OASIS PROJECT FINAL REPORT

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EXECUTIVE SUMMARY

Project OASIS (Operation of Advanced Structures, Interfaces and Sub-components for MEAs) was a 12 month project that ran from 1st September 2008 to 31st August 2009, and was managed by the Department of Energy Office of Science, Chicago Office, as Award No DE-FG02-08ER64658, with Johnson Matthey Fuel Cells Inc. as the sole contractor. The project was completed on schedule, with technical successes (details below) and payment of the full grant award made by DOE.

The aim of the project was the development of membrane electrode assemblies (MEAs) for H₂/air polymer electrolyte membrane (PEM) fuel cells that would give higher performance under hot/dry and dry operating conditions, ideally with no loss of performance under wet conditions. Reducing or eliminating the need for humidifying the incoming gases will allow significant system cost and size reduction for many fuel cell applications including automotive, stationary and back-up power, and portable systems. Portable systems are also of particular interest in military markets.

In previous work Johnson Matthey Fuel Cells had developed very stable, corrosion-resistant catalysts suitable for resisting degradation by carbon corrosion in particular. These materials were applied within the OASIS project as they are considered necessary for systems such as automotive where multiple start-stop events are experienced. These catalysts were contrasted with more conventional materials in the design of catalyst layers and novel microporous layers (MPLs) and gas diffusion layer (GDL) combinations were also explored.

Early on in the work it was shown how much more aggressive high temperature operation is than dry operation. At the same humidity, tests at 110°C caused much more dehydration than tests at 80°C and the high temperature condition was much more revealing of improvements made to MEA design.

Alloy catalysts were introduced and compared with Pt catalysts with a range of particle sizes. It was apparent that the larger particle sizes of the alloy catalysts led to a reduction in performance that offset much of their kinetic advantage. The Pt-only materials clearly showed that small particles are beneficial to good performance under hot/dry conditions, because of their higher surface area, although they are known to be less stable to cyclic operation. An ex-situ water vapour sorption technique was developed that showed a very clear correlation with in-cell performance: catalyst powders that absorbed more water gave better performance in-cell. It was shown that alloy catalysts could give a 25 mV advantage over Pt-only at 1 Acm⁻².

GDL design was also shown to influence performance and more permeable GDLs on the anode allowed better membrane hydration and therefore conductivity. A very impermeable GDL on the cathode caused cathode flooding even under dry conditions, but a novel cathode MPL incorporating ionomer and operating at 110°C, 33/17% RH showed a 150 mV gain at 800 mAcm⁻² over the conventional MPL.

This project has increased the understanding of the factors that influence performance loss under dry conditions, including the development of an insightful ex-situ characterisation technique (Dynamic Vapour Sorption). All the approaches investigated can be readily

implemented in state-of the-art MEAs, although optimisation would be needed to integrate the new designs with existing MEA types and to tune to the exact range of operating conditions. The work is thus expected to benefit the public by feeding through more condition-tolerant production MEAs to a range of applications and thereby accelerate the commercialisation of fuel cell technology.

In summary, a number of specific catalyst, catalyst layer, MPL and GDL improvements were made during this project. Often the best designs under dry conditions translated to some performance loss under wet conditions, but compromise situations were also found where dry performance was improved with no loss of wet performance. The components studied in this project were then taken forward into the follow-on project (Contract no. DUDR.101155.000.030) in 2009/10 with Foster Miller, Inc. (subsequently QinetiQ North America, Inc.) and Protonex Technology Corporation and incorporated into MEAs for application in a new Protonex 750W stack design.

CONTENTS

EXECUTIVE SUMMARY.....	2
CONTENTS	4
1. Introduction	5
2. Methods and Materials.....	6
a. Catalysts.....	6
b. Ionomers	6
c. Membranes.....	6
d. Gas Diffusion Layers.....	6
e. MEA Preparation	6
f. Cell Testing.....	7
g. Dynamic Vapour Sorption (DVS).....	7
h. Mercury Intrusion Porosimetry (MIP)	7
3. Summary of Experimental Findings	8
a. Temperature versus Humidity.....	8
b. Influence of the GDL/MPL	8
c. Effect of Cathode Catalyst Type	10
d. Anode Catalyst Layer Design Effects	12
e. Cathode Catalyst Layer Design Effects.....	13
4. Summary of Accomplishments versus Objectives	17
5. Other Products and Technology Transfer	18

1. Introduction

Current polymer electrolyte membrane fuel cells (PEMFCs) operate at low temperatures and high relative humidity (R_H) owing to limitations in cell component technology and membrane electrode assembly (MEA) engineering. Substantial system advantages (such as reducing the stack heat rejection – currently needed so that the required low operating temperatures are maintained, and improved tolerance to fuel/oxidant impurities) would result from the development of PEMFC systems capable of operating at higher temperatures. One of the barriers to high temperature operation when utilizing today's perfluorosulfonic acid based membranes and ionomers is the difficulty in ensuring that the MEA remains sufficiently humidified for optimum proton conductivity, which is necessary for high cell performance. A reduced requirement to provide external humidification to the fuel cell, as per current technologies, would generate a significant reduction in fuel cell stack complexity and cost.

Johnson Matthey Fuel Cells Inc. (JMFCI) has previously developed a range of fuel cell catalysts specifically designed for high temperature PEMFC operation (120 °C vs. 80 °C employed in today's systems) which show both improved catalyst stability and increased corrosion resistance. The objective of the current project was to investigate the effect of the components within the MEA (excluding the membrane) and their configuration, with the aim of improving performance under dry operating conditions, to complement the existing stabilised catalysts. The challenge is to minimize cell dry-out, which results in increased ohmic resistance and correspondingly fuel cell performance loss, under high temperature, low R_H operating conditions, whilst ensuring that the MEA can also maintain high performance under the low temperature, high R_H conditions that may be experienced in operation i.e. during stack start-up/shut-down cycles. The approaches used focused on utilising the water produced within the MEA by tailoring the properties of the anode and cathode electrode structures, i.e. modifying electrode porosity, functionality and, correspondingly, hydrophobicity. Additionally, the ability of the gas diffusion layers (GDLs) and microporous layers (MPLs) were assessed for ability to limit dry out. The invention of new ionomeric components for improved proton mobility is not included in this work.

The MEA is the key enabler in the development of PEMFC systems for commercial applications, which includes replacement of the internal combustion engine in light-duty vehicles. JMFCI's research plan for this project related directly to the goals and objectives established by the DoE to enable use of PEMFCs in automotive applications.

2. Methods and Materials

a. Catalysts

Catalysts were all prepared in-house using proprietary Pt deposition chemistry on a range of commercially available and developmental carbon supports, to examine the effect of the support on the catalyst layer structure and the Pt dispersion. Pt black was also used to study layers with no carbon support.

- 60% Pt/Ketjen
- 60% Pt graphitised Ketjen 300J at 1800°C and 2500°C
- 60% Pt/Ketjen annealed in H₂/N₂
- 40% Pt/Ketjen annealed in H₂
- 60% Pt/developmental carbon supports
- Pt black

b. Ionomers

A range of commercially available ionomers were used to examine the effect of equivalent weight (EW), and other parameters, including Nafion 1100EW and Nafion 885EW.

c. Membranes

The membrane type was kept constant throughout this work and the material used was SH-30, a 30 micron thick membrane available from Asahi Glass.

d. Gas Diffusion Layers

Two main commercial GDLs were used: Toray TGP-H-060 with an in-house MPL applied and SGL 35BC, which came supplied with a MPL. Experimental MPL and GDLs, with reduced permeability or altered water handling, were also used.

e. MEA Preparation

Catalyst coated membrane (CCM) type MEAs were used for this work to eliminate the effect of the GDL/MPL on catalyst layer structure in the case where the catalyst layer is deposited into the MPL/GDL.

To make the CCMs, catalyst layers were deposited onto PTFE sheet and decal transferred to the membrane by hot pressing. The full MEA was assembled at the time of cell testing by placing GDLs either side of the CCM and mounting in the cell hardware.

f. Cell Testing

MEAs were prepared and tested at the 50 cm² active area scale ('screener' size) using a variety of test conditions and in-cell diagnostics to resolve flooding, membrane drying, catalyst activity etc. Humidities were typically 100%, 50% or 30% R_H both sides, but some more extreme conditions were also used. Similarly, the standard testing temperature was 80°C, but conditions up to 110°C were used. In most cases, variables were compared under the 30/30% R_H conditions.

g. Dynamic Vapour Sorption (DVS)

Dynamic vapour sorption – or DVS – is a gravimetric technique used to measure the amount and rate of water uptake by a material under controlled conditions of temperature and humidity. An adsorption-desorption isotherm can be recorded by stepwise increases in the water partial pressure in the nitrogen gas stream, holding until equilibrium is reached, then followed by stepwise decreases to record the desorption isotherm. Using the TA Instruments Q5000SA moisture sorption analyser, the isotherms were recorded at 60°C with 10% steps up in relative humidity from 0-90% R_H, followed by 10% steps down in R_H from 90-10% R_H. The method measures both adsorbed and absorbed water, and at low R_H values the amount of water sorbed depends on the attraction of water to the surface, the temperature, the water concentration and the amount of exposed surface area. At high R_H values the amount of water uptake also depends upon capillary condensation within the pore structure, which can show hysteresis, and can occur in an otherwise hydrophobic material.

This technique does not seem to have been applied to fuel cell MEA components previously and was developed to this end within this project.

h. Mercury Intrusion Porosimetry (MIP)

Mercury porosimetry is a characterisation technique used to provide information on the pore size distribution of a material over a wide range of pore diameters. The technique is based on the non-wetting properties of mercury (for most materials) which means that it will not enter the pores in a solid by capillary action, but must be forced in by the application of an external pressure. Using the Micromeritics Autopore IV 9520 porosimeter, the pressure was increased from 0.5 to 60,000 psia in small steps, with accompanying measurements of the volume of mercury intruded into the sample. The pore size distribution was then calculated using the Washburn equation, which relates the applied pressure to the diameter of the pores into which mercury is intruded, thereby giving the amount of porosity in pores from ~300 microns to 3 nm in diameter.

3. Summary of Experimental Findings

A lot of data was generated during the course of this work and the summary below is not meant to be in any way comprehensive, rather just an illustration of some of the more important findings.

a. Temperature versus Humidity

A very clear finding from this work was that temperature has a much more profound effect on dry-out than relative humidity (RH) under the range of conditions considered likely for automotive fuel cell operation. This can be explained by the change in outlet humidity because of changes in temperature, as shown in Table 1.

Temperature	Inlet RH (%)	Outlet RH(%)
80°C	30	80
110°C	30	40

Table 1: Comparison of outlet humidities for screener hardware with a 30% RH inlet, at two different cell temperatures.

In some cases, strategies for reducing the deleterious impact of dry conditions only showed their benefit under more extreme hot (>80°C) and dry conditions; an example of this was the use of ionomer to treat the MPL (see below). In general in this work, 30% RH on anode and cathode at 80°C was used to assess dry performance, in comparison to performance at 100 and 50% RH at 80°C, to avoid the time penalties involved with changing temperature on the test stands used.

b. Influence of the GDL/MPL

A direct comparison was made between SGL35BC supplied with a MPL and Toray TGP-H-060, which had an in-house MPL applied. Under fully humidified conditions, the CCMs tested with the SGL material gave significantly improved performance above about 1 Acm⁻² with a range of cathode layer designs, due to reduced flooding. It was hoped that this effect could be exploited by using this GDL/MPL to improve the performance under dry conditions by using cathode layers that would normally flood under wet conditions; for example by using high ionomer contents in the cathode. This was, however, not seen; the flooding under wet conditions was reduced as described, but under dry conditions no benefit was seen. Examination of the in-cell diagnostics, especially the helox gains, explained why this was the case. The helox gains are shown in Figure 1 for fully humidified conditions and it is clear that the benefit of the SGL material was almost exclusively a lowering of the helox gains.

Figure 1: Helox gains for fully humidified conditions comparing the effect of different GDL/MPL types.

The oxygen gains are not shown, but were almost un-affected by GDL type. This is interpreted as the SGL material having a much lower gas-phase mass transport resistance due to a difference in the way liquid water is handled. It is beyond the scope of this report to explain this effect, but the key point is that no benefit is seen under dry conditions because water removal to the cathode exhaust occurs exclusively in the gas phase under the dry conditions used (30/30% RH), rather than removal as liquid water. In a further iteration, it was found that an extra small improvement in performance under dry conditions could be obtained by using Toray on the anode and SGL on the cathode.

To restrict water removal in the vapour phase, a GDL/MPL with lower gas permeability was thought necessary. According to SGL's data sheets, the 35BC material has a porosity of about 80% and the Toray GDL/MPL combination was measured in-house using MIP to have a porosity of about 77%. Thus, a GDL/MPL with much lower porosity (53%) was obtained and tested. Unfortunately, when used on the cathode side of the MEA, this material did not allow the cell to run under any of the standard conditions; the cell performance was too low. Use of this low permeability GDL/MPL did not offer benefits when used on the anode either; performance was reduced by about 75 mV at 1.4 Acm⁻² across the range of humidities.

More positive results were obtained with a MPL created using Nafion instead of PTFE as the fluoropolymer additive and binder, since Nafion can exhibit quite pronounced surface hydrophobicity. Although this led to mild flooding under full R_H conditions, there was a slight benefit under dry conditions (30/30% R_H). Under hot, dry conditions (110°C, 33/17%

R_H), however, a tremendous advantage was seen; the Nafion-treated MPL showed a 150 mV gain at 800 mAcm⁻² over the conventional PTFE-treated MPL, due to reduced oxygen gains and CI resistance, which implies a better hydrated catalyst layer and membrane.

c. Effect of Cathode Catalyst Type

A large number of Pt catalysts were evaluated in this work, differing primarily in the type of carbon support and in the Pt surface area. In some cases, lower metal areas were obtained after catalysation due to the nature of the support; in others, the Pt metal area was reduced by heat treatment of the catalyst. The Pt metal area was found to be a key parameter affecting the behaviour of the cathode catalyst layers under dry operating conditions, bearing in mind that it was not possible to create catalyst layers with invariant structure using different catalyst types. The effect is illustrated in Figure 2, where the performance at 30/30% RH is plotted versus catalyst metal area. The data is corrected for membrane resistance differences and it is clear that better performance under dry conditions is obtained with higher Pt metal areas. It is also clear that an advantage of about 25 mV can be achieved by using a PtCr alloy catalyst

Figure 2: Air performance at 30/30% RH versus catalyst metal area for a wide range of Pt catalysts on different carbon supports.

To further probe the origin of this effect of metal area of the cathode catalysts, DVS data was generated on the water uptake of a wide range of materials at 60°C (the upper limit of the instrument) and 30% RH. Figure 3 shows the correlation between metal area, as determined by the number of CO molecules adsorbed on the surface, and the number of water molecules

adsorbed. The straight line shows the case of a 1:1 correlation where the number of CO and water molecules would be the same. Only two of the catalysts approach this 1:1 line; the others have lower water uptake than CO uptake. Note that the water uptake by the carbons in the catalysts is small in comparison to the Pt (typically 5 to 10%). This data shows that the increase in catalyst hydrophilicity is indeed due to the increase in Pt metal area, although this may not be the only effect of metal area on performance under dry conditions. For example, higher metal areas lead to lower local current densities, which will lead to lower iR drops in the (imperfectly hydrated) catalyst layer ionomer.

Figure 3: Correlation between metal area and number of water molecules adsorbed by a range of catalysts. Note that by far the majority of the water is taken up by the Pt and not the carbon.

Alloy catalysts offer clear performance benefits on pure oxygen under fully hydrated operation, promising opportunities to thrift metal loadings. The benefit on air under a wider range of humidification is less well established. Cell tests using 40%PtCr/Ketjen catalysts showed the expected benefit on oxygen, and air at low current densities. Under dry conditions, the performance suffered significantly compared to a conventional 60%Pt/Ketjen catalyst and so 0.2 mgPtcm⁻² PtCr layers were tested with a higher lamination pressure and with a low EW ionomer (900 EW) replacing Nafion 1100 EW. Both these approaches gave significant improvements at 1Acm⁻² under dry conditions (+60 mV for high pressure lamination, +100 mV for the low EW ionomer), but both also caused substantial flooding under full humidification.

Mercury porosimetry showed the layers laminated at high pressure to have substantially less porosity than the low pressure versions (1.0 cm³g⁻¹ versus 1.5 cm³g⁻¹), in the size range 35 to 60 nm.

d. Anode Catalyst Layer Design Effects

Following the reasoning that the ionic resistance in a thin catalyst layer should be lower than in a thicker one, especially under dry conditions, Pt black anodes were tested in comparison to the conventional carbon-supported catalysts as anode layers. Under the conditions tested (100/100, 50/50 and 30/30% RH on anode/cathode), however, no difference was seen. This work also showed the difficulty of depositing a uniform layer of Pt black at a 0.1 mgPtcm^{-2} loading; visual uniformity could only be achieved at a loading of about 0.2 mgPtcm^{-2} .

Another anode strategy employed to try and improve the performance under dry conditions was to use low EW ionomer, at the same loading as the Nafion 1100EW. The expectation was that the increased number of sulphonic groups should reduce the ionic resistance and increase the hydration of the anode. Summary results are shown in Figure 4, showing that the benefit of the low EW ionomer on the anode is only seen under more severely drying conditions at 100°C and 110°C . The benefit was mostly due to a lower membrane resistance (measured by current-interrupt), which is attributed to a higher water concentration on the anode leading to a higher water content in the membrane. In this scenario, the water back-diffusion from cathode to anode is reduced and the water content of the membrane is higher, due to the shallower concentration gradient between cathode and anode. Alternatively, the water dragged from the anode into the membrane by electro-osmosis is considered to have increased. Under fully humidified conditions and high current densities, the low EW anodes caused cathode flooding, presumably due to the same effect on water movement, in comparison to when the standard 1100EW ionomer was used on the anode. Whatever the mechanism, the benefit was clear: about 60 mV improvement at 800 mAcm^{-2} under the hottest, driest condition.

Figure 4: Air performance of low EW ionomer anode at different conditions.e. Cathode Catalyst Layer Design Effects

The temperature of drying of the cathode catalyst layer has a significant effect on performance under dry conditions, the magnitude of which is dependent on other components within the MEA. Figure 5 shows that drying the cathode catalyst layer under ambient conditions gave better performance than drying at 150°C, but that the effect is more pronounced when using SGL GDL than Toray GDL. Under wet conditions though, the *higher* drying temperature conferred better performance (not shown).

Figure 5: Effect of drying temperature of the cathode layer on the performance under 30/30% RH conditions with two different GDLs.

Spraying and screen printing were compared as deposition methods for the same catalyst-ionomer combination and cell test results at 30% RH showed that the printed layer gave better performance (see Figure 6). This was shown using MIP to be due to larger pores within the sprayed layer, rather than any significant difference in total porosity, leading to faster water removal by evaporation. Note that the pore size has a very influential effect on the permeability of a porous layer (proportional to median pore diameter squared).

Figure 6: Comparison of printing and spraying deposition methods for the cathode catalyst layer in their effect on performance under dry conditions.

It was reasoned that Pt black layers may work well under dry conditions due the hydrophilicity of Pt (see above) and the thinness of the layer formed in the absence of a carbon support. Layers were made with a range of ionomer loadings and in some cases blended with plain carbon, of the type used as a catalyst support, to see if this influenced the performance by altering the layer thickness. In all cases the Pt black layers were significantly worse than Pt/C ones, giving a performance range of 425 to 480 mV at 1 A/cm² under 30/30% RH, compared to 610 mV for a typical high surface area supported Pt catalyst. In part, this can be explained by the low surface area of the Pt in the Pt black (see above). The other contributing factor was found to be the high porosity and large pores seen within the Pt black layers, as revealed by mercury porosimetry. These combine to give a layer with high permeability and thus high rates of water removal under dry conditions.

An obvious way to try and improve performance under dry conditions was to add more ionomer to the catalyst layer, although this can lead to a significant loss of porosity, which has a very damaging effect on performance, especially under wet operating conditions. Low EW ionomers could be used, to try and help to reduce the volumetric effect of extra ionomer addition. In this work the effect of different types and amounts of ionomer was investigated by measuring the effect on water uptake by DVS. In general, the more moles of sulphonic acid groups, the higher the water uptake under dry conditions, as shown in Figure 7. Note that the x-axis is expressed in moles of protons and it is assumed that one proton is associated with one sulphonic acid group. There is, however, no simple improvement in performance by adding more sulphonic acid groups. For a single catalyst type, performance tends to improve up to a maximum, where the increasing water uptake is due solely to an increase in number of

sulphonic acid groups, as shown in Figure 8. Note that a maximum is reached above which further increases do not improve performance. For a range of catalyst types, there is no simple correlation between number of sulphonic groups in the layer and dry performance; other factors come into play, such as the metal area, the layer porosity, the carbon type and the layer thickness.

Figure 7: Effect number of sulphonic acid groups on water uptake for a wide range of catalysts.

Figure 8: Effect of catalyst layer water uptake on air performance, where uptake is increased by adding more ionomer to the same catalyst type.

One of the key activities in this work was to improve the performance of the stabilised catalysts developed previously. The nature of the stabilised support made it difficult to increase the Pt metal area significantly and as the above data has shown, this sets a limit to the performance under dry conditions. Progress was made however and Figure 9 shows the trade-offs that were possible for a highly stabilised carbon support. The performance could be increased by about 75mV at 1 Acm⁻² over what had been achieved previously (350mV), without any loss of performance under wet conditions. To achieve further improvements under dry conditions however, losses were incurred under wet conditions. The best improvement seen was just over 150 mV at 30/30% RH, but with a penalty of about 80 mV at fully humidified conditions. This may be acceptable for certain applications, depending on the need for the stabilised catalyst and the balance of time spent under the different operating conditions.

Figure 9: Effect of cathode layer design modifications, using stabilised catalysts, on the MEA performance at high current density under a range of humidities. An MEA with an un-stabilised cathode catalyst included for comparison.

4. Summary of Accomplishments versus Objectives

The broad objectives of this project were to improve overall MEA performance under dry/hot operating conditions by tailoring the MEA components for such conditions, with the exception of the membrane. In detail, the following objectives and corresponding accomplishments were realised:

Objective	Accomplishment
Test thin, metal-only layers for better performance under dry conditions.	Layers tested, but no benefit seen on anode or cathode.
Increase dry performance by increasing cathode layer ionomer content, without causing flooding under wet conditions.	Accomplished. There is a catalyst-dependent limit for this approach: adding too much ionomer does not further increase dry performance and causes flooding when wet.
Show that GDLs can improve dry performance.	Accomplished. Less permeable GDLs can reduce dry out without causing flooding. An ionomer-bound MPL showed large improvements under hot/dry conditions.
Understand catalyst contribution to behaviour under dry conditions.	Accomplished. The key property of Pt-based catalysts that affects dry performance is the metal area (higher is better). The role of the carbon is secondary.
Demonstrate effect of cathode layer structure on water removal.	Accomplished. Dominated by median pore size with a lesser effect from total porosity. Performance dry can be improved by reducing pore sizes and total porosity but can rapidly lead to flooding under wet conditions.
Investigate layer alternative deposition methods and their effect on dry performance.	Accomplished. The effects are due to changes in the catalyst layer structure as described above.
Significantly improve the high current density performance of the highly stabilised catalysts under 30/30% RH conditions using, with minimal impact on fully humidified performance.	Accomplished, but with the caveat that the dry performance can only be increased up to a point with the stabilised catalyst, without affecting the wet performance (see Figure 9). The low metal areas on the stabilised catalysts limit the performance under dry conditions and attempts to increase the Pt area only gave modest improvements.

5. Other Products and Technology Transfer

Technique Development

a. Dynamic Vapour Sorption

It was not an objective of this work at the outset to develop an ex-situ method for determining water uptake by MEA components, but it became clear during the work that de-convoluting the effect of changes within the catalyst layer would require very large experimental matrices of in-cell testing. Thus the approach was taken to develop the DVS method to try and find correlations between in-cell behaviour and ex-situ affinity for water vapour under similar R_H and temperature conditions. This approach was very successful and has led to insights into the relative roles of carbon, Pt or other metal, and ionomer within the catalyst layer in holding water under dry conditions.

b. Pore Hydrophobicity Quantification

The method developed here is considered proprietary and was invented before the project started. It was used and improved during this project, however, to identify where changes in catalyst layer behaviour were due to changes in structure, as determined by mercury porosimetry, and where the changes were due to changes in hydrophobicity within the pores of the catalyst layer.

Technology Transfer

The immediate transfer of the technology developed in this project was into the activity with Protonex for use in their 750W stack for UAV applications. Other beneficiaries include customers of JMFC that operate their stacks under dry conditions, either at low or high temperature. As might be expected, some of the improvements developed in this work can be applied to systems that run very dry, as opposed to hot and dry.