The 3-rd JASTIP-WP2 Annual Workshop
Feb. 5, 2018(Novotel Bangkok on Siam Square Hotel)

Extension of Solvent Treatment Method Developed by SATREPS Program to ASEAN Region

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Members of our group (tentative)

Hideaki Ohgaki, Proferssor, Institute of Advanced Energy, Kyoto University
Ryuichi Ashida, Lecturer, Graduate School of Engineering, Kyoto University
Janewit Wannapeera, Researcher, Institute of Advanced Energy, Kyoto University
Katsuyasu Sugawara, Professor, Akita University
Nakorn Worasunarak, Assoc. Professor, JGSEE/KMUTT
Suneerat Fukuda, Assoc. Professor, JGSEE/KMUTT

Japan-Thailand SATREPS Project

Development of clean and efficient utilization of low rank coals and biomass by solvent treatment

Dec. 20, 2013 – Jan. 7, 2019

Kouichi Miura
Institute of Advanced Energy,
Kyoto University

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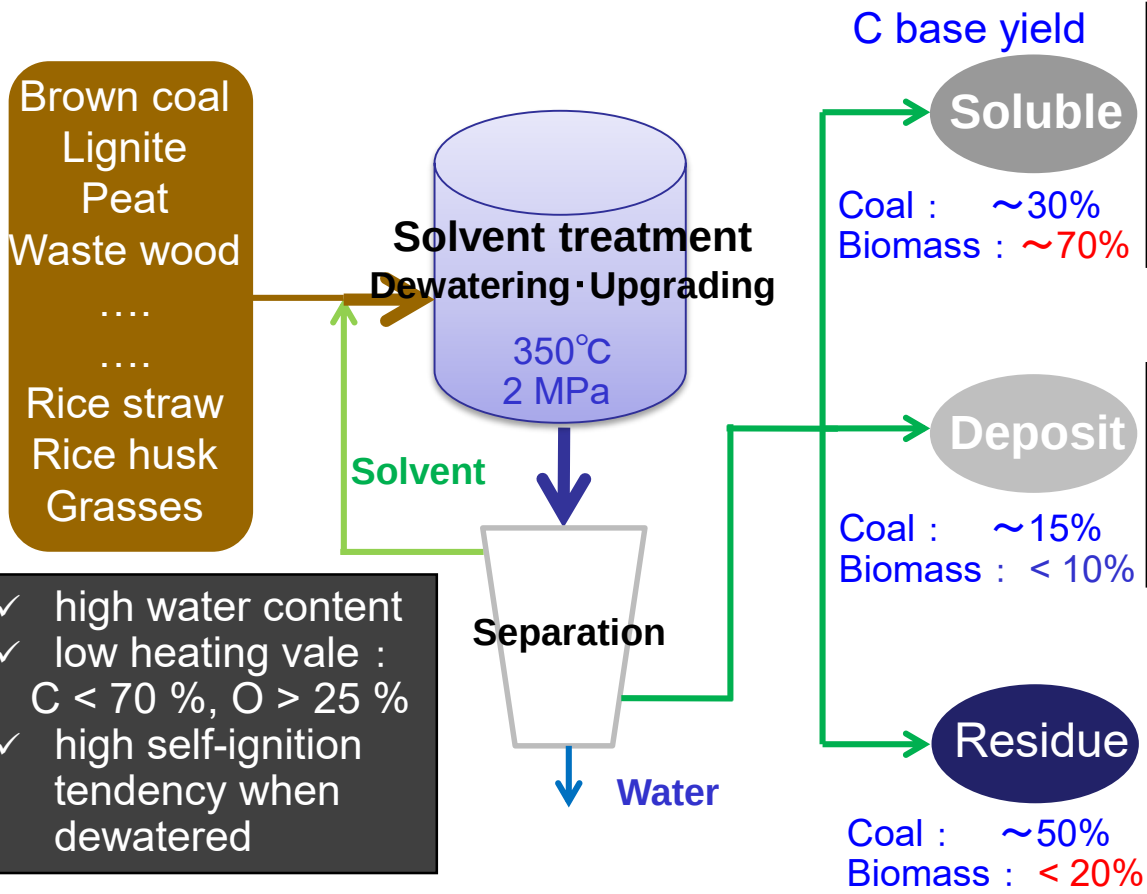
JGSEE/King Mongkut's University of Technology Thonburi

Purposes of the SATREPS Project

1. To establish a technology converting low rank coals and/or biomass wastes using a new method called "Degradative Solvent Extraction", which was developed by Kyoto University group, to raw material independent small molecular weight components called "Soluble" and Residue.
2. To develop technologies for utilizing Soluble and Residue effectively.
   eg. Preparation of value added materials such as carbon fiber, clean fuel, chemicals, etc. Effective methods to combust/gasify Residue
3. To assist the development of human resources and research capabilities in Thailand by conducting joint research.

- The technologies developed under cooperative researches will contribute to reduce the emission of global warming gases as well as environmental pollutants.
- The technologies developed will be disseminated to ASEAN countries which need such technologies.

Core technology is “Degradative Solvent Extraction”



Raw material independent property
high HHV：C≒82 %, H ≒9 %, O ≒9%
low MW: 100-500, peak at ca. 300
80 % are volatile components
melts below 100 °C
free from water and ash

Raw material independent property
C ≒76 %, H ≒5.5 %, O ≒15.5%
melts below 250 °C
free from water
low ash content：＜1.4 %

High reactivity to combustion
low water content
Concentrated mineral components

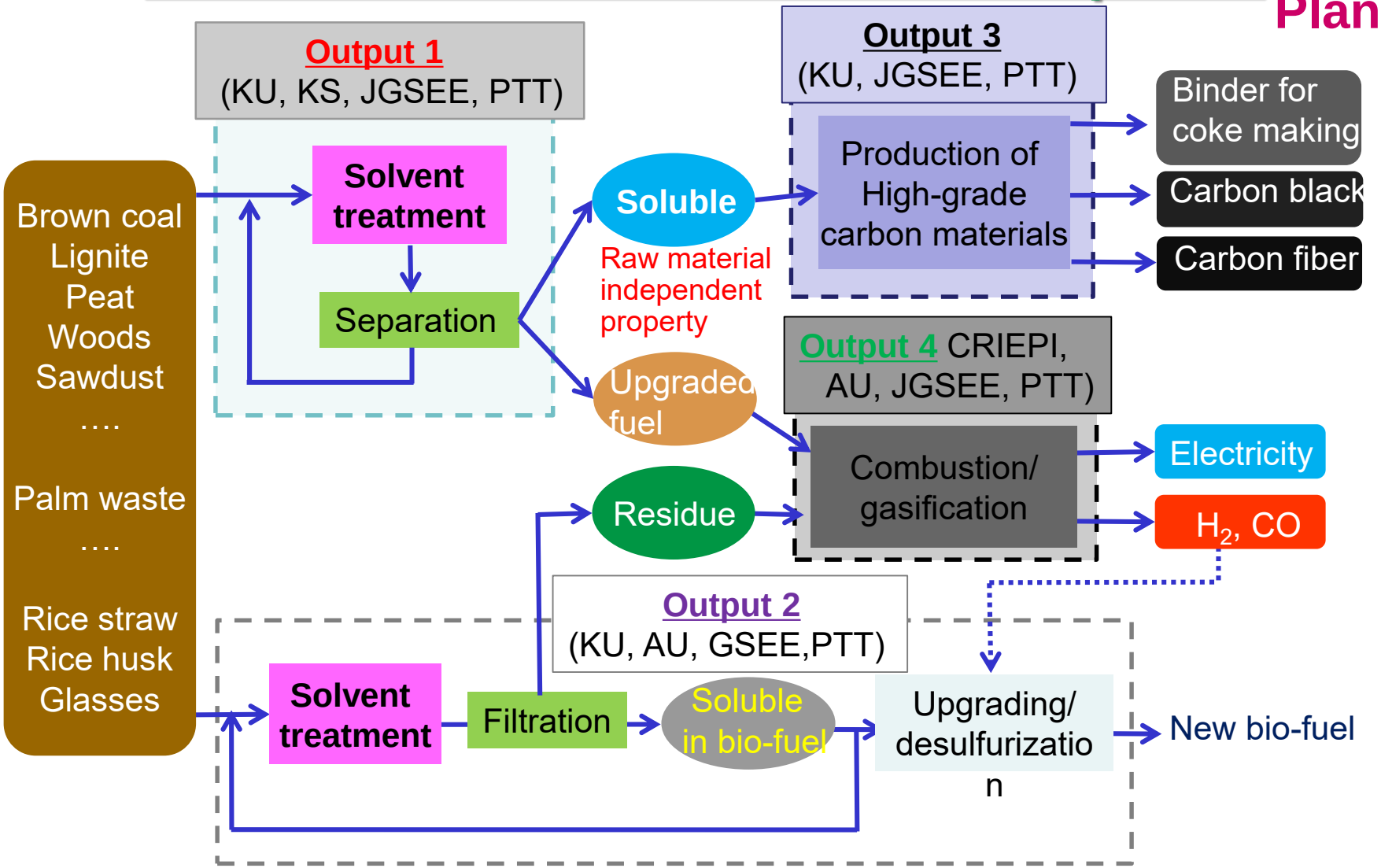
- ✓ high water content
- ✓ low heating vale：
  C < 70 %, O > 25 %
- ✓ high self-ignition tendency when dewatered

The method dewaters and upgrades various low grade carbonaceous resources, producing high quality extract in high yield under mild conditions.

- ➢ Almost no heating value loss through the treatment
- ➢ Soluble and Deposit have raw material independent properties

Structure of Research and Development

Planned



Output 1: Upgrading of low rank coals and biomass by solvent treatment
Output 2: Production of new bio-fuel from biomass wastes and effective upgrading
Output 3: Production of high-grade carbon materials from the Solubles
Output 4: Combustion/gasification of upgraded fuels/residues

Cooperative Structure of our project

Japan

Head Investigator: Kouichi Miura
Research fund: 178 million yen from JST

Kyoto University: Miura Gr.
Kouichi Miura, Specially App. Prof.
Hideaki Ohgaki, Prof
Ryuichi Ashida, Assist. Prof.
Motoaki Kawase, Prof.
Taro Sonobe, Research Administrator
Janewit Wannapeera, Dr.
Trairat Muangthong-on, PhD cand.

Akita University: Sugawara Gr.
Katsuyasu Sugawara, Prof.
Takahiro, Kato, Assis. Prof.
Kenji Murakami, Prof.

CRIEPI: Makino Gr.
Hisao Makino, Dr.
Kenji Tanno, Dr.
Satoshi Umemoto, Dr.
Atsushi Ikeda, Mr.
Shiro Kajitani, Dr.

Kobe Steel Co. Ltd: Okuyama Gr
Noriyuki Okuyama, Dr.
Takuya Yoshida, Dr.
Shigeru Kinoshia, Mr.
Koji Sakai, Mr.

Thailand

Head Investigator: Bundit Fungtammasan
Research fund: 300 million yen from ODA

JGSEE/KMUTT: Bundit Gr.
Assoc.Prof. Bundit Fungtammasan
Assoc.Prof. Sirintornthep Tawprayoon
Assoc.Prof. Nakorn Worasuwannarak
Assoc.Prof. Suneerat Fukuda
Dr. Supachita Krerkkaiwan
Ms. Sasithorn Buranatrevedhya
Mr. Supachai Jadsadajerm
Mr.Jaggapan Sanduang
Ms.Thitima Sornpitak
Mr.Kaweewong Wongaiyara

PTT-RTI, PTT Public Company Ltd: Arunratt Gr.
Arunratt Wuttimongkolchai, Ms.
Suttipong Tunyapisetsak, Mr.
Suchada Butnark, Dr.
Anurak Winitsorn, Dr.
Suriya Porntangjitlikit, Mr.
Kornthape Prasirtsiripham, Mr.

Four research groups from Japan and two research groups from Thailand are involved in this project.

More than 30 researchers from academy and industry contribute to this project

Planned Schedule of Research and Development

Activity	Schedule (from 2014 to 2018)																				Group in charge	
	2014				2015				2016				2017				2018				Japan	Thailand
	1Q	2Q	3Q	4Q	1Q	2Q	3Q	4Q	1Q	2Q	3Q	4Q	1Q	2Q	3Q	4Q	1Q	2Q	3Q	4Q		
Task 1. Upgrading of low rank coals and biomass by solvent treatment																						
1.1 Production of Solubles from low rank coals and biomass using a batch autoclave																					KU	JGSEE
1.2 To optimize the production of Solubles from low rank coals and biomass																					KU	JGSEE
1.3 To characterize the properties of Solubles and Residues from low rank coals and biomass																					KU	JGSEE
1.4 To design and construct the semi-continuous extraction process (1 kg/h)																					KS	PTT
1.5 Production of Solubles from low rank coals and biomass using the semi-continuous extraction process																					KS	PTT
1.6 Conceptual process design for constructing a pilot plant of 10 ton/day																					KS	PTT
Task 2. Production of new liquid biofuels from solubles																						
2.1 Optimization of production of liquid biofuels using batch reactor (5 L)																					KU	JGSEE, PTT
2.2 Upgrading liquid products to liquid biofuels by hydroprocessing																					AU	PTT
2.3 Combustion test in gas turbine engine																					CRIEPI, KS	PTT
2.4 Cost estimation, feasibility study and scale-up plant (in case of technical soundness)																					KS	PTT
Task 3. Production of high-grade carbonaceous materials from Solubles																						
3.1 Characterization of Solubles as a raw material for high performance carbon materials																					KU	JGSEE
3.2 Design and construct a small apparatus producing carbon fiber/carbon black																					KU	JGSEE
3.3 Production of carbon fiber from Solubles																					KU	JGSEE
3.4 Design and construct a small continuous spinning apparatus (0.1 kg/h)																					KU	JGSEE
3.5 Production of carbon fiber using a small continuous spinning apparatus																					KU	JGSEE PTT
3.6 Conceptual process design for a pilot plant																					KU	JGSEE
Task 4: Combustion/gasification of upgraded fuels/residues																						
4-1 Fundamental Examination of combustion/gasification behaviors of upgraded fuels/residues in TG																					CRIEPI,AU	JGSEE,PTT
4-2 Examination of combustion/gasification behaviors of upgraded fuels/residues in Entrained bed reactor																					CRIEPI,AU	JGSEE,PTT
4-3 Examination of combustion behaviors of upgraded fuels/residues in Fluidized bed reactor																					CRIEPI	JGSEE

Dispatch of researchers
Acceptance of researchers

History of exchange

Year	Number of dispatch researchers	Number x Day (man-day)	Number of accepted researchers	Number x Day (man-day)
2013	11	55	1	60
2014	39	311	11	255
2015	27	249	9	123
2016	19	197	12	154
2017	22	184	12	276*
Total	**118**	**996**	**45**	**868**

*) includes the acceptance by other funds

Visit COOLGEN by Thai members (Osaki, Feb. 6, 2017)

JST Review Meeting at Kita-Kyushu (Feb. 7, 2017)

On Feb. 8 Thai members had a chance to visit the Yahata work of Nippon Steel & Sumitomo Metal Co. Ltd.

Joint Coordinating Committee Meeting(Nov.22)

Provision of equipment

Introduction of equipment by the JICA fund

Equipment	Specification	Installation site	Installation date
CHN analyzer	J-Science, JM-10	JGSEE/KMUTT	2014/9/1
Micro balance	AND, BM-20	JGSEE/KMUTT	2014/9/1
Rotary evaporator	Buchi, R-210/V-850	JGSEE/KMUTT	2014/9/1
TMA analyzer	Shimadzu, TMA-60	JGSEE/KMUTT	2014/9/1
Dryer	Yamato, DKN402	JGSEE/KMUTT	2014/9/1
Vacuum dryer	Yamato, ADP300	JGSEE/KMUTT	2014/9/1
Autoclave (0.5 L)	Nitto-Koatsu, Custom made	JGSEE/KMUTT	2014/9/1
Autoclave (0.5 L)	Nitto-Koatsu, Custom made	PTT-RTI	2014/9/1
Carbon fiber spinning machine	Musashino Kikai, Custom made	JKGSEE/KMUTT	2015/5/26
SEM with EDS analyzer	JEOL, JCM6000	JSEE/KMUTT	2015/5/26
Thermobalance	Shimadzu, TG50H	JGSEE/KMUTT	2015/5/26
Steam generator	Hirota, KJ-1505	JGSEE/KMUTT	2015/5/26
Atomic absorption spectrometer	Shimadzu, AA-7000G	JGSEE/KMUTT	2015/5/26
Sulfur/Carbon analyzer	Horiba, EMIA-220V2	JGSEE/KMUTT	2015/5/26
Drop tube furnace	Elec. Techno-System, Cusom made	JGSEE/KMUTT	2016/1/4

Introduction of equipment by the JICA fund

Equipment	Specification	Installation site	Installation date
Testing machine	Shimadzu, EZ-SX 5N	JGSEE/ KMUTT	2016/5/25
GCMS	Shimadzu,GCMS-QP2010 SE	JGSEE/ KMUTT	2016/5/25
Thermobalance	Shimadzu, TG50H	JGSEE/ KMUTT	2016/5/25
Micro gaschromatograph	GL Science, 490AB-GC	JGSEE/ KMUTT	2016/5/25
Plasma asher	J-SCIENCE, JPA300	JGSEE/ KMUTT	2016/5/25
Semi-cont. extractor	**Custom made**	**PTT-RTI**	**2017/10**

- **In total 23 equipment were introduced to JGSEE/KMUTT and PTT-RTI.**
- **One big bench plant was introduced to PTT-RTI this year.**

Arriving of the apparatus at PTT-RTI

(Oct. 3, 2017)

Completion of Acceptance Inspection at PTT-RTI (Oct. 5, 2017)

Training by Kobe Steel members at PTT-RTI (Nov. 1-21, 2017)

Training by Kobe Steel members at PTT-RTI (Nov. 1-21, 2017)

Construction and Operation Schedule of Semi-continuous unit (2)

	November, 2017													
week	12	13	14	15	16	17	18	19	20	21	22	23	24	25
date	Sun	Mon	Tue	Wed	Thu	Fri	Sat	Sun	Mon	Tue	Wed	Thu	Fri	Sat
Event		Exp. using only A150		Act. operation (Rice straw)					Act. operation (Rice straw)		JCC meeting & Facility tour	Project proceed meeting		
Content		Flash operation		Carried out extraction filtration and flash distillation					Sample recovery	Clean-up				

Leakage trouble
Emergency shut down

Briquette charging
Efficient way

Got Soluble sample

V-05 bottom (Solidified extract)

Extract recovered

Soluble

Members visiting the semi-cont. extractor (Nov.22)

Members visiting the semi-cont. extractor (Nov.22)

Research Activities in 2017

Task 3: Production of High-Grade Carbon Materials from the Soluble

2013-2016: Characterization of Soluble as a raw material for high performance carbon materials

- ✓ Examine the softening/melting behaviors of Solubles
- ✓ Characterize the chemical properties of Solubles as carbon fiber precursor

2014-2017: Examination of possibility of production of carbon fiber from Soluble

- ✓ Optimization of carbon fiber production from several raw materials in laboratory scale
- ✓ Investigate the parameters effecting the hollow formation in carbon fiber
- ✓ Examine the possibility of production of activated carbon fiber from Soluble

2016-2018 : Production of carbon fiber using a small continuous spinning apparatus

⇨ Production of carbon fiber from the Mae Moh coal Soluble

⇨ Production of carbon fiber from the rice straw Soluble prepared by using a semi-continuous solvent extraction process (A150 as a Solvent)

Production of Carbon Fiber from Rice straw (RS) and Mae Moh (MM) coal Solubles

Carbon fiber production process

Soluble

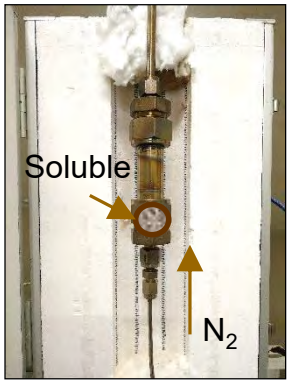
Rice straw:
C=81.1%, H=7.0%, N=2.1%, O=9.4%

Mae Moh coal:
C=81.4%, H=7.1%, N=1.7%, O+S=9.8%

Melting point: less than 80 ° C

Pretreatment

Pitch-like precursor



Vertical tube furnace

N_2, 10 ° C/min, 280-320 ° C, 80-100 min

Melting point: around 170 ° C

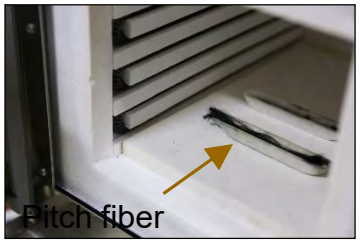
Melt Spinning

Pitch fiber

Melt-spinning machine: N_2, 280 ° C, 180 m/min

Stabilization

Stabilized fiber

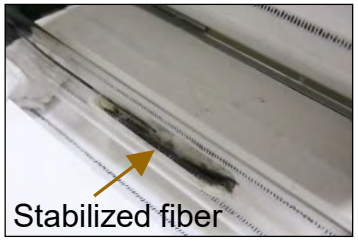


Muffle furnace

Air, 0.5 ° C/min, 300 ° C, 60 min

Carbonization

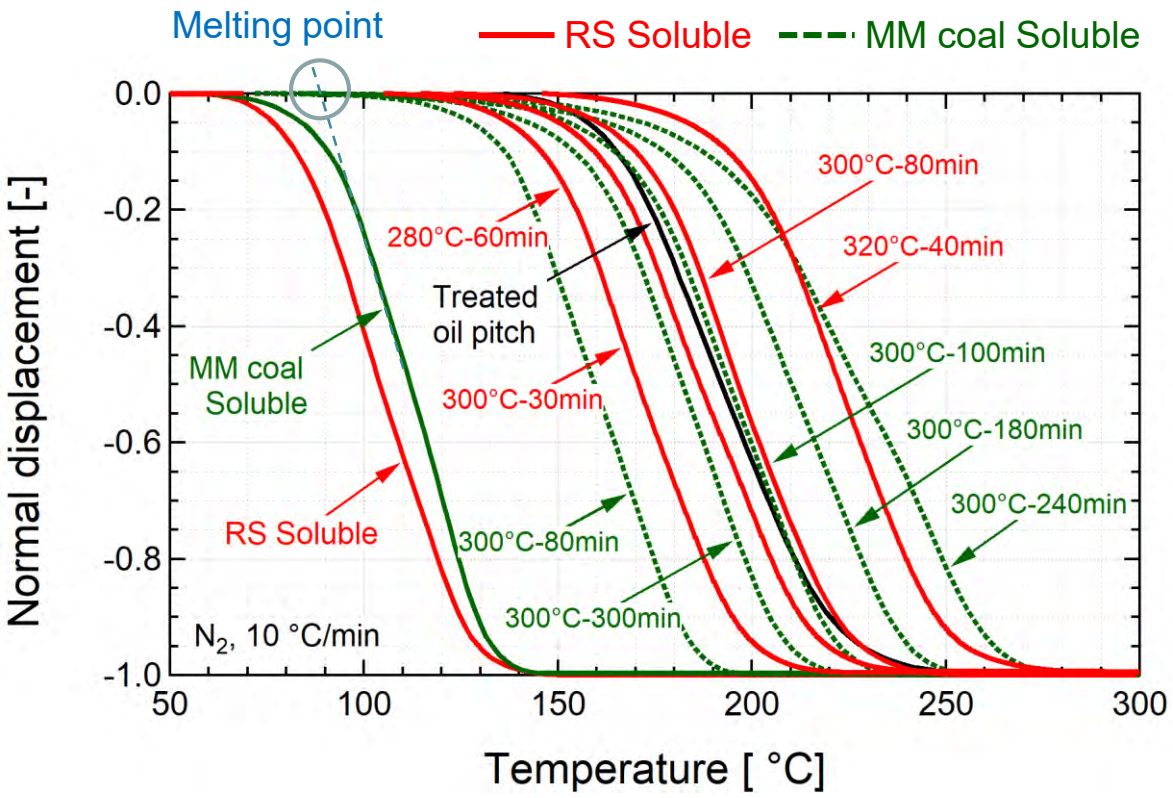
Carbon fiber

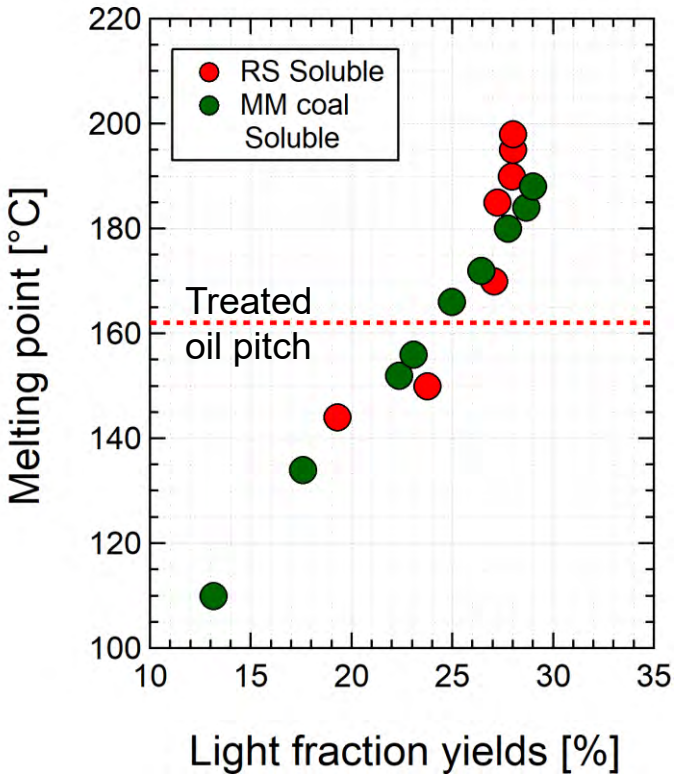


Horizontal tube furnace

N_2, 5 ° C/min, 900 ° C, 60 min

Pitch-like Precursor: Treated Oil Pitch as a Reference





At melting point = Treated oil pitch (170° C)

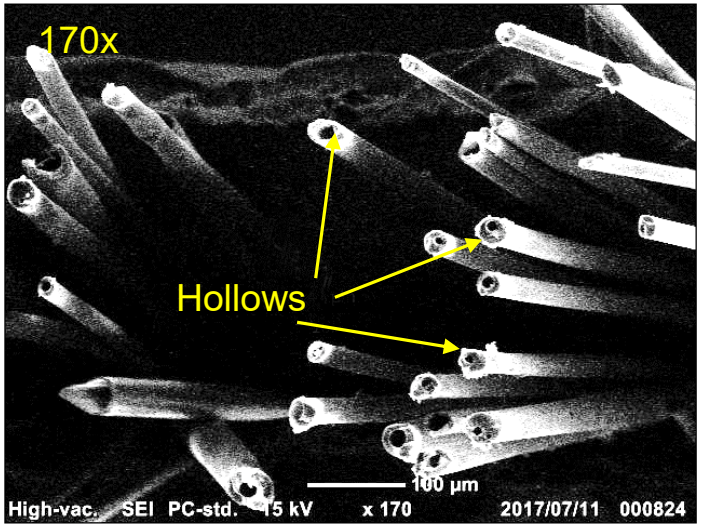
RS 300° C-80 min: Yield = 72.9%, C=83.0%, H=6.5%, N=2.2%, O=8.3%

MM 300° C-100 min: Yield = 75.0%, C=89.3%, H=0.3%, N=1.9%, O+S=8.4%

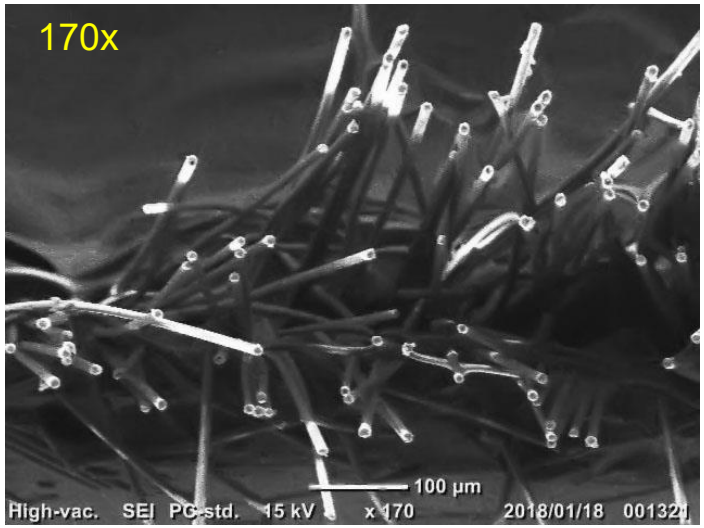
Pitch Fiber

- Hollows form after melt-spinning process of rice straw Soluble

Pitch fiber spun at 180 m/min

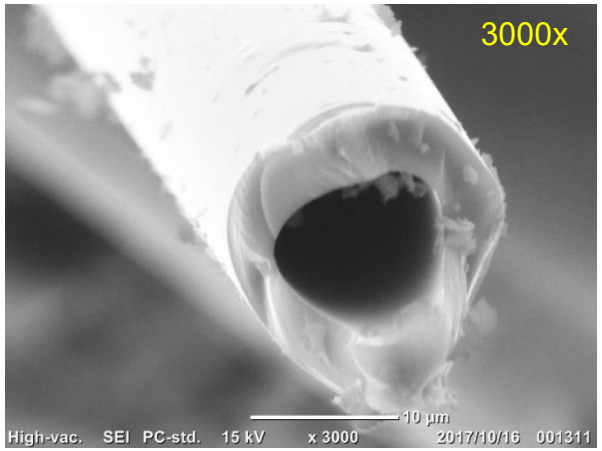


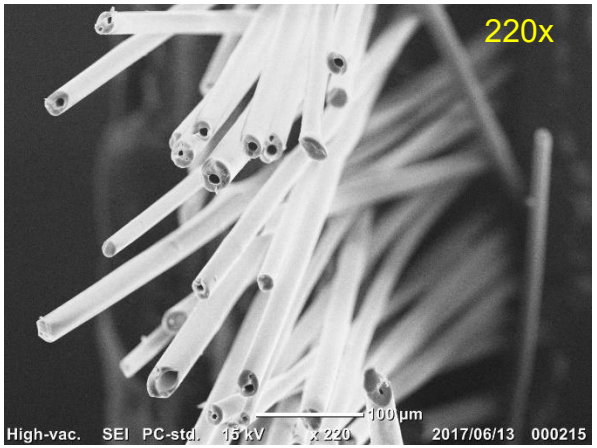
Rice straw Soluble

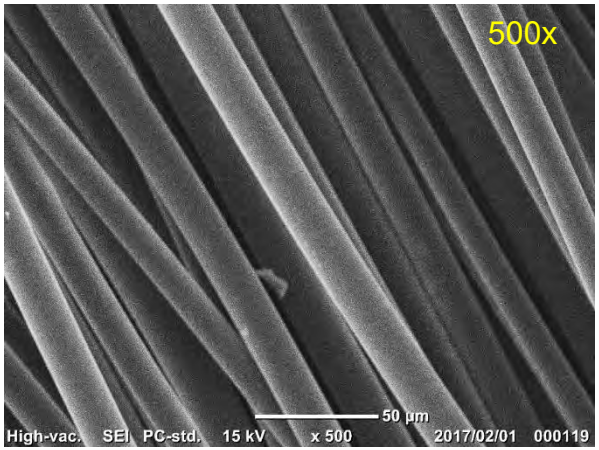


Mae Moh coal Soluble

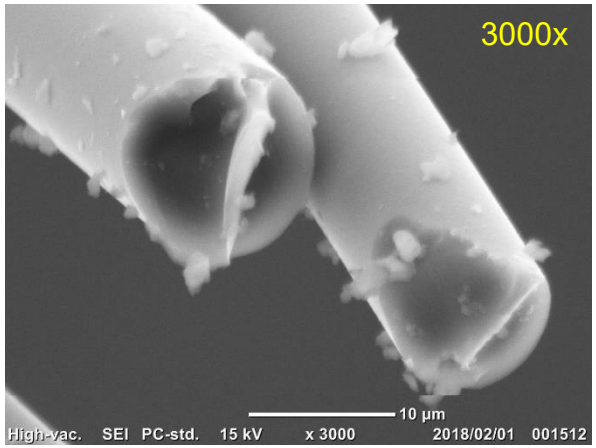
Carbon Fiber

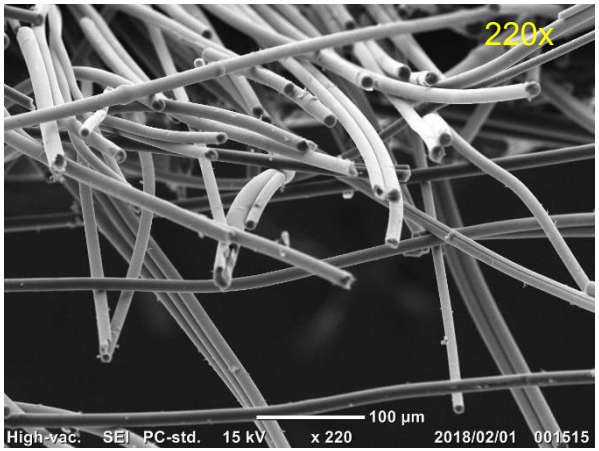






Rice straw carbon fiber: Yield = 33.6% on Soluble basis, Avg. dia. = 20±3 µm, Tensile strength = 148±36 MPa,
C = 85.9%, H = 1.7%, N = 2.7%, O = 9.7%

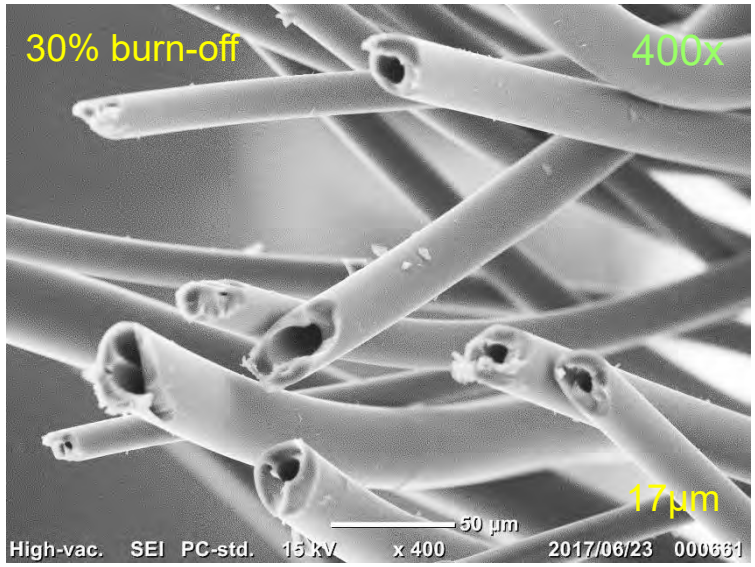


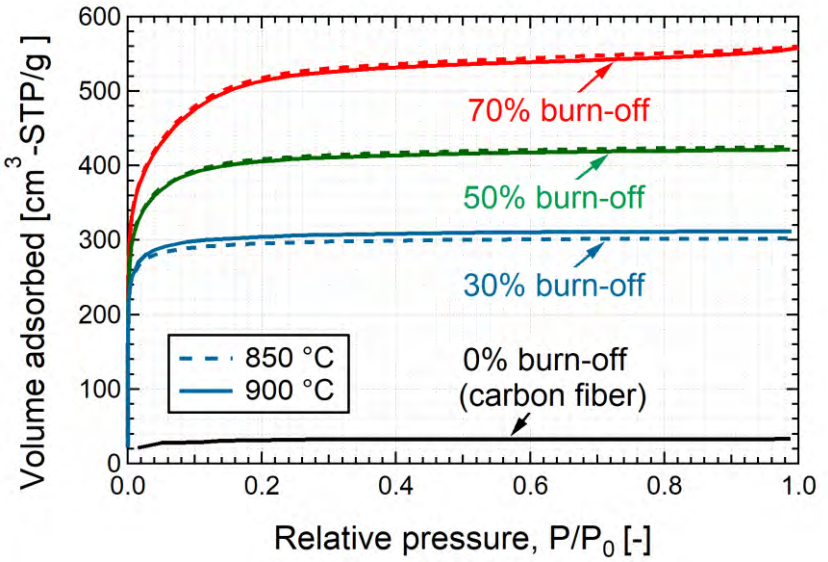


Mae Moh coal carbon fiber: Yield = 33.9% on Soluble basis, Avg. dia. = 12±0.8 µm,
C = 89.3%, H = 0.3%, N = 1.9%, O+S = 8.4%

Production of Activated Carbon Fiber

- Rice straw carbon fibers: 80 mg
- Activation temperature: 850˚ C, 900˚ C
- Steam partial pressure : 0.5
- Burn-off level: 30%, 50%, 70%





BET surface area (ACF850˚ C)

0% burn-off : 114 m^2/g

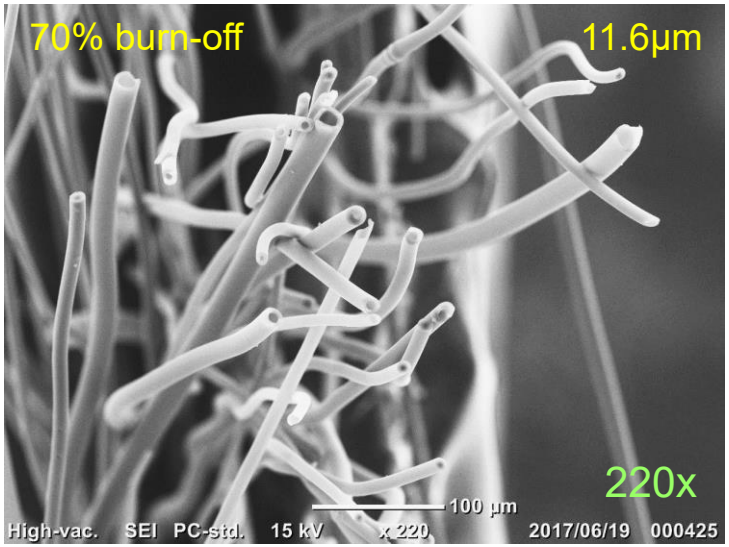
30% burn-off : 1149 m^2/g

50% burn-off : 1540 m^2/g

70% burn-off : 1882 m^2/g

Summaries of Present Works

1. The hollow carbon fiber can be produced by using rice straw Soluble and the formation of hollow could be controlled. The rheological properties of precursor and the spinning conditions are the important parameters effecting the formation of hollow.
2. Preparation of the hollow activated carbon fiber was achieved. The surface areas of activated carbon fibers reach 1900 m^3/g after steam activation at 850 ° C and burn-off level of 70%.



Hollow activated carbon fiber produced from rice straw Soluble

Activities extending the SATREPS project

Human Resources Development (1)

Mr. Trairat Muangthong-on, supported by the Monbu-sho scholarship through JST, got phD degree from Kyoto University on Sep.25, 2017.

Thesis title:

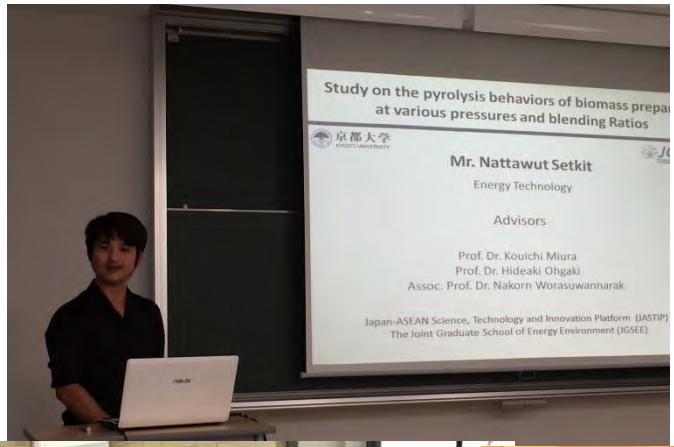
"Effect of Solvent on the Degradative Solvent Extraction of Low Rank Coal and Examination of Propensity to Spontaneous Heating of the Solvent Treated Coal and Residue"
（低品位炭の溶剤改質に対する溶剤種の影響と溶剤改質炭と抽出残渣物の自然発火性に関する研究）

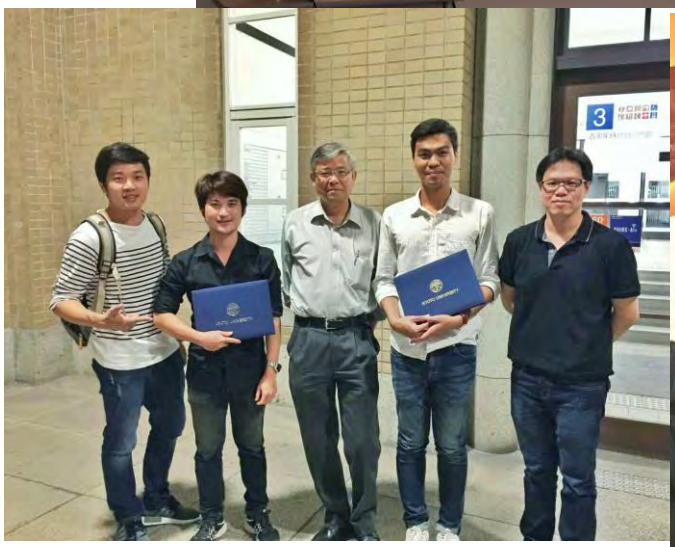
Supervisor: Prof. Hideaki Ohgaki

Publications related to phD:

1. Trairat Muangthong-on, Janewit Wannapeera, Hideaki Ohgaki, Kouichi Miura, TG-DSC Study To Measure Heat of Desorption of Water during theThermal Drying of Coal and To Examine the Role of Adsorption of Water Vapor for Examining Spontaneous Heating of Coal over 100 ° C
   Energy Fuels, DOI: 10.1021/acs.energyfuels.7b01836
2. Trairat Muangthong-on, Janewit Wannapeera, Hideaki Ohgaki, Kouichi Miura, Examination of Interactions of Solvent-Treated Coal with Oxygen and Water Vapor at Over 100 ° C Using TG-DSC for Examining Propensity to Spontaneous Heating of the Solvent-Treated Coal
   Energy Fuels, DOI: 10.1021/acs.energyfuels.7b01906
3. Trairat Muangthong-on, Janewit Wannapeera, Supachai Jadsadajerm, Nakorn Worasuwannarak, Hideaki Ohgaki, Kouichi Miura, Effect of Solvent on the Degradative Solvent Extraction of Low Rank Coal.
   Energy Fuels, DOI: 10.1021/acs.energyfuels.7b02352

Human Resources Development (2)





Mrs. Supachai Jadsadajerm, Setkit Nattawut, and Ruangdet Fugtuan have stayed 1 to 6 months at Kyoto University to perform experimental works.

Human Resources Development (3)

Training of Dr. Xayalak from Laos National University

Kyoto University (Jan. 9 – Feb. 7)

Dr. Xayalak is using a set of thermal analysis equipment

JGSEE (Aug. 1 – 31)

Dr. Xayalak performed analyses of Lao biomass samples using the equipment introduced by the SATREPS project

Expected Outcome/Future of our Project

- Implementation of a new technology for utilizing low rank coals and biomass wastes in Thailand
- Human building in both Japan and Thailand for effective utilization of biomass waste/low rank coal

- JGSESS/KMUTT and PTT-RTI help dissimilation of the technologies developed to ASEAN countries
- JGSESS/KMUTT works as a center of biomass conversion technology development and human resource building in ASEAEN countries

Japanese members will assist the activities through JASTIP (Japan ASEAN Science and Technology Innovation Platform) program .