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High-Grade Biosilica Isolated from Diluted Acids Leached Rice Husk

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Abstract

Rice husk which consists of ~20% silica seems to be quite promising to be the biosilica resources. As one of the most abundant agricultural wastes derived from paddy milling sites, rice husks have been generally directly burn on sites causing air pollution and environmental problems. Rice husk silica is amorphous in nature which is quite reactive and has a great potential to be used as a raw material for various silica based products. The aim of this experiment was to study the efficacy of diluted acids such as HCl and citric acid in order to obtain high-grade silica. The characterisation of silica ashes included the analysis of composition and structure using Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), X-ray Fluorescence (XRF). The specific surface area of silica was determined using Brunauer, Emmet, Teller-Surface Area Analyzer (BET-SAA). Silica produced from these experiments had an amorphous form. The highest silica purity with Si content of 96.2% was obtained from rice husk treated using diluted HCl. However, the highest specific surface area of ~253 m^2/g was derived from diluted citric acid treated rice husks. Diluted acids seemed to be quite effective in removing metallic impurities enabling to produce high grade biosilica.

Keywords: Amorphous silica, rice husk, dilute acids, leaching.

1. Introduction

The rice paddy production in the world reached 748 million tons in 2016 of which ~ 90% was produced in Asia (FAO, 2016). In Indonesia, about 75 million tons was produced in 2015 (BPS, 2016). Around 20% of rice paddy is rice husk (Blissett, 2017), thus around 15 million tons of rice husks are produced annually in Indonesia. Rice husk is produced during the paddy milling and regarded as the second largest wastes after rice straws. Rice husk is generally accumulated and burnt on sites thus increasing air pollution. Rice husks have been considered to be the amorphous silica resources which have been widely used in many industrial applications such as cements (Sinyoung, 2017; Aprianti, 2017), fillers (Hsieh, 2017; Noushad, 2016), polymer composites (Yaakob, 2017), adsorbent (Deokar, 2016), catalyst support (Kumar, 2016), and drug delivery (Rajanna, 2017). Besides that, there have been several attempts to harness rice husk as biomass derived fuels (Blissett, 2017) and as precursors for silica-based materials synthesis such as zeolite (Mohamed, 2015), Si (Malino, 2016), SiC (Seraji, 2016), and Si_3N_4 (Qadri, 2016) The high content of silica in rice husk of ~20% makes it promising to be the low cost and renewable resources of biosilica with significantly increased added value product.

Rice husk silica could be isolated in several ways, such as alkaline extraction using NaOH (Kalapathy, 2000; Liou, 2011) or NH_4OH (Della, 2005) followed by precipitation process using acids. Silica could be simply obtained from direct combustion of rice husk resulting in silica rich ashes. However, direct combustion of rice husk without any pretreatment has resulted in low-grade silica which might also be converted into crystalline form due to the reactions between silica and alkaline salts forming eutectic mixtures of silicates. In the last decade, there have been many attempts of conducting pretreatment of rice husk using different acids solutions such as HCl, H_2SO_4, HNO_3, acetic acid, oxalic acid, citric acid (Chandrasekhar, 2005; Umeda, 2007; Umeda, 2008; Umeda 2009; Liou, 2010; Sapei, 2012; Rafiee, 2012; Sapei, 2015; Bakar, 2016) for the removal of metallic impurities prior to combustion in order to obtain silica with high purity. There have been a tendency of using diluted strong acid of 1-5% (Umeda, 2007; Rafiee, 2012) instead of strong acid solution with ~ 5-20% (Umeda, 2007) and even by using organic acid such as citric acid of 1-7% (Umeda, 2008; Umeda, 2009) for a green and more environmentally benign process. In this research, a more diluted HCl and citric acid of 0.01 M were used as the reagents during acid leaching prior to thermal treatment in order to obtain silica with high purity. The resulted silica was characterized using several methods, such as XRF, FTIR, XRD, and BET-SAA. Silica obtained from this mild process was expected to fulfill the criteria of high-grade amorphous silica with high purity and high specific surface area.

2. Materials and Methods

2.1 Materials

Rice husks were derived from paddy milling processing plant in Mojokerto, East Java. Citric acid p.a. (Sigma Aldrich) and hydrochloric acid 37% (Merck). Aquades.

2.2 Isolation of Rice Husk Silica

Impurities such as sand and rice straws were removed out from rice husks prior to water rinsing and drying at 105^oC in the oven (Memmert, Germany) for 2 hours. The dried clean rice husks were then subjected to a leaching process using diluted acids, i.e. 0.01 M HCl and 0.01 M citric acid. Leaching process was conducted at 100^oC for 60 minutes in a stirred flask with reflux. Afterwards, rice husks were rinsed several times using aquades, filtered, and then dried in the oven at 105^oC for 2 hours. The dried leached rice husks were then thermally treated in a furnace (Ney VULCAN D-550, Dentsply Ceramco, USA) at 750^oC for 5 hours for the organics removal. The remaining ash after combustion contained highly pure silica. Rice husk without acid leaching was also thermally treated as the control.

2.3 Characterisation of Rice Husk Silica

Rice husk ashes which were mainly silica were characterised using several techniques such as XRF (X-ray Fluorescence), FTIR (Fourier Transform Infrared Spectroscopy), XRD (X-ray Diffraction), and BET-SAA (Brunauer, Emmet, Teller-Surface Area Analyzer). Elemental composition of the ashes was determined using (XRF) (PANalytical, MiniPal4, Netherlands) at Physics Laboratory, State University of Malang, Malang. The functional groups of chemical constituents of the ashes were analysed using FTIR (Bruker Tensor, Germany) at Polymer and Membrane Laboratory, University of Surabaya, Surabaya. Samples were prepared using a KBr pellet at dry atmosphere at 25℃. Each spectrum was derived from an average of 32 scans with a resolution of 64 cm^{-1} within the wavenumber range of 400–4000 cm^{-1}. XRD (X'Pert Pro PACAnalytical, Netherlands) measurement was conducted within $2\theta = 5–70^{o}$ at Energy Laboratory, Institut Teknologi Sepuluh November, Surabaya in order to study the crystallinity of the ashes. Finally, the specific surface areas of the ashes were determined using BET-SAA (Quantachrome, USA) at Energy Laboratory, Institut Teknologi Sepuluh November, Surabaya.

3. Results and Discussion

The ash obtained from thermally treated rice husk was about 20% of the native rice husk and ~90% of the ash was silica based on the gravimetric analysis. The ash of untreated rice husk was grey in color, whereas those obtained from diluted treated acids showed a white color as could be seen in Figure 1. The white color reflected silica of high purity, whereas the grey color indicated some trapped carbon inside the silica network due to the eutectic reactions between silica and metallic impurities (Umeda, 2009). Potassium has been the main impurities of which presence disturbed the isolation process of pure silica (Sapei, 2008). The pretreatments of rice husk using diluted acids of both HCl and citric acid prior to thermal treatment have successfully increased the whiteness and brightness of the ashes. This visual observation was supported by the results obtained from XRF analysis.

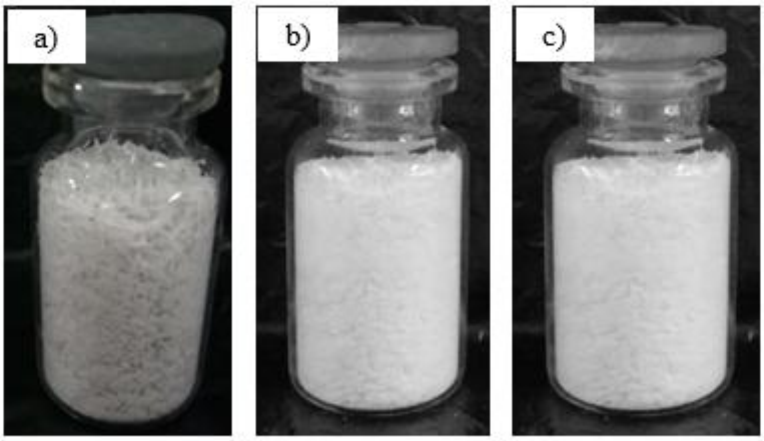

Figure 1. Silica ashes isolated from acid pretreated rice husk followed by thermal treatment at 750°C for 5 hours. a) control; b) diluted HCl treated; c) diluted citric acid treated.

Table 1. Elemental analysis of rice husk silica

Element/ Samples	Content (%)		
	Control	Diluted HCl treated	Diluted citric acid treated
Si	84.7	96.2	94.7
K	4.68	0.18	0.57
Ca	5.83	1.59	1.92
Mn	0.92	0.11	0.18
Fe	1.22	0.26	0.49
Ni	1.56	1.02	1.36
Cu	0.22	0.17	0.21
others	0.87	0.47	0.57

The Si content was increased from ~85% to ~ 95-96% (Table 1) when rice husk was previously treated using diluted acids. The corresponding silica purity as SiO_2 obtained from XRF measurement were 98.6% and 98% for diluted HCl treated and diluted citric acid treated rice husk, respectively. The Si content in biosilica isolated from diluted HCl rice husk was slightly higher than those isolated from diluted citric acid. However, both showed a quite high silica purity of ~98%. These results showed that much diluted acids of 0.01 M of both HCl and citric acids were already sufficient in removing the metallic impurities mainly K in order to obtain high-grade biosilica. There have been about 96% and 88% decrease of K after leaching with diluted HCl and diluted citric acid, respectively. It turned out that diluted HCl seemed to be more powerful in removing the metal impurities compared to diluted citric acid.

HCl as a strong acid was almost fully ionized when mixed with water producing hydrogen ion which was responsible for the dissolution of metallic impurities. During the citric acid leaching, metallic impurities were removed from rice husks due to the chelate reaction between -COOH groups and metallic impurities. These carboxyl groups, tend to donate proton (H^+), resulting in the negatively charged carboxyl group that is capable of forming stable complexes with several metal ions such as K^+ and Ca^{2+} (Pa, 2016). Silica purity of 99.3% was obtained when 1% citric acid was used (Umeda, 2007). In this experiment, 5 times more diluted citric acid was used and comparable result was obtained. Diluted acid leaching proved to be significantly useful and effective in substantially removing the metallic impurities and increase the purity of rice husk silica while reducing environmental problems.

The FTIR spectra of all samples (Figure 2) including control seemed identical and showed a fingerprint of amorphous silica with typical siloxane bands at 460-487 cm^{-1}, 808-823 cm^{-1}, and 1064-1089 cm^{-1}. The peaks at wavenumber 1100-1000 and 800 cm^{-1} were attributed to –$(SiO)_n$– due to asymmetric and symmetric stretching vibration from Si–O–Si, respectively (Socrates, 2001). Absorption band at ~460 cm^{-1} was assigned to bending vibration of O–Si–O

(Moenke, 1974). There was some physically adsorbed water in the biosilica indicated by a small peak at 1635-1658 cm^{-1} due to the vibration of H–O–H bending (Moenke, 1974). A broad hump between 3460-3471 cm^{-1} was assigned to –OH group stretching which could derived from silanol (Si–OH) or H_2O (Moenke, 1974).

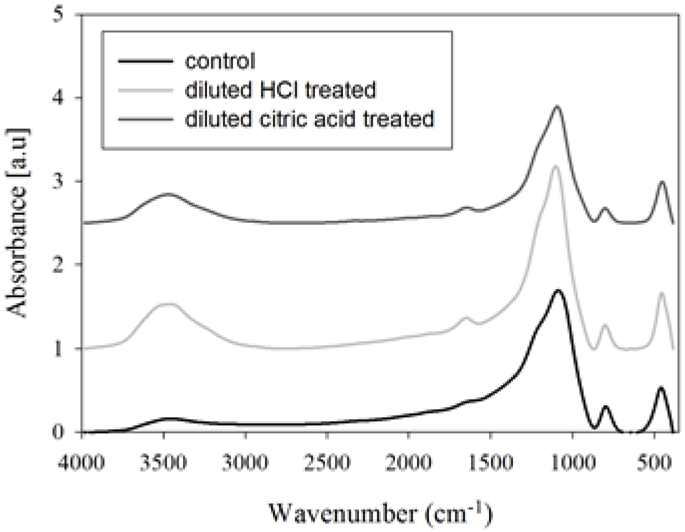

Figure 2. FTIR spectra of rice husk silica

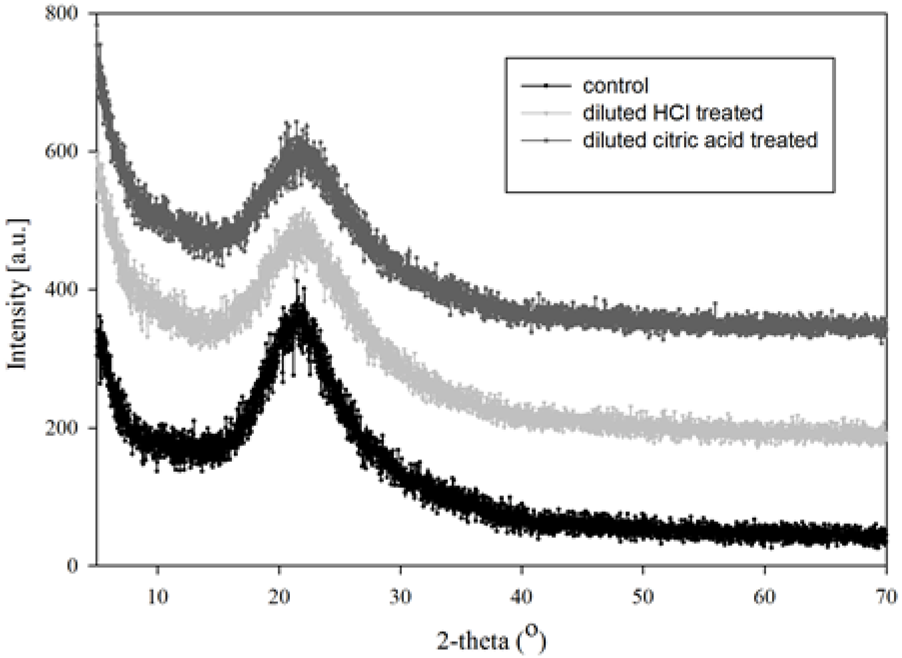

Figure 3. XRD patterns of rice husk silica

All silica ashes both treated with diluted acids and non-treated showed an amorphous form according to XRD analysis as shown in Figure 3. A broad hump with a peak at $2\theta = 22^o$ was a typical characteristics of amorphous silica. This was in line with the previous investigation stating that silica present in rice husk was amorphous in nature (Sapei, 2012; Umeda, 2008).

Furthermore, silica accumulated in plants such as in *Equisetum hyemale* was also amorphous (Sapei, 2007).

Table 2. Specific area of rice husk silica

Sample	BET Specific Area (m^2/g)
Control	17.909
Diluted HCl treated	165.765
Diluted citric acid treated	253.283

The specific surface area of biosilica isolated from diluted acid leached rice husks could be seen in Table 2. The non-treated sample (control) showed the lowest specific surface area of ~18 m^2/g. This was due to the formation of eutectics as the results of the reaction between silica and alkaline metal mainly potassium during the thermal treatment. The low melting point eutectics were easily melted during the thermal treatment thus collapsing the pores of biosilica. Leaching with diluted acids significantly enhanced the specific surface area of about 9 to 14 times. These specific areas were more or less comparable with those obtained using higher concentrations of corresponding acids (Bakar, 2016; Alyosef, 2013; Rafiee, 2012). Interestingly, silica obtained from diluted citric acid treated rice husk showed a much higher specific surface area compared to that obtained from diluted HCl treated rice husk. Citric acid was able to hydrolyze the main organic materials in the husk, such as cellulose and hemicelluloses into simple structure of monosaccharides such as furfural and levoglucosan (Alyosef, 2013). Most probably the hydrolysis of organics by diluted citric acid was more efficient than that in the presence of diluted HCl. The enhanced area was thereby attributed to some pores formed due to the removal of organics. Organic acid could be considered as an alternative chemical to replace the strong acid in the leaching process since organic acid is less corrosive, less hazardous and more environmentally benign compared to HCl. Furthermore, much diluted acid was sufficient in substantially removing metallic impurities in rice husk in order to obtain high-grade silica thus reducing the production cost and environmental problems.

4. Conclusion

Biosilica isolated from diluted acid leached rice husk was amorphous according to XRD analysis. The FTIR spectra of all samples showed the fingerprint of amorphous silica. The Si contents were improved from ~85% to ~95-96% when rice husks were previously treated using diluted acids of both HCl and citric acid prior to thermal treatments. Although the purity of silica derived from HCl treated rice husk had a slightly higher purity, its surface area was about 65% lower than that obtained from citric acid treated rice husk which had a relatively high surface area of 253 m^2/g. The purity and surface area of silica derived from this mild process was comparable with those obtained using more concentrated acids. This proved that leaching using much diluted citric acid seemed to be sufficiently efficient and economical in order to produce high grade silica while also decreasing the environmental problems.

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Content

Content 3
Welcome Message 7
General Information for Participants 8
International Committees 10
International Committee of Nature Sciences 10
Conference Venue Information 13
Conference Schedule 15
Natural Sciences Keynote Speech 18
Oral Sessions 19
Environmental Sciences (1) 19
ICCBES-0026 21
ICCBES-0011 34
ICCBES-0046 35
ICCBES-0101 38
ICCBES-0108 42
Civil Engineering / Electrical Engineering 47
ICEAI-0013 49
ICEAI-0019 63
ICEAI-0021 71
ICEAI-0032 82
ICEAI-0020 91
ICEAI-0044 102
Environmental Sciences (2) 108
ICCBES-0021 110
ICCBES-0049 120
ICCBES-0054 122
ICCBES-0058 124
ICCBES-0076 133
ICCBES-0070 138
Computer Science / Information Engineering / Information Management / Information Technology 141
ICEAI-0040 143
ICEAI-0041 144
ICCIS-0006 146
ICEAI-0006 154
ICEAI-0015 157

ICCIS-0005 159
ICCIS-0003 172
ICCIS-0004 185
Biological Engineering / Biological Sciences / Chemical Engineering 188
ICEAI-0018 190
ICCBES-0066 201
ICEAI-0029 209
ICEAI-0056 211
ICCBES-0115 220
Industrial Engineering 225
ICEAI-0010 227
ICEAI-0012 237
ICEAI-0025 250
ICEAI-0031 260
ICEAI-0033 270
ICEAI-0038 279
ICEAI-0043 291
Chemical Sciences 302
ICCBES-0019 304
ICCBES-0020 307
ICCBES-0025 310
ICCBES-0032 318
ICCBES-0080 320
Energy Engineering 321
ICEAI-0030 323
ICEAI-0034 332
ICEAI-0042 344
ICEAI-0045 354
ICEAI-0052 362
ICEAI-0060 370
Environmental Sciences (3) 372
ICCBES-0073 374
ICCBES-0111 377
ICCBES-0112 379
ICCBES-0113 381
ICCBES-0043 383
Poster Sessions (2) 388
Electrical Engineering / Biological Sciences(1) / Chemical Sciences / Computer Science / Civil Engineering 388

ICEAI-0008 393
ICEAI-0011 402
ICEAI-0022 413
ICEAI-0024 415
ICEAI-0026 422
ICEAI-0035 425
ICCBES-0059 434
ICCBES-0068 436
ICCBES-0071 438
ICCBES-0084 440
ICCBES-0090 441
ICCBES-0091 443
ICCBES-0094 445
ICCBES-0095 447
ICCBES-0022 449
ICCBES-0031 451
ICCBES-0042 454
ICCBES-0047 456
ICCBES-0065 465
ICCBES-0069 468
ICCBES-0077 471
ICCBES-0081 474
ICCBES-0105 476
ICEAI-0027 478
ICEAI-0036 480
ICEAI-0055 483
Poster Sessions (3) 494
Biological Sciences (2) / Information Engineering / Environmental Sciences / Environmental Engineering / Energy Engineering / Industrial Engineering 494
ICCBES-0009 500
ICCBES-0010 502
ICCBES-0013 503
ICCBES-0014 505
ICCBES-0023 507
ICCBES-0028 509
ICCBES-0034 525
ICCBES-0055 527
ICCBES-0064 529
ICCBES-0078 531

ICEAI-0014 .... **534**
ICEAI-0037 .... **537**
ICEAI-0054 .... **542**
ICCIS-0002 .... **553**
ICCBES-0027 .... **564**
ICCBES-0051 .... **567**
ICCBES-0056 .... **569**
ICCBES-0057 .... **570**
ICCBES-0079 .... **571**
ICCBES-0088 .... **572**
ICCBES-0098 .... **580**
ICEAI-0062 .... **582**
ICEAI-0003 .... **594**
ICEAI-0049 .... **596**
ICEAI-0039 .... **598**

Welcome Message

Local Host

Michelle Kawamura Ph.D
Associate Professor
Ritsumeikan University

Dear scholars and friends,

On behalf of Higher Education Forum I would like to welcome you all to Kyoto, Japan. Kyoto is a city representing a mixture of traditional Japanese cultures and modernization. From the well preserved constructions over centuries old, thousands of Buddhist temples and Shinto shrines to the untouched nature, this one of a kind place is unique in today's world. It is also these elements which attract people who appreciate and value historical surroundings. Beyond the various fascinating historical and traditional sites in Kyoto, the city also offers convenient transportation, comfortable accommodation, delicacies from all parts of the world and most of all, the hospitality of Kyoto. People in Kyoto welcome tourists to explore their culture, experience their customs and traditions. All these reasons led to an increasing number of foreign visitors to Kyoto every year. Some people visit for the first time and many others visit more than once.

Each year, many scholastic conferences of various disciplines take place in Kyoto. Kyoto has over 33 national, public and private universities. It also has over 57 vocational and technical schools. Including graduate schools and public and private research institutions, Kyoto takes pride in its education system, scholastic and research development. This is one of the reasons that many domestic and international conferences select Kyoto to host their conferences.

This is my third time, in three years to proudly act as the local host of Kyoto for the Higher Education Forum to welcome all the guests from all over the world. I have presented in 2014 and gave a welcome and keynote speech last year. I have met many professionals from different fields and exchanged knowledge from our researches to cultural experiences. The opportunity here is beyond scholastic exchange, but to also connect us to various cultures to become more aware of cultural differences, empathy and tolerance for others. I have gained new friends and learned profoundly here in the past 2 years participating in the conferences held by the Higher Education Forum. I sincerely hope you will enjoy your stay in Kyoto and bring home lots of experiences and knowledge from this conference.

See you soon~

Michelle Kawamura

General Information for Participants

Registration

The registration desk will be situated on the **4th Floor** at the **Kyoto Research Park** during the following time:

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Participants are requested to arrive in a timely fashion for all addresses. Presenters are reminded that the time slots should be divided fairly and equally by the number of presentations, and that they should not overrun. The session chair is asked to assume this timekeeping role and to summarize key issues in each topic.

Sandals or Slippers

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A certificate of attendance includes participant's name and affiliation, certifying the participation in the conference. A certificate of presentation indicates a presenter's name, affiliation and the paper title that is presented in the scheduled session.

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Oral presenters will receive a certificate of presentation from the session chair after their presentations or at the end of the session. Poster presenters will receive a certificate of presentation from the conference staff at the end of their poster session.

The certificate of presentation will not be issued, either at or after the conference, to authors whose papers are registered but not presented. Instead, the certificate of attendance will be provided after the conference.

■ Preparation for Oral Presentations

All presentation rooms are equipped with a screen, an LCD projector, and a laptop computer installed with Microsoft PowerPoint. You will be able to insert your USB flash drive into the computer and double check your file in PowerPoint. We recommend you to bring two copies of the file in case that one fails. You may also connect your own laptop to the provided projector; however please ensure you have the requisite connector.

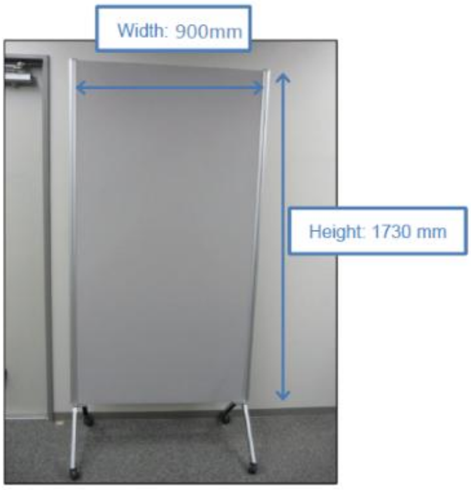
Preparation for Poster Presentation

Materials Provided by the Conference Organizer:

1. Poster Panel (90cm×173cm)
2. Push pins

Materials Prepared by the Presenters:

1. Home-made poster(s)
2. Material: not limited, can be posted on the poster panel
3. Maximum poster size is A0



(Example)

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Michiko Miyamoto	Akita Prefectural University	Japan
Minagawa, Masaru	Tokyo City University	Japan

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Sergei Gorlatch	University of Muenster	Germany
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Yee-Wen Yen	National Taiwan University of Science and Technology	Taiwan
Yoshida Masafumi	Tokyo City University	Japan
Youngjune Park	Gwangju Institute of Science and Technology	South Korea
Yuan-Lung Lo	Tamkang University	Taiwan

Conference Venue Information

Kyoto Research Park

Tel: +81-75-322-7800

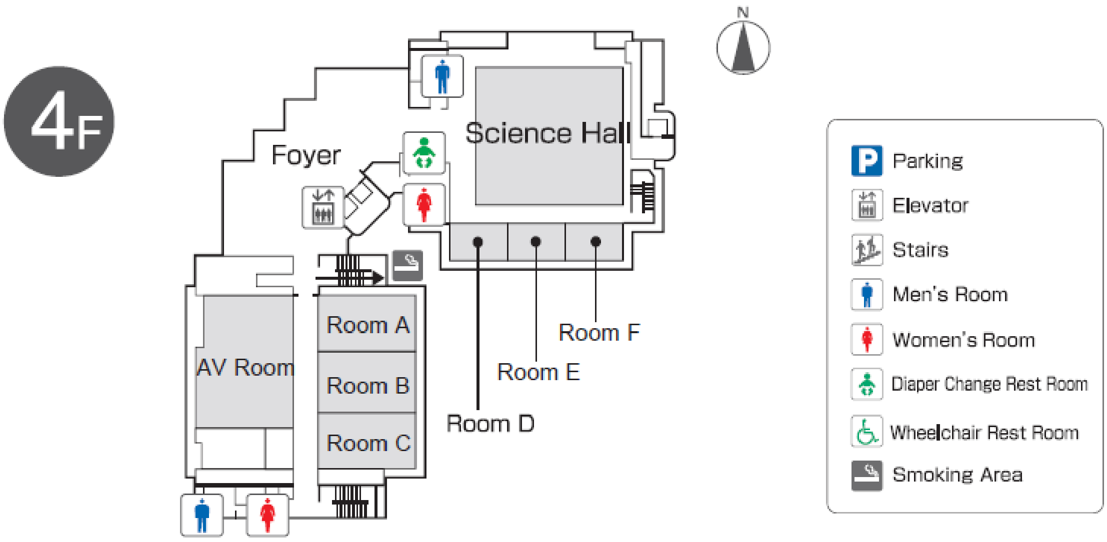
Address: 134, Chudoji Minami-machi, Shimogyo-ku, Kyoto 600-8813, Japan

Located at the ancient capital of Japan, Kyoto Research Park (KRP) is set at a convenient spot. KRP has a wide variety of networks involving universities, industries, administrative institutes, public research institutes, and economic bodies. KRP offers various of conference rooms, offices, and labs.

The conference will be held at Hall 1 inside the research park. Hall 1 is near to JR Tanbaguchi-Eki.

Floor Map (4th Floor)

Floor Map of Bldg. #1



Conference Schedule

Tuesday, May 09, 2017
Internal Training

Wednesday, May 10, 2017		
Oral Session (4th Floor)		
Time	**Schedule**	**Venue**
08:10-16:00	**Registration**	Foyer Area
08:30-10:00	Environmental Sciences (1)	Room B
	Civil Engineering / Electrical Engineering	Room C
10:00-10:20	**Tea Break & Networking**	Foyer Area
10:20-12:30	Environmental Sciences (2)	Room C
	Welcome Speech & Networking(10:20-10:30) **Prof. Michelle Kawamura** Ritsumeikan University -- **Nature Science Keynote Speech (10:30-11:30)** **Prof. Simona Vasilache** University of Tsukuba Topic: *Multicultural Perspectives on Software Engineering*	AV Room
12:30-13:30	**Lunch**	Atrium
15:00-15:20	**Tea Break & Networking**	Foyer Area
15:20-16:50	Computer Science / Information Engineering / Information Management / Information Technology	Room B

Wednesday, May 10, 2017	
Poster Session (Foyer Area, 4th floor)	
Time	**Schedule**
14:00-15:00	Poster Session (2)
	Electrical Engineering / Biological Sciences (1) / Chemical Sciences / Computer Science / Civil Engineering

Thursday, May 11, 2017		
Oral Session (4th Floor)		
Time	**Schedule**	**Venue**
08:30-16:30	**Registration**	Foyer Area
08:40-10:10	Biological Engineering / Biological Sciences / Chemical Engineering	Room B
	Industrial Engineering	Room C
10:10-10:30	**Tea Break & Networking**	Foyer Area
10:30-12:00	Chemical Sciences	Room B
	Energy Engineering	Room C
12:00-13:00	**Lunch**	Atrium
13:00-14:30	Environmental Sciences (3)	Room C
14:30-14:50	**Tea Break & Networking**	Foyer Area
16:20-16:30	**Tea Break & Networking**	Foyer Area

Thursday, May 11, 2017	
Poster Session (Foyer Area, 4th floor)	
Time	**Schedule**
11:00-12:00	Poster Session (3)
	Biological Sciences (2) / Information Engineering / Environmental Sciences / Environmental Engineering / Energy Engineering / Industrial Engineering

Natural Sciences Keynote Speech

AV Room

10:30-11:30 Wednesday, May 10, 2017

Topic:
Multicultural Perspectives on Software Engineering

Prof. Simona Vasilache
Dept. of Computer Science
Graduate School of Systems and Information
University of Tsukuba
Japan

Abstract
Globalization plays a major role in all aspects of our 21st century life, influencing software engineering practice, as well as software engineering education.
Software engineering is a relatively young engineering discipline and it is under constant development and evolution; software development teams often include members from various cultural backgrounds. At the same time, the multicultural nature of education proves to be a challenge in all educational aspects, including those related to software engineering .
This talk will highlight some of the challenges posed by multiculturalism in the field of software engineering, both in industry and in academia.

Introduction of Prof. Simona Vasilache
Simona Vasilache is an assistant professor in the Graduate School of Systems and Information Engineering at the University of Tsukuba in Japan. She completed her undergraduate studies at the Politechnica University of Bucharest, Romania, and she obtained her PhD from the University of Tsukuba in Japan. Her research interests include human computer interaction, software engineering, formal methods, cultural aspects of e-learning, as well as intercultural communication.
For the past 5 years she has been teaching a culturally diverse group of international students in Japan.

Environmental Sciences (1)

Wednesday, May 10, 2017 08:30-10:00 Room B

Session Chair: *Prof. Ayca Erdem*

ICCBES-0026

Investigation and Assessment of Energy and Resource Integration into Sewage Treatment and Waste Heat Recovery

Kuang-Chih Chang | *Ministry of Economic Affairs*
Ya-Feng Wang | *Chung Yuan Christian University*
Tien-Chin Chang | *National Taipei University of Technology*
Lin-Han Chiang Hsieh | *Chung Yuan Christian University*
Ciao-En Hu | *National Taipei University of Technology*

ICCBES-0011

Seasonal Evaluation of the Sanitary Quality of the Alexandrian Mediterranean Coast Using Microbial Indicators

Amira M. Hamdan | *Alexandria University*

ICCBES-0046

Current Research Trends in Environmental Engineering: Content Analysis of Publications on Micropollutants

Merve Ozkaleli | *Akdeniz University*
Ayse Gunay | *Akdeniz University*
Meltem Asilturk | *Akdeniz University*
Cigdem Moral | *Akdeniz University*
Ayca Erdem | *Akdeniz University*

ICCBES-0101

Effect of Dopants on Pt/γ-Al_2O_3 Catalyst in Propane Dehydrogenation

Nattanon Chartsakulsaka | *Chulalongkorn University*
Adisak Guntidaa | *Chulalongkorn University*
Joongjai Panpranota | *Chulalongkorn University*
Kongkiat Suriyeb | *SCG Chemicals*
Piyasan Praserthdama | *Chulalongkorn University*

ICCBES-0108

The Effect of Loading Sequence of the La-Promoted WO_3/Sio_2 Catalysts in the Cross-Metathesis of Ethylene and 2-Butene and Self-Metathesis of Propylene

Thotsapan Kornpitak｜*Chulalongkorn University*
Joongjai Panpranot｜*Chulalongkorn University*
Piyasan Praserthdam｜*Chulalongkorn University*

Biological Engineering / Biological Sciences / Chemical Engineering

Thursday, May 11, 2017 08:40-10:10 Room B

Session Chair: *Prof. Lanny Sapei*

ICEAI-0018

Optimized Hot Air Flow Distribution System for Increasing the Dialyzer Drying Process Performance

Mohammad Fard | *Novatisteb Company*

Ali Rezaee | *Novatisteb Company*

ICCBES-0066

A Method for the Quantification of 18F-FDOPA in PET Imaging for Parkinson's Disease Correlation to Brain Structures

Printaporn Sanguansuttigul | *King Mongkut's University of Technology Thonburi*

Teerapol Saleewong | *King Mongkut's University of Technology Thonburi*

Sayaphat Suksai | *Srinakharinwirot University*

Kitiwat Khamwan | *Chulalongkorn University*

Saknan Bongsebandhu-Phubhakdi | *Chulalongkorn University*

ICEAI-0029

Simulations of Geometrical Effects on Transport Properties Through Micro- and Nanopores

Attaphon Chaimanatsakun | *Kasetsart University*

Deanpen Japrung | *Thailand Science Park*

Prapasiri Pongprayoon | *Kasetsart University*

ICEAI-0056

High-Grade Biosilica Isolated from Diluted Acids Leached Rice Husk

Lanny Sapei | *University of Surabaya*

Natalia Suseno | *University of Surabaya*

Karsono Samuel Padmawijaya | *University of Surabaya*

Jessica Natalia Wibowo | *University of Surabaya*

Chandra Widjaja | *University of Surabaya*

ICCBES-0115

Effects of Natural Steroid Sand Fish (*Holothuria Scabra*) for Uterus Development of Post Menopausal Women

Etty Riani｜*Bogor Agricultural University*
Chairunissa｜*Bogor Agricultural University*
Hera Maheswari｜*Bogor Agricultural University*
Farah Bilqistiputri｜*Lampung University*
Nastiti Kusumorini｜*Bogor Agricultural University*