SYNTHESIS AND STRUCTURAL STUDIES OF COVALENT ORGANIC FRAMEWORKS PREPARED FROM POLYHEDRAL OLIGOSILSESQUIOXANE FOR NAPROXEN ADSORPTION

By

BALA SULEIMAN

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirement for the Degree of Doctor of Philosophy

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DEDICATION

To my lovely parents and my beloved, Late Mall Bala Musa and Malama Hajara Wakili Umar Gaya who always kept praying for me day and night to achieve my goal

To my family members:

and

To all my friends who supported me all these years

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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Faculty : Science

Covalent organic frameworks (COFs) are porous crystalline materials made up of organic components joined by strong reversible covalent bonds that have a persistent influence on the geometry and permeability of the arrangement. These substances are totally composed of light components such as H, B, C, N, O, and Si. Pharmaceuticals and personal care products (PPCPs) are an emerging problem as environmental contaminants. An "emerging toxin" such as naproxen which a nonsteroidal anti-inflammatory drug, is a toxic compound that has conquered or is manufactured in significant quantities in an ecosystem, causing some persistence and harm to living species. Hence, the development of polyhedral oligomeric silesquioxane (POSS) COFs as adsorbents for the removal of naproxen is crucial. POSS octa(phenyl) silesquioxane (OPS) was nitrated to produce octa(nitrophenyl) silesquioxane, which was then reduced to yield octa(aminophenyl)silesquioxane (OAPS). Four newly POSS COFs with various linkers, namely, COF-S4, OAPS with 1,5-dihydroxyanthraquionone (1,5-DHAQ, L_1); COF-S7, OAPS with 2-methylanthraquionone (2-MeAQ, L_2); COF-S12, OAPS with Terephthalaldehyde (TPA, L_3); COF-S14, OAPS with 1,8-dihydroxyanthraquionone (1,8-DHAQ. L_4) were successfully synthesised by solvothermal condensation method using Schiff base reaction ($R_1R_2C=NR'$), with a molar ratio 1:8 for OAPS to linker (L_1, L_2, L_3 and L_4), at temperature 120, 125, 100 and 120˚C for COF-S4, COF-S7, COF-S12 and COF-S14 respectively. The nanomaterials obtained were investigated using numerous spectroscopy techniques. The formation of large crystal lattice unit cells of the COFs frameworks was indicated by the peaks observed at low angles of less than 10°. The functional groups were investigated by FTIR which exhibited that the formation of the frameworks was attained through the Schiff base formation (C=N). Similarly, the Si−O−Si bonds for the synthesised COFs were all shown, which further proved that the materials were formed. ^{13}C and ^{28}Si CP-MAS NMR analysis confirmed the formation of the COFs through the C=O peaks in the range 180-200 ppm for the linkers and the existence of the C=N peaks in the range of 160-180 ppm for the nanomaterials produced. ^{28}Si NMR further affirmed the retention of silicone in the compounds after the synthesis. The COFs displayed excellent thermal durability for up to 400˚C for COF-S4 and COF-

S14, and 600°C for COF-S7 and COF-S12, respectively. The structural morphology FESEM of the compounds obtained displayed that the materials were nano crystals with nano-grain size pores and demonstrated the presence of all the expected elemental composition via EDX analysis. N_2 physisorption (BET) analysis demonstrated that the materials showed Type IV isotherm, and H3 hysteresis loop, which is a characteristic of mesoporous material. The remedied effluent was investigated, and a significant performance was recorded in the removal capability of the naproxen over COF-S4, COF-S7, COF-S12, and COF-S14 as 76%, 70%, 86% and 77% at a contact time of 210, 210, 270, and 270 min, respectively, at a constant dose of 0.05 g and pH 7. The maximum adsorption capabilities of the compounds were found to be 37, 35, 42, and 38 mg/g. The pH effect signifies that there is steady exclusion with a rise in pH to 9. At pH 9, the drop value was achieved for all COFs except for COF-S12 which was observed at pH 11, owing to the further negative charge, consequential to the repulsion between the synthesised COFs and naproxen solution. Investigation of the as-synthesised materials demonstrated admirable performance in reusability in the adsorption removal of naproxen. The as-synthesised COFs are envisioned as future adsorbents for removing anti-inflammatory drugs (AIDs) from water due to their ease of production, notable adsorption effectiveness, and admirable reusability.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

SINTESIS DAN PENCIRIAN STRUKTUR KERANGKA KOVALEN-ORGANIK DIHASILKAN DARIPADA POLIHIDRAL OLIGOSILSESKUIOXAN UNTUK PENJERAPAN NAPROSIN

Oleh

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Kerangka organik kovalen (COFs) ialah bahan hablur berliang yang terdiri daripada komponen organik yang dicantumkan oleh ikatan kovalen berbalik yang kuat yang mempunyai pengaruh berterusan ke atas geometri dan kebolehtelapan susunan. Bahan-bahan ini sepenuhnya terdiri daripada komponen ringan seperti H, B, C, N, O, dan Si. Produk farmaseutikal dan penjagaan diri (PPCPs) merupakan masalah yang muncul sebagai bahan cemar alam sekitar. "Toksin yang muncul" seperti naproxen yang merupakan ubat anti-radang bukan steroid, ialah sebatian toksik yang telah ditakluki atau dihasilkan dalam kuantiti yang ketara dalam ekosistem, menyebabkan beberapa ketekalan dan kemudaratan kepada spesies hidup. Oleh itu, pembangunan polihedral oligomerik silseskuioksana (POSS) COFs sebagai penjerap untuk penyingkiran naproxen adalah penting. POSS okta(fenil)silseskuioksana (OPS) telah dinitratkan untuk menghasilkan okta(nitrofenil)silseskuioksana, yang kemudiannya diturunkan untuk menghasilkan okta(aminofenil)silseskuioksana (OAPS). Empat COF POSS baharu dengan pelbagai penghubung, iaitu, COF-S4, OAPS dengan 1,5-dihidroksiantrakuinon (1,5-DHAQ, L_1); COF-S7, OAPS dengan 2-metilantrakuinon (2-MeAQ, L_2); COF-S12, OAPS dengan tereftalaldehid (TPA, L_3); COF-S14, OAPS dengan 1,8-dihidroksiantrakuionon (1,8-DHAQ. L_4) berjaya disintesis dengan kaedah pemeluwapan solvoterma menggunakan tindak balas asas bes Schiff ($R_1R_2C{=}NR'$), dengan nisbah molar 1:8 untuk OAPS kepada penyambung (L_1, L_2, L_3 dan L_4), pada suhu 120, 125, 100 dan 120°C masing-masing untuk COF-S4, COF-S7, COF-S12 dan COF-S14. Bahan nano yang diperolehi telah disiasat menggunakan pelbagai teknik spektroskopi. Pembentukan sel unit kekisi hablur besar kerangka COFs ditunjukkan oleh puncak yang diperhatikan pada sudut rendah kurang daripada 10°. Kumpulan berfungsi telah disiasat oleh FTIR yang menunjukkan bahawa pembentukan kerangka telah dicapai melalui pembentukan bes Schiff (C=N). Begitu juga, ikatan Si−O−Si untuk COF yang disintesis semuanya ditunjukkan, yang seterusnya membuktikan bahawa bahan telah terbentuk. Analisis CP-MAS NMR ^{13}C dan ^{28}Si mengesahkan pembentukan COF melalui puncak C=O dalam julat 180-200 ppm untuk penghubung dan kewujudan puncak C=N dalam julat 160-180 ppm untuk bahan nano yang dihasilkan. ^{28}Si NMR seterusnya

mengesahkan pengekalan silikon dalam sebatian selepas sintesis. COF menunjukkan ketahanan terma yang sangat baik sehingga 400°C untuk COF-S4 dan COF-S14, dan 600°C untuk COF-S7 dan COF-S12, masing-masing. Morfologi struktur FESEM bagi sebatian yang diperoleh menunjukkan bahawa bahan tersebut adalah hablur nano dengan liang saiz butiran nano dan menunjukkan kehadiran semua komposisi unsur yang dijangka melalui analisis EDX. Analisis fisierapan N_2 (BET) menunjukkan bahawa bahan menunjukkan isoterma Jenis IV, dan gelung histeresis H3, yang merupakan ciri bahan mesoporus. Efluen yang telah diperbaiki telah disiasat, dan prestasi yang ketara telah direkodkan dalam keupayaan penyingkiran naproxen ke atas COF-S4, COF-S7, COF-S12, dan COF-S14 sebagai 76%, 70%, 86% dan 77% pada masa sentuh masing-masing 210, 210, 270, dan 270 min, pada dos tetap 0.05 g dan pH 7. Keupayaan penjerapan maksimum sebatian didapati 37, 35, 42, dan 38 mg/g. Kesan pH menandakan bahawa terdapat pengecualian yang mantap dengan kenaikan pH kepada 9. Pada pH 9, nilai kejatuhan dicapai untuk semua COF kecuali COF-S12 yang diperhatikan pada pH 11, disebabkan oleh cas negatif selanjutnya, berbangkit tolakan antara COF tersintesis dan larutan naproxen. Penyiasatan terhadap bahan yang disintesiskan menunjukkan prestasi yang mengagumkan dalam kebolehgunaan semula dalam penyingkiran penjerapan naproxen. COF yang disintesis sebagai dibayangkan sebagai penjerap masa hadapan untuk mengeluarkan ubat anti-radang (AID) daripada air kerana kemudahan pengeluarannya, keberkesanan penjerapan yang ketara dan kebolehgunaan semula yang mengagumkan.

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This thesis submitted to the Senate of Universiti Putra Malaysia has been accepted as fulfillment of the requirements for the degree of Doctor of Philosophy. The members of Supervisory Committee are as follows:

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This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012- 2013) are adhered to

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>	More than
1,5-DHAQ	1,5-dihydroxyanthraquionone
1,8-DHAQ	1,8-dihydroxyanthraquionone
2MeAQ	2-Methylanthraquinone
AIDs	Anti-inflammatory drugs
ATR	Attenuated total reflection
BDBA	benzene diboronic acid
BET	Brunauer-Emmet-Teller
BUs	benzoylurea insecticides
CCOF 7	COF prepared 6,6′-dichloro-2,2′- diethoxy-1,1′-binaphthyl-4,4′-dialdehyde and orthogonal chiral tetrakis(4-aminophenyl) ethene
CCOF 8	COF synthesised from 6,6′-dichloro-2,2′- diethoxy-1,1′-binaphthyl-4,4′-dialdehyde and 1,3,5-tris(4-amino-3,5-diisopropylphenyl) benzene
C_f	Final concentration
C_O	Initial concentration
COF-1	COF made from self-condensation of benzene diboronic acid
COF-10	COF produced by condensation hexahydroxytriphenylene and biphenyldiboronic acid
COF-102	COF made by co-condensation tetrahedral tetra(4-dihydroxyborylphenyl) methane and of triangular 2,3,6,7,10,11-hexahydroxytriphenylene
COF-108	COF made from tetra(4-(dihydroxy) borylphenyl) methane with 2,3,6,7,10,11- hexahydroxytriphenylene
COF-300	COF generated by tetra-(4-anilyl) methane and terephthalaldehyde by condensation
COF-42	COF fabricated with 2,5-diethoxyterephthalohydrazide and 1,3,5-triformylbenzene

COF-43	COF prepared by condensation of 2,5-diethoxyterephthalohydrazide and 1,3,5-Tris(4-formylphenyl) benzene
COF-5	COF made from co-condensation of benzene diboronic acid and hexahydroxy tetriphenylene
COF-6	from boronic acid building blocks and 2,3,6,7,10,11-hexahydroxytriphenylene
COF-76	COF made from 1,3,6,8-tetrakis(p-formylphenyl) pyrene with the three-coordinate tris (4- aminophenyl) amine
COF-77	COF prepared from 1,3,6,8-tetrakis(p-formylphenyl) pyrene and benzene-1,4-dialdehyde
COF-78	COF made from 1,3,6,8-tetrakis(p-formylphenyl) pyrene pyromellitic dianhydride
COF-DL229	COF synthesised by the condensation reaction of 1,3,5,7-tetrakis(4-aminophenyl)-adamantane and 1,4-phthalaldehyde
COF-ETBA-DAB	4,4',4'',4'''-(ethane-1,1,2,2-tetrayl) tetra benzaldehyde (ETBA) and 1,4-diaminobenzene (DAB)
COF-LZU1	COF made from the co-condensation of 1,3,5-triformylbenzene and 1,4-diaminobenzene
COF-S12	COF prepared by condensation of octa (aminophenyl) silsesquioxane and terephthalaldehyde
COF-S14	COF prepared by condensation of octa(aminophenyl)silsesquioxane and 1,8-dihydroxyanthraquionone
COF-S4	COF prepared by condensation of octa (aminophenyl) silsesquioxane and 1,5-dihydroxyanthraquionone
COF-S7	COF prepared by condensation of octa (aminophenyl) silsesquioxane and 2-methylanthraquionone
COF-TpAzo	COF made from 1,3,5-triformylphloroglucinol and 4,4-azodianiline
CP-MAS	Cross Polarisation Magic Angle Spinning
Cr-MIL-101	MOF made from chromium (III) nitrate nanohydrate and terephthalic acid

CTF-1	COF made from trimerization of dicyanobenzene in molten $ZnCl_2$ to trimers and oligomers
CTF-2	COF synthesised via the condensation of 2,6-naphthalenedicarbonitrile in zinc chloride
Cu-MOF	MOF made from copper (II) nitrate trihydrate and 1,4-benzenedioic
CuP-SQ COF	COF processed by squaraine acid and 5,10,15,20-tetrakis(4-aminophenyl) porphyrin copper (II) as linkers
DCC	Dynamic covalent chemistry
DHAQ	Dihydroxyanthraquinone
DMAc	Dimethylacetamide
DMF	Dimethylformamide
DMSO	Dimethylsulphuroxide
FWHM	Full weight at half maximum
HHTP	Hexahydroxytriphenylene
HHTP-DPB COF	COF prepared from hexadroxytriphenylene and diphenyl boron
IRMOF-16	MOF generated from 1,4-di (4-carboxy-2-hydroxyphenyl) benzene as organic ligand and zinc nitrate hexahydrate
IRMOF-3	MOF prepared by refluxing 2-aminoterephthalic acid and zinc nitrate hexahydrate
K_F	Freundlich constant
K_L	Langmuir constant
KTP	Ketoprofen
LAG	Liquid-assisted grinding
MC	Mechanochemical
MTMS	Methyltrimethoxysilane
n	Heterogeneity of adsorption
NAP	Naproxen

NMP	N-methyl-2-pyrrolidone
NSAID	Non-steroidal anti-inflammatory drug
NU-125	MOF made from Cu (II) sulphate monohydrate and a hexa-carboxylic acid linker
OAPS	Octa(aminophenyl)silsesquioxane
ONPS	Octa(nitrophenyl)silsesquioxane
OPS	Octa(phenylsilsesquioxane)
Pa	*p*-phenylenediamine
PAHs	Polyaromatic hydrocarbons
PCBs	Polychlorinated biphenyls
Pc-PBBA-COF	COF made up of phthalocyanine macrocycles joined by 1,4-phenylene bis (boronic acid) linkers
PhACs	Pharmaceutical active compounds
PI-COF-4	COF made by pyromellitic dianhydride, reacts with the tetrahedral 1,3,5,7-tetraaminoadamantane
PI-COF-5	COF made from pyromellitic dianhydride and tetrahedral tetra(4-aminophenyl) methane
POPs	Persistent organic pollutants
POSS	Polyhedral oligomeric silsesquioxane
PPCPs	Pharmaceuticals and personal care products
PR (%)	Protein retention
PTA	1,4-phthaldehyde
PTSA	p-toluene sulphonic acid
PVC	Polyvinyl chloride
q_e	Adsorption capacity
q_{max}	Maximum adsorption capacity
q_t	Adsorption at equilibrium time

R^2	Correlation coefficient
RE	Removal efficiency
SBUs	Secondary building units
SCC-DFB	Self-consistent charge-density function tight -binding
$scCO_2$	Supercritical CO_2 activation
SPE	Solid-phase extraction
SPIO@COF	COF formed from 1,3,5-Tris (4-aminophenyl) benzene and 2,5-divinylterephthalaldehyde
SPIOsCOF	COF formed from 1,3,5-Tris (4-aminophenyl) benzene and 2,5-divinylterephthalaldehyde coated on the surface of superparamagnetic iron oxide nanoparticles
ST	Solvothermal
STPs	Samples of stormwater treatment practices
TAPA	Tetrahedral 1,3,5,7-tetrakis(4-aminophenyl)-adamantane
TFMS	Trifluoromethanesulfonic acid
THBP	Tetra(4-hydroxyborylphenyl) methane
Tp	2,4,6- triformylphloroglucinol
TPA	Terephthalaldehyde
TPBD	1,3,5-triformylphloroglucinol and benzidine
TpPa-COF-1	COF made from condensation of 2,4,6- triformylphloroglucinol and p-Phenylenediamine
TpPa-COF-2	COF made from condensation of 2,4,6- triformylphloroglucinol and 2,5-dimethyl-p-phenyldiamine
TPT-BD COF	COF modulated, through aldehyde-amine polycondensation process of 2,4,6-tris(4-formylphenoxy) and 1,3,5-triazine as vertices and 3,3'-dihydroxybenzidine
TPT-DHBD COF	Modulated, through aldehyde-amine polycondensation process of 2,4,6-tris(4-formylphenoxy) and 1,3,5-triazine (TPT-CHO) as vertices and benzidine (BD)

WTPs	Water treatment plants
WWTPs	Wastewater treatment plants
ZIF-8	Zeolitic imidazole framework made from zinc metal and 2-methylimidazole ligands

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