

The Design and Study of Porous Metal Organic Framework (MOF) Structures.

By

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Declaration

I declare that this dissertation is my own, unaided work. It is being submitted for the degree of Master of Science in the University of the Witwatersrand, Johannesburg. It has not been submitted before for any degree or examination in any other university.

(Signature of candidate)

_____ day of _____ 201____.

Abstract

Metal Organic Frameworks are hybrid materials that can be modified by altering their fundamental components. This capability enables them to be tailored to suit specific applications, which range from catalysis to sensor technologies. Sensor based materials using MOFs technology have received a great deal of interest over recent years due to the potential advantages they offer with regard to monitoring devices. Therefore, in this project we attempted to systematically design and synthesise porous solid-state MOFs sensors using charge transfer (CT) phenomena as a basis for its “sensory” abilities.

CT host molecules/MOF linkers used in this work were based on pyromellitic diimide derivatives. These host molecules contain electrophilic as well as *trans* arranged carboxylic acid components, which allows the formation of CT complexes through CT $\pi \cdots \pi$ interactions and extended hydrogen bonding or metal coordination through the carboxylic acids. Two pyromellitic linkers were synthesised through condensation reactions, namely *N,N'*-bis(glyciny)-pyromellitic diimide (gly-L) and *N,N'*-bis(γ -aminobutyric)-pyromellitic diimide (but-L). The smaller gly-L host successfully formed CT complexes with all four aromatic hydrocarbons used in the work (naphthalene, anthracene, phenanthrene and perylene), whereas the larger but-L ligand selectively formed two novel CT complexes with phenanthrene and perylene.

All CT complexes obtained crystallised in the triclinic *P*-1 crystal system with the exception of gly-ANT (gly-L + anthracene) and but-PERY (but-L + perylene). The aromatic hydrocarbons formed 1:1 molecular complexes with each host molecule, thereby forming a stacked 2D layer. A $R^4_4(12)$ hydrogen bonding pattern was observed in the gly-ANT structure due to the incorporation of two solvent methanol molecules within the carboxylic acid bridges, whereas all other CT complexes formed conventional $R^2_2(8)$ dimers. Besides gly-ANT and but-PERY, all CT complexes form 2D parallel sheets with stabilisation in the third dimension achieved by various

intermolecular CH $\cdots$ O hydrogen bonding interactions between the host-host and host-guest molecules.

Lattice energy calculations using Gavezzotti's OPIX program suite were used to find common molecular arrangements as well as the relative stability of these arrangements in all the CT complexes. These included $\pi\cdots\pi$ stacking, and various hydrogen bond interactions. Various analysis techniques (X-Ray, thermal and spectroscopical) were employed to further assess the physical properties of these materials.

The *trans* arranged carboxylic acid groups of the CT host/linker molecules are somewhat unusual when compared to the usual linear linker approach utilised in MOF production. Both host linker molecules were utilised in MOF formation, however under the same synthetic conditions, gly-L showed an affinity to MOF formation, producing four new structures, whereas but-L did not. The use of divalent zinc and cadmium nitrates produced large MOF crystals at room temperature, while a cobalt (II) nitrate reaction mixture had to be cooled down to produce suitable crystals. SCXRD was successfully utilised to identifying the structural topology and bonding interactions of each MOF.

All metals used in this study, adopted typical coordination environments for *d*-block metals, with each structure containing solvent molecules within its unit cell. Solvent molecules play a vital role in the overall extension of the each structure through various hydrogen bonding interactions. With the exception of one zinc based MOF structure (MOF-Zn2), all structures contain bridging linkers that enable two dimensional extension leading to herringbone (MOF-Zn1) and step-like arrangements (MOF-Cd1 and MOF-Co1). The bonding characteristics and structural features of gly-L linker component were retained within all the MOF frameworks. Of the four structures obtained, only MOF-Zn2 and MOF-Cd1 formed 1D open pores of 56Å³ and 29Å³ respectively. Unfortunately due to structural instability and poor yields further inroads into MOFs with linkers using CT complexing for sensory capabilities could

not be achieved. This project illustrates many of the concepts and thoughts into applying rational design to the synthesis of functional MOF materials and the many problems associated with such studies.

Dedication

To my parents Abdul Carriem and Khadija, thank you for granting me this opportunity in furthering my education. For always providing me with assistance and continued motivation in undertaken this path. To my wife and daughter for all their support and help through the difficult times, and lastly to my brother and sisters, thanks for always understanding and putting up with me.

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Glossary

ArH	: Aromatic hydrogen.
Charge transfer	: CT.
Dimethyl formamide	:DMF.
but-L	: <i>N,N'</i> -bis(γ -butyric)-pyromellitic diimide.
but-PHEN	: Charge transfer complex containing phenanthrene.
but-PERY	: Charge transfer complex containing perylene.
CB	: Coordination bonding.
DSC	: Differential Scanning Calorimetry.
GC	: Glyciny carbonyl.
gly-L	: <i>N,N'</i> -bis(glyciny)-pyromellitic diimide.
gly-NAP	: Charge transfer complex containing naphthalene.
gly-ANT	: Charge transfer complex containing anthracene.
gly-PHEN	: Charge transfer complex containing phenanthrene.
gly-PERY	: Charge transfer complex containing perylene.
H	: Hydrogen.
HB	: Hydrogen bonding.
H-bond	: hydrogen bonds.
IC	: Imide carbonyl.
IR	: Infrared.
MOFs	: Metal Organic Frameworks.
MOF-Zn1	: First Metal Organic Framework containing zinc (II) metal.
MOF-Zn2	: Second Metal Organic Framework containing zinc (II) metal.
MOF-Cd1	: Metal Organic Framework containing cadmium (II) metal.
MOF-Co1	: Metal Organic Frameworks containing cobalt (II) metal.
PXRD	: Powder X-ray diffraction.
SBU	: Secondary building unit.
SCXRD	: Single crystal X-ray diffraction.

Solv	: Solvent.
TEA	: Triethylamine.
TGA	: Thermogravimetric analysis.
UV-Vis	: Ultraviolet-visible.