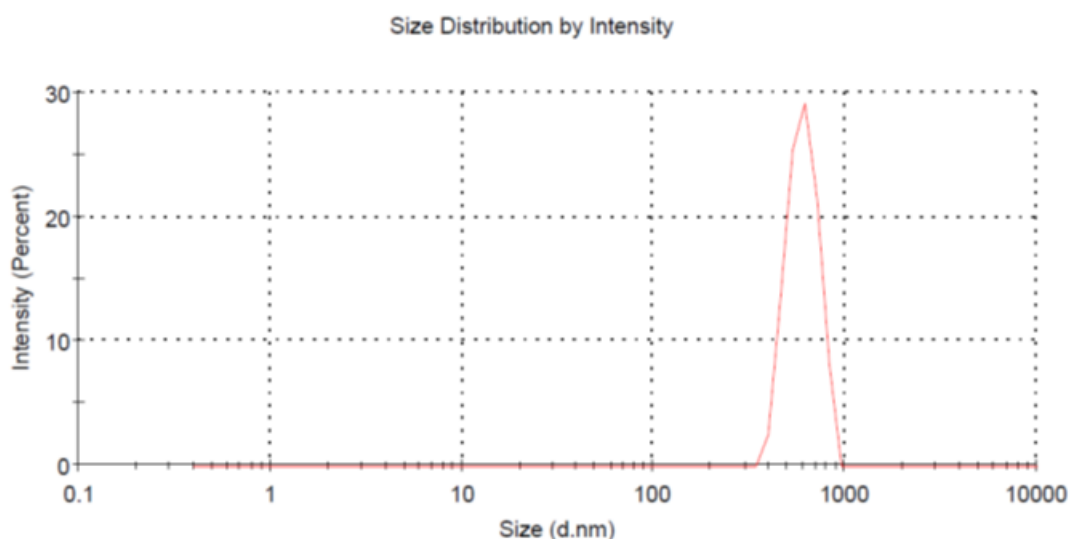


Figure 4.1: A sample particle size distribution of co-doped graphene in DI water

#### 4.3.1.2 Zeta-potential measurements

The graphene was characterized by zeta sizer to determine its zeta potential value across the entire pH range. Zeta potential values greater than 30 mV or less than -30 mV is an indicator of stability. The graphene dispersed in DI water showed zeta potential values less than -30mV which indicates stability (as shown in Figure 4.3). Stability in polar solvents like water is a good indicator that the graphene will be stable in other polar solvents like ethylene glycol and isopropyl alcohol during Pt deposition and catalyst ink formulation steps. This characterization was done using Malvern Zetasizer Nano ZS.

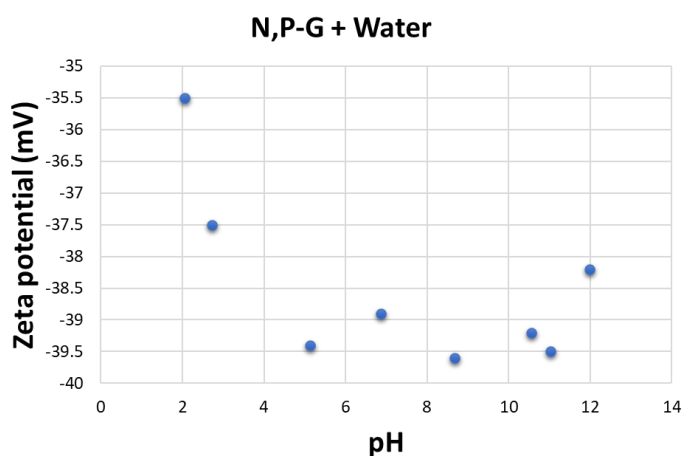


Figure 4.2: Zeta potential measurements of co-doped graphene in DI water

#### 4.3.1.3 Determination of Pt loading

Pt nanoparticles were deposited on the synthesized graphene (Pt/G) by modified polyol reduction method, and it was characterized using TGA, SEM and EDX methods. The graphene was characterized by TGA to quantify the platinum loading (wt %) on the graphene support. After Pt was deposited on the co-doped graphene by modified polyol method, we confirmed the Pt/G % to be 12.2%. The target was 10% Pt/G. The actual loading was higher than the target either due to measurement error or loss of graphene material during Pt deposition step. This characterization was done using Mettler Toledo TGA/DSC3+.

#### 4.3.1.4 Determination of Pt distribution

The Pt/G was characterized by SEM to qualitatively analyze its morphology to see the size of graphene and Pt particles and also the degree of Pt distribution on the surface of graphene. SEM analysis of Pt/G showed the presence of Pt nanoparticles on the surface of graphene (as shown in Figure 4.4). This characterization was done using Phenom Pro X. However, further confirmation of particle size, distribution and uniformity is required using a higher resolution SEM and TEM.

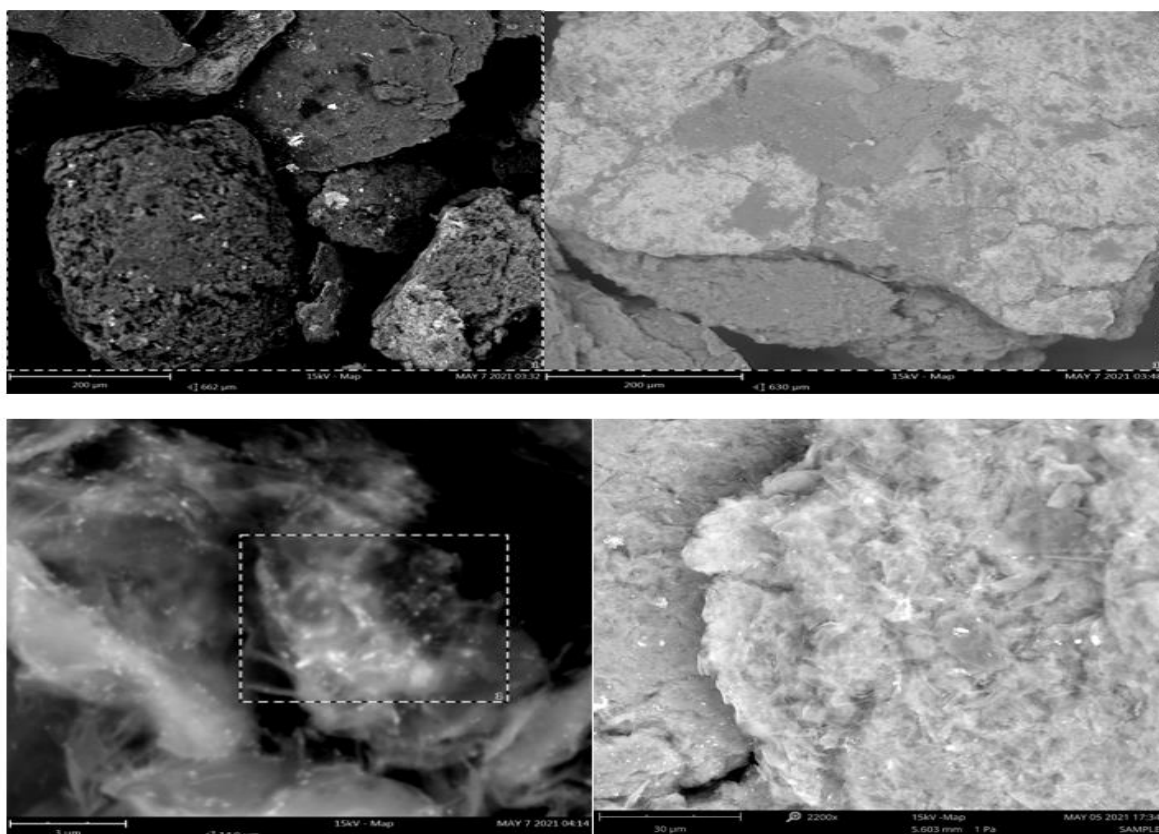


Figure 4.3: SEM analysis of Pt/G catalyst

#### 4.3.1.5 Elemental analysis of Pt/G

EDX was used for elemental analysis and to confirm the presence of dopant heteroatoms, Pt and oxygen functional groups. Elemental analysis of Pt/G confirmed the presence of carbon, Pt as well as phosphorus, nitrogen and oxygen functional groups (as shown in table 4.4). However, further confirmation of the elemental composition is required by X-ray photoelectron spectroscopy (XPS).

Element Symbol	Atomic Conc.	Weight Conc.	Oxide Symbol	Stoich. Conc.
C	60.51	33.64	C	84.41
O	28.31	20.97		
P	4.72	6.77	P	6.59
Pt	4.11	37.10	Pt	5.73
N	2.34	1.52	N	3.27

Table 4.4: EDX analysis of Pt/G

#### 4.3.2 In-situ fuel cell characterization

An MEA containing the CL made with Pt/G catalyst was fabricated and tested using a PEM fuel cell setup.

##### 4.3.2.1 Determination of ECSA

The ECSA of the cathode CL at different RH conditions was confirmed using CV. Figure 4.5(a) shows the presence of Pt peaks in the hydrogen desorption region of the CV curve which confirms the presence of electrochemically active Pt particles. Figure 4.5(b) shows the effect of RH on ECSA of Pt/G versus Pt/C. In both cases, the ECSA increases linearly with relative humidity. Figure 4.5(c) shows the effect of RH on roughness factor (RF) of Pt/G versus Pt/C.

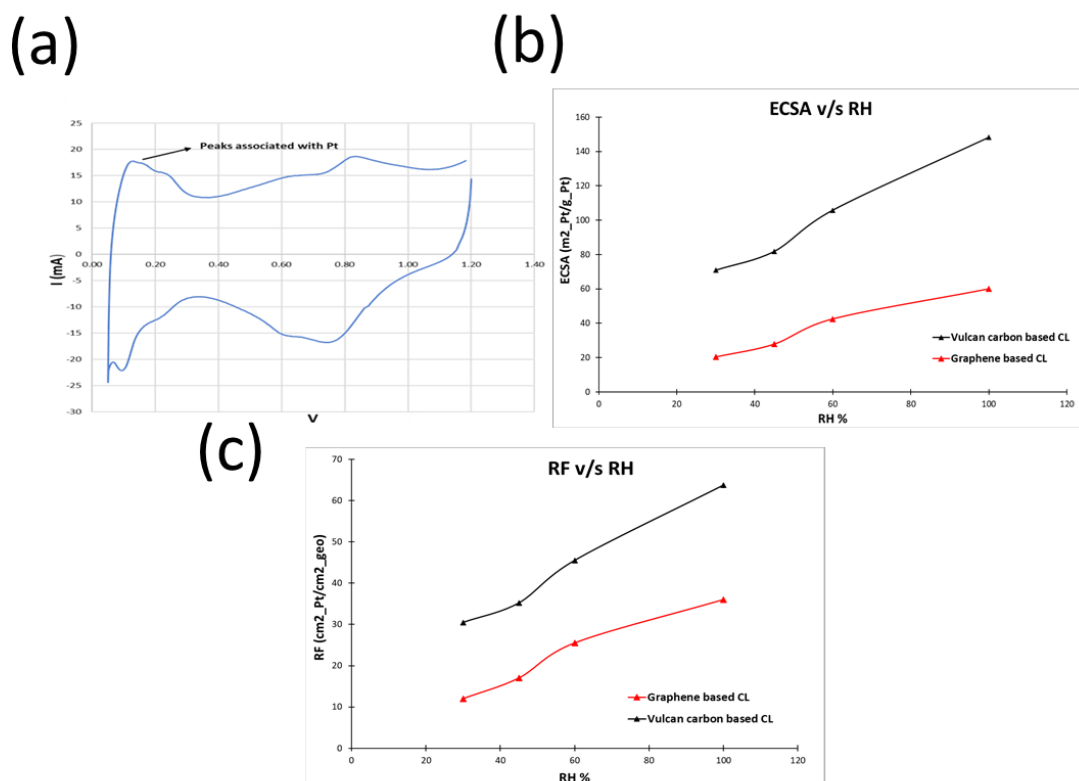


Figure 4.4: (a) In-situ cyclic voltammetry at 100% RH in  $\text{H}_2/\text{N}_2$ ; (b) RH dependent ECSA measurements; (c) RH dependant RF measurements

#### 4.3.2.2 Determination of double layer capacitance

The double layer capacitance from the anode side was quantified from the CV plots. For both CLs, the DL capacitance values increases with RH as shown in Fig 4.6. However, we should note the significantly low DL capacitance values of the graphene based CL which could hint towards poor ionomer coverage in the CL.

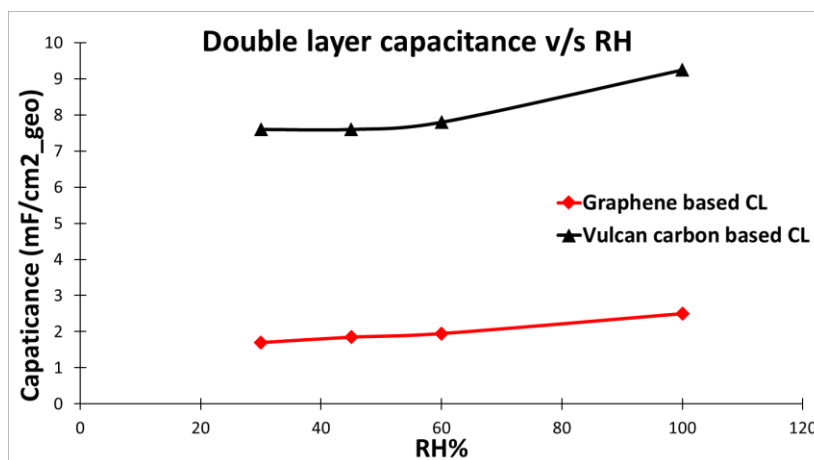


Fig 4.5: Double layer capacitance at different RHs

#### 4.3.2.3 Determination of H<sub>2</sub> cross over current

The H<sub>2</sub> cross over from anode side was quantified using LSV. The H<sub>2</sub> cross over current values at different RHs were low indicating that there was minimal cross over of H<sub>2</sub> from anode to cathode (as shown in Figure 4.7). The RH dependant H<sub>2</sub> cross over current values of both Pt/G and Pt/C were low.

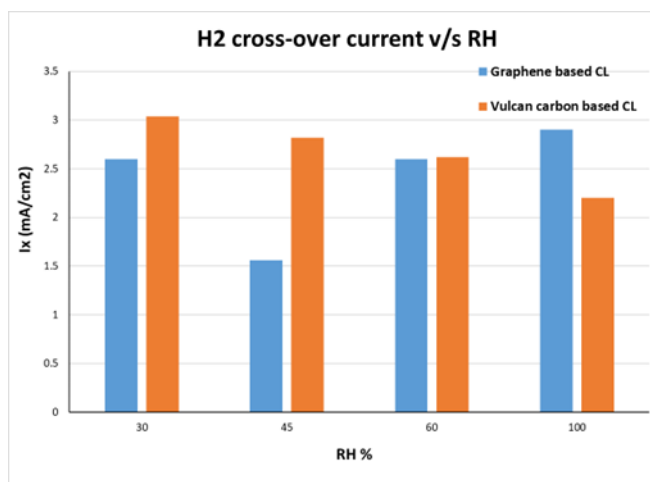


Figure 4.6: H<sub>2</sub> cross over currents at different RHs

#### 4.3.2.4 Determination of CL ionomer protonic resistance and HFR

The catalyst layer ionomer resistance ( $R_{CL,H^+}$ ) and HFR of the membrane was determined using EIS. In comparison to conventional Pt/C catalysts, the  $R_{CL,H^+}$  of Pt/G was too high (as shown in Figure 4.8). This could mean that the ionomer coverage in the CL was non-uniform. Future work will focus on mitigating this issue by altering the ionomer to carbon and solid to liquid ratios of the catalyst ink. The HFR values of the membrane was within normal range.

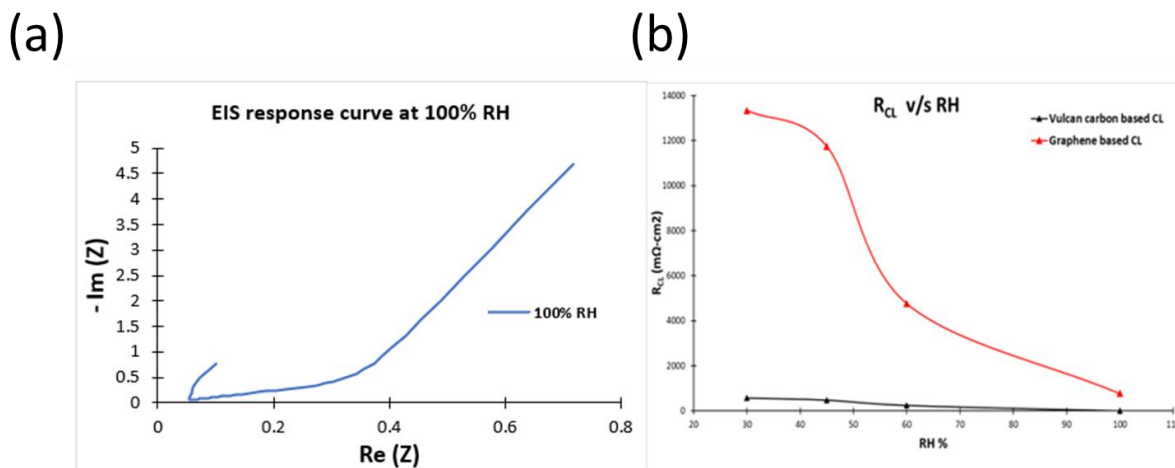


Figure 4.7: a) EIS response curve of Pt/G at 100%RH, b) RH dependence of CL ionomer protonic resistance

#### 4.3.2.5 In-situ performance tests

As seen in figure 4.9, the performance of Pt/G was poor in comparison to conventional Pt/C [111]. This could be due to non-uniform ionomer coverage in the CL and re-stacking of graphene layers in the CL which hinders the transport of reactant gas to the active sites. This problem will be mitigated in future work.

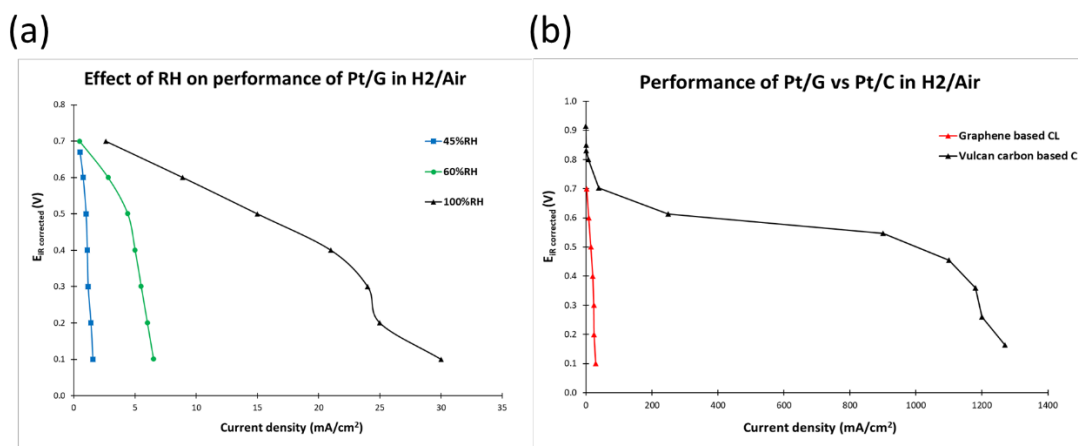


Figure 4.8: a) Effect of RH on the performance of Pt/G in air, b) Performance comparison of Pt/G and Pt/C in air and 100% RH

#### 4.3.2.6 Limiting current study to determine CL oxygen transport resistance

No limiting current was observed during this test which means that the performance is not limited by mass transport issues in the CL.

## 4.4 Conclusion

One of the main goals of the work described in Chapter 3 was to establish a baseline performance with a Vulcan based CL and then compare it with the graphene based CL as described in Chapter 4. The Vulcan carbon based CL outperformed the graphene based CL in almost every performance metric. In chapter 4, although the CV showed the presence of electrochemically active Pt in the CL, the poor performance, lack of limiting current and the high CL protonic conductivity indicates that reactant gas is unable to reach the active sites due to poor CL porosity possibly due to re-stacking of graphene layers. Also, protonic transport is hindered by non-uniform ionomer coverage in the CL as seen in Fig 4.6. Thus, the goal of future work will be to redesign the CL in order to resolve the above issues.

# Chapter 5. Final conclusions and recommendations for future work

## 5.1 Final conclusions

The following research objectives for this study were all accomplished successfully:

- Establish a baseline of PEMFC properties such as ECSA, CL ionomer protonic resistance, performance, and O<sub>2</sub> transport resistance with a conventionally used Vulcan based CL.
- Study the effect of ionomer equivalent weight, ionomer side chain length and relative humidity (RH) on PEMFC CL properties.
- Use co-doped electrochemically exfoliated graphene as a PEMFC cathode CL and develop a catalyst ink recipe for it.
- Examine the effect of graphitization on the properties of the PEMFC catalyst layers such as ECSA, CL ionomer protonic resistance, performance and O<sub>2</sub> transport resistance.
- Investigate the performance and properties of an alternative cathode catalyst layer architecture and compare it to the conventionally used vulcan carbon based CL.

In conclusion, by examining some of the very significant current roadblocks preventing the wide adoption of PEMFC technology, such as catalyst activity, CL durability and local O<sub>2</sub>-transport, this thesis has made several contributions to the advancement of the PEMFC research field. The knowledge that has been added to PEMFC science can be summarized below:

- In chapter 3, the CL prepared with shorter side chain (Aq-825) exhibited higher ECSA, higher CL ionic conductivity, higher CL double-layer capacitance, and higher CL local O<sub>2</sub>-transport resistance compared to the CL prepared with longer side chain (Naf-1100).
- In-situ analysis of RH-dependent oxygen reduction reaction (ORR) kinetics for CLs with different ionomers was done for the first time in this study. The discovery that the ORR kinetic activity ( $A/cm^2_{Pt}$ ) for the Aquivion-825 CL is higher than for the Nafion-1100 CL is significant. The reason for this is that the two CL's Pt/ionomer interfaces have different interfacial protonic contents.



- In chapter 4, one step electrochemically exfoliated co-doped graphene was successfully fabricated into functional catalyst support and it demonstrated a significant amount of ECSA using in-situ PEMC characterization.

Furthermore, the problems that have been identified have been summarised below:

- In chapter 4, although the CV showed the presence of electrochemically active Pt in the graphene based CL, the poor performance, lack of limiting current and the high CL protonic conductivity indicates that reactant gas is unable to reach the active sites due to poor CL porosity possibly due to re-stacking of graphene layers.
- Also, protonic transport is hindered by non-uniform ionomer coverage in the CL. Before we can compare and test the durability of a graphene based CL with conventional CLs, these issues need to be resolved.

## 5.2 Recommendations for future work

The work in chapter 3 examined the impact of ionomer side chain length (or EW) on the electrochemical interfacial characteristics and mass transport parameters, including long-range proton transport, local O<sub>2</sub>-transport, Pt utilisation, double-layer capacitance, and ORR reaction kinetics. In conclusion, compared to the CL prepared with longer side chain, the CL prepared with shorter side chain (Aq-825) showed greater ECSA, CL ionic conductivity, CL double-layer capacitance, and CL local O<sub>2</sub>-transport resistance (Naf-1100). The discrepancies in these properties can be explained based on variations in the EW and side chain length. However, variations in the microstructure of the catalyst layer, such as ionomer coverage and connectivity or pore size, may also be responsible. In the future, to determine the cause of the observed variations in catalyst layer properties, a comprehensive investigation integrating microstructural characterisation and catalyst layer properties is required.

In reference to the work in chapter 4, the characterization of Pt/G must be done using high resolution SEM and TEM to test the degree of uniformity, particle size and distribution of Pt nanoparticles on the graphene surface. Also, due to its higher accuracy, XPS must be used to confirm the elemental composition of Pt/G sample. In addition, the catalyst ink recipe could be further optimized to improve the uniformity and coatability of the catalyst ink. The catalyst ink parameters like Ionomer to carbon ratio, solid to liquid ratio, etc should be optimized to improve the ionomer coverage and porosity in the CL. Furthermore, after the incorporation of the above recommendations, if the performance of the Pt/G CL still does not improve, the structure of the Pt/G CL should be modified by adding 'spacers' between the graphene layers to mitigate the restacking issue and to

improve CL porosity for better reactant gas permeability. After the incorporation of these recommendations, if the performance of the graphene based CL can match or even exceed the performance of conventional CLs, durability testing of the Pt/G CL should be done using the protocol dictated by the USDOE.

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Publication Title	Nature Energy	Publication Type	e-Journal
Article Title	New roads and challenges for fuel cells in heavy-duty transportation	Start Page	462
		End Page	474
Date	01/01/2016	Issue	5
Language	English	Volume	6
Country	United Kingdom of Great Britain and Northern Ireland		
Rightsholder	Springer Nature BV		

## REQUEST DETAILS

Portion Type	Chart/graph/table/figure	Distribution	Worldwide
Number of Charts / Graphs / Tables / Figures Requested	1	Translation	Original language of publication
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Lifetime Unit Quantity	Up to 750,000	Currency	CAD
Rights Requested	Main product		

## NEW WORK DETAILS

Title	Effect of Graphitization on PEMFC Catalyst Layer Properties	Institution Name	University of Calgary
Instructor Name	Sushmit Poojary	Expected Presentation Date	2023-01-27

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The Requesting  
Person/Organization to  
Appear on the License

Sushmit Poojary

## REQUESTED CONTENT DETAILS

<b>Title, Description or Numeric Reference of the Portion(s)</b>	Fig. 2: Summary of fuel-cell targets and lifetimes	<b>Title of the Article/Chapter the Portion Is From</b>	New roads and challenges for fuel cells in heavy-duty transportation
<b>Editor of Portion(s)</b>	Cullen, David A.; Neyerlin, K. C.; Ahluwalia, Rajesh K.; Mukundan, Rangachary; More, Karren L.; Borup, Rodney L.; Weber, Adam Z.; Myers, Deborah J.; Kusoglu, Ahmet	<b>Author of Portion(s)</b>	Cullen, David A.; Neyerlin, K. C.; Ahluwalia, Rajesh K.; Mukundan, Rangachary; More, Karren L.; Borup, Rodney L.; Weber, Adam Z.; Myers, Deborah J.; Kusoglu, Ahmet
<b>Volume of Serial or Monograph</b>	6	<b>Publication Date of Portion</b>	2021-04-30
<b>Page or Page Range of Portion</b>	462-474		

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*Last updated October 2022*



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## LICENSED CONTENT

Publication Title	Current Opinion in Electrochemistry	Publication Type	Journal
Article Title	PEFC catalyst layer: Recent advances in materials, microstructural characterization, and modeling	Start Page	27
		End Page	35
		Issue	1
		Volume	5
Date	01/01/2017		
Language	English		
Country	United Kingdom of Great Britain and Northern Ireland		
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## NEW WORK DETAILS

Title	Effect of Graphitization on PEMFC Catalyst Layer Properties	Institution Name	University of Calgary
Instructor Name	Kunal Karan	Expected Presentation Date	2023-01-27

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Order Reference Number	N/A	The Requesting Person/Organization to Appear on the License	Sushmit Poojary
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## REQUESTED CONTENT DETAILS

Title, Description or Numeric Reference of the Portion(s)	Figure 1. Depiction of local CL structure comprising porous carbon support with graphitic micro-domains, Pt nanoparticles on surface and in micropore—capped by ionomer film and uncovered	Title of the Article/Chapter the Portion Is From	PEFC catalyst layer: Recent advances in materials, microstructural characterization, and modeling
		Author of Portion(s)	Karan, Kunal
		Issue, if Republishing an Article From a Serial	1
Editor of Portion(s)	Karan, Kunal	Publication Date of Portion	2017-09-30
Volume of Serial or Monograph	5		
Page or Page Range of Portion	27-35		

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i) The copies and anthologies created under this License may be made and assembled by faculty members individually or at their request by on-campus bookstores or copy centers, or by off-campus copy shops and other similar entities.

ii) No License granted shall in any way: (i) include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied) (ii) permit "publishing ventures" where any particular anthology would be systematically marketed at multiple institutions.

iii) Subject to any Publisher Terms (and notwithstanding any apparent contradiction in the Order Confirmation arising from data provided by User), any use authorized under the academic pay-per-use service is limited as follows:

A) any License granted shall apply to only one class (bearing a unique identifier as assigned by the institution, and thereby including all sections or other subparts of the class) at one institution;

B) use is limited to not more than 25% of the text of a book or of the items in a published collection of essays, poems or articles;

C) use is limited to no more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

D) no User may sell or distribute any particular anthology, whether photocopied or electronic, at more than one institution of learning;

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F) any permission granted shall expire at the end of the class. No permission granted shall in any way include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied).

iv) Books and Records; Right to Audit. As to each permission granted under the academic pay-per-use Service, User shall maintain for at least four full calendar years books and records sufficient for CCC to determine the numbers of copies made by User under such permission. CCC and any representatives it may designate shall have the right to audit such books and records at any time during User's ordinary business hours, upon two days' prior notice. If any such audit shall determine that User shall have underpaid for, or underreported, any photocopies sold or by three percent (3%) or more, then User shall bear all the costs of any such audit; otherwise, CCC shall bear the costs of any such audit. Any amount determined by such audit to have been underpaid by User shall immediately be paid to CCC by User, together with interest thereon at the rate of 10% per annum from the date such amount was originally due. The provisions of this paragraph shall survive the termination of this License for any reason.

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C) use is limited to not more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

D) no User may sell or distribute any particular materials, whether photocopied or electronic, at more than one institution of learning;

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*Last updated October 2022*


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<b>Order Date</b>	20-Dec-2022	<b>Type of Use</b>	Republish in a
<b>Order License ID</b>	1303074-1		thesis/dissertation
<b>ISSN</b>	1615-6854	<b>Publisher Portion</b>	WILEY-VCH
			Image/photo/illustration

## LICENSED CONTENT

<b>Publication Title</b>	Fuel cells	<b>Publication Type</b>	e-Journal
<b>Article Title</b>	Methodology for Evaluation of Contributions of Ostwald Ripening and Particle Agglomeration to Growth of Catalyst Particles in PEM Fuel Cells	<b>Start Page</b>	487
		<b>End Page</b>	498
		<b>Issue</b>	4
		<b>Volume</b>	20
		<b>URL</b>	http://www3.interscience. wiley.com/cgi- bin/jhome/84502986
<b>Author/Editor</b>	Wiley InterScience (Online service)		
<b>Date</b>	01/01/2001		
<b>Language</b>	English		
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<b>Title</b>	Effect of Graphitization on PEMFC Catalyst Layer Properties	<b>Institution Name</b>	University of Calgary
		<b>Expected Presentation Date</b>	2023-01-27
<b>Instructor Name</b>	Kunal Karan		

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<b>Title, Description or Numeric Reference of the Portion(s)</b>	Figure 1. ECSA loss mechanisms- Oswald ripening vs particle agglomeration	<b>Title of the Article/Chapter the Portion Is From</b>	Methodology for Evaluation of Contributions of Ostwald Ripening and Particle Agglomeration to Growth of Catalyst Particles in PEM Fuel Cells
<b>Editor of Portion(s)</b>	Kregar, A.; Kravos, A.; Kutrašnik, T.		
<b>Volume of Serial or Monograph</b>	20	<b>Author of Portion(s)</b>	Kregar, A.; Kravos, A.; Kutrašnik, T.
<b>Page or Page Range of Portion</b>	487-498	<b>Publication Date of Portion</b>	2020-08-26

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**11) Limitation of Liability.** UNDER NO CIRCUMSTANCES WILL CCC OR THE RIGHTSHOLDER BE LIABLE FOR ANY DIRECT, INDIRECT, CONSEQUENTIAL, OR INCIDENTAL DAMAGES (INCLUDING WITHOUT LIMITATION DAMAGES FOR LOSS OF BUSINESS PROFITS OR INFORMATION, OR FOR BUSINESS INTERRUPTION) ARISING OUT OF THE USE OR INABILITY TO USE A WORK, EVEN IF ONE OR BOTH OF THEM HAS BEEN ADVISED OF THE POSSIBILITY OF SUCH DAMAGES. In any event, the total liability of the Rightsholder and CCC (including their respective employees and directors) shall not exceed the total amount actually paid by User for the relevant License. User assumes full liability for the actions and omissions of its principals, employees, agents, affiliates, successors, and assigns.

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**13) Effect of Breach.** Any failure by User to pay any amount when due, or any use by User of a Work beyond the scope of the License set forth in the Order Confirmation and/or the Terms, shall be a material breach of such License. Any breach not cured within 10 days of written notice thereof shall result in immediate termination of such License without further notice. Any unauthorized (but licensable) use of a Work that is terminated immediately upon notice thereof may be liquidated by payment of the Rightsholder's ordinary license price therefor; any unauthorized (and unlicensable) use that is not terminated immediately for any reason (including, for example, because materials containing the Work cannot reasonably be recalled) will be subject to all remedies available at law or in equity, but in no event to a payment of less than three times the Rightsholder's ordinary license price for the most closely analogous licensable use plus Rightsholder's and/or CCC's costs and expenses incurred in collecting such payment.

**14) Additional Terms for Specific Products and Services.** If a User is making one of the uses described in this Section 14, the additional terms and conditions apply:

**a) *Print Uses of Academic Course Content and Materials (photocopies for academic coursepacks or classroom handouts).*** For photocopies for academic coursepacks or classroom handouts the following additional terms apply:

i) The copies and anthologies created under this License may be made and assembled by faculty members individually or at their request by on-campus bookstores or copy centers, or by off-campus copy shops and other similar entities.

ii) No License granted shall in any way: (i) include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied) (ii) permit "publishing ventures" where any particular anthology would be systematically marketed at multiple institutions.

iii) Subject to any Publisher Terms (and notwithstanding any apparent contradiction in the Order Confirmation arising from data provided by User), any use authorized under the academic pay-per-use service is limited as follows:

A) any License granted shall apply to only one class (bearing a unique identifier as assigned by the institution, and thereby including all sections or other subparts of the class) at one institution;

B) use is limited to not more than 25% of the text of a book or of the items in a published collection of essays, poems or articles;

C) use is limited to no more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

D) no User may sell or distribute any particular anthology, whether photocopied or electronic, at more than one institution of learning;

E) in the case of a photocopy permission, no materials may be entered into electronic memory by User except in order to produce an identical copy of a Work before or during the academic term (or analogous period) as to which any particular permission is granted. In the event that User shall choose to retain materials that are the subject of a photocopy permission in electronic memory for purposes of producing identical copies more than one day after such retention (but still within the scope of any permission granted), User must notify CCC of such fact in the applicable permission request and such retention shall constitute one copy actually sold for purposes of calculating permission fees due; and

F) any permission granted shall expire at the end of the class. No permission granted shall in any way include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied).

iv) Books and Records; Right to Audit. As to each permission granted under the academic pay-per-use Service, User shall maintain for at least four full calendar years books and records sufficient for CCC to determine the numbers of copies made by User under such permission. CCC and any representatives it may designate shall have the right to audit such books and records at any time during User's ordinary business hours, upon two days' prior notice. If any such audit shall determine that User shall have underpaid for, or underreported, any photocopies sold or by three percent (3%) or more, then User shall bear all the costs of any such audit; otherwise, CCC shall bear the costs of any such audit. Any amount determined by such audit to have been underpaid by User shall immediately be paid to CCC by User, together with interest thereon at the rate of 10% per annum from the date such amount was originally due. The provisions of this paragraph shall survive the termination of this License for any reason.

**b) *Digital Pay-Per-Uses of Academic Course Content and Materials (e-coursepacks, electronic reserves, learning management systems, academic institution intranets).*** For uses in e-coursepacks, posts in electronic reserves, posts in learning management systems, or posts on academic institution intranets, the following additional terms apply:

i) The pay-per-uses subject to this Section 14(b) include:

A) **Posting e-reserves, course management systems, e-coursepacks for text-based content**, which grants authorizations to import requested material in electronic format, and allows electronic access to this material to members of a designated college or university class, under the direction of an instructor designated by the college or university, accessible only under appropriate electronic controls (e.g., password);

B) **Posting e-reserves, course management systems, e-coursepacks for material consisting of photographs or other still images not embedded in text**, which grants not only the authorizations described in Section 14(b)(i)(A) above, but also the following authorization: to include the requested material in course materials for use consistent with Section 14(b)(i)(A) above, including any necessary resizing, reformatting or modification of the resolution of such requested material (provided that such modification does not alter the underlying editorial content or meaning of the requested material, and provided that the resulting modified content is used solely within the scope of, and in a manner consistent with, the particular authorization described in the Order Confirmation and the Terms), but not including any other form of manipulation, alteration or editing of the requested material;

C) **Posting e-reserves, course management systems, e-coursepacks or other academic distribution for audiovisual content**, which grants not only the authorizations described in Section 14(b)(i)(A) above, but also the following authorizations: (i) to include the requested material in course materials for use consistent with Section 14(b)(i)(A) above; (ii) to display and perform the requested material to such members of such class in the physical classroom or remotely by means of streaming media or other video formats; and (iii) to "clip" or reformat the requested material for purposes of time or content management or ease of delivery, provided that such "clipping" or reformatting does not alter the underlying editorial content or meaning of the

requested material and that the resulting material is used solely within the scope of, and in a manner consistent with, the particular authorization described in the Order Confirmation and the Terms. Unless expressly set forth in the relevant Order Confirmation, the License does not authorize any other form of manipulation, alteration or editing of the requested material.

ii) Unless expressly set forth in the relevant Order Confirmation, no License granted shall in any way: (i) include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied or, in the case of Works subject to Sections 14(b)(1)(B) or (C) above, as described in such Sections) (ii) permit "publishing ventures" where any particular course materials would be systematically marketed at multiple institutions.

iii) Subject to any further limitations determined in the Rightsholder Terms (and notwithstanding any apparent contradiction in the Order Confirmation arising from data provided by User), any use authorized under the electronic course content pay-per-use service is limited as follows:

A) any License granted shall apply to only one class (bearing a unique identifier as assigned by the institution, and thereby including all sections or other subparts of the class) at one institution;

B) use is limited to not more than 25% of the text of a book or of the items in a published collection of essays, poems or articles;

C) use is limited to not more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

D) no User may sell or distribute any particular materials, whether photocopied or electronic, at more than one institution of learning;

E) electronic access to material which is the subject of an electronic-use permission must be limited by means of electronic password, student identification or other control permitting access solely to students and instructors in the class;

F) User must ensure (through use of an electronic cover page or other appropriate means) that any person, upon gaining electronic access to the material, which is the subject of a permission, shall see:

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- a statement to the effect that such copy was made pursuant to permission,
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v) Books and Records; Right to Audit. As to each permission granted under the electronic course content Service, User shall maintain for at least four full calendar years books and records sufficient for CCC to determine the numbers of copies made by User under such permission. CCC and any representatives it may designate shall have the right to audit such books and records at any time during User's ordinary business hours, upon two days' prior notice. If any such audit shall determine that User shall have underpaid for, or underreported, any electronic



copies used by three percent (3%) or more, then User shall bear all the costs of any such audit; otherwise, CCC shall bear the costs of any such audit. Any amount determined by such audit to have been underpaid by User shall immediately be paid to CCC by User, together with interest thereon at the rate of 10% per annum from the date such amount was originally due. The provisions of this paragraph shall survive the termination of this license for any reason.

c) ***Pay-Per-Use Permissions for Certain Reproductions (Academic photocopies for library reserves and interlibrary loan reporting) (Non-academic internal/external business uses and commercial document delivery)***. The License expressly excludes the uses listed in Section (c)(i)-(v) below (which must be subject to separate license from the applicable Rightsholder) for: academic photocopies for library reserves and interlibrary loan reporting; and non-academic internal/external business uses and commercial document delivery.

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- v) republication in any different form. Please obtain authorizations for these uses through other CCC services or directly from the rightsholder.

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d) ***Electronic Reproductions in Online Environments (Non-Academic-email, intranet, internet and extranet)***. For "electronic reproductions", which generally includes e-mail use (including instant messaging or other electronic transmission to a defined group of recipients) or posting on an intranet, extranet or Intranet site (including any display or performance incidental thereto), the following additional terms apply:

- i) Unless otherwise set forth in the Order Confirmation, the License is limited to use completed within 30 days for any use on the Internet, 60 days for any use on an intranet or extranet and one year for any other use, all as measured from the "republication date" as identified in the Order Confirmation, if any, and otherwise from the date of the Order Confirmation.
- ii) User may not make or permit any alterations to the Work, unless expressly set forth in the Order Confirmation (after request by User and approval by Rightsholder); provided, however, that a Work consisting of photographs or other still images not embedded in text may, if necessary, be resized, reformatted or have its resolution modified without additional express permission, and a Work consisting of audiovisual content may, if necessary, be "clipped" or reformatted for purposes of time or content management or ease of delivery (provided that any such resizing, reformatting, resolution modification or "clipping" does not alter the underlying editorial content or meaning of the Work used, and that the resulting material is used solely within the scope of, and in a manner consistent with, the particular License described in the Order Confirmation and the Terms.

#### 15) Miscellaneous.

a) User acknowledges that CCC may, from time to time, make changes or additions to the Service or to the Terms, and that Rightsholder may make changes or additions to the Rightsholder Terms. Such updated Terms will replace the prior terms and conditions in the order workflow and shall be effective as to any subsequent Licenses but shall not apply to Licenses already granted and paid for under a prior set of terms.

b) Use of User-related information collected through the Service is governed by CCC's privacy policy, available online at [www.copyright.com/about/privacy-policy/](http://www.copyright.com/about/privacy-policy/).

c) The License is personal to User. Therefore, User may not assign or transfer to any other person (whether a natural person or an organization of any kind) the License or any rights granted thereunder; provided, however, that, where applicable, User may assign such License in its entirety on written notice to CCC in the event of a transfer of all or substantially all of User's rights in any new material which includes the Work(s) licensed under this Service.

d) No amendment or waiver of any Terms is binding unless set forth in writing and signed by the appropriate parties, including, where applicable, the Rightsholder. The Rightsholder and CCC hereby object to any terms contained in any writing prepared by or on behalf of the User or its principals, employees, agents or affiliates and purporting to govern or otherwise relate to the License described in the Order Confirmation, which terms are in any way inconsistent with any Terms set forth in the Order Confirmation, and/or in CCC's standard operating procedures, whether such writing is prepared prior to, simultaneously with or subsequent to the Order Confirmation, and whether such writing appears on a copy of the Order Confirmation or in a separate instrument.

e) The License described in the Order Confirmation shall be governed by and construed under the law of the State of New York, USA, without regard to the principles thereof of conflicts of law. Any case, controversy, suit, action, or proceeding arising out of, in connection with, or related to such License shall be brought, at CCC's sole discretion, in any federal or state court located in the County of New York, State of New York, USA, or in any federal or state court whose geographical jurisdiction covers the location of the Rightsholder set forth in the Order Confirmation. The parties expressly submit to the personal jurisdiction and venue of each such federal or state court.

*Last updated October 2022*



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Order Date	21-Dec-2022	Type of Use	Republish in a
Order License ID	1303461-1		thesis/dissertation
ISSN	0378-7753	Publisher Portion	ELSEVIER S.A. Chart/graph/table/figure

## LICENSED CONTENT

Publication Title	Journal of power sources	Publication Type	Journal
Article Title	PEM fuel cell electrocatalyst durability measurements	Start Page	76
		End Page	81
Date	01/01/1976	Issue	1
Language	English, French, French, German, German	Volume	163
Country	Switzerland		
Rightsholder	Elsevier Science & Technology Journals		

## REQUEST DETAILS

Portion Type	Chart/graph/table/figure	Distribution	Worldwide
Number of Charts / Graphs / Tables / Figures Requested	2	Translation	Original language of publication
Format (select all that apply)	Electronic	Copies for the Disabled?	Yes
Who Will Republish the Content?	Academic institution	Minor Editing Privileges?	Yes
Duration of Use	Life of current edition	Incidental Promotional Use?	Yes
Lifetime Unit Quantity	Up to 44,999	Currency	CAD
Rights Requested	Main product and any product related to main product		

## NEW WORK DETAILS

Title	Effect of Graphitization on PEMFC Catalyst Layer Properties	Institution Name	University of Calgary
		Expected Presentation Date	2023-01-27
Instructor Name	Kunal Karan		

## ADDITIONAL DETAILS

Order Reference Number	N/A	The Requesting Person/Organization to Appear on the License	Sushmit Poojary
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## REQUESTED CONTENT DETAILS

Title, Description or Numeric Reference of the Portion(s)	Figures 2 and 6	Title of the Article/Chapter the Portion Is From	PEM fuel cell electrocatalyst durability measurements
Editor of Portion(s)	Borup, Rod L.; Davey, John R.; Garzon, Fernando H.; Wood, David L; Inbody, Michael A.	Author of Portion(s)	Borup, Rod L.; Davey, John R.; Garzon, Fernando H.; Wood, David L; Inbody, Michael A.
Volume of Serial or Monograph	163	Issue, if Republishing an Article From a Serial	1
Page or Page Range of Portion	76-81	Publication Date of Portion	2005-12-31

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"Order Confirmation" is the confirmation CCC provides to the User at the conclusion of each Marketplace transaction. "Order Confirmation Terms" are additional terms set forth on specific Order Confirmations not set forth in the General Terms that can include terms applicable to a particular CCC transactional licensing service and/or any Rightsholder-specific terms.

"Rightsholder(s)" are the holders of copyright rights in the Works for which a User obtains licenses via the Marketplace platform, which are displayed on specific Order Confirmations.

"Terms" means the terms and conditions set forth in these General Terms and any additional Order Confirmation Terms collectively.

"User" or "you" is the person or entity making the use granted under the relevant License. Where the person accepting the Terms on behalf of a User is a freelancer or other third party who the User authorized to accept the General Terms on the User's behalf, such person shall be deemed jointly a User for purposes of such Terms.

"Work(s)" are the copyright protected works described in relevant Order Confirmations.

2) **Description of Service.** CCC's Marketplace enables Users to obtain Licenses to use one or more Works in accordance with all relevant Terms. CCC grants Licenses as an agent on behalf of the copyright rightsholder identified in the relevant Order Confirmation.

3) **Applicability of Terms.** The Terms govern User's use of Works in connection with the relevant License. In the event of any conflict between General Terms and Order Confirmation Terms, the latter shall govern. User acknowledges that Rightsholders have complete discretion whether to grant any permission, and whether to place any limitations on any grant, and that CCC has no right to supersede or to modify any such discretionary act by a Rightsholder.

4) **Representations; Acceptance.** By using the Service, User represents and warrants that User has been duly authorized by the User to accept, and hereby does accept, all Terms.

5) **Scope of License; Limitations and Obligations.** All Works and all rights therein, including copyright rights, remain the sole and exclusive property of the Rightsholder. The License provides only those rights expressly set forth in the terms and conveys no other rights in any Works

6) **General Payment Terms.** User may pay at time of checkout by credit card or choose to be invoiced. If the User chooses to be invoiced, the User shall: (i) remit payments in the manner identified on specific invoices, (ii) unless otherwise specifically stated in an Order Confirmation or separate written agreement, Users shall remit payments upon receipt of the relevant invoice from CCC, either by delivery or notification of availability of the invoice via the Marketplace platform, and (iii) if the User does not pay the invoice within 30 days of receipt, the User may incur a service charge of 1.5% per month or the maximum rate allowed by applicable law, whichever is less. While User may exercise the rights in the License immediately upon receiving the Order Confirmation, the License is automatically revoked and is null and void, as if it had never been issued, if CCC does not receive complete payment on a timely basis.

7) **General Limits on Use.** Unless otherwise provided in the Order Confirmation, any grant of rights to User (i) involves only the rights set forth in the Terms and does not include subsequent or additional uses, (ii) is non-exclusive and non-transferable, and (iii) is subject to any and all limitations and restrictions (such as, but not limited to, limitations on duration of use or circulation) included in the Terms. Upon completion of the licensed use as set forth in the Order Confirmation, User shall either secure a new permission for further use of the Work(s) or immediately cease any new use of the Work(s) and shall render inaccessible (such as by deleting or by removing or severing links or other locators) any further copies of the Work. User may only make alterations to the Work if and as expressly set forth in the Order Confirmation. No Work may be used in any way that is unlawful, including without limitation if such use would violate applicable sanctions laws or regulations, would be defamatory, violate the rights of third parties (including such third parties' rights of copyright, privacy, publicity, or other tangible or intangible property), or is otherwise illegal, sexually explicit, or obscene. In addition, User may not conjoin a Work with any other material that may result in damage to the reputation of the Rightsholder. Any unlawful use will render any licenses hereunder null and void. User agrees to inform CCC if it becomes aware of any infringement of any rights in a Work and to cooperate with any reasonable request of CCC or the Rightsholder in connection therewith.

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10) **Indemnity.** User hereby indemnifies and agrees to defend the Rightsholder and CCC, and their respective employees and directors, against all claims, liability, damages, costs, and expenses, including legal fees and expenses, arising out of any use of a Work beyond the scope of the rights granted herein and in the Order Confirmation, or any use of a Work which has been altered in any unauthorized way by User, including claims of defamation or infringement of rights of copyright, publicity, privacy, or other tangible or intangible property.

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13) **Effect of Breach.** Any failure by User to pay any amount when due, or any use by User of a Work beyond the scope of the License set forth in the Order Confirmation and/or the Terms, shall be a material breach of such License. Any breach not cured within 10 days of written notice thereof shall result in immediate termination of such License without further notice. Any unauthorized (but licensable) use of a Work that is terminated immediately upon notice thereof may be liquidated by payment of the Rightsholder's ordinary license price therefor; any unauthorized (and unlicensable) use that is not terminated immediately for any reason (including, for example, because materials containing the Work cannot reasonably be recalled) will be subject to all remedies available at law or in equity, but in no event to a payment of less than three times the Rightsholder's ordinary license price for the most closely analogous licensable use plus Rightsholder's and/or CCC's costs and expenses incurred in collecting such payment.

14) **Additional Terms for Specific Products and Services.** If a User is making one of the uses described in this Section 14, the additional terms and conditions apply:

a) ***Print Uses of Academic Course Content and Materials (photocopies for academic coursepacks or classroom handouts).*** For photocopies for academic coursepacks or classroom handouts the following additional terms apply:

i) The copies and anthologies created under this License may be made and assembled by faculty members individually or at their request by on-campus bookstores or copy centers, or by off-campus copy shops and other similar entities.

ii) No License granted shall in any way: (i) include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied) (ii) permit "publishing ventures" where any particular anthology would be systematically marketed at multiple institutions.

iii) Subject to any Publisher Terms (and notwithstanding any apparent contradiction in the Order Confirmation arising from data provided by User), any use authorized under the academic pay-per-use service is limited as follows:

A) any License granted shall apply to only one class (bearing a unique identifier as assigned by the institution, and thereby including all sections or other subparts of the class) at one institution;

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C) use is limited to no more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

D) no User may sell or distribute any particular anthology, whether photocopied or electronic, at more than one institution of learning;

E) in the case of a photocopy permission, no materials may be entered into electronic memory by User except in order to produce an identical copy of a Work before or during the academic term (or analogous period) as to which any particular permission is granted. In the event that User shall choose to retain materials that are the subject of a photocopy permission in electronic memory for purposes of producing identical copies more than one day after such retention (but still within the scope of any permission granted), User must notify CCC of such fact in the applicable permission request and such retention shall constitute one copy actually sold for purposes of calculating permission fees due; and

F) any permission granted shall expire at the end of the class. No permission granted shall in any way include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied).

iv) Books and Records; Right to Audit. As to each permission granted under the academic pay-per-use Service, User shall maintain for at least four full calendar years books and records sufficient for CCC to determine the numbers of copies made by User under such permission. CCC and any representatives it may designate shall have the right to audit such books and records at any time during User's ordinary business hours, upon two days' prior notice. If any such audit shall determine that User shall have underpaid for, or underreported, any photocopies sold or by three percent (3%) or more, then User shall bear all the costs of any such audit; otherwise, CCC shall bear the costs of any such audit. Any amount determined by such audit to have been underpaid by User shall immediately be paid to CCC by User, together with interest thereon at the rate of 10% per annum from the date such amount was originally due. The provisions of this paragraph shall survive the termination of this License for any reason.

**b) *Digital Pay-Per-Uses of Academic Course Content and Materials (e-coursepacks, electronic reserves, learning management systems, academic institution intranets).*** For uses in e-coursepacks, posts in electronic reserves, posts in learning management systems, or posts on academic institution intranets, the following additional terms apply:

i) The pay-per-uses subject to this Section 14(b) include:

A) **Posting e-reserves, course management systems, e-coursepacks for text-based content**, which grants authorizations to import requested material in electronic format, and allows electronic access to this material to members of a designated college or university class, under the direction of an instructor designated by the college or university, accessible only under appropriate electronic controls (e.g., password);

B) **Posting e-reserves, course management systems, e-coursepacks for material consisting of photographs or other still images not embedded in text**, which grants not only the authorizations described in Section 14(b)(i)(A) above, but also the following authorization: to include the requested material in course materials for use consistent with Section 14(b)(i)(A) above, including any necessary resizing, reformatting or modification of the resolution of such requested material (provided that such modification does not alter the underlying editorial content or meaning of the requested material, and provided that the resulting modified content is used solely within the scope of, and in a manner consistent with, the particular authorization described in the Order Confirmation and the Terms), but not including any other form of manipulation, alteration or editing of the requested material;

C) **Posting e-reserves, course management systems, e-coursepacks or other academic distribution for audiovisual content**, which grants not only the authorizations described in Section 14(b)(i)(A) above, but also the following authorizations: (i) to include the requested material in course materials for use consistent with Section 14(b)(i)(A) above; (ii) to display and perform the requested material to such members of such class in the physical classroom or remotely by means of streaming media or other video formats; and (iii) to "clip" or reformat the requested material for purposes of time or content management or ease of delivery, provided that such "clipping" or reformatting does not alter the underlying editorial content or meaning of the requested material and that the resulting material is used solely within the scope of, and in a manner consistent with, the particular authorization described in the Order Confirmation and the Terms. Unless expressly set forth in the relevant Order Confirmation, the License does not authorize any other form of manipulation, alteration or editing of the requested material.

ii) Unless expressly set forth in the relevant Order Confirmation, no License granted shall in any way: (i) include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied or, in the case of Works subject to Sections 14(b)(1)(B) or (C) above, as described in such Sections) (ii) permit "publishing ventures" where any particular course materials would be systematically marketed at multiple institutions.

iii) Subject to any further limitations determined in the Rightsholder Terms (and notwithstanding any apparent contradiction in the Order Confirmation arising from data provided by User), any use authorized under the electronic course content pay-per-use service is limited as follows:

A) any License granted shall apply to only one class (bearing a unique identifier as assigned by the institution, and thereby including all sections or other subparts of the class) at one institution;

B) use is limited to not more than 25% of the text of a book or of the items in a published collection of essays, poems or articles;

C) use is limited to not more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

D) no User may sell or distribute any particular materials, whether photocopied or electronic, at more than one institution of learning;

E) electronic access to material which is the subject of an electronic-use permission must be limited by means of electronic password, student identification or other control permitting access solely to students and instructors in the class;

F) User must ensure (through use of an electronic cover page or other appropriate means) that any person, upon gaining electronic access to the material, which is the subject of a permission, shall see:

- a proper copyright notice, identifying the Rightsholder in whose name CCC has granted permission,
- a statement to the effect that such copy was made pursuant to permission,
- a statement identifying the class to which the material applies and notifying the reader that the material has been made available electronically solely for use in the class, and
- a statement to the effect that the material may not be further distributed to any person outside the class, whether by copying or by transmission and whether electronically or in paper form, and User must also ensure that such cover page or other means will print out in the event that the person accessing the material chooses to print out the material or any part thereof.

G) any permission granted shall expire at the end of the class and, absent some other form of authorization, User is thereupon required to delete the applicable material from any electronic storage or to block electronic access to the applicable material.

iv) Uses of separate portions of a Work, even if they are to be included in the same course material or the same university or college class, require separate permissions under the electronic course content pay-per-use Service. Unless otherwise provided in the Order Confirmation, any grant of rights to User is limited to use completed no later than the end of the academic term (or analogous period) as to which any particular permission is granted.

v) Books and Records; Right to Audit. As to each permission granted under the electronic course content Service, User shall maintain for at least four full calendar years books and records sufficient for CCC to determine the numbers of copies made by User under such permission. CCC and any representatives it may designate shall have the right to audit such books and records at any time during User's ordinary business hours, upon two days' prior notice. If any such audit shall determine that User shall have underpaid for, or underreported, any electronic copies used by three percent (3%) or more, then User shall bear all the costs of any such audit; otherwise, CCC shall bear the costs of any such audit. Any amount determined by such audit to have been underpaid by User shall immediately be paid to CCC by User, together with interest thereon at the rate of 10% per annum from the date such amount was originally due. The provisions of this paragraph shall survive the termination of this license for any reason.



c) ***Pay-Per-Use Permissions for Certain Reproductions (Academic photocopies for library reserves and interlibrary loan reporting) (Non-academic internal/external business uses and commercial document delivery)***. The License expressly excludes the uses listed in Section (c)(i)-(v) below (which must be subject to separate license from the applicable Rightsholder) for: academic photocopies for library reserves and interlibrary loan reporting; and non-academic internal/external business uses and commercial document delivery.

- i) electronic storage of any reproduction (whether in plain-text, PDF, or any other format) other than on a transitory basis;
- ii) the input of Works or reproductions thereof into any computerized database;
- iii) reproduction of an entire Work (cover-to-cover copying) except where the Work is a single article;
- iv) reproduction for resale to anyone other than a specific customer of User;
- v) republication in any different form. Please obtain authorizations for these uses through other CCC services or directly from the rightsholder.

Any license granted is further limited as set forth in any restrictions included in the Order Confirmation and/or in these Terms.

d) ***Electronic Reproductions in Online Environments (Non-Academic-email, intranet, internet and extranet)***. For "electronic reproductions", which generally includes e-mail use (including instant messaging or other electronic transmission to a defined group of recipients) or posting on an intranet, extranet or Intranet site (including any display or performance incidental thereto), the following additional terms apply:

- i) Unless otherwise set forth in the Order Confirmation, the License is limited to use completed within 30 days for any use on the Internet, 60 days for any use on an intranet or extranet and one year for any other use, all as measured from the "republication date" as identified in the Order Confirmation, if any, and otherwise from the date of the Order Confirmation.
- ii) User may not make or permit any alterations to the Work, unless expressly set forth in the Order Confirmation (after request by User and approval by Rightsholder); provided, however, that a Work consisting of photographs or other still images not embedded in text may, if necessary, be resized, reformatted or have its resolution modified without additional express permission, and a Work consisting of audiovisual content may, if necessary, be "clipped" or reformatted for purposes of time or content management or ease of delivery (provided that any such resizing, reformatting, resolution modification or "clipping" does not alter the underlying editorial content or meaning of the Work used, and that the resulting material is used solely within the scope of, and in a manner consistent with, the particular License described in the Order Confirmation and the Terms.

## 15) Miscellaneous.

a) User acknowledges that CCC may, from time to time, make changes or additions to the Service or to the Terms, and that Rightsholder may make changes or additions to the Rightsholder Terms. Such updated Terms will replace the prior terms and conditions in the order workflow and shall be effective as to any subsequent Licenses but shall not apply to Licenses already granted and paid for under a prior set of terms.

b) Use of User-related information collected through the Service is governed by CCC's privacy policy, available online at [www.copyright.com/about/privacy-policy/](http://www.copyright.com/about/privacy-policy/).

c) The License is personal to User. Therefore, User may not assign or transfer to any other person (whether a natural person or an organization of any kind) the License or any rights granted thereunder; provided, however, that, where applicable, User may assign such License in its entirety on written notice to CCC in the event of a transfer of all or substantially all of User's rights in any new material which includes the Work(s) licensed under this Service.

d) No amendment or waiver of any Terms is binding unless set forth in writing and signed by the appropriate parties, including, where applicable, the Rightsholder. The Rightsholder and CCC hereby object to any terms contained in any writing prepared by or on behalf of the User or its principals, employees, agents or affiliates and purporting to govern or otherwise relate to the License described in the Order Confirmation, which terms are in any way inconsistent with any Terms set forth in the Order Confirmation, and/or in CCC's standard operating procedures, whether such writing is prepared prior to, simultaneously with or subsequent to the Order Confirmation, and whether such writing appears on a copy of the Order Confirmation or in a separate instrument.

e) The License described in the Order Confirmation shall be governed by and construed under the law of the State of New York, USA, without regard to the principles thereof of conflicts of law. Any case, controversy, suit, action, or proceeding arising out of, in connection with, or related to such License shall be brought, at CCC's sole discretion, in any federal or state court located in the County of New York, State of New York, USA, or in any federal or state court whose geographical jurisdiction covers the location of the Rightsholder set forth in the Order Confirmation. The parties expressly submit to the personal jurisdiction and venue of each such federal or state court.

*Last updated October 2022*



Marketplace

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Article Title	Effect of operating conditions on carbon corrosion in polymer electrolyte membrane fuel cells	Start Page	575
		End Page	579
		Issue	2
		Volume	193
Date	01/01/1976		
Language	English, French, French, German, German		
Country	Switzerland		
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Title	Effect of Graphitization on PEMFC Catalyst Layer Properties	Institution Name	University of Calgary
		Expected Presentation Date	2023-01-27

Instructor Name

Kunal Karan

## ADDITIONAL DETAILS

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## Marketplace Permissions General Terms and Conditions

The following terms and conditions ("General Terms"), together with any applicable Publisher Terms and Conditions, govern User's use of Works pursuant to the Licenses granted by Copyright Clearance Center, Inc. ("CCC") on behalf of the applicable Rightsholders of such Works through CCC's applicable Marketplace transactional licensing services (each, a "Service").

1) **Definitions.** For purposes of these General Terms, the following definitions apply:

"License" is the licensed use the User obtains via the Marketplace platform in a particular licensing transaction, as set forth in the Order Confirmation.

"Order Confirmation" is the confirmation CCC provides to the User at the conclusion of each Marketplace transaction. "Order Confirmation Terms" are additional terms set forth on specific Order Confirmations not set forth in the General Terms that can include terms applicable to a particular CCC transactional licensing service and/or any Rightsholder-specific terms.

"Rightsholder(s)" are the holders of copyright rights in the Works for which a User obtains licenses via the Marketplace platform, which are displayed on specific Order Confirmations.

"Terms" means the terms and conditions set forth in these General Terms and any additional Order Confirmation Terms collectively.

"User" or "you" is the person or entity making the use granted under the relevant License. Where the person accepting the Terms on behalf of a User is a freelancer or other third party who the User authorized to accept the General Terms on the User's behalf, such person shall be deemed jointly a User for purposes of such Terms.

"Work(s)" are the copyright protected works described in relevant Order Confirmations.

**2) Description of Service.** CCC's Marketplace enables Users to obtain Licenses to use one or more Works in accordance with all relevant Terms. CCC grants Licenses as an agent on behalf of the copyright rightsholder identified in the relevant Order Confirmation.

**3) Applicability of Terms.** The Terms govern User's use of Works in connection with the relevant License. In the event of any conflict between General Terms and Order Confirmation Terms, the latter shall govern. User acknowledges that Rightsholders have complete discretion whether to grant any permission, and whether to place any limitations on any grant, and that CCC has no right to supersede or to modify any such discretionary act by a Rightsholder.

**4) Representations; Acceptance.** By using the Service, User represents and warrants that User has been duly authorized by the User to accept, and hereby does accept, all Terms.

**5) Scope of License; Limitations and Obligations.** All Works and all rights therein, including copyright rights, remain the sole and exclusive property of the Rightsholder. The License provides only those rights expressly set forth in the terms and conveys no other rights in any Works

**6) General Payment Terms.** User may pay at time of checkout by credit card or choose to be invoiced. If the User chooses to be invoiced, the User shall: (i) remit payments in the manner identified on specific invoices, (ii) unless otherwise specifically stated in an Order Confirmation or separate written agreement, Users shall remit payments upon receipt of the relevant invoice from CCC, either by delivery or notification of availability of the invoice via the Marketplace platform, and (iii) if the User does not pay the invoice within 30 days of receipt, the User may incur a service charge of 1.5% per month or the maximum rate allowed by applicable law, whichever is less. While User may exercise the rights in the License immediately upon receiving the Order Confirmation, the License is automatically revoked and is null and void, as if it had never been issued, if CCC does not receive complete payment on a timely basis.

**7) General Limits on Use.** Unless otherwise provided in the Order Confirmation, any grant of rights to User (i) involves only the rights set forth in the Terms and does not include subsequent or additional uses, (ii) is non-exclusive and non-transferable, and (iii) is subject to any and all limitations and restrictions (such as, but not limited to, limitations on duration of use or circulation) included in the Terms. Upon completion of the licensed use as set forth in the Order Confirmation, User shall either secure a new permission for further use of the Work(s) or immediately cease any new use of the Work(s) and shall render inaccessible (such as by deleting or by removing or severing links or other locators) any further copies of the Work. User may only make alterations to the Work if and as expressly set forth in the Order Confirmation. No Work may be used in any way that is unlawful, including without limitation if such use would violate applicable sanctions laws or regulations, would be defamatory, violate the rights of third parties (including such third parties' rights of copyright, privacy, publicity, or other tangible or intangible property), or is otherwise illegal, sexually explicit, or obscene. In addition, User may not conjoin a Work with any other material that may result in damage to the reputation of the Rightsholder. Any unlawful use will render any licenses hereunder null and void. User agrees to inform CCC if it becomes aware of any infringement of any rights in a Work and to cooperate with any reasonable request of CCC or the Rightsholder in connection therewith.

**8) Third Party Materials.** In the event that the material for which a License is sought includes third party materials (such as photographs, illustrations, graphs, inserts and similar materials) that are identified in such material as having been used by permission (or a similar indicator), User is responsible for identifying, and seeking separate licenses (under this Service, if available, or otherwise) for any of such third party materials; without a separate license, User may not use such third party materials via the License.

**9) Copyright Notice.** Use of proper copyright notice for a Work is required as a condition of any License granted under the Service. Unless otherwise provided in the Order Confirmation, a proper copyright notice will read substantially as follows: "Used with permission of [Rightsholder's name], from [Work's title, author, volume, edition number and year of copyright]; permission conveyed through Copyright Clearance Center, Inc." Such notice must be provided in a reasonably legible font size and must be placed either on a cover page or in another location that any person, upon gaining access to the material which is the subject of a permission, shall see, or in the case of republication Licenses, immediately adjacent to the Work as used (for example, as part of a by-line or footnote) or in the place where substantially all other credits or notices for the new work containing the republished Work are located. Failure to include the required notice results in



loss to the Rightsholder and CCC, and the User shall be liable to pay liquidated damages for each such failure equal to twice the use fee specified in the Order Confirmation, in addition to the use fee itself and any other fees and charges specified.

**10) Indemnity.** User hereby indemnifies and agrees to defend the Rightsholder and CCC, and their respective employees and directors, against all claims, liability, damages, costs, and expenses, including legal fees and expenses, arising out of any use of a Work beyond the scope of the rights granted herein and in the Order Confirmation, or any use of a Work which has been altered in any unauthorized way by User, including claims of defamation or infringement of rights of copyright, publicity, privacy, or other tangible or intangible property.

**11) Limitation of Liability.** UNDER NO CIRCUMSTANCES WILL CCC OR THE RIGHTSHOLDER BE LIABLE FOR ANY DIRECT, INDIRECT, CONSEQUENTIAL, OR INCIDENTAL DAMAGES (INCLUDING WITHOUT LIMITATION DAMAGES FOR LOSS OF BUSINESS PROFITS OR INFORMATION, OR FOR BUSINESS INTERRUPTION) ARISING OUT OF THE USE OR INABILITY TO USE A WORK, EVEN IF ONE OR BOTH OF THEM HAS BEEN ADVISED OF THE POSSIBILITY OF SUCH DAMAGES. In any event, the total liability of the Rightsholder and CCC (including their respective employees and directors) shall not exceed the total amount actually paid by User for the relevant License. User assumes full liability for the actions and omissions of its principals, employees, agents, affiliates, successors, and assigns.

**12) Limited Warranties.** THE WORK(S) AND RIGHT(S) ARE PROVIDED "AS IS." CCC HAS THE RIGHT TO GRANT TO USER THE RIGHTS GRANTED IN THE ORDER CONFIRMATION DOCUMENT. CCC AND THE RIGHTSHOLDER DISCLAIM ALL OTHER WARRANTIES RELATING TO THE WORK(S) AND RIGHT(S), EITHER EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION IMPLIED WARRANTIES OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE. ADDITIONAL RIGHTS MAY BE REQUIRED TO USE ILLUSTRATIONS, GRAPHS, PHOTOGRAPHS, ABSTRACTS, INSERTS, OR OTHER PORTIONS OF THE WORK (AS OPPOSED TO THE ENTIRE WORK) IN A MANNER CONTEMPLATED BY USER; USER UNDERSTANDS AND AGREES THAT NEITHER CCC NOR THE RIGHTSHOLDER MAY HAVE SUCH ADDITIONAL RIGHTS TO GRANT.

**13) Effect of Breach.** Any failure by User to pay any amount when due, or any use by User of a Work beyond the scope of the License set forth in the Order Confirmation and/or the Terms, shall be a material breach of such License. Any breach not cured within 10 days of written notice thereof shall result in immediate termination of such License without further notice. Any unauthorized (but licensable) use of a Work that is terminated immediately upon notice thereof may be liquidated by payment of the Rightsholder's ordinary license price therefor; any unauthorized (and unlicensable) use that is not terminated immediately for any reason (including, for example, because materials containing the Work cannot reasonably be recalled) will be subject to all remedies available at law or in equity, but in no event to a payment of less than three times the Rightsholder's ordinary license price for the most closely analogous licensable use plus Rightsholder's and/or CCC's costs and expenses incurred in collecting such payment.

**14) Additional Terms for Specific Products and Services.** If a User is making one of the uses described in this Section 14, the additional terms and conditions apply:

**a) *Print Uses of Academic Course Content and Materials (photocopies for academic coursepacks or classroom handouts).*** For photocopies for academic coursepacks or classroom handouts the following additional terms apply:

i) The copies and anthologies created under this License may be made and assembled by faculty members individually or at their request by on-campus bookstores or copy centers, or by off-campus copy shops and other similar entities.

ii) No License granted shall in any way: (i) include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied) (ii) permit "publishing ventures" where any particular anthology would be systematically marketed at multiple institutions.

iii) Subject to any Publisher Terms (and notwithstanding any apparent contradiction in the Order Confirmation arising from data provided by User), any use authorized under the academic pay-per-use service is limited as follows:

A) any License granted shall apply to only one class (bearing a unique identifier as assigned by the institution, and thereby including all sections or other subparts of the class) at one institution;

B) use is limited to not more than 25% of the text of a book or of the items in a published collection of essays, poems or articles;

C) use is limited to no more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

D) no User may sell or distribute any particular anthology, whether photocopied or electronic, at more than one institution of learning;

E) in the case of a photocopy permission, no materials may be entered into electronic memory by User except in order to produce an identical copy of a Work before or during the academic term (or analogous period) as to which any particular permission is granted. In the event that User shall choose to retain materials that are the subject of a photocopy permission in electronic memory for purposes of producing identical copies more than one day after such retention (but still within the scope of any permission granted), User must notify CCC of such fact in the applicable permission request and such retention shall constitute one copy actually sold for purposes of calculating permission fees due; and

F) any permission granted shall expire at the end of the class. No permission granted shall in any way include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied).

iv) Books and Records; Right to Audit. As to each permission granted under the academic pay-per-use Service, User shall maintain for at least four full calendar years books and records sufficient for CCC to determine the numbers of copies made by User under such permission. CCC and any representatives it may designate shall have the right to audit such books and records at any time during User's ordinary business hours, upon two days' prior notice. If any such audit shall determine that User shall have underpaid for, or underreported, any photocopies sold or by three percent (3%) or more, then User shall bear all the costs of any such audit; otherwise, CCC shall bear the costs of any such audit. Any amount determined by such audit to have been underpaid by User shall immediately be paid to CCC by User, together with interest thereon at the rate of 10% per annum from the date such amount was originally due. The provisions of this paragraph shall survive the termination of this License for any reason.

**b) *Digital Pay-Per-Uses of Academic Course Content and Materials (e-coursepacks, electronic reserves, learning management systems, academic institution intranets).*** For uses in e-coursepacks, posts in electronic reserves, posts in learning management systems, or posts on academic institution intranets, the following additional terms apply:

i) The pay-per-uses subject to this Section 14(b) include:

A) **Posting e-reserves, course management systems, e-coursepacks for text-based content**, which grants authorizations to import requested material in electronic format, and allows electronic access to this material to members of a designated college or university class, under the direction of an instructor designated by the college or university, accessible only under appropriate electronic controls (e.g., password);

B) **Posting e-reserves, course management systems, e-coursepacks for material consisting of photographs or other still images not embedded in text**, which grants not only the authorizations described in Section 14(b)(i)(A) above, but also the following authorization: to include the requested material in course materials for use consistent with Section 14(b)(i)(A) above, including any necessary resizing, reformatting or modification of the resolution of such requested material (provided that such modification does not alter the underlying editorial content or meaning of the requested material, and provided that the resulting modified content is used solely within the scope of, and in a manner consistent with, the particular authorization described in the Order Confirmation and the Terms), but not including any other form of manipulation, alteration or editing of the requested material;

C) **Posting e-reserves, course management systems, e-coursepacks or other academic distribution for audiovisual content**, which grants not only the authorizations described in Section 14(b)(i)(A) above, but also the following authorizations: (i) to include the requested material in course materials for use consistent with Section 14(b)(i)(A) above; (ii) to display and perform the requested material to such members of such class in the physical classroom or remotely by means of streaming media or other video formats; and (iii) to "clip" or reformat the requested material for purposes of time or content management or ease of delivery, provided that such "clipping" or reformatting does not alter the underlying editorial content or meaning of the requested material and that the resulting material is used solely within the scope of, and in a manner consistent with, the particular authorization described in the Order Confirmation and the Terms. Unless

expressly set forth in the relevant Order Confirmation, the License does not authorize any other form of manipulation, alteration or editing of the requested material.

ii) Unless expressly set forth in the relevant Order Confirmation, no License granted shall in any way: (i) include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied or, in the case of Works subject to Sections 14(b)(1)(B) or (C) above, as described in such Sections) (ii) permit "publishing ventures" where any particular course materials would be systematically marketed at multiple institutions.

iii) Subject to any further limitations determined in the Rightsholder Terms (and notwithstanding any apparent contradiction in the Order Confirmation arising from data provided by User), any use authorized under the electronic course content pay-per-use service is limited as follows:

A) any License granted shall apply to only one class (bearing a unique identifier as assigned by the institution, and thereby including all sections or other subparts of the class) at one institution;

B) use is limited to not more than 25% of the text of a book or of the items in a published collection of essays, poems or articles;

C) use is limited to not more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

D) no User may sell or distribute any particular materials, whether photocopied or electronic, at more than one institution of learning;

E) electronic access to material which is the subject of an electronic-use permission must be limited by means of electronic password, student identification or other control permitting access solely to students and instructors in the class;

F) User must ensure (through use of an electronic cover page or other appropriate means) that any person, upon gaining electronic access to the material, which is the subject of a permission, shall see:

- a proper copyright notice, identifying the Rightsholder in whose name CCC has granted permission,
- a statement to the effect that such copy was made pursuant to permission,
- a statement identifying the class to which the material applies and notifying the reader that the material has been made available electronically solely for use in the class, and
- a statement to the effect that the material may not be further distributed to any person outside the class, whether by copying or by transmission and whether electronically or in paper form, and User must also ensure that such cover page or other means will print out in the event that the person accessing the material chooses to print out the material or any part thereof.

G) any permission granted shall expire at the end of the class and, absent some other form of authorization, User is thereupon required to delete the applicable material from any electronic storage or to block electronic access to the applicable material.

iv) Uses of separate portions of a Work, even if they are to be included in the same course material or the same university or college class, require separate permissions under the electronic course content pay-per-use Service. Unless otherwise provided in the Order Confirmation, any grant of rights to User is limited to use completed no later than the end of the academic term (or analogous period) as to which any particular permission is granted.

v) Books and Records; Right to Audit. As to each permission granted under the electronic course content Service, User shall maintain for at least four full calendar years books and records sufficient for CCC to determine the numbers of copies made by User under such permission. CCC and any representatives it may designate shall have the right to audit such books and records at any time during User's ordinary business hours, upon two days' prior notice. If any such audit shall determine that User shall have underpaid for, or underreported, any electronic copies used by three percent (3%) or more, then User shall bear all the costs of any such audit; otherwise, CCC shall bear the costs of any such audit. Any amount determined by such audit to have been underpaid by User



shall immediately be paid to CCC by User, together with interest thereon at the rate of 10% per annum from the date such amount was originally due. The provisions of this paragraph shall survive the termination of this license for any reason.

c) ***Pay-Per-Use Permissions for Certain Reproductions (Academic photocopies for library reserves and interlibrary loan reporting) (Non-academic internal/external business uses and commercial document delivery)***. The License expressly excludes the uses listed in Section (c)(i)-(v) below (which must be subject to separate license from the applicable Rightsholder) for: academic photocopies for library reserves and interlibrary loan reporting; and non-academic internal/external business uses and commercial document delivery.

- i) electronic storage of any reproduction (whether in plain-text, PDF, or any other format) other than on a transitory basis;
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- v) republication in any different form. Please obtain authorizations for these uses through other CCC services or directly from the rightsholder.

Any license granted is further limited as set forth in any restrictions included in the Order Confirmation and/or in these Terms.

d) ***Electronic Reproductions in Online Environments (Non-Academic-email, intranet, internet and extranet)***. For "electronic reproductions", which generally includes e-mail use (including instant messaging or other electronic transmission to a defined group of recipients) or posting on an intranet, extranet or Intranet site (including any display or performance incidental thereto), the following additional terms apply:

- i) Unless otherwise set forth in the Order Confirmation, the License is limited to use completed within 30 days for any use on the Internet, 60 days for any use on an intranet or extranet and one year for any other use, all as measured from the "republication date" as identified in the Order Confirmation, if any, and otherwise from the date of the Order Confirmation.
- ii) User may not make or permit any alterations to the Work, unless expressly set forth in the Order Confirmation (after request by User and approval by Rightsholder); provided, however, that a Work consisting of photographs or other still images not embedded in text may, if necessary, be resized, reformatted or have its resolution modified without additional express permission, and a Work consisting of audiovisual content may, if necessary, be "clipped" or reformatted for purposes of time or content management or ease of delivery (provided that any such resizing, reformatting, resolution modification or "clipping" does not alter the underlying editorial content or meaning of the Work used, and that the resulting material is used solely within the scope of, and in a manner consistent with, the particular License described in the Order Confirmation and the Terms.

## 15) Miscellaneous.

a) User acknowledges that CCC may, from time to time, make changes or additions to the Service or to the Terms, and that Rightsholder may make changes or additions to the Rightsholder Terms. Such updated Terms will replace the prior terms and conditions in the order workflow and shall be effective as to any subsequent Licenses but shall not apply to Licenses already granted and paid for under a prior set of terms.

b) Use of User-related information collected through the Service is governed by CCC's privacy policy, available online at [www.copyright.com/about/privacy-policy/](http://www.copyright.com/about/privacy-policy/).

c) The License is personal to User. Therefore, User may not assign or transfer to any other person (whether a natural person or an organization of any kind) the License or any rights granted thereunder; provided, however, that, where applicable, User may assign such License in its entirety on written notice to CCC in the event of a transfer of all or substantially all of User's rights in any new material which includes the Work(s) licensed under this Service.

d) No amendment or waiver of any Terms is binding unless set forth in writing and signed by the appropriate parties, including, where applicable, the Rightsholder. The Rightsholder and CCC hereby object to any terms contained in any writing prepared by or on behalf of the User or its principals, employees, agents or affiliates and purporting to govern

or otherwise relate to the License described in the Order Confirmation, which terms are in any way inconsistent with any Terms set forth in the Order Confirmation, and/or in CCC's standard operating procedures, whether such writing is prepared prior to, simultaneously with or subsequent to the Order Confirmation, and whether such writing appears on a copy of the Order Confirmation or in a separate instrument.

e) The License described in the Order Confirmation shall be governed by and construed under the law of the State of New York, USA, without regard to the principles thereof of conflicts of law. Any case, controversy, suit, action, or proceeding arising out of, in connection with, or related to such License shall be brought, at CCC's sole discretion, in any federal or state court located in the County of New York, State of New York, USA, or in any federal or state court whose geographical jurisdiction covers the location of the Rightsholder set forth in the Order Confirmation. The parties expressly submit to the personal jurisdiction and venue of each such federal or state court.

*Last updated October 2022*



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<b>Order Date</b>	21-Dec-2022	<b>Type of Use</b>	Republish in a
<b>Order License ID</b>	1303475-1		thesis/dissertation
<b>ISSN</b>	0013-4686	<b>Publisher Portion</b>	PERGAMON
			Chart/graph/table/figure

## LICENSED CONTENT

<b>Publication Title</b>	Electrochimica acta	<b>Rightsholder</b>	Elsevier Science & Technology Journals
<b>Article Title</b>	Corrosion resistance and sintering effect of carbon supports in polymer electrolyte membrane fuel cells	<b>Publication Type</b>	Journal
		<b>Start Page</b>	6515
		<b>End Page</b>	6521
<b>Author/Editor</b>	INTERNATIONAL SOCIETY OF ELECTROCHEMISTRY.	<b>Issue</b>	26
		<b>Volume</b>	54
<b>Date</b>	01/01/1959		
<b>Language</b>	English, French, German		
<b>Country</b>	United Kingdom of Great Britain and Northern Ireland		

## REQUEST DETAILS

<b>Portion Type</b>	Chart/graph/table/figure	<b>Distribution</b>	Worldwide
<b>Number of Charts / Graphs / Tables / Figures Requested</b>	2	<b>Translation</b>	Original language of publication
<b>Format (select all that apply)</b>	Print, Electronic	<b>Copies for the Disabled?</b>	Yes
<b>Who Will Republish the Content?</b>	Academic institution	<b>Minor Editing Privileges?</b>	Yes
<b>Duration of Use</b>	Life of current edition	<b>Incidental Promotional Use?</b>	Yes
<b>Lifetime Unit Quantity</b>	Up to 44,999	<b>Currency</b>	CAD
<b>Rights Requested</b>	Main product and any product related to main product		

## NEW WORK DETAILS

<b>Title</b>	Effect of Graphitization on PEMFC Catalyst Layer Properties	<b>Institution Name</b>	University of Calgary
		<b>Expected Presentation Date</b>	2023-01-27
<b>Instructor Name</b>	Kunal Karan		

## ADDITIONAL DETAILS

<b>Order Reference Number</b>	N/A	<b>The Requesting Person/Organization to Appear on the License</b>	Sushmit Poojary
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## REQUESTED CONTENT DETAILS

<b>Title, Description or Numeric Reference of the Portion(s)</b>	Effect of operating conditions on carbon corrosion in polymer electrolyte membrane fuel cells	<b>Title of the Article/Chapter the Portion Is From</b>	Corrosion resistance and sintering effect of carbon supports in polymer electrolyte membrane fuel cells
<b>Editor of Portion(s)</b>	Oh, Hyung-Suk; Lim, Katie Heeyum; Roh, Bumwook; Hwang, Inchul; Kim, Hansung	<b>Author of Portion(s)</b>	Oh, Hyung-Suk; Lim, Katie Heeyum; Roh, Bumwook; Hwang, Inchul; Kim, Hansung
<b>Volume of Serial or Monograph</b>	54	<b>Issue, if Republishing an Article From a Serial</b>	26
<b>Page or Page Range of Portion</b>	6515-6521	<b>Publication Date of Portion</b>	2009-10-31

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"Order Confirmation" is the confirmation CCC provides to the User at the conclusion of each Marketplace transaction. "Order Confirmation Terms" are additional terms set forth on specific Order Confirmations not set forth in the General Terms that can include terms applicable to a particular CCC transactional licensing service and/or any Rightsholder-specific terms.

"Rightsholder(s)" are the holders of copyright rights in the Works for which a User obtains licenses via the Marketplace platform, which are displayed on specific Order Confirmations.

"Terms" means the terms and conditions set forth in these General Terms and any additional Order Confirmation Terms collectively.

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"Work(s)" are the copyright protected works described in relevant Order Confirmations.

**2) Description of Service.** CCC's Marketplace enables Users to obtain Licenses to use one or more Works in accordance with all relevant Terms. CCC grants Licenses as an agent on behalf of the copyright rightsholder identified in the relevant Order Confirmation.

**3) Applicability of Terms.** The Terms govern User's use of Works in connection with the relevant License. In the event of any conflict between General Terms and Order Confirmation Terms, the latter shall govern. User acknowledges that Rightsholders have complete discretion whether to grant any permission, and whether to place any limitations on any grant, and that CCC has no right to supersede or to modify any such discretionary act by a Rightsholder.

**4) Representations; Acceptance.** By using the Service, User represents and warrants that User has been duly authorized by the User to accept, and hereby does accept, all Terms.

**5) Scope of License; Limitations and Obligations.** All Works and all rights therein, including copyright rights, remain the sole and exclusive property of the Rightsholder. The License provides only those rights expressly set forth in the terms and conveys no other rights in any Works

**6) General Payment Terms.** User may pay at time of checkout by credit card or choose to be invoiced. If the User chooses to be invoiced, the User shall: (i) remit payments in the manner identified on specific invoices, (ii) unless otherwise specifically stated in an Order Confirmation or separate written agreement, Users shall remit payments upon receipt of the relevant invoice from CCC, either by delivery or notification of availability of the invoice via the Marketplace platform, and (iii) if the User does not pay the invoice within 30 days of receipt, the User may incur a service charge of 1.5% per month or the maximum rate allowed by applicable law, whichever is less. While User may exercise the rights in the License immediately upon receiving the Order Confirmation, the License is automatically revoked and is null and void, as if it had never been issued, if CCC does not receive complete payment on a timely basis.

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**14) Additional Terms for Specific Products and Services.** If a User is making one of the uses described in this Section 14, the additional terms and conditions apply:

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i) The copies and anthologies created under this License may be made and assembled by faculty members individually or at their request by on-campus bookstores or copy centers, or by off-campus copy shops and other similar entities.

ii) No License granted shall in any way: (i) include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied) (ii) permit "publishing ventures" where any particular anthology would be systematically marketed at multiple institutions.

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A) any License granted shall apply to only one class (bearing a unique identifier as assigned by the institution, and thereby including all sections or other subparts of the class) at one institution;

B) use is limited to not more than 25% of the text of a book or of the items in a published collection of essays, poems or articles;

C) use is limited to no more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

D) no User may sell or distribute any particular anthology, whether photocopied or electronic, at more than one institution of learning;

E) in the case of a photocopy permission, no materials may be entered into electronic memory by User except in order to produce an identical copy of a Work before or during the academic term (or analogous period) as to which any particular permission is granted. In the event that User shall choose to retain materials that are the subject of a photocopy permission in electronic memory for purposes of producing identical copies more than one day after such retention (but still within the scope of any permission granted), User must notify CCC of such fact in the applicable permission request and such retention shall constitute one copy actually sold for purposes of calculating permission fees due; and

F) any permission granted shall expire at the end of the class. No permission granted shall in any way include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied).

iv) Books and Records; Right to Audit. As to each permission granted under the academic pay-per-use Service, User shall maintain for at least four full calendar years books and records sufficient for CCC to determine the numbers of copies made by User under such permission. CCC and any representatives it may designate shall have the right to audit such books and records at any time during User's ordinary business hours, upon two days' prior notice. If any such audit shall determine that User shall have underpaid for, or underreported, any photocopies sold or by three percent (3%) or more, then User shall bear all the costs of any such audit; otherwise, CCC shall bear the costs of any such audit. Any amount determined by such audit to have been underpaid by User shall immediately be paid to CCC by User, together with interest thereon at the rate of 10% per annum from the date such amount was originally due. The provisions of this paragraph shall survive the termination of this License for any reason.

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B) **Posting e-reserves, course management systems, e-coursepacks for material consisting of photographs or other still images not embedded in text**, which grants not only the authorizations described in Section 14(b)(i)(A) above, but also the following authorization: to include the requested material in course materials for use consistent with Section 14(b)(i)(A) above, including any necessary resizing, reformatting or modification of the resolution of such requested material (provided that such modification does not alter the underlying editorial content or meaning of the requested material, and provided that the resulting modified content is used solely within the scope of, and in a manner consistent with, the particular authorization described in the Order Confirmation and the Terms), but not including any other form of manipulation, alteration or editing of the requested material;

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ii) Unless expressly set forth in the relevant Order Confirmation, no License granted shall in any way: (i) include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied or, in the case of Works subject to Sections 14(b)(1)(B) or (C) above, as described in such Sections) (ii) permit "publishing ventures" where any particular course materials would be systematically marketed at multiple institutions.

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B) use is limited to not more than 25% of the text of a book or of the items in a published collection of essays, poems or articles;

C) use is limited to not more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

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- ii) User may not make or permit any alterations to the Work, unless expressly set forth in the Order Confirmation (after request by User and approval by Rightsholder); provided, however, that a Work consisting of photographs or other still images not embedded in text may, if necessary, be resized, reformatted or have its resolution modified without additional express permission, and a Work consisting of audiovisual content may, if necessary, be "clipped" or reformatted for purposes of time or content management or ease of delivery (provided that any such resizing, reformatting, resolution modification or "clipping" does not alter the underlying editorial content or meaning of the Work used, and that the resulting material is used solely within the scope of, and in a manner consistent with, the particular License described in the Order Confirmation and the Terms.

## 15) Miscellaneous.

a) User acknowledges that CCC may, from time to time, make changes or additions to the Service or to the Terms, and that Rightsholder may make changes or additions to the Rightsholder Terms. Such updated Terms will replace the prior terms and conditions in the order workflow and shall be effective as to any subsequent Licenses but shall not apply to Licenses already granted and paid for under a prior set of terms.

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*Last updated October 2022*



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Order Date	21-Dec-2022	Type of Use	Republish in a
Order License ID	1303478-1		thesis/dissertation
ISSN	2046-2069	Publisher Portion	RSC Publishing
			Image/photo/illustration

## LICENSED CONTENT

Publication Title	RSC advances	Publication Type	e-Journal
Article Title	Enhanced PEMFC durability with graphitized carbon black cathode catalyst supports under accelerated stress testing	Start Page	19417
		End Page	19425
		Issue	32
		Volume	11
Date	01/01/2011	URL	http://pubs.rsc.org/en/Journals/JournalIssues/RA
Language	English		
Country	United Kingdom of Great Britain and Northern Ireland		
Rightsholder	Royal Society of Chemistry		

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Portion Type	Image/photo/illustration	Distribution	Worldwide
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Duration of Use	Life of current edition	Incidental Promotional Use?	Yes
Lifetime Unit Quantity	Up to 99,999	Currency	CAD
Rights Requested	Main product and any product related to main product		

## NEW WORK DETAILS

Title	Effect of Graphitization on PEMFC Catalyst Layer Properties	Institution Name	University of Calgary
		Expected Presentation Date	2023-01-27
Instructor Name	Kunal Karan		

## ADDITIONAL DETAILS

Order Reference Number	N/A	The Requesting Person/Organization to Appear on the License	Sushmit Poojary
------------------------	-----	-------------------------------------------------------------	-----------------

## REQUESTED CONTENT DETAILS

Title, Description or Numeric Reference of the Portion(s)	Figure 4d. ECSA decay rates	Title of the Article/Chapter the Portion Is From	Enhanced PEMFC durability with graphitized carbon black cathode catalyst supports under accelerated stress testing
Editor of Portion(s)	Xue, Qiong; Huang, Jian-biao; Yang, Dai-jun; Li, Bing; Zhang, Cun-man	Author of Portion(s)	Xue, Qiong; Huang, Jian-biao; Yang, Dai-jun; Li, Bing; Zhang, Cun-man
Volume of Serial or Monograph	11		
Page or Page Range of Portion	19417-19425	Issue, if Republishing an Article From a Serial	32
		Publication Date of Portion	2021-05-26

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C) use is limited to no more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

D) no User may sell or distribute any particular anthology, whether photocopied or electronic, at more than one institution of learning;

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C) use is limited to not more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

D) no User may sell or distribute any particular materials, whether photocopied or electronic, at more than one institution of learning;

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d) No amendment or waiver of any Terms is binding unless set forth in writing and signed by the appropriate parties, including, where applicable, the Rightsholder. The Rightsholder and CCC hereby object to any terms contained in any writing prepared by or on behalf of the User or its principals, employees, agents or affiliates and purporting to govern or otherwise relate to the License described in the Order Confirmation, which terms are in any way inconsistent with any Terms set forth in the Order Confirmation, and/or in CCC's standard operating procedures, whether such writing is prepared prior to, simultaneously with or subsequent to the Order Confirmation, and whether such writing appears on a copy of the Order Confirmation or in a separate instrument.

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*Last updated October 2022*



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Order Date	21-Dec-2022	Type of Use	Republish in a
Order License ID	1303479-1		thesis/dissertation
ISSN	0360-5442	Publisher Portion	PERGAMON
			Chart/graph/table/figure

## LICENSED CONTENT

Publication Title	Energy : technologies, resources, reserves, demands, impact, conservation, management, policy	Rightsholder	Elsevier Science & Technology Journals
		Publication Type	Journal
		Start Page	120318
Article Title	A structured catalyst support combining electrochemically exfoliated graphene oxide and carbon black for enhanced performance and durability in low-temperature hydrogen fuel cells	Volume	226
		URL	<a href="http://www.journals.elsevier.com/energy/">http://www.journals.elsevier.com/energy/</a>
Date	01/01/1976		
Language	English		
Country	United Kingdom of Great Britain and Northern Ireland		

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Portion Type	Chart/graph/table/figure	Distribution	Worldwide
Number of Charts / Graphs / Tables / Figures Requested	3	Translation	Original language of publication
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Duration of Use	Life of current edition	Incidental Promotional Use?	Yes
Lifetime Unit Quantity	Up to 44,999	Currency	CAD
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## NEW WORK DETAILS

<b>Title</b>	Effect of Graphitization on PEMFC Catalyst Layer Properties	<b>Institution Name</b>	University of Calgary
		<b>Expected Presentation Date</b>	2023-01-27
<b>Instructor Name</b>	Kunal Karan		

## ADDITIONAL DETAILS

<b>Order Reference Number</b>	N/A	<b>The Requesting Person/Organization to Appear on the License</b>	Sushmit Poojary
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## REQUESTED CONTENT DETAILS

<b>Title, Description or Numeric Reference of the Portion(s)</b>	Figure 7 and 9	<b>Title of the Article/Chapter the Portion Is From</b>	A structured catalyst support combining electrochemically exfoliated graphene oxide and carbon black for enhanced performance and durability in low-temperature hydrogen fuel cells
<b>Editor of Portion(s)</b>	Ji, Zhaoqi; Perez-Page, Maria; Chen, Jianuo; Rodriguez, Romeo Gonzalez; Cai, Rongsheng; Haigh, Sarah J.; Holmes, Stuart M.		
<b>Volume of Serial or Monograph</b>	226	<b>Author of Portion(s)</b>	Ji, Zhaoqi; Perez-Page, Maria; Chen, Jianuo; Rodriguez, Romeo Gonzalez; Cai, Rongsheng; Haigh, Sarah J.; Holmes, Stuart M.
<b>Page or Page Range of Portion</b>	120318		
		<b>Issue, if Republishing an Article From a Serial</b>	N/A
		<b>Publication Date of Portion</b>	2021-06-30

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The following terms and conditions ("General Terms"), together with any applicable Publisher Terms and Conditions, govern User's use of Works pursuant to the Licenses granted by Copyright Clearance Center, Inc. ("CCC") on behalf of the applicable Rightsholders of such Works through CCC's applicable Marketplace transactional licensing services (each, a "Service").

1) **Definitions.** For purposes of these General Terms, the following definitions apply:

“License” is the licensed use the User obtains via the Marketplace platform in a particular licensing transaction, as set forth in the Order Confirmation.

“Order Confirmation” is the confirmation CCC provides to the User at the conclusion of each Marketplace transaction. “Order Confirmation Terms” are additional terms set forth on specific Order Confirmations not set forth in the General Terms that can include terms applicable to a particular CCC transactional licensing service and/or any Rightsholder-specific terms.

“Rightsholder(s)” are the holders of copyright rights in the Works for which a User obtains licenses via the Marketplace platform, which are displayed on specific Order Confirmations.

“Terms” means the terms and conditions set forth in these General Terms and any additional Order Confirmation Terms collectively.

“User” or “you” is the person or entity making the use granted under the relevant License. Where the person accepting the Terms on behalf of a User is a freelancer or other third party who the User authorized to accept the General Terms on the User’s behalf, such person shall be deemed jointly a User for purposes of such Terms.

“Work(s)” are the copyright protected works described in relevant Order Confirmations.

**2) Description of Service.** CCC’s Marketplace enables Users to obtain Licenses to use one or more Works in accordance with all relevant Terms. CCC grants Licenses as an agent on behalf of the copyright rightsholder identified in the relevant Order Confirmation.

**3) Applicability of Terms.** The Terms govern User’s use of Works in connection with the relevant License. In the event of any conflict between General Terms and Order Confirmation Terms, the latter shall govern. User acknowledges that Rightsholders have complete discretion whether to grant any permission, and whether to place any limitations on any grant, and that CCC has no right to supersede or to modify any such discretionary act by a Rightsholder.

**4) Representations; Acceptance.** By using the Service, User represents and warrants that User has been duly authorized by the User to accept, and hereby does accept, all Terms.

**5) Scope of License; Limitations and Obligations.** All Works and all rights therein, including copyright rights, remain the sole and exclusive property of the Rightsholder. The License provides only those rights expressly set forth in the terms and conveys no other rights in any Works

**6) General Payment Terms.** User may pay at time of checkout by credit card or choose to be invoiced. If the User chooses to be invoiced, the User shall: (i) remit payments in the manner identified on specific invoices, (ii) unless otherwise specifically stated in an Order Confirmation or separate written agreement, Users shall remit payments upon receipt of the relevant invoice from CCC, either by delivery or notification of availability of the invoice via the Marketplace platform, and (iii) if the User does not pay the invoice within 30 days of receipt, the User may incur a service charge of 1.5% per month or the maximum rate allowed by applicable law, whichever is less. While User may exercise the rights in the License immediately upon receiving the Order Confirmation, the License is automatically revoked and is null and void, as if it had never been issued, if CCC does not receive complete payment on a timely basis.

**7) General Limits on Use.** Unless otherwise provided in the Order Confirmation, any grant of rights to User (i) involves only the rights set forth in the Terms and does not include subsequent or additional uses, (ii) is non-exclusive and non-transferable, and (iii) is subject to any and all limitations and restrictions (such as, but not limited to, limitations on duration of use or circulation) included in the Terms. Upon completion of the licensed use as set forth in the Order Confirmation, User shall either secure a new permission for further use of the Work(s) or immediately cease any new use of the Work(s) and shall render inaccessible (such as by deleting or by removing or severing links or other locators) any further copies of the Work. User may only make alterations to the Work if and as expressly set forth in the Order Confirmation. No Work may be used in any way that is unlawful, including without limitation if such use would violate applicable sanctions laws or regulations, would be defamatory, violate the rights of third parties (including such third parties’ rights of copyright, privacy, publicity, or other tangible or intangible property), or is otherwise illegal, sexually explicit, or obscene. In addition, User may not conjoin a Work with any other material that may result in damage to the reputation of the Rightsholder. Any unlawful use will render any licenses hereunder null and void. User agrees to inform CCC if it becomes aware of any infringement of any rights in a Work and to cooperate with any reasonable request of CCC or the Rightsholder in connection therewith.



8) **Third Party Materials.** In the event that the material for which a License is sought includes third party materials (such as photographs, illustrations, graphs, inserts and similar materials) that are identified in such material as having been used by permission (or a similar indicator), User is responsible for identifying, and seeking separate licenses (under this Service, if available, or otherwise) for any of such third party materials; without a separate license, User may not use such third party materials via the License.

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10) **Indemnity.** User hereby indemnifies and agrees to defend the Rightsholder and CCC, and their respective employees and directors, against all claims, liability, damages, costs, and expenses, including legal fees and expenses, arising out of any use of a Work beyond the scope of the rights granted herein and in the Order Confirmation, or any use of a Work which has been altered in any unauthorized way by User, including claims of defamation or infringement of rights of copyright, publicity, privacy, or other tangible or intangible property.

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13) **Effect of Breach.** Any failure by User to pay any amount when due, or any use by User of a Work beyond the scope of the License set forth in the Order Confirmation and/or the Terms, shall be a material breach of such License. Any breach not cured within 10 days of written notice thereof shall result in immediate termination of such License without further notice. Any unauthorized (but licensable) use of a Work that is terminated immediately upon notice thereof may be liquidated by payment of the Rightsholder's ordinary license price therefor; any unauthorized (and unlicensable) use that is not terminated immediately for any reason (including, for example, because materials containing the Work cannot reasonably be recalled) will be subject to all remedies available at law or in equity, but in no event to a payment of less than three times the Rightsholder's ordinary license price for the most closely analogous licensable use plus Rightsholder's and/or CCC's costs and expenses incurred in collecting such payment.

14) **Additional Terms for Specific Products and Services.** If a User is making one of the uses described in this Section 14, the additional terms and conditions apply:

a) ***Print Uses of Academic Course Content and Materials (photocopies for academic coursepacks or classroom handouts).*** For photocopies for academic coursepacks or classroom handouts the following additional terms apply:

i) The copies and anthologies created under this License may be made and assembled by faculty members individually or at their request by on-campus bookstores or copy centers, or by off-campus copy shops and other similar entities.



ii) No License granted shall in any way: (i) include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied) (ii) permit "publishing ventures" where any particular anthology would be systematically marketed at multiple institutions.

iii) Subject to any Publisher Terms (and notwithstanding any apparent contradiction in the Order Confirmation arising from data provided by User), any use authorized under the academic pay-per-use service is limited as follows:

A) any License granted shall apply to only one class (bearing a unique identifier as assigned by the institution, and thereby including all sections or other subparts of the class) at one institution;

B) use is limited to not more than 25% of the text of a book or of the items in a published collection of essays, poems or articles;

C) use is limited to no more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

D) no User may sell or distribute any particular anthology, whether photocopied or electronic, at more than one institution of learning;

E) in the case of a photocopy permission, no materials may be entered into electronic memory by User except in order to produce an identical copy of a Work before or during the academic term (or analogous period) as to which any particular permission is granted. In the event that User shall choose to retain materials that are the subject of a photocopy permission in electronic memory for purposes of producing identical copies more than one day after such retention (but still within the scope of any permission granted), User must notify CCC of such fact in the applicable permission request and such retention shall constitute one copy actually sold for purposes of calculating permission fees due; and

F) any permission granted shall expire at the end of the class. No permission granted shall in any way include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied).

iv) Books and Records; Right to Audit. As to each permission granted under the academic pay-per-use Service, User shall maintain for at least four full calendar years books and records sufficient for CCC to determine the numbers of copies made by User under such permission. CCC and any representatives it may designate shall have the right to audit such books and records at any time during User's ordinary business hours, upon two days' prior notice. If any such audit shall determine that User shall have underpaid for, or underreported, any photocopies sold or by three percent (3%) or more, then User shall bear all the costs of any such audit; otherwise, CCC shall bear the costs of any such audit. Any amount determined by such audit to have been underpaid by User shall immediately be paid to CCC by User, together with interest thereon at the rate of 10% per annum from the date such amount was originally due. The provisions of this paragraph shall survive the termination of this License for any reason.

b) ***Digital Pay-Per-Uses of Academic Course Content and Materials (e-coursepacks, electronic reserves, learning management systems, academic institution intranets).*** For uses in e-coursepacks, posts in electronic reserves, posts in learning management systems, or posts on academic institution intranets, the following additional terms apply:

i) The pay-per-uses subject to this Section 14(b) include:

A) **Posting e-reserves, course management systems, e-coursepacks for text-based content**, which grants authorizations to import requested material in electronic format, and allows electronic access to this material to members of a designated college or university class, under the direction of an instructor designated by the college or university, accessible only under appropriate electronic controls (e.g., password);

B) **Posting e-reserves, course management systems, e-coursepacks for material consisting of photographs or other still images not embedded in text**, which grants not only the authorizations described in Section 14(b)(i)(A) above, but also the following authorization: to include the requested material in course materials for use consistent with Section 14(b)(i)(A) above, including any necessary resizing, reformatting or modification of the resolution of such requested material (provided that such modification does not alter the underlying

editorial content or meaning of the requested material, and provided that the resulting modified content is used solely within the scope of, and in a manner consistent with, the particular authorization described in the Order Confirmation and the Terms), but not including any other form of manipulation, alteration or editing of the requested material;

**C) Posting e-reserves, course management systems, e-coursepacks or other academic distribution for audiovisual content**, which grants not only the authorizations described in Section 14(b)(i)(A) above, but also the following authorizations: (i) to include the requested material in course materials for use consistent with Section 14(b)(i)(A) above; (ii) to display and perform the requested material to such members of such class in the physical classroom or remotely by means of streaming media or other video formats; and (iii) to "clip" or reformat the requested material for purposes of time or content management or ease of delivery, provided that such "clipping" or reformatting does not alter the underlying editorial content or meaning of the requested material and that the resulting material is used solely within the scope of, and in a manner consistent with, the particular authorization described in the Order Confirmation and the Terms. Unless expressly set forth in the relevant Order Confirmation, the License does not authorize any other form of manipulation, alteration or editing of the requested material.

ii) Unless expressly set forth in the relevant Order Confirmation, no License granted shall in any way: (i) include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied or, in the case of Works subject to Sections 14(b)(1)(B) or (C) above, as described in such Sections) (ii) permit "publishing ventures" where any particular course materials would be systematically marketed at multiple institutions.

iii) Subject to any further limitations determined in the Rightsholder Terms (and notwithstanding any apparent contradiction in the Order Confirmation arising from data provided by User), any use authorized under the electronic course content pay-per-use service is limited as follows:

A) any License granted shall apply to only one class (bearing a unique identifier as assigned by the institution, and thereby including all sections or other subparts of the class) at one institution;

B) use is limited to not more than 25% of the text of a book or of the items in a published collection of essays, poems or articles;

C) use is limited to not more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

D) no User may sell or distribute any particular materials, whether photocopied or electronic, at more than one institution of learning;

E) electronic access to material which is the subject of an electronic-use permission must be limited by means of electronic password, student identification or other control permitting access solely to students and instructors in the class;

F) User must ensure (through use of an electronic cover page or other appropriate means) that any person, upon gaining electronic access to the material, which is the subject of a permission, shall see:

- a proper copyright notice, identifying the Rightsholder in whose name CCC has granted permission,
- a statement to the effect that such copy was made pursuant to permission,
- a statement identifying the class to which the material applies and notifying the reader that the material has been made available electronically solely for use in the class, and
- a statement to the effect that the material may not be further distributed to any person outside the class, whether by copying or by transmission and whether electronically or in paper form, and User must also ensure that such cover page or other means will print out in the event that the person accessing the material chooses to print out the material or any part thereof.

G) any permission granted shall expire at the end of the class and, absent some other form of authorization, User is thereupon required to delete the applicable material from any electronic storage or to block electronic

access to the applicable material.

iv) Uses of separate portions of a Work, even if they are to be included in the same course material or the same university or college class, require separate permissions under the electronic course content pay-per-use Service. Unless otherwise provided in the Order Confirmation, any grant of rights to User is limited to use completed no later than the end of the academic term (or analogous period) as to which any particular permission is granted.

v) Books and Records; Right to Audit. As to each permission granted under the electronic course content Service, User shall maintain for at least four full calendar years books and records sufficient for CCC to determine the numbers of copies made by User under such permission. CCC and any representatives it may designate shall have the right to audit such books and records at any time during User's ordinary business hours, upon two days' prior notice. If any such audit shall determine that User shall have underpaid for, or underreported, any electronic copies used by three percent (3%) or more, then User shall bear all the costs of any such audit; otherwise, CCC shall bear the costs of any such audit. Any amount determined by such audit to have been underpaid by User shall immediately be paid to CCC by User, together with interest thereon at the rate of 10% per annum from the date such amount was originally due. The provisions of this paragraph shall survive the termination of this license for any reason.

c) ***Pay-Per-Use Permissions for Certain Reproductions (Academic photocopies for library reserves and interlibrary loan reporting) (Non-academic internal/external business uses and commercial document delivery).*** The License expressly excludes the uses listed in Section (c)(i)-(v) below (which must be subject to separate license from the applicable Rightsholder) for: academic photocopies for library reserves and interlibrary loan reporting; and non-academic internal/external business uses and commercial document delivery.

i) electronic storage of any reproduction (whether in plain-text, PDF, or any other format) other than on a transitory basis;

ii) the input of Works or reproductions thereof into any computerized database;

iii) reproduction of an entire Work (cover-to-cover copying) except where the Work is a single article;

iv) reproduction for resale to anyone other than a specific customer of User;

v) republication in any different form. Please obtain authorizations for these uses through other CCC services or directly from the rightsholder.

Any license granted is further limited as set forth in any restrictions included in the Order Confirmation and/or in these Terms.

d) ***Electronic Reproductions in Online Environments (Non-Academic-email, intranet, internet and extranet).*** For "electronic reproductions", which generally includes e-mail use (including instant messaging or other electronic transmission to a defined group of recipients) or posting on an intranet, extranet or Intranet site (including any display or performance incidental thereto), the following additional terms apply:

i) Unless otherwise set forth in the Order Confirmation, the License is limited to use completed within 30 days for any use on the Internet, 60 days for any use on an intranet or extranet and one year for any other use, all as measured from the "republication date" as identified in the Order Confirmation, if any, and otherwise from the date of the Order Confirmation.

ii) User may not make or permit any alterations to the Work, unless expressly set forth in the Order Confirmation (after request by User and approval by Rightsholder); provided, however, that a Work consisting of photographs or other still images not embedded in text may, if necessary, be resized, reformatted or have its resolution modified without additional express permission, and a Work consisting of audiovisual content may, if necessary, be "clipped" or reformatted for purposes of time or content management or ease of delivery (provided that any such resizing, reformatting, resolution modification or "clipping" does not alter the underlying editorial content or meaning of the Work used, and that the resulting material is used solely within the scope of, and in a manner consistent with, the particular License described in the Order Confirmation and the Terms.

## 15) Miscellaneous.

a) User acknowledges that CCC may, from time to time, make changes or additions to the Service or to the Terms, and that Rightsholder may make changes or additions to the Rightsholder Terms. Such updated Terms will replace the

prior terms and conditions in the order workflow and shall be effective as to any subsequent Licenses but shall not apply to Licenses already granted and paid for under a prior set of terms.

b) Use of User-related information collected through the Service is governed by CCC's privacy policy, available online at [www.copyright.com/about/privacy-policy/](http://www.copyright.com/about/privacy-policy/).

c) The License is personal to User. Therefore, User may not assign or transfer to any other person (whether a natural person or an organization of any kind) the License or any rights granted thereunder; provided, however, that, where applicable, User may assign such License in its entirety on written notice to CCC in the event of a transfer of all or substantially all of User's rights in any new material which includes the Work(s) licensed under this Service.

d) No amendment or waiver of any Terms is binding unless set forth in writing and signed by the appropriate parties, including, where applicable, the Rightsholder. The Rightsholder and CCC hereby object to any terms contained in any writing prepared by or on behalf of the User or its principals, employees, agents or affiliates and purporting to govern or otherwise relate to the License described in the Order Confirmation, which terms are in any way inconsistent with any Terms set forth in the Order Confirmation, and/or in CCC's standard operating procedures, whether such writing is prepared prior to, simultaneously with or subsequent to the Order Confirmation, and whether such writing appears on a copy of the Order Confirmation or in a separate instrument.

e) The License described in the Order Confirmation shall be governed by and construed under the law of the State of New York, USA, without regard to the principles thereof of conflicts of law. Any case, controversy, suit, action, or proceeding arising out of, in connection with, or related to such License shall be brought, at CCC's sole discretion, in any federal or state court located in the County of New York, State of New York, USA, or in any federal or state court whose geographical jurisdiction covers the location of the Rightsholder set forth in the Order Confirmation. The parties expressly submit to the personal jurisdiction and venue of each such federal or state court.

*Last updated October 2022*



## Marketplace

This is a License Agreement between Sushmit Poojary ("User") and Copyright Clearance Center, Inc. ("CCC") on behalf of the Rightsholder identified in the order details below. The license consists of the order details, the Marketplace Order General Terms and Conditions below, and any Rightsholder Terms and Conditions which are included below.

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ISSN	0008-6223	Publisher	PERGAMON
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Article Title	Synthesis of a high-temperature stable electrochemically exfoliated graphene	Publication Type	Journal
		Start Page	681
		End Page	692
Author/Editor	AMERICAN CARBON COMMITTEE.	Volume	157
Date	01/01/1963		
Language	English, English, French, German		
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## NEW WORK DETAILS

Title	Effect of Graphitization on PEMFC Catalyst Layer Properties	Institution Name	University of Calgary
		Expected Presentation Date	2023-01-27



Instructor Name

Kunal Karan

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The Requesting  
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Sushmit Poojary

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**4) Representations; Acceptance.** By using the Service, User represents and warrants that User has been duly authorized by the User to accept, and hereby does accept, all Terms.

**5) Scope of License; Limitations and Obligations.** All Works and all rights therein, including copyright rights, remain the sole and exclusive property of the Rightsholder. The License provides only those rights expressly set forth in the terms and conveys no other rights in any Works

**6) General Payment Terms.** User may pay at time of checkout by credit card or choose to be invoiced. If the User chooses to be invoiced, the User shall: (i) remit payments in the manner identified on specific invoices, (ii) unless otherwise specifically stated in an Order Confirmation or separate written agreement, Users shall remit payments upon receipt of the relevant invoice from CCC, either by delivery or notification of availability of the invoice via the Marketplace platform, and (iii) if the User does not pay the invoice within 30 days of receipt, the User may incur a service charge of 1.5% per month or the maximum rate allowed by applicable law, whichever is less. While User may exercise the rights in the License immediately upon receiving the Order Confirmation, the License is automatically revoked and is null and void, as if it had never been issued, if CCC does not receive complete payment on a timely basis.

**7) General Limits on Use.** Unless otherwise provided in the Order Confirmation, any grant of rights to User (i) involves only the rights set forth in the Terms and does not include subsequent or additional uses, (ii) is non-exclusive and non-transferable, and (iii) is subject to any and all limitations and restrictions (such as, but not limited to, limitations on duration of use or circulation) included in the Terms. Upon completion of the licensed use as set forth in the Order Confirmation, User shall either secure a new permission for further use of the Work(s) or immediately cease any new use of the Work(s) and shall render inaccessible (such as by deleting or by removing or severing links or other locators) any further copies of the Work. User may only make alterations to the Work if and as expressly set forth in the Order Confirmation. No Work may be used in any way that is unlawful, including without limitation if such use would violate applicable sanctions laws or regulations, would be defamatory, violate the rights of third parties (including such third parties' rights of copyright, privacy, publicity, or other tangible or intangible property), or is otherwise illegal, sexually explicit, or obscene. In addition, User may not conjoin a Work with any other material that may result in damage to the reputation of the Rightsholder. Any unlawful use will render any licenses hereunder null and void. User agrees to inform CCC if it becomes aware of any infringement of any rights in a Work and to cooperate with any reasonable request of CCC or the Rightsholder in connection therewith.

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**14) Additional Terms for Specific Products and Services.** If a User is making one of the uses described in this Section 14, the additional terms and conditions apply:

**a) *Print Uses of Academic Course Content and Materials (photocopies for academic coursepacks or classroom handouts).*** For photocopies for academic coursepacks or classroom handouts the following additional terms apply:

i) The copies and anthologies created under this License may be made and assembled by faculty members individually or at their request by on-campus bookstores or copy centers, or by off-campus copy shops and other similar entities.

ii) No License granted shall in any way: (i) include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied) (ii) permit "publishing ventures" where any particular anthology would be systematically marketed at multiple institutions.

iii) Subject to any Publisher Terms (and notwithstanding any apparent contradiction in the Order Confirmation arising from data provided by User), any use authorized under the academic pay-per-use service is limited as follows:

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B) use is limited to not more than 25% of the text of a book or of the items in a published collection of essays, poems or articles;

C) use is limited to no more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

D) no User may sell or distribute any particular anthology, whether photocopied or electronic, at more than one institution of learning;

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C) use is limited to not more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

D) no User may sell or distribute any particular materials, whether photocopied or electronic, at more than one institution of learning;

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*Last updated October 2022*

**Order Number:** 1349615**Order Date:** 27 Apr 2023

## Payment Information

Sushmit Poojary  
sushmit.poojary@ucalgary.ca  
**Payment method:** Invoice

**Billing Address:**  
Mr. Sushmit Poojary  
2442 7 Avenue NW  
Calgary, AB T2N1A2  
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**Customer Location:**  
Mr. Sushmit Poojary  
2442 7 Avenue NW  
Calgary, AB T2N1A2  
Canada

## Order Details

### 1. Advanced functional materials

**Billing Status:**  
Open

**Article:** Development and Simulation of Sulfur<sup>2</sup>doped Graphene Supported Platinum with Exemplary Stability and Activity Towards Oxygen Reduction

<b>Order License ID</b>	1349615-1	<b>Type of use</b>	Republish in a thesis/dissertation
<b>Order detail status</b>	Completed	<b>Publisher</b>	WILEY - V C H VERLAG GMBH & CO. KGAA
<b>ISSN</b>	1616-301X	<b>Portion</b>	Chart/graph/table/figure
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<b>Publication Title</b>	Advanced functional materials	<b>Publication Type</b>	Journal
<b>Article Title</b>	Development and Simulation of Sulfur <sup>2</sup> doped Graphene Supported Platinum with Exemplary Stability and Activity Towards Oxygen Reduction	<b>Start Page</b>	4325
		<b>End Page</b>	4336
		<b>Issue</b>	27
		<b>Volume</b>	24
		<b>URL</b>	http://onlinelibrary.wiley.com/journal/10.1002/(ISSN)1616-3028
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<b>Title</b>	Fabrication and Performance of Fuel Cell Catalyst Layer Made with Graphitic and Carbon Black Catalyst Supports	<b>Institution Name</b>	University of Calgary
		<b>Expected Presentation Date</b>	2023-05-01
<b>Instructor Name</b>	Kunal Karan		

## ADDITIONAL DETAILS

**The Requesting Person/Organization to Appear on the License** Sushmit Poojary

## REQUESTED CONTENT DETAILS

<b>Title, Description or Numeric Reference of the Portion(s)</b>	Fig 3. CV curves initially and after ADT for a) Pt/SG, b) Pt/G, c) Pt/C, and d) normalized ECSA remaining throughout ADT.	<b>Title of the Article/Chapter the Portion Is From</b>	Development and Simulation of Sulfur doped Graphene Supported Platinum with Exemplary Stability and Activity Towards Oxygen Reduction
<b>Editor of Portion(s)</b>	Higgins, Drew; Hoque, Md Ariful; Seo, Min Ho; Wang, Rongyue; Hassan, Fathy; Choi, Ja? Yeon; Pritzker, Mark; Yu, Aiping; Zhang, JiuJun; Chen, Zhongwei		



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14) **Additional Terms for Specific Products and Services.** If a User is making one of the uses described in this Section 14, the additional terms and conditions apply:

a) ***Print Uses of Academic Course Content and Materials (photocopies for academic coursepacks or classroom handouts).*** For photocopies for academic coursepacks or classroom handouts the following additional terms apply:

i) The copies and anthologies created under this License may be made and assembled by faculty members individually or at their request by on-campus bookstores or copy centers, or by off-campus copy shops and other similar entities.

ii) No License granted shall in any way: (i) include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied) (ii) permit "publishing ventures" where any particular anthology would be systematically marketed at multiple institutions.

iii) Subject to any Publisher Terms (and notwithstanding any apparent contradiction in the Order Confirmation arising from data provided by User), any use authorized under the academic pay-per-use service is limited as follows:

A) any License granted shall apply to only one class (bearing a unique identifier as assigned by the institution, and thereby including all sections or other subparts of the class) at one institution;

B) use is limited to not more than 25% of the text of a book or of the items in a published collection of essays, poems or articles;

C) use is limited to no more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

D) no User may sell or distribute any particular anthology, whether photocopied or electronic, at more than one institution of learning;

E) in the case of a photocopy permission, no materials may be entered into electronic memory by User except in order to produce an identical copy of a Work before or during the academic term (or analogous period) as to which any particular permission is granted. In the event that User shall choose to retain materials that are the subject of a photocopy permission in electronic memory for purposes of producing identical copies more than one day after such retention (but still within the scope of any permission granted), User must notify CCC of such fact in the applicable permission request and such retention shall constitute one copy actually sold for purposes of calculating permission fees due; and

F) any permission granted shall expire at the end of the class. No permission granted shall in any way include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied).

iv) Books and Records; Right to Audit. As to each permission granted under the academic pay-per-use Service, User shall maintain for at least four full calendar years books and records sufficient for CCC to determine the numbers of copies made by User under such permission. CCC and any representatives it may designate shall have the right to audit such books and records at any time during User's ordinary business hours, upon two days' prior notice. If any such audit shall determine that User shall have underpaid for, or underreported, any photocopies sold or by three percent (3%) or more, then User shall bear all the costs of any such audit; otherwise, CCC shall bear the costs of any such audit. Any amount determined by such audit to have been underpaid by User shall immediately be paid to CCC by User, together with interest thereon at the rate of 10% per annum from the date such amount was originally due. The provisions of this paragraph shall survive the termination of this License for any reason.

**b) *Digital Pay-Per-Uses of Academic Course Content and Materials (e-coursepacks, electronic reserves, learning management systems, academic institution intranets).*** For uses in e-coursepacks, posts in electronic reserves, posts in learning management systems, or posts on academic institution intranets, the following additional terms apply:

i) The pay-per-uses subject to this Section 14(b) include:

A) **Posting e-reserves, course management systems, e-coursepacks for text-based content**, which grants authorizations to import requested material in electronic format, and allows electronic access to this material to members of a designated college or university class, under the direction of an instructor designated by the college or university, accessible only under appropriate electronic controls (e.g., password);

B) **Posting e-reserves, course management systems, e-coursepacks for material consisting of photographs or other still images not embedded in text**, which grants not only the authorizations described in Section 14(b)(i)(A) above, but also the following authorization: to include the requested

material in course materials for use consistent with Section 14(b)(i)(A) above, including any necessary resizing, reformatting or modification of the resolution of such requested material (provided that such modification does not alter the underlying editorial content or meaning of the requested material, and provided that the resulting modified content is used solely within the scope of, and in a manner consistent with, the particular authorization described in the Order Confirmation and the Terms), but not including any other form of manipulation, alteration or editing of the requested material;

**C) Posting e-reserves, course management systems, e-coursepacks or other academic distribution for audiovisual content**, which grants not only the authorizations described in Section 14(b)(i)(A) above, but also the following authorizations: (i) to include the requested material in course materials for use consistent with Section 14(b)(i)(A) above; (ii) to display and perform the requested material to such members of such class in the physical classroom or remotely by means of streaming media or other video formats; and (iii) to "clip" or reformat the requested material for purposes of time or content management or ease of delivery, provided that such "clipping" or reformatting does not alter the underlying editorial content or meaning of the requested material and that the resulting material is used solely within the scope of, and in a manner consistent with, the particular authorization described in the Order Confirmation and the Terms. Unless expressly set forth in the relevant Order Confirmation, the License does not authorize any other form of manipulation, alteration or editing of the requested material.

ii) Unless expressly set forth in the relevant Order Confirmation, no License granted shall in any way: (i) include any right by User to create a substantively non-identical copy of the Work or to edit or in any other way modify the Work (except by means of deleting material immediately preceding or following the entire portion of the Work copied or, in the case of Works subject to Sections 14(b)(1)(B) or (C) above, as described in such Sections) (ii) permit "publishing ventures" where any particular course materials would be systematically marketed at multiple institutions.

iii) Subject to any further limitations determined in the Rightsholder Terms (and notwithstanding any apparent contradiction in the Order Confirmation arising from data provided by User), any use authorized under the electronic course content pay-per-use service is limited as follows:

A) any License granted shall apply to only one class (bearing a unique identifier as assigned by the institution, and thereby including all sections or other subparts of the class) at one institution;

B) use is limited to not more than 25% of the text of a book or of the items in a published collection of essays, poems or articles;

C) use is limited to not more than the greater of (a) 25% of the text of an issue of a journal or other periodical or (b) two articles from such an issue;

D) no User may sell or distribute any particular materials, whether photocopied or electronic, at more than one institution of learning;

E) electronic access to material which is the subject of an electronic-use permission must be limited by means of electronic password, student identification or other control permitting access solely to students and instructors in the class;

F) User must ensure (through use of an electronic cover page or other appropriate means) that any person, upon gaining electronic access to the material, which is the subject of a permission, shall see:

- a proper copyright notice, identifying the Rightsholder in whose name CCC has granted permission,
- a statement to the effect that such copy was made pursuant to permission,

- a statement identifying the class to which the material applies and notifying the reader that the material has been made available electronically solely for use in the class, and
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v) Books and Records; Right to Audit. As to each permission granted under the electronic course content Service, User shall maintain for at least four full calendar years books and records sufficient for CCC to determine the numbers of copies made by User under such permission. CCC and any representatives it may designate shall have the right to audit such books and records at any time during User's ordinary business hours, upon two days' prior notice. If any such audit shall determine that User shall have underpaid for, or underreported, any electronic copies used by three percent (3%) or more, then User shall bear all the costs of any such audit; otherwise, CCC shall bear the costs of any such audit. Any amount determined by such audit to have been underpaid by User shall immediately be paid to CCC by User, together with interest thereon at the rate of 10% per annum from the date such amount was originally due. The provisions of this paragraph shall survive the termination of this license for any reason.

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ii) User may not make or permit any alterations to the Work, unless expressly set forth in the Order Confirmation (after request by User and approval by Rightsholder); provided, however, that a Work consisting of photographs or other still images not embedded in text may, if necessary, be resized, reformatted or have its resolution modified without additional express permission, and a Work consisting of audiovisual content may, if necessary, be "clipped" or reformatted for purposes of time or content management or ease of delivery (provided that any such resizing, reformatting, resolution modification or "clipping" does not alter the underlying editorial content or meaning of the Work used, and that the resulting material is used solely within the scope of, and in a manner consistent with, the particular License described in the Order Confirmation and the Terms.

#### 15) Miscellaneous.

a) User acknowledges that CCC may, from time to time, make changes or additions to the Service or to the Terms, and that Rightsholder may make changes or additions to the Rightsholder Terms. Such updated Terms will replace the prior terms and conditions in the order workflow and shall be effective as to any subsequent Licenses but shall not apply to Licenses already granted and paid for under a prior set of terms.

b) Use of User-related information collected through the Service is governed by CCC's privacy policy, available online at [www.copyright.com/about/privacy-policy/](http://www.copyright.com/about/privacy-policy/).

c) The License is personal to User. Therefore, User may not assign or transfer to any other person (whether a natural person or an organization of any kind) the License or any rights granted thereunder; provided, however, that, where applicable, User may assign such License in its entirety on written notice to CCC in the event of a transfer of all or substantially all of User's rights in any new material which includes the Work(s) licensed under this Service.

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e) The License described in the Order Confirmation shall be governed by and construed under the law of the State of New York, USA, without regard to the principles thereof of conflicts of law. Any case, controversy, suit, action, or proceeding arising out of, in connection with, or related to such License shall be brought, at CCC's sole discretion, in any federal or state court located in the County of New York, State of New York, USA, or in any federal or state court whose geographical jurisdiction covers the location of the Rightsholder set forth in the Order Confirmation. The parties expressly submit to the personal jurisdiction and venue of each such federal or state court.

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