



**NOVEL MATERIALS PROCESS FOR
ALCOHOL BASED FUEL CELLS**

Final Report

Contract Number: F/02/00272/REP

URN NUMBER: 05/1480

dti

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**APPLICATION OF THE 'ONE-STEP' PRODUCTION METHOD TO NEW
MATERIALS**

FES F/02/00272/00/REP
URN Number: 05/1480

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EXECUTIVE SUMMARY

This report is the summation of a development programme conducted by ITM Power Plc, in conjunction with Cranfield University, to develop cheap novel materials and processes for alcohol based fuel cells. These devices are of commercial interest as they offer the prospect of power sources with a high efficiency, high energy density and rapid refuelling times for a range of electronic devices such as mobile phones, laptops and MP3 players. The market for such fuel cells is estimated to be worth \$800 million by 2010 [1].

At present, the key reason for the poor sales of fuel cells is their unit cost. This is due to the price of two key components:

- The ion exchange membrane, which separates the fuel and oxidant. Currently, the market is dominated by Nafion™, a product developed by DuPont, for which the cheapest available price is ~£270/m² (\$500/m²). This is an acidic membrane which separates the fuel and oxidant, but is permeable to hydrogen ions (protons).
- The catalyst. This is invariably made from platinum, which is currently trading for £15,250/kg on the commodity markets. Despite its high cost, for alcohol fuels, this is the most effective catalyst for the acidic chemistry of Nafion.

The majority of companies working to develop fuel cells use Nafion, and hence platinum. The only realistic way they can reduce the unit cost of their fuel cells is to try to increase the power output per cm² of membrane (and hence per gram of catalyst). Many papers and research projects have been dedicated to increasing their power density with the general conclusion that it can only be achieved through working at elevated temperatures (~70°C) and high fuel / oxidant pressures and flow rates. Unfortunately, these working conditions are not suitable for the small portable device market for which the fuel cells are developed.

During this project, ITM Power have sought to take a fresh approach to the problem by developing two new categories of cheaper ion exchange membranes; thus negating the requirement for Nafion. The membranes developed at ITM are based on ionic hydrophilic polymers, made by bulk co-polymerisation from solution, (henceforth referred to as 'Type 1' conductive polymers), while Cranfield University have been contracted for their expertise in imparting polymers with ionic properties through radiation grafting (henceforth referred to as 'Type 2' conductive polymers). By approaching the problem through the development of two distinct novel ion permeable membranes, the company sought to increase the chance of project success, while expanding its suite of materials.

During the project, each type of conductive polymer was developed as both acidic (with similar chemistry to Nafion) and alkaline based membranes. The latter have the potential to use cheaper catalysts than platinum, creating a further cost saving.

The key objective of this project was not to obtain power densities comparable with Nafion, which has been constantly refined over recent decades. Instead, the emphasis has been on the development of a range of *cheap* fuel cells. If

this can be achieved, the requirement for high power densities is largely negated for many commercial applications. Furthermore, results reported within this document were not obtained under conditions designed to maximise the power figures, but were assessed under more realistic conditions found inside a portable device – ambient temperatures and with low pressures and flow rates for fuel and oxidant.

For the 'Type 1' hydrophilic polymers, the specific project objectives were to further refine the existing Cation Exchange (CE) membranes (which are acidic and allow protons to permeate – hence they are often referred to in the literature as Proton Exchange Membranes) and develop the completely novel Anion Exchange (AE) membranes (which are alkaline and allow hydroxide ions to permeate). Furthermore, these unique materials allowed the production of a 'One-Shot' fuel cell, where electrodes, catalysts and membrane, are all assembled and adhered together into a Membrane Electrode Assembly (MEA), in a single stage process. This ability is unique within the fuel cell industry, and represents a significant manufacturing cost saving over the current technologies. Specific features of the 'One-Shot' system (such as catalyst application method) were to be refined throughout the project.

For the 'Type 2', radiation grafted hydrocarbon technology, the project objectives were to adapt the existing acidic CE membranes for use with alcohol fuels and develop novel alkaline AE membranes.

The first work package within the project detailed the development of acidic CE membranes using both Type 1 and Type 2 production methodologies. Nearly thirty variations of ITM's original Type 1 CE membrane were assessed for ionic conductivity, changing such properties as water content, cross-linking agent and acidic content. From the tested compositions, two had ionic conductivities of 1.4 and 2.25 times that of Nafion 117¹ and were taken forward for further study. Fourteen Type 2 CE compositions were also assessed for ionic conductivity relative to Nafion 117. Of these, eight formulations based on styrene-sulphonic acid grafts were taken forward for further study, with the best being 2.44 times the conductivity of Nafion.

The second work package involved the development of AE membranes using Type 1 and Type 2 technologies. For these experiments, Nafion 117 AE² was used as a baseline. The conductivity of several Type 1 AE chemistries were measured, with the best exceeding the performance of alkaline conditioned Nafion by a factor of 1.42. Three variations were carried forward for further development. Twelve Type 2 AE membranes were tested for ionic conductivity, with the best having an ionic conductivity of 3.63 times that of alkaline conditioned Nafion.

The membranes taken forward from previous units were analysed for fuel crossover in the third work package. It is desired that fuel cross over should be reduced as it acts to decrease the fuel cell output. For CE materials, the Type 1 samples had varying levels of success, the best having 1.5 times better

¹ Nafion 117 is the type of Nafion used for comparison during this project

² No commercially available anion exchange material is available. Thus, 'Nafion 117 AE' is made by conditioning Nafion 117 in an alkaline environment. Contrary to what many papers say, this is not a true anion exchange membrane, unlike those developed within this project; however, it is the closest available for comparison.

crossover than the Nafion 117 benchmark. The Type 2 membranes were more successful, with nearly all of the samples tested performing considerably better than Nafion 117, with the best having only 3.5 times better crossover. For the AE materials, the Type 1 membranes showed a large variation in methanol permeability, with the best having a four time worse crossover than Nafion 117. The Type 2 membranes, all outperformed Nafion 117, with the best having a six times better crossover than Nafion 117.

An additional commonly used comparator between membranes is the ratio of conductivity to methanol crossover, although ITM not been able to confirm or define how this is related to fuel cell performance. The best CE and AE ratios are 7.9 and 16.5 times that of Nafion 117.

The next work package focussed on the use of catalysts with different membrane chemistries. This section of work was largely based on a report commissioned for ITM by Dr Kucernak of Imperial College, London. It was concluded that for methanol fuel, platinum was best for the oxidant side, while a mixture of platinum and ruthenium was best for the fuel side. However, it was suggested that for alkaline chemistries (the AE membranes), cheaper, alternative catalysts such as nickel and palladium may give reasonable performance. Based on these recommendations, different methods were proposed for how to attach the catalyst to the polymer during ITM's patented 'One-Shot' process. These included a chemical reaction with the polymer, coating porous materials which could then be cured in contact with the membrane, and application to a sacrificial material that could be polymerised in contact with the membrane then peeled / dissolved away.

During the subsequent work package, the various options for catalyst deposition were attempted to identify those methods which were most effective. It was found that chemical deposition of the catalyst onto the membrane after polymerisation was not successful; however, platinum plating of the electrode and the sacrificial transfer methods appeared viable options to take forward to MEA production.

The final work unit involved the production and testing of MEAs. Based on the conductivity and crossover data, a total of four materials were tested; an AE and a CE material of each ion permeable membrane technology. These were compared to the performance of Nafion 117 AE and CE materials. For reasons of reproducibility, the catalyst selected for the comparison was commercial loaded carbon cloths of Platinum (Pt) for the oxidant side, and Platinum-Ruthenium (Pt-Ru) for the fuel side. The MEAs were tested using a variety of methanol concentrations at room temperature. The performance was assessed by conducting a series of polarisations and noting the peak power during polarisation. As this is a commercial project, for comparison reasons, results are presented as membrane or MEA £/kW at this peak power.

For CE materials, the cheapest membrane per kW output was the Type 1 membrane at £316/kW, compared to Nafion 117 at £1815/kW. For AE materials, the Type 1 membrane was again the cheapest at £1019/kW, compared with Nafion 117 AE at £6923/kW. It was also considered that because the Type 1

membranes were up to 20 times thicker than the Nafion 117 and Type 2 membranes, the development of thin Type 1 membranes should result in significant increases in power.

Tests were conducted using ITM's patented 'One-Shot' process, in which the MEA is created in a single polymerisation process, which should reduce manufacturing costs. To achieve this, stainless steel expanded mesh was coated in Pt (as recommended in the catalyst report), placed in the liquid monomer, and cured. However, the cost/kW calculations suggested that for methanol fuels, this particular method was not advantageous.

Further tests were conducted into the use of palladium as a cheaper alternative catalyst to platinum. It was found that palladium (Pd) gave the same power density as Pt when used on the fuel side of the cell, giving a lower MEA £/kW than Pt when used on the fuel side of the cell. Experiments comparing Pt and Pt-Ru confirmed that the catalysis of pure Pt is greatly assisted by the presence of ruthenium (Ru), indicating that a Pd-Ru system could lead to higher power densities and lower catalyst costs both of which would lead to further reductions in the MEA £/kW. As a further test, a liquid oxidant was used in a Pd cell, resulting in an increase in power density, further reducing the MEA £/kW.

It was concluded that this project has been successful. The objectives were to produce cheap fuel cells using novel materials. The results demonstrate that this has been achieved using a combination of two alternative ion permeable membrane technologies.

Based on the results presented here, ITM have applied for five new patents that have resulted in whole or in part from this project (patent filing numbers, 0329459.2, 0417911.5, 0420961.5, 0504460.7 and one newly filed still awaiting an application number), at least two of these applications will undergo PCT application. Many of the membranes discussed have been shown to work highly successfully using alternative liquid fuels, which are outside the scope of this report, and ITM are seeking their rapid commercialisation.

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1. INTRODUCTION

Fuel cells offer the potential of a power source that lasts far longer than is possible with battery technology. Furthermore, once depleted, while batteries require several hours plugged into a mains supply to recharge, fuel cells only require a quick refill from a fuel cassette and they are ready for use. With the portable electronic device market worth several billion pounds per annum, these advantages have caused fuel cells to receive wide coverage over the last few years in academic, government and commercial research establishments. However, in general, the impact of the developments has been limited, due to uncompetitive costs and poor performance.

Fuel cells work by having an electrode/catalyst immersed in a fuel source, methanol in the case of this project, and a second electrode/catalyst in the presence of an oxidant, often gaseous oxygen. The fuel and oxidant are separated by a membrane which allows ions generated at the catalyst to permeate, but prevents the transfer of fuel, oxidant and electricity. Electrons generated at the catalyst are extracted by the electrode, may be forced to do work in a circuit (for example by powering a mobile phone or laptop) and are then returned to the fuel cell at the second electrode.

The uncompetitive costs of fuel cells arise primarily from the material used to make the ion permeable membrane. The industry standard is a product called Nafion, made by DuPont. This product is based on acidic chemistry, and provides reasonable power outputs. However, its cost is ~£270 (\$500) per m², which makes it uneconomical to use in most situations. A further problem is that due to the acidic chemistry of Nafion, the catalyst required for alcohol fuels is limited to platinum, which costs ~£15,250/kg.

Using current technology, the only way of reducing the cost of the Membrane Electrode Assemblies (MEAs) is to refine the system so that the power output per unit area is increased; thus, for a given power requirement, less membrane and catalyst is required. Hence, the fuel cell industry has become obsessed with achieving ever higher power densities, even if the conditions under which the measurements are taken are not representative of those found in small portable devices for which their use is intended. For example, most papers and research projects publish results obtained at 70°C with high fuel and oxidant flow rates and pressures, which are completely unrealistic for most commercial applications.

ITM Power Plc, working with Cranfield University, have taken a novel approach to this problem, by focussing on producing cheap fuel cells, by utilising two different membrane technologies. One is based on making ionic hydrophilic polymers, while the other uses radiation to graft ionic sites onto polymers. Henceforth, these will be referred to as Type 1 and Type 2 technologies, respectively. By using two novel technologies the chances of a successful outcome to the project are increased. These methods can potentially reduce the material cost of the membrane to ~1% of Nafion.

Further advantage is found from using alkaline ionic sites, rather than acidic. Despite what is stated in many research papers, this can not be achieved with Nafion - it can only be conditioned in an alkaline environment to make a pseudo alkaline membrane. However, the novel materials developed within

this project can easily have the ionic sites changed from acidic to alkaline, making a true alkaline ion permeable membrane. The main advantage of using such alkaline chemistry is that it opens the possibility of using far cheaper catalysts than platinum, such as palladium and nickel.

Extra saving could also be obtained through improving the manufacturing route. The Type 1 materials are formed into a membrane by pouring liquid monomers into a thin mould, then curing to this shape. It allows the option of adding the electrodes and catalysts to the mould, pouring in the liquid monomer and creating the entire MEA (or even a stack of MEAs) in a single 'One-Shot' curing process. This simplified production route should yield further savings over Nafion.

Hence, through this use of cheap technology, there is no requirement to constantly drive for higher power densities. Instead, by focussing on lowering costs and developing fuel cells for real-world applications, ITM Power have created technology which can allow the potential of fuel cells to be realised.

2. PREVIOUS WORK

It has been shown in previous studies that the use of Nafion membranes in Direct Methanol Fuel Cells (DMFC) can result in poor performance [2,3]. This problem is largely ascribed to the high permeation of methanol through the membrane into the cathode side of the cell, where it is oxidised without generating electrical power. It was believed that the two technologies developed within this report would offer significant improvements.

2.1 Type 1 Technology (Hydrophilic Solid Polymer Electrolyte)

ITM power had previously developed a hydrophilic Cationic Exchange Solid Polymer Electrolyte (CESPE) material, which was known to have properties enabling its use as a hydrogen-oxygen fuel cell membrane. The ultimate aim of this project was to demonstrate a material or range of materials suitable for use in an alcohol fuel cell, requiring the Solid Polymer Electrode (SPE) to operate in a quite different environment. As such, the optimum material for use in a hydrogen system may have been different to the optimum material for an alcohol system. In particular, materials of lower equilibrium water content offered the potential to lower alcohol crossover, and thus may have been better suited to alcohol fuel applications.

2.2 Type 2 Technology (Radiation Grafted Solid Polymer Electrolyte)

Previous research at Cranfield University has shown that materials prepared by the process of radiochemical copolymerisation (otherwise known as radiation grafting) have found application in primary and secondary batteries and proton exchange membrane fuel cells (PEMFC). With expertise in this field, Cranfield University were contracted to develop these membranes for use in Direct Methanol Fuel Cells.

Studies have shown that these materials in PEMFCs can offer cell performances which are equal to and, in some circumstances, significantly better than those achievable with the benchmark Nafion 117 membranes. The high conductivity Cranfield membranes, developed specifically for PEMFCs, when used in DMFCs, have been shown in un-published work to result in similar limitations. Previous unreported Cranfield work has also shown that membranes based on hydrocarbon polymers may possess a lower methanol permeability than the more usual fluorocarbon-based membranes used in fuel cells (Nafion).

Thus, the project aimed to use radiation grafting technology to synthesise a new range of membranes that, whilst having good ionic conductivity, significantly reduced the problem of methanol crossover.

3. METHODOLOGY

3.1 Samples Tested

3.1.1 Type 1 Technology CE Samples

A Type 1 CE sample had previously been developed for use with hydrogen as a fuel, thus only alteration for the methanol fuel was required. Changes made to the existing polymer included:

- Changing the hydrophobic : hydrophilic monomer ratio
- Changing the water content
- Changing the cross linker content
- Changing the acid concentration

3.1.2 Type 2 Technology CE Samples

Four commercial polymers were chosen for this study:

- 125 μ Low Density Polyethylene (LDPE) film
- 125 μ Ethylene Tetrafluoroethylene film (ETFE)
- 50 μ Low Density LDPE film
- 50 μ ETFE film

These were then radiation grafted using monomers based on styrene and acrylic acid.

3.1.3 Type 1 Technology AE Samples

Work was undertaken on an extensive formulation exercise in which 25 candidate AESPE materials were prepared, using a combination of four monomers and six bases.

3.1.4 Type 2 Technology AE Samples

Two commercially-sourced base polymers were selected:

- 50 μ Low Density Polyethylene (LDPE) film
- 50 μ Ethylene Tetrafluoroethylene film (ETFE)

Two radiation-grafting processes were used to prepare the AESPE materials, the Mutual Irradiation Grafting (MIG) and the Post Irradiation Grafting (PIG) techniques. Following the grafting treatments the materials were thoroughly washed to remove unwanted by-products and dried.

The monomers chosen for membrane preparation were:

- Vinyl benzyl chloride (VBC)
- 2-(dimethylamino)ethyl methacrylate (DEAM)

2-[(Methacryloyloxy)ethyl]trimethylammonium chloride (MATAM)
4-Vinyl pyridine (VPy) and Acrylic Acid (AA)

3.2 Conductivity Measurements

The ionic conductivity was measured using an AC impedance technique comprising a HP 4274A Multi-Frequency LCR Meter connected to a custom-designed cell. The cell was thermostatically controlled at 25°C and is shown schematically in Figure 1. A small dish of water inside the sample chamber ensured the humidity remained constant.

Samples under test were conditioned in de-ionised water for 24 hours prior to being torque-clamped between the stainless steel electrodes. Due to the fact that Nafion 117 and the Type 2 membranes were far less compliant compared to the Type 1 materials, different methods were followed for securing them between the electrodes. The less compliant materials were held firmly, the electrodes being tightened using a torque wrench. The Type 1 materials required far less pressure to achieve adequate contact with the electrodes; indeed some formulations were damaged in attempts to secure them as firmly as required for Nafion 117. Therefore, the Type 1 materials were squeezed between the electrodes until they were compressed by approximately 10%.

The samples were allowed to equilibrate in the test cell for an hour prior to the commencement of the test. The sample resistance was computed via an algorithm from measurements taken at six different frequencies ranging from 1Hz to 100KHz. The membrane conductivity is the reciprocal of the resistance, (after accounting for the electrode area), multiplied by the membrane thickness.

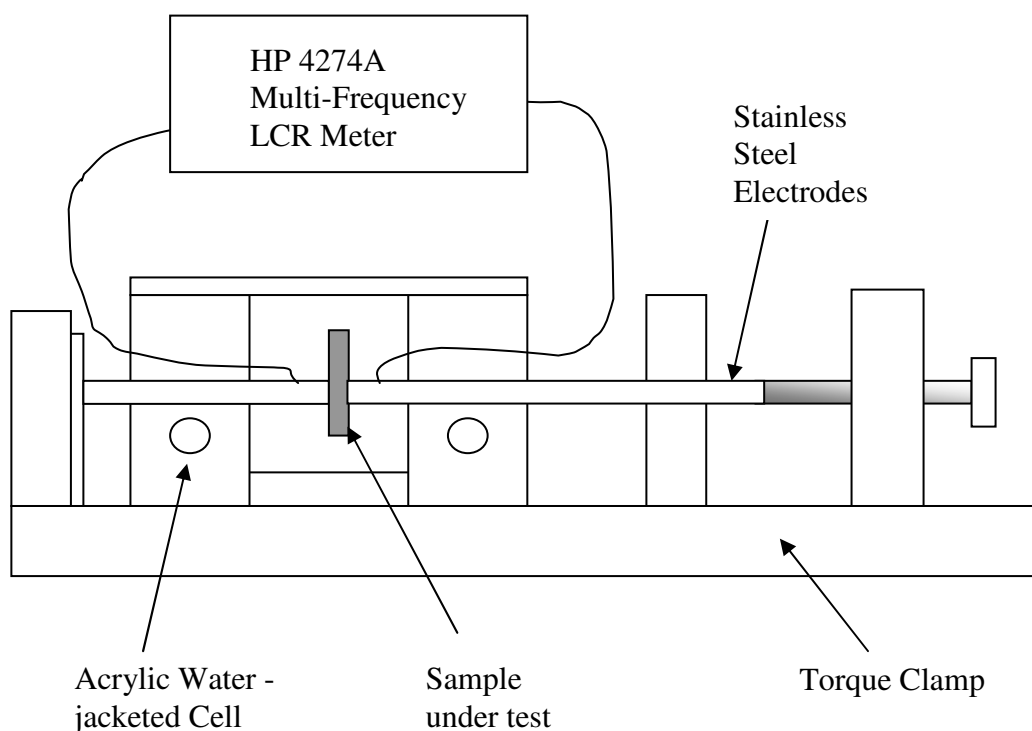


Figure 1. AC Conductivity Measurement Apparatus

3.3 Water Uptake Measurements

The water uptake is the equilibrium amount of water held by the membrane after soaking for 16 hours and was measured gravimetrically. It is expressed as a percentage of the dry sample weight.

3.4 Methanol Permeability Measurement Technique

There are two principal methods for measuring methanol-in-water concentrations, Refractive Index (RI) and Gas Chromatography (GC). However, provided chemically pure liquids are used, the RI technique is as accurate as GC and quicker to perform. It was therefore the preferred method for this project.

Before methanol permeability measurements were taken, the refractometer was calibrated using accurately made solutions of methanol in water. The graph of methanol/water concentration against RI in the range 0% - 100% methanol is sigmoid in nature. The method used for analysing the data involves dividing the curve into a series of straight lines, obtaining the equation of the lines in the form $y = mx + c$, from which, if the RI is y , the methanol concentration x , can be calculated.

3.4.1 Type 1 and Type 2 CE Methanol Permeability

The CE membranes created using either novel technology were placed in glass bottles of demineralised water and conditioned in a water bath maintained at 25°C until tested. Samples were removed from the bottles and patted dry before being placed in a water-jacketed, two-compartment test cell. The membrane was placed between two cell halves, one containing water and the other 100% methanol. The cell temperature was maintained at 25°C. Both chambers were continuously stirred to prevent concentration gradient effects. At the beginning of each run the refractive index of pure water (RI_w) was measured and aliquots were then withdrawn from the water side of the cell at intervals over a period of at least 2 hours. The refractive index (RI_m) was measured using a Bellingham and Stanley Refractometer (Model No.60) and the difference in refractive index ($RI_w - RI_m$) was calculated. The difference in refractive index was then related to concentration using the calibration data. The concentration values were plotted against time and the apparent diffusion coefficient (DK) was calculated from the slope of the resulting graph.

3.4.2 Type 1 AE Methanol Permeability

The AE Type 1 membranes were placed in bottles of demineralised water and equilibrated in a water bath at 25°C. For testing the membrane was removed from the water, patted dry, clamped into the cell and measured using the same procedure as for the CESPE materials.

Samples were also placed in bottles of 2M NaOH equilibrated in a water bath at 25°C. For testing, the membrane was removed from the NaOH, rinsed in demineralised water, patted dry, clamped into the cell and measured using the same procedure as for the CESPE materials.

3.4.3 Type 2 AE Methanol Permeability

The AESPE membranes were conditioned by heating in 2M NaOH at 90°C for 10 minutes and allowed to cool. The resulting membranes were placed in

bottles of 2M NaOH and equilibrated in a water bath at 25°C. For testing the membrane was removed from the NaOH, rinsed in demineralised water, patted dry, clamped into the cell and measured using the same procedure as for the CESPE materials.

3.5 Catalyst Activity Assessment

The catalyst materials identified in the catalyst report commissioned by ITM and written by Dr Kucernak of Imperial College London, have been assessed for their activity with the preferred SPE materials and a methanol fuel. Pt:Ru serves as a benchmark for comparison.

The analysis was conducted using cyclic voltammetry, with the test cell housing a sample of the SPE material, approximately 60mm diameter. A microelectrode (the tip consisting of the catalyst material under test) was brought into intimate contact with the SPE. The electrode was supplied with an electrical potential that is swept from 0.2V to 1.2V and back again at a rate of 10mV/s. The oxidation current was monitored and analysed to indicate activity.

3.6 MEA Testing

3.6.1 Testing

The materials studied in this work package were:

Type 1 CE

Type 1 AE

Type 2 CE

Type 2 AE

Nafion 117 (CE)

Nafion 117 AE (No commercial material is available for comparison, so the acidic Nafion 117 has been conditioned in alkali, in accordance with standard practice)

A range of methods were investigated to bond the Type 2 membranes to the catalyst-coated cloth. These include the well-documented use of Nafion solution and compression at elevated temperature, the use of di-functional crosslinking agents such as divinyl benzene (DVB) and glycidyl methacrylate (GMA), the use of ionically conducting adhesives such as acrylic acid sprays, and the development of special ionically conducting copolymer solutions, e.g. based on PVDF-g-AA and PVDF-g-DMAM. Included in the processing was hydraulic pressing at 80°C and 25kg/cm².

In order to evaluate the bond strength, each MEA was subjected to immersion in boiling demineralised water and then methanol. Those MEAs that did not show signs of delamination when subjected to the boiling water and methanol were further equilibrated in 2M MeOH/0.1M NaOH solution for 16 hours prior to their ionic conductivity being measured. For convenience the conductivity measurements were taken in the 'dry' cell.

The results showed that although there was significant conductivity, the values were in each case lower than that of the membrane incorporated in the MEA, indicating that either the use of the bonding agent had affected the ionic

migration, or that during the bonding process, the membrane had dehydrated to a level which could not be fully recovered in the equilibration process.

For the subsequent fuel cell tests new MEAs were pressed. These were conditioned by boiling in water for 1 hour and heating to 95°C in 0.1M NaOH for 5 minutes. The MEAs were then placed in their respective fuel solutions i.e., for the AESPE this was 2M MeOH / 0.1M NaOH and for the CESPE 2M MeOH.

In the first series of fuel cell tests both the CESPE and the Nafion 117 (control) MEAs gave poor power density performances and showed a lower than expected Open Cell Voltage (OCV). Circulating fuel through the cell for up to a week slightly improved the performance, perhaps indicating that during the MEA formation dehydration of the membrane had occurred, and that an even longer conditioning period was required. However, time constraints precluded this and a limit of 48 hours conditioning was imposed. Thus, in the re-tests, each MEA was made by simply clamping the catalyst cloth to the membranes in the test cell.

The Type 1 MEAs were prepared using two methods, firstly by clamping the catalyst cloth to the membrane in the test cell, and secondly by embedding a catalyst mesh into the surface of the membrane during polymerisation. The only pre-treatment required was the room temperature hydration of the membranes in demineralised water, for CE membranes, and 1M NaOH, for AE membranes.

MEAs using both technologies were studied in a cell with an active area of 5cm² on an Arbin Instruments fuel cell testing facility at ITM's Sheffield laboratories. Aqueous solutions of methanol in concentrations of 2M, 4M and 6M were used for the tests. For AE MEAs, NaOH was added to the fuel to give a concentration of 1M. This ensured that the AE membranes and fuel were chemically equivalent³. For most tests, oxygen was used as the oxidant; however, specific experiments were conducted using acidified hydrogen peroxide. The fuel and oxidant were supplied at a pressure of 5psi, temperature of 30°C and flow rates of 20cc/min and 100cc/min, respectively.

Based on the results of the catalyst report, initial tests were conducted using platinum-ruthenium (Pt-Ru) catalyst on the fuel side and platinum black on the oxidant side, as this was believed to be the combination that would be most active. It was decided to use commercial Pt and Pt-Ru loaded cloths as they ensured repeatability of tests and a fair comparison between different materials. However, the catalyst report suggested alternative, cheaper, catalysts may be used; thus some tests were conducted using a commercial palladium (Pd) loaded cloth.

One proposed key advantage of ITM's materials is that they hold the potential to allow the creation of 'One-Shot' MEAs, i.e. have the membrane, catalyst and current collector created in a single stage process. Various options for this were considered, and it was decided that the method offering most potential was to coat a stainless steel expanded mesh with platinum and embed this into the polymer during curing. This was compared to the results using commercial

³ It should be noted that for AE systems, this results in an intrinsically ionic membrane hydrated in an ionic liquid. Understanding the dynamics of the resulting system is not trivial, and forms part of ITM's future core work.

Pt cloth. However, it should be noted that hydrogen peroxide was used as the oxidant for both tests.

All tests were measured using the following schedule:

- 1) Wait until environmental parameters are met by the system.
- 2) Rest for 15 minutes at OCV
- 3) Perform initial polarisation - discharge 5mA from the cell every 3 seconds until the voltage reaches a level of <50mV
- 4) Rest for 10 minutes at OCV
- 5) Perform 2nd polarisation
- 6) Repeat steps 4 and 5 until 4 polarisations have been performed
- 7) Rest for 10minutes at OCV
- 8) End of schedule

3.6.2 Reporting of MEA Results

As discussed in Section 1, the fuel cell industry is currently based on using Nafion, a product with a very high cost per unit area. Thus, most scientific papers focus on producing as much power per unit area as possible. However, as this is a commercial project comparing three very different membrane technologies, it is felt that mW/cm² tells little about the commercial viability of potential products – only their size. Thus, the cost per kW has been used throughout this report, as it is felt to be a far more useful comparator.

However, when comparing on a basis of cost, it is important that the costs are clearly defined. Throughout, most of this report, the same catalyst has been used for all three membrane technologies - pressed on commercial Pt loaded carbon cloth. Thus, as catalyst cost is constant, when membranes from different technologies are compared the price / kW values quoted are for the cost of the membranes only. A further problem is that while Nafion is in mass production, the ITM membranes have only been created on a laboratory scale, and any attempt to estimate full scale production costs would be outside the scope of this project. Thus, to keep calculations simple, the figures quoted for ITM membranes are based on the raw materials required for laboratory scale production – enough to make ~50m² of membranes. It should be noted that full scale production would probably result in an order of magnitude decrease in the material costs. An estimate of membrane production costs has been made at ~100% of material costs. Thus, the 'membrane cost' quoted is the sum of the material and production costs.

To allow comparison with Nafion 117, a value of £270 (\$500)/m² has been used as a basis for its cost. While this figure may not be the lowest possible cost obtainable, it still requires a large purchase to obtain it at this value, and is felt a fair comparison to the cost of laboratory scale production that have been used for the ITM materials.

When comparing different catalysts, oxidants and manufacturing process routes the £/kW is based on the entire MEA structure, rather than just the membrane cost.

Costings were based on the following assumptions:

- The membrane cost per m² was based on the raw material costs and processing.
- Processing was set at 100% of the raw materials cost.
- The MEA £/m², was based on the membrane costs and the catalyst electrode costs.
- The catalyst cloth costs were based on using catalyst costs, cloth costs, binder costs and mark-up costs, using a method suggested by Imperial College in a report commissioned by ITM, the Catalyst costs have been updated from the report to take into account fluctuations in prices over the last year.
- The platinum, palladium and ruthenium catalyst costs are based on average prices from August 2004 to July 2005, sources from JM, Platinum today.

This project was not concerned with establishing the ideal catalysts for the oxidant side of the fuel cell. The result was that for all tests (other than the 'One-Shot' test) all the MEAs constructed used Pt cloth on the oxidant side. This is the most expensive catalyst and has a large effect on the MEA cost/kW value. The catalyst report ITM commissioned has suggested cheaper alternative oxidant catalysts, but their testing is outside the scope of this project.

4. RESULTS

4.1 Cationic Exchange (CE) Materials

4.1.1 Preliminary Testing

Before the project started, CE materials created using the Type 1 and Type 2 methods had been developed for use with alternative fuels. To refine the materials for use with methanol based fuel, a series of alterations were made to their chemistry, which are detailed in Sections 3.11 & 3.12.

4.1.1.1 Preliminary Testing of the Type 1 Polymer

The new Type 1 polymer chemistries were assessed initially through conductivity measurements, and then the most promising samples were tested for methanol crossover. A summary of the best conductivity results are presented in Figure 2 and the crossover results in Figure 3

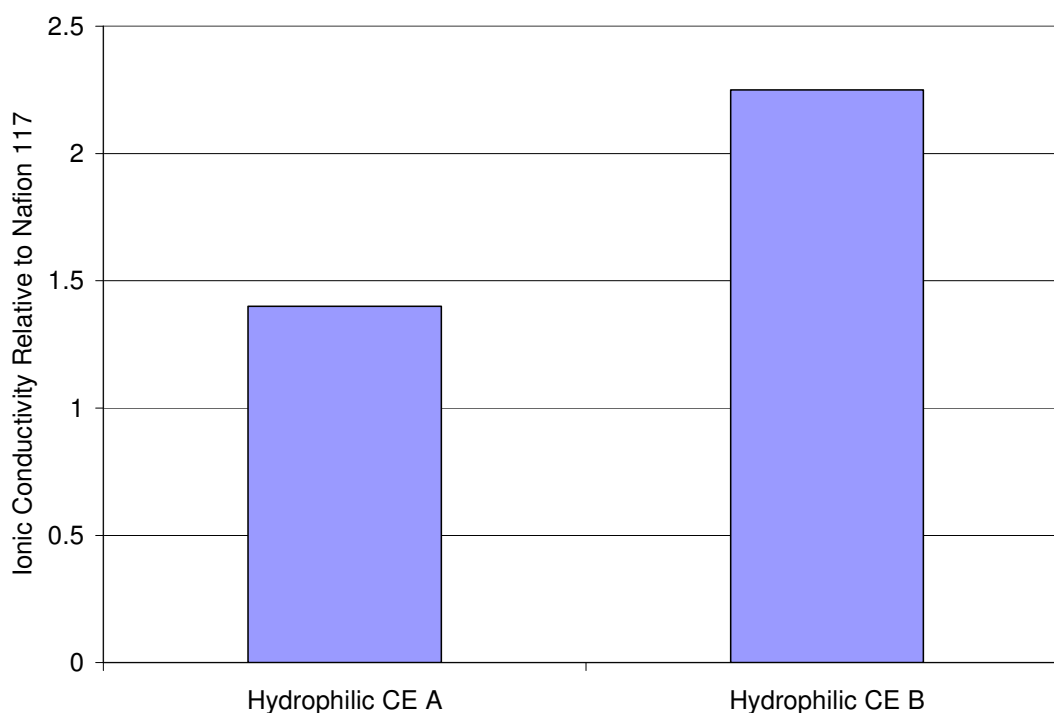


Figure 2. A graph comparing the conductivity of the two best Type 1 hydrophilic CE membranes relative to Nafion 117. The conductivity of Sample B is over double that of Nafion 117.

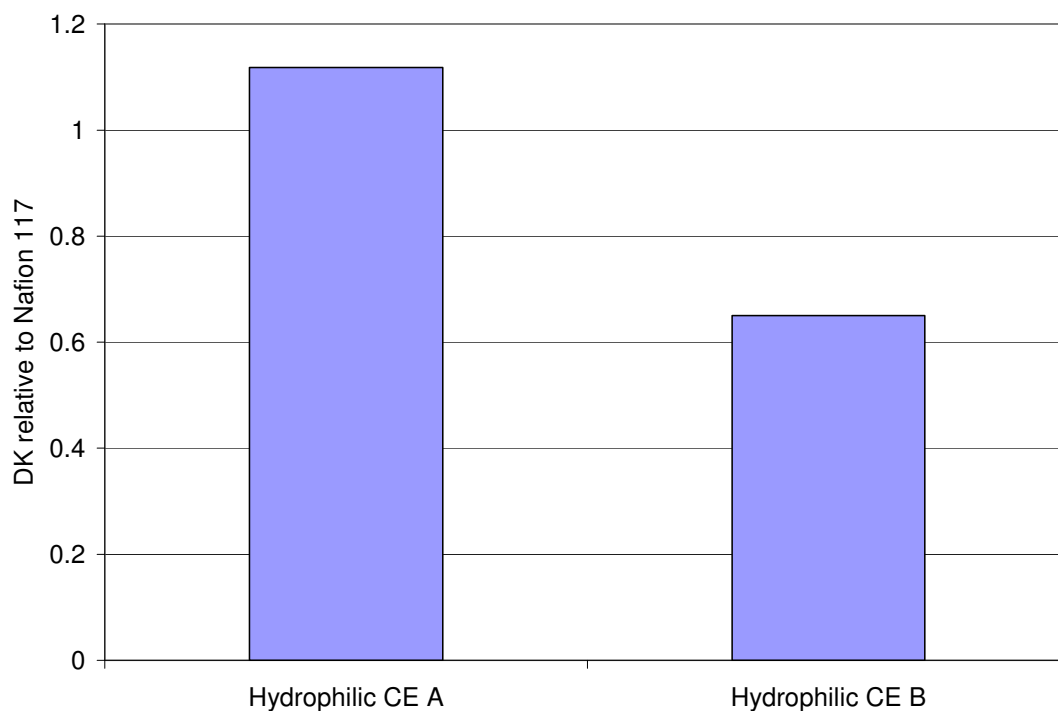


Figure 3. Results showing the methanol permeability (DK) of the preferred Type 1 hydrophilic CE compositions relative to Nafion 117.

The conductivity and crossover results were combined and expressed as a fraction of conductivity over methanol permeability (relative to Nafion 117). The results are presented in Figure 4.

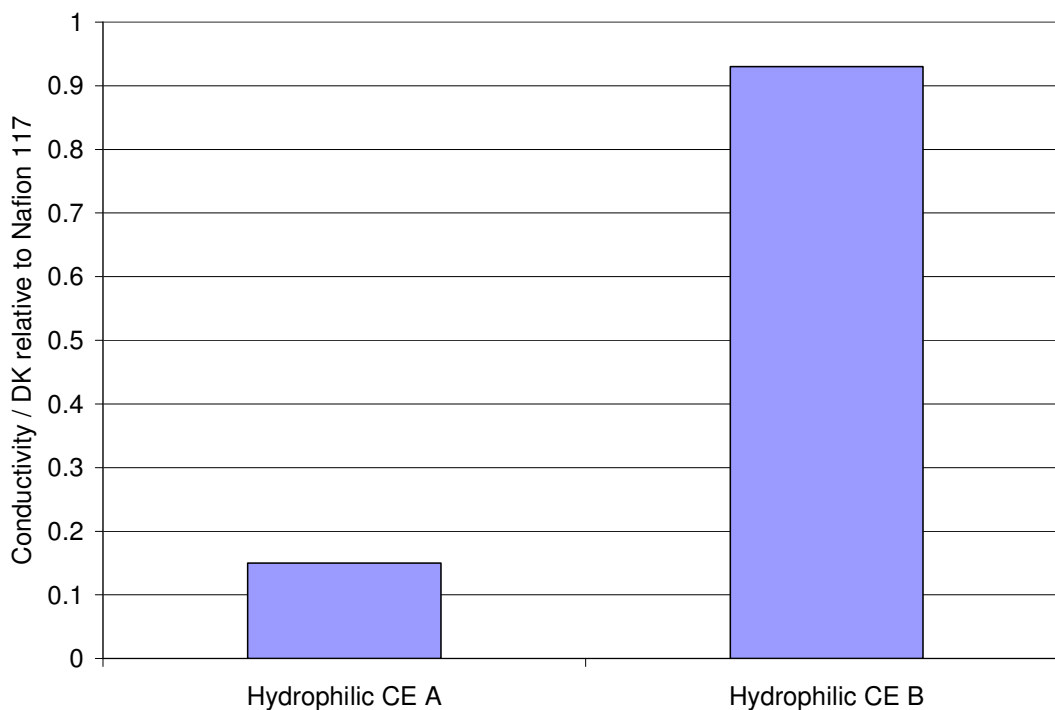


Figure 4. Conductivity / methanol permeability of Type 1 hydrophilic CE samples relative to Nafion 117.

Based on this, the Type 1 sample taken forward for further study was Sample B.

4.1.1.2 Preliminary Testing of the Type 2 Membranes

A total of 14 Type 2 radiation grafted samples of various chemistries (detailed in Section 3.2) were tested for conductivity. The results are presented in Figure 5.

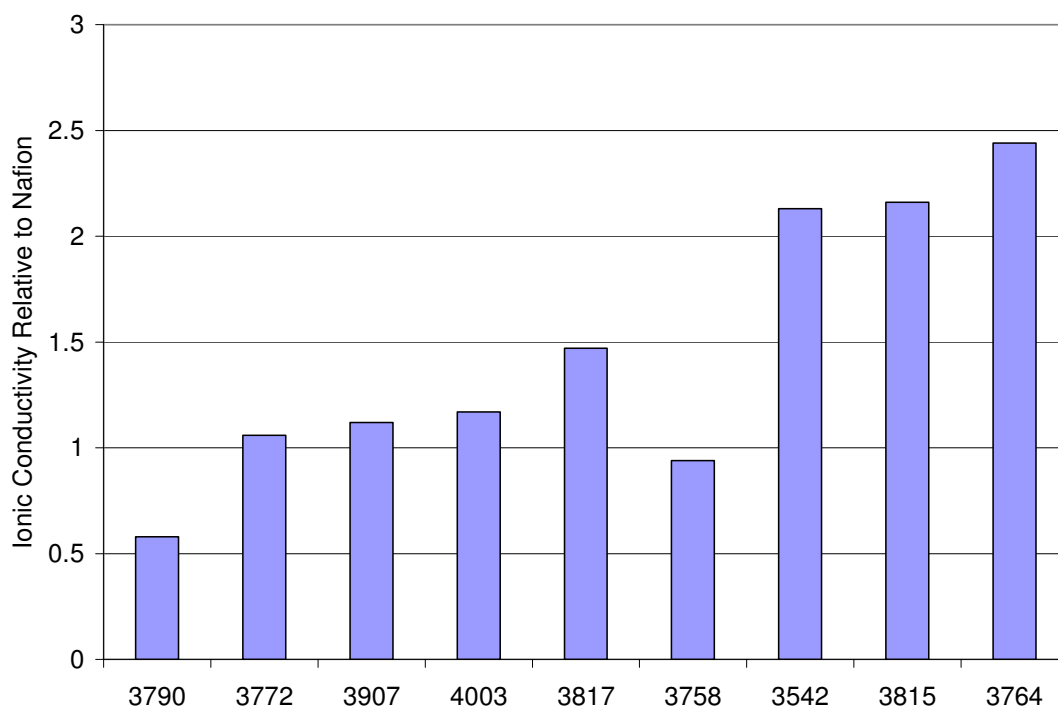


Figure 5. A graph showing the conductivity of various Type 2 radiation grafted CE samples, relative to Nafion. Several of the samples have over double the conductivity of Nafion.

This shows that most of the samples either have similar or significantly higher conductivity than Nafion 117. These samples were then tested for methanol permeability, and the results presented in Figure 6.

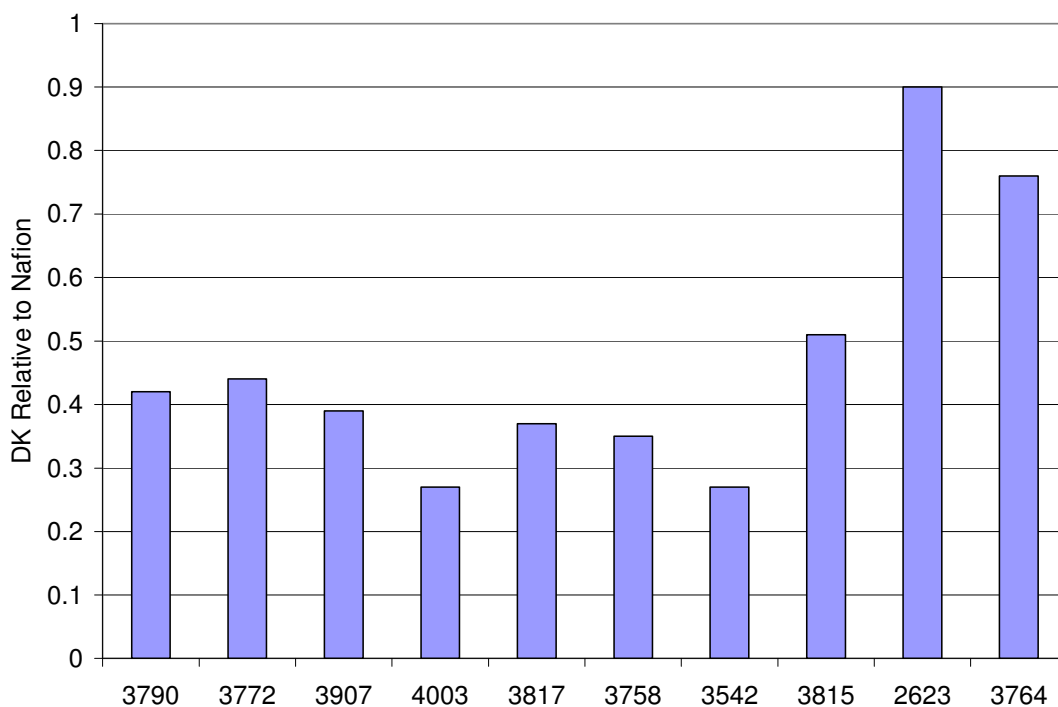


Figure 6. A graph showing the methanol permeability of various Type 2 radiation grafted CE samples, relative to Nafion. It is clear that all of the samples have considerably less crossover than Nafion.

All of the samples tested had lower methanol permeability than Nafion 117. As with the Type 1 samples, the data were combined on a graph of conductivity / methanol permeability, shown in Figure 7.

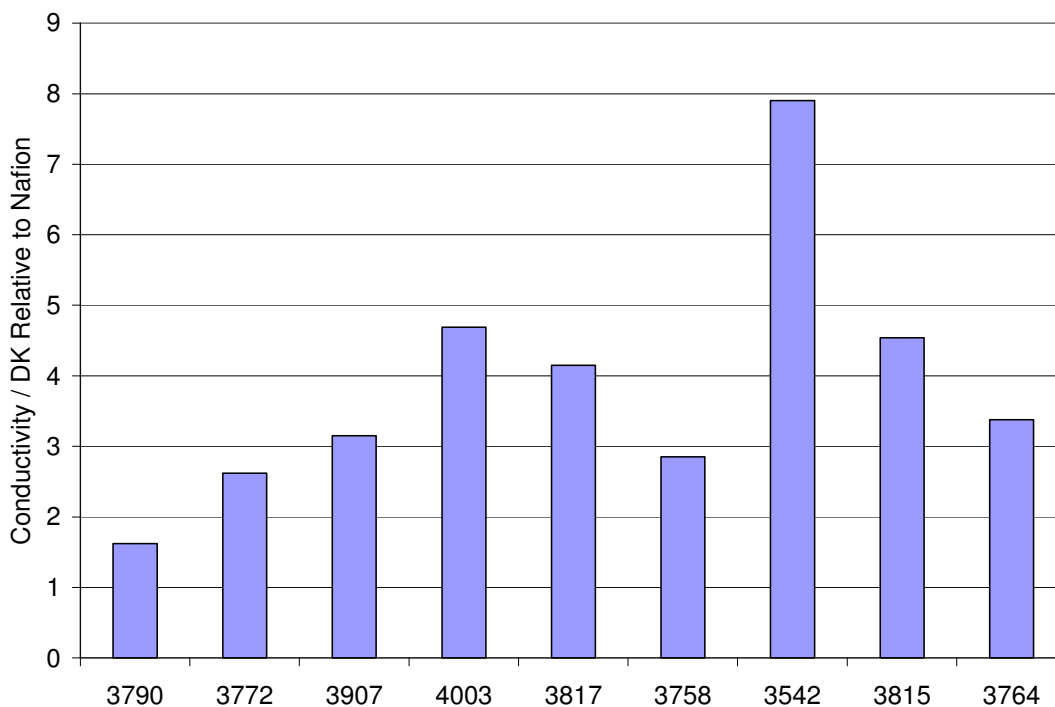


Figure 7. A graph showing the fraction of conductivity / methanol permeability for various Type 2, compared with Nafion 117. This suggests that nearly all of the samples should be superior to Nafion on operation; however, 3542 appears to offer the most potential.

Nearly all of the samples had a conductivity / methanol permeability value higher than Nafion 117; however, based on the data, sample '3542' was taken forward for further study in MEAs.

4.1.1.3 CE Catalyst Study Results

A report on suitable catalysts for methanol cells was written for ITM by Dr Kucernak of Imperial College London. He concluded that for CE materials, the best combination of catalysts was Pt & Ru on the fuel side and Pt on the oxidant side. It was suggested that there would be little activity from cheaper catalysts such as nickel and palladium.

While the Type 2 samples are limited in the methods available for applying catalysts, the Type 1 polymers have the advantage that they are poured into a mould and polymerised in-situ either thermally or by gamma radiation. This allows the prospect of the electrodes and catalysts being placed in the mould prior to polymerisation, allowing the entire membrane electrode assembly (MEA) to be created in a single procedure. This is known as ITM's 'One-Shot' process. Various methods for achieving a 'One-Shot' process were considered⁴, and of these several were attempted and the (OCV) recorded. These methods included:

- Applying commercial platinum black powder to a precursor material which was removed sacrificially after polymerisation. The following sacrificial materials were attempted: polythene, corona discharge treated polythene, silicone, mechanically roughened silicone. The best results were from the corona discharge treated polythene, which achieved an OCV of 947mV
- Chemical deposition of Pt to the steel electrode. This achieved an OCV of 415mV

As a further study, methods of adding catalyst after polymerisation were also considered. The results were:

- A metallic ink consisting of a suspension of platinum black powder in water was created and applied to the membrane surface. Once dried, the level of platinum adherence was measured through washing the surface with water. Through using this method, an OCV of 840mV was obtained.
- Chemical deposition of the Pt directly to the membrane surface. It was found that a fine (<50nm particle diameter) dispersion of platinum could be achieved on the membrane surface through the reaction of the membrane with silver nitrate and a platinum salt.

⁴ It should be noted that the methods described in this section apply equally to the Type 1 AE materials discussed in Section 4.2.

4.1.2 CE MEA Testing

4.1.2.1 Comparing Different CE Membrane Technologies

MEAs were tested as described in Section 3.5. For each membrane, a variety of fuel concentrations were tested; however, only the results of the fuel concentration giving the peak power for each membrane are presented in Figure 8.

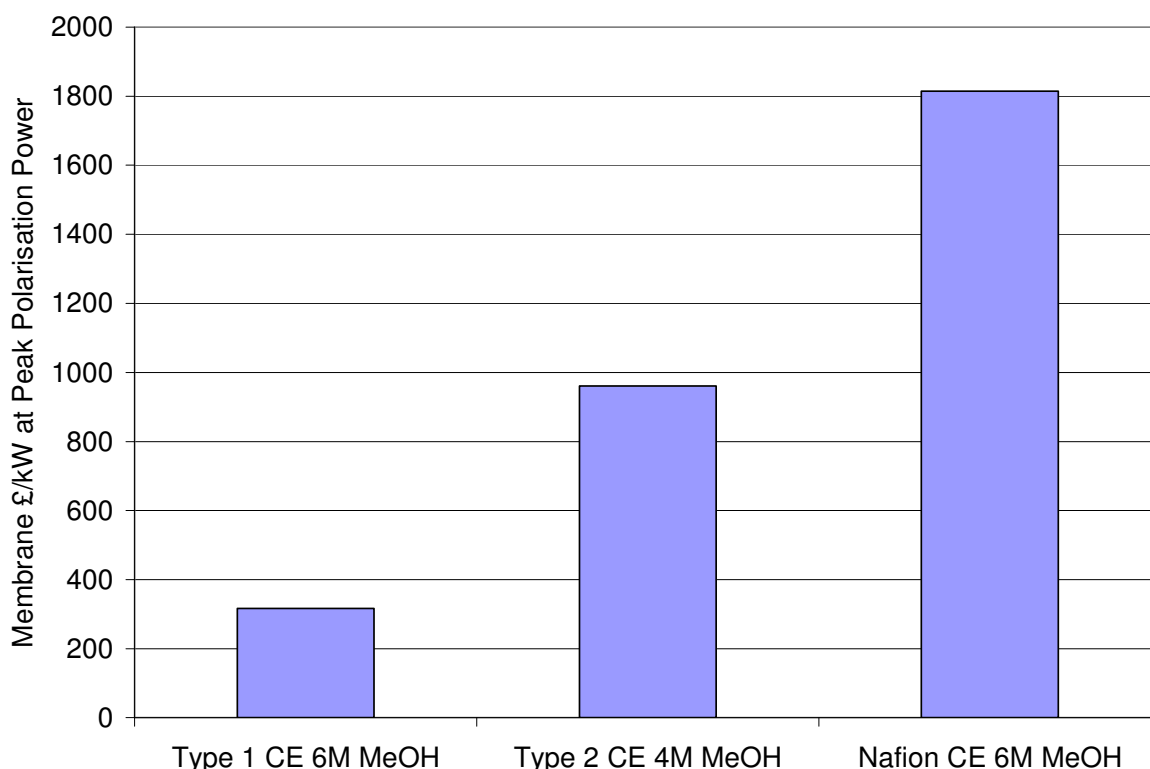


Figure 8. A bar chart showing the £/kW for the different CE membrane technologies at the fuel concentration that gives maximum power for each material.

Figure 8 clearly shows that the Type 1 and Type 2 CE materials are considerably cheaper per kW output at their peak polarisation power than Nafion 117.

4.1.2.2 'One-Shot' Results

To test the viability of the 'One-Shot' process, an expanded mesh was coated with Pt and placed in contact with the Type 1 CE material as it polymerised. Due to the low surface area of mesh in 3-phase contact with the membrane and oxygen, a liquid oxidant was used (hydrogen peroxide).

The results indicated that that the cloth is 2% of the MEA £/kW than the expanded mesh. However, two points should be considered:

- The most effective catalyst for methanol is Pt-Ru, not Pt. Thus, the presence of 50% Ru in the cloth not only makes it considerably cheaper,

but more effective than the Pt mesh. The addition of Ru to the mesh is being investigated.

- The expanded mesh has approximately 50% open projected area; thus, catalyst is only exposed to half of the membrane, suggesting the power of the mesh could, in theory, be doubled.

4.2 Anionic Exchange (AE) Materials

4.2.1 Preliminary Testing

Before the project started, no AE materials using either Type 1 or Type 2 technology had been developed. Therefore, materials were created based on the existing CE chemistries, but using groups which facilitate hydroxide ion transport, rather than protons.

4.2.1.1 Preliminary Testing of the Type 1 AE Polymer

Combinations of three monomers and six bases were chosen for conductivity measurements. Many of the formulations attempted did not polymerise, and many of those that did had poor conductivity. The best results are presented in Figure 9. These are quoted relative to Nafion 117 which had been pre-treated in an alkaline environment, subsequently referred to as 'alkaline conditioned Nafion'.

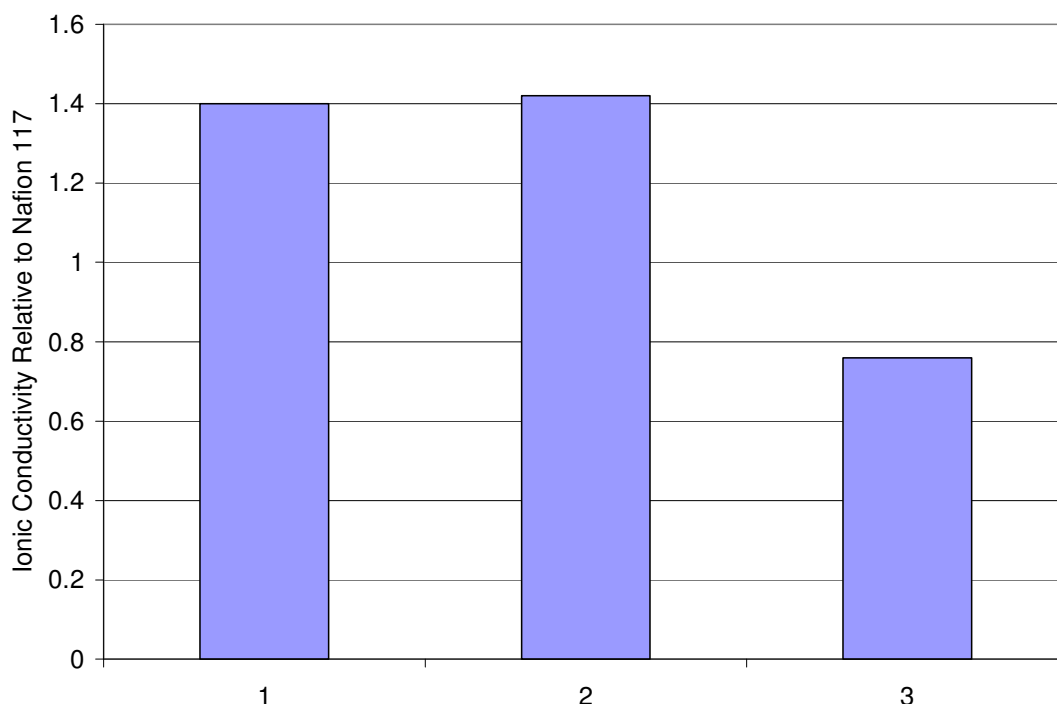


Figure 9. The conductivity of the best Type 1 AE membranes relative to alkaline conditioned Nafion 117.

These Type 1 AE samples had conductivity similar to or better than Nafion 117.

The samples were then tested for methanol permeability, and the results are presented in Figure 10.

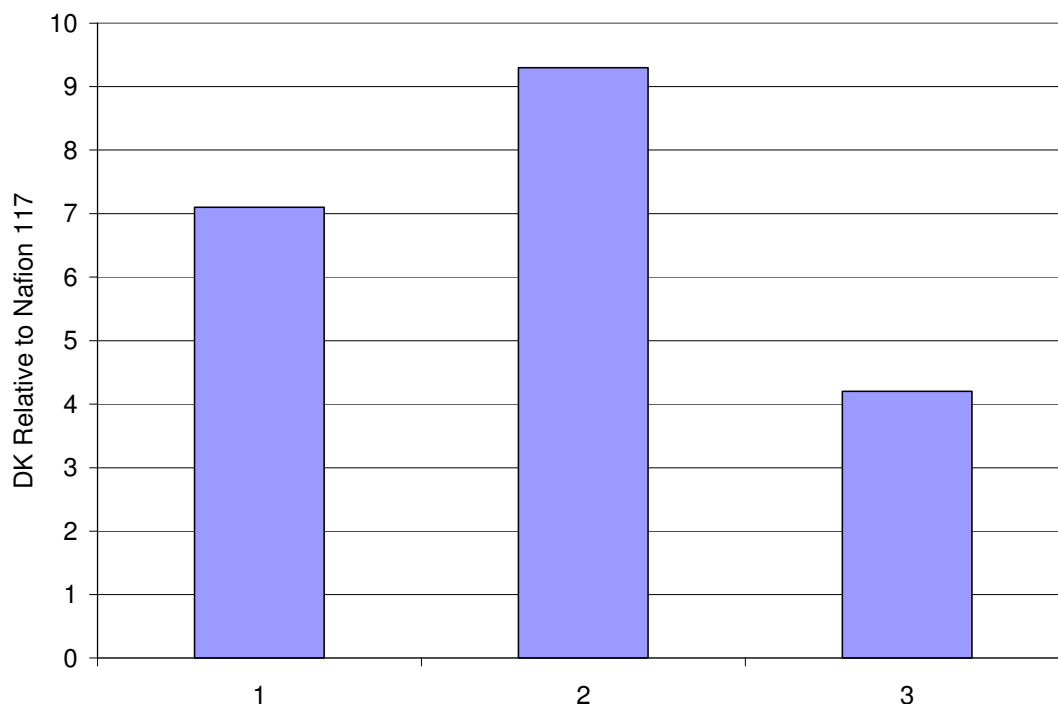


Figure 10. Methanol permeability (DK) relative to alkaline conditioned Nafion, for three hydrophilic AE materials.

All of the Type 1 AE materials have considerably greater crossover than Nafion 117.

As with the CE materials, the conductivity and crossover data were expressed as a fraction of conductivity / methanol crossover relative to Nafion 117. The results are presented in Figure 11.

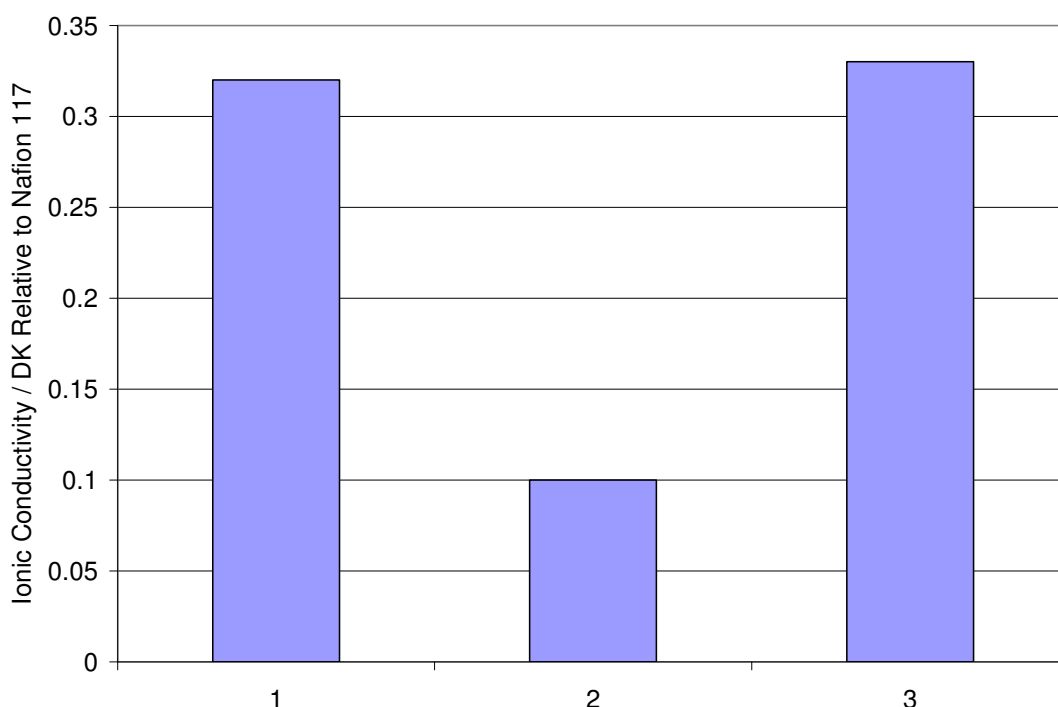


Figure 11. Conductivity / methanol permeability relative to alkaline conditioned Nafion for three Type 1 AE membranes.

Based on the results presented in Figure 11, AE chemistry '3' was taken forward for further study in MEAs.

4.2.1.2 Preliminary Testing of the Type 2 AE Polymer

A total of twelve Type 2 AESPE membranes were assessed and compared to Nafion 117 conditioned in an alkaline environment, for ionic conductivity and methanol crossover. The conductivity results for the best eight are presented in Figure 12.

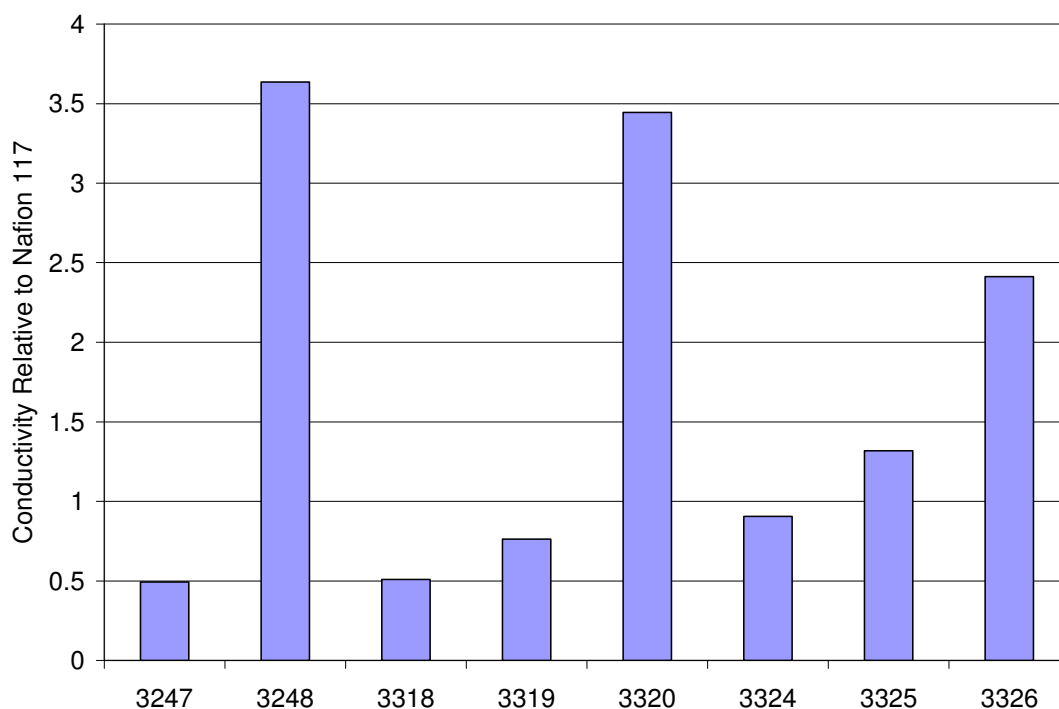


Figure 12. The conductivity of different radiation grafted Type 2 AE polymers, relative to alkaline conditioned Nafion 117.

The ionic conductivity results indicate that several of the Type 2 polymer samples have ionic conductivity values considerably higher than Nafion 117.

The results from the methanol permeability tests are presented in Figure 13.

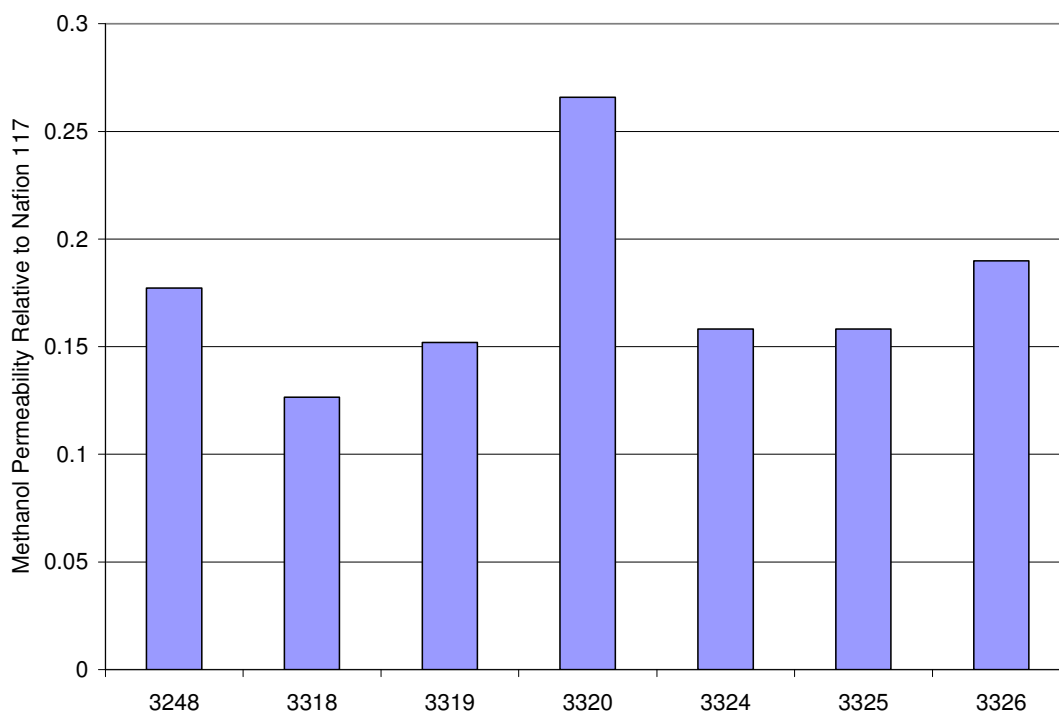


Figure 13. The methanol permeability of the Type 2 polymer AE samples, relative to alkaline conditioned Nafion 117.

As was done with previous samples, the conductivity and methanol permeability was expressed as a single fraction of conductivity / methanol permeability compared to Nafion 117. The results are presented in Figure 14.

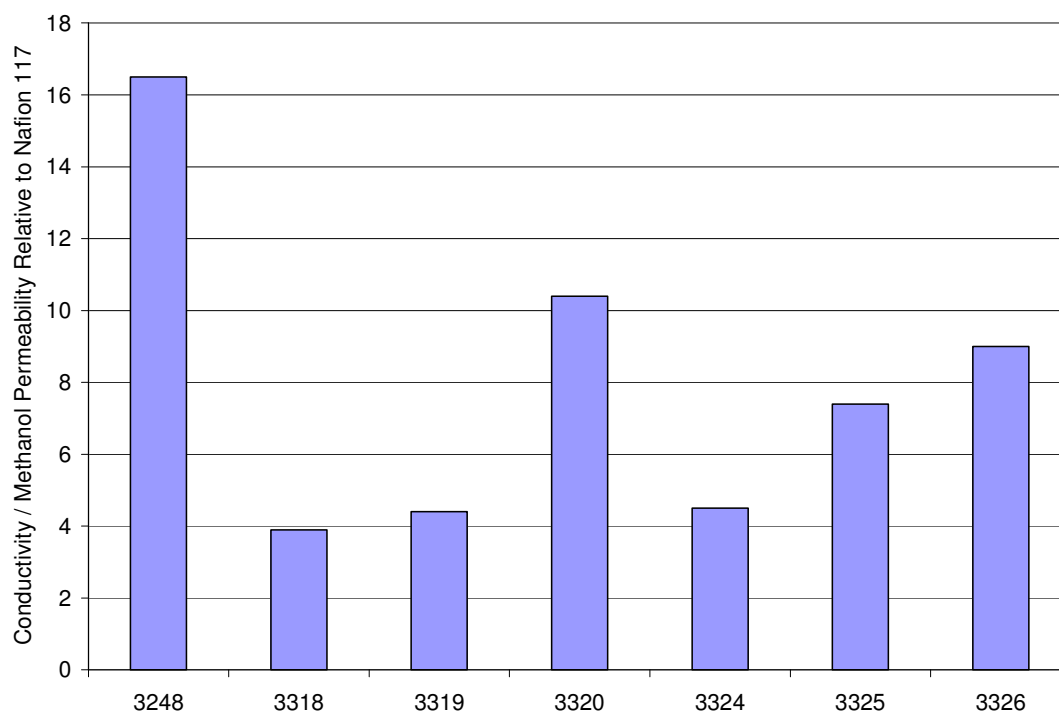


Figure 14. The fraction of conductivity / methanol permeability for the Type 2 AE samples, relative to alkaline conditioned Nafion.

Based on this data, sample '3248' was taken forward for further study in MEAs.

4.2.1.3 Catalyst Study Results

The conclusions of the catalyst report commissioned by ITM for AE materials were similar to those for CE materials, as discussed in Section 4.1.1.3. It was recommended that for both ionic systems, Pt was the best catalyst on the oxidant side, while Pt-Ru was the most active on the fuel side. However, when using alkaline chemistry, cheaper catalysts such as Pd and Ni were predicted to give reasonable performance. This premise is tested in Section 4.2.2.

4.2.2 AE MEA Testing

4.2.2.1 Comparing Different AE Membrane Technologies

AE MEAs were tested as described in Section 3.5. For each membrane, a variety of fuel concentrations were used; however, only the results of the fuel giving the peak power for each membrane are presented in Figure 15. It should be noted that as the membranes were hydrated in 2M NaOH, a similar concentration was added to the fuels to stabilise the membranes.

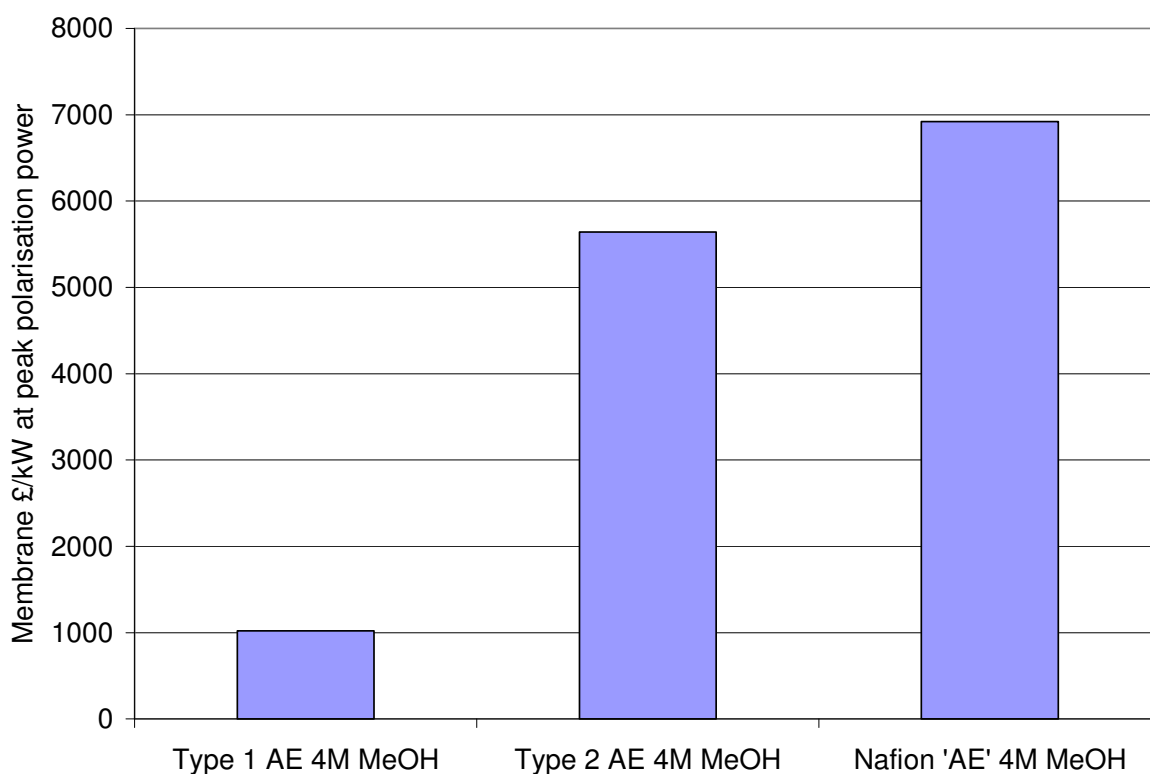


Figure 15. Comparison between the cost per kW output for each membrane at the peak polarisation power of different AE technologies using the fuel concentration that provides maximum power for each membrane type.

Figure 15 shows that both the Type 1 and Type 2 AE technologies are significantly cheaper per kW than alkaline conditioned Nafion 117.

4.2.2.2 Comparing Palladium with Platinum Catalyst

The results from Section 4.2.1.3 suggested that palladium could be used as a replacement for platinum on the fuel side of the cell, as this could offer significant cost advantages.

A Pt loaded cloth on the fuel side of a Type 1 AE MEA was compared to commercial Pd loaded cloth, there was no detriment in the power density. The results indicated that using Pd cloth would give a 36% reduction in MEA £/kW when compared to Pt cloth. This indicates the alkaline chemistry does indeed allow the use of cheaper alternative catalysts. It should be noted that both of the cloths tested did not contain Ru, which has been shown to significantly improve catalytic activity. This is due to problems with locating Pd-Ru cloth. The presence of Ru would further decrease the cost per kW, as Ru is cheaper than Pd, and, based on the performance difference between Pt and Pt-Ru, the power density in a Pd-Ru system is expected to be higher than in a Pd only system.

4.2.2.3 Using Hydrogen Peroxide (H₂O₂) as an Oxidant

As part of ITM's independent research with other fuel and oxidant systems, the use of a liquid oxidant has been investigated. As the results from these alternative systems have been promising, some experiments were conducted where oxygen was replaced with hydrogen peroxide. When using Pd as a fuel side catalyst, the MEA £/kW output at peak polarisation power fell by 66% when H₂O₂ was added. Again, it should be noted that Pd cloth was used, not Pd-Ru.

The commercial advantages of using two liquids, offer considerable potential for military and other specialist applications. As such this has been identified as an area of interest to ITM, and considerable time and finance will be allocated to allow further work in this area.

5. DISCUSSION OF RESULTS

The commonly used comparators for different membranes when using alcohol fuels are the 'ionic conductivity' and the 'ratio of conductivity to methanol crossover'. For Type 1 and Type 2 technologies, CE membranes were found to have ionic conductivities of 2.25 and 2.44 times that of Nafion 117, while the AE membranes had values of 1.42 and 3.63 times that of alkaline conditioned Nafion 117. These values show ITM's technology to be a considerable improvement over the current industry standard.

The best ratio of conductivity to methanol crossover values for CE and AE materials were 7.9 and 16.5 times that of Nafion 117. Although ITM found little evidence that the ratio of conductivity to methanol crossover was directly related to fuel cell performance, these measurements are often quoted in research papers. Nevertheless, the values obtained for the Type 2 materials are a significant improvement over Nafion 117.

The results comparing the £/kW for the various membrane technologies are positive. For the CE materials, the Type 1 and Type 2 membranes were approximately 17% and 53% of the membrane £/kW of Nafion 117. For the AE materials, the Type 1 and Type 2 were 15% and 81% of the membrane £/kW of alkaline conditioned Nafion 117.

Although these results are a significant improvement over the industry standard, there are several points that should be considered:

These are the first results obtained for these materials using methanol, and yet they have superior performance than Nafion 117, which has been in development for many decades. It is highly likely that with a year's further refinement, the cost/kW could be further reduced by a factor of two.

The conditioning regime of the Type 2 membranes prior to MEA testing was not part of the development and has not been perfected; thus there may be potential for significant future improvements.

All of the membrane £/kW data was obtained using values based on the manufacture of ~50m² of material, and yet it is compared to Nafion 117 which is in mass production. It is probable that were the manufacture of the novel membranes to begin, the material costs would fall by an order of magnitude.

Every test that was performed (except the 'One-Shot' experiment) used Pt cloth on the oxidant side, which is the most expensive form of catalyst. This was because this project was not directly involved with the development of oxidant catalysts. This resulted in unrealistically high MEA costs.

The Type 2 and Nafion 117 membranes are approximately 200microns in thickness, while the Type 1 membranes hydrate to approximately 2mm. The conductivity of the membranes is inversely proportional to their thickness. This implies that order of magnitude increases in performance could be possible with the Type 1 membranes if the manufacturing process could be developed to produce membranes of similar thickness to Nafion 117. However, there are both engineering and scientific

barriers to this target. To enable thinner membranes, the physical strength of the Type 1 materials may need to be significantly increased. Even if this could be achieved, because the Type 1 polymers are not thermoplastic, fine control will be required to cure to the lower thickness. Work in this area has been included in ITM's future research plan.

Replacing Pt with a Pd catalyst produced an improvement in the MEA £/kW value of ~36%. The presence of Ru in the Pt-Ru catalyst results in another step improvement in performance, and it is predicted that a Pd-Ru catalyst would bring further benefits. However, given time constraints, it has not been possible to obtain this material and test it for this report.

The addition of hydrogen peroxide to the palladium catalysed AE cell increased performance, which had the effect of reducing the MEA cost/kW by 66%. Furthermore, it is predicted that oxidising a Pd-Ru cell with H₂O₂ could reduce the MEA £/kW even further. The use of hydrogen peroxide within this project could be considered controversial, as the original markets identified for the materials were generally small portable devices in which a reservoir of hydrogen peroxide would not be desirable due to its additional size and weight. However, this data represents a significant performance increase and it is the company's belief that some markets will be more sensitive to component cost than to component size. Furthermore, in some military situations, having a dual liquid system could be considered advantageous. Clearly, more work with methanol and alternative oxidants is required before firm conclusions can be drawn; although, this is outside the scope of the current work package.

The 'One-Shot' MEA performed worse than expected. However, it must be considered that the catalyst applied to the expanded mesh surface was Pt, not Pt-Ru. As discussed previously, in the absence of Ru, the catalysis of methanol is inefficient, thus reduced performance is to be expected. Furthermore, the Pt coating of the sample tested had not been perfected. Figure 16a, shows an SEM micrograph of the coating used for the measurements, which illustrates a microscopically smooth surface. However, Figure 16b is an image of a Pt coating which has recently been developed for ITM and is currently in the assessment phase. Its surface is comprised of Pt spheres of diameter ranging from 1micron to less than 50nm, giving it a higher active area; however, the time restrictions of this work package mean that it has not been possible to test this mesh in a methanol cell.

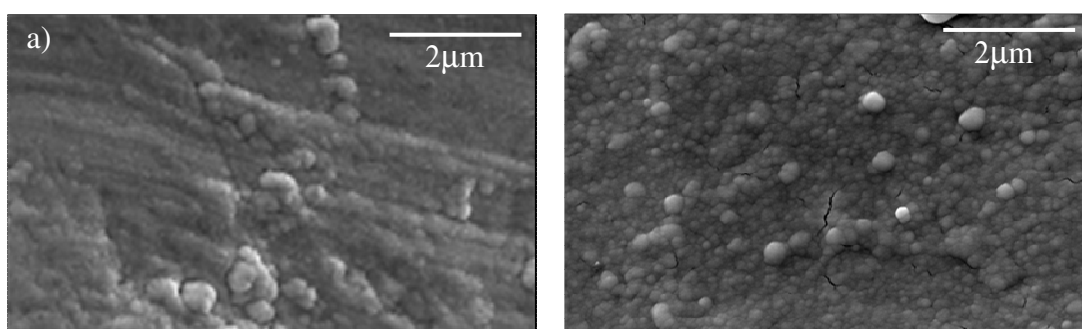


Figure 16. (a) shows an SEM micrograph of the Pt coating used for the 'One-Shot' MEA, while (b) illustrates a high surface area coating which is currently in development.

Based on work ITM has conducted using other liquid fuels, it is believed that once the coating morphology is perfected and Ru is added, the performance of the embedded mesh will surpass the commercial loaded cloths. Furthermore, it will have the advantage of a cheap 'One-Shot' manufacturing route while, as no degradation of the electrode/catalyst with time has been found, recycling of this expensive cell component will consist of peeling it off one membrane and polymerising it with another.

6. CONCLUSIONS

Cationic exchange (CE) and anionic exchange (AE) membranes have been developed using novel technologies based around the polymerisation of hydrophilic copolymers from monomer solution (Type 1 technology) and radiation grafted co-polymers (Type 2 technology). These were assessed for conductivity and fuel crossover. It was found that the Type 1 and Type 2 CE membranes had a conductivity of 2.25 and 2.44 times that of Nafion 117. The Type 1 and Type 2 AE membranes had a conductivity of 1.42 and 3.63 times that of Nafion 117 conditioned in an alkaline environment. The ratio of conductivity to methanol crossover was also calculated, although ITM have been unable to confirm or define how this relates to fuel cell performance. The best CE and AE ratios are 7.9 and 16.5 times that of Nafion 117.

From the conductivity and crossover data, the most promising membranes were tested in a direct methanol fuel cell, and compared to Nafion 117, the current industry standard. A variety of fuel concentrations, oxidants and catalysts were tested at room temperature and low flow rates, with results quoted on a £/kW basis to facilitate commercial comparison between the very different materials and processes. See section 3.5.2 for details of costing assumptions and calculations.

For CE materials, the Type 1 and Type 2 membranes were calculated to cost £316/kW and £960/kW, respectively. These compare favourably with the cost of Nafion 117, at £1815/kW. See section 4.1.2.1.

For AE materials, the Type 1 and Type 2 membranes were calculated to cost £1019/kW and £5641/kW, respectively. Again, these are both cheaper per kW than Nafion 117 conditioned in an alkaline environment, at £6923/kW. See section 4.2.2.1.

As, unlike Nafion, the AE membranes developed in this project are true anion exchange materials, they offer the possibility of using cheaper catalysts. Therefore, fuel cells were tested where platinum (Pt) was replaced by palladium (Pd) on the fuel side of the cell. This was successful and resulted in a 36% reduction in the cost per kW. It has been predicted that a Pd-Ru cell would be about further reduce the cost/ kW of a Pt-Ru cell. However, this is yet to be tested. See section 4.2.2.2.

Alternative oxidants were also briefly examined. It was found that replacing oxygen with hydrogen peroxide (H_2O_2) when using Pd as the catalyst produced a 66% reduction in £/kW. Again, it is predicted that a Pd-Ru catalyst with H_2O_2 will further reduce the cost/kW.

The objective of this project was to develop two alternative membrane technologies which can produce power at a significant cost reduction. This objective has been achieved. Within ITM, the polymers developed during this project have been utilised on other research programs and now form an integral part of our expanding portfolio of materials. The AE Type 1 materials appear to offer considerable potential in the electrolyser market, which ITM proposes to exploit.

7. FURTHER WORK

The success of this project has highlighted areas which could offer significant commercial advantage. Those areas that could benefit from further development include:

-The AE Type 1 membranes appear to have considerable potential in electrolyzers. However, it is probable that the membranes will require some refinement for this new role.

-Further work would be desirable towards perfecting the alternative catalysts / oxidants. This was outside the scope of the MEA assessment work package and limited time was available to refine the systems. However, the data produced for the cost / kW is so encouraging that ITM will encompass this work into its overall work plan.

-It would be desirable to reduce the thickness of the Type 1 membranes to ~0.1mm. This should result in an order of magnitude increase in performance, and reduce the cost of the membrane per m². This has been included as part of ITM's core research plan and work will be ongoing to identify the effect of thickness on performance in a variety of systems and different materials.

-It is predicted that Pd-Ru will bring the greatest cost per kW benefits. Thus, ITM are working to obtain some Pd-Ru loaded cloths and coated meshes.

8. REFERENCES

- 1) World Fuel Cells- An Industry Profile with Market Prospects to 2010, Elsevier Advanced Technology, 2002
- 2) Sundmacher K, Scott K, Chem. Eng. Sci., 54, (1999), 2927-2936
- 3) Scott K, Taama W et al, J. Power Sources, 83, (1999), 204-216