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POROSIMETRY EQUIPMENT FOR THE ELUCIDATION OF THE STRUCTURAL ARCHITECTURE OF NOVEL MATERIALS DEVELOPED FOR FUEL CELL TECHNOLOGIES

SM Holmes and E P L Roberts

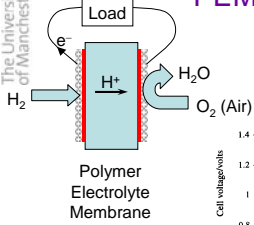
School of Chemical Engineering
University of Manchester

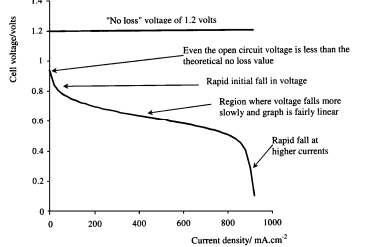


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PEM Fuel cells




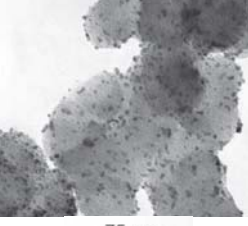


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Conventional catalyst structure





75 nm

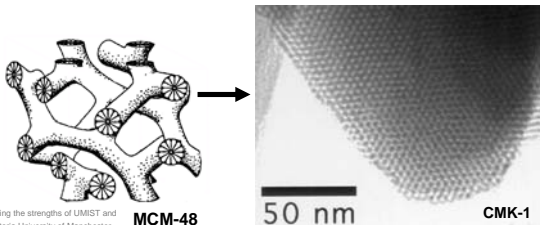
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Plan

- Use templated carbons and catalyst.
- Improved connectivity, utilisation & reactant access
- e.g. mesoporous carbons
R. Ryoo, S. H. Joo and S. Jun, *J. Phys. Chem. B*, 1999, **103**, 7743)



MCM-48

50 nm

CMK-1

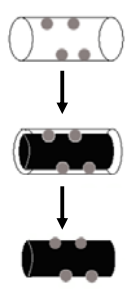
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Plan

- Mesoporous silica
- Metal salt impregnation
- Metal Oxidation/reduction
- Impregnation with sucrose
- Carbonisation
- Leaching of Silica template

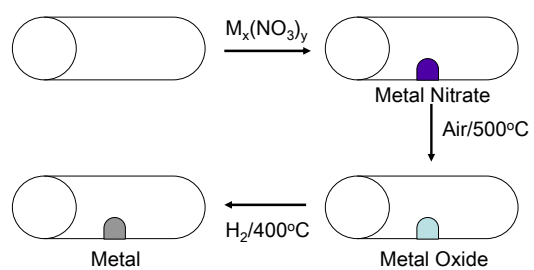


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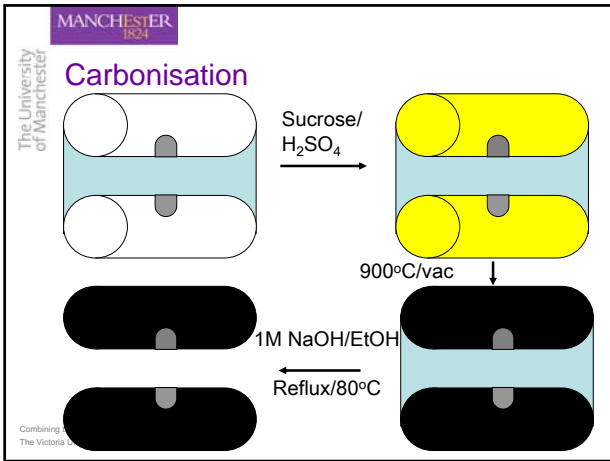
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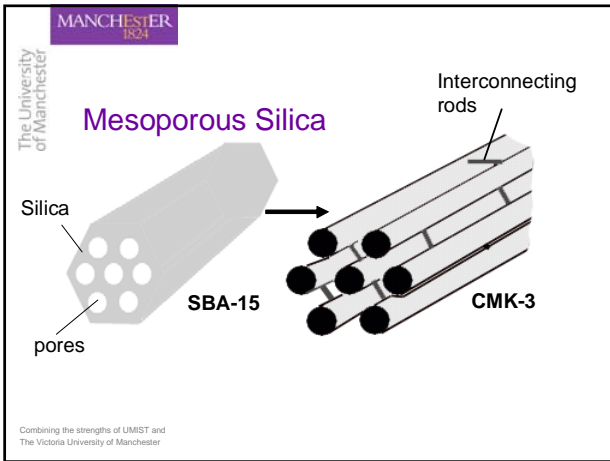
Metal Impregnation

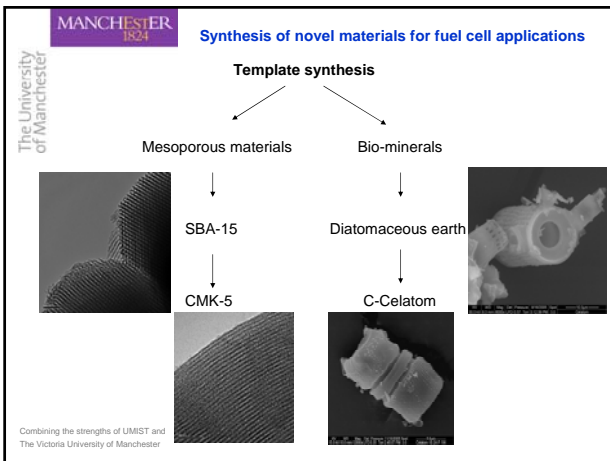


$M_x(NO_3)_y$
 Metal Nitrate
 Air/500°C
 Metal Oxide
 $H_2/400^\circ C$
 Metal

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Metal impregnation into the nanostructures

Two methods of metal impregnation were used: ***before and after carbonisation**

***S. M. Holmes, P. Foran, E. P. L. Roberts, and J. M. Newton, "Encapsulation of metal particles within the wall structure of mesoporous carbons," Chemical Communications, 2005, pages 1912-1913**

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CMK-5 electrocatalyst

Pt particles follows the pore structure of the carbon support

| Material | BET (m ² g ⁻¹) | Pore Diameter (nm) | Pore Volume (cm ³ g ⁻¹) |
|-----------------|---------------------------------------|--------------------|--|
| Plain CMK-5 | 1581 | 3.00 | 1.2 |
| 13 wt% Pt/CMK-5 | 900 | 2.8 | 0.62 |
| 23 wt% Pt/CMK-5 | 290 | 3.00 | 0.22 |

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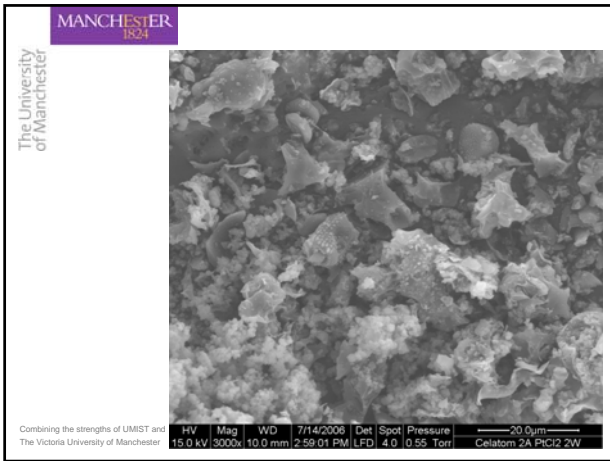
C-Celatom electrocatalyst

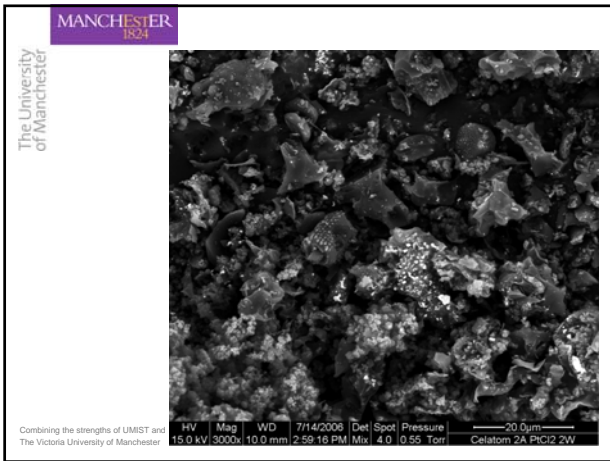
Carbon negative (C-Celatom)
Pt particles uniformly distributed throughout the novel carbon support

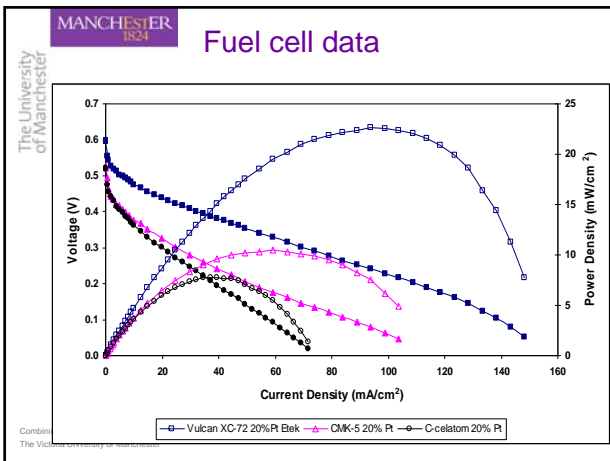
| Material | BET (m ² g ⁻¹) |
|------------------|---------------------------------------|
| Plain C-Celatom | 312 |
| 16 wt% Pt/C-Cela | 252 |
| 28 wt% Pt/C-Cela | 218 |

Vulcan XC-72 BET Surface Area 250 m²g⁻¹

S. M. Holmes, B. E. Granjel-Garcia, P. Foran, P. Hill, E. P. L. Roberts, B. H. Sakakini and J.M. Newton
 "A novel porous carbon based on diatomaceous earth"
 Chemical Communications, 2006, pages 2662 - 2663







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| Sample | Vulcan XC-72 20%Pt | CMK-5 20% Pt | C-celatom 20% Pt |
|--|-----------------------|------------------------|-----------------------|
| BET Surface area support only (N₂ ads) | 250 m ² /g | 1581 m ² /g | 312 m ² /g |
| Surface area platinum only (CO ads) | 31 m ² /g | 8 m ² /g | 0.5 m ² /g |
| Pt particle size | From TEM ~ 2 nm | From TEM ~ 3 nm | From SEM ~ 125 nm |

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Nafion-Mordenite Composite Membrane for Direct Methanol Fuel Cell Application

To develop a membrane with lower methanol permeability in order to achieve a superior DMFC performance to Nafion

Nafion

Mordenite

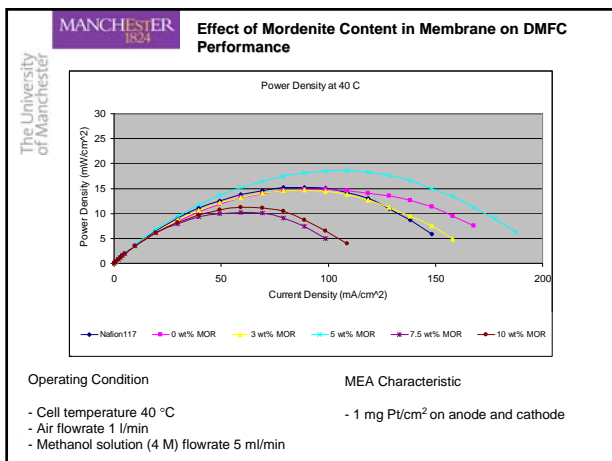
Nafion-Mordenite Composite Membrane

H⁺

CH₃OH

Nafion matrix

Mordenite



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A series of hierarchical porous structures have been synthesised using natural products as templates or scaffolds.

Initial work based around the growth of silicalite on to the surface of diatomaceous earth has been advanced by the development of the synthesis of zeolite Y, through a seeding / hydrothermal growth process

Seeding with colloidal zeolites

90 nm

Hydrothermal growth

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The growth of zeolite coatings using seeds prepared by ball milling of commercial zeolites opens up the production of these materials to a range of zeolitic structures which can be tailored for the ionic contaminant to be targeted.

Carbonised Olive pips
(before and after zeolite growth)

Carbonising natural structures offers a wide range of highly ordered supports with large contact areas. These materials show affinity for the growth of zeolites within their natural cavities.

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Coconut shell

Raw material

After carbonisation and synthesis

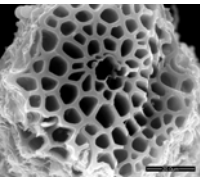
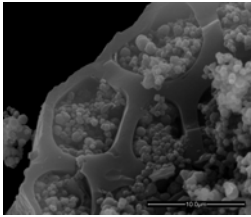
Additionally, the carbon supports can be removed by calcination, producing negative structures of their porous system.

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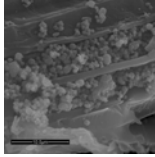
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Coconut fibres

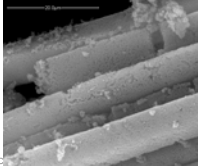
Coconut fibres are hierarchical structures with the highest surface area of all the tested materials

Zeolite crystals fill up the pores of the fibre



Once the carbon is removed, the crystals retain the cylindrical shape of the pore



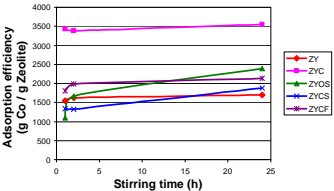
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These materials have been demonstrated to be very effective in the removal of **cobalt** ions from aqueous solutions.


For the case of **arsenic**, the adsorbents have achieved a **complete removal** when working with concentrations commonly found in polluted streams.

Hg porosimetry will be a powerful aid to characterise the nature of the composites' surfaces, in order to explain the mechanics behind their adsorption capacities.



| Stirring time (h) | ZY (red) | ZYC (green) | ZYOS (blue) | ZYCS (purple) | ZYCF (magenta) |
|-------------------|----------|-------------|-------------|---------------|----------------|
| 0 | 1500 | 1800 | 1600 | 1700 | 3500 |
| 5 | 1600 | 1900 | 1700 | 1800 | 3500 |
| 10 | 1700 | 2000 | 1800 | 1900 | 3500 |
| 15 | 1800 | 2100 | 1900 | 2000 | 3500 |
| 20 | 1900 | 2200 | 2000 | 2100 | 3500 |
| 25 | 2000 | 2300 | 2100 | 2200 | 3500 |

The aluminosilicate composition of the materials means that encapsulation of the cobalt ions is extremely effective when the material is vitrified to a glass at high temperature.




Leaching tests have proven that no ions are released even when exchangeable ions are present.

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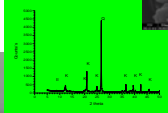
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REFINEMENT OF AHOKO NIGERIAN KAOLIN FOR ZEOLITES SYNTHESIS : STUDY BY PASCAL 140/240

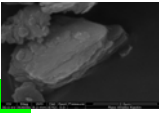
MERCURY POROSIMETRY




Nigeria map showing location



XRD pattern of Ahoko kaolin



SEM image of Ahoko kaolin



Representative sample of as-shipped Ahoko kaolin

COLLECTION OF REPRESENTATIVE SAMPLES OF AHOKO NIGERIAN KAOLIN FROM LOCATION IN NIGERIA AND ITS ANALYSIS BY XRD AND SEM


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Thank you!

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