Volume 1788

Conference collection

International Conference on Engineering, Science and Nanotechnology 2016 (ICESNANO 2016) – Sebelas Maret University (UNS) Indonesia and Universiti Tun Hussein Onn (UTHM) Malaysia

Solo, Indonesia
3-5 August 2016
Editors
Budi Kristiawan et al.

AIP | Conference Proceedings

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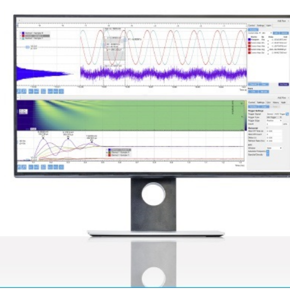
Preface: International Conference on Engineering, Science and Nanotechnology 2016 (ICESNANO 2016)

Cite as: AIP Conference Proceedings **1788**, 010001 (2017); https://doi.org/10.1063/1.4968247
Published Online: 03 January 2017



PREFACE: International Conference on Engineering, Science and Nanotechnology 2016 (ICESNANO 2016)

Dear colleagues,

On behalf of the Committees, It is our great pleasure to welcome you to Solo for International Conference on Engineering, Science and Nanotechnology 2016 (ICESNANO 2016) held at The Alana Hotel & Convention Center - Solo, INDONESIA on August 3 (Wed) ~ 5 (Fri), 2016. The joint committee between Mechanical Engineering Department, Sebelas Maret University (UNS) and Microelectronics & Nanotechnology - Shamsuddin Research Centre or MiNT-SRC, Universiti Tun Hussein Onn Malaysia (UTHM) are very proud to be performing the first ICESNANO 2016. In this year, the conference theme is "*Empowering innovation in engineering, science and nanotechnology"*. This conference aims to communicate and distribute knowledge of fundamental and applied research in the field of engineering, science and nanotechnology. It also provides the premier interdisciplinary forum for participants to present and discuss the most recent innovations and practical challenges in this field.

We are very proud and honored to have a welcoming and opening speech by Prof. Dr. Ravik Karsidi, M.S. (Rector of UNS) and Prof. Datuk Dr. Mohd Noh Dalimin (Vice-chancellor of UTHM), respectively. We would like to great thank the keynote speakers given by Prof. Abdul Latif Ahmad (Universiti Sains Malaysia), Prof. Akio Miyara (Saga University) and Assoc Prof. Takahiko Miyazaki (Kyushu University), who will present their recent work and will give new insights and ideas to the conference participants. The committees are very grateful to the invited speakers, i.e. Assoc Prof. Keishi Kariya (Saga University), Dr. Koichi Nakaso (Kyushu University), Prof. Masaya Ichimura (Nagoya Institute of Technology), Prof. Dr. Dwi Aries Himawanto (Sebelas Maret University) and Dr. Ir. Astu Unadi, M.Eng. (Director of ICAERD, Indonesian Center for Agricultural Engineering Research and Development - Ministry of Agriculture) who present their innovative works.

The organization of ICESNANO 2016 is very much a team effort. I want to especially thank all the members of the conference committee, who have carried out a huge and complicated workload. I also wish to acknowledge the members of the scientific committee, who had the arduous task of peer review process for a lot of the submitted abstracts. I also wish to thank the Ministries of Research, Technology, and Higher Education Republic of Indonesia for an international conference grant. We are also very grateful to our sponsors and exhibitors i.e. Preston Shipyard Sdn. Bhd., REI, and PT. Horiba Indonesia. Finally, let me wish you are going to enjoy this exciting conference regarding both its academic and social programs.

Kind regards,

Dr. Budi Kristiawan
Conference Chair, ICESNANO 2016Solo, INDONESIA
August, 3rd 2016

International Conference on Engineering, Science and Nanotechnology 2016 (ICESNANO 2016)
AIP Conf. Proc. 1788, 010001-1–010001-1; doi: 10.1063/1.4968247
Published by AIP Publishing. 978-0-7354-1452-5/$30.00

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AIP Conference Proceedings

Table of Contents

INTERNATIONAL CONFERENCE ON ENGINEERING, SCIENCE AND NANOTECHNOLOGY 2016 (ICESNANO 2016)

PREV NEXT

Conference date: 3–5 August 2016
Location: Solo, Indonesia
ISBN: 978-0-7354-1452-5
Editors:
Volume number: 1788
Published: Jan 3, 2017

DISPLAY : 20 50 100 all

PRELIMINARY

BROWSE VOLUMES

Open . January 2017

Preface: International Conference on Engineering, Science and Nanotechnology 2016 (ICESNANO 2016)

AIP Conference Proceedings **1788**, 010001 (2017); https://doi.org/10.1063/1.4968247

Open . January 2017

Group Photo: International Conference on Engineering, Science and Nanotechnology 2016 (ICESNANO 2016)

AIP Conference Proceedings **1788**, 010002 (2017); https://doi.org/10.1063/1.4968248

PLENARY AND INVITED PAPERS

Open . January 2017

Heat transfer characteristics of various kinds of ground heat exchangers for ground source heat pump system

A. Miyara, K. Kariya, Md. H. Ali, S. B. Selamat and Jalaluddin

AIP Conference Proceedings **1788**, 020001 (2017); https://doi.org/10.1063/1.4968249

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

Study toward high-performance thermally driven air-conditioning systems

Takahiko Miyazaki, Jin Miyawaki, Tomonori Ohba, Seong-Ho Yoon, Bidyut Baran Saha and Shigeru Koyama

AIP Conference Proceedings **1788**, 020002 (2017); https://doi.org/10.1063/1.4968250

SHOW ABSTRACT

Open . January 2017

Transport properties measurement on low GWP alternative refrigerants

Keishi Kariya, Mohammad Ariful Islam, Alam Md Jahangir, Hirotaka Ishida and Akio Miyara

AIP Conference Proceedings **1788**, 020003 (2017); https://doi.org/10.1063/1.4968251

SHOW ABSTRACT

Open . January 2017

Generation of high-temperature steam from unused thermal energy by a novel adsorption heat pump

Koichi Nakaso, Shotaro Eshima and Jun Fukai

AIP Conference Proceedings **1788**, 020004 (2017); https://doi.org/10.1063/1.4968252

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

Deposition of p-type wide-gapsemiconductor Cu_xZn_yS

Masaya Ichimura

AIP Conference Proceedings **1788**, 020005 (2017); https://doi.org/10.1063/1.4968253

SHOW ABSTRACT

CONTRIBUTED ORAL PAPERS

Open . January 2017

Performance and driveline analyses of engine capacity in range extender engine hybrid vehicle

Achmad Praptijanto, Widodo Budi Santoso, Arifin Nur, Bambang Wahono and Yanuandri Putrasari

AIP Conference Proceedings **1788**, 030001 (2017); https://doi.org/10.1063/1.4968254

SHOW ABSTRACT

Open . January 2017

Numerical investigation of laminar convective heat transfer for TiO_2/water nanofluids using two-phase mixture model (Eulerian approach)

Budi Kristiawan, Budi Santoso, Wibawa Endra Juwana, Raden Mahesa Ramadhan and Ivan Riandana

AIP Conference Proceedings **1788**, 030002 (2017); https://doi.org/10.1063/1.4968255

BROWSE VOLUMES

SHOW ABSTRACT

Open . January 2017

Improving ion-exchange membrane properties by the role of nanoparticles

Danu Ariono and Khoiruddin

AIP Conference Proceedings **1788**, 030003 (2017); https://doi.org/10.1063/1.4968256

SHOW ABSTRACT

Open . January 2017

Performance of the drag type of Horizontal Axis Water Turbine (HAWT) as effect of depth to width ratio of blade

Syamsul Hadi, Rio Jevri Apdila, Arif Hidayat Purwono, Eko Prasetya Budiana and Dominicus Danardono Dwi Prija Tjahjana

AIP Conference Proceedings **1788**, 030004 (2017); https://doi.org/10.1063/1.4968257

SHOW ABSTRACT

Open . January 2017

Concentric tube heat exchanger installed by twisted tapes using various wings with alternate axes

Indri Yaningsih and Agung Tri Wijayanta

AIP Conference Proceedings **1788**, 030005 (2017); https://doi.org/10.1063/1.4968258

BROWSE VOLUMES

SHOW ABSTRACT

Open . January 2017

A modified Newton's method used to solve a steady flow problem based on the shallow water equations

Sudi Mungkasi and Juliani Sihotang

AIP Conference Proceedings **1788**, 030006 (2017); https://doi.org/10.1063/1.4968259

SHOW ABSTRACT

Open . January 2017

Comparison between SVPWM two level in internal and external circle of hexagon

Andhika Giyantara and Mochammad Rameli

AIP Conference Proceedings **1788**, 030007 (2017); https://doi.org/10.1063/1.4968260

SHOW ABSTRACT

Open . January 2017

Experimental study on optimization of curvature blade impeller pump as turbine which functioned as power plant picohydro

Dwi Aries Himawanto, D. D. D. P. Tjahjana and Hantarum

AIP Conference Proceedings **1788**, 030008 (2017); https://doi.org/10.1063/1.4968261

BROWSE VOLUMES

Open . January 2017

Knock detection system to improve petrol engine performance, using microphone sensor

Agus Sujono, Budi Santoso and Wibawa Endra Juwana

AIP Conference Proceedings **1788**, 030009 (2017); https://doi.org/10.1063/1.4968262

SHOW ABSTRACT

Open . January 2017

Effect of sintering time on the performance of turmeric dye-sensitized solar cells

Basuki, R. Lullus Lambang G. Hidajat, Suyitno, Budi Kristiawan and Rendy Adhi Rachmanto

AIP Conference Proceedings **1788**, 030010 (2017); https://doi.org/10.1063/1.4968263

SHOW ABSTRACT

Open . January 2017

Exergy analysis of a dual-evaporator refrigeration systems

Matheus M. Dwinanto, Suhanan and Prajitno

AIP Conference Proceedings **1788**, 030011 (2017); https://doi.org/10.1063/1.4968264

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

Three-dimensional flow in the vicinity of a circular cylinder mounted to a flat plate at high Reynolds number

Galih Bangga, Andri Ashfahani, Erik Sugianto, Devy Sa'adiyah, Tiara Putri, Eva Jost and Thorsten Lutz

AIP Conference Proceedings **1788**, 030012 (2017); https://doi.org/10.1063/1.4968265

SHOW ABSTRACT

Open . January 2017

Numerical simulation of flow through pipe with magnitude of valve opening as variant at Re 2×10^5

Mahmud and Wawan Aries Widodo

AIP Conference Proceedings **1788**, 030013 (2017); https://doi.org/10.1063/1.4968266

SHOW ABSTRACT

Open . January 2017

Comparison of wake behind finned cylinders with fin pitch variations in cross-flow

Sudirman and Ruslim

AIP Conference Proceedings **1788**, 030014 (2017); https://doi.org/10.1063/1.4968267

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

Performance analysis of the electric vehicle air conditioner by replacing hydrocarbon refrigerant

Budi Santoso and D. D. D. P. Tjahjana

AIP Conference Proceedings **1788**, 030015 (2017); https://doi.org/10.1063/1.4968268

SHOW ABSTRACT

Open . January 2017

Computational study: The influence of omni-directional guide vane on the flow pattern characteristic around Savonius wind turbine

Yoga Arob Wicaksono and D. D. D. P. Tjahjana

AIP Conference Proceedings **1788**, 030016 (2017); https://doi.org/10.1063/1.4968269

SHOW ABSTRACT

Open . January 2017

Development of briquette fuel from cashew shells and rice husk mixture

Eflita Yohana, Arijanto, Ivan Edgar Kalyana and Andy Lazuardi

AIP Conference Proceedings **1788**, 030017 (2017); https://doi.org/10.1063/1.4968270

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

The study of membrane formation via phase inversion method by cloud point and light scattering experiment

Nasrul Arahman, Teuku Maimun, Mukramah and Syawaliah

AIP Conference Proceedings **1788**, 030018 (2017); https://doi.org/10.1063/1.4968271

SHOW ABSTRACT

Open . January 2017

Ternary (liquid + liquid) equilibria of (diethyl carbonate + ethanol or 1-propanol + water) systems at 303.15 K under atmospheric pressure

Rizqy Romadhona Ginting, Asalil Mustain, Ignatius Gunardi and Gede Wibawa

AIP Conference Proceedings **1788**, 030019 (2017); https://doi.org/10.1063/1.4968272

SHOW ABSTRACT

Open . January 2017

A numerical study of condensation heat transfer for R-134a in annular flow regime inside horizontal tube

Teguh Hady Ariwibowo, Fifi Hesty Solihah and Bambang Harjanto

AIP Conference Proceedings **1788**, 030020 (2017); https://doi.org/10.1063/1.4968273

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

Mass transfer in stirred tank for phenolic extraction from coal tar

Dewi Selvia Fardhyanti, Bahy Wibowo and Muhammad Rafiqi

AIP Conference Proceedings **1788**, 030021 (2017); https://doi.org/10.1063/1.4968274

SHOW ABSTRACT

Open . January 2017

High crystalline Cu_2ZnSnS_4 semiconductor prepared from low toxicity ethanol-based precursors

Badrul Munir, Bayu Eko Prastyo, Dwi Marta Nurjaya, Ersan Yudhapratama Muslih and Sahri Karim Alfauzan

AIP Conference Proceedings **1788**, 030022 (2017); https://doi.org/10.1063/1.4968275

SHOW ABSTRACT

Open . January 2017

Experimental evaluation on the use of capillary tube and thermostatic expansion valve with heat recovery hot spot water heater in air source refrigeration system

Azridjal Aziz, Rahmat Iman Mainil, Afdhal Kurniawan Mainil and Eko Saputra

AIP Conference Proceedings **1788**, 030023 (2017); https://doi.org/10.1063/1.4968276

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

Effect of water temperature and air stream velocity on performance of direct evaporative air cooler for thermal comfort

Azridjal Aziz, Rahmat Iman Mainil, Afdhal Kurniawan Mainil and Hendra Listiono

AIP Conference Proceedings **1788**, 030024 (2017); https://doi.org/10.1063/1.4968277

SHOW ABSTRACT

Open . January 2017

Effect of concave rectangular winglet vortex generator on convection coefficient of heat transfer

Syaiful, Gladys Sugiri, Maria F. Soetanto and Myung-whan Bae

AIP Conference Proceedings **1788**, 030025 (2017); https://doi.org/10.1063/1.4968278

SHOW ABSTRACT

Open . January 2017

Wind energy potential assessment to estimate performance of selected wind turbine in northern coastal region of Semarang-Indonesia

B. S. Premono, D. D. D. P. Tjahjana and S. Hadi

AIP Conference Proceedings **1788**, 030026 (2017); https://doi.org/10.1063/1.4968279

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

Numerical study of 3D brine flow across ice can to analyze heat transfer characteristics

Arrad Ghani Safitra and Prabowo

AIP Conference Proceedings **1788**, 030027 (2017); https://doi.org/10.1063/1.4968280

SHOW ABSTRACT

Open . January 2017

Thermal analysis of disc brakes using finite element method

Jaenudin, J. Jamari and M. Tauviqirrahman

AIP Conference Proceedings **1788**, 030028 (2017); https://doi.org/10.1063/1.4968281

SHOW ABSTRACT

Open . January 2017

Bifunctional catalyst of graphite-encapsulated iron compound nanoparticle for magnetic carbon nanotubes growth by chemical vapor deposition

Teguh Endah Saraswati, Oktaviana Dewi Indah Prasiwi, Abu Masykur and Miftahul Anwar

AIP Conference Proceedings **1788**, 030029 (2017); https://doi.org/10.1063/1.4968282

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

Effect of annealing temperature of titanium dioxide thin films on structural and electrical properties

A. S. Bakri, M. Z. Sahdan, F. Adriyanto, N. A. Raship, N. D. M. Said, S. A. Abdullah and M. S. Rahim

AIP Conference Proceedings **1788**, 030030 (2017); https://doi.org/10.1063/1.4968283

SHOW ABSTRACT

Open . January 2017

Effect of electrode and weld current on the physical and mechanical properties of cast iron welding

M. Chamim, Triyono and Kuncoro Diharjo

AIP Conference Proceedings **1788**, 030031 (2017); https://doi.org/10.1063/1.4968284

SHOW ABSTRACT

Open . January 2017

Performance prediction of serpentine type compact magnetorheological brake prototype

Ubaidillah, A. Wibowo, D. Adiputra, D. D. D. P. Tjahjana, M. A. A. Rahman and S. A. Mazlan

AIP Conference Proceedings **1788**, 030032 (2017); https://doi.org/10.1063/1.4968285

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

Degradation activation energy determination of PEG 4000-quartz composites using dynamic mechanical analyzer (DMA) measurements

Teuku Andi Fadly, Nur Aini Fauziyah, Allif Rosyidy, Mashuri and Suminar Pratapa

AIP Conference Proceedings **1788**, 030033 (2017); https://doi.org/10.1063/1.4968286

SHOW ABSTRACT

Open . January 2017

Fins effectiveness and efficiency with position function of rhombus sectional area in unsteady condition

Tito Dwi Nugroho and P. K. Purwadi

AIP Conference Proceedings **1788**, 030034 (2017); https://doi.org/10.1063/1.4968287

SHOW ABSTRACT

Open . January 2017

The effect of thermal cycles during aluminum casting on the intermetallic layer formation at the interface of steel crucible

Dedy Hernady, Triyono and Nurul Muhayat

AIP Conference Proceedings **1788**, 030035 (2017); https://doi.org/10.1063/1.4968288

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

Rheological properties of a reclaimed waste tire rubber through high-pressure high-temperature sintering

Ubaidillah, N. A. Yunus, S. A. A. Aziz, N. A. A. Wahab and S. A. Mazlan

AIP Conference Proceedings **1788**, 030036 (2017); https://doi.org/10.1063/1.4968289

SHOW ABSTRACT

Open . January 2017

Combination of ternary $Fe_3O_4/TiO_2/CuO$ nanocomposites and nanographene platelets: High performance photo and sonocatalysis

Ardiansyah Taufik and Rosari Saleh

AIP Conference Proceedings **1788**, 030037 (2017); https://doi.org/10.1063/1.4968290

SHOW ABSTRACT

Open . January 2017

Gear distortion analysis due to heat treatment process

Natalino F. D. S. Guterres, Rusnaldy and Achmad Widodo

AIP Conference Proceedings **1788**, 030038 (2017); https://doi.org/10.1063/1.4968291

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

Microstructure and mechanical properties of micro-SiC_p particles reinforced magnesium matrix composites with semi-solid stir casting method

E. I. Bhiftime and J. B. Belo

AIP Conference Proceedings **1788**, 030039 (2017); https://doi.org/10.1063/1.4968292

SHOW ABSTRACT

Open . January 2017

Effect of wt.% SiC_p and TiB on the mechanical properties in SiC_p/AZ81A magnesium matrix composite by the method semi solid stir casting

E. I. Bhiftime, Natalino F. D. S. Guterres and R. Atmaja

AIP Conference Proceedings **1788**, 030040 (2017); https://doi.org/10.1063/1.4968293

SHOW ABSTRACT

Open . January 2017

Study the effect of active carbon modified using HNO_3 for carbon electrodes in capacitive deionization system

Ernes Josias Blegur and Endarko

AIP Conference Proceedings **1788**, 030041 (2017); https://doi.org/10.1063/1.4968294

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

Ultrasonic irradiation-driven sonocatalytic degradation of methylene blue by ternary Fe_3O_4/ZnO/NGP nanocomposites

Faurul Fitri Harno, Ardiansyah Taufik and Rosari Saleh

AIP Conference Proceedings **1788**, 030042 (2017); https://doi.org/10.1063/1.4968295

SHOW ABSTRACT

Open . January 2017

Mechanical behaviour of biophotocomposite materials: An experimentally validated micromechanics model for tensile strength

Joko Triyono, Alva Edy Tontowi, Widowati Siswomihardjo and Rochmadi

AIP Conference Proceedings **1788**, 030043 (2017); https://doi.org/10.1063/1.4968296

SHOW ABSTRACT

Open . January 2017

Effect of anneal temperature on fluorine doped tin oxide (FTO) nanostructured fabricated using hydrothermal method

M. K. Ahmad, N. A. Marzuki, C. F. Soon, N. Nafarizal, R. Sanudin, A. B. Suriani, A. Mohamed, M. Shimomura, K. Murakami, M. H. Mamat and M. F. Malek

AIP Conference Proceedings **1788**, 030044 (2017); https://doi.org/10.1063/1.4968297

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

The influence of co-sintering Bi_2O_3 on $Yb_{0.2}Ce_{0.8}O_{2-\delta}$ ceramic SOFC

B. Budiana and S. Suasmoro

AIP Conference Proceedings **1788**, 030045 (2017); https://doi.org/10.1063/1.4968298

SHOW ABSTRACT

Open . January 2017

The influence of the number and position of the carbon fiber lamina on the natural frequency and damping ratio of the carbon-glass hybrid composite

Julian Tri Utomo, Didik Djoko Susilo and Wijang Wisnu Raharja

AIP Conference Proceedings **1788**, 030046 (2017); https://doi.org/10.1063/1.4968299

SHOW ABSTRACT

Open . January 2017

The influence of surface modification on sound absorption coefficient of albizzia wood absorber

Kuncoro Diharjo, Anditya E. Prabowo, Jamasri and Neng Sri Suharty

AIP Conference Proceedings **1788**, 030047 (2017); https://doi.org/10.1063/1.4968300

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

The effect of polymer concentration on flux stability of polysulfone membrane

D. Ariono, P. T. P. Aryanti, S. Subagjo and I. G. Wenten

AIP Conference Proceedings **1788**, 030048 (2017); https://doi.org/10.1063/1.4968301

SHOW ABSTRACT

Open . January 2017

Effect of acetylation treatment and soaking time to bending strength of sugar palm fiber composite

Kuncoro Diharjo, Andy Permana, Robbi Arsada, Gundhi Asmoro, Herru Santosa Budiono and Yohanes Firdaus

AIP Conference Proceedings **1788**, 030049 (2017); https://doi.org/10.1063/1.4968302

SHOW ABSTRACT

Open . January 2017

Enhancing on bending properties of sugar palm fiber composite using alkali treatment

Kuncoro Diharjo, Sahid Bayu Setiajit, Setyo Rojikin, Hammar Ilham Akbar, Ilham Taufik Maulana, Dimas. M. Natsir and Yohanes Waloyo

AIP Conference Proceedings **1788**, 030050 (2017); https://doi.org/10.1063/1.4968303

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

The effect of orientation difference in fused deposition modeling of ABS polymer on the processing time, dimension accuracy, and strength

Yopi Y. Tanoto, Juliana Anggono, Ian H. Siahaan and Wesley Budiman

AIP Conference Proceedings **1788**, 030051 (2017); https://doi.org/10.1063/1.4968304

SHOW ABSTRACT

Open . January 2017

Experimental study of flexural capacity on bamboo ori strip notched v reinforced concrete beams

Agus Setiya Budi, A. P. Rahmadi and Endang Rismunarsi

AIP Conference Proceedings **1788**, 030052 (2017); https://doi.org/10.1063/1.4968305

SHOW ABSTRACT

Open . January 2017

The engineering of soft ferromagnetic plane by AISI 304 hardening process

Naila Mubarok, Hamdan Akbar Notonegoro, Kemas Ahmad Zaini Thosin and Azwar Manaf

AIP Conference Proceedings **1788**, 030053 (2017); https://doi.org/10.1063/1.4968306

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

A simple temperature evaluation in high-pressure magnetron sputtering plasma using optical emission spectroscopy (OES) technique

Soo Ren How, Nafarizal Nayan and Jais Lias

AIP Conference Proceedings **1788**, 030054 (2017); https://doi.org/10.1063/1.4968307

SHOW ABSTRACT

Open . January 2017

The use of sugarcane bagasse in PP matrix composites: A comparative study of bagasse treatment using calcium hydroxide and sodium hydroxide on composite strength

Juliana Anggono, Suwandi Sugondo, Sanjaya Sewucipto, Hariyati Purwaningsih and Steven Henrico

AIP Conference Proceedings **1788**, 030055 (2017); https://doi.org/10.1063/1.4968308

SHOW ABSTRACT

Open . January 2017

Application of sandwich honeycomb carbon/glass fiber-honeycomb composite in the floor component of electric car

I. C. Sukmaji, W. R. Wijang, S. Andri, K. Bambang and T. Teguh

AIP Conference Proceedings **1788**, 030056 (2017); https://doi.org/10.1063/1.4968309

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

The effect of heating temperature in static thermal tensioning (STT) welding on mechanical properties and fatigue crack propagation rate of FCAW in steel A 36

N. Subeki, Jamasri, M. N. Ilman and P. T. Iswanto

AIP Conference Proceedings **1788**, 030057 (2017); https://doi.org/10.1063/1.4968310

SHOW ABSTRACT

Open . January 2017

The effect of core thickness variation of sandwich composite cantala rHDPE on mechanical strength of bending test

Andri Setiadi, Wijang Wisnu Raharjo and Teguh Triyono

AIP Conference Proceedings **1788**, 030058 (2017); https://doi.org/10.1063/1.4968311

SHOW ABSTRACT

Open . January 2017

Effect of codoping cobalt and aluminum on enhancing the piezoelectricity properties of fiber-based zinc oxide

Dedi Subagiyo, M. Thoyib, Suyitno, Syamsul Hadi, Anif Jamaluddin and R. L. L. G. Hidayat

AIP Conference Proceedings **1788**, 030059 (2017); https://doi.org/10.1063/1.4968312

SHOW ABSTRACT

BROWSE VOLUMES

Open . January 2017

Effects of alkali and steaming on mechanical properties of snake fruit (*Salacca*) fiber

Seno Darmanto, Heru S. B. Rochardjo, Jamasri and Ragil Widyorini

AIP Conference Proceedings **1788**, 030060 (2017); https://doi.org/10.1063/1.4968313

SHOW ABSTRACT

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Adhesion strength study of sintered silver for power electronic devices application

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Ratna Dewi Kusumaningtyas, Naomi Ratrianti, Indah Purnamasari, Arief Budiman, et al.

Kinetics Study of Jatropha Oil Esterification with Ethanol in the Presence of Tin (II) Chloride Catalyst for Biodiesel Production

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Abstract. Jatropha oil is one of the promising feedstocks for biodiesel production. Jatropha oil is non-edible oil hence utilization of this oil would not compete with the needs of food. However, crude jatropha oil usually has high free fatty acid (FFA) content. Due to this fact, direct alkaline-catalyzed transesterification of crude jatropha oil for biodiesel production cannot be performed. FFA in crude jatropha oil will react with a base catalyst, resulting in soap as by product and hindering methyl ester (biodiesel) production. Therefore, prior to a transesterification reaction, it is crucial to run a pretreatment step of jatropha oil which can lower the FFA content in the oil. In this work, the pretreatment process was conducted through the esterification reaction of FFA contained in crude jatropha oil with ethanol over tin (II) chloride catalyst to reduce the acid value of the feedstock. The feedstock was Indonesia crude jatropha oil containing 12.03% of FFA. The esterification reaction was carried out in a batch reactor with a molar ratio of FFA to ethanol was 1:60 and total reaction time was 180 minutes. Tin (II) chloride catalyst was varied at 2.5, 5, 7.5, and 10% wt, whereas the effect of the reaction temperature was studied at 35, 34, 55, and 65 °C. The best reaction conversion was 71.55%, achieved at the following condition: a reaction temperature of 65 °C, catalyst concentration of 10% wt, the reaction time of 180 min, and the molar ratio of FFA to ethanol was 1:60. Kinetics study was also conducted in this work. It was found that esterification reaction of jatropha oil FFA with ethanol catalyzed by tin(II) chloride fitted the first-order pseudo-homogeneous kinetics model. It was also revealed that the frequency factor (A) and the activation energy (Ea) were 4.3864×10^6 min^{-1} and 56.2513 kJ/mole, respectively.

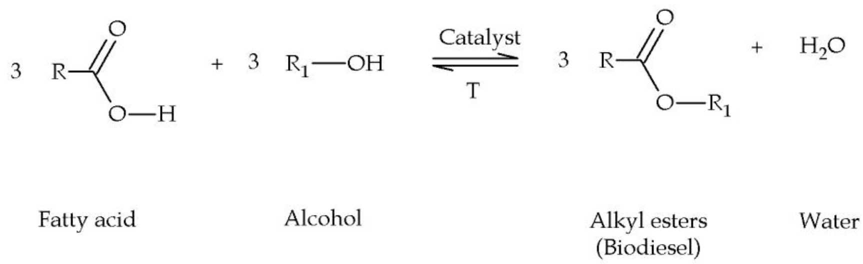
INTRODUCTION

The rapid growth in industrialization and transportation has increased the energy demand all over the world [1]. In accordance with this global trend, fossil fuel is the most used energy source to date. However, recently, there are two important issues related to the fossil fuel utilization. First, fossil fuel depletion has been known as a future challenge of the energy security. Secondly, related to the environmental aspect, fossil fuel burning releases CO_2 emission which is among the primary greenhouse gasses. Greenhouse gasses are the principal contributor to the global warming and climate change [2, 3]. Thus, an efficient strategy is needed to reduce the consumption of fossil fuel as the main CO2 emission source. To overcome this problem, it is essential to develop alternative energy which is renewable and has eco-friendly characteristic. Among the most prospective alternative energy source is biodiesel. Biodiesel is clean energy source which holds many advantages. It has comparable properties to fossil-based diesel fuel, can be produced in large quantity, and it includes to the low carbon energy source. Due to its benefits, biodiesel

International Conference on Engineering, Science and Nanotechnology 2016 (ICESNANO 2016)
AIP Conf. Proc. 1788, 030086-1–030086-7; doi: 10.1063/1.4968339
Published by AIP Publishing. 978-0-7354-1452-5/$30.00

is the top priority of the national biofuel development policy in Indonesia as stated in the Presidential Regulation No. 5/ 2016.

Biodiesel can be produced from vegetable oils, both edible and non-edible oils, as raw material. However, due to the competition of edible oil for food and biofuel production, utilization of non-edible vegetable oil as biodiesel feedstocks is more favorable [4]. In this work, biodiesel production using jatropha oil was carried out. Jatropha oil is non-edible oil which is available in Indonesia. Biodiesel is generally produced via alkaline-catalyzed transesterification reaction of vegetable oil. However, jatropha oil contains a high amount of free fatty acid (FFA) which is not suitable for a direct transesterification. The high content of FFA will react with the base catalyst, resulting in soap byproduct and hindering methyl ester (biodiesel production). Therefore, pretreatment step to reduce the FFA content in the feedstock is necessary. The pretreatment process can be performed through the esterification reaction of FFA using alcohol in the presence of acid catalyst [5]. In this work, esterification reaction of jatropha oil was conducted using ethanol over tin (II) chloride catalyst. Tin (II) chloride is solid Lewis acid catalyst which has many benefits. It is cheap, active, less corrosion, easy to separate with the reaction product of the reaction, but it acts alike a homogeneous catalyst during the reaction [6]. The esterification reaction of jatropha oil with alcohol is depicted in Equation (1). In this research, the effects of main parameter of the reaction were evaluated. Kinetics study was also carried out to obtain the kinetics constants of the reaction.



(1)

EXPERIMENTAL METHOD

The feedstock used in this work was jatropha oil with FFA content of 30.57, which was obtained from PT Pura Energi Kudus, Indonesia and absolute ethanol (Merck). The catalyst was tin (II) chloride ($SnCl_2.2H_2O$) was purchased from Merck. The esterification reaction was conducted in a 250 mL batch reactor equipped with a condenser and magnetic stirrer. Initially, 65.5 g jatropha oil was introduced to the reactor and heated up to 35°C. In the same time, 100 mL ethanol was heated separately until it reached the identical temperature, and subsequently, it was discharged to the reactor to react with the jatropha oil. Tin (II) chloride as much as 3.3 g (5% wt) was then added to the reactor. Samples of the reaction were withdrawn at following reaction time: 0, 15, 30, 45, 60, 90, 120, and 180 minutes. The experiments were conducted at various temperature and catalyst concentration. Estimation of the reaction conversion was conducted based on the FFA content in the reaction mixture by using standard NaoH titration [7]. The data obtained was then utilized for kinetics study and kinetics parameter evaluation. Determination of the FFA content was carried out using the following formula:

$$\% \text{ acid} = \frac{(Mr)(N\ KOH)(V\ KOH)}{(W)(1000)} \times 100\% \qquad (2)$$

$$\% \text{ FFA} = \% \text{ acid of the sample} - \% \text{ acid of tin (II) chloride} \qquad (3)$$

Where,
Where,	% acid of the sample	= acid content of (FFA+ tin (II) chloride) in the sample, %wt
	% acid of tin (II) chloride	= acid content of tin (II) chloride in the sample, %wt
	% FFA	= free fatty acid content resulted in the esterification reaction, %wt
	Mr	= molecular weight of oleic acid, g/mol
	N KOH	= normality of KOH solution used as titrant, N
	V KOH	= volume of KOH solution used for sample titration, mL
	W	= weight of oil sample, g

FFA conversion was determined using the following equation:

$$X_A = \left|\frac{\%\ FFA\ initial - \%\ FFA\ sample}{\%\ FFA\ initial}\right| x\ 100\% \tag{4}$$

Where, X_A is reaction conversion, %.

RESULTS AND DISCUSSION

In this work, esterification reaction of jatropha oil with ethanol was carried out in a batch reactor in the presence of tin (II) chloride catalyst. The molar ratio of FFA to ethanol was 1:60. Variables studied in this work were reaction temperature and catalyst concentration.

Effect of Reaction Temperature

The reaction temperature was varied at 34, 45, 55, and 65 °C. On the other hand, molar ratio of FFA: ethanol and catalyst concentration were maintained at 1:60 and 5%, respectively. Effect of the reaction temperature on the reaction conversion is shown in Fig. 1.

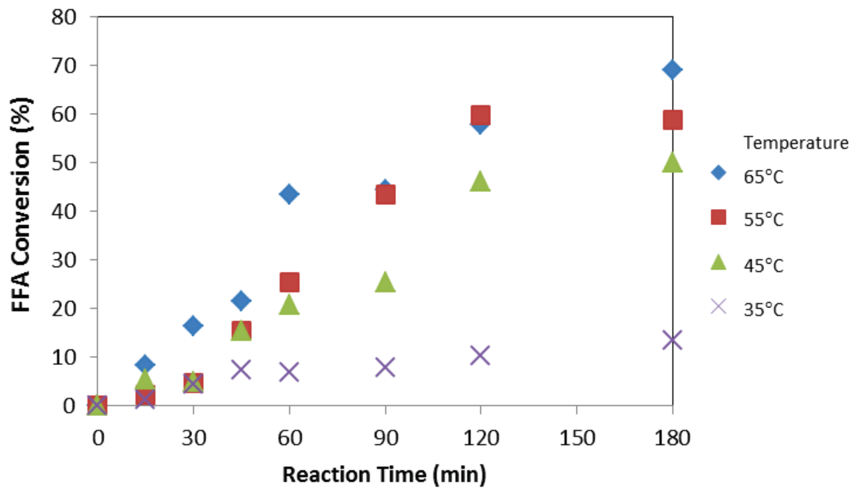


FIGURE 1. Effects of Temperature on the FFA Conversion at Different Reaction Temperatures

Figure 2 revealed that the FFA conversion increased with the increasing of the temperature. It was due to the fact that esterification reaction is an exothermic reaction. Based on the Arrhenius equation, the higher reaction temperature will enhance the value of the reaction rate coefficient, causing the higher reaction rate and conversion. Among the experiments conducted at various temperature and reaction time, the best conversion was 68.96%, which was obtained at the temperature of 65°C with the reaction time of 180 minutes. The optimal reaction temperature obtained was in accordance with the result reported in the literature for FFA esterification using acid catalyst [8]. Therefore, this temperature was employed for the subsequent experiments which studied the influence of catalyst concentration.

Kinetic Study

The data of reaction conversion at different reaction time and temperature were utilized for the reaction kinetics determination. To develop the kinetics model of the reaction, Equation (1) was rewritten to Equation (5).

$$\underset{(FFA)}{A} + \underset{(Ethanol)}{E} \rightleftharpoons \underset{(Methyl\ Ester)}{D} + \underset{(Water)}{W} \tag{5}$$

At stoichiometric condition, esterification reaction is usually considered as a second-order reversible reaction, which can be formulated as demonstrated in Equation (6).

$$-r_A = k_1 C_A C_E - k_2 C_w C_D \tag{6}$$

However, in this work, a far excess of alcohol was applied, indicated by the molar ratio of FFA to alcohol of 1:60. Hence, it could be assumed that the reaction equilibrium shifted to the completion of product formation and the reverse reaction could be neglected. Furthermore, since ethanol in the reaction system was great excess with respect to FFA, its concentration could be considered to remain constant throughout the reaction time. Therefore, the ethanol concentration could be included in the rate constant, resulting in a pseudo-first-order reaction rate model [6]. The reaction rate thus can be abridged to one exhibited in Equation (7).

$$-r_A = k\ C_A \tag{7}$$

To obtain the parameters of the reaction rate, mass balance of A (FFA) in batch reactor was developed:

Rate of mass A in – Rate of mass A out – Rate of A consumed in the reaction = Rate of accumulation of A (8)

$$N_{A0} - N_A - (-r_A)\,V = -\frac{d\,C_A.V}{dt} \tag{9}$$

$$0 - 0 - (-r_A)\,V = -\frac{d\,C_A.V}{dt} \tag{10}$$

$$(-r_A) = -\frac{d\,C_A}{dt} \tag{11}$$

Substitution of Equation (7) to Equation (11) resulted in Equation (12):

$$k\ \ C_A = -\frac{d\,C_A}{dt} \tag{12}$$

Based on the stoichiometric equation, C_A can be expressed is the function of reaction conversion:

$$C_A = C_{A0}\,(1-\frac{X_A}{100}) \tag{13}$$

Substitution of Equation (13) to Equation (12) resulted in the following equation:

$$k.C_{A0}\,(1-\frac{X_A}{100}) = -\frac{d\,C_{A0}(1-\frac{X_A}{100})}{dt} \tag{14}$$

$$k.C_{A0}\,(1-\frac{X_A}{100}) = -\,C_{A0}\,\frac{d(1-\frac{X_A}{100})}{dt} \tag{15}$$

$$k.dt \quad = -\frac{1}{(1-\frac{X_A}{100})}\,d\,(1-\frac{X_A}{100}) \tag{16}$$

$$k\int_0^t dt \quad = -\int_0^{X_A} \frac{1}{(1-\frac{X_A}{100})}\,d\,(1-\frac{X_A}{100}) \tag{17}$$

$$k\,.\,t \ = -\ln\,(1-\frac{X_A}{100}) \tag{18}$$

The value of reaction rate constant can be obtained using linear regression method. The values of Sum of Square Error (SSE) were also measured. The calculation was solved numerically using Matlab program. The values of reaction rate constant (k) obtained are demonstrated in Table 1. It was revealed that the SSE value for each temperature was small, indicating that the calculation was accurate.

TABLE 1. Reaction Rate Coefficient at Various Reaction Temperatures

Temperature (K)	$k.10^3$(1/min)	$SSE.10^2$
308	0.9066	0.19528
318	3.9716	1.2979
328	5.4821	3.6370
338	6.9330	1.3651

Furthermore, the fitting of the reaction kinetics model to the experimental data is exhibited in Fig. 2. It is shown that the calculated values estimated based on the kinetic model were closed to the experimental data. It means that the kinetic model proposed (pseudo-first order model) was appropriate for this esterification reaction system.

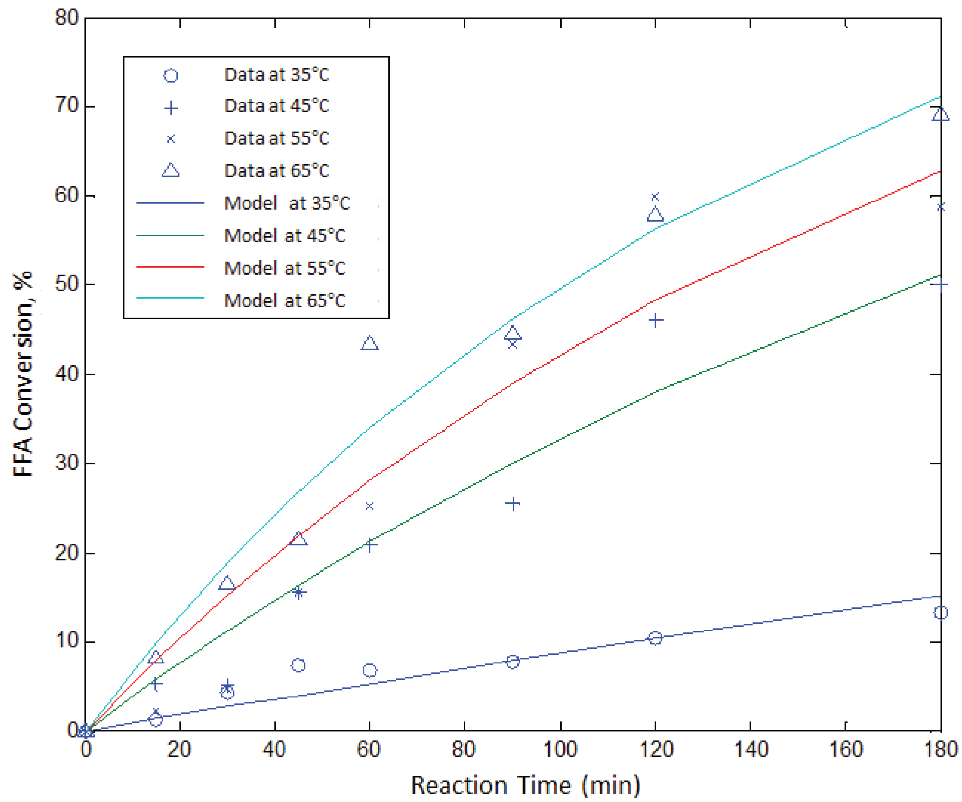


FIGURE 2. Comparison of the Calculated Value Resulted from the Model and the Experimental Data

The reaction rate constant obtained was then utilized to find the activation energy (Ea) and frequency factor (A) values in the Arrhenius equation using linear regression method. Arrhenius stated that correlation between reaction rate and temperature is written as:

$$k = A \exp(-\frac{E}{RT}) \tag{19}$$

By composing correlation of ln k and (1/T) using linear graph fitting as shown in Fig. 3, it was found that the value of A was 4386414.8912 min^{-1} and the activation energy (E) was 56.2513 kJ/mol. This value of activation energy was comparable to those reported in the literature for acid catalyst esterification, which starting 46.69 kJ/mol [6], 50.74 kJ/mol and 42.76 kJ/mol [9]. The result of this research was considered accurate since the coefficient of determinant (R^2) was 0.8427, which was closed to 1. The relative error in this modeling was 5.088%, and correlation of reaction rate coefficient and the reaction temperature was formulated in the Equation (20).

$$k = 4386414{,}8912 \exp(-\frac{56{,}2513}{RT}) \tag{20}$$

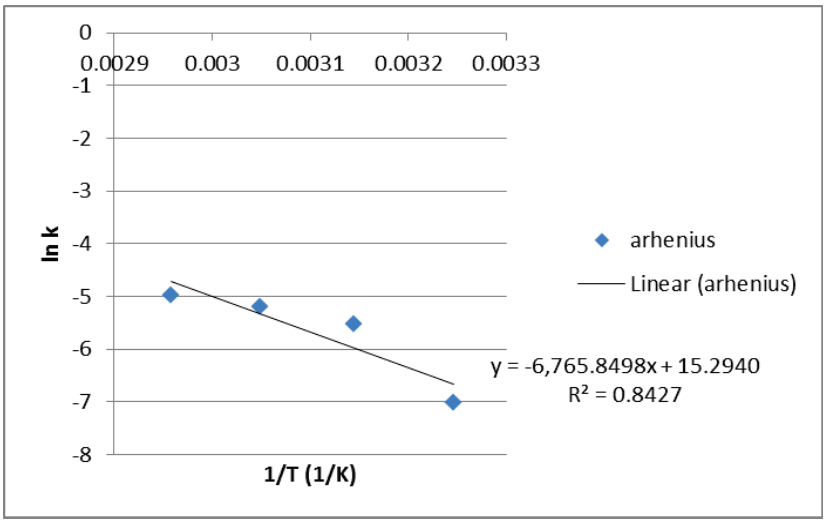


FIGURE 3. Correlation of Reaction Rate Coefficient and Reaction Temperature

Effect of Catalyst Concentration

To evaluate the effect of a catalyst on the reaction conversion, the concentration of the catalyst employed was varied at 2.5, 5, 7.5, and 10% wt. Reactions were conducted at the fixed molar ratio of FFA to ethanol and reaction temperature of 1:60 and 65 °C, respectively. The result is presented in Fig. 4. This Figure has shown that the increasing of the catalyst concentration brought the higher FFA conversion. It is because catalyst can provide an alternative route of the reaction which requires a lower activation energy to result in the product. It consequently brought about the higher reaction rate and higher conversion. Figure 4 demonstrated that the reaction conversion enhanced significantly when the catalyst concentration was increased from 2.5 to 5% wt. However, the addition of the catalyst concentration to the higher amount than 5% resulted in a relatively constant conversion. It is due to the fact that at 5% wt of catalyst, the amount of catalyst has almost reached the maximum amount of catalyst which is required to activate the carbonyl group of the FFA. The best reaction conversion was 71.55% provided by the catalyst concentration of 10% wt. This result is comparable to the reaction performed using a homogeneous sulfuric acid catalyst which yielded reaction conversion of around 77% [8]. On the other hand, in this work, 5% wt of catalyst provided 68.96%. Therefore, the employment of 5% wt of the catalyst is considered more efficient.

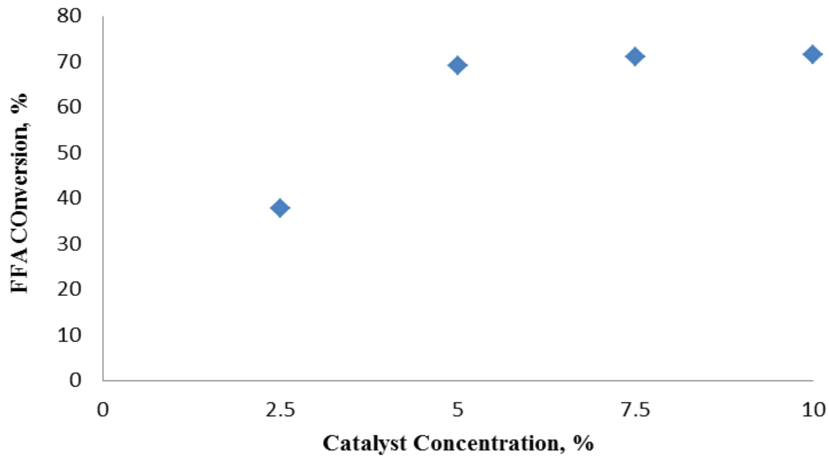


FIGURE 4. FFA Conversion at Different Catalyst Concentration

CONCLUSIONS

Based on the result of the research, it can be concluded that the higher reaction temperature, the higher value of reaction rate coefficient was obtained, resulting in the higher conversion of FFA. On the other hand, the increasing of catalyst concentration employed in the reaction led to the higher reaction conversion achieved. The study on the reaction kinetics showed that the reaction fit the first order pseudo-homogeneous model. Parameter values obtained in this works were: frequent factor (A) was 4.3864 x 10^6 min^{-1}, activation energy (E) was 56.2513 kJ/mol. The highest reaction conversion achieved in the experiments of jatropha oil esterification with ethanol over tin (II) chloride catalyst was 71.55% at the reaction temperature of 65°C using 10% concentration of catalyst at the molar ratio of FFA to ethanol of 1:60.

ACKNOWLEDGMENT

We would like to thank to Directorate Research and Community Service-Indonesian Directorate General of Higher Education, Ministry of Research, Technology, and Higher Education for supporting the research funding through Hibah Bersaing Grant scheme.

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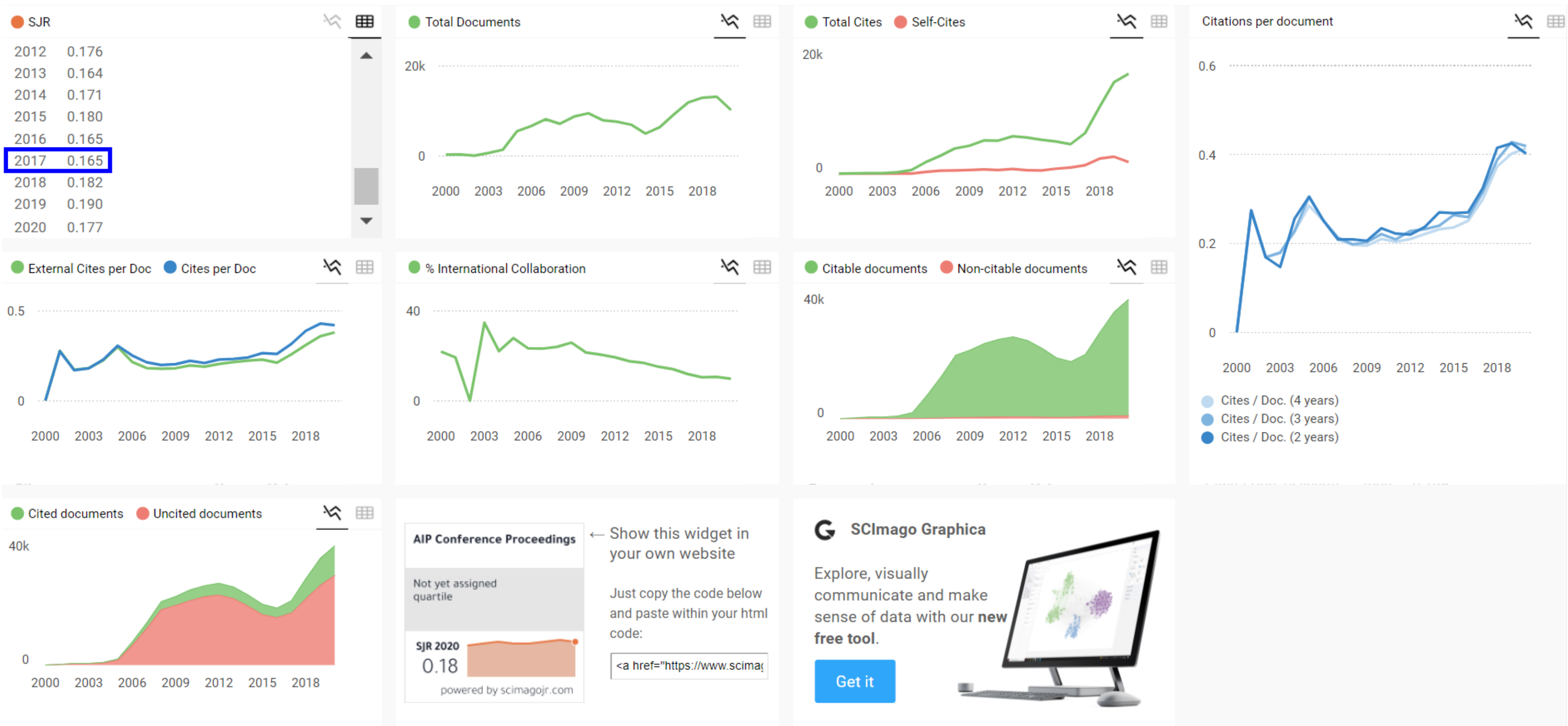

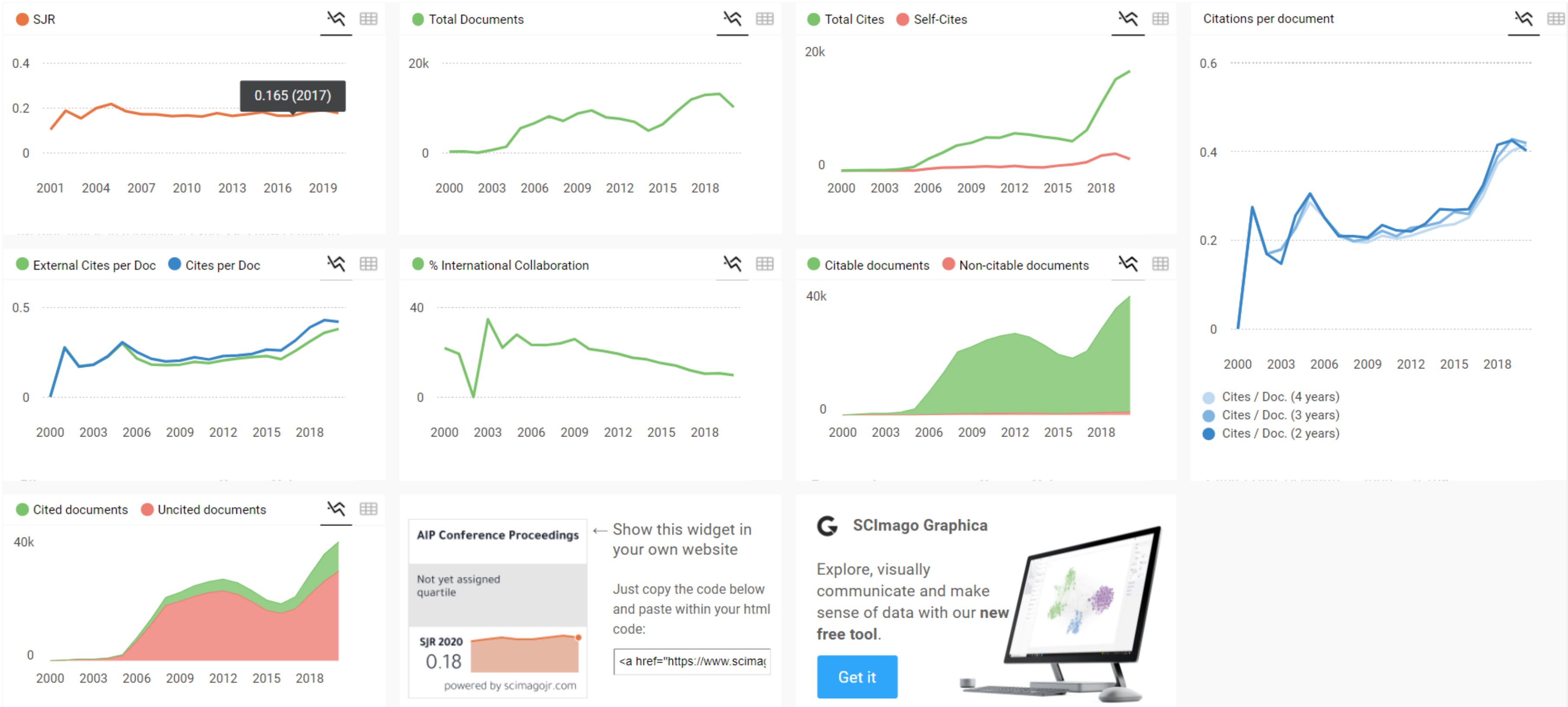
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