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Rasayan J. Chem.  
ISSN (Print) : 0974-1496  
ISSN (Online) : 0976-0083  
CODEN : RJCABP

Volume- 9 | Number- 3 | 300 - 549 | July - September | 2016

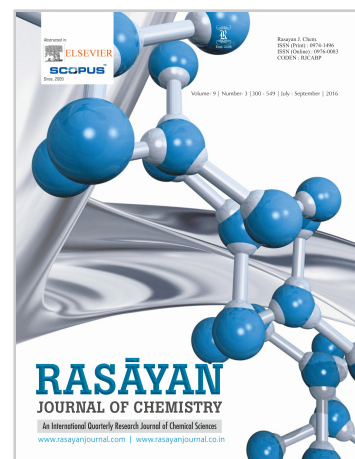
# RASĀYAN

## JOURNAL OF CHEMISTRY

An International Quarterly Research Journal of Chemical Sciences

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## Current Issue



### Volume 11, Number 1, 1-439, January - March (2018)

#### MERCURY UPTAKE AND TRANSLOCATION BY INDIGENOUS PLANTS

*S. Jameer Ahammad*<sup>1</sup>, *S. Sumithra*<sup>1,\*</sup> and *P. Senthilkumar*<sup>2</sup>

Rasayan J. Chem., 11(1), 1-12 (2018)

**Keywords:** Mercury, native plants, phytoremediation, proline and MDA.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111726>

#### MOLECULAR STRUCTURE, SPECTROSCOPIC (FT-IR, FT-RAMAN) AND HOMO-LUMO ANALYSES OF SOME ACNE VULGARIS DRUGS

*Jamelah S. Al-Otaibi*

Rasayan J. Chem., 11(1), 80-102(2018)

**Keywords:** DFT, FT-IR, FT-Raman, HOMO, LUMO, Salicylic acid, Benzoyl peroxide

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111980>

#### STUDIES ON ENHANCING THE EFFICIENCY OF ZLD PLANT FOR TANNERY EFFLUENT BY IMPLEMENTING LOW-COST AMBIENT AIR EVAPORATOR SYSTEM

*R. Rajkumar*<sup>1</sup>, *S. Sathish*<sup>2,\*</sup> and *P. Senthilkumar*<sup>2</sup>

Rasayan J. Chem., 11(1), 13-17 (2018)

**Keywords:** Tannery effluent, ZLD, Ambient air evaporator, Waste management.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111893>

#### IMPACT OF PEG6000 ON THE PHYSICAL PROPERTIES OF MICROWAVE-ASSISTED ZnO NANOSTRUCTURES USING WET CHEMICAL SYNTHESIS

*A. Kiruthiga*<sup>1</sup>, *R. Kannan*<sup>2,\*</sup> and *T. Krishnakumar*<sup>3</sup>

Rasayan J. Chem., 11(1), 18-22 (2018)

**Keywords:** Zinc oxide, SEM, PEG6000, wet chemical synthesis, and surfactants.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111935>

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## APPLICATION OF BEKONANG CLAY AND ANDISOL SOIL COMPOSITES AS COPPER (II) METAL ION ADSORBENT IN METAL CRAFTS WASTEWATER

*Pranoto Pranoto*<sup>1,\*</sup>, *C. Purnawan*<sup>1</sup> and *T. Utami*<sup>1</sup>

Rasayan J. Chem., 11(1), 23-31 (2018)

**Keywords:** Bekonang Clay, Andisol Soil, Copper, Adsorption.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111939>

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## AN OPTIMIZED GLUCOSE BIOSENSOR AS A POTENTIAL MICRO-FUEL CELL

*Laksmi Ambarsari*<sup>1</sup>, *Akhiruddin Maddu*<sup>2</sup>, *Titi Rohmayati*<sup>1</sup> and *Waras Nurcholis*<sup>1,\*</sup>

Rasayan J. Chem., 11(1), 32-36 (2018)

**Keywords:** Biosensor, Carbon paste, Electrode, Glucose oxidase, Polyaniline nanofiber.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111981>

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## ASSESSMENT OF RADON GAS CONCENTRATIONS LEVELS AND RADIATION HAZARDS IN THE DWELLINGS OF BAGHDAD PROVINCE, IRAQ

*Laith A. Najam*<sup>1,\*</sup>, *Sameera A. Ebrahiem*<sup>2</sup>, *Shaema A. Abbas*<sup>2</sup> and *Hind A. Mahdi*<sup>2</sup>

Rasayan J. Chem., 11(1), 37-40 (2018)

**Keywords:** Radon gas, Rad-7 detector, Indoor CRn, Baghdad province.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111696>

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## USING SDS FOR PLASMID CURING TO STUDY THE ANTIBIOTIC RESISTANCE OF BACTERIA ISOLATED FROM ACUTE SUPPURATIVE OTITIS MEDIA SUBJECTS IN EGYPT

*Maryam Kotb*<sup>1,\*</sup>, *Maged Bahgat*<sup>2</sup>, *Shabaan Hashem*<sup>3</sup>, *Hussein Sabit*<sup>4</sup> and *Marwan ElBagoury*<sup>5</sup>

Rasayan J. Chem., 11(1), 41-45 (2018)

**Keywords:** Antibiotic resistance- Sodium Dodecyl Sulphate- Plasmid Curing- Chromosomal resistance- Plasmidic resistance- Acute suppurative otitis media- Otitis media.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111958>

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## ASSESSMENT OF WATER QUALITY IN COAL MINES: A QUANTITATIVE APPROACH

*H. L. Yadav* and *A. Jamal*

Rasayan J. Chem., 11(1), 46-52 (2018)

**Keywords:** Water Quality Index (WQI), main sump water, seepage water, Physico-chemical parameters, coal mines.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111961>

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## HYDROTHERMAL SYNTHESIS OF ULTRA VIOLET-VISIBLE LIGHT RESPONSIVE RUTHENIUM DOPED TITANIA (RuXTi1-XO2) NANOSHEET AND ITS EFFECT ON PHOTOCATALYTIC DEGRADATION OF REACTIVE RED DYES

*Ashwini Ashok*<sup>1</sup>, *Kalaivani Raman*<sup>1,\*</sup>, *Shanmugaraj Andikkadu Masilamani*<sup>2</sup> and *Raghu Subash Chandra Bose*<sup>2</sup>

Rasayan J. Chem., 11(1), 53-62 (2018)

**Keywords:** hydrothermal process, titania nanocrystal, ruthenium dioxide, wastewater treatment, photocatalysis.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111969>

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## ANTIBACTERIAL ACTIVITY OF SILVER NANOPARTICLES COATED INTRAVASCULAR CATHETERS (AgNPs-IVC) AGAINST BIOFILM PRODUCING PATHOGENS

*R. Sengodan*<sup>1</sup>, *R. Ranjithkumar*<sup>2</sup>, *K. Selvam*<sup>3</sup> and *B. handarshekar*<sup>4</sup>

Rasayan J. Chem., 11(1), 63-68 (2018)

**Keywords:** Silver nanoparticles, AgNPs-ICV, Biofilm, ZOI, SPTT

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111934>

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## SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL SCREENING OF 2-(2-ARYLIDENEHYDRAZINYL)-4-(3-CHLOROPHENYL)-6-HYDROXYPYRIMIDINE-5-CARBONITRILE

*Pravin T. Tryambake*

Rasayan J. Chem., 11(1), 69-73 (2018)

**Keywords:** Pyrimidine, antibacterial, antifungal activity.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111615>

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## NEUTRALIZATION OF ACIDIC MINE WATER USING FLYASH AND OVERBURDEN

*Saba Shirin\* and Aarif Jamal*

Rasayan J. Chem., 11(1), 74-79 (2018)

**Keywords:** Flyash, Overburden, Acidic water, Neutralization, Environmental problem.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111957>

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## AN EXPERIMENTAL AND COMPUTATIONAL STUDY ON THERMAL CONDUCTIVITY OF MARBLE PARTICLE FILLED EPOXY COMPOSITES

*Subhrajit Ray1,\* , Arun Ku. Rout2 and Ashok Ku. Sahoo3*

Rasayan J. Chem., 11(1), 80-87 (2018)

**Keywords:** Thermal conductivity, Epoxy, marble powder, FEM

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111952>

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## INFLUENCE OF D-GLUCOSE ON CORROSION RESISTANCE OF SS316 L IN PRESENCE OF ARTIFICIAL SALIVA

*R. Saranya1,\* and Susai Rajendran2*

Rasayan J. Chem., 11(1), 103-110 (2018)

**Keywords:** Corrosion of metal, artificial saliva (AS), SS316L, D-Glucose.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111747>

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## EXPERIMENTAL STUDY ON EFFECTS OF STABILIZATION OF CLAYEY SOIL USING COPPER SLAG AND GGBS

*M. Kavisri1,\* , P. Senthilkumar2, M. S. Gurukumar3 and Karunian J. Pushparaj4*

Rasayan J. Chem., 11(1), 111-117 (2018)

**Keywords:** Copper slag, GGBS, Optimum Moisture Content, Maximum Dry Density, Unconfined Compressive Strength.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111805>

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## IN-SILICO DESIGN, SYNTHESIS AND PHARMACOLOGICAL EVALUATION OF PYRROLIDINE-2-CARBONITRILE DERIVED ANTI-DIABETIC AGENTS

*B. V. Udugade1,\* and S. P. Gawade2*

Rasayan J. Chem., 11(1), 118-126 (2018)

**Keywords:** 3D QSAR; pharmacophore; docking Design; synthesis; pyrrolidine- 2-carbonitrile, acute toxicity; chronic toxicity; anti-diabetic ligands.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111860>

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## DFT BASED ANALYSIS OF N-(3-METHYL-2, 6-DIPHENYLPYPERIDIN-4-YLIDINE)-N'-PHENYL HYDRAZINE (3-MDPYP) MOLECULE

*M. Dinesh Kumar1\*, P. Rajesh2 , M. Ezhil Inban3 and P. Kumaradhas4*

**Keywords:** 3-MDPYP, DFT, Bond topology, Deformation density, Laplacian electron density

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111820>

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## ACTIVITY ANALYSIS OF ANTHOCYANIN FROM SYZYGIUM CUMINI (L.) SKEELS AS A NATURAL INDICATOR IN ACID- BASE TITRATION

*Muhammad Zulfajri\* and Muttakin*

Rasayan J. Chem., 11(1), 135-141 (2018)

**Keywords:** Syzygium cumini, Anthocyanin, Natural Indicator, Acid-Base, Titration

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111983>

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## BIOSORPTION OF COPPER (II) ON TO THE WASTE LEAVES OF KAFAL (*Myrica esculenta*)

*Naveen Chandra Joshi<sup>1,\*</sup> and Vivekanand Bahuguna<sup>2</sup>*

Rasayan J. Chem., 11(1), 142-150 (2018)

**Keywords:** Biosorption, Kafal leaves, Batch operation, Optimized conditions, Isotherms

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1112008>

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## A NOVEL APPROACH OF MEMBRANE ELECTRODE ASSEMBLY FOR APPLICATION IN MICROBIAL FUEL CELL

*S. Mulijani<sup>1,\*</sup>, G. Syahbirin<sup>1</sup> and A.Wulanawati<sup>1</sup>*

Rasayan J. Chem., 11(1), 151-154 (2018)

**Keywords:** Polymer electrolyte fuel cells, nafion, membrane electrode assembly, sulfonated polysulfone, titanium dioxide, microbial fuel cell.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111930>

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## SOLVENT EFFECTS ON OUTER-SPHERE ELECTRON TRANSFER BETWEEN $\text{Co}(\text{L})_3^{3+}$ - $\text{Fe}(\text{II})$ IONS IN AQUEOUS- ORGANIC SOLVENT MEDIA: A SPECIATION APPROACH

*L. Devaraj Stephen*

Rasayan J. Chem., 11(1), 155-165 (2018)

**Keywords:** Solvation, Charge-transfer Reactions, Energy Dynamics, Regression effects.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111770>

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## MIXED LIGAND COMPLEXES CONTAINING SCHIFF BASES AND THEIR BIOLOGICAL ACTIVITIES: A SHORT REVIEW

*M. M. El-ajaily<sup>1</sup>, A. A. Maihub<sup>2</sup>, U. K. Mahanta<sup>3</sup>, G. Badhei<sup>4</sup>, R. K. Mohapatra<sup>5,\*</sup> and P. K. Das<sup>6</sup>*

Rasayan J. Chem., 11(1), 166-174 (2018)

**Keywords:** Schiff base, Mixed ligand complexes, Synthesis, Biological activities

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111988>

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## SYNTHESIS OF 2-AMINO-3-PHENYLPROPAN-1-OL COMPOUNDS FROM BAYLIS-HILLMAN DERIVATIVES, CARBON NANOTUBE, KINETIC, LIFETIME AND BIOLOGICAL STUDIES

*P. Senthilkumar<sup>1</sup>, V. Srinivas<sup>1</sup> and M.N. Sivakumar<sup>2,\*</sup>*

Rasayan J. Chem., 11(1), 175-180 (2018)

**Keywords:** Baylis-Hillman adducts, Carbon nanotube, Iron acidic acid, (E)-2-nitro-3-phenylprop-2-en-1-ol, 2-amino-3-phenylpropane-1-ol.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111864>

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## PRELIMINARY SAMPLE ANALYSIS IN ENNORE OIL SPILL AREA

*R. Nagalakshmi*<sup>1,\*</sup>, *P. M. Rameshwaran*<sup>2</sup> and *R. Santhosh*<sup>2</sup>

Rasayan J. Chem., 11(1), 181-186 (2018)

**Keywords:** oil spill, Ennore Port, trajectory model, sea surface temperature, metric ton, heavy metals

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111774>

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## DEVELOPMENT OF ENZYMATIC BIODIESEL FROM VEGETABLE OIL AND QUANTIFICATION OF FATTY ACID BUTYL ESTERS

*Dilip Kumar*<sup>\*</sup> and *Bhawna Verma*

Rasayan J. Chem., 11(1), 187-194 (2018)

**Keywords:** Lipase, Transesterification, Butyl biodiesel, and Soybean oil.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111813>

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## EFFECT OF TiO<sub>2</sub> NANOFILLER ON OPTICAL AND IONIC CONDUCTIVITY STUDIES OF (1-X) PVP: X (CH<sub>3</sub>COOK) POLYMER ELECTROLYTE FILMS

*R. Naveen*, *SK. Shahenoor Basha* and *M.C. Rao*

Rasayan J. Chem., 11(1), 195-202 (2018)

**Keywords:** Nanocomposite polymer electrolyte films, Solution cast technique, UV-visible, Ionic conductivity, Transport properties and Discharge studies.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1112016>

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## USE OF PLANT BIOMASS FOR REMOVAL OF MALACHITE GREEN FROM AQUEOUS SOLUTION AND OPTIMIZATION USING CENTRAL COMPOSITE DESIGN (CCD)

*P. Pallavi*, *P. King* and *Y. Prasanna Kumar*

Rasayan J. Chem., 11(1), 203-218 (2018)

**Keywords:** Plumbago Zeylancia, Malachite Green, Biosorption, Isotherms, Kinetics, Response Surface Methodology.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111971>

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## MALACHITE GREEN DYE DEGRADATION USING ZnCl<sub>2</sub> ACTIVATED RICINUS COMMUNIS STEM BY SUNLIGHT IRRADIATION

*V. NirmalaDevi*, *M. Makeswari* and *T. Santhi*

Rasayan J. Chem., 11(1), 219-227 (2018)

**Keywords:** Ricinus Communis stem, ZnCl<sub>2</sub>, degradation capacity, Malachite Green, Adsorption isotherms, Kinetics.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111986>

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## PHYTOCHEMICAL INVESTIGATION OF DIFFERENT SOLVENT EXTRACTS OF Berberis lycium FRUITS

*Ajay Singh* and *Mansi Gupta*

Rasayan J. Chem., 11(1), 228-231 (2018)

**Keywords:** Antimicrobial activity, Antifungal, Biosynthetic, Berberidaceae, Bacterial strains.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111999>

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## EVALUATION OF ANTIOXIDANT ACTIVITY AND MINERALS VALUE FROM WATERCRESS (*Nasturtium officinale* R.Br.)

*G. Haro*, *I. Iksen*<sup>1,\*</sup>, *R.M. Rumanti*, *N. Marbun*, *R. P. Sari* and *R. P. J. Gultom*

Rasayan J. Chem., 11(1), 232-237 (2018)

**Keywords:** Watercress, Antioxidant, DPPH, Mineral, Extract, pectrophotometry.

## ANTIMICROBIAL ACTIVITY OF SOME BIOCIDES AGAINST MICROORGANISMS ISOLATED FROM A SHARED STUDENT KITCHEN

*Karim Hassan and Marwan El Bagoury*

Rasayan J. Chem., 11(1), 238-244 (2018)

**Keywords:** Biocides, shared kitchens, 4-chloro-3,5-dimethylphenol, benzalkonium chloride, sodium hypochlorite

DOI: <http://dx.doi.org/10.7324/RJC.2018.1112019>

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## JOINED CLOUD POINT EXTRACTION WITH SOLVATION FOR SEPARATION, PRECONCENTRATION AND EXTRACTION LANTHANUM (III)

*S. K. Jawad \* , M. O. Kadhim and A. S. Alwan*

Rasayan J. Chem., 11(1), 245-253 (2018)

**Keywords:** Solvation, 2,4-Dimethyl pentane-3- one, Lanthanum(III), Salting out, Cloud Point Extraction.

DOI: <http://dx.doi.org/10.7324/RJC.2018.1111912>

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## Ca@Al-BENTONITE: A NEW MATERIAL FOR CHALCONE PRODUCTION

*Bayu Ardiansah, Ridla Bakri \* , Agustino Zulys and Gerry Kosamagi*

Rasayan J. Chem., 11(1), 254-259 (2018)

**Keywords:** bentonite, Ca@Al-Bentonite, chalcone, Aldol condensation, solid catalyst.

DOI: <http://dx.doi.org/10.7324/RJC.2018.1112023>

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## SYNERGISTIC ABILITY OF PSf AND PVDF TO DEVELOP HIGH-PERFORMANCE PSf/PVDF COATED MEMBRANE FOR WATER TREATMENT

*N. Kusumawati<sup>1</sup>, P. Setiarso<sup>2</sup>, S. Muslim<sup>3</sup> and N. Purwidiani<sup>4</sup>*

Rasayan J. Chem., 11(1), 260-279 (2018)

**Keywords:** membranes; coating; polysulfone; polyvinylidene fluoride; composite

DOI: <http://dx.doi.org/10.7324/RJC.2018.1112018>

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## PREPARATION AND CHARACTERISTICS OF HIGHLY MICROPOROUS ACTIVATED CARBON DERIVED FROM EMPTY FRUIT BUNCH OF PALM OIL USING KOH ACTIVATION

*Allwar Allwar*

Rasayan J. Chem., 11(1), 280-286 (2018)

**Keywords:** Empty fruit bunch, activated carbon, potassium hydroxide, micropores, surface area

DOI: <http://dx.doi.org/10.7324/RJC.2018.1112000>

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## DEVELOPMENT OF GAS-GENERATOR CHEMICAL CARTRIDGES WORKING IN THE MODE OF NON-EXPLOSIVE DESTRUCTIVE MIXTURE

*M. I. Tulepov<sup>1</sup>, Sh. E. Gabdrashova<sup>1</sup>, N. M. Rakhova<sup>1</sup>, L. R. Sassykova<sup>1\*</sup>, D. A. Baiseitov<sup>1</sup>, Zh. Elemesova<sup>1</sup>, M. A. Korchagin<sup>2</sup>, S. Sendilvelan<sup>3</sup>, I. O. Pustovalov<sup>1</sup> and Z.A. Mansurov<sup>1</sup>*

Rasayan J. Chem., 11(1), 287-293 (2018)

**Keywords:** combustion, gas-generator chemical cartridge, non-explosive destructive mixture, carbon-containing nanostructured material

DOI: <http://dx.doi.org/10.7324/RJC.2018.1112013>

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## SULFATION OF PALM SEED (*Arenga pinnata* Merr.) GALACTOMANNAN: ANTIMICROBIAL ACTIVITY AND TOXICITY TEST

Rasayan J. Chem., 11(1), 294-299 (2018)

**Keywords:** Aren Seed, Arenga pinnata, antibacterial, antifungal, sulfation, galactomannan, acut toxicity.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1112039>

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## DEVELOPMENT AND VALIDATION OF A RP-HPLC METHOD FOR THE ANALYSIS OF RIMANTADINE HYDROCHLORIDE IN MEDICINAL FORM

*J. Mamatha 1,\** and *N. Devanna 2*

Rasayan J. Chem., 11(1), 300-306 (2018)

**Keywords:** RP-HPLC, Method validation, Derivatization (DRT), ICH.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1112007>

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## A STUDY ON THE DESIGN OF THERMOSYPHON EVAPORATOR USED IN NUCLEAR WASTE VOLUME REDUCTION METHOD

*M. Arul Jayan 1,\**, *C. Marimuthu 2*, *V. Thiyagarajan 3* and *S. Senthil Velavan 4*

Rasayan J. Chem., 11(1), 307-311 (2018)

**Keywords:** Nuclear waste, Volume reduction, Evaporation, Thermosyphon evaporator

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111964>

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## PERFORMANCE OF MAGNESIUM HYDROXIDE FLUORIDES AS HETEROGENEOUS ACID CATALYST FOR BIODIESEL PRODUCTION

*S. Indrayanah 1*, *A. Rosyidah 1*, *H. Setyawati 2* and *I.K. Murwani 1\**

Rasayan J. Chem., 11(1), 312-320 (2018)

**Keywords:** MgF x (OH) 2-x, Aging temperature, Heterogeneous acid catalyst, Biodiesel, Waste cooking oil (WCO).

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111904>

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## SYNTHESIS OF 3-(3- METHOXY-2- NITRO-3- PHENYLPROPYL)-9H- CARBAZOLE FROM FRIEDEL-CRAFTS REACTION

*A.Margaret Clementpia 1*, *P. Nithya 1* and *M. N. Sivakumar 2, \**

Rasayan J. Chem., 11(1), 321-323 (2018)

**Keywords:** Friedel-Crafts reaction, nitroolefin, carbazole, Methanol, K<sub>2</sub>CO<sub>3</sub>, nitro methane, methane sulphonic acid, benzaldehyde.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111759>

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## PREPARATION OF MECHANICALLY ACTIVATED MIXTURES OF TITANIUM WITH THE CARBON NANOTUBES AND STUDY OF THEIR PROPERTIES UNDER THERMAL EXPLOSION

*Sh.E. Gabdrashova 1*, *N.M. Rakhova 1*, *I.O. Pustovalov 1*, *Zh. Elemesova 1*, *M.I. Tulepov 1*, *M.A. Korchagin 2*, *L.R. Sassykova 1*, *S. Sendilvelan 3* and *D.A. Baiseitov 1*

Rasayan J. Chem, 11 (1), 324 - 330 (2018)

**Keywords:** mechanoactivation, thermal explosion, titanium, carbon nanotubes, planetary ball mill

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1112017>

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## COMPARITIVE STUDY ON PHYSICO-CHEMICAL AND BACTERIOLOGICAL ANALYSIS OF HARVESTED RAINWATER AND NON HARVESTED GROUNDWATER

*Suparna Deshmukh*

Rasayan J. Chem, 11 (1), 331 - 338 (2018)

**Keywords:** Water crisis, Rainwater harvesting, Physic-chemical analysis, Bacteriological analysis.



## VIRTUAL SCREENING STUDIES OF TWO CLOSELY RELATED WITHANOLIDES TO CONTROL CELL PROLIFERATION AND INDUCTION OF CELL SENESCENCE

*S. Rashmi , S. Nivethitha , C. N. Hemalatha and M. Vijey Aanandhi*

Rasayan J. Chem, 11 (1), 339 - 344 (2018)

**Keywords:** Withania somnifera, Withaferin A, Withanolides, cellular senescence

DOI: <http://dx.doi.org/10.7324/RJC.2018.1111848>

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## DEVELOPMENT OF Ti4+-IMMOBILIZED NANOPOROUS MONOLITHIC POLYMER FOR SELECTIVE SEPARATION AND DETECTION OF PHOSPHOPEPTIDES

*S. F. Raeni , I. Allwicher , E. D. Iftitah , and A. Sabarudin*

Rasayan J. Chem, 11 (1), 345 - 354 (2018)

**Keywords:** Chromatography; Monolith; Phosphopeptide; Protein; Ti4+-immobilized.

DOI: <http://dx.doi.org/10.7324/RJC.2018.1111967>

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## EXPERIMENTAL ANALYSIS OF ALUMINIUM ALLOY METAL MATRIX COMPOSITE WITH TUNGSTEN CARBIDE BY IN-SITU METHOD USING SEM

*M. Rajaram Narayanan1\* and S. Nallusamy2*

Rasayan J. Chem, 11 (1), 355 - 360 (2018)

**Keywords:** FSP, Aluminium Matrix Composite, Tungsten Carbide, Mechanical Properties, SEM.

DOI: <http://dx.doi.org/10.7324/RJC.2018.1112047>

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## P-E STUDIES OF Nd MODIFIED SrBi4Ti4O15 CERAMICS

*G. Rajashekhar , Rizwana , and P. Sarah*

Rasayan J. Chem, 11 (1), 361 - 364 (2018)

**Keywords:** X-ray diffraction, solid-state reaction method, Scanning Electron Micrograph, Polarization, ferroelectrics

DOI: <http://dx.doi.org/10.7324/RJC.2018.1112057>

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## EFFECT OF THERMO GRAVIMETRIC AND FT-IR ANALYSIS ON FRICTION STIR PROCESSED MG-ZE42 ALLOY

*A. K. Darwins1,\* , M. Satheesh1 and G.Ramanan2*

Rasayan J. Chem, 11 (1), 365 - 371 (2018)

**Keywords:** Magnesium alloy, Microstructure, XRD, EDAX, TGA/DSC, FT-IR

DOI: <http://dx.doi.org/10.7324/RJC.2018.1112030>

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## EMISSION CHARACTERISTICS OF BIODIESEL DERIVED FROM USED COOKING OIL BLENDED WITH DIESEL IN THE PRESENCE OF POTASSIUM HYDROXIDE (KOH) CATALYST AS ALTERNATIVE FUEL FOR DIESEL ENGINES

*M. Saravanakumar, M. Prabhakar , Sangeetha Krishnamoorthi and S. Sendilvelan*

Rasayan J. Chem, 11 (1), 372 - 377 (2018)

**Keywords:** FSP, Aluminium Matrix Composite, Tungsten Carbide, Mechanical Properties, SEM.

DOI: <http://dx.doi.org/10.7324/RJC.2018.1112024>

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*Irvan1,2,\* , B. Trisakti1,2, S. Maulina1,2 and H. Daimon3*

Rasayan J. Chem, 11 (1), 378 - 385 (2018)

**Keywords:** Palm oil mill effluent, biogas, anaerobic, fermentation, thermophilic, pilot scale.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1112028>

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Rasayan J. Chem, 11 (1), 386 - 391 (2018)

**Keywords:** cashew apple bagasse, skin, lipids, fatty acids, valuation, chemical composition

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111858>

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Rasayan J. Chem, 11 (1), 392 - 400 (2018)

**Keywords:** Sofosbuvir, Velpatasvir, RP-HPLC, Validation, ICH Guidelines.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1111931>

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*A. Manimaran, Seenu Santhosh and P.T.Ravichandran*<sup>\*</sup>

Rasayan J. Chem., 11(1), 401- 404(2018)

**Keywords:** CBR value, GGBS, Free Swell Test.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1112044>

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Rasayan J. Chem., 11(1), 405- 412(2018)

**Keywords:** 82M; Free Energy Calculations; HIV-1 entry; gp120 binding; MD simulations

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1112050>

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Rasayan J. Chem, 11 (1), 413 - 425 (2018)

**Keywords:** Biodegradable, Antioxidant and Hydrolytic degradation.

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1112002>

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*KushalGhosh*<sup>\*</sup> and *ParthaGhosh*

Rasayan J. Chem., 11(1), 426-439 (2018)

**Keywords:**

**DOI:** <http://dx.doi.org/10.7324/RJC.2018.1112036>

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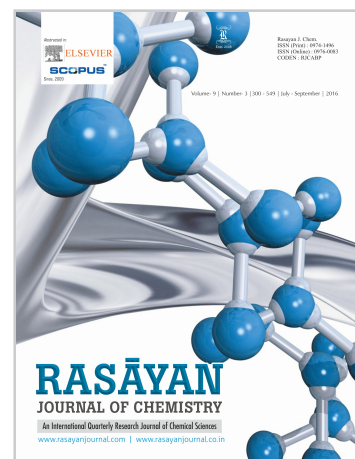
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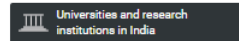
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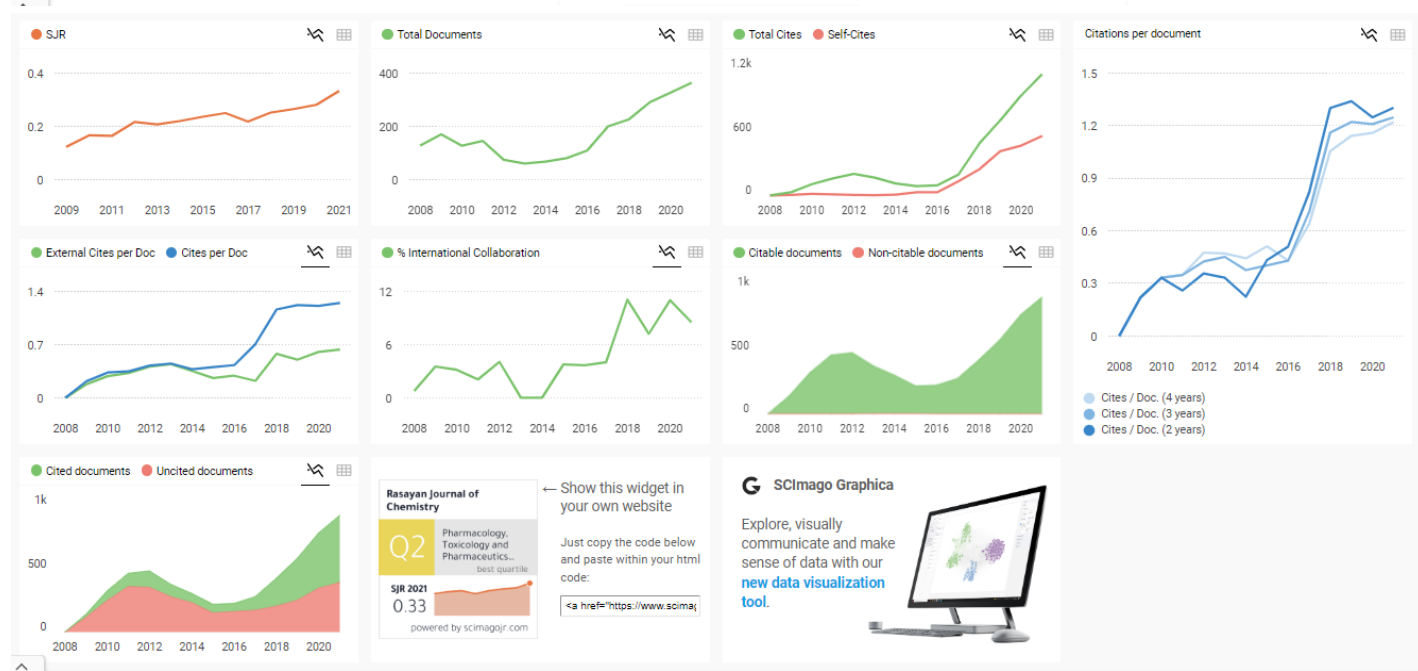
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# PERFORMANCE OF MAGNESIUM HYDROXIDE FLUORIDES AS HETEROGENEOUS ACID CATALYST FOR BIODIESEL PRODUCTION

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## ABSTRACT

The magnesium hydroxide fluorides,  $MgF_x(OH)_{2-x}$  with different aging temperatures have been synthesized by the sol-gel method. The resulting materials were characterized by various techniques such as XRD, FT-IR, TG/DTG,  $N_2$  adsorption-desorption, pyridine FT-IR and SEM, respectively. The catalytic evaluation of samples was investigated in the simultaneous transesterification and esterification between waste cooking oil (WCO) containing different %FFA and methanol to yield biodiesel. The performance of the catalyst, especially  $MgF_x(OH)_{2-x}$  aged at room temperature (RT) showed higher biodiesel yield compared to  $MgF_x(OH)_{2-x}$  with increasing of aging temperature. The higher catalytic activity could be attributed to Brønsted acid site, pore diameter size and volume of the materials. Moreover, the yield of biodiesel was not correlated with the surface area of  $MgF_x(OH)_{2-x}$ . The reusability or lifetime of catalyst decreased after three runs used.

**Keywords:**  $MgF_x(OH)_{2-x}$ , Aging temperature, Heterogeneous acid catalyst, Biodiesel, Waste cooking oil (WCO).

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## INTRODUCTION

The biodiesel has been considered as alternative energy source for fossil-based fuel. Compared with fossil-derived fuel, biodiesel produced by transesterification of triglycerides from vegetable oils or animal fats with short-chained alcohols showed many advantages such as high biodegradability, non-toxicity and renewability. However, the biodiesel obtained from refined vegetable oils or animal fats was uneconomical and impractical because of high raw materials price. In order to reduce the production cost of biodiesel, it is necessary to develop innovative technology using alternative feedstock. Based on the previous research, one of the most attractive alternative raw materials was low-quality oil including waste cooking oil (WCO) due to less expensive. Moreover, the use of WCO will not only reduce the price of biodiesel production, but it will also convert the wastes into resources<sup>1-6</sup>.

Conventionally, the biodiesel obtained from low-quality feedstocks required two processes of reaction. The esterification of high FFA using homogeneous acid catalysts such as  $H_2SO_4$ ,  $H_3PO_4$  and HCl was the first step and transesterification reaction of triglycerides in the presence of a base catalysts like NaOH or KOH was the last stage<sup>7-8</sup>. However, the homogeneous catalyst was not used due to requiring other stages and thus it increased operational cost. Moreover, the catalyst generated much wastewater during product washing, not recoverable and corrosive so that creating other problems to the environment. Therefore, the homogeneous catalyst system for the production of biodiesel became unfavorable.

The use of heterogeneous catalyst system could be used as an alternative. There were many studies to yield biodiesel using heterogeneous base catalysts such as CaO and Dolomite. The processes were cheaper, less corrosive, more environmentally friendly and easily regenerated<sup>9-10</sup>. However, they were not adaptive to free fatty acids (FFA) and water in low-quality oils. Therefore, the heterogeneous acid catalysts became more popular because they were not sensitive to FFA and water contents<sup>11</sup>. Moreover, they could be used

as a catalyst in the simultaneous transesterification and esterification for biodiesel production from low-grade feedstock in a one step. Some of the heterogeneous acid catalysts have been used to produce biodiesel such as  $V_2O_5$  phosphate<sup>12</sup>, Amberlyst-15 and Nafion<sup>13-14</sup>, Zirconium based catalysts<sup>15-16</sup>, Heteropoly acids<sup>17-18</sup> and Zeolites<sup>19</sup>. However, these catalysts showed many disadvantages like the inability to high-temperature reaction (Amberlyst-15 and Nafion), mass transfer limitation and deactivation (Zeolites), loss of activity in polar solvents due to high solubility (Heteropoly acids), high cost (Zirconium) and the easy of being deactivated ( $V_2O_5$  phosphate).

Currently, there is no study on the production of biodiesel from waste cooking oils (WCO) using magnesium hydroxide fluorides,  $MgF_x(OH)_{2-x}$  as a heterogeneous acid catalyst. Therefore, the purpose of this research is to synthesise, characterize and investigate the influence of different aging temperatures of  $MgF_x(OH)_{2-x}$  in the simultaneous transesterification and esterification between WCO containing different %FFA and methanol to yield biodiesel. The catalytic activity of the resulting materials,  $MgF_x(OH)_{2-x}$  with various aging temperatures is monitored in terms of biodiesel yield obtained from the simultaneous reaction of transesterification and esterification between WCO and methanol in a single stage. Moreover, the durability or lifetime of the catalyst is also evaluated in the optimum conditions of reaction with the same catalyst for production of biodiesel.

## EXPERIMENTAL

### Chemicals

Mg turnings (98%) was obtained from Aldrich Co. Methanol dry, 48 wt.-% HF, and methanol for HPLC were obtained from Merck Co. Ethanol and acetone were analytically pure or higher and also purchased from Merck Co. The Waste Cooking Oils (WCO) with various %FFA was obtained from Restoran Sederhana, Surabaya, Indonesia.

### Preparation of the Catalyst

The samples of  $MgF_x(OH)_{2-x}$  (containing a molar ratio of Mg alkoxide to HF = 1:2) with different aging temperatures were prepared by using Mg as a precursor and sol-gel technique. Mg turnings (98%) (31.68 mmol, 0.77 g) was reacted with 50 mL dry methanol for 3 h. Then the amount of 48 wt.-% HF was added and stirred for 3 h. The sol or gel obtained from the previous step was aged at different temperatures i.e room temperature (RT), 65, 100 and 150 °C for 12 h. Furthermore, the solvent was removed under vacuum. Finally, the resulting samples were dried under reduced pressure for 5 h at a temperature of 70 °C.

### Characterization of the Catalyst

The phase and structure of samples prepared by sol-gel method were characterized by X-ray diffraction with Cu- $K_\alpha$  radiation source at  $\lambda = 1.54056$  and range of  $2\theta$  about 20-80°. Reflections obtained from this characterization were compared with database of JCPDS-PDF. The chemical bonding and substitutes of materials were recorded with a spectrophotometer FT-IR Shimadzu in the range 4000-400  $cm^{-1}$  using KBR pellets method. The porosity characteristics of the resulting materials were measured by  $N_2$  adsorption-desorption using a micromeritics Quantachrome Instruments. The first, the powder was degassed under reduced pressure at a temperature of 70 °C and the surface area of solid was determined using BET method. The thermal analysis of the samples was investigated by TG/DTG apparatus, METTLER TOLEDO with a Pt/PtRh10 thermocouple in  $N_2$  atmosphere. The morphology or topology of the samples was studied using scanning electron microscopy (SEM) (ZEISS EVO MA 10). The surface acidity was determined by FTIR-pyridine adsorption method. The pyridine with a purity  $\geq 99.5\%$  was supplied by Aldrich Co. The first, the powder (15 mg) was pressed and activated for 3 h under vacuum at 70 °C. After heating to a temperature of 150 °C, the probe is injected during 10 minutes. Furthermore, the spectrum of pyridine adsorbed/desorbed samples were recorded with a spectrophotometer FT-IR Shimadzu in the range from 1300 to 1600  $cm^{-1}$  with an activated sample as a blank.

### Catalytic Test

The catalytic activity of the resulting samples was tested in the simultaneous reaction of transesterification and esterification between waste cooking oils (WCO) and methanol. The reaction of WCO and methanol



in a single step was carried out in a Teflon-lined 200 mL autoclave equipped. The first stage, the reactor was charged with 10 g of WCO and 48 mL methanol (corresponding to the molar ratio of WCO and methanol of 1:30) with 0.5 g catalyst (5% of WCO). Then, the mixtures were heated to 150 °C (oil batch) under constant stirring (600 rpm) for 5 hours. Later, the mixtures were allowed to cool down and the catalyst was separated by centrifugation. The residual methanol was removed with a rotary evaporator and methyl esters, known as biodiesel, was performed with GC chromatography equipped (HP6890) equipped FID detector. Instrument setting: oven (Ramps temperature) = 140 °C, injector temperature = 200 °C and detector temperature = 250 °C.

In this research, information about the stability or durability of catalyst was very important. Therefore, to investigate the reusability of the catalyst, the simultaneous transesterification and esterification between WCO and methanol were performed with the same catalyst and the reaction conditions (WCO to methanol molar ratio of 1:30 at temperature 150 °C for 5 hours under stirring speed 600 rpm with 5 wt.% catalyst). After the reaction finished, mixtures were allowed to cool down and the catalyst was separated by centrifugation. Further the catalyst was separated, washed with ethanol, acetone and distilled water to remove the impurities and used for the subsequent runs.

## RESULTS AND DISCUSSION

### X-ray diffraction (XRD)

The sol-gel technique has been used for synthesis of  $MgF_x(OH)_{2-x}$  with different aging temperatures. In this method, it happens two-step reactions (hydrolysis magnesium as precursor and fluorosis to obtain Mg-F bond<sup>20</sup>). The structure and phase of  $MgF_x(OH)_{2-x}$  with different aging temperatures were characterized by X-ray diffraction. The XRD patterns of  $MgF_x(OH)_{2-x}$  with different aging temperatures are summarized in Figure-1. The diffractogram patterns of the resulting samples as shown in Figure-1 shows main peaks at  $2\theta = 27.2^\circ$ ;  $40.4^\circ$ ; and  $53.5^\circ$ . These peaks show characteristic peaks of  $MgF_2$  ( $\diamond$ ), which are indexed to the (110), (111) and (211) planes of rutile structure of pure  $MgF_x(OH)_{2-x}$  (matched with the standard JCPDS card No. 70-2269), respectively. The X-ray diffraction technique is also used to determine changes in the structure, phase and crystallinity of the modified  $MgF_x(OH)_{2-x}$  with different aging temperatures. As shown in Figure-1, X-ray diffractogram patterns of  $MgF_x(OH)_{2-x}$  with different aging temperatures are similar. Based on the results of characterization using X-ray diffraction technique showed that different aging temperatures up to 150° C does not change the structure of  $MgF_x(OH)_{2-x}$ .

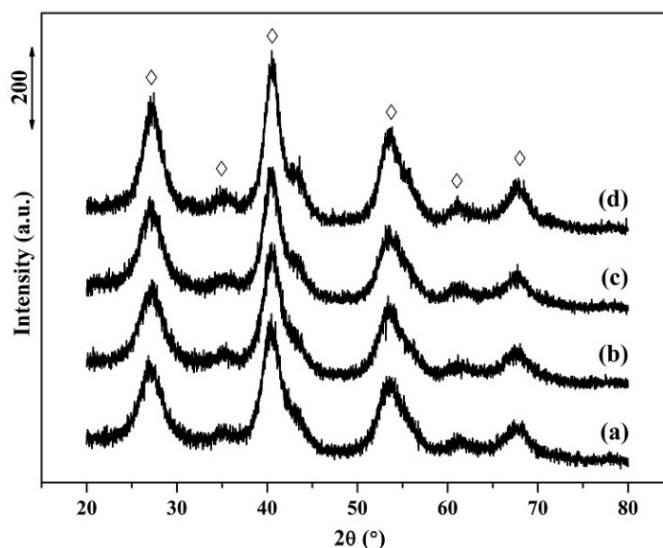


Fig.-1: XRD patterns of  $MgF_x(OH)_{2-x}$  with different aging temperatures: (a) RT, (b) 65, (c) 100 and (d) 150 °C

### Fourier-Transform Infrared Spectroscopy (FT-IR)

The materials prepared by sol-gel method with different aging temperatures were also characterized by FT-

IR spectroscopy. The resulting spectra were interpreted to determine of chemical bonding based on its vibrations. The spectra of  $\text{MgF}_x(\text{OH})_{2-x}$  with different aging temperatures are shown in Figure-2. All samples show broad O-H bands ( $\blackspadesuit$ ) located at  $3400\text{-}3700\text{ cm}^{-1}$  that are stretching vibration of water, while the bending vibration of H-O-H and bridged OH group appear about  $1640\text{ cm}^{-1}$  ( $\bullet$ ). The infrared spectra of  $\text{MgF}_x(\text{OH})_{2-x}$  with different aging temperatures as presented in the Figure-2 exhibit the same vibration at wavenumber about  $490\text{ cm}^{-1}$  ( $\blacklozenge$ ) which indicate  $\nu_{\text{Mg-F}}$  vibration. It is clear that  $\text{MgF}_x(\text{OH})_{2-x}$  which are synthesized by different aging temperatures does not destroy the structure or chemical bonding of  $\text{MgF}_x(\text{OH})_{2-x}$  sample, similar to the XRD result.

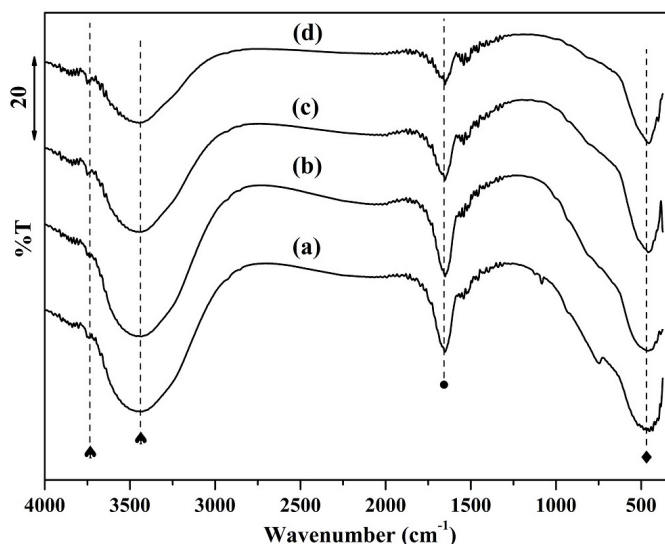


Fig.-2: FTIR spectra of  $\text{MgF}_x(\text{OH})_{2-x}$  with different aging temperatures: (a) RT, (b) 65, (c) 100 and (d) 150 °C

### Thermal Analysis

Thermal analysis of  $\text{MgF}_x(\text{OH})_{2-x}$  with different aging temperatures was carried out to investigate the thermal stability of materials, especially the influence of different aging temperatures. The TG (A)/DTG (B) curves of the samples are shown in the Fig.-3. The curves of all samples as demonstrated in Fig.-3 show very simple one-step destruction phase at a temperature about  $80\text{-}300^\circ\text{C}$  that indicate the release of MeOH from  $\text{Mg}(\text{OCH}_3)_2 \cdot 2\text{CH}_3\text{OH}$  and loss of the -OH group on the surface of solids. Further both TG (A) and DTG (B) curves as shown in Fig.-3, the decomposition and removal of HF which are appeared at a temperature about  $600\text{-}700^\circ\text{C}$  is not observed, indicating that the resulting materials are thermally stable. Based on the result, it can be concluded that  $\text{MgF}_x(\text{OH})_{2-x}$  with different aging temperatures exhibit the same thermal stability.

### $\text{N}_2$ Adsorption-Desorption Isotherms

The  $\text{N}_2$  adsorption-desorption isotherms of  $\text{MgF}_x(\text{OH})_{2-x}$  with various aging temperatures are shown in Fig.-4. All samples increase sharply at relatively high pressure, indicating the presence of mesopores. As shown in Figure-4, the adsorption of nitrogen molecules begin to occur in the relative pressure  $P/P_0$  from zero to  $<0.1$ . All samples start an interaction in the pore when the pressure reaches from the  $P/P_0 = 0$  to  $P/P_0 = 0.9$ , whereas at higher pressure,  $P/P_0 < 1$  isothermal curve shape are higher. This result indicated that the adsorption occurs at great pressure. At the moment the pressure is lowered to desorption, the isothermal curve shows a hysteresis loop. This is expected because the adsorption is not the same as the desorption. In addition, hysteric loop also occurs due to capillary condensation of nitrogen molecules. Through capillary condensation can be obtained information about the type of pores. Based on the IUPAC classification, all materials have typical IV Brunauer isotherm with H1 hysteric loop, which showed cylindrical pores. Furthermore, the pore diameter size and volume of  $\text{MgF}_x(\text{OH})_{2-x}$  with different aging

temperatures decreased from 3.63 to 2.67 nm and from 0.1 to 0.089 cc/g after increasing of aging temperatures as shown in Table-1, respectively.

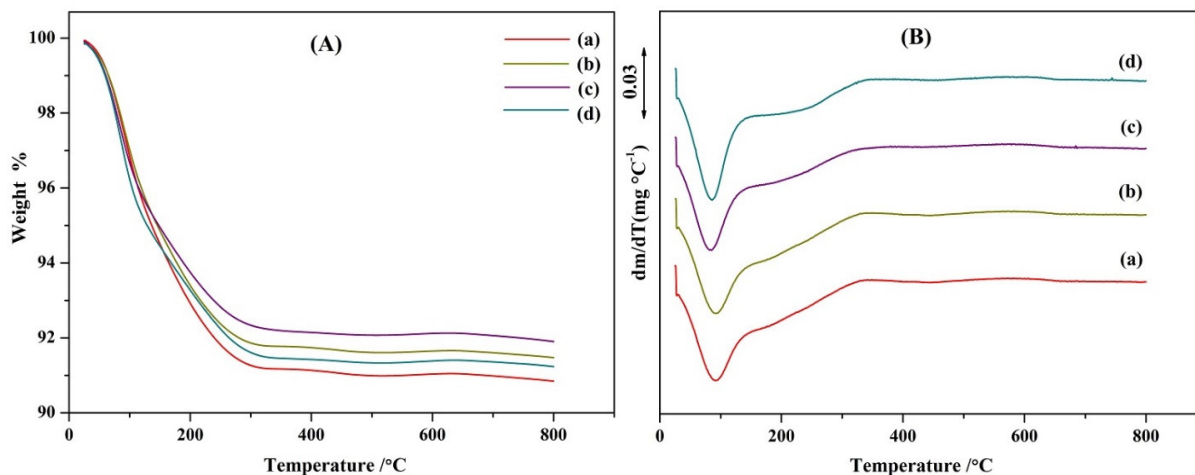


Fig.-3: TG (A) and DTG (B) curves of  $MgF_x(OH)_{2-x}$  with different aging temperatures: (a) RT, (b) 65, (c) 100 and (d) 150 °C

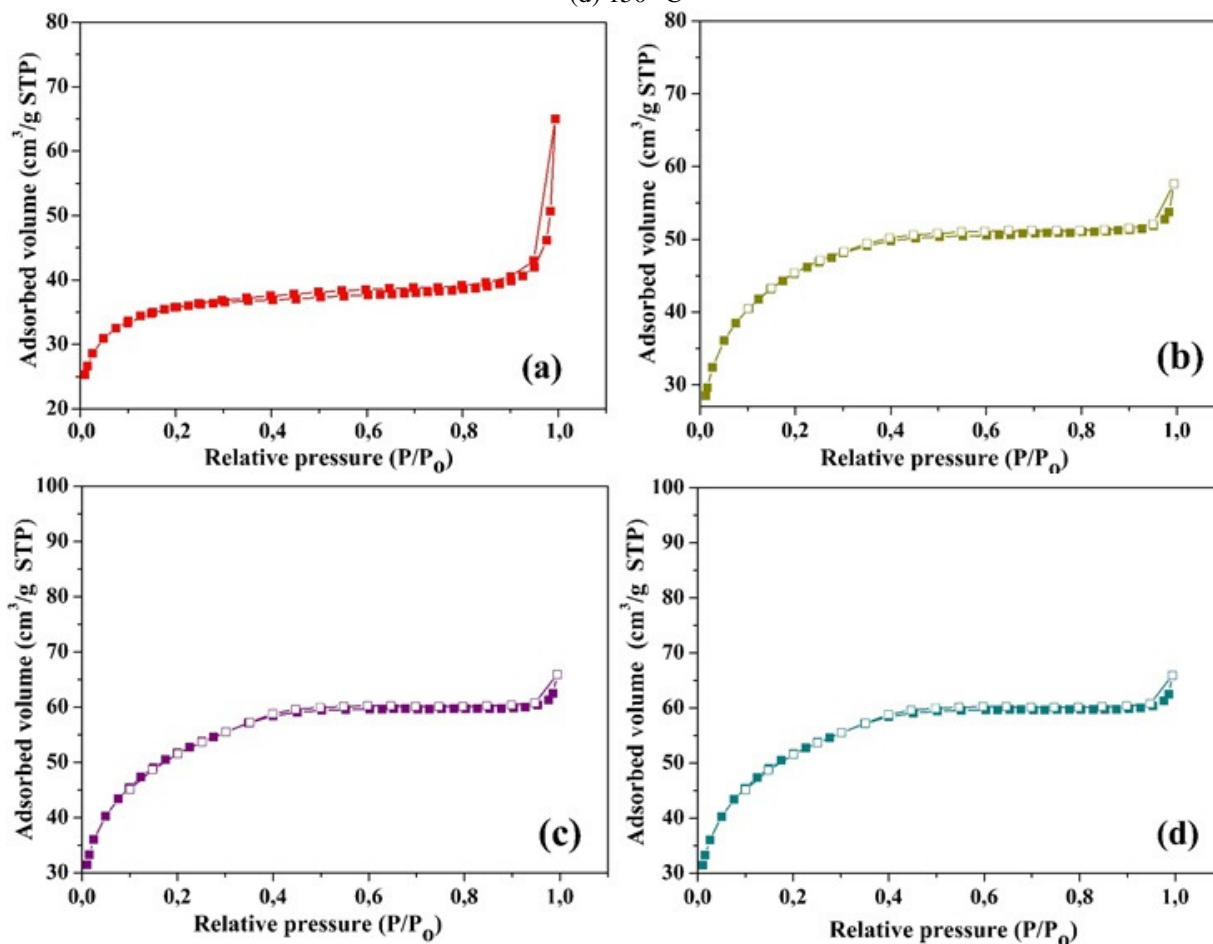


Fig.-4: N<sub>2</sub> adsorption-desorption isotherm of  $MgF_x(OH)_{2-x}$  with different aging temperatures: (a) RT, (b) 65, (c) 100 and (d) 150 °C

This result indicated that the increase in aging temperature can decrease the average pore size and pore

volume of samples. Generally, the specific surface area (BET) of the materials increased with increasing of aging temperatures. Especially for aging temperature of 150 °C, the specific surface area of  $\text{MgF}_x(\text{OH})_{2-x}$  is 171.88 ( $\text{m}^2/\text{g}$ ) as shown in Table-1. This indicated that the increase of aging temperatures in the sol-gel method increase the surface area of  $\text{MgF}_x(\text{OH})_{2-x}$ .

Table-1: Physicochemical properties of catalysts

Catalysts	$S_{\text{BET}}$ ( $\text{m}^2/\text{g}$ )	Pore diameter size (nm)	Pore volume (nm)	Acid sites	
				Lewis (mmol/g)	Brønsted (mmol/g)
$\text{MgF}_x(\text{OH})_{2-x}$ (RT)	110.97	3.63	0.100	1.458	2.319
$\text{MgF}_x(\text{OH})_{2-x}$ (65 °C)	150.55	2.82	0.107	1.549	1.723
$\text{MgF}_x(\text{OH})_{2-x}$ (100 °C)	148.70	2.40	0.102	1.515	0.717
$\text{MgF}_x(\text{OH})_{2-x}$ (150 °C)	171.88	2.67	0.089	1.704	0.523

### Acidity Analysis

The FTIR spectra with pyridine as probe and the values of Lewis/Brønsted acid sites of the resulting materials,  $\text{MgF}_x(\text{OH})_{2-x}$  are shown in Figure-5 and Table-1. All samples show absorption peaks at 1442-1450  $\text{cm}^{-1}$ , indicating the presence of Lewis acid site. In addition, there are peaks at 1540-1550  $\text{cm}^{-1}$ , which is characteristic peak of Brønsted acid site. With increase in aging temperatures, the peak of Brønsted acid site decrease. This result indicated that the existence of -OH group decrease with increasing of aging temperatures. The presence of Lewis and Brønsted acid sites in all samples is magnified with a peak at 1490-1500  $\text{cm}^{-1}$ .

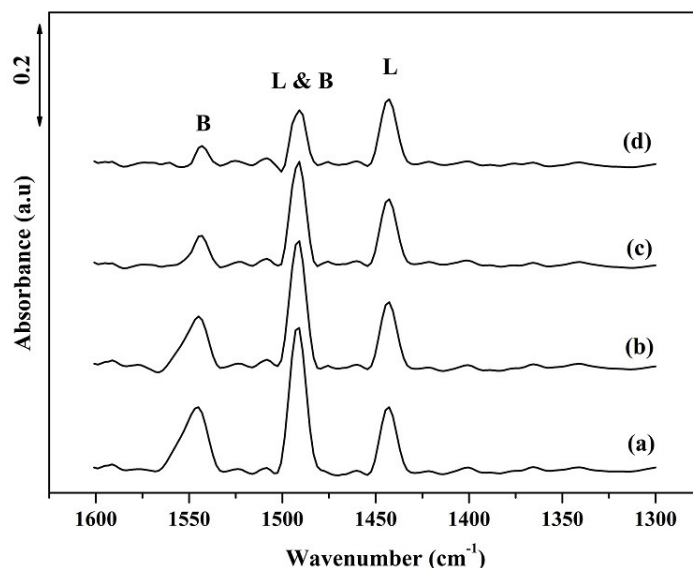


Fig.-5: FTIR spectra for pyridine adsorption on  $\text{MgF}_x(\text{OH})_{2-x}$  with different aging temperatures: (a) RT, (b) 65, (c) 100 and (d) 150 °C

### SEM Analysis

Study of morphology and topography of the samples was determined by SEM. Figure-6 shows the SEM  $\text{MgF}_x(\text{OH})_{2-x}$  with different aging temperatures. As shown in Fig.-6, it can be obtained that the morphology of the samples is dissimilar from that of the sample with an increase in aging temperatures. The particle shapes change also from spired to more spherical and homogeneous with an increase in aging temperatures (Figure-6. b-d). However, the particle size of  $\text{MgF}_x(\text{OH})_{2-x}$  samples are similar, around 0.2-3  $\mu\text{m}$ . Moreover, the agglomerate increases with increasing of aging temperatures as shown in Fig.-6 (a-d).

### Catalytic Activity

To study the catalytic activity of  $MgF_x(OH)_{2-x}$  with different aging temperatures as a heterogeneous acid catalyst, the simultaneous reaction of transesterification and esterification between WCO (containing 4, 7, 10, 13 and 17 %FFA) and methanol to yield biodiesel have been used as a probe reaction. The use of methanol in this reaction was because of more reactive and cheaper than other alcohols. The simultaneous transesterification and esterification were evaluated under the optimum conditions of reaction. Based on the previous experiment, the higher yield of biodiesel resulted from the optimum conditions (oil/methanol molar ratio of 1:30 at a temperature of reaction about 150 °C for 5 hours with catalyst/oil mass ratio of 5% wt. and speed of stirring about 600 rpm). Therefore, these parameter conditions were used, and the results are summarized in Tabel-2. The data are shown in Table-2 demonstrate that the biodiesel yield increases from 26.82% to 38.31, 50.14, 75.29% with increasing of the free fatty acids concentration of waste cooking oils, respectively. However, the yield of biodiesel decreases with increasing of FFA up to 17% (48.45% biodiesel yield). Based on the results, it is concluded that  $MgF_x(OH)_{2-x}$  catalyst which aged at room temperature is very adaptive to FFA in waste cooking oil. The presence of acid site, especially Brønsted acid site is believed to improve the biodiesel yield.

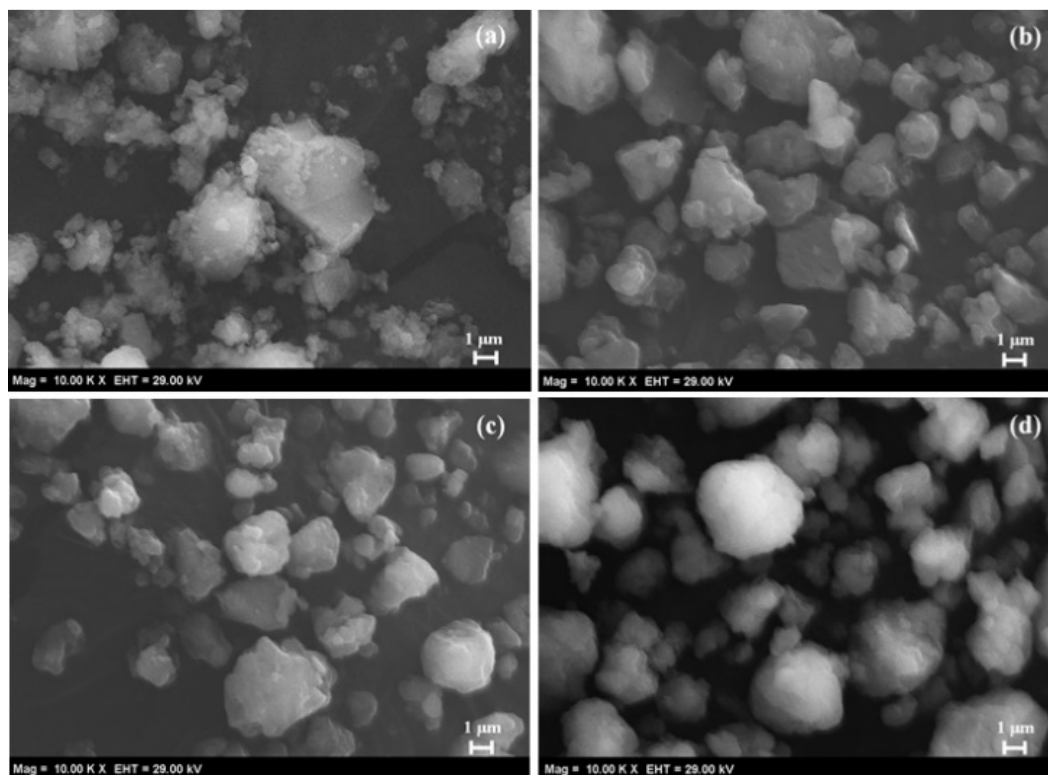


Fig.-6: SEM images of  $MgF_x(OH)_{2-x}$  with different aging temperatures: (a) RT, (b) 65, (c) 100 and (d) 150 °C

Furthermore, to investigate the effect of different aging temperatures on the  $MgF_x(OH)_{2-x}$ , the reaction of WCO containing 13% FFA is carried out with optimum reaction condition and the results are presented in Tabel 3. As shown in Tabel 3, the biodiesel yield decreases with increasing of aging temperatures from 75.29% to 62.11, 51.42, 23.89 %, respectively. The higher activity of  $MgF_x(OH)_{2-x}$  which aged at room temperature could be correlated with pore diameter size, volume and surface acidity. This result demonstrated that the larger diameter size of pore and volume can absorb more reactants and thus increase its catalytic activity. Moreover, catalyst with a higher total acidity, especially Brønsted acid site is able to capture a large number of FFA in the active site. According to this result, it is clear that the Bronsted acid site is more influence than Lewis acid site in the simultaneous reaction between WCO and methanol in a

single step. On the other hand, although the surface area of  $MgF_x(OH)_{2-x}$  with increasing of aging temperature is greater than  $MgF_x(OH)_{2-x}$  aged at room temperature, but it can not help improve the contact between WCO and methanol. This shows that the catalytic activity of these samples does not correlate to surface area. The combination of pore diameter and the high surface acidity although the lower surface area is able to provide a high biodiesel production.

Table-2: The effects of FFA (%) on the yield of biodiesel\*

Waste Cooking Oil (WCO)	FFA (%)	Biodiesel yield (%)
WCO 1	4	26.82
WCO 2	7	38.31
WCO 3	10	50.14
WCO 4	13	75.29
WCO 5	17	48.45

\*Reaction conditions: WCO/methanol molar ratio 1:30; reaction temperature 150 °C; reaction time 5 h; catalyst amount 5 wt.% of  $MgF_x(OH)_{2-x}$  (RT); and stirring speed 600 rpm.

Table-3. The effects of different aging temperatures of  $MgF_x(OH)_{2-x}$  on the yield of biodiesel\*

Catalysts	Biodiesel yield (%)
$MgF_x(OH)_{2-x}$ (RT)	75.29
$MgF_x(OH)_{2-x}$ (65 °C)	62.11
$MgF_x(OH)_{2-x}$ (100 °C)	51.42
$dMgF_x(OH)_{2-x}$ (150 °C)	23.89

\*Reaction conditions: WCO(13%FFA)/methanol molar ratio 1:30; reaction temperature 150 °C; reaction time 5 h; catalyst amount 5 wt.%; and stirring speed 600 rpm.

The loss of catalyst activity of materials was an important parameter for the heterogeneous catalytic system in simultaneous transesterification and esterification. The reusability of the catalyst is evaluated by the same reaction conditions and  $MgF_x(OH)_{2-x}$  aged at room temperature as a catalyst. After each reaction cycles, the used catalyst is recovered by centrifugation and washed with ethanol, acetone and distilled water. The results of the durability of catalysts in simultaneous reactions are displayed in Tabel-4. As summarized in Tabel-4. The yield of biodiesel decreases from 75.29% to 69.23, 51.95% after being used for three runs, respectively. These results show mass loss during the recovery process and the existence of impurities such as fatty acid and byproducts (glycerol), which can cover the pore of the catalyst.

Table-4: Reusability of  $MgF_x(OH)_{2-x}$  (RT) catalyst\*

Cycles	Biodiesel yield (%)
1 <sup>st</sup> use	75.29
2 <sup>nd</sup> use	69.23
3 <sup>rd</sup> use	51.95

\*Reaction conditions: WCO (13%FFA)/methanol molar ratio 1:30; reaction temperature 150 °C; reaction time 5 h; catalyst amount 5 wt.%; and stirring speed 600 rpm.

## CONCLUSION

The magnesium hydroxide fluorides,  $MgF_x(OH)_{2-x}$  with different aging temperatures have been successfully obtained from the sol-gel method. Based on the characterization results indicated that all materials showed

similar structure, thermal stability, and the particle size. However, the pore diameter size, volume and surface acidity, especially Brønsted acid site decreased with increasing of aging temperatures. Catalytic evaluation in the simultaneous transesterification and esterification reaction between WCO and methanol over the resulting materials as heterogeneous acid catalyst indicated that  $MgF_x(OH)_{2-x}$  aged at room temperature (RT) exhibited higher biodiesel yield compared to  $MgF_x(OH)_{2-x}$  with an increase in aging temperature. The higher activity of catalyst,  $MgF_x(OH)_{2-x}$  aged at room temperature could be attributed to surface acidity, especially Brønsted acid site, pore size and pore volume. Moreover, the durability of the  $MgF_x(OH)_{2-x}$  catalyst decreased after three cycles.

### ACKNOWLEDGEMENT

The authors thank Directorate of Higher Education (DIKTI) for granting the scholarship and Graduate Research Programme of LPPM-ITS 2017 for financial support.

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[RJC-1904/2017]