The 2nd JASTIP-WP2 Annual Workshop
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Extension of Solvent Treatment Method Developed by SATREPS Program to ASEAN Region

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Katsuyasu Sugawara, Professor, Akita University
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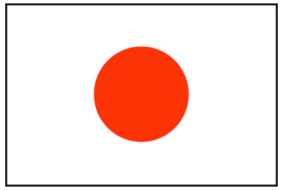
Suneerat Fukuda, Assoc. Professor, JGSEE/KMUTT



Japan-Thailand SATREPS Project

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

Dec. 20, 2013 – Dec. 19, 2018

Kouichi Miura
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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.

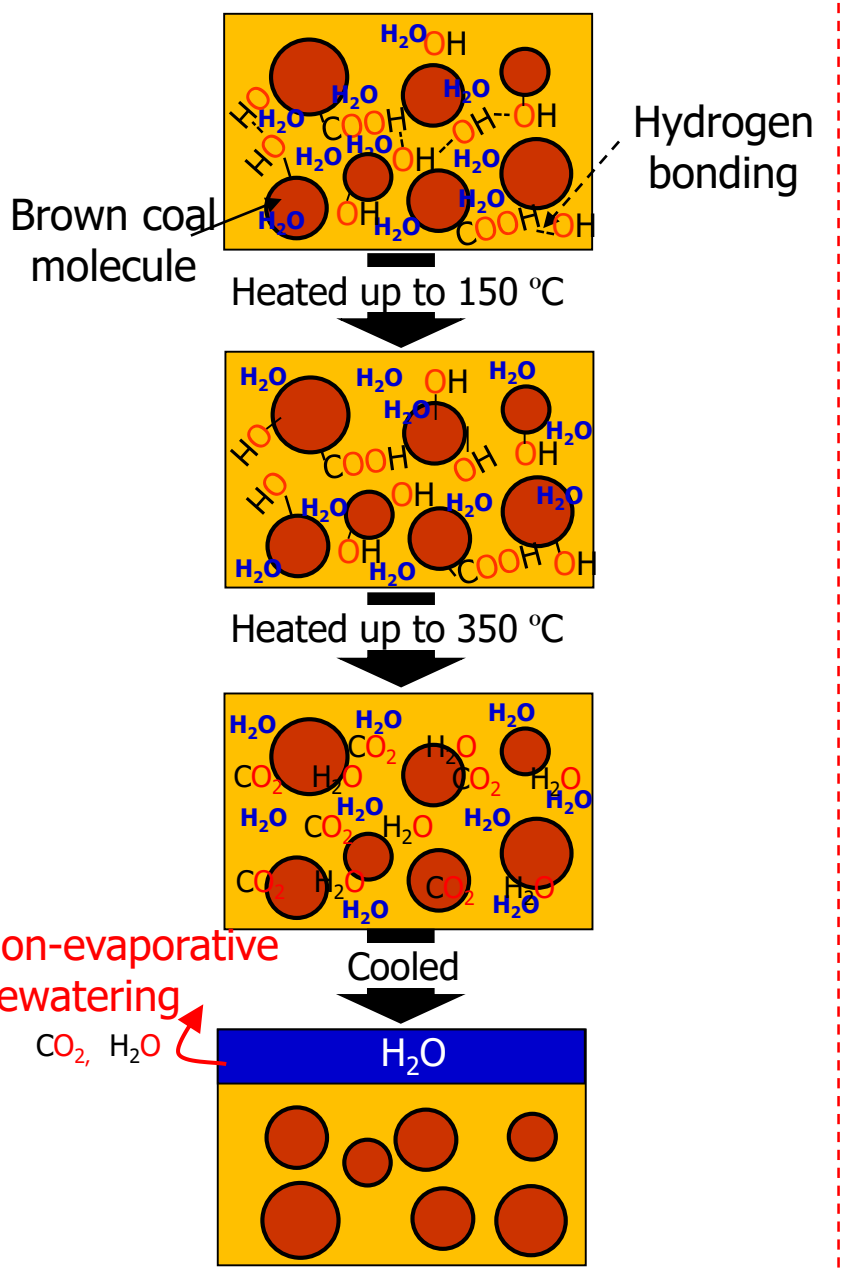
- The technologies developed under cooperative researches will contribute to reduce the emission of global warming gases as well as environmental pollutants.
- The technologies developed will be disseminated to ASEAN countries which need such technologies.

What is the

“Degradative Solvent Extraction”?

Proposed method

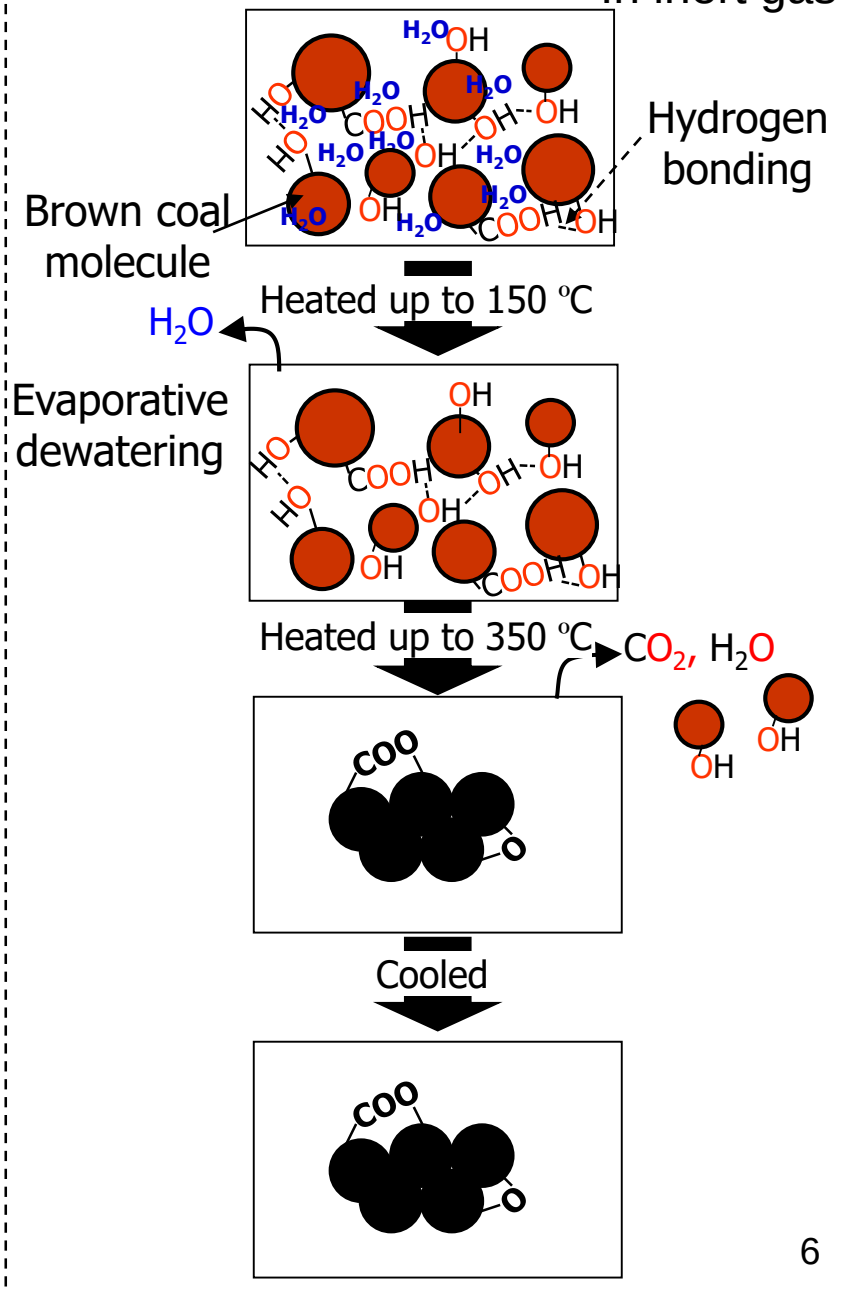
In non-polar solvent



Upgrading without cross-linking reactions.

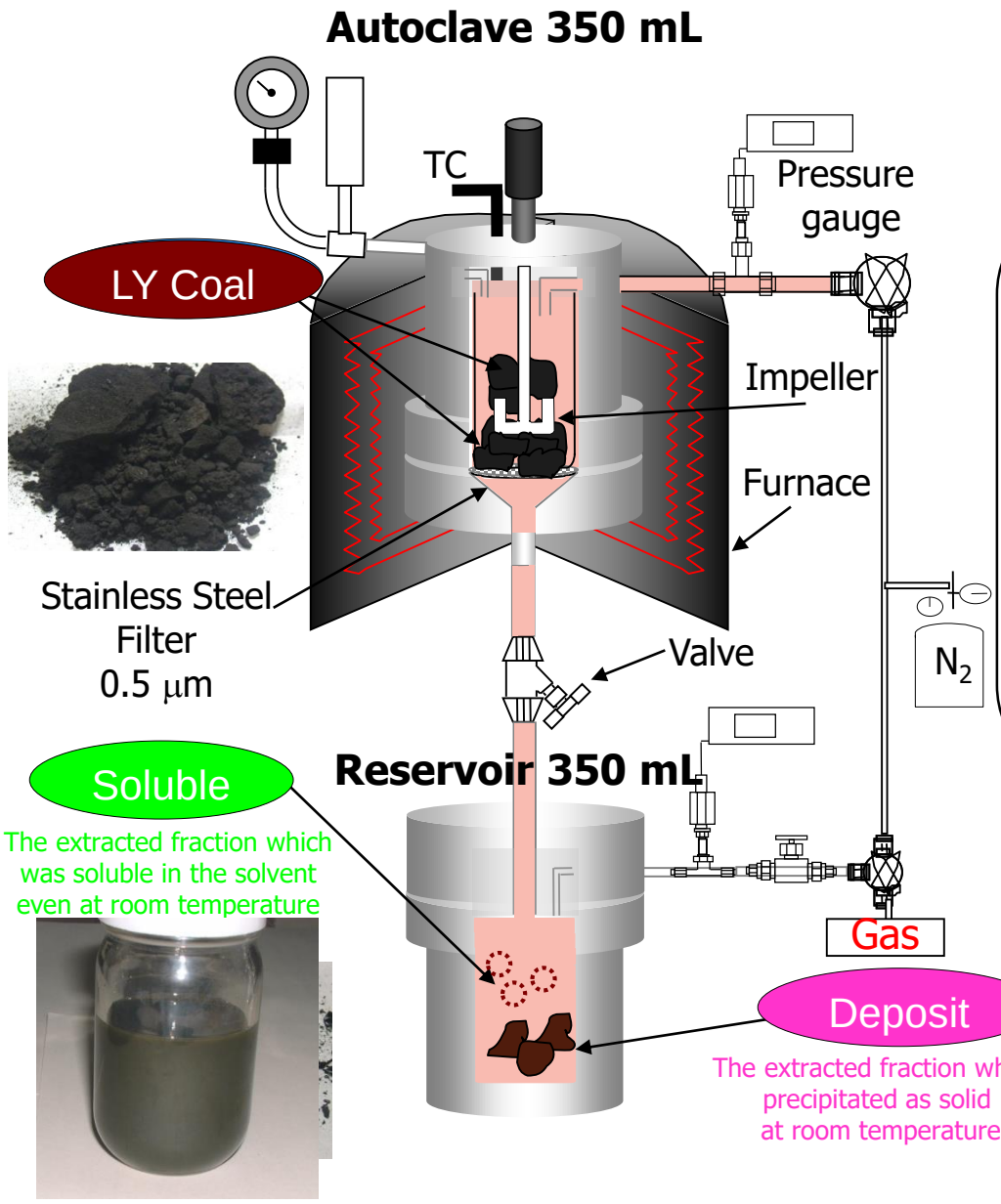
Conventional heat-treatment

In inert gas



Significant enlargement of the coal molecules.

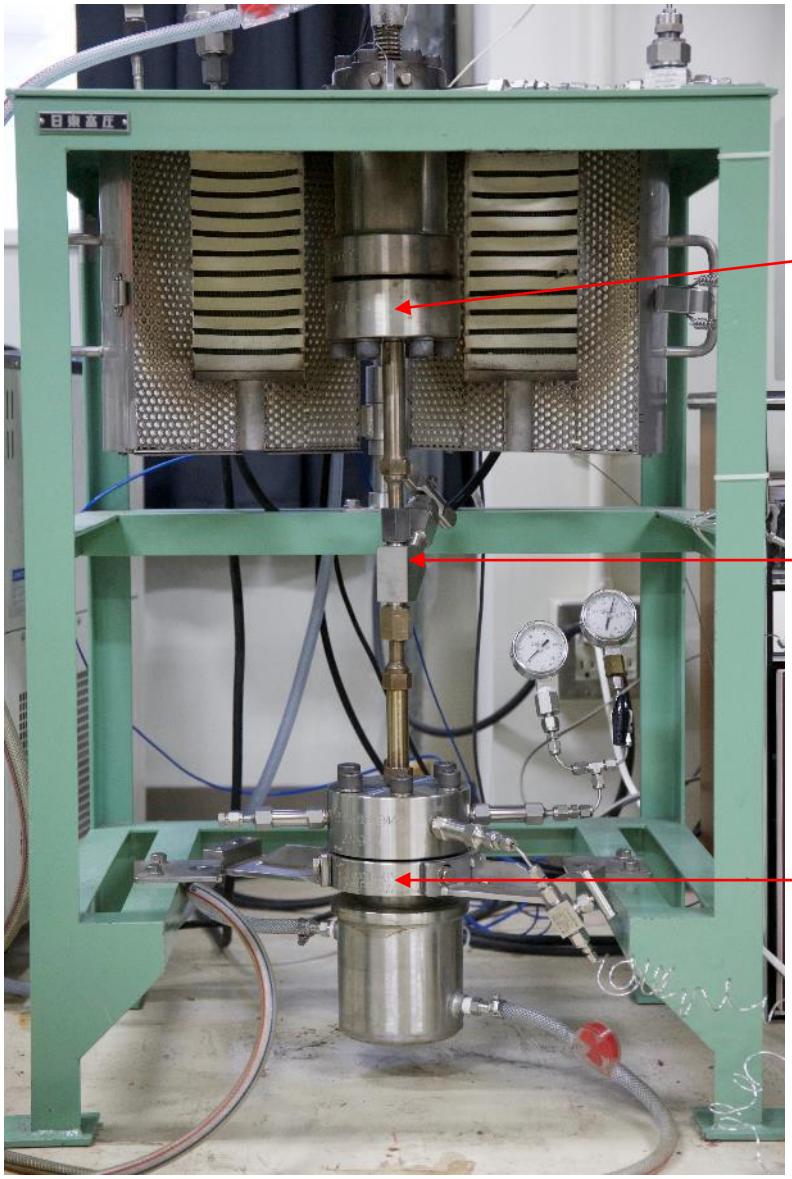
Apparatus and procedure



Experimental conditions

- Coal: 14 g-d.a.f.
  (Charged as received)
- 1-MN : 300 mL
- Temperature : 350°C
- Holding time : 0, 1, 2, 3 h
- Final pressure : 2.3, 6.8, 15.7 MPa

Apparatus used



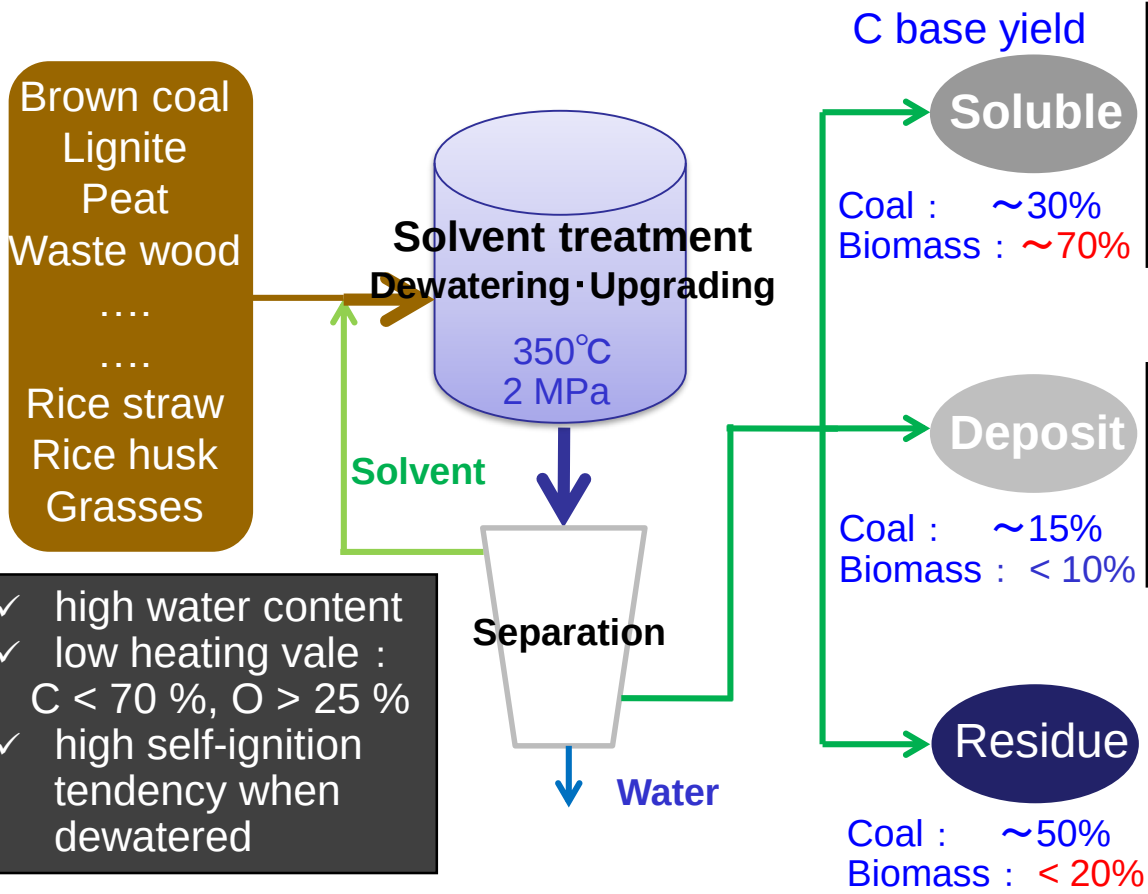
Raw materials used

Brown coal (Loy Yang)

Rice straw

Leucaena

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

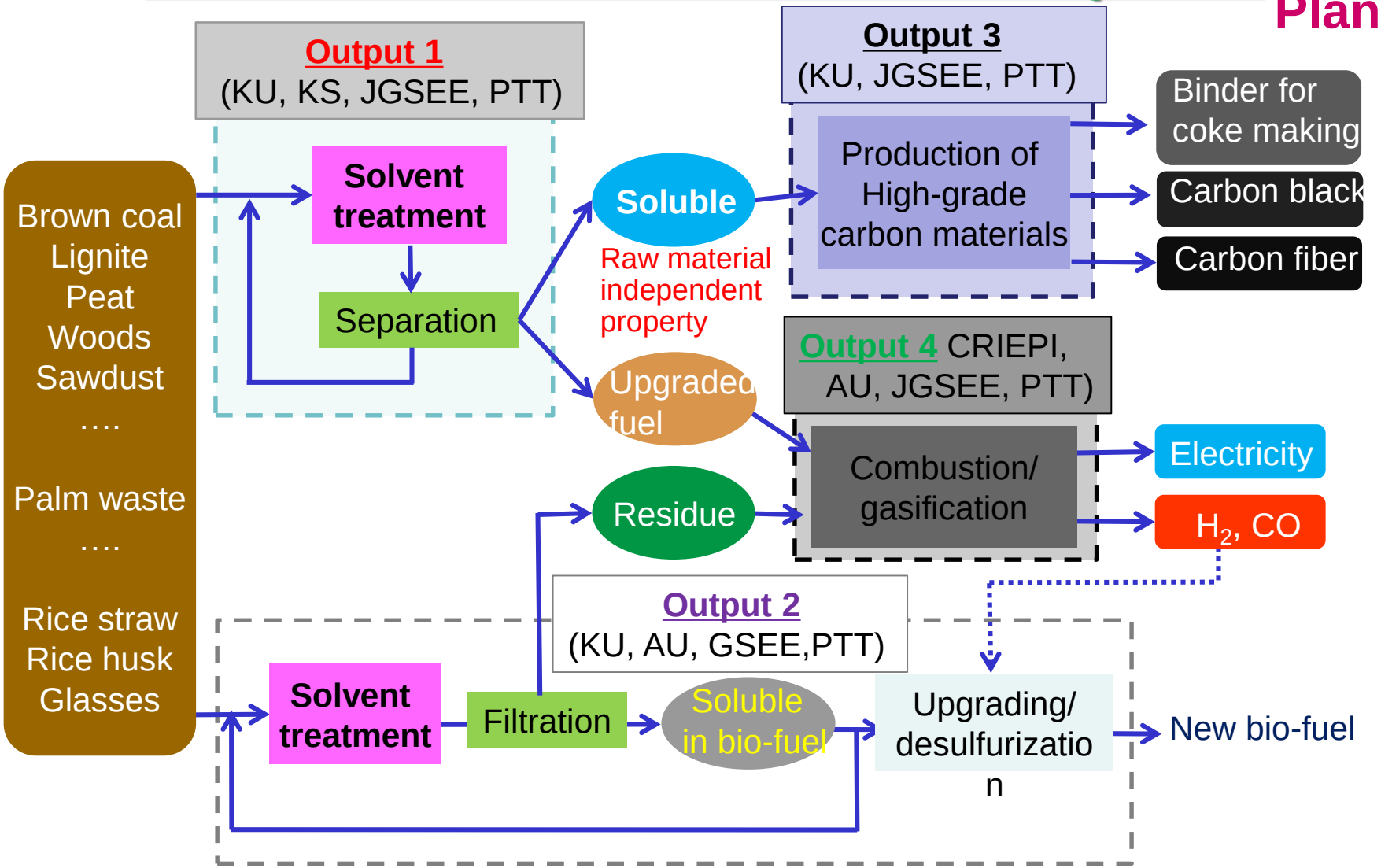
- ✓ high water content
- ✓ low heating vale：
  C < 70 %, O > 25 %
- ✓ high self-ignition tendency when dewatered

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.

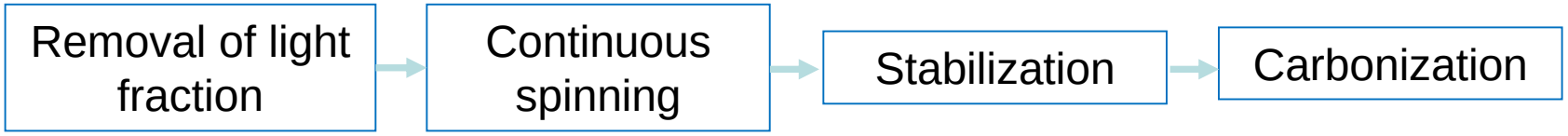
Four research groups from Japan and two research groups from Thailand are involved in this project.

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

Preparation of carbon fiber from Soluble – Task 3 -



-20% of light fraction was removed by heat treatment

-20% of Soluble can be utilized as oil without treatment

Spinning using a mono-hole continuous spinner at -200℃

Oxidation treatment in air at -300℃

Heat treatment at -800℃

Continuous spinning of the modified Soluble

Modified Soluble is heated to 285 °C

Pitch fiber coming out from the mono-hole

Pitch fibers collected

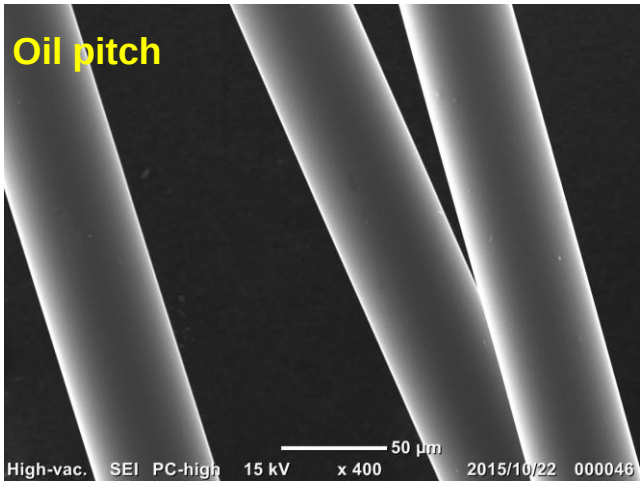
Rotating drum (16 cmϕ)
(rotating at 600 – 1000 rpm)

Fig. Mono-hole spinning machine

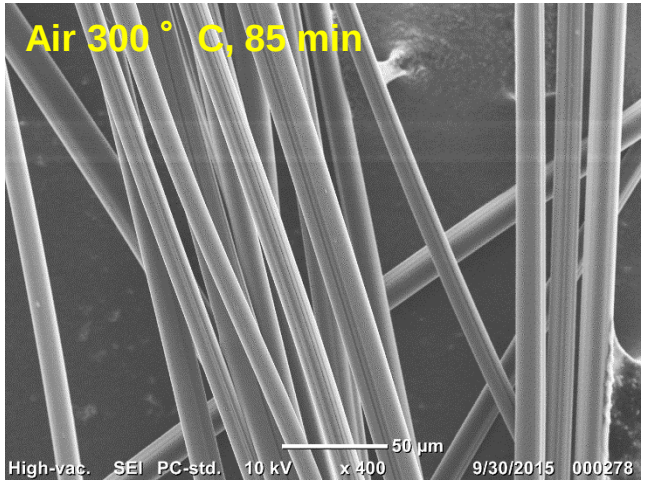
Carbon fibers: J-RS Soluble

- SEM images of carbon fibers (400x)

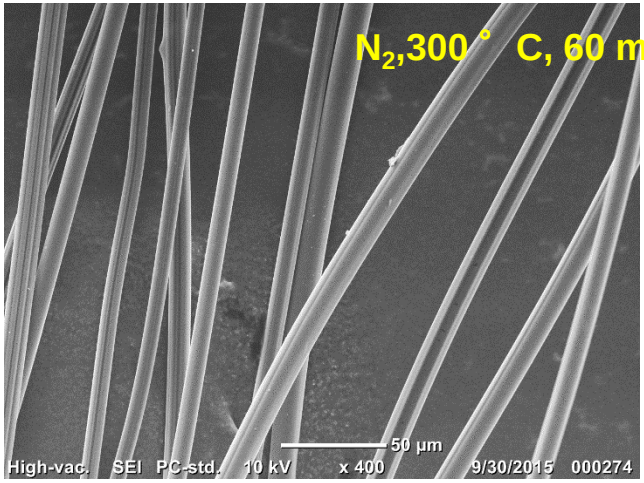
Fibers dia.
51.7-54.7 µm



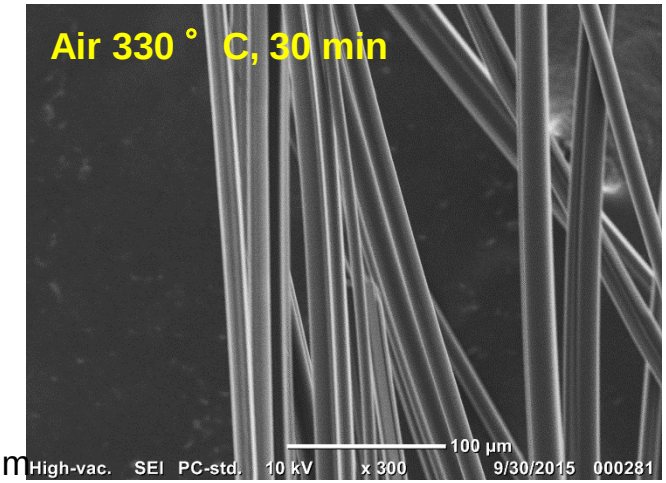
Fibers dia.
11.4-16.7 µm



Fibers dia.
6.6-13.8 µm

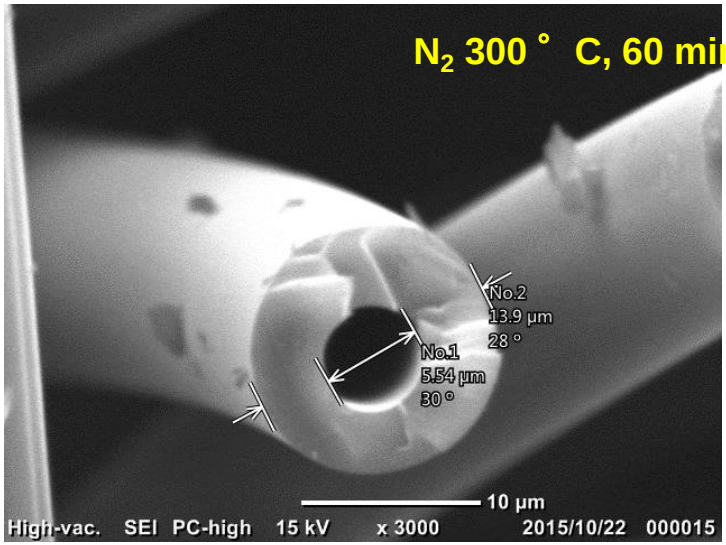


Fibers dia.
12.1-16.8 µm

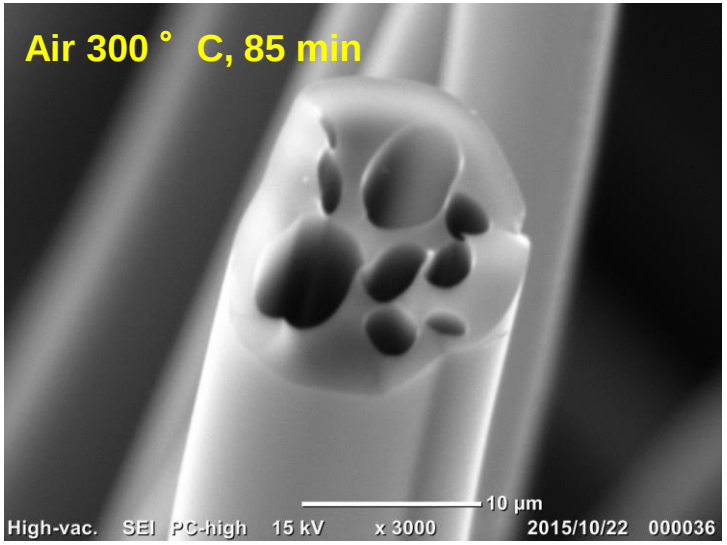


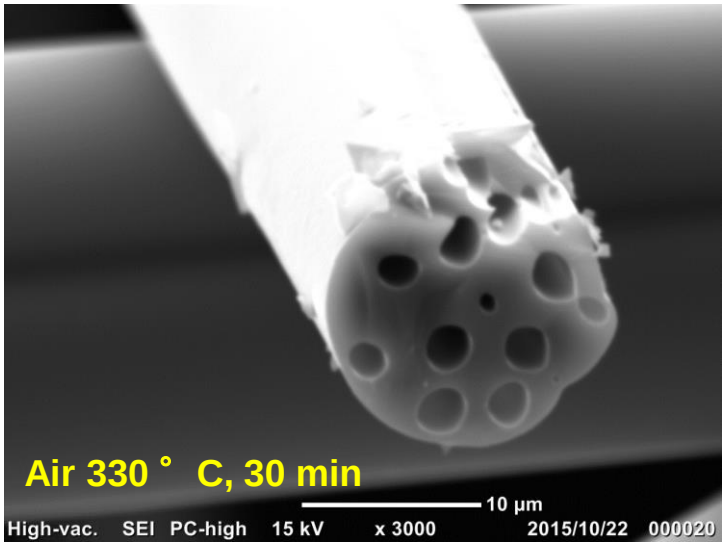
Carbon fibers: J-RS Soluble

- SEM cross-sectional images of carbon fibers (3000x)



  - Only one hollow was observed from the fibers prepared from Soluble treated by the N_2 purge.
  - Several hollows were observed from the fibers prepared from Soluble treated by the air oxidation.



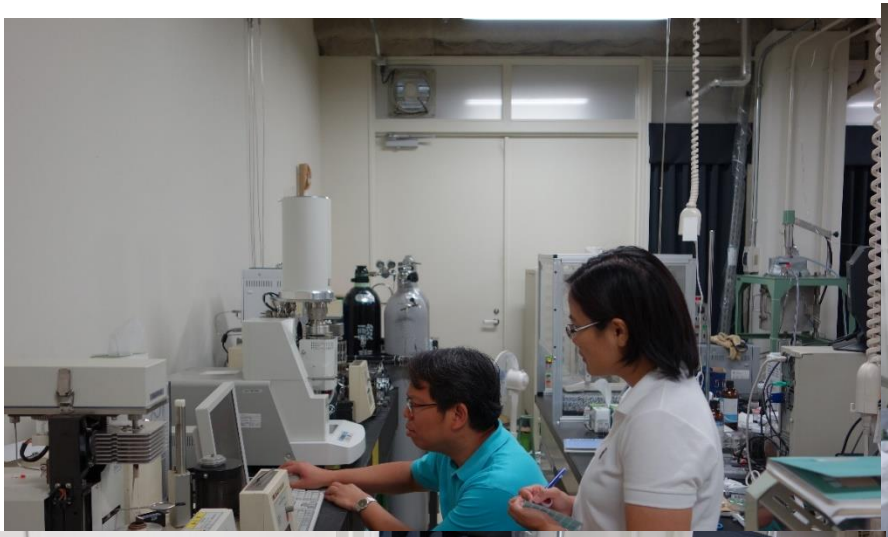


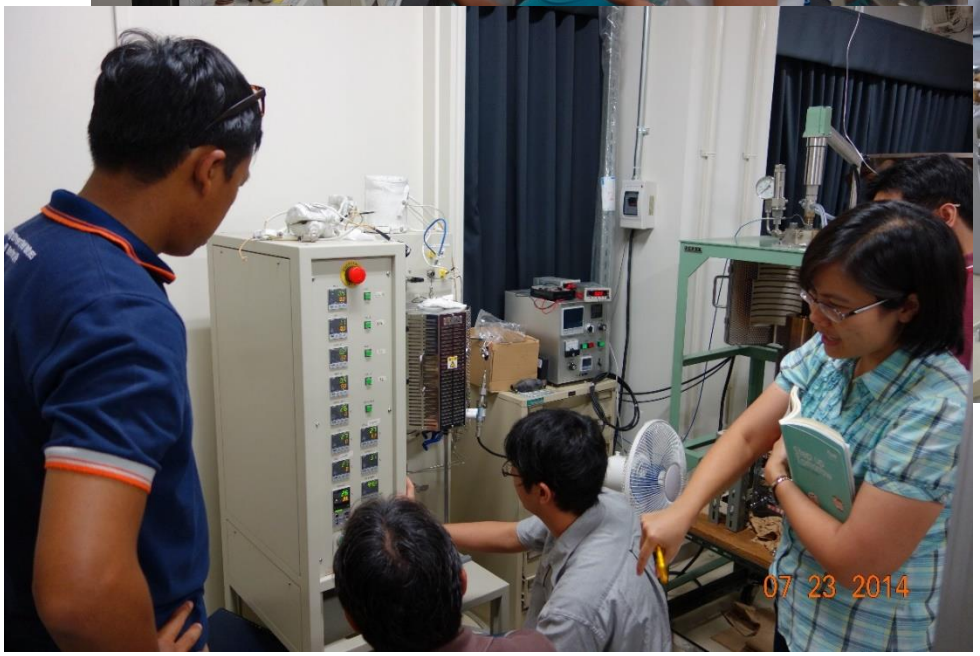
Dispatch of researchers
Acceptance of researchers

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
Total	**96**	**812**	**33**	**592**

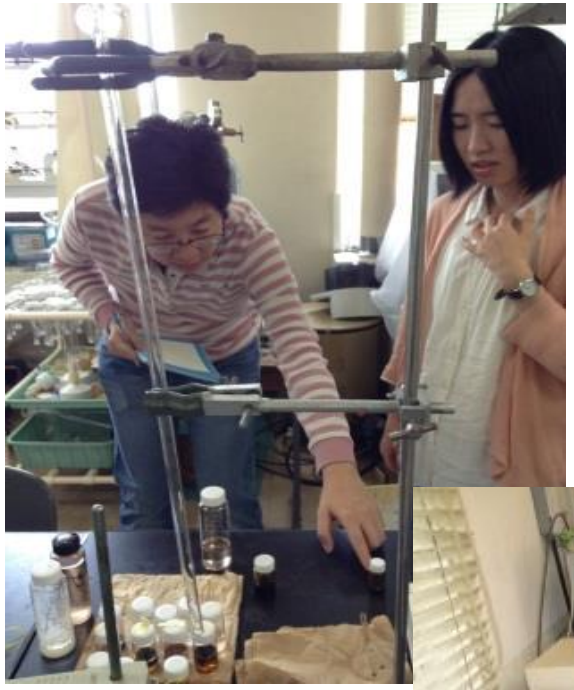
Visit Kyoto University (July. 17 – Aug. 3, 2014)





Training of solvent extraction and carbon fiber preparation

Akita University (June, July, 2014)



Training of solvent desulfurization experiments

Visit CRIEPI

(June. 15-17, 2015)

Training of DTF operation

(Feb. 1, 2015)

Plant tour at Kobe Steel (Aug. 4, 2014)

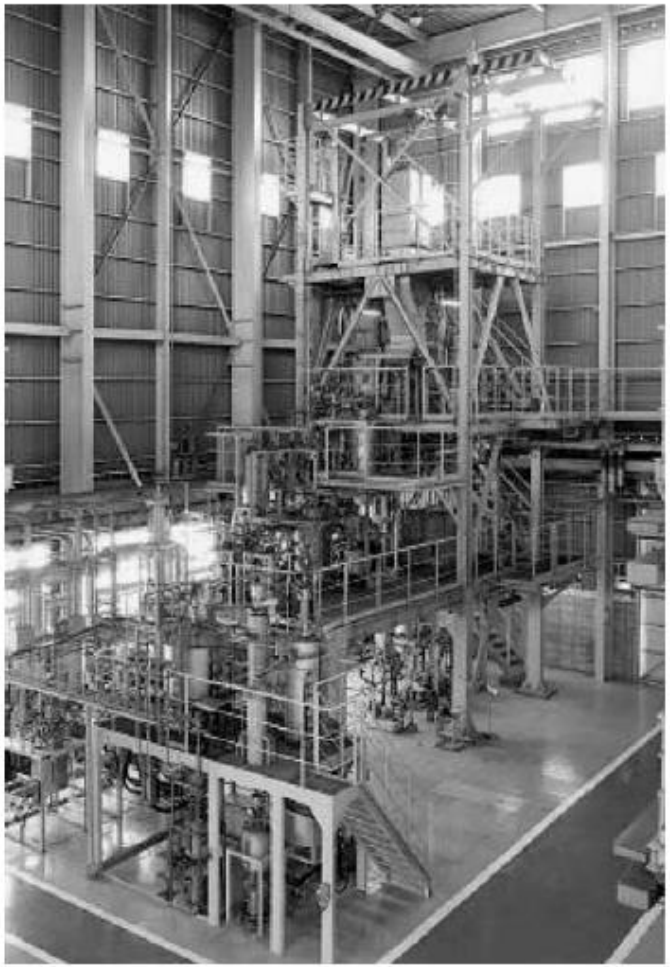


図 3 0.1t/d HPC 連続製造試験装置
Fig. 3 0.1t/d HPC Bench scale unit

The Thai members had a opportunity to see the continuous HPC production facility

Solvent Extraction Plant tour at Kobe Steel (April, 2015)