## Malaysian Journal of Analytical Sciences



## (MJAS)

Published by Malaysian Analytical Sciences Society

ISSN: 1394-2506



About this Journal V Info V MJAS Online

ANALIS

▼ Miscellaneous ▼ Contact



Malaysian Journal of Analytical Sciences (MJAS) ISSN: 1394-2506

**About this Journal** 

*Malaysian Journal of Analytical Sciences* (MJAS) is a peer-reviewed research journal that is devoted to the dissemination of new and original knowledge in all branches of analytical chemistry. Manuscripts submitted may be experimentally based or may be entirely theoretical and should contribute to any phase of analytical measurements and concepts including electrochemistry, mass spectrometry, environmental analysis, separation techniques, spectroscopy, instrumentation, surface analysis, bioanalysis, structures, chemometrics and data processing. Manuscripts dealing with conventional analytical methods should offer a significant, original application of the method, a noteworthy improvement or results on an important analyte.

*Malaysian Journal of Analytical Sciences* is indexed in **MyAIS** (Malaysian Abstracting and Indexing System), International Nuclear Information System (INIS Database) and SCOPUS (Yearly SJR 2016 0.152, SiteScore 0.32, Impact per Publication (0.245), Quartile Q4 (Analytical Chemistry) and H-index of 6). MJAS is also cited in MyCite, an indexing system owned by Malaysian Citation Centre (MCC), the Ministry of Higher Education (MOHE) Malaysia, with a 5-year Impact Factor of 0.034 (2016), Cited half-life of 3.8 and H-index of 3.

This Journal is published by the Malaysian Analytical Sciences Society or ANALIS as the sole scientific publication of the Society since 1995. From 2015, the Journal is published in six issues per volume (per year) i.e. in February, April, June, August, October and December.

*Malaysian Journal of Analytical Sciences* provides immediate open access to its content on the principle that making published articles freely access and downloadable to the public supports a greater global exchange of knowledge. Researchers from any countries are invited to publish their research articles in this journal.

#### **International Advisors**

Fortunato Servilla III (Philippines) Ganden Supriyanto (Indonesia) Hassan Y. Aboul-Enein (Egypt) Lee Hian Kee (Singapore) Muhammad Idiris Saleh (Malaysia) R. Narayanaswamy (U. Kingdom) Sukiman Sarmani (Malaysia) Wan Md Zin Wan Yunus (Malaysia)

#### **Editorial Board 2017**

Md Pauzi AbdullahEditorAhmad SaatMohdAmran Ab. MajidNor ABahruddin SaadNorh:LeeYook HengSuhaiMansor AhmadWanMhd Radzi AbasZaniMohd Marsin SanagiHarun Haji HamzahMan Mohd Afiq Wan Mohd KhalikExecutor

Editor in Chief Mohd Talib Latif Nor Azah Yusof Norhayati Mohd Tahir Suhaimi Suratman Wan Aini Wan Ibrahim Zani @ Zaini Assim

Managing Editor Executive Editor

© 2018 by The Malaysian Analytical Sciences Society Last update: 02 January 2018 by hhh



# Malaysian Journal of Analytical Sciences (MJAS)

Published by Malaysian Analytical Sciences Society ISSN: 1394-2506





### Malaysian Journal of Analytical Sciences Vol 21 No 6 2017

Articles 11 - 19 are selected papers from the "The 16th Asia-Pacific International Symposium on Microscale Separations and Analysis (APCE 2016), 7-10 November 2016, KSL Hotel & Resort, Johor Bahru, Johor; organised by Universiti Teknologi Malaysia and Malaysian Institute of Chemistry."

Guest Editor: Wan Aini Wan Ibrahim

Articles 20 - 26 are selected papers from the "Malaysia-Thailand-Japan International Conference on Science and Technology (MTJICST), 28 & 29 September 2016, UKM Graduate Centre; organised by Polymer Research Center (PORCE), School of Chemical Sciences and Food Technology, School of Applied Physics, Graduate Centre, Universiti Kebangsaan Malaysia." Guest Editor: Chia Chin Hua

Page

1210

 FLUORESCENCE AND EVAPORATIVE LIGHT SCATTERING HPLC PROFILING OF INTRACELLULAR ASPARAGINE (N)-LINKED OLIGOSACCHARIDES FROM Saccharomyces cerevisiae USING THE alg8 MUTANT (Pemprofilan KCPT Pendarfluor dan Penyerakan Cahaya Sejatan Oligosakarida Terpaut-(N) Asparagina Intrasel daripada Saccharomyces cerevisiae Menggunakan Mutan alg8) Iqbal Jalaludin, Amirul Husna Sudin, Ikram Mohd Said, Kamalrul Azlan Azizan, Syarul Nataqain Baharum, Abdul Munir Abdul Murad, Farah Diba Abu Bakar, Nor Muhammad Mahadi, Mark R. Wormald, Dominic S. Alonzi, Mukram Mohamed Mackeen

#### Malaysian Journal of Analytical Sciences Vol 21 No 6 2017

Articles 11 - 19 are selected papers from the "The 16th Asia-Pacific International Symposium on Microscale Separations and Analysis (APCE 2016), 7-10 November 2016, KSL Hotel & Resort, Johor Bahru, Johor; organised by Universiti Teknologi Malaysia and Malaysian Institute of Chemistry."

Guest Editor: Wan Aini Wan Ibrahim

Articles 20 - 26 are selected papers from the "Malaysia-Thailand-Japan International Conference on Science and Technology (MTJICST), 28 & 29 September 2016, UKM Graduate Centre; organised by Polymer Research Center (PORCE), School of Chemical Sciences and Food Technology, School of Applied Physics, Graduate Centre, Universiti Kebangsaan Malaysia." Guest Editor: Chia Chin Hua

1.	FLUORESCENCE AND EVAPORATIVE LIGHT SCATTERING HPLC PROFILING OF INTRACELLULAR ASPARAGINE (N)-LINKED OLIGOSACCHARIDES FROM <i>Saccharomyces cerevisiae</i> USING THE <i>alg8</i> MUTANT (Pemprofilan KCPT Pendarfluor dan Penyerakan Cahaya Sejatan Oligosakarida Terpaut-(N) Asparagina Intrasel daripada <i>Saccharomyces cerevisiae</i> Menggunakan Mutan <i>alg8</i> ) <i>Iqbal Jalaludin, Amirul Husna Sudin, Ikram Mohd Said, Kamalrul Azlan Azizan, Syarul Nataqain Baharum, Abdul Munir Abdul Murad, Farah Diba Abu Bakar, Nor Muhammad Mahadi, Mark R. Wormald, Dominic S. Alonzi, Mukram Mohamed Mackeen   Abstract &amp; References    Full Text: pdf ( 797 K )    DOI: 10.17576/mjas-2017-2106-01  </i>	1210
2.	SYNTHESIS OF BENZOTHIAZOLE DERIVATIVES USING ULTRASONIC PROBE IRRADIATION (Sintesis Terbitan Benzothiazola Dengan Menggunakan Prob Penyinaran Ultrasonik) <i>Su-Yin Kan, Wei Sem Yiong, Fu Siong Julius Yong, Poh Wai Chia</i>   Abstract & References    Full Text: pdf (549 K )    DOI: 10.17576/mjas-2017-2106-02	1219
3.	SYNTHESIS OF THIOUREA DERIVATIVES AND BINDING BEHAVIOR TOWARDS THE MERCURY ION (Sintesis Sebatian Terbitan Tiourea dan Sifat Pengikatan Terhadap Ion Merkuri) <i>Fatimatul Akma Awang Ngah, Emma Izzati Zakariah, Nurul Izzaty Hassan, Bohari Yamin, Suhaila Sapari, Siti Aishah Hasbullah</i>   Abstract & References    Full Text: pdf ( 475 K )    DOI: 10.17576/mjas-2017-2106-03	1226
4.	ENCAPSULATION OF GADOLINIUM NANOPARTICLES IN AMINO ACID BASED VESICLES (Pengkapsulan Nanozarah Gadolinium ke dalam Vesikel Berasaskan Asid Amino) <i>Muhammad Zul Azri Muhammad Jamil, Faizal Mohamed, Nur Ratasha Alia Md. Rosli, Irman Abdul Rahman</i>   Abstract & References    Full Text: pdf ( 1047 K )    DOI: 10.17576/mjas-2017-2106-04	1235
5.	DIKETOPIPERAZINE PRODUCED BY PSYCHROPHILIC YEAST <i>Glaciozyma antarctica</i> PI12 (Diketopiperazin Dihasilkan oleh Yis Psikropilik <i>Glaciozyma antarctica</i> PI12) <i>Andi Rifki Rosandy, Muntaz Abu Bakar, Nurul Nadiah Abdul Rahman, Abdul Munir Abdul Murad, Azira Muhamad, Rozida Mohd. Khalid</i> [Abstract & References    Full Text: pdf ( 446 K )    DOI: 10.17576/mjas-2017-2106-05	1243
6.	SOLVENT EXTRACTION OF THORIUM FROM RARE EARTH ELEMENTS IN MONAZITE THORIUM CONCENTRATE (Pengekstrakan Pelarut Torium daripada Unsur Nadir Bumi dalam Pekatan Torium Monazit) <i>Wadeeah M. Al-Areqi, Che Nor Aniza Che Zainul Bahri, Amran Ab. Majid, Sukiman Sarmani</i>   Abstract & References    Full Text: pdf (740 K )    DOI: 10.17576/mjas-2017-2106-06	1250
7.	PHOTOSTABILITY OF PLASTICIZED POLYVINYL CHLORIDE MEMBRANES: A THEORETICAL STUDY (Kestabilan Foto bagi Membran Plastik Polivinil Klorida: Satu Kajian Teori) <i>Oksana Fizer, Maksym Fizer, Yaroslav Studenyak</i>   Abstract & References    Full Text: pdf (1405 K)    DOI: 10.17576/mjas-2017-2106-07	1257
8.	POTENTIOMETRIC STUDY OF RHENIUM(V) COMPLEX FORMATION WITH AZATHIOPRINE AND CEFTRIAXONE (Kajian Potentiometri Penghasilan Kompleks Rhenium(V) dengan Azatioprina dan Ceftriaxon) <i>Gamal Abdelaziz Hussein Gouda, Gomaa Abdelgawad Mohammed Ali</i>   Abstract & References    Full Text: pdf ( 862 K )    DOI: 10.17576/mjas-2017-2106-08	1266
9.	SINTESIS MnO <sub>2</sub> NANOBUNGA POROS MENGGUNAKAN TEMPLAT SILIKA-APTES (Synthesis of Porous MnO <sub>2</sub> Nanoflower using Silica-APTES Template) <i>Siti Zubaidah Hasan, Mohamed Rozali Othman, Muhammad Rahimi Yusop</i>   Abstract & References II Full Text: pdf ( 826 K ) II DOI: 10.17576/mias-2017-2106-09	1276
10.	EXTRACTION OF METHYLPARABEN IN COSMETICS USING DISPERSIVE LIQUID-LIQUID MICROEXTRACTION BASED ON SOLIDIFICATION OF FLOATING ORGANIC DROP COUPLED WITH GAS CHROMATOGRAPHY FLAME IONIZATION DETECTOR (Pengekstrakan Metilparaben dalam Kosmetik Menggunakan Pengekstrakan Mikro Cecair-Cecair Serakan Berdasarkan Pemejalan Organik Terampai Berganding dengan Kromatografi Gas Pengesan Nyala Pengionan) Dyia Syaleyana Md Shukri, Nurul Asma Hamedan, Nur Shakirah Hussin, Wan Izhan Nawawi   Abstract & References    Full Text: pdf ( 672 K )    DOI: 10.17576/mjas-2017-2106-10	1289
11.	SENSORY EVALUATION AND QUANTITATIVE ANALYSIS OF MINERAL AND HEAVY METAL CONTENT OF PROTEIN SUPPLEMENT INCORPORATED WITH HERBAL EXTRACT (Penilaian Rasa dan Analisa Kuantitatif Kandungan Mineral dan Logam Berat bagi Makanan Tambahan Protin Digabungkan dengan Ekstrak Herba) Noor Azwani Zainol, Nor Farahiyah Aman Nor, HarisunYa'akob, Siti Nor Azlina Abd Rashid, Siti Hajar Mat Sarip, Ramlan Aziz LAbstract & References II Full Text: pdf (519 K) II DOI: 10.17576/mias-2017-2106-11 J	1299
12.	MOLECULARLY IMPRINTED TIO <sub>2</sub> INORGANIC FILM AND PVDF/TIO <sub>2</sub> COMPOSITE FILM AS SENSORS FOR THE DETECTION OF	1307

	CHEMICAL THREAT AGENTS USING QUARTZ CRYSTAL MICROBALANCE	
	(1102 FILETTI Tak Organik uati Filetti Komposit PVDF/1102 Molekui Tercetak sebagai Sensor untuk Mengesati Agen Kimia berbahaya Monggunakan Ponimbang Mikro Kristal Kuarza)	
	Abstract & References    Full Text: pdf ( 764 K )    DOI: 10.17576/mjas-2017-2106-12	
12		1216
15.	(Karbon Nitrida Taruhah Suai Kunnum Sebagai Sencor Dendafluor Ion Nitrat)	1210
	Siti Marvam Jasman. Hendrik Q. Lintang, Siew Ling Lee, Lenv Yuliati	
	Abstract & References    Full Text: pdf ( 1435 K )    DOI: 10.17576/mjas-2017-2106-13	
14.	MOLECULARLY IMPRINTED POLYMER-COATED QUARTZ CRYSTAL MICROBALANCE FOR DETECTION OF PARATHION	1327
	(Polimer Molekul Tercetak Saduran Kristal Kuarza Penimbang Mikro bagi Penentuan Parathion)	
	Xiamin Cheng, Qihui Zhang, Sam Fong Yau Li, Bin Liu	
	Abstract & References    Full Text: pdf ( 1039 K )    DOI: 10.17576/mjas-2017-2106-14	
15.	DETECTION OF BIOMOLECULES BY A NOVEL FLUORESCENCE APTASENSOR BASED ON POLYDOPAMINE SPHERES	1335
	(Pengesanan Biomolekul oleh Aptasensor Pendarfluor Novel Berasaskan Sfera Polidopamina)	
	Haryan Li and Sam Fong Yau Li	
	Abstract & References    Full Text: pdf ( 796 K )    DOI: 10.17576/mjas-2017-2106-15	10.10
16.	FLUORESCENCE QUENCHING ON MESOPOROUS CARBON NITRIDE BY PHENOL AND ANILINE (Relindanan Dendarfluor nada Karbon Nitrida Mesoliang oleh Fenel dan Anilina)	1342
	(Pelindapan) Pelidarindor pada Karbon Nitrida Mesoliarig Oleh Feliol dari Animita)	
	Abstract & References    Full Text: ndf ( 823 K )    DOI: 10.17576/mias-2017-2106-16	
17	EFFECT OF BUFFER COMPOSITION ON THE ANALYSIS OF LIDOPOLYSACCHARIDES FROM Escherichia coli 055-B5 AND LIT189 BY	1352
17.	CAPILLARY ELECTROPHORESIS WITH DIRECT UV DETECTION	1552
	(Kesan Komposisi Penimbal ke atas Analisis Lipopolisakarida dari Escherichia coli 055:B5 dan UT189 oleh Elektroforesis Kapilari	
	dengan Pengesan UV)	
	Fun Man Fung, Min Su, Sam Fong Yau Li	
	Abstract & References    Full Text: pdf ( 1296 K )    DOI: 10.1/5/6/mjas-201/-2106-1/	
18.	SPECIES AND SEROTYPE-SPECIFIC DETERMINATION OF ENDOTOXING USING LC-MS	1366
	(Penentuan Spesies dan Serotip-Khusus Endotoksin Menggunakan LC-MS)	
	Anna Karen Carrasco Laserna, Huatao Feng, Sam Fong Yau Li Abstract & References II Full Text: pdf ( 1030 K ) II DOI: 10.17576/miac-2017-2106-18 I	
10	SUBJEACE DI ASMONI DESONIANCE SENSOD FOD DETECTION OF DADATHION METHYL IN WATED	1272
19.	SURFACE PLASIMON RESONANCE SENSOR FOR DETECTION OF PARALITION METHTLIN WATER	13/3
	Baisheng Chen, Douglas Chiang Yon Chong, Xiamin Cheng, Bin Liu, Sam Fong Yau Li	
	Abstract & References    Full Text: pdf ( 960 K )    DOI: 10.17576/mjas-2017-2106-19	
20.	IN SITU HYDROGENATION OF LIQUID EPOXIDIZED NATURAL RUBBER USING DIIMIDE	1380
	(Penghidrogenan In Situ Getah Asli Cecair Terepoksida Menggunakan Diimida)	
	Nur Hanis Adila Azhar, Hamizah Md Rasid, Nur Aidasyakirah Mohd Tahir, Siti Fairus M. Yusoff	
	Abstract & References    Full Text: pdf ( 733 K )    DOI: 10.17576/mjas-2017-2106-20	
21.	ISOLATION OF TWO BOTRYANES FROM Hypoxylon rickii AND IDENTIFICATION OF THE ENCODING GENES	1389
	(Permencilan Sebatian Botrien dari Hypoxylon rickii dan Pengenalpastian Pengekodan Gen)	
	Allialii Alwi, Alui Kliki Kosalidy, Falali Diba Abu bakal, Kozula Pioliti Klialiu	
22		1200
22.	(Nanoemulsi Berasaskan Olein Sawit Sebagai Pembawa Vitamin E)	1399
	Suria Ramli, Norhakimah Norhman, Norhidayu Zainuddin, Safiah Mohamad Ja'afar, Irman Abdul Rahman	
	Abstract & References    Full Text: pdf ( 658 K )    DOI: 10.17576/mjas-2017-2106-22	
23.	STUDY ON THE EFFECT OF OIL PHASE AND CO-SURFACTANT ON MICROEMULSION SYSTEMS	1409
	(Kajian Kesan Fasa Minyak dan Ko-Surfaktan Terhadap Sistem Mikroemulsi)	
	Muhammad Asri Abd Sisak, Rusli Daik, Suria Ramli	
	Abstract & References    Full Text: pdf ( 1569 K )    DOI: 10.1/5/6/mjas-201/-2106-23	
24.	TENSILE AND THERMAL BEHAVIOURS OF POLY(LACTIC ACID)/LIQUID NATURAL RUBBER BLEND MATRIX INCORPORATED NICKEL	1417
	ZINC FERRITE NANOPARTICLES	
	(Silat Regaligali dali terma Matriks Adunan Asid Polilakuk/Cecali Getan Asil Diperkuat Nahozaran Niker Zirik Ferit) Ruev Shan Chen, Dalila Shahdan, Sahrim Ahmad	
	Abstract & References    Full Text: pdf ( 548 K )    DOI: 10.17576/mias-2017-2106-24	
25		1423
201	(Pengubahsuaian Kimia Minyak Sawit Terepoksida Untuk Aplikasi Biopelincir)	1120
	Nurazira Mohd Nor, Darfizzi Derawi, Jumat Salimon	
	Abstract & References    Full Text: pdf ( 1372 K )    DOI: 10.17576/mjas-2017-2106-25	
26.	CHARCOAL BASE METALLIC COMPOSITE ELECTRODE FOR WASTEWATER TREATMENT	1432
	(Elektrod Komposit Logam Berasaskan Arang Bagi Rawatan Air Buangan)	
	Majd Ahmed Jumaah, Mohamed Rozali Othman, Muhammad Rahimi Yusop, Jumat Salimon, Baydaa Khalaf, Wan Mohd Afiq Wan	
	Mona Knallk	
	ADBURGE & NEICECEUCES      UII TEAL, PUL ( OUT K )    DOI: 10.1/3/0/11/05-201/-2100-20	

 $\leftarrow$ 

Simago Journal & Country Rank Enter Journal Title, ISSN or Publisher Name

Home

Journal Rankings

ngs Counti

Country Rankings

Help

Viz Tools

About Us

## **Malaysian Journal of Analytical Sciences**

Country	Malaysia	6
Subject Area and Category	Chemistry Analytical Chemistry	U
Publisher	Faculty of Science and Technology, Universiti Kebangsaan Malaysia	H Index
Publication type	Journals	
ISSN	13942506	
Coverage	2010-ongoing	

#### Quartiles

highest values and Q4 (red) the lowest values.

Category	Year	Quartile
Analytical Chemistry	2011	Q4
Analytical Chemistry	2012	Q4
Analytical Chemistry	2013	Q4
Analytical Chemistry	2014	Q4
Analytical Chemistry	2015	Q4
Analytical Chemistry	2016	Q4

SJR journal i	is.		$\leftarrow$	Citations per document	+
Year	SJR				
2011	0.102				
2012	0.123				
2013	0.138				
2014	0.134				
2015	0.158				
2016	0.152				
_					
Tota	l Cites	Self-Cites	+		







 Show this widget in your own website 0

2011

2012

Just copy the code below and paste within your html code:

<a href="http://www.scimagojr.



2013

2014

2015

2016



MALAYSIAN JOURNAL OF ANALYTICAL SCIENCES

Published by The Malaysian Analytical Sciences Society

ISSN 1394 - 2506

#### FLUORESCENCE QUENCHING ON MESOPOROUS CARBON NITRIDE BY PHENOL AND ANILINE

(Pelindapan Pendarfluor pada Karbon Nitrida Mesoliang oleh Fenol dan Anilina)

Leny Yuliati<sup>1,2,3</sup>\*, Ahmad Hanami Abd Kadir<sup>4</sup>, Siew Ling Lee<sup>3,4</sup>, Hendrik O. Lintang<sup>1,2,3</sup>

<sup>1</sup>Ma Chung Research Center for Photosynthetic Pigments <sup>2</sup>Department of Chemistry, Faculty of Science and Technology Universitas Ma Chung, Villa Puncak Tidar N-01, Malang 65151, East Java, Indonesia <sup>3</sup>Centre for Sustainable Nanomaterials, Ibnu Sina Institute for Scientific and Industrial Research <sup>4</sup>Department of Chemistry, Faculty of Science Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia

\*Corresponding author: leny.yuliati@machung.ac.id

Received: 7 November 2016; Accepted: 18 September 2017

#### Abstract

In this study, fluorescence quenching on mesoporous carbon nitride (MCN) was examined in the presence of phenol and aniline. The MCN was synthesized by thermal polymerization of cyanamide using nanocolloidal silica (7 nm) as a hard template and characterized by X-ray diffractometer (XRD), Fourier transform infrared (FTIR), transmission electron microscope (TEM), specific surface area, pore size distribution, and fluorescence spectrophotometer. The MCN showed two excitation wavelengths at 275 and 370 nm, owing to the presence of N=C and N-C terminal groups, respectively. These two excitation wavelengths gave only one emission peak at around 460 nm. Either in the presence of phenol or aniline, the emission intensity of the MCN was confirmed to be quenched in a linear function towards the concentration of phenol or aniline as the quencher molecule. N-C terminal groups were found to have slightly better interactions to phenol and aniline as compared to the N=C groups. Since the emission intensity of MCN was more quenched by phenol molecules than the aniline, it was proposed that the MCN would have stronger interaction to phenol than aniline. This result was also supported by the adsorption data, in which the MCN gave higher adsorption towards phenol than the aniline.

Keywords: mesoporous carbon nitride, phenol, aniline, fluorescence quenching, adsorption

#### Abstrak

Dalam kajian ini, pelindapan pendarfluor pada karbon nitrida mesoliang (MCN) telah diperiksa dengan kehadiran fenol dan anilina. MCN telah disintesis secara pempolimeran haba sianamida menggunakan silika nanokoloid (7 nm) sebagai templat keras dan telah dicirikan dengan pembelauan sinar-X (XRD), inframerah transformasi Fourier (FTIR), mikroskop elektron transmisi (TEM), luas permukaan, taburan saiz liang, dan spektroskopi pendafluor. MCN menunjukkan dua panjang gelombang pengujaan pada 275 dan 370 nm oleh kerana kehadiran masing – masing N=C dan N-C sebagai kumpulan terminal. Kedua-dua panjang gelombang pengujaan memberikan hanya satu puncak pelepasan pada kira-kira 460 nm.Sama ada dengan kehadiran fenol atau anilina, keamatan pemancaran daripada MCN disahkan mengalami pelindapan dalam fungsi linear terhadap kepekatan fenol atau anilina berbanding dengan kumpulan N=C. Oleh kerana keamatan pemancaran MCN didapati lebih baik sedikit untuk fenol dan anilina dengan anilina, adalah dicadangkan bahawa MCN berinteraksi lebih kuat dengan fenol dibandingkan dengan anilina. Keputusan ini juga disokong oleh data penjerapan, dengan MCN memberikan penjerapan yang lebih tinggi terhadap fenol dibandingkan dengan anilina.

Kata kunci: karbon nitrida mesoliang, fenol, anilina, pelindapan pendarfluor, penjerapan

#### Introduction

Carbon nitrides (CN) are carbon materials in which some carbon atoms in the graphite-like structure are replaced by some nitrogen atoms in a regular manner. Generally, CN stands for a large family of related compounds  $(C_xN_y)$  [1 – 3]. Even though the CN has been reported since the 1990s, this material still receives considerable attention because of its versatility until nowadays. The CN is used in various applications while the material is just composed of C, N and H atoms [4]. Other factors that make CN being well explored is because of the abundance of these atoms on earth, non-toxic and easy to be synthesized into a desired shape where the pore of carbon nitride is tunable to form mesoporous carbon nitride (MCN) [1, 5, 6]. MCN has a larger specific surface area than the CN and it facilitated better performance for various applications [7 – 12]. Moreover, CN has many advantageous properties that make it an interesting material, such as high thermal and chemical stability, and great optical properties [13 – 15].

Recently, application of CN and MCN as fluorescence sensor has been a significant interest. Owing to the presence of fluorescent active sites (tri-*s*-triazine units), CN and MCN are potential materials to be used as an optical sensor. Despite of the fact that these materials have a strong fluorescence property, less attention was paid on its sensing ability until there was a report showing the utilities of cubic MCN as all-in-one chemosensor for sensing of trace amount of metal ions in aqueous solution [16]. Since the cubic MCN has the highest sensitivity and selectivity towards copper ions, further modification was also carried out by the research group on the cubic MCN incorporating copper ions for sensing of cyanide ions, which are very poisonous and used abundantly in metal plating industry [17]. CN has been also reported as an efficient fluorosensor for detection of copper ions. It was suggested that the fluorescence quenching was due to strong coordination of the Lewis basic sites on the CN with the Cu ions [18]. Besides metal and cyanide ions, recent studies showed that nitrogen-containing compounds can be also detected by fluorescence quenching. It was reported that MCN was able to be used as a fluorescence sensor for *N*-nitrosopyrrolidone (NPYR) [7]. The surface interaction was proposed to involve electrostatic interactions between the electropositive N and electropositive N at the NPYR molecules and terminal N-C groups of the MCN, respectively. CN was also reported as a fluorescence chemical sensor for nitrate ions [19].

In this study, fluorescence quenching on MCN was investigated in the presence of aromatic compounds, which are phenol and aniline. Phenol is considered as one of the organic pollutants that can be easily found in industrial wastewater [20 - 22]. The wastewater containing phenol may cause the contamination of groundwater and it will give bad effects for ecosystems and also for human. Meanwhile, aniline is also recognized as one of the high priority pollutants that are released from several industries, such as various manufacturers of dyes, and rubber, which caused wide distribution of aniline in the environment [23 - 25]. As an adsorbent, MCN possesses well-ordered porosity, high surface area, and basic groups (NH and NH<sub>2</sub>) in the structures, which are the important criteria for the removal of organic pollutants. Due to the nitrogen rich structure of MCN with basicity property, it showed remarkable adsorptions towards one of the acidic persistent organic pollutants, *i.e.* phenol [26]. On the other hand, the MCN was also reported showing superior capability to adsorb aniline than the CN and MCM-41 [8, 9]. Therefore, based on these reported studies, the fluorescence quenching on MCN by phenol and aniline is feasible. This study demonstrated that MCN can be also used as a fluorescence sensor for the aromatic compounds.

#### **Materials and Methods**

#### **Preparation of MCN**

The MCN was prepared by a thermal polymerization of cyanamide as the precursor, in the similar way to the method reported in other literatures [7 - 9]. The cyanamide was mixed with the colloidal silica as a hard template 7 nm (mass ratio was 1:1), followed by stirring and heating at 363 K until a white powder was formed. The obtained white powder was ground and then heated at a temperature of 823 K for 4 hours. In order to obtain the MCN, the silica template was removed by treating the MCN powder with the NH<sub>4</sub>HF<sub>2</sub> solution (4 M, 400 mL) under vigorous stirring and also with ethanol and distilled water. This washing step was repeated for three times. After washing and filtration, the powder was dried at 343 K overnight in an oven.

#### Characterizations

The structure of the MCN was determined by an X-ray diffractometer (XRD) on a Bruker D8 Advance Diffractometer at ambient temperature, with a scan rate of 0.05 s<sup>-1</sup> using Cu-K $\alpha$  radiation where  $\lambda$  equals to

1.5406 Å at 40 kV and 40 mA. To determine the structural information and functional groups of the MCN, a Fourier transform infrared spectroscopy (FTIR) was employed using a FTIR Nicolet iS50 Thermo Scientific. For pellet preparation, a small amount of the MCN was mixed with potassium bromide (KBr) and put under a pellet press. To obtain a better background correction, nitrogen gas was purged into the system before the analysis to remove all humidity. Morphology of the MCN was measured using a transmission electron microscope (TEM) on a JEOL JEM-2100. The MCN powder was first dispersed well in acetone, and then put onto the copper grid to be observed with the TEM. The surface area of the MCN was determined by using the Brunauer-Emmet-Teller (BET) equation, while the pore size of the MCN was determined using the Barret-Joyner-Halenda (BJH) from adsorption-desorption isotherm at 77 K obtained on a Quanta chrome Autosorb-1 instrument. The fluorescence properties were measured using a JASCO Spectrofluorometer FP-8500 to obtain the excitation and emission spectra. The excitation wavelengths used to monitor the emission spectra were 275 and 370 nm, while the emission wavelength used to monitor the excitation spectra was 460 nm. The analysis was carried out at room temperature.

#### **Fluorescence quenching**

The quenching tests were carried out with various concentrations of phenol and aniline on the MCN (50 mg) at room temperature. The excitation wavelengths at 275 and 370 nm of the MCN were determined by observing the emission spectrum at 460 nm. Meanwhile, the emission wavelength was determined by observing excitation spectra at both excitation wavelengths. From various amounts of phenol solutions (300 - 600 ppm),  $20 \mu$ L of the solution was taken and then introduced onto the MCN for the quenching test for phenol. The changes in the emission spectra of the MCN were then measured at excitation wavelengths of 275 and 370 nm. As for the quenching test for aniline, the same steps were followed using various concentrations of aniline solution (200 - 700 ppm).

#### **Adsorption tests**

The adsorption process of phenol or aniline onto the MCN was conducted under dark condition using various concentrations of phenol (60 - 400 ppm) and aniline (100 - 500 ppm) at room temperature. Different series of concentrations were selected due to the different behaviors of the observable adsorption. A magnetic bar was put in a 30 mL beaker and 10 mL of phenol or aniline solution was poured into the beaker together with the MCN (50 mg). Inside a closed box, the beaker containing the mixture was placed on the stirring plate and stirred for 6 hours. After filtration and separation from the MCN, the solution was analysed using gas chromatography with a flame ionization detector (GC-FID) on an Agilent 7820AGC-FID. The adsorbed amount was determined by subtracting the initial phenol or aniline with the amounts of remaining phenol or aniline after the adsorption test.

#### **Properties of MCN**

#### **Results and Discussion**

Shown in Figure 1(a) is the XRD pattern of the prepared MCN, which gave two diffraction peaks at  $2\theta$  of 13.1 and 27.4°. The peak at  $2\theta$  of 13.1° was corresponding to the distance of 0.66 nm, which indicated the in-planar repeating units and structural packing motif, such as hole-to-hole distance of the nitride pores, while the intense diffraction peak at the  $2\theta$  of 27.4° could be indexed as (002) for graphitic materials, giving an interlayer distance of 0.325 nm for the graphite-like stacking of the conjugated aromatic CN layers. This result is in good agreement with values reported elsewhere [4, 7 – 11, 13, 14]. Therefore, it can be concluded that the MCN sample was successfully synthesized.

The functional groups present on the MCN were investigated by FTIR spectroscopy. As shown in Figure 1(b), the absorption peaks of the MCN were observable at the region of 809, 1200 - 1700 and 3000 - 3700 cm<sup>-1</sup>. The bands at 809, 1252, 1319, 1428 and 1646 cm<sup>-1</sup> could be attributed to the vibration modes of heterocyclic tri-*s*-triazine ring (C<sub>6</sub>N<sub>7</sub>) units, while the broad peak at 3163 cm<sup>-1</sup> could be assigned to two overlapping bands of stretching modes of N-H and O-H groups [9, 11, 27]. The peak at 2177 cm<sup>-1</sup> could be attributed to the presence of C=N or N=C=N group, which was reported in MCN prepared via thermal polymerization reaction [27]. These FTIR spectra supported that the MCN sample was successfully prepared.



Figure 1. (a) XRD pattern and (b) FTIR spectrum of MCN

The morphology of the prepared MCN was observed by TEM as shown in Figure 2. It can be seen that the MCN has uneven layers due to the graphitic structures of the MCN. Figure 3(a) shows the nitrogen adsorption-desorption isotherms and Figure 3(b) shows the Barrett-Joyner-Halenda (BJH) pore size distribution of the MCN. The results suggested that the MCN has type IV isotherm for mesoporous material with hysteresis loop at relative pressure of 0.5 - 1 due to the capillary condensation in pores. The BET specific surface area of the MCN was determined to be 287 m<sup>2</sup> g<sup>-1</sup>. Meanwhile, for BJH pore size distribution, the MCN showed a narrow and sharp pore size of 7 nm. This pore size reflected the size of the silica template that was used for the preparation of the MCN.



Figure 2. TEM images of MCN shown in different magnification scale bars, (a) 100 nm and (b) 20 nm



Figure 3. (a) Nitrogen adsorption-desorption isotherm and (b) BJH pore size distribution of MCN

Figure 4(a) displays the excitation spectrum of the MCN. It was clear that the MCN exhibited two excitation peaks at 275 and 370 nm. The excitation peak at 275 nm can be attributed to the  $\pi$  to  $\pi^*$  electronic transition of the N=C groups in the aromatic 1,3,5-triazine ring of MCN. Meanwhile, the other excitation peak at 370 nm can be attributed to *n* to  $\pi^*$  electronic transition of a terminal N-C group involving lone pairs of nitrogen atoms. As can be seen from Figure 4(b), the MCN gave one emission peak at a wavelength of 460 nm when the MCN was excited either at 275 or 370 nm. These results agree well with previous reports [7, 9, 19].



Figure 4. (a) Excitation and (b) emission spectra of MCN that were monitored at emission wavelength of 460 nm and excitation wavelengths of 275 and 370 nm

#### **Fluorescence quenching**

Quenching tests were conducted on the MCN by the addition of various concentrations of phenol or aniline in order to investigate the sites where the interactions occurred either at N=C groups or terminal N-C groups. The quenching tests were determined by observing the changes in the emission intensity of the MCN sample before and after the addition of phenol or aniline. Since one emission spectrum at around 460 nm was observed for both excitations at 275 and 370 nm, the quenching tests were carried out by monitoring the intensity of the emission peak at 460 nm excited at both excitation wavelengths. As illustrated in Figure 5(a) and (b), the emission intensities of the MCN

excited at both excitation wavelengths of 275 and 370 nm (N=C and terminal N-C emission sites), respectively decreased with the addition of the phenol. This result showed that phenol acted as a quenching agent to deactivate the emission sites. When the added phenol concentration increased, the emission intensity was further decreased. The decrease in the emission intensity would be due to the interactions between the emission sites of MCN at an emission wavelength of 460 nm and the added phenol.



Figure 5. Fluorescence quenching on MCN in the absence and presence of phenol (0–600 ppm) when excited at (a) N=C and (b) terminal N-C groups

In order to have a better understanding about the interaction of MCN with phenol, the quenching results were plotted according to the Stern-Volmer plot following the equation 1:

$$Io/I = K_{SV}[Q] + 1 \tag{1}$$

where *Io* and *I* are fluorescence intensities observed in the absence and presence of phenol, Q is the concentration of phenol, and  $K_{SV}$  is the Stern-Volmer quenching constant.

Based on the Stern-Volmer equation, the relative emission intensity would be expressed as the function of phenol concentration when the same amount of MCN and the constant illumination intensity were taken in account. Figure 6 shows the Stern-Volmer plots of the relative emission intensity of MCN with various concentrations of phenol. In the range of 300 - 600 ppm, the relative emission intensity excited at both emission sites gave a linear Stern-Volmer plot with the concentrations of phenol. The linear Stern-Volmer plot indicated that the MCN could detect phenol using the fluorescence spectroscopy. Meanwhile, the quenching efficiency of phenol on the emission site of MCN could be obtained from the slope of the linear plot, which was matched to the quenching rate constant ( $K_{SV}$  value). It was shown that the  $K_{SV}$  values for the emission sites monitored at excitation wavelengths of 275 and 370 nm were  $4 \times 10^{-4}$  and  $5 \times 10^{-4}$  ppm<sup>-1</sup>, respectively. These results suggested that the emission sites of MCN excited at 275 and 370 nm have almost equal interactions with phenol, which the terminal N-C groups gave slightly better interaction than the N=C groups.

Figure 7(a) and (b) show the changes on the emission intensities excited at 275 and 375 nm, respectively in the presence of aniline. Similar to the phenol addition, the addition of aniline quenched the intensity of the MCN. Aniline also acted as a quenching agent that deactivated the emission sites of MCN. When the concentration of aniline increased, the emission intensity was also further reduced. The decrease in the emission intensity of the MCN suggested that there were interactions between the N=C and terminal N-C emission sites and the aniline. As previously mentioned, the MCN has two excitation sites at 275 and 370 nm, which belong to the N=C groups

 $(\pi - \pi^*)$  in the aromatic 1,3,5-triazine and the terminal N-C groups  $(n - \pi^*)$ , respectively. In order to investigate which sites were favoured to the aniline, the Stern-Volmer plot was performed.



Figure 6. Stern Volmer plots of phenol quenching on MCN at N=C and terminal N-C groups



Figure 7. Fluorescence quenching on MCN in the absence and presence of aniline (0 - 700 ppm) when excited at (a) N=C and (b) terminal N-C groups

Shown in Figure 8 is the linear Stern-Volmer plots between the relative emission intensity of MCN with various concentrations of aniline in the range of 200 –700 ppm. Both emission sites, the N=C and terminal N-C groups showed the linear Stern-Volmer plots with the addition of aniline.  $K_{SV}$  values obtained were  $3 \times 10^{-4}$  and  $4 \times 10^{-4}$  ppm<sup>-1</sup> for the N=C and terminal N-C groups, respectively. The slightly higher  $K_{SV}$  value of the terminal N-C group suggested that similar to the results when using phenol as the quencher agent, aniline also favored the terminal N-C groups. Based on the fluorescence quenching tests, it was demonstrated that the MCN showed good interactions with phenol and aniline.



Figure 8. Stern Volmer plots of aniline quenching on MCN at N=C and terminal N-C groups

#### **Adsorption tests**

Adsorption capability of the MCN towards phenol or aniline was examined by using different concentrations. The adsorption test for phenol was carried out at room temperature with the concentration of phenol was varied from 60 to 400 ppm. Figure 9 presents the results of the adsorption test of phenol on the MCN. The amount of adsorbed phenol increased with the increase of the phenol concentration when it was below 100 ppm. Further increase of the phenol concentration at more than 100 ppm did not affect much the amount of adsorbed phenol. Figure 9 also shows the results of aniline adsorption on the MCN with various concentrations from 100 to 500 ppm. Separated experiments confirmed that no aniline adsorption occurred when the aniline concentration was lower than 100 ppm. At concentration of 100 ppm and above, the MCN showed an increased amount of adsorption with the increase of aniline concentration up to 500 ppm. Compared to the adsorption of phenol, higher amount of aniline was required for MCN to act as an adsorbent.



Figure 9. Adsorption of phenol and aniline with various concentrations on MCN at room temperature

From the adsorption test analyses, it can be proposed that phenol and aniline were successfully adsorbed on the MCN. However, under the same concentration for instance at 100 ppm, the MCN gave better adsorption towards phenol than the aniline, which gave adsorbed amounts of 2.16 and 0.02 ppm, respectively. The higher adsorbed

amount of phenol than aniline might be due to the fact that phenol is a weak acid, while aniline is a weak base. Therefore, basicity of the MCN would favor phenol rather than aniline. It can be proposed that phenol would give stronger interaction to the MCN, which in turn gave higher  $K_{SV}$  value than the aniline. As has been reported elsewhere [7], the presence of electronegative N at the terminal N-C groups of the MCN provided the possibility for the formation of electrostatic interactions towards the analyte molecules. As phenol is a weak acid, the acid-base electrostatic interactions would occur between the electronegative N at the terminal N-C groups of the MCN and the electronegative phenolic proton. As for aniline, the electrostatic interactions would occur between the electronegative H at the NH<sub>2</sub> group of the aniline, which was less electropositive than the phenolic proton.

#### Conclusion

Characterizations by XRD, FTIR, TEM, surface area and pore size analyzer, and fluorescence spectroscopy confirmed that the MCN was prepared successfully by thermal polymerization of cyanamide using nanocolloidal silica as the hard template. The MCN was shown to have good interactions with phenol and aniline, where its emission intensity was quenched almost linearly with the addition of these aromatic compounds. It was obtained that the terminal N-C groups gave slightly higher quenching efficiency than the N=C groups for both phenol and aniline. Phenol gave better interactions towards the emission sites of the MCN, owing to the stronger adsorption of phenol on to the MCN than the aniline caused by the acid-base electrostatic attractive force.

#### Acknowledgement

This work was financially supported by the Ministry of Higher Education (MOHE, Malaysia) and the Universiti Teknologi Malaysia (UTM, Malaysia) through Flagship Research University Grant (cost center code: Q.J130000.2426.00G07).

#### References

- 1. Vinu, A. (2008). Two-dimensional hexagonally-ordered mesoporous carbon nitrides with tunable pore diameter , surface area and nitrogen content. *Advanced Functional Materials*, 18(5): 816 827.
- 2. Teter, D. M. and Hemley R. J. (1996). Low-compressibility carbon nitride. Science, 271(5245): 53 55.
- 3. Corkill, J. L. and Cohen, M. L. (1993). Calculated quasiparticle band gap of β-C<sub>3</sub>N<sub>4</sub>. *Physical Review B*, 48(23): 17622 17624.
- 4. Goettmann, F., Fischer, A., Antonietti, M. and Thomas, A. (2006). Chemical synthesis of mesoporous carbon nitrides using hard templates and their use as a metal-free catalyst for Friedel-Craft reaction of benzene. *Angewandte Chemie International Edition*, 45(27): 4467 4471.
- 5. Gillan, E. G. (2000). Synthesis of nitrogen-rich carbon nitride networks from an energetic molecular azide precursor. *Chemistry of Materials*, 12(12): 3906 3912.
- 6. Komatsu, T. (2001). Attempted chemical synthesis of graphite-like carbon nitride. *Journal of Materials Chemistry*, 11: 799 801.
- Sam, M. S., Lintang, H. O., Sanagi, M. M., Lee, S. L. and Yuliati, L. (2014). Mesoporous carbon nitride for adsorption and fluorescence sensor of N-nitrosopyrrolidine. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, 124: 357 – 364.
- 8. Sam, M. S., Lintang, H. O., Sanagi, M. M., Lee, S. L. and Yuliati, L. (2014). Adsorption of aniline using novel mesoporous carbon nitride. *Advanced Materials Research*, 95: 135–139.
- 9. Sam, M. S., Tiong, P., Lintang, H. O., Sanagi, M. M., Lee, S. L. and Yuliati, L. (2015). Mesoporous carbon nitride as a metal-free catalyst for the removal of aniline. *RSC Advances*, 5: 44578 44586.
- Lee, S.C., Lintang, H.O. and Yuliati, L. (2012). A urea precursor to synthesize carbon nitride with meso porosity for enhanced activity in the photocatalytic removal of phenol. *Chemistry An Asian Journal*, 7(9): 2139 - 2144.
- 11. Cui, Y., Zhang, J., Zhang, G., Huang, J., Liu, P., Antonietti, M. and Wang X. (2011). Synthesis of bulk and nanoporous carbon nitride polymers from ammonium thiocyanate for photocatalytic hydrogen evolution. *Journal of Materials Chemistry*, 21: 13032 13039.
- Wang, X., Maeda, K., Chen, X., Takanabe, K., Domen, K., Hou, Y., Fu, X. and Antonietti, M. (2009). Polymer semiconductors for artificial photosynthesis: hydrogen evolution by mesoporous graphitic carbon nitride with visible light. *Journal of the American Chemical Society*, 131(5): 1680 – 1681.

- Wang, Y., Wang, X. and Antonietti, M. (2012). Polymeric graphitic carbon nitrides as a heterogeneous organocatalyst: from photochemistry to multipurpose catalysis to sustainable chemistry. *Angewandte Chemie International Edition*, 51(1): 68 – 89.
- Thomas, A., Fischer, A., Goettmann, F., Antonietti, M., Müller, J.-O., Schlögl, R. and Carlsson, J. M. (2008). Graphitic carbon nitride materials: Variation of structure and morphology and their use as metal-free catalyst. *Journal of Materials Chemistry*, 18(41): 4893 – 4908.
- 15. Zhang, Y., Mori, T. and Ye, J. (2012). Polymeric carbon nitrides: Semiconducting properties and emerging applications in photocatalysis and photoelectrochemical energy conversion. *Science of Advanced Materials*, 4(2): 282 291.
- Lee, E. Z., Jun, Y. S., Hong, W. H., Thomas, A. and Jin, M. M. (2010). Cubic mesoporous graphitic carbon(IV) nitride: an all-in-one chemosensor for selective optical sensing of metal ions. *Angewandte Chemie International Edition*, 49 (50): 9706 9710.
- Lee, E. Z., Lee, S. U., Heo, N. S., Stucky, G. D., Jun, Y. S. and Hong, W. H. (2012). A fluorescent sensor for selective detection of cyanide using mesoporous graphitic carbon(IV) nitride. *Chemical Communications*, 48(33): 3942 3944.
- Tian, J., Liu, Q., Asiri, A. M., Al Youbi, A. O. and Sun, X. (2013). Ultrathin graphitic carbon nitride nanosheet: a highly efficient fluorosensor for rapid, ultrasensitive detection of Cu<sup>2+</sup>. *Analytical Chemistry*, 85(11): 5595 – 5599.
- Alim, N. S., Lintang, H. O. and Yuliati, L. (2015). Fabricated metal-free carbon nitride characterizations for fluorescence chemical sensor of nitrate ions. *Jurnal Teknologi*, 76(13): 1 – 6.
- Dąbrowski, A., Podkościelny, P., Hubicki, Z. and Barczak, M. (2005). Adsorption of phenolic compounds by activated carbon - A critical review. *Chemosphere*, 58(8): 1049 – 1070.
- Busca, G., Berardinelli, S., Resini, C. and Arrighi, L. (2008). Technologies for the removal of phenol from fluid streams: A short review of recent developments. *Journal of Hazardous Materials*, 160(2-3): 265 – 288.
- Ahmed, S., Rasul, M. G., Martens, W. N., Brown, R. and Hashib, M.A. (2010). Heterogeneous photocatalytic degradation of phenols in wastewater: a review on current status and developments. *Desalination*, 261(1–2): 3 18.
- 23. Soares, B. G., Amorim, G. S., Souza Jr, F. G., Oliveira M. G. and da Silva, J. E. P. (2006). The *in situ* polymerization of aniline in nitrile rubber. *Synthetic Metals*, 156 (2–4): 91 98.
- Zhao, R., Tan, C., Xie, Y., Gao, C., Liu, H. and Jiang, Y. (2011). One step synthesis of azo compounds from nitroaromatics and anilines, *Tetrahedron Letter*, 52(29): 3805 – 3809.
- Puig, A., Ormad, P., Roche, P., Sarasa, J., Gimeno, E. and Ovelleiro, J. L. (1996). Wastewater from the manufacture of rubber vulcanization accelerators: Characterization, downstream monitoring and chemical treatment. *Journal of Chromatography A*, 733 (1–2): 511 – 522.
- Haque, E., Jun, J. W., Talapaneni, S. N., Vinu, A. and Jhung, S. H. (2010). Superior adsorption capacity of mesoporous carbon nitride with basic CN framework for phenol. *Journal of Materials Chemistry*, 20(48): 10801 – 10803.
- 27. Miller, D. R., Wang, J. and Gillan, E. G. (2002). Rapid, facile synthesis of nitrogen-rich carbon nitride powders. *Journals of Materials Chemistry*, 12(8): 2463 2469.