# 超臨界流体と共に歩んだ名古屋と熊本での38年
# 38 years in Nagoya and Kumamoto along with supercritical fluids


名古屋大学

大学院工学研究科　物質プロセス工学専攻

後藤元信


工学研究科1号館2階121講義室
2022年3月10日14:00-15:00

# Biodata (Motonobu Goto)

1984 Dr. Eng., Nagoya University
1984 Assistant Professor, Nagoya University

1988 Associate Professor, Kumamoto University
1988 California University, Davis（15 months)
2001 Professor, Kumamoto University

2012 Professor, Nagoya University
2013 Super Critical Technology Centre Co. Ltd.

Vice-President of The Society of Chemical Engineers, Japan (2016-7)
Vice-President of The International Society for Advancement of Supercritical Fluids
Associate Editor of The Journal of Supercritical Fluids
President of Research Association For Feedstock Recycling of Plastics Japan (2016-7)

# Event in my researcher life on SCF

Prof. T. Hirose

1988 Nagoya University名大→ Kumamoto University熊大
1988 California University, Davis（15 months)

1993 NATO Advanced Study Institute on Supercritical Fluids（Turkey）
1996 JSPS Research for the Future Program未来開拓学術研究推進事業 【1996-2000】荒井康彦
1997 MESC Scientific Research on Priority Areas重点領域研究【1997-2000】超臨界
2003 21 century COE Program (Kumamoto Univ)【2003-2007】
2006 8th International Symposium on Supercritical Fluids (Kyoto) Organizer
2008 Global COE Program (Kumamoto Univ)【2008-2012】
2009 MEXT Scientific Research on Innovative Areas新学術領域研究【2009-2013】プラズマ

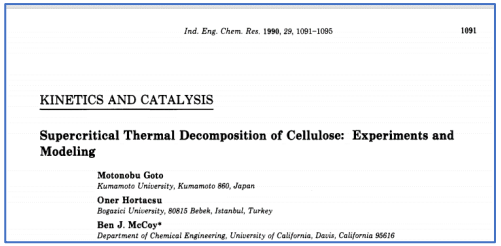
2012 Kumamoto University 熊大→ Nagoya University名大
2013 Workshop on Supercritical fluids and Energy in Brazil
2013 Super Critical Technology Centre Co. Ltd. 超臨界技術センター㈱ 設立 名大発ベンチャー
2017 Supergreen (10th International Conference on Supercritical Fluids) Nagoya

# University of California, Davis 1988.10-1989.12

NAGOYA UNIVERSITY

Benjamin J. McCoy J. M. Smith


## Supercritical fluid Mathematical Modeling

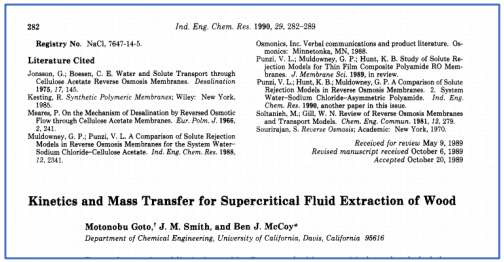


Ind. Eng. Chem. Res. 1990, 29, 1091–1095 1091

KINETICS AND CATALYSIS

**Supercritical Thermal Decomposition of Cellulose: Experiments and Modeling**


**Motonobu Goto**
*Kumamoto University, Kumamoto 860, Japan*
**Oner Hortacsu**
*Bogazici University, 80815 Bebek, Istanbul, Turkey*
**Ben J. McCoy***
*Department of Chemical Engineering, University of California, Davis, California 95616*

282 Ind. Eng. Chem. Res. 1990, 29, 282–289

Registry No. NaCl, 7647-14-5.

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Jonsson, G.; Bossen, C. E. Water and Solute Transport through Cellulose Acetate Reverse Osmosis Membranes. *Desalination* **1978**, *17*, 145.
Kesting, R. *Synthetic Polymeric Membranes*; Wiley: New York, 1985.
Meares, P. On the Mechanism of Desalination by Reversed Osmotic Flow through Cellulose Acetate Membranes. *Bur. Polm. J.* **1966**, *2*, 241.
Muldowney, G. P.; Punni, V. L. A Comparison of Solute Rejection Models in Reverse Osmosis Membranes for the System Water–Sodium Chloride–Cellulose Acetate. *Ind. Eng. Chem. Res.* **1988**, *12*, 2341.
Osmonics, Inc. Verbal communications and product literature. Osmonics: Minnetonka, MN, 1988.
Punni, V. L.; Muldowney, G. P.; Hunt, K. B. Study of Solute Rejection Models for Thin Film Composite Polyamide RO Membranes. *J. Membrane Sci.* **1989**, in review.
Punni, V. L.; Hunt, K. B.; Muldowney, G. P. A Comparison of Solute Rejection Models in Reverse Osmosis Membranes. 2. System Water–Sodium Chloride–Asymmetric Polyamide. *Ind. Eng. Chem. Res.* **1990**, another paper in this issue.
Soltanieh, M.; Gill, W. N. Review of Reverse Osmosis Membranes and Transport Models. *Chem. Eng. Commun.* **1981**, *12*, 279.
Sourirajan, S. *Reverse Osmosis*; Academic: New York, 1970.



*Received for review* May 9, 1989
*Revised manuscript received* October 6, 1989
*Accepted* October 20, 1989


**Kinetics and Mass Transfer for Supercritical Fluid Extraction of Wood**


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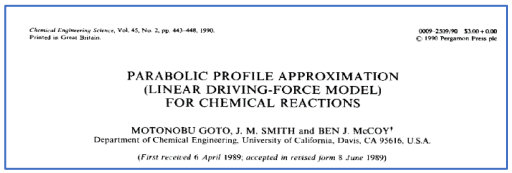


*Chemical Engineering Science*, Vol. 45, No. 2, pp. 443–448, 1990. Printed in Great Britain. 0009-2509/90 $3.00 + 0.00



PARABOLIC PROFILE APPROXIMATION
(LINEAR DRIVING-FORCE MODEL)
FOR CHEMICAL REACTIONS


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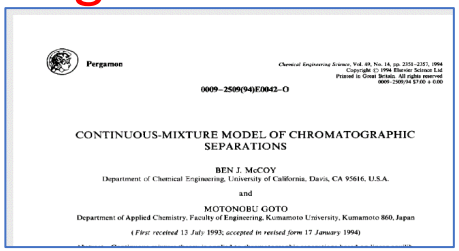
(*First received* 6 *April* 1989; *accepted in revised form* 8 *June* 1989)

Pergamon


*Chemical Engineering Science*, Vol. 49, No. 14, pp. 2355–2357, 1994



0009–2509/94 $7.00 + 0.00
0009–2509(94)E0042–O


CONTINUOUS-MIXTURE MODEL OF CHROMATOGRAPHIC SEPARATIONS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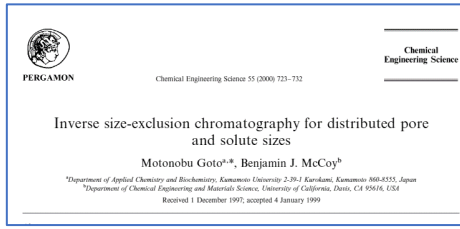
(*First received* 13 *July* 1993; *accepted in revised form* 17 *January* 1994)

PERGAMON


Chemical Engineering Science 55 (2000) 723–732

Chemical Engineering Science


Inverse size-exclusion chromatography for distributed pore and solute sizes


Motonobu Goto[a,*], Benjamin J. McCoy[b]
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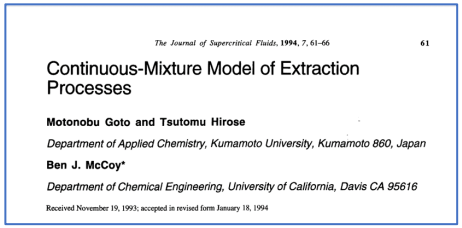
Received 1 December 1997; accepted 4 January 1999


*The Journal of Supercritical Fluids*, **1994**, *7*, 61–66 61


Continuous-Mixture Model of Extraction Processes


**Motonobu Goto and Tsutomu Hirose**
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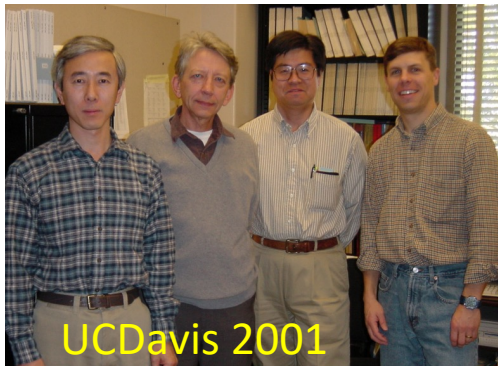
Received November 19, 1993; accepted in revised form January 18, 1994



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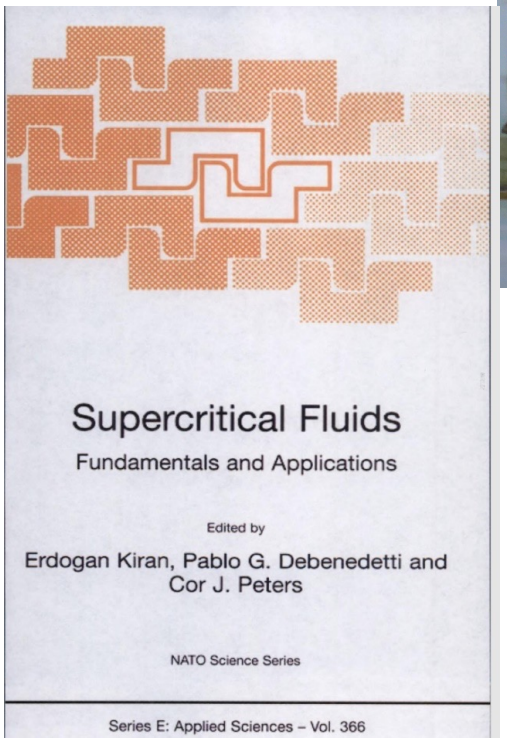
# NATO Advanced Study Institute on Supercritical Fluids

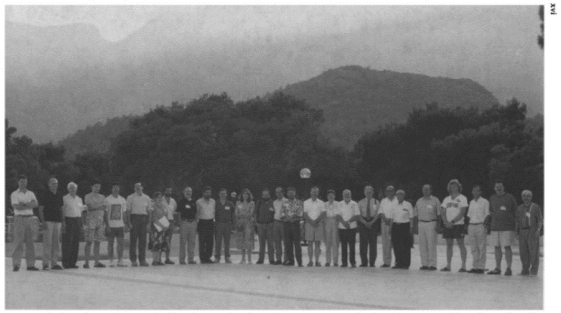
## Kemer, Antalya, Turkey, 1993.7.18-31

Erdogan Kiran




Feral Temelli

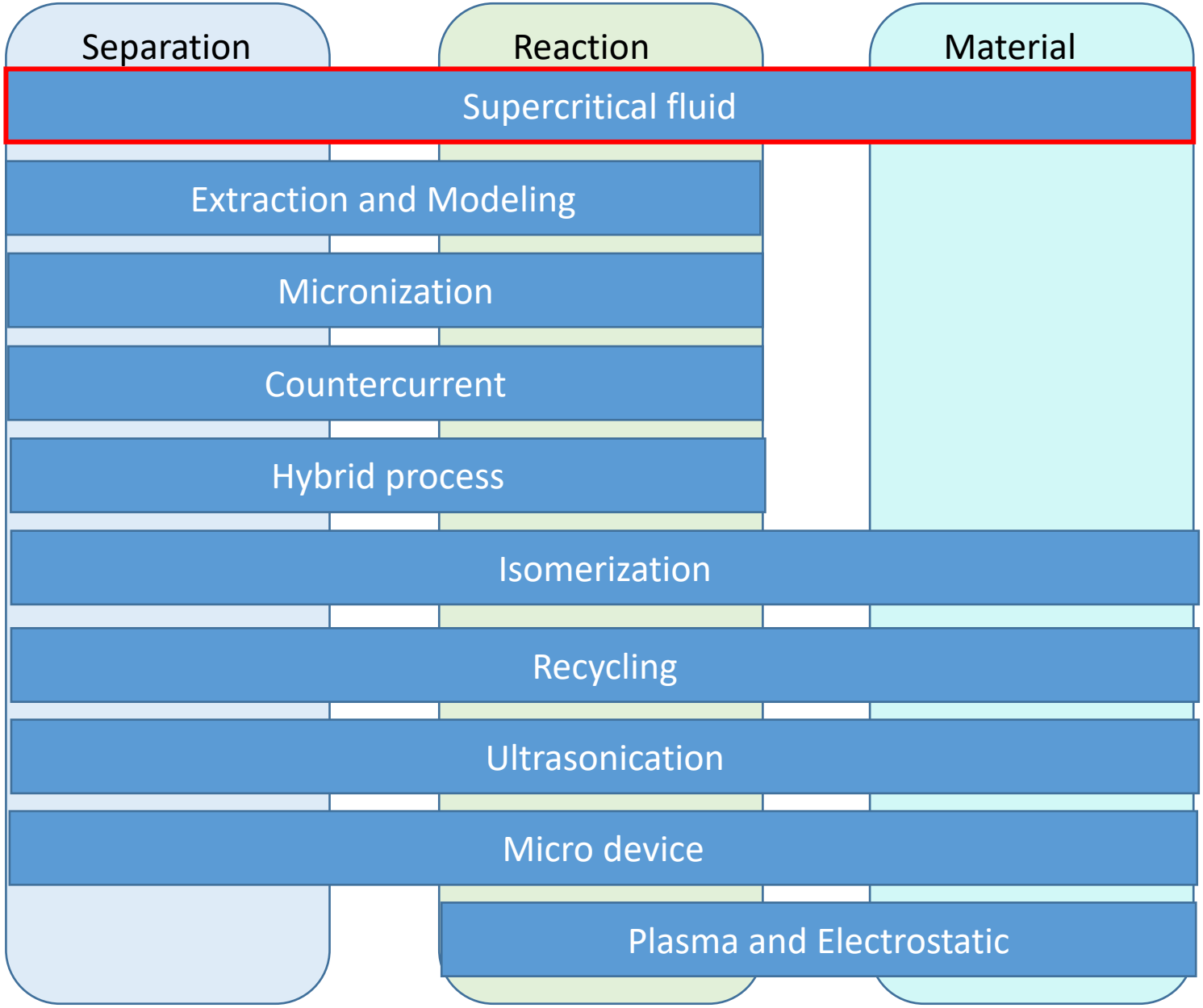
Stephane Sarrade



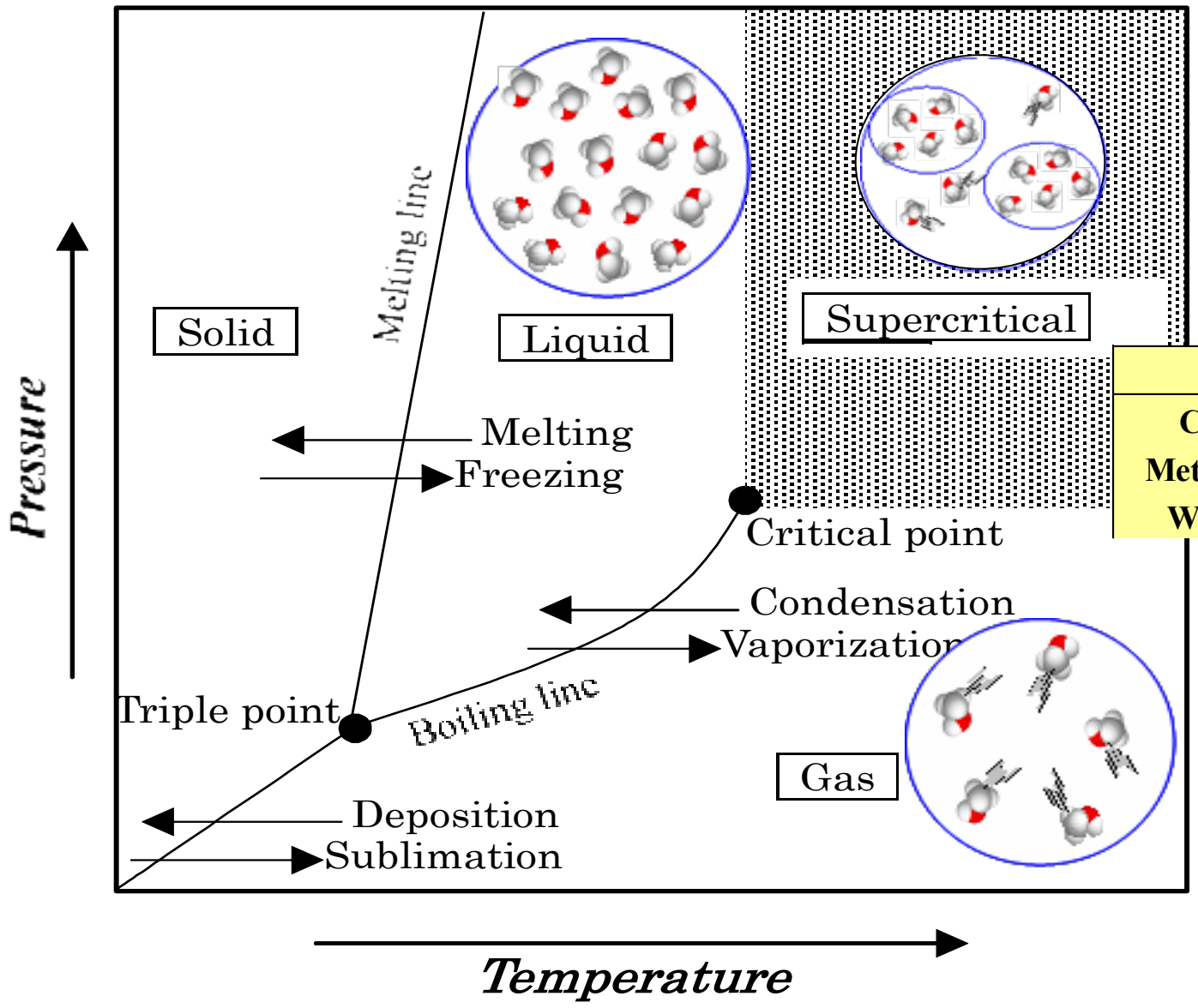
**Organizing Committee Members and the Contributors**

*From left-to-right:* de Loos, Clifford, Heidemann, Meroni, Vesovic, Given, Brand, Lomba, Sandler, Panagiotopoulos, Page, Kruse, Debenedetti, Peters, Kleintjens, O'Connell, Levelt Sengers, Sengers, Schneider, Brunner, Peter, Buback, Howdle, Orbey, Cummings, and Kiran. (Missing from the picture are Akgerman and Lee).

"Supercritical Fluids Fundamental and Applications",
Erdogan Kiran, Pablo G. Debenedetti, Cor J. Peters,
NATO Science Series(NSSE) volume366
Springer, 2000

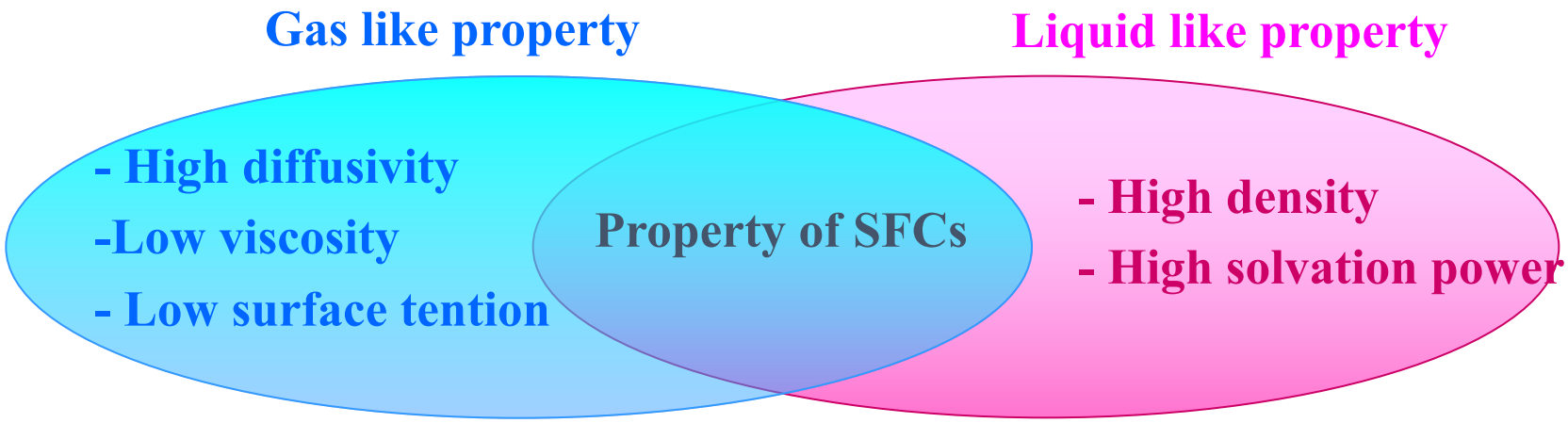

Separation
Reaction
Material
Supercritical fluid
Extraction and Modeling
Micronization
Countercurrent
Hybrid process
Isomerization
Recycling
Ultrasonication
Micro device
Plasma and Electrostatic

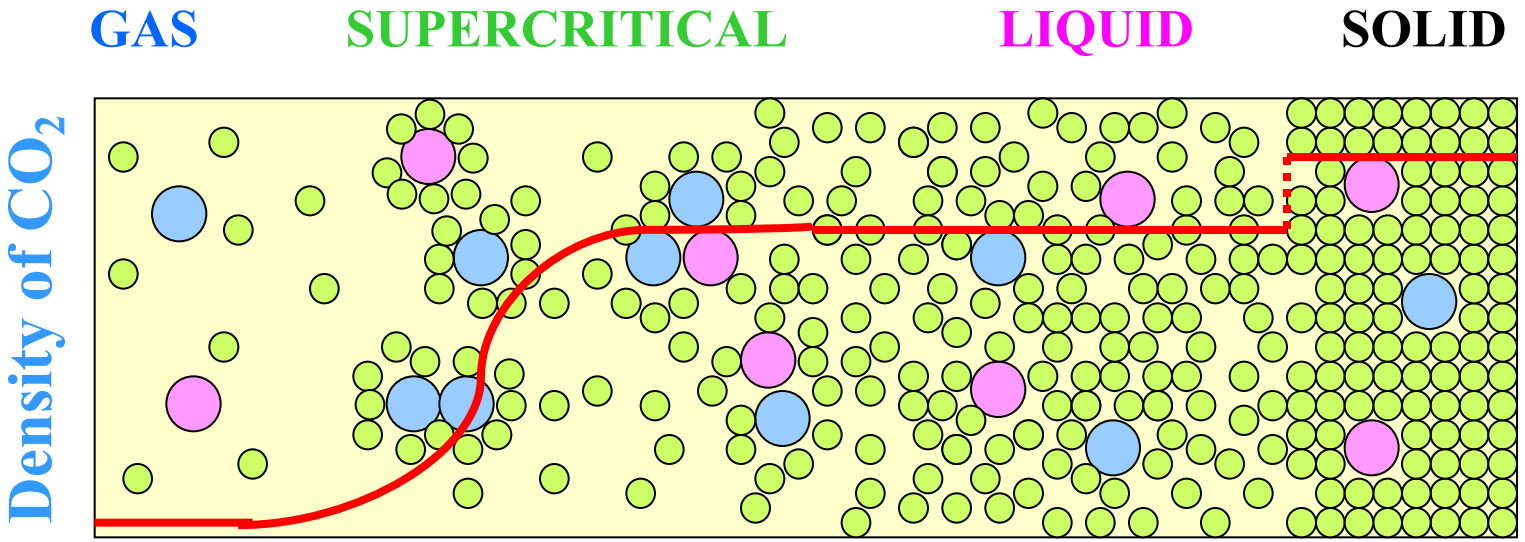
# Supercritical Fluid




| | Tc [K] | Pc [MPa] |
|---|---|---|
| CO2 | 304.15 | 7.38 |
| Methanol | 512.6 | 8.09 |
| Water | 647.3 | 22.12 |

# Supercritical Fluid



# Extraction

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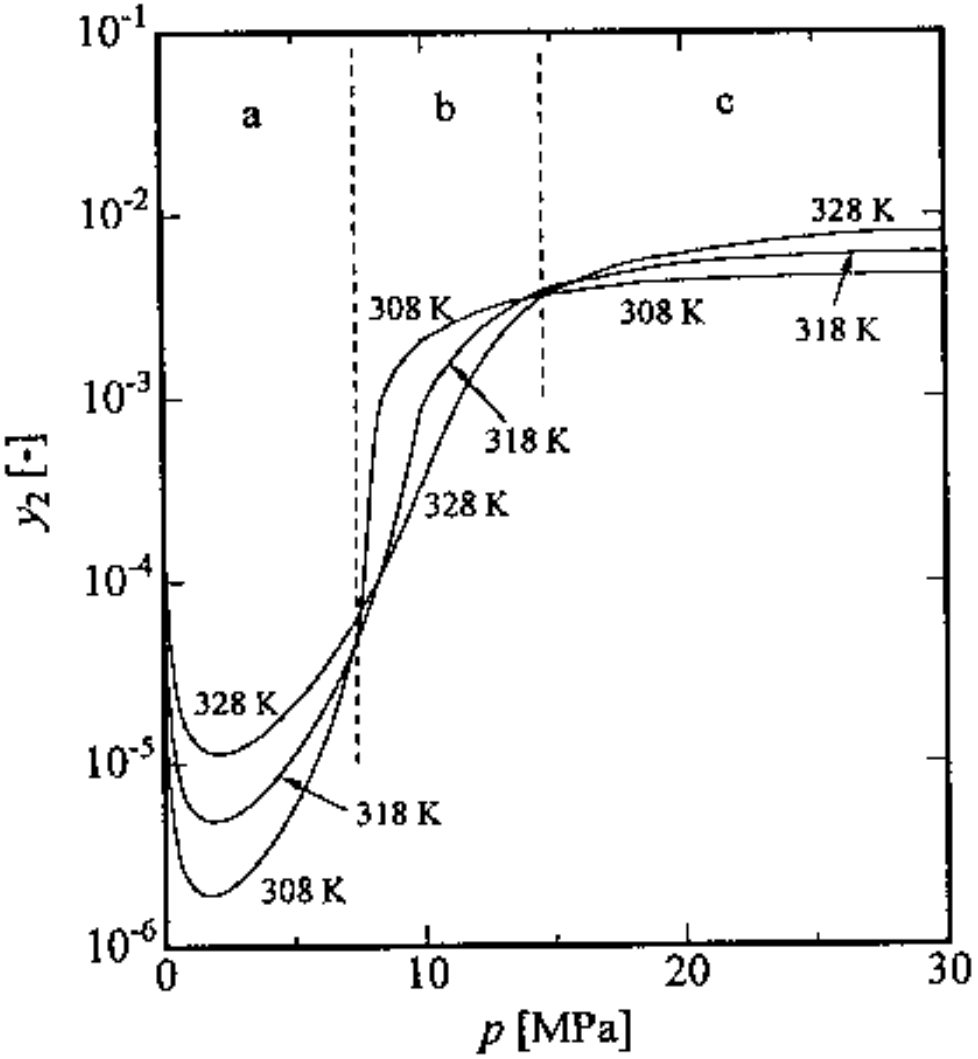
## Solubility of 2,3-dimethylnaphthalene in carbon dioxide




Density of solvent

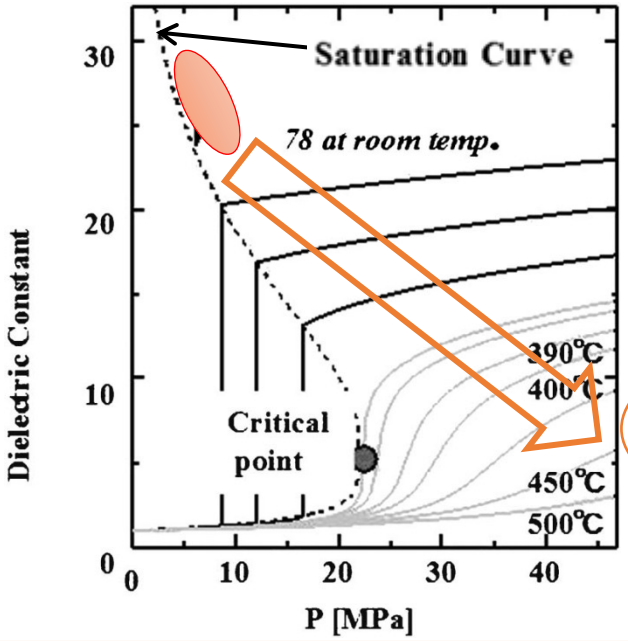
Vapor pressure

Solubility

# Supercritical Fluid

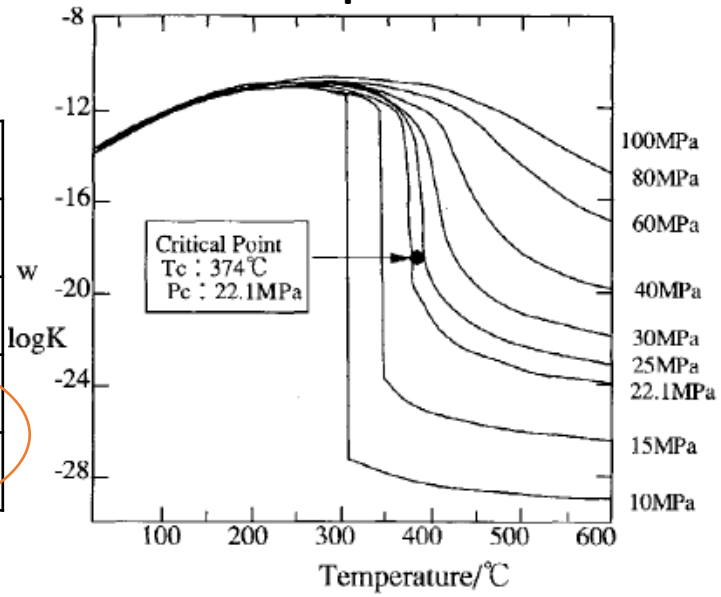
## Property of water

**Dielectric constant**




| | |
|---|---|
| hexisane | 1.8(20℃) |
| benzene | 2.28(20℃) |
| acetone | 20.70(25℃) |
| ethanol | 24.55(25℃) |
| methanol | 32.63(25℃) |

**Ion product**




High dissolution power comparable to organic solvent

Hydrolysis proceed w/o catalyst due to the strong acidic and basic property

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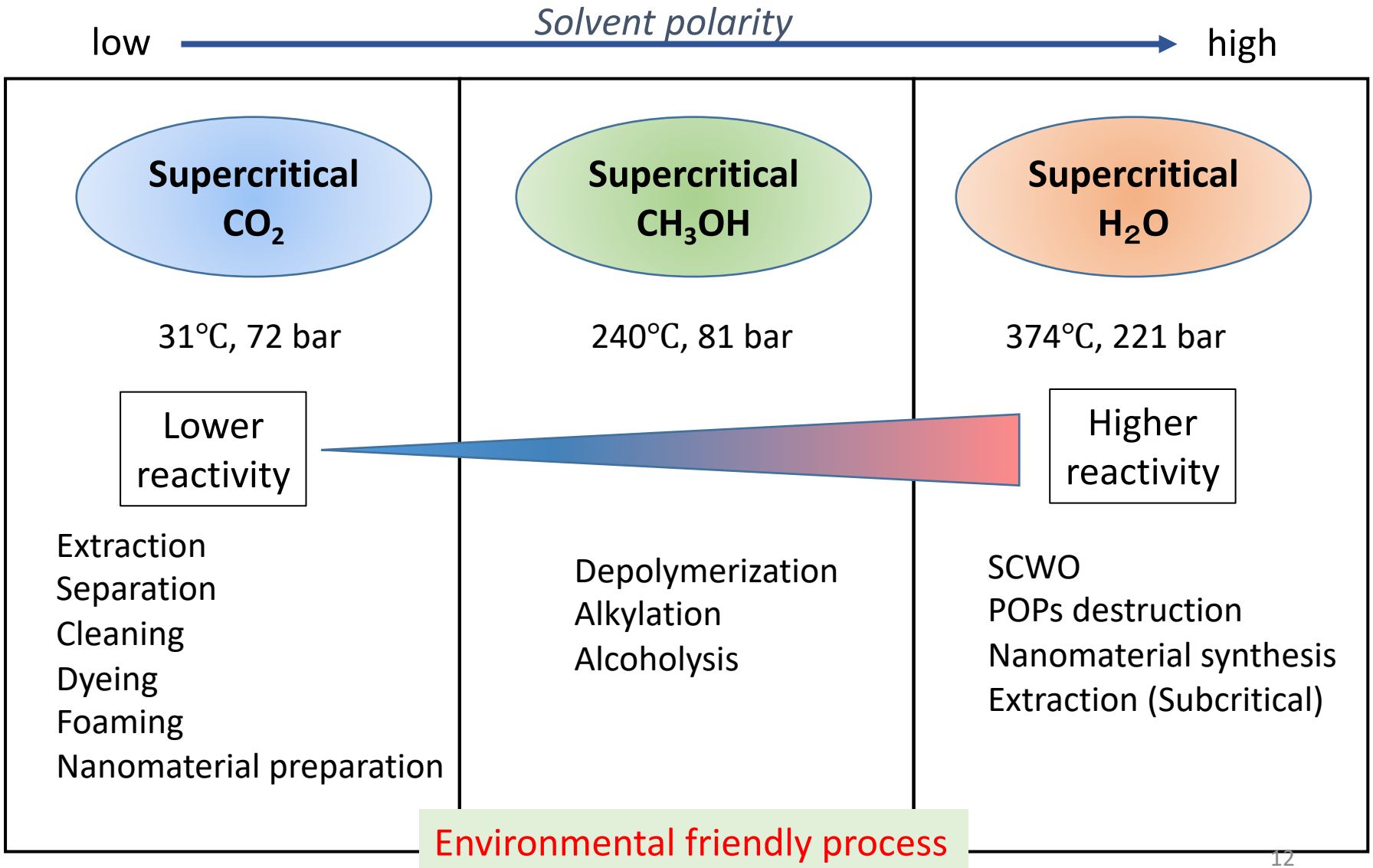
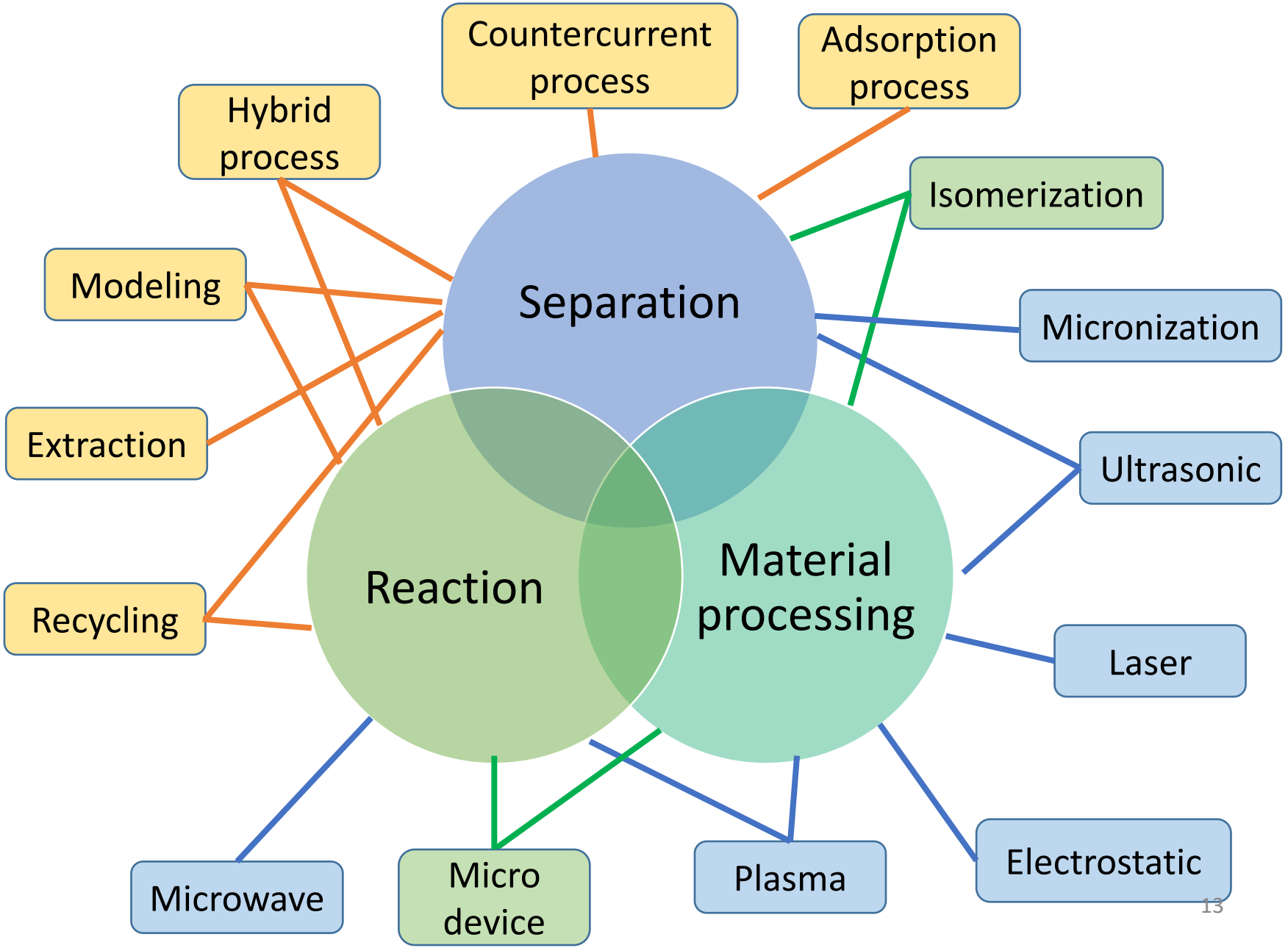
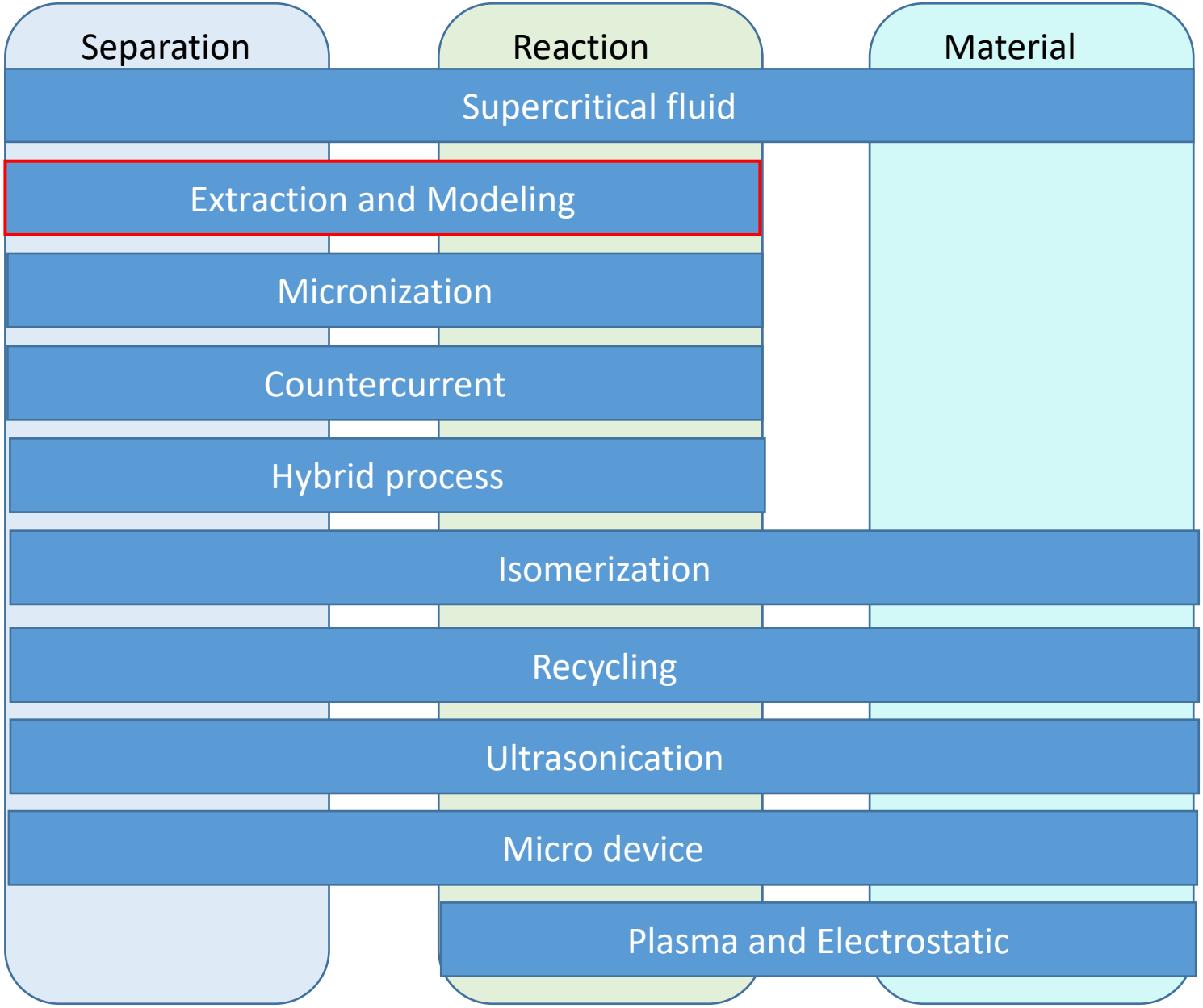
# Typical Application of SCFs

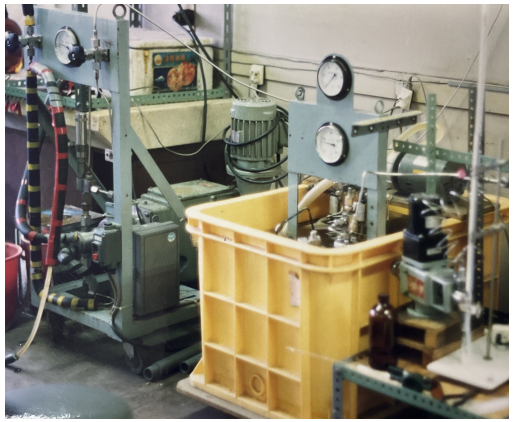


# Supercritical Fluid




Separation
Reaction
Material
Supercritical fluid
Extraction and Modeling
Micronization
Countercurrent
Hybrid process
Isomerization
Recycling
Ultrasonication
Micro device
Plasma and Electrostatic

# Start-up SCF laboratory at Kumamoto University (1990)

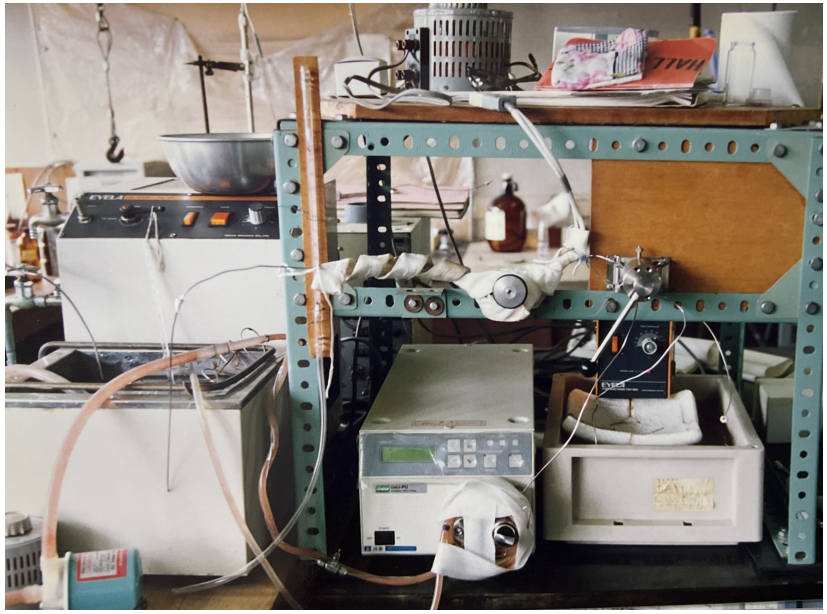


Prof. T. Kobayashi

Prof. T. Hirose

Extraction apparatus from Prof. Kobayashi

1998



Supercritical CO2 extraction apparatus

HPLC

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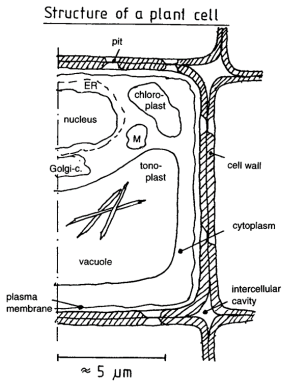
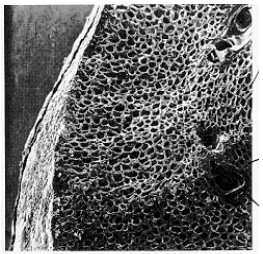
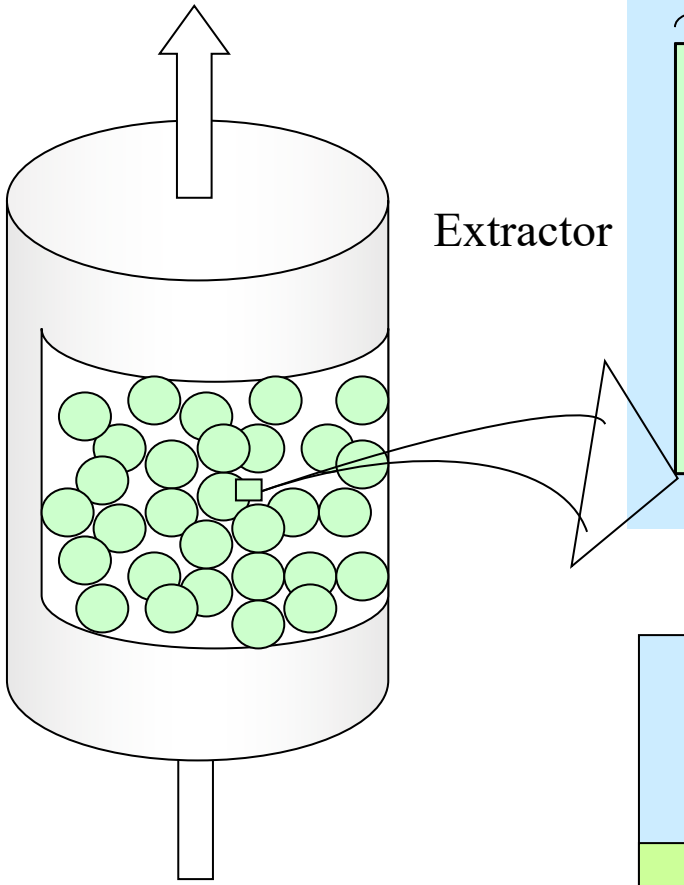
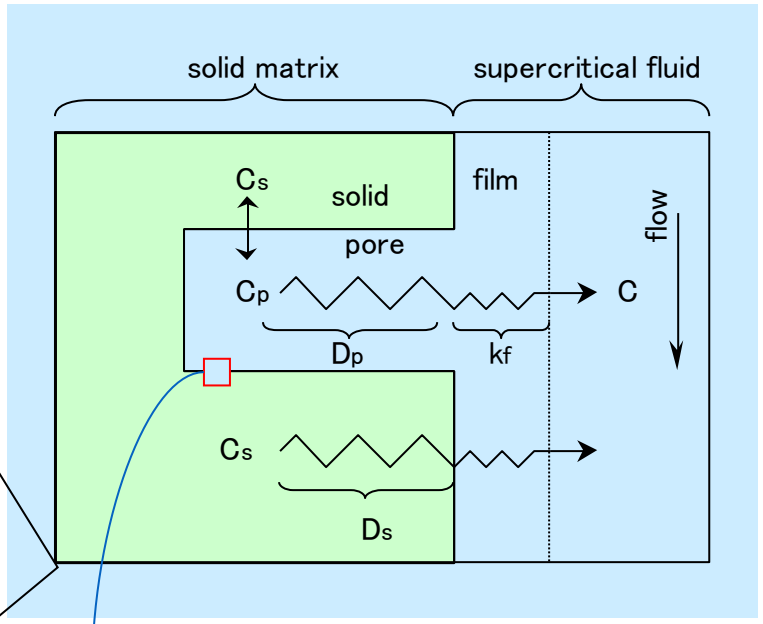
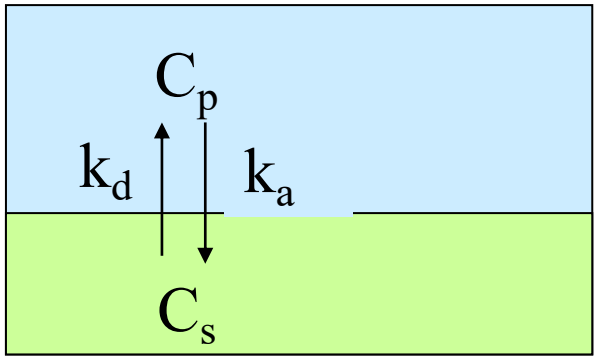
# Extraction modeling





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# Extraction models

- Diffusion model
  - M. Goto, M. Sato and T. Hirose, "Extraction of Peppermint Oil by Supercritical Carbon Dioxide", J. Chem. Eng. Japan, 26, 4, 401-407, 1993
- Shrinking-core model
  - M. Goto, B. C. Roy, and T. Hirose, "Shrinking-Core Leaching Model for Supercritical Fluid Extraction", J. Supercritical Fluids, 9, 2, 128-133, 1996
- Heterogeneous model
  - B. C. Roy, M. Goto, T. Hirose, O. Navaro and O. Hortacsu, "Extraction Rates of Oil from Tomato Seeds with Supercritical Carbon Dioxide", J. Chem. Eng. Japan, 27, 6, 769-773, 1994
- Solute-solid interaction model
  - M. Goto, B. C. Roy, A. Kodama, and T. Hirose, "Modeling Supercritical Fluid Extraction Process Involving Solute-Solid Interaction", J. Chem. Eng. Japan, 31, 2, 171-177, 1998
- Solute-solute interaction model
  - N. T. Dunford, M. Goto, and F. Temelli, "Modeling of Oil Extraction with Supercritical CO2 from Atlantic Mackerel (scomber scombrus) at Different Moisture Contents", J. Supercritical Fluids, 13, 13, 303-309, 1998
- Continuous-mixture model
  - M. Goto, T. Hirose, and B. J. McCoy, "Continuous-Mixture Model of Extraction Processes", J. Supercritical Fluids, 7, 61-66, 1994

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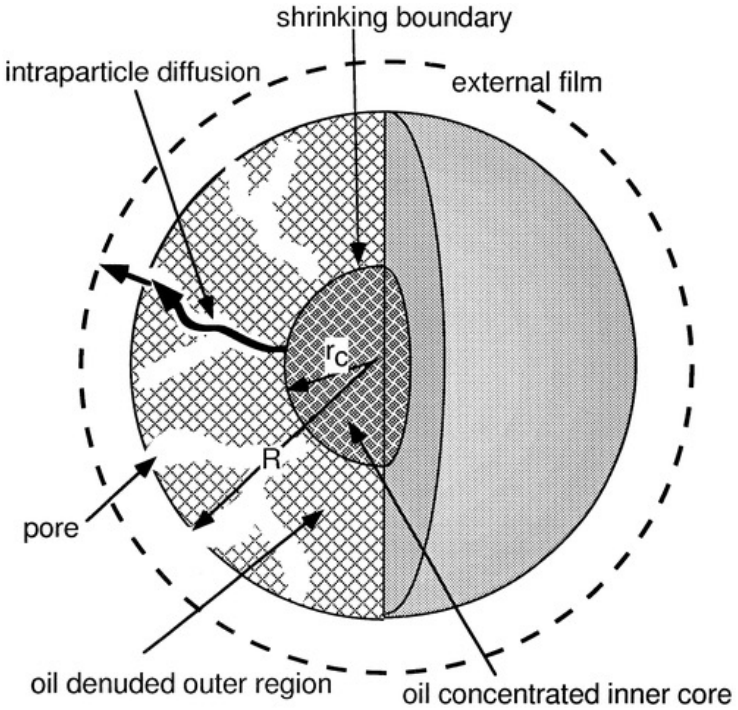
# Shrinking-core leaching model




$$\frac{\partial x}{\partial \theta} + a\frac{\partial x}{\partial Z} = \frac{a}{Pe}\frac{\partial^2 x}{\partial Z^2} - \frac{1-\epsilon}{\epsilon}\frac{3Bi(x-1)}{1-Bi(1-1/\xi_c)}$$

$$\frac{\partial \xi_c}{\partial \theta} = \frac{bBi(x-1)}{\xi_c{}^2[1-Bi(1-1/\xi_c)]}$$

$$\bar{y} = \xi_c{}^3$$

Boundary and initial conditions are

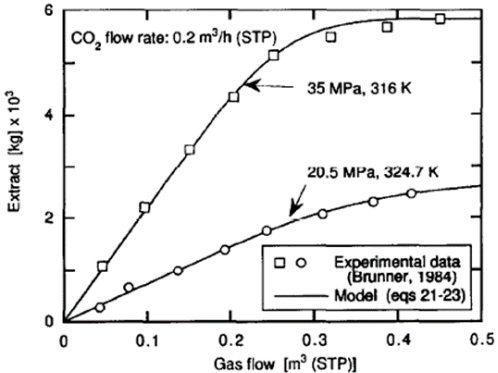
$$\left\{\begin{array}{lll} x - \dfrac{1}{Pe}\dfrac{\partial x}{\partial Z} = 0 & \text{at} & Z = 0 \\ \dfrac{\partial x}{\partial Z} = 0 & \text{at} & Z = 1 \\ x = 0 & \text{at} & \theta = 0 \\ x_i = x_0 & \text{at} & \theta = 0 \\ \xi_c = 1 & \text{at} & \theta = 0 \end{array}\right\}$$

$$\frac{D_e}{r^2}\frac{\partial}{\partial r}\left(r^2\frac{\partial C_i}{\partial r}\right) = 0$$

$$C_{s,av}/C_{s,0} = (r_c/R)^3$$

$$C_i(r = r_c) = C_r$$

$$r_c(t = 0) = R$$



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# Continuous-Mixture Model of Extraction Processes

$$\frac{\alpha dC}{dt}+\frac{C}{\tau}=-(1-\alpha)k_{\mathrm{L}}\left(C-mC_{\mathrm{p}}\right) \tag{3}$$

$$\frac{\beta dC_{\mathrm{p}}}{dt}=k_{\mathrm{L}}\left(C-mC_{\mathrm{p}}\right)-(1-\beta)\frac{dC_{\mathrm{s}}}{dt} \tag{4}$$

$$\frac{dC_{\mathrm{s}}}{dt}=k_{\mathrm{a}}\left(C_{\mathrm{p}}-C_{\mathrm{s}}/K\right) \tag{5}$$

where $C(x, t)$, $C_{\mathrm{P}}(x, t)$, and $C_{\mathrm{S}}(x, t)$ are frequency distribution functions for the extraparticle, the intraparticle, and the sorbed species, respectively. The initial conditions are

$$C(x,\ t=0)=0 \tag{6}$$

$$C_{\mathrm{p}}(x,\ t=0)=C_{\mathrm{po}}(x) \tag{7}$$

$$C_{\mathrm{s}}(x,\ t=0)=C_{\mathrm{so}}(x)\ . \tag{8}$$

The expression for the mass transfer coefficient, $k_{\mathrm{L}} = (15k_{\mathrm{f}}/r)/(5 + mBi)$, in terms of the Biot number $Bi = k_{\mathrm{f}}r/D_{\mathrm{e}}$, includes both external and intraparticle diffusional resistances.[3] When adsorption–desorption equilibrium is established instantaneously, $C_{\mathrm{s}}$ can be eliminated with $C_{\mathrm{p}} = C_{\mathrm{s}}/K$. For this equilibrium adsorption case we define dimensionless distribution functions in terms of the total lumped initial concentration

$$c_{\mathrm{o}}=\int_0^{\infty}C_{\mathrm{o}}(x)dx \tag{9}$$

where

$$C_{\mathrm{o}}(x)=C_{\mathrm{po}}(x)\left[\beta+K(1-\beta)\right]\ . \tag{10}$$

Thus,

$$y(x,\ \theta)=\frac{C(x,\ \theta)}{c_{\mathrm{o}}} \tag{11}$$

and

$$y_{\mathrm{p}}(x,\ \theta)=\frac{C_{\mathrm{p}}(x,\ \theta)}{c_{\mathrm{o}}} \tag{12}$$

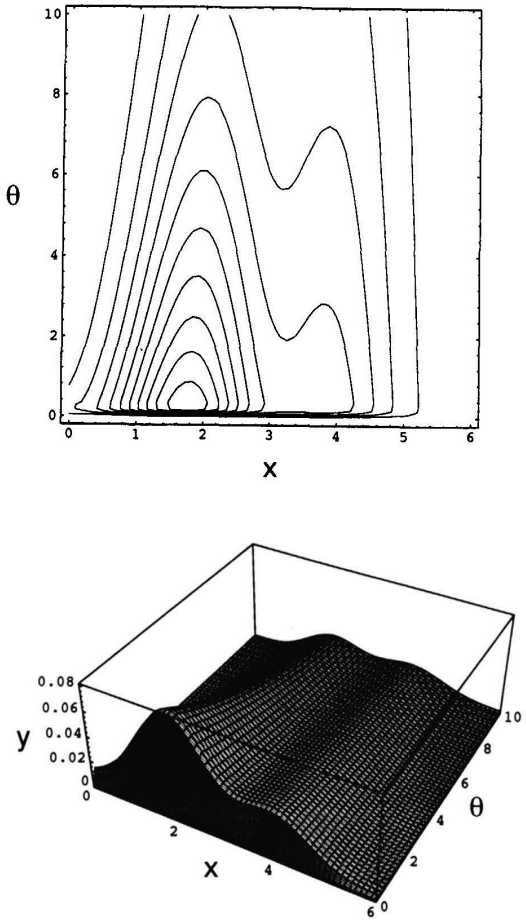



**Figure 1.** Contour and three-dimensional plots of dimensionless concentration $y$, molecular property $x$, and dimensionless time $\theta$ when $K = x/0.1$.

UCD/Ben. J. McCoy

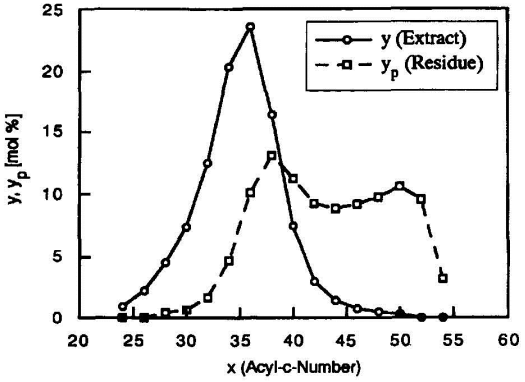



**Figure 3.** Molar distribution of triglycerides for different acyl-C-numbers in extract and residue at equilibrium for a batch extraction.[5]



M. Goto, T. Hirose, B. J. McCoy, J. Supercritical Fluids, 7, 61, 1994

# Extraction – Modeling -

## Article Influence

### Extraction

Food Bioproc. Technol.
**Bioactive Compounds**
Meireles, 2010
346

Crit. Rev. Food Sci.
**Oilseed Extraction Models**
Del Valle, 2006
111

Chem. Eng. Res. Design
**Kinetic Models**
C.M.Silva, 2011
113

JSF
**Shrinking Core Model**
Goto, 1996
(R)
122

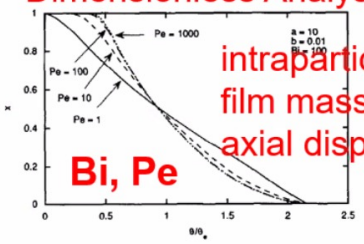
JSF
**Mathematical Models for SFE**
M.B.King, 1999
117

JSF
**Vegetable Matrices**
De Melo & C.M.Silva, 2014
284

**Cites**

(R) = Research Paper

Dimensionless Analysis

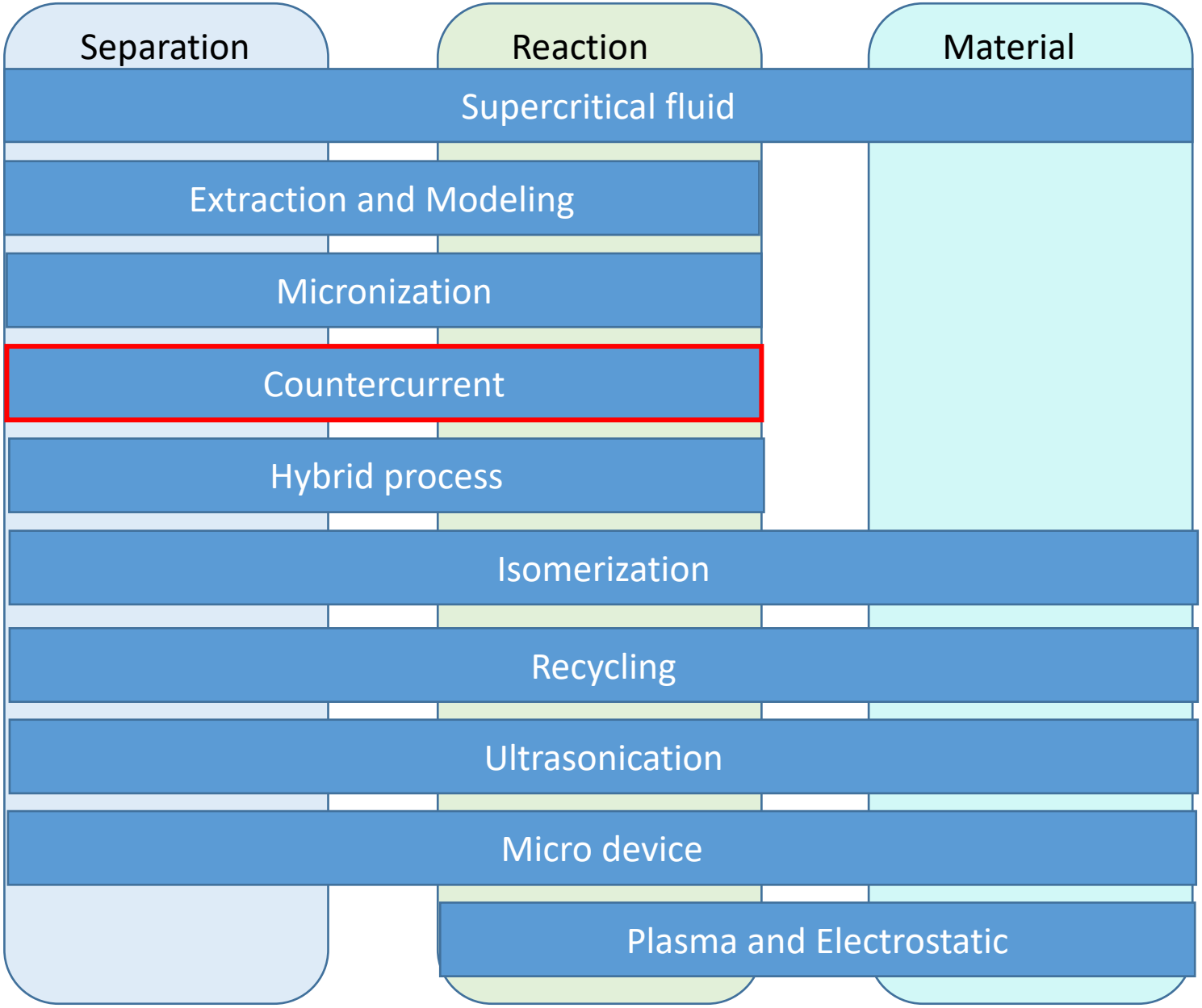



intraparticle diffusion
film mass transfer
axial dispersion

M. Goto, B.C. Roy, T. Hirose, Shrinking-core leaching model for supercritical-fluid extraction, Journal of Supercritical Fluids 9 (1996) 128-133. https://doi.org/10.1016/S0896-8446(96)90009-1



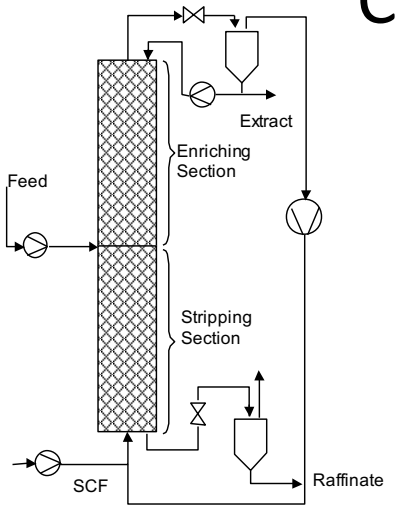
By Prof. Richard Smith

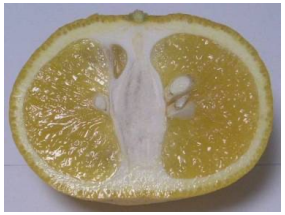

Separation
Reaction
Material
Supercritical fluid
Extraction and Modeling
Micronization
Countercurrent
Hybrid process
Isomerization
Recycling
Ultrasonication
Micro device
Plasma and Electrostatic

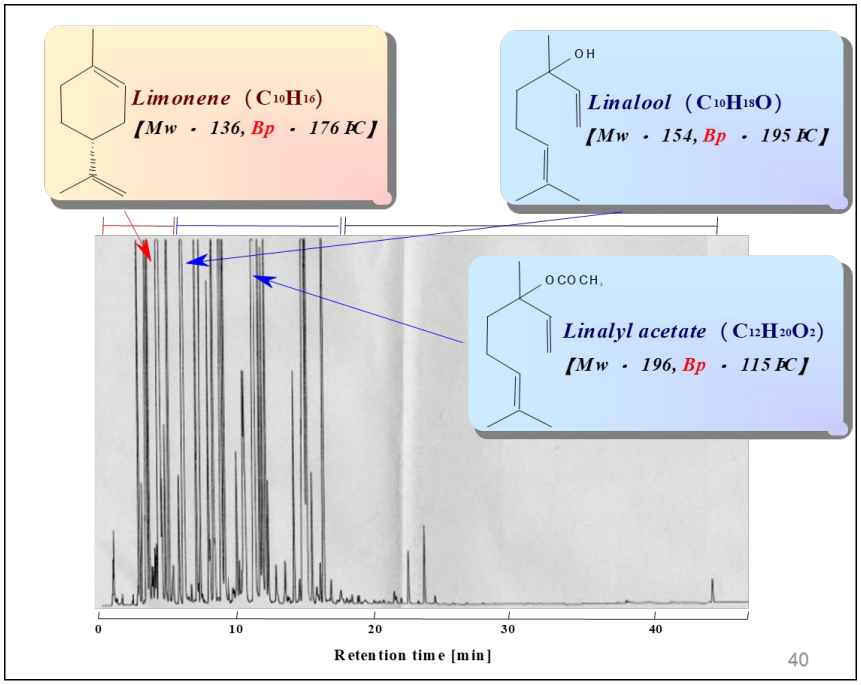
NAGOYA UNIVERSITY

# Countercurrent extraction

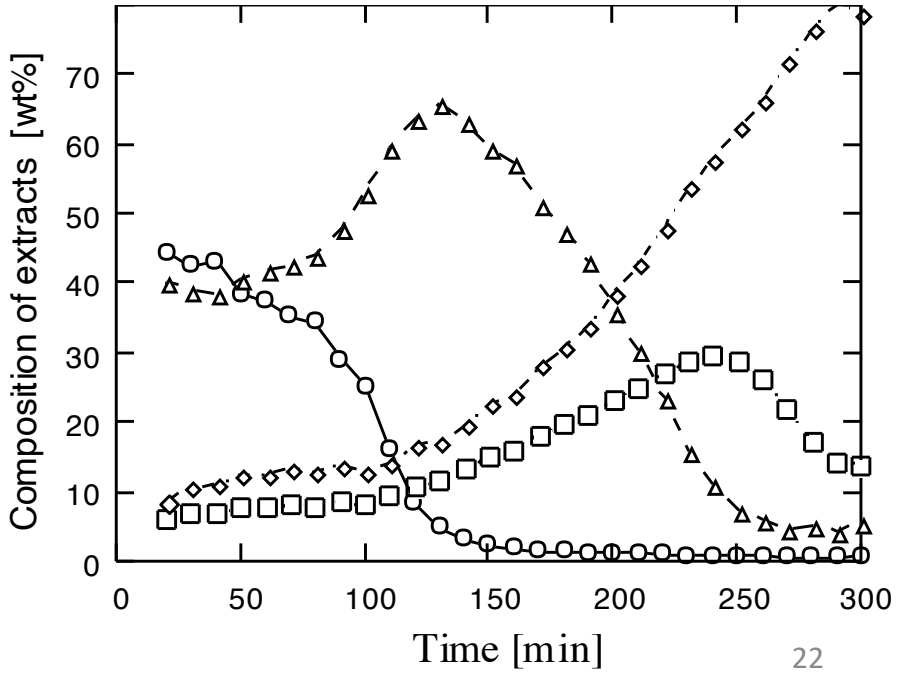
## Deterpenation of citrus peel oil







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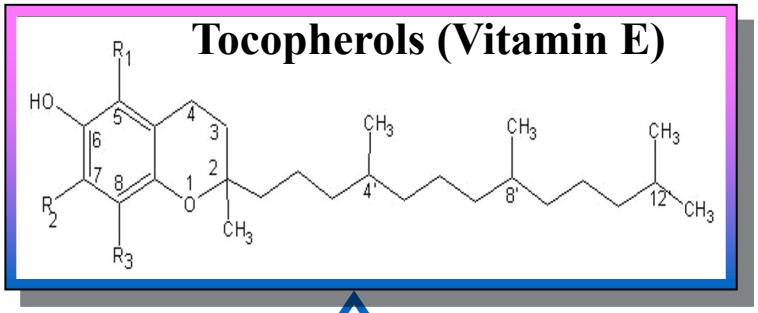
# Separation of Fatty Acid Methyl Esters and Tocopherols with Supercritical $CO_2$

Dr. Tao Fang




Supercritical $CO_2$

Molecular or vacuum distillation

Methyl esterified DOD (ME-DOD)

Supercritical MeOH

Methyl esterification ( $H_2SO_4$)

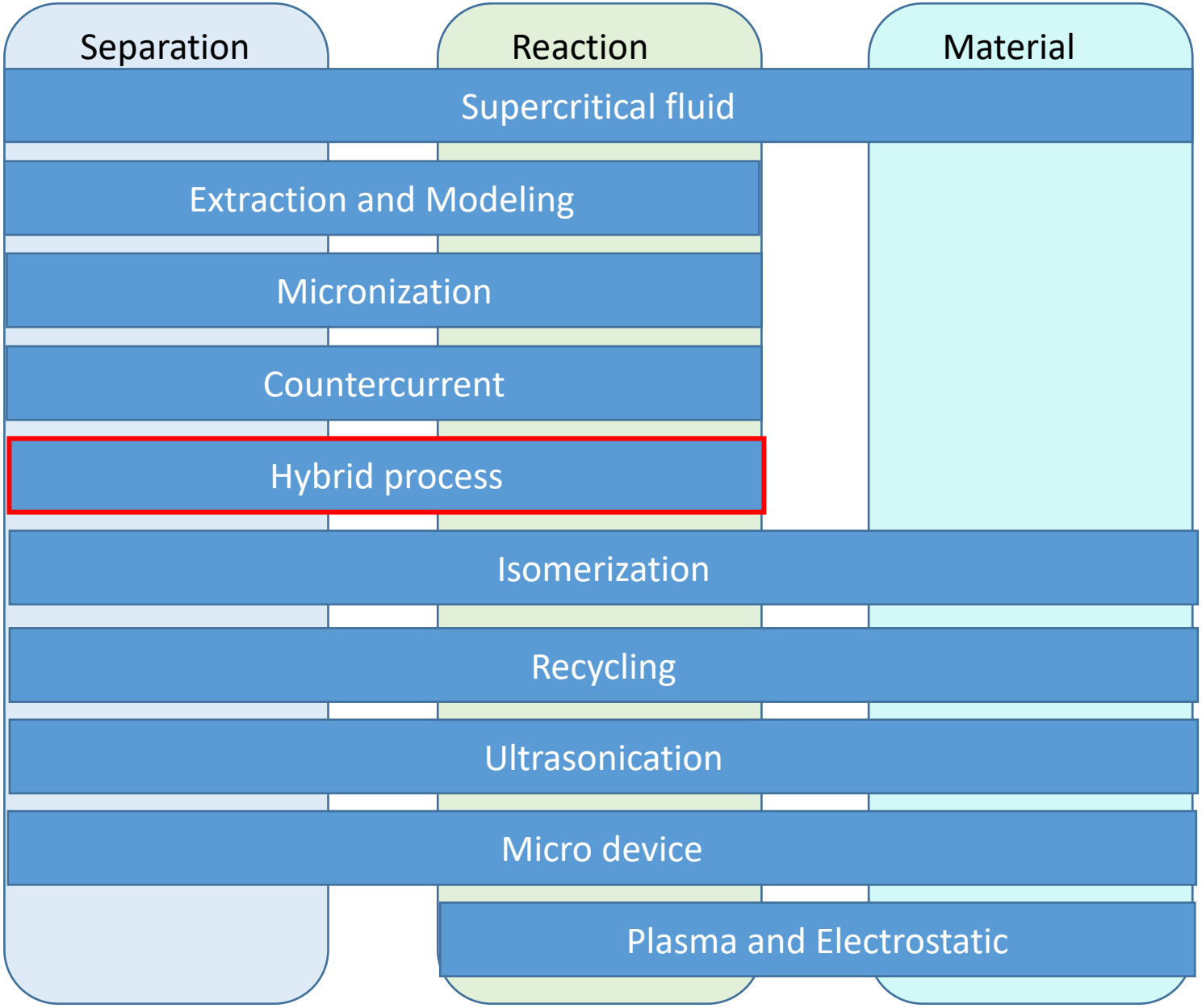
Sterols

Soybean deodorizer distillate (abbreviated as "DOD" )

Soybean oil industry

China

Korea


Separation
Reaction
Material
Supercritical fluid
Extraction and Modeling
Micronization
Countercurrent
Hybrid process
Isomerization
Recycling
Ultrasonication
Micro device
Plasma and Electrostatic

# Separation - Adsorption -

## Supercritical Pressure Swing Adsorption Process

◇*Experiment*◆

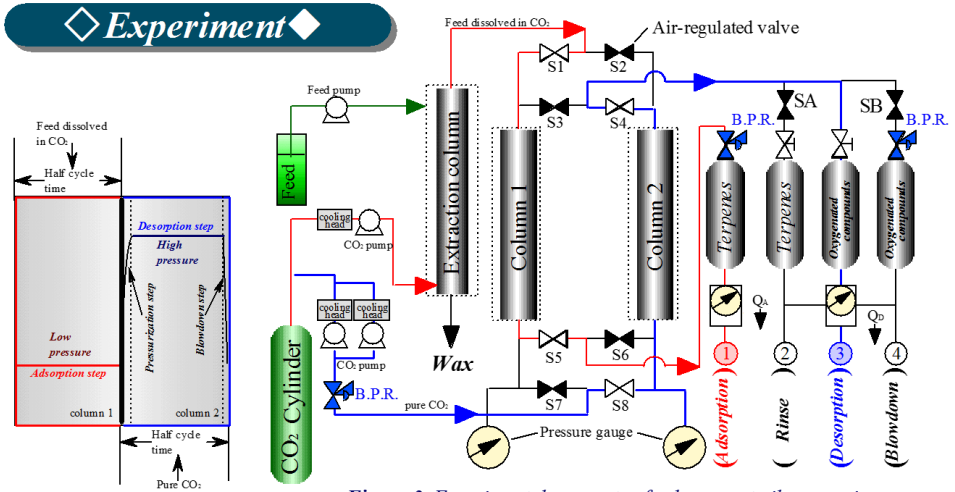
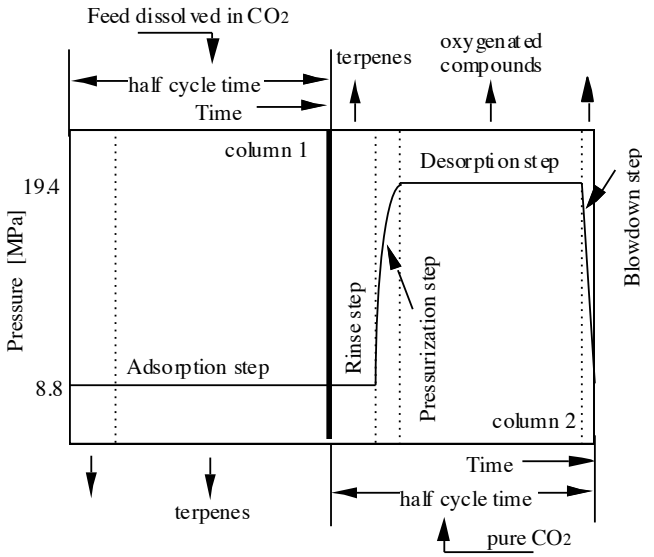
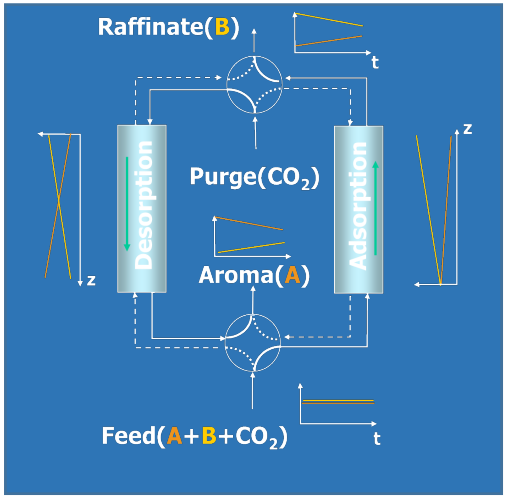


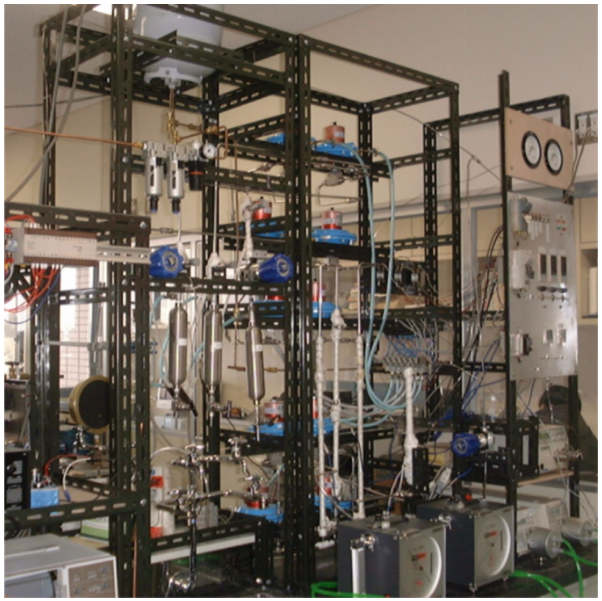

*Figure 3* *Experimental apparatus for bergamot oil processing.*

***Extraction column*** : 9mm $\phi$ ×600mm
***Adsorption column*** : 9mm $\phi$ ×500mm
***Temperature*** : ***313 K***




- ***Oxygenated compounds*** in the effluents collected were weighed and analyzed by ***a capillary gas chromatograph equipped with FID***

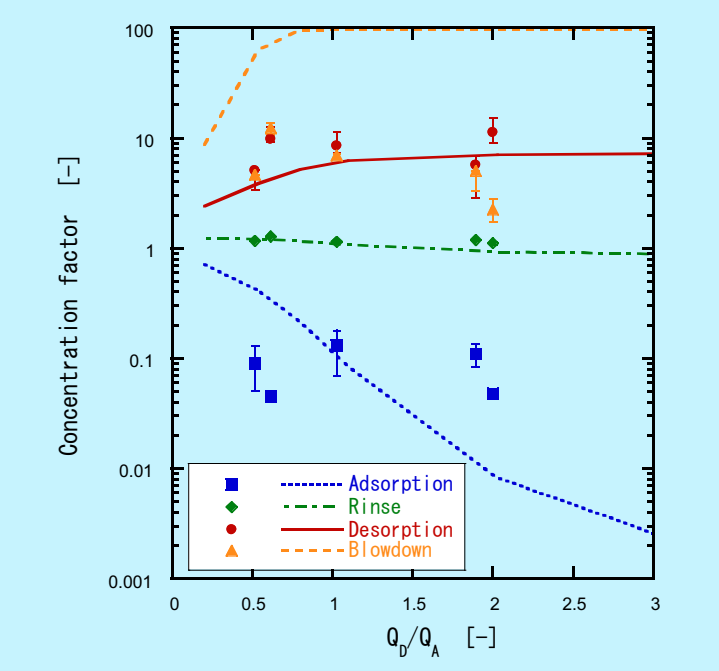


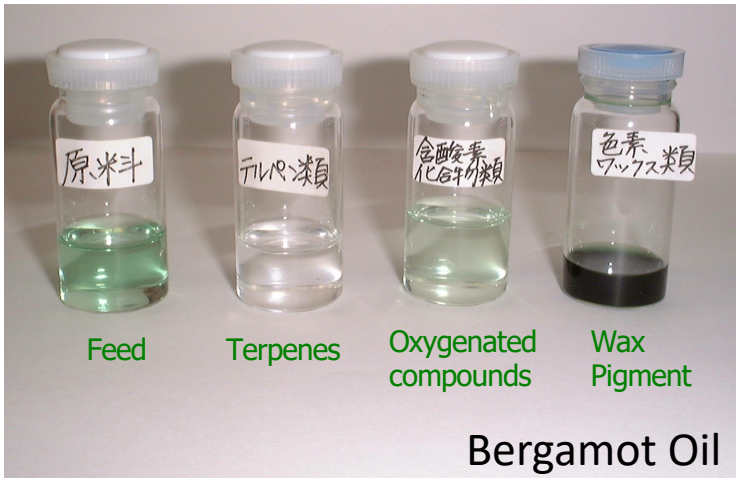



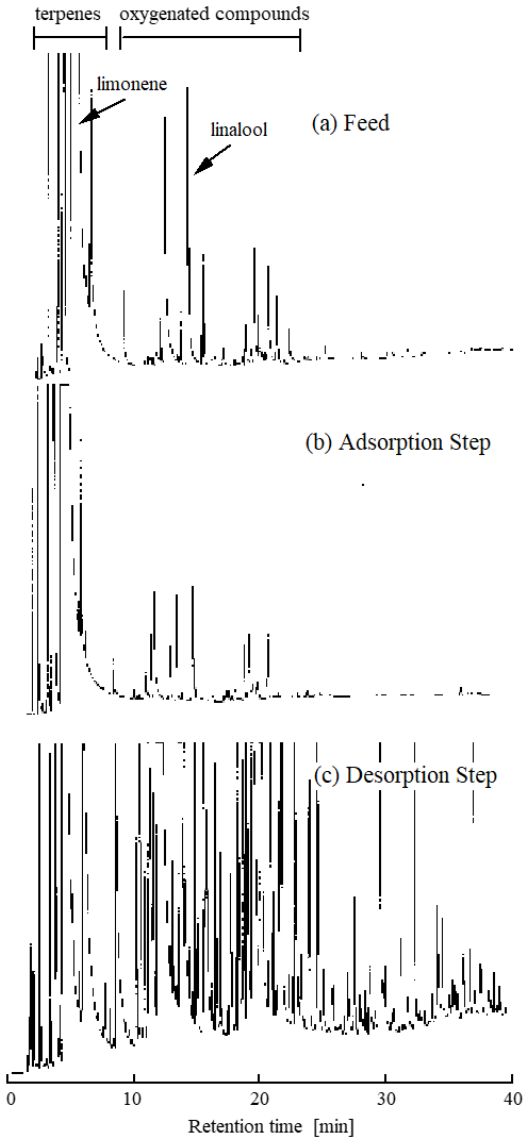
Dr. Masaki Sato

# Separation - Adsorption -

## Supercritical Pressure Swing Adsorption Process



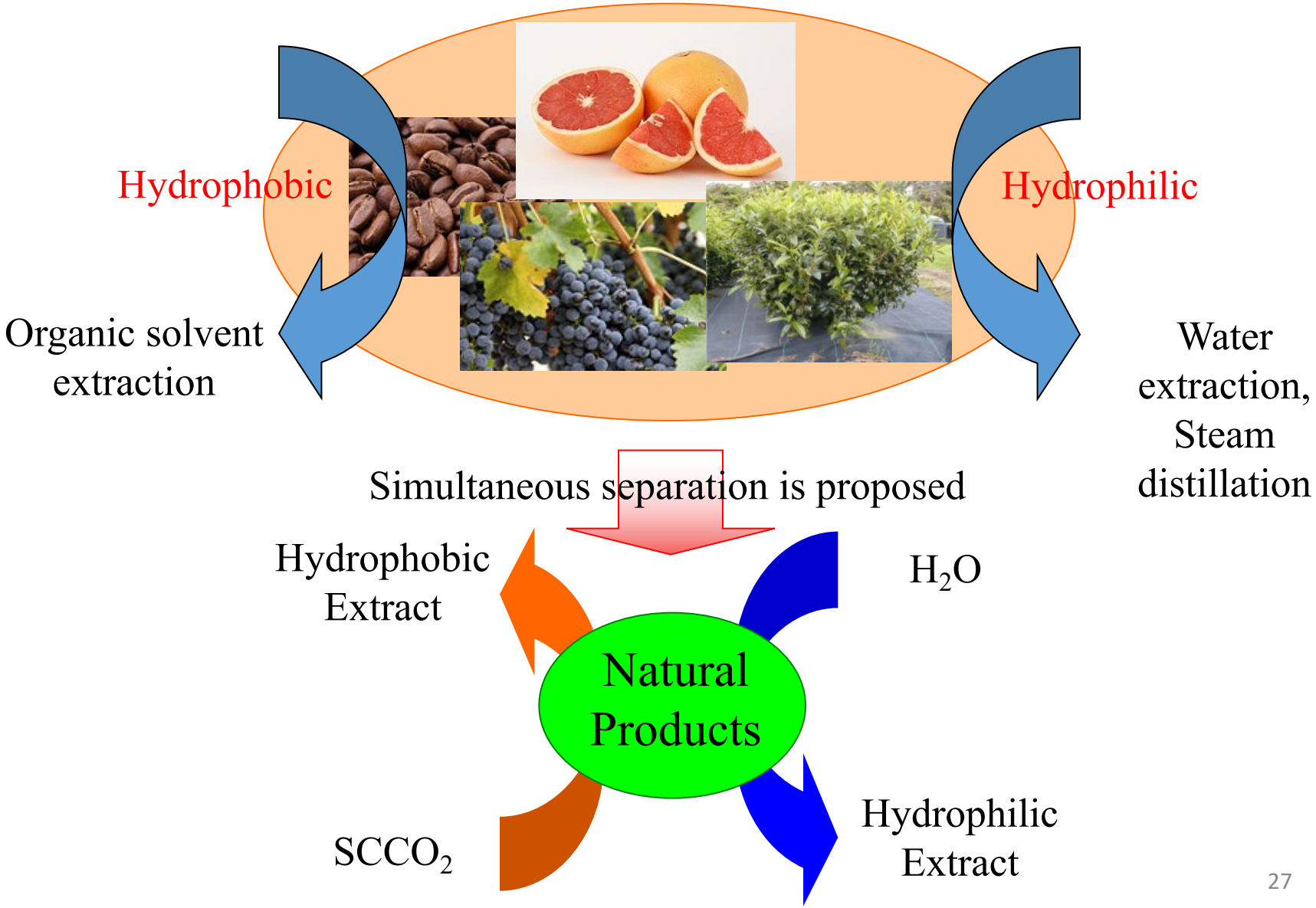

collaboration

Givaudan

Lemon oil
Orange oil
Bergamot oil
Squalene/tocopherol

# Separation - Hybrid process-

## Hybrid extraction process (sc-$CO_2$+Liquid Water)



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# Hybrid SFE with $CO_2$ and water

**$scCO_2$ Extraction**

**Hot-water Extraction (subcritical water)**

**Target Material**

**Polarity ·Molecular weight**

- Essential Oil
- Terpenes
- Flavonoid
- Protein
- lipids
- Saccharides
- Glycoside

**Hybrid SFE with $CO_2$ and water**

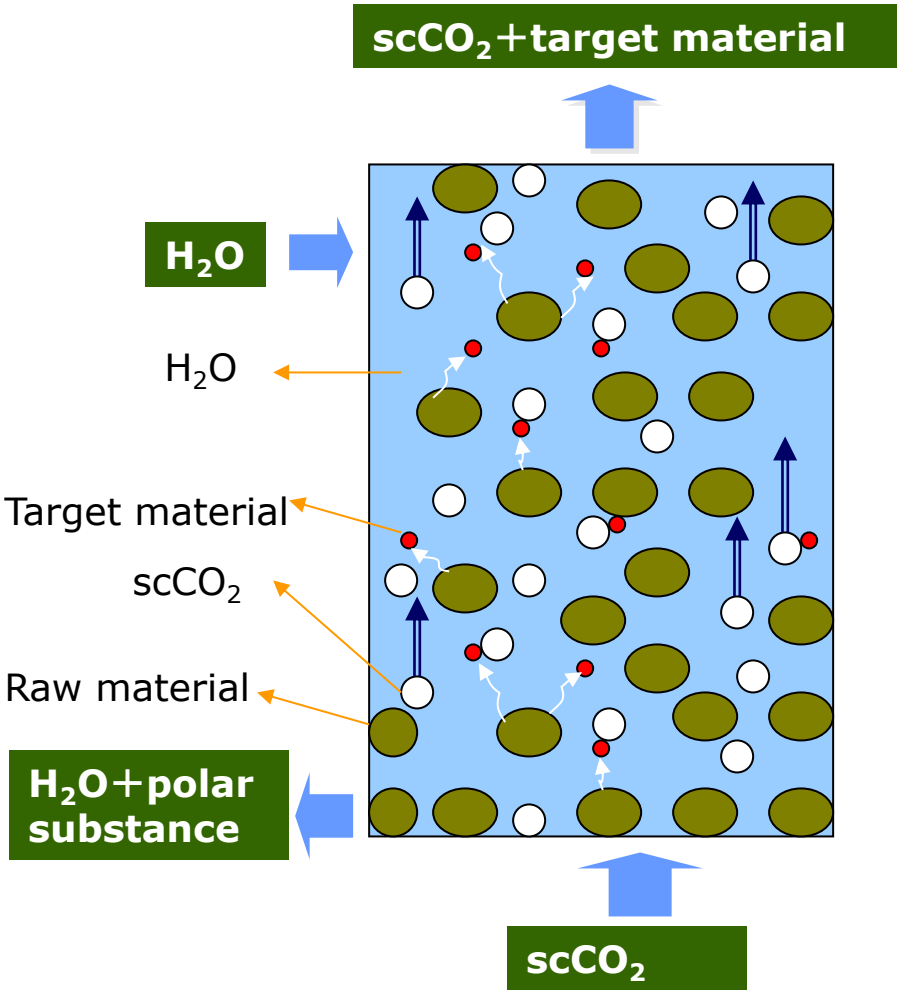
Green solvent process

Halal production process

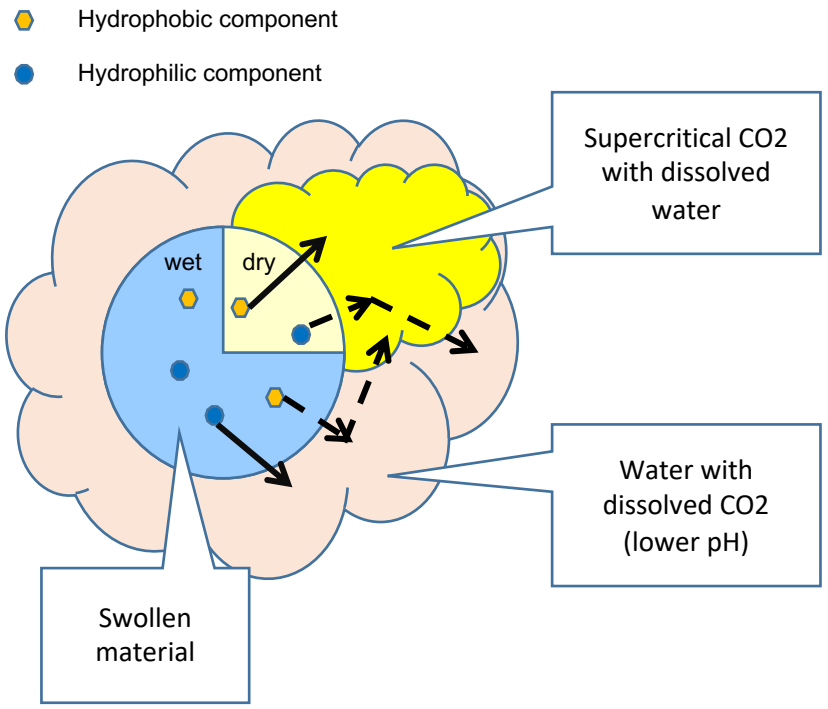
https://halalfocus.net/eu-introducing-the-european-halal-initiative/ 2022.5.27

# Separation - Hybrid process-

## Hybrid extraction process (sc-$CO_2$+Liquid Water)




[continuous;H2O, dispersion;scCO2]



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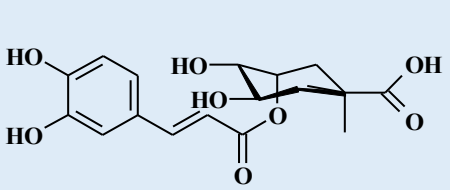
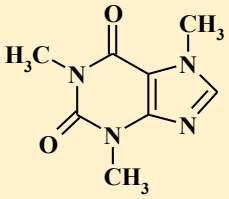
# Hybrid extraction process

Chlorogenic acid and caffeine from green coffee bean

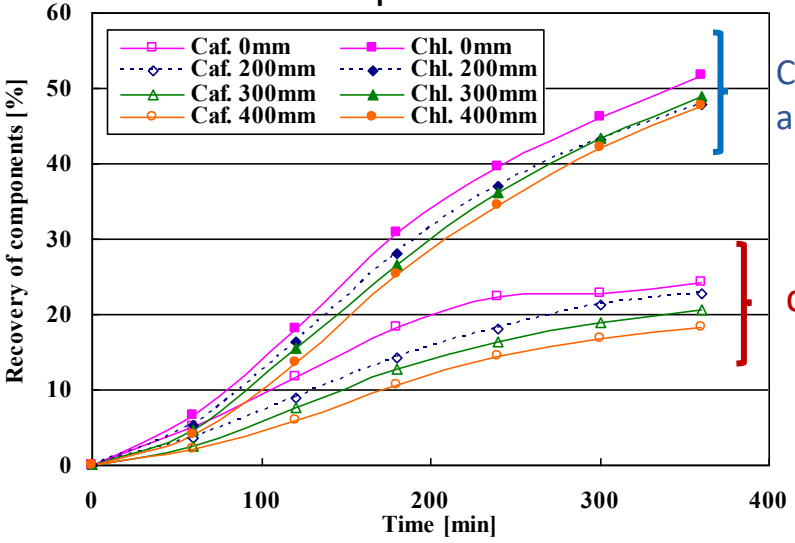


Chlorogenic acid



Caffeine

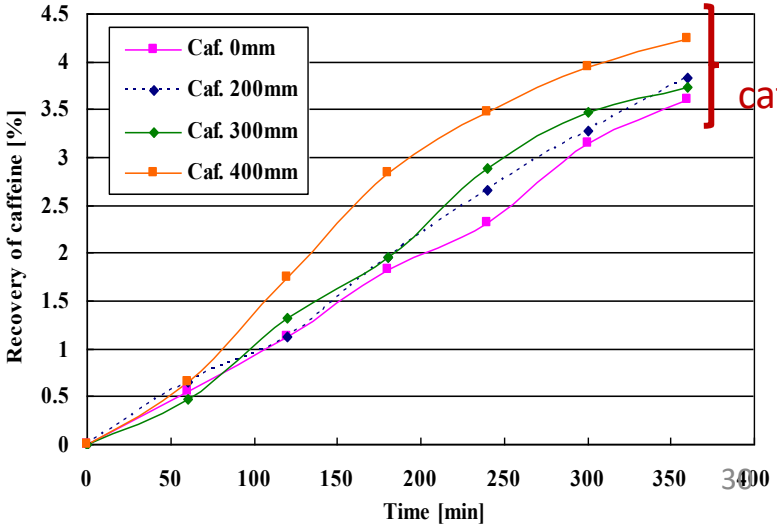
Water phase




Chlorogenic acid

caffeinec

$CO_2$ phase

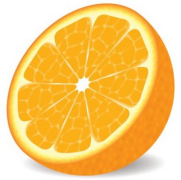



caffeine

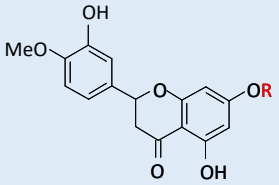
# Separation - Hybrid process-

## Hybrid extraction process

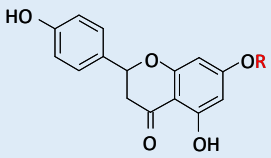
Flavonoids extraction from citrus peel



### High polarity

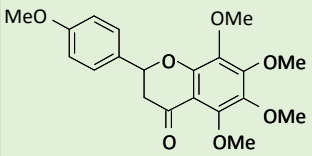



Hesperidin
(R: rutinose)

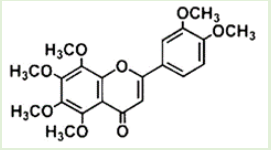



Naringin
(R: neohesperidose)

### Low polarity

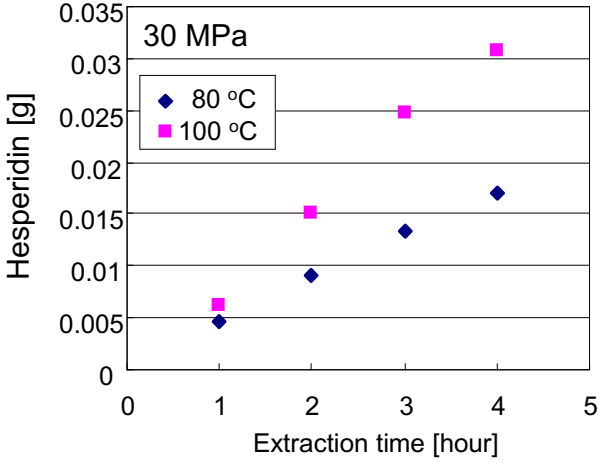



Tangeretin

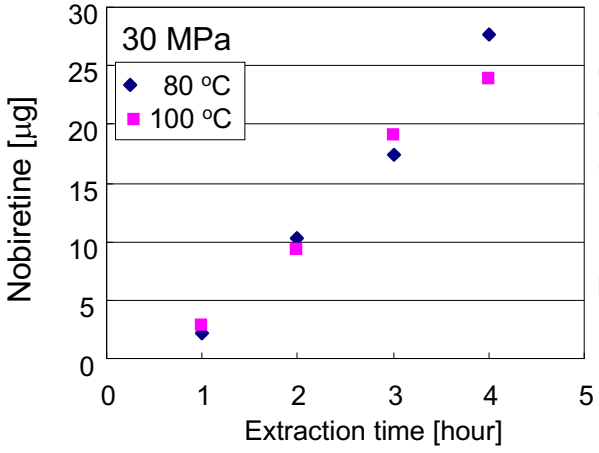


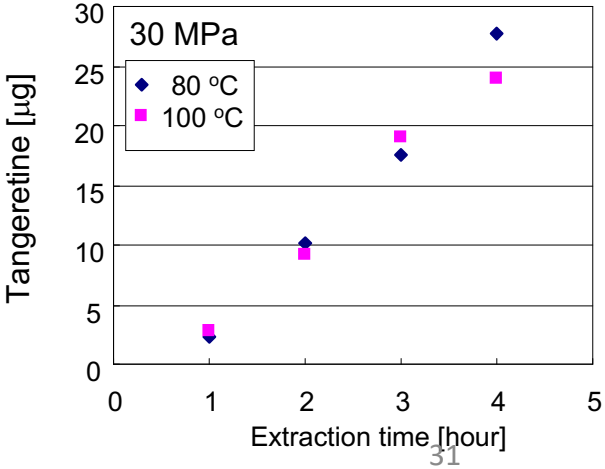

Nobiretine

**Hesperidin in water stream**



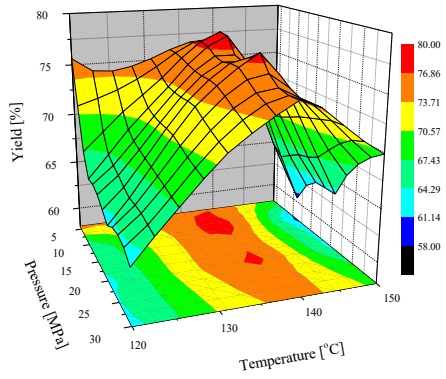

**Polymethoxyflavonoid in $CO_2$ stream**



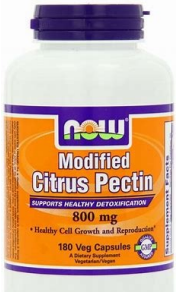
# Separation - Hybrid process-

## Extraction of pectin with subcritical water and $CO_2$

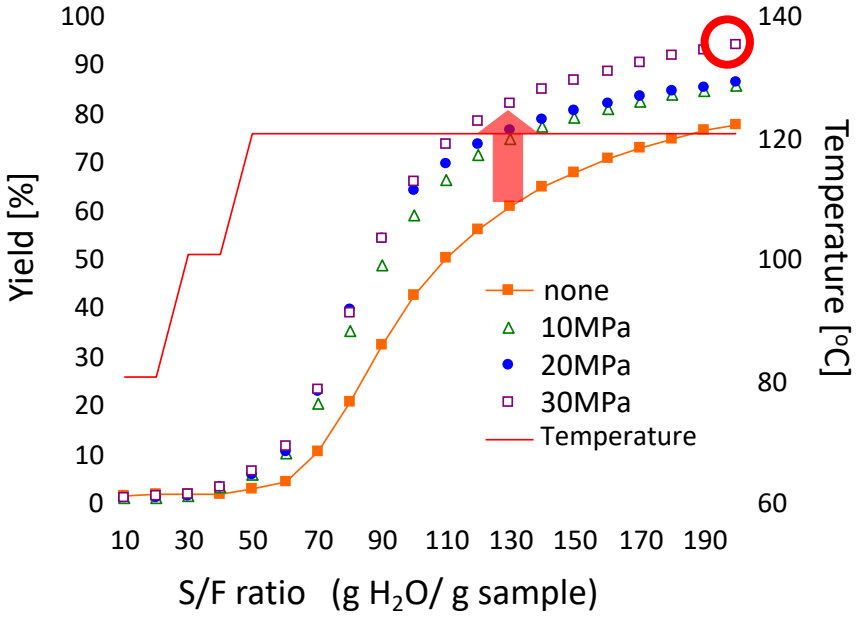



78 %

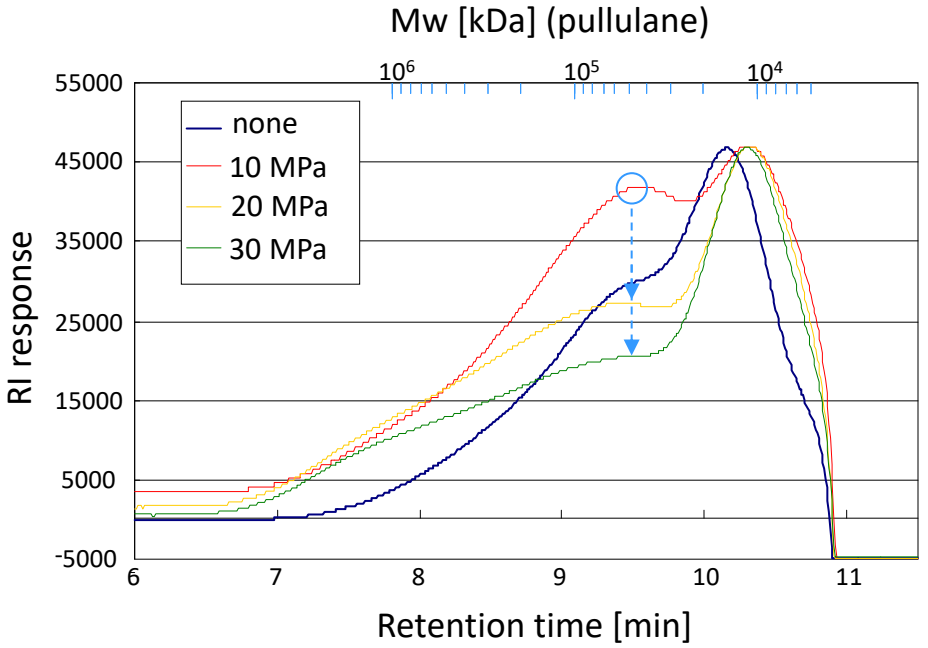
The hydrolysis of pectin molecule was enhanced by increasing in the concentration of carbonic acid in subcritical water.




92 %




Extraction yield of pectin increased with pressure (dissolved $CO_2$)



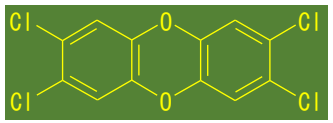

Molecular weight of pectin was controlled by pressure (dissolved $CO_2$)

# Separation - Hybrid process-

Destruction of Dioxins by combined process of extraction, adsorption, and SCWO

Dr. S. Kawajiri

Solid containing dioxins



extraction

detoxified solid

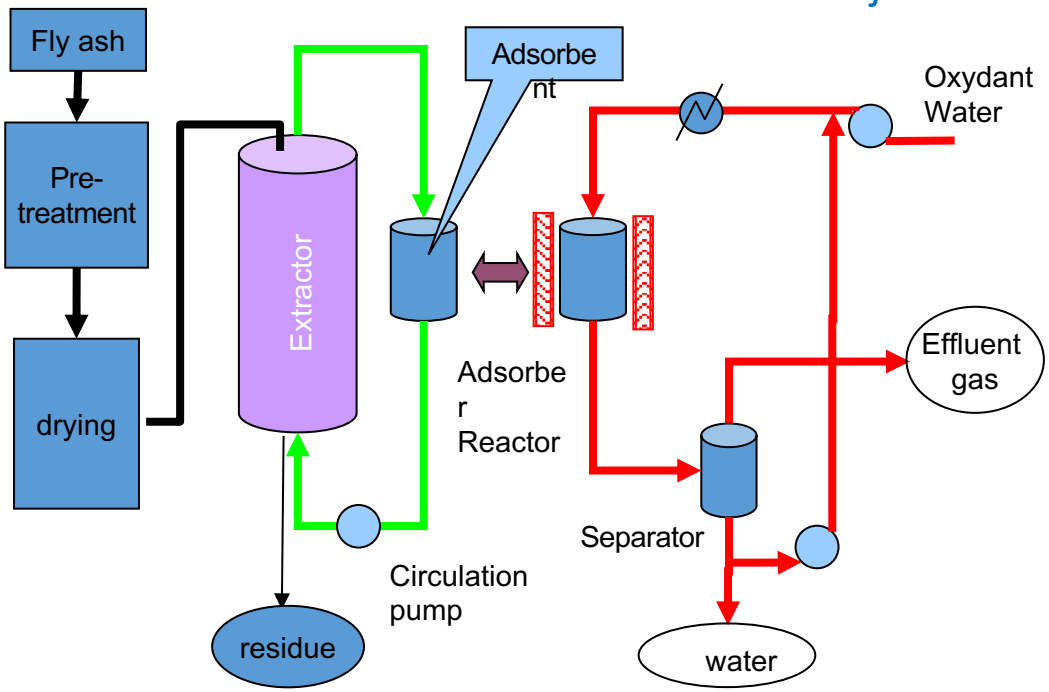
Sc-CO2

adsorption

concentration

destruction

SCWO




NEDO Project [2000-2004]

*Nagasaki Ryoden Technica*

*Kumamoto University*

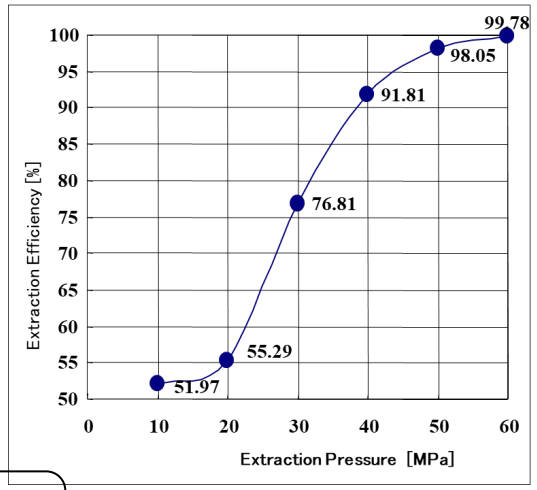
*Shizuoka University*

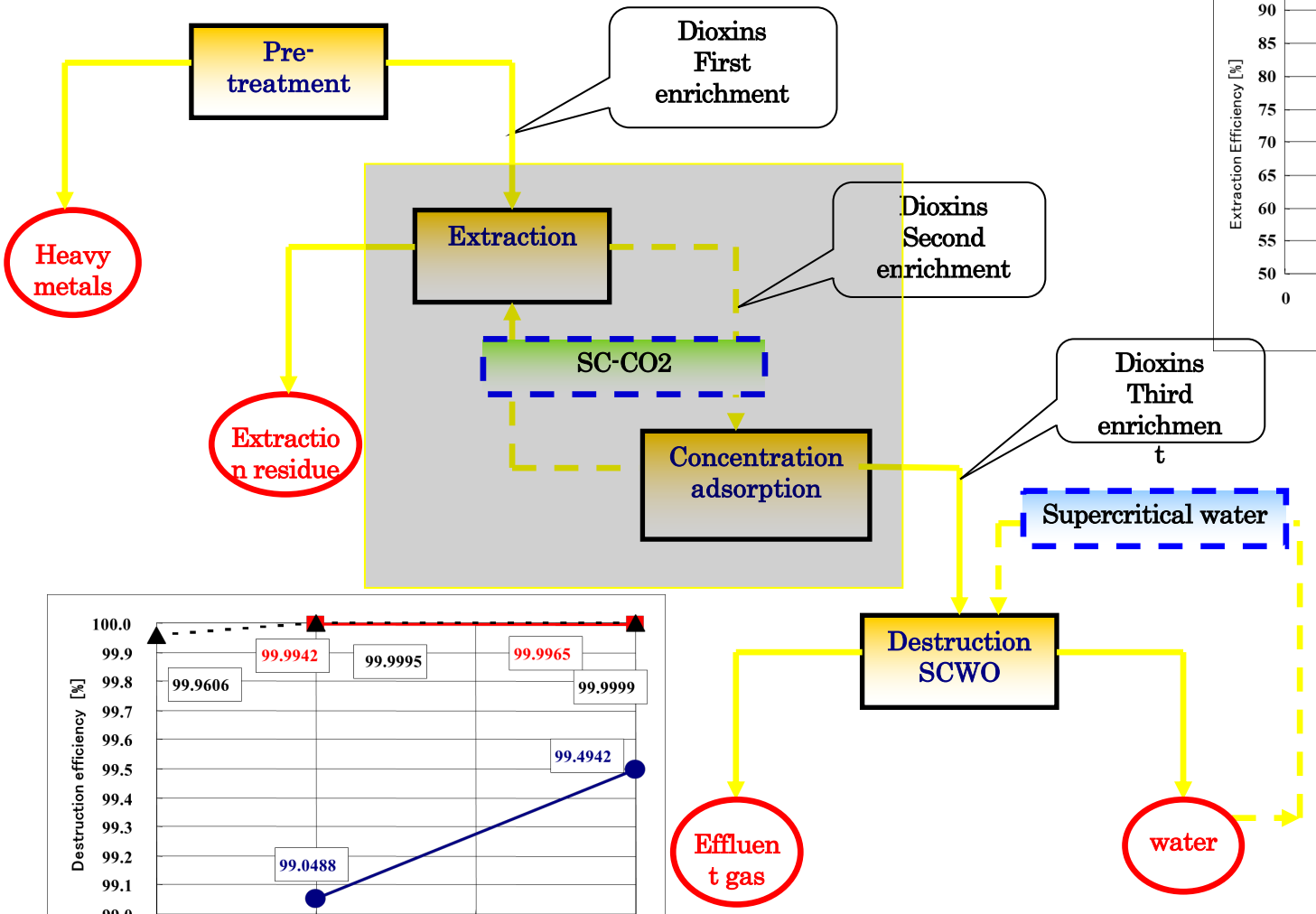


長崎菱電テクニカ株式会社

# Separation - Hybrid process-

## Destruction of Dioxins by combined process of extraction, adsorption, and SCWO



# Separation - Hybrid process-

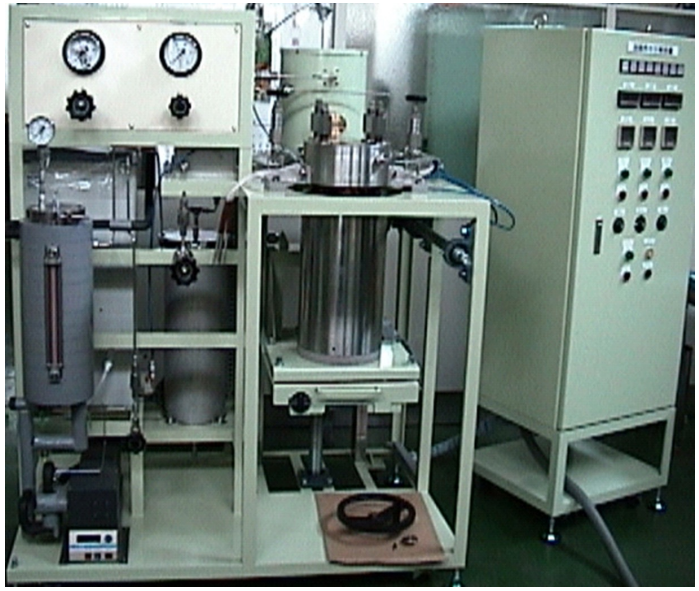
Destruction of Dioxins by combined process of extraction, adsorption, and SCWO

*Nagasaki Ryoden Technika / Shizuoka Univ.*

## Bench Plant

Extractor: 30 litter
Adsorber: 300 ml

SCWO reactor: 2 litter

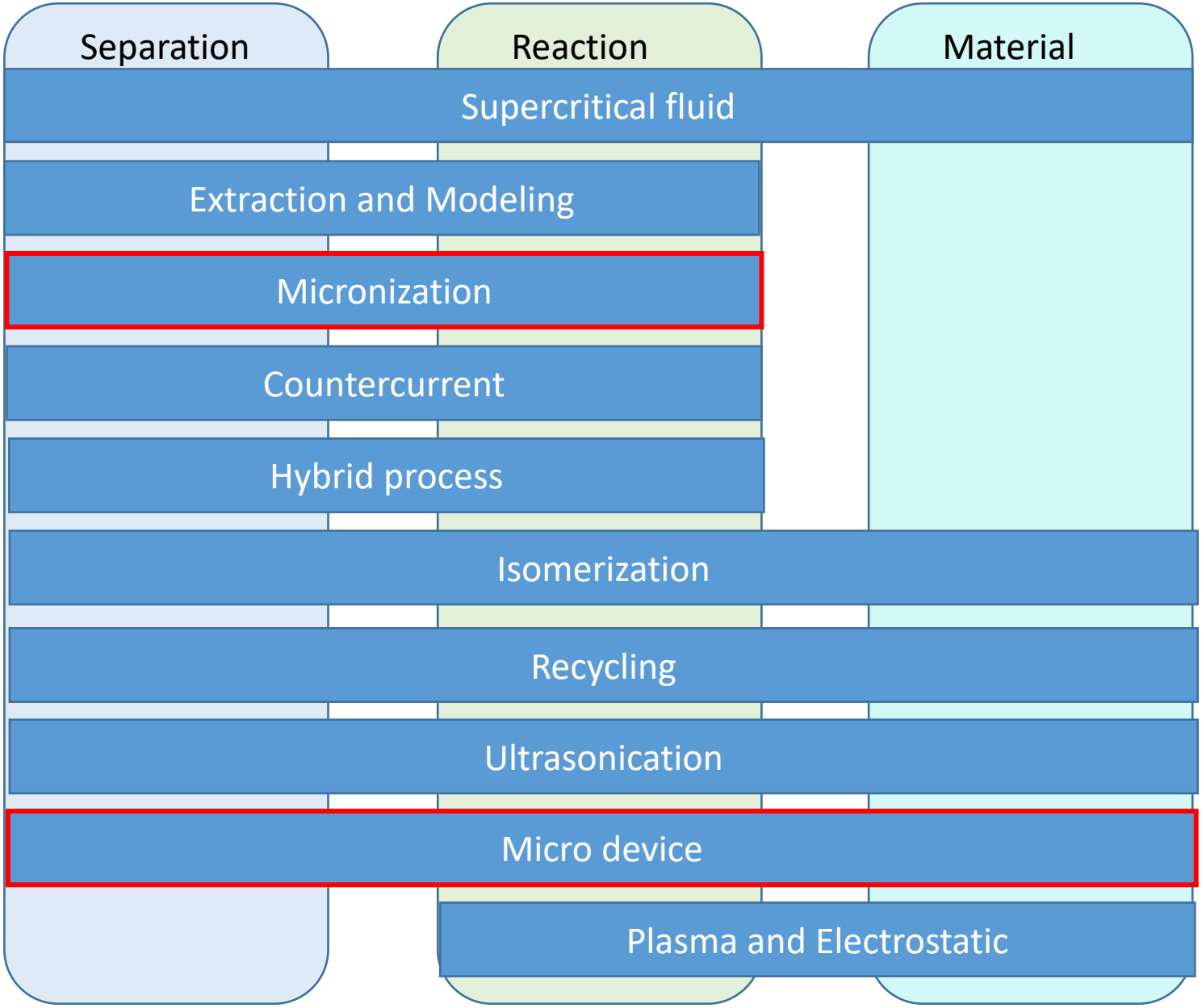


Extraction/Adsorption

SCWO



長崎菱電テクニカ株式会社


Separation
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Isomerization
Recycling
Ultrasonication
Micro device
Plasma and Electrostatic

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# Particle Design with Supercritical Fluids Technology

Prof. Youn-Woo Lee (SNU)

RESS (Rapid Expansion of Supercritical Solutions)

GAS (Gas Anti-Solvent)

SAS (Supercritical fluid Anti-Solvent)

ASES (Aerosol Solvent Extraction System)

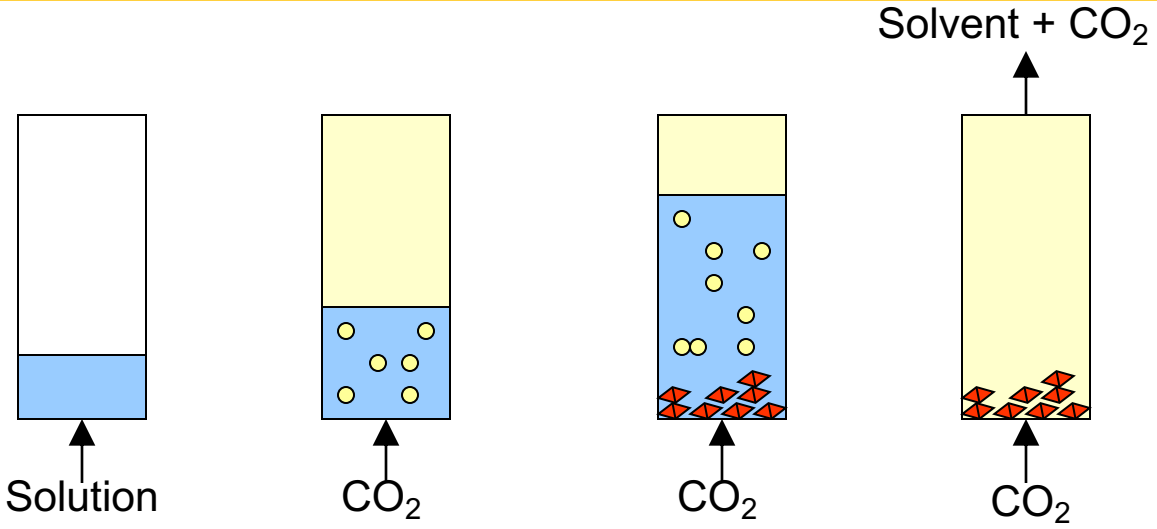
SEDS (Solution Enhanced Dispersion by Supercritical Fluids)

PGSS (Particles from Gas Saturated Solutions)

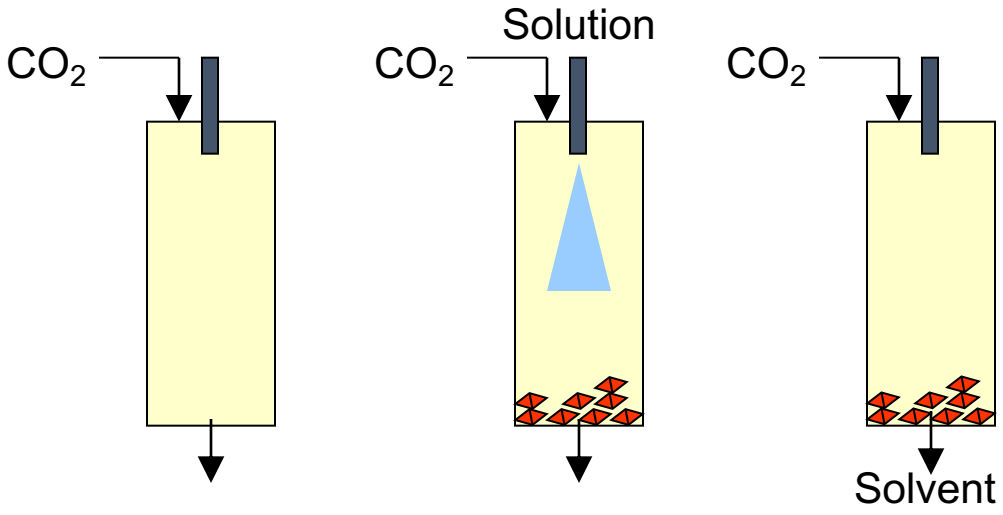
NAGOYA UNIVERSITY

# SAS: Supercritical Anti-Solvent Crystallization

GAS
(Gas Ant-Solvent)




ASES
(Aerosol Solvent Extraction System)



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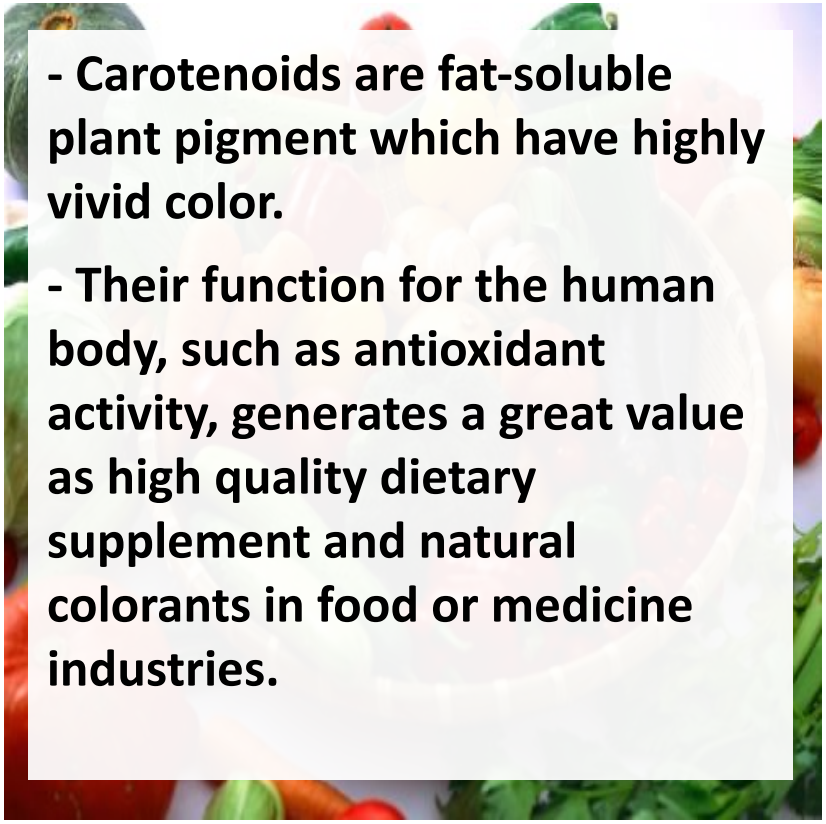
# Carotenoids

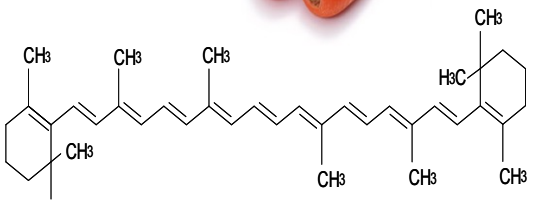


- Carotenoids are fat-soluble plant pigment which have highly vivid color.
- Their function for the human body, such as antioxidant activity, generates a great value as high quality dietary supplement and natural colorants in food or medicine industries.

## β-carotene ($C_{40}H_{56}$)

MW: 536.87

**Activity**
- Anti-oxidant
- Anti-cancer
- dermatitis prevention

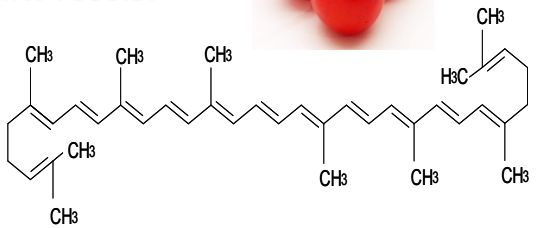



chemical structure of β-carotene

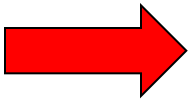
## Lycopene ($C_{40}H_{56}$)

MW : 536.87

**Activity**
- Anti-oxidant
- Anti-cancer
- Maintain bone density




chemical structure of Lycopene



Dissolve in organic solvent and oil

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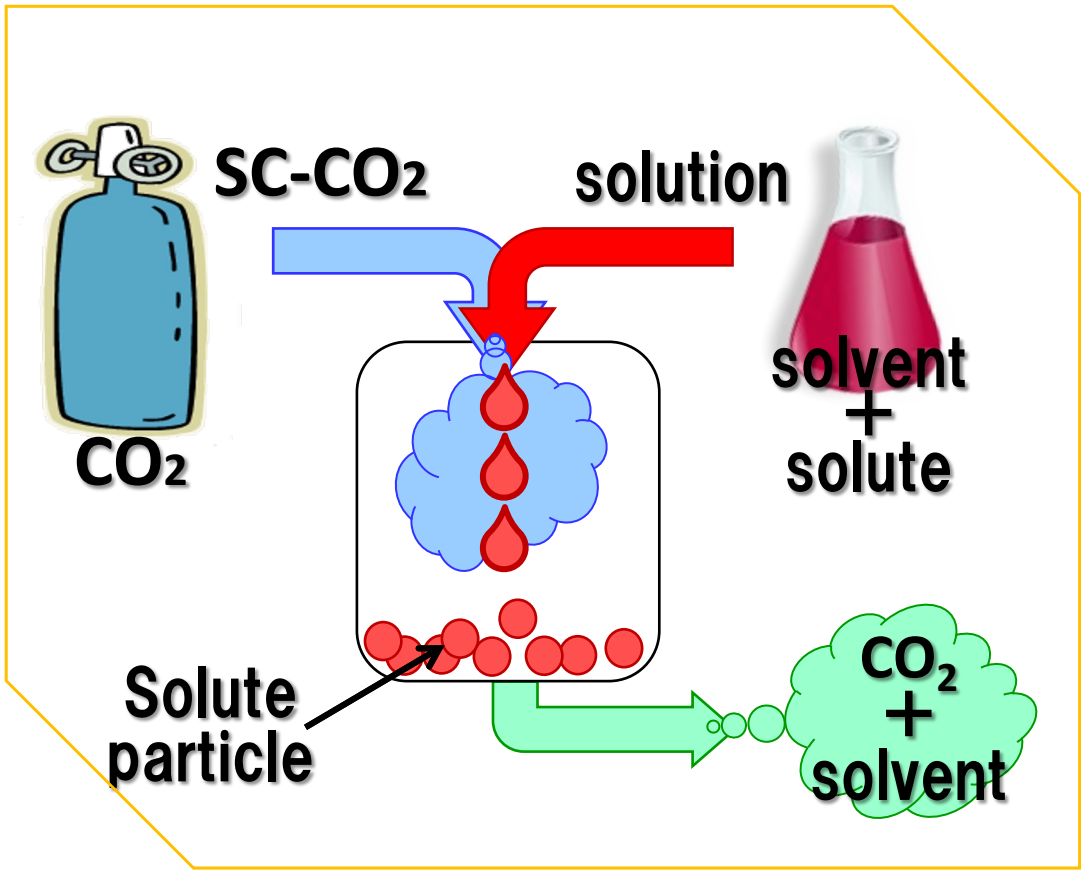
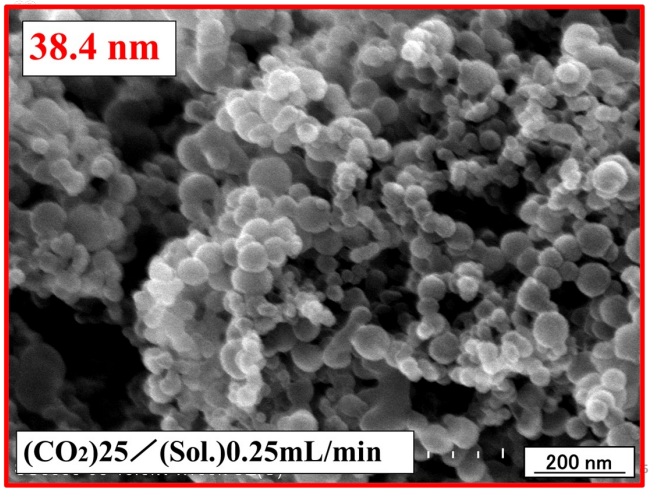
# SEDS process

Dr. Hazuki Nerome




lycopene/β-CyD



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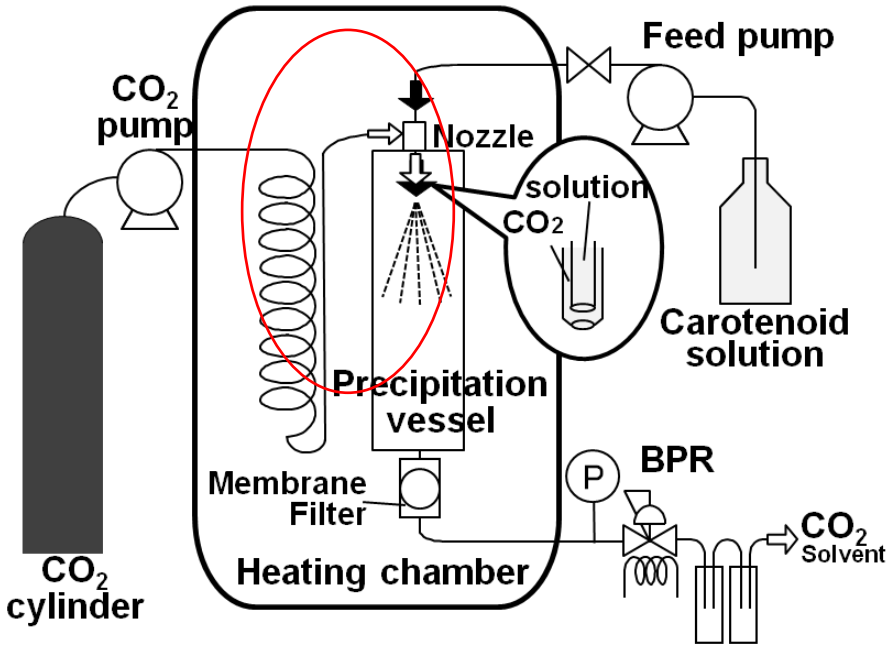
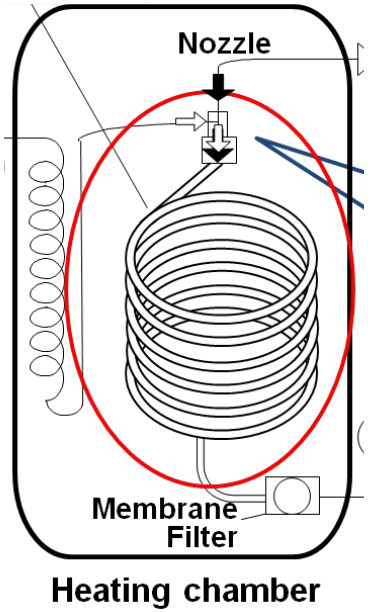
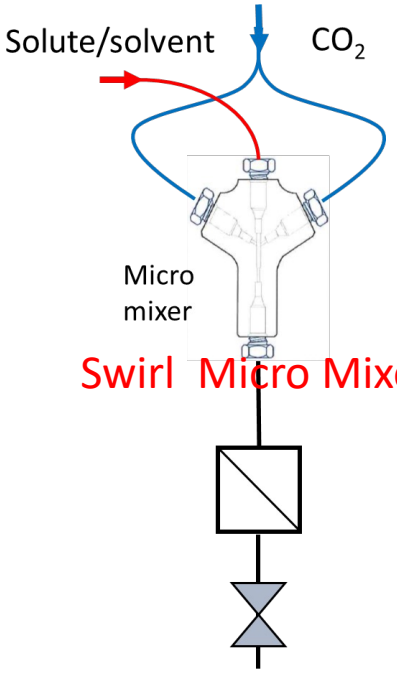
# Design of precipitator cell




Vessel type

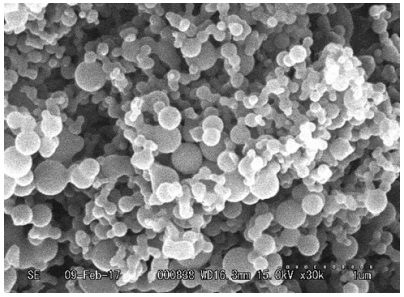



Tube type

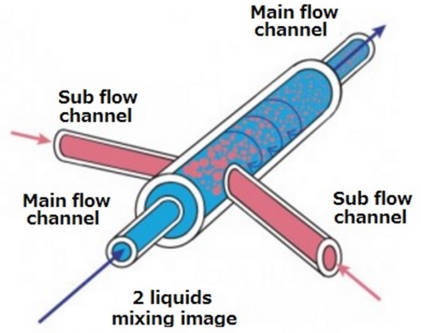


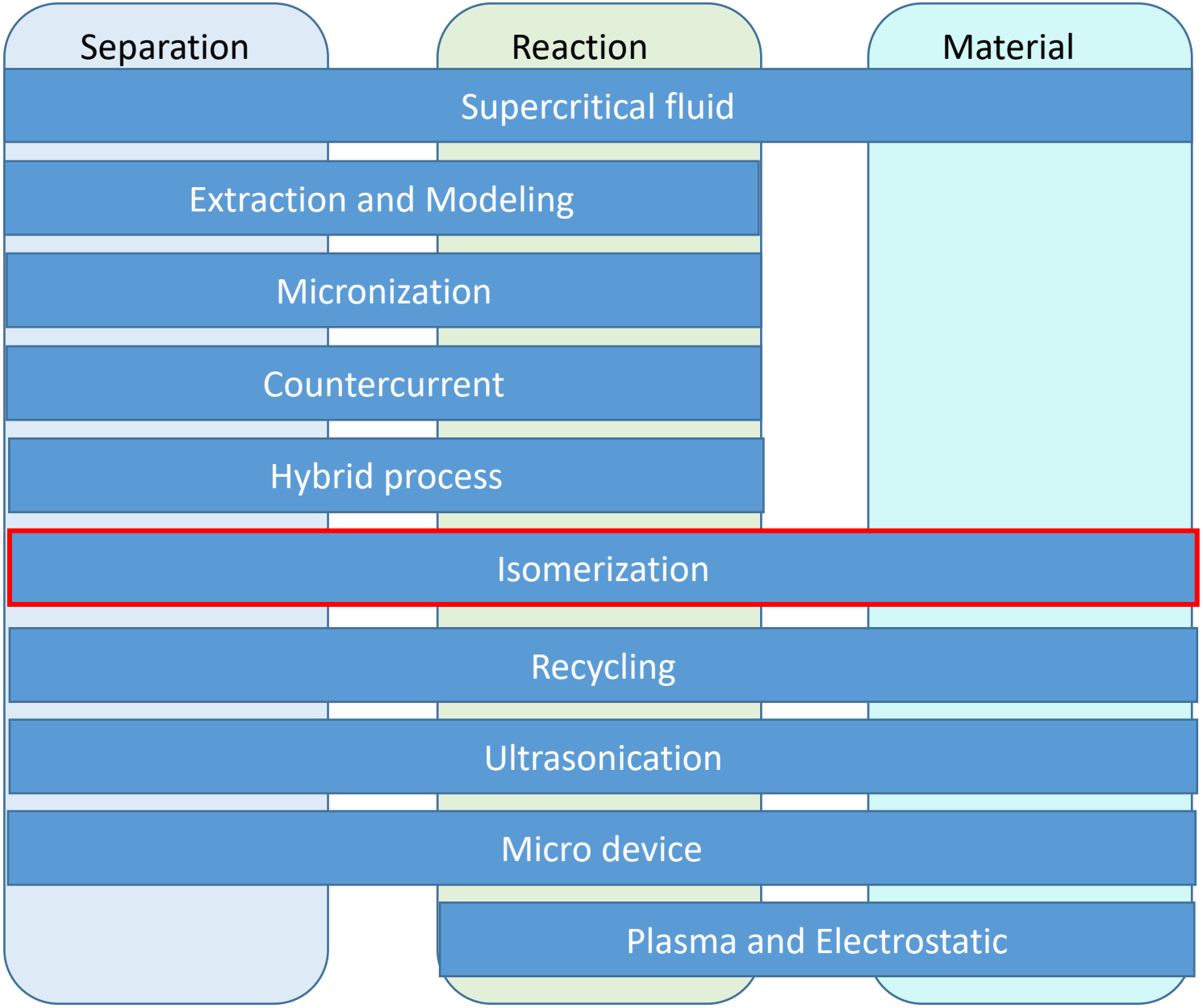

Swirl Micro Mixer

Micromixer type



Curcumin/PVP particle

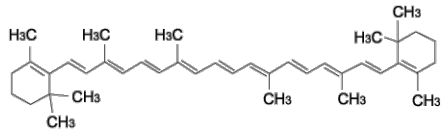


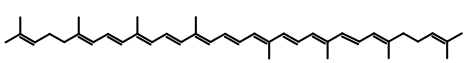

Separation
Reaction
Material
Supercritical fluid
Extraction and Modeling
Micronization
Countercurrent
Hybrid process
Isomerization
Recycling
Ultrasonication
Micro device
Plasma and Electrostatic

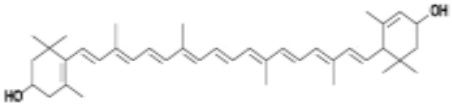
# Isomerization

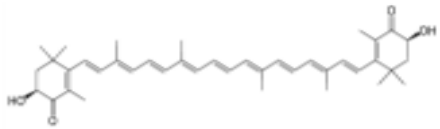
## Z-isomerization in Supercritical Fluid Processing of Carotenoids

Dr. Masaki Honda

β-carotene  

Lycopene  

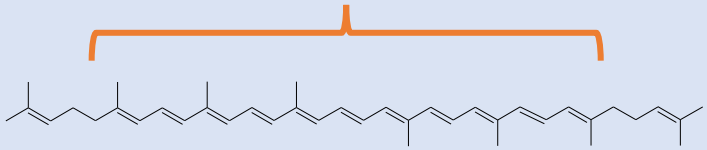
Lutein  

Astaxanthin  

Improvement of functionalities
→ absorbability and antioxidant capacity

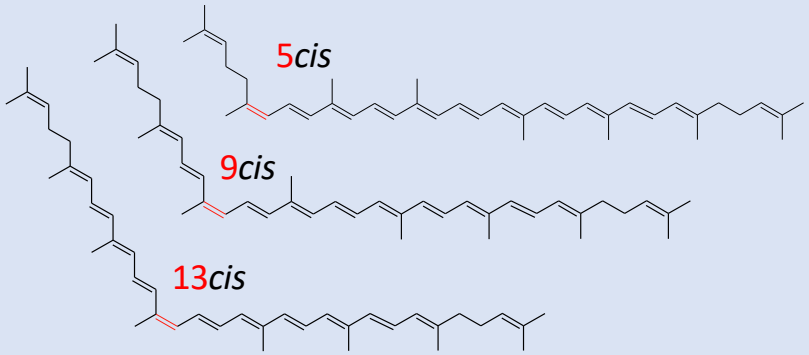
Change of physical properties
→ melting point, crystallinity and solubility

11 conjugated double bonds



**all-*trans*-lycopene**

・ plants




***cis*-isomers of lycopene**

・ living animals・ processed foods

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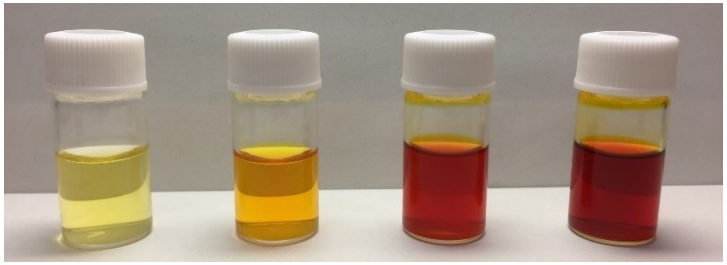
# Solubility of lycopene

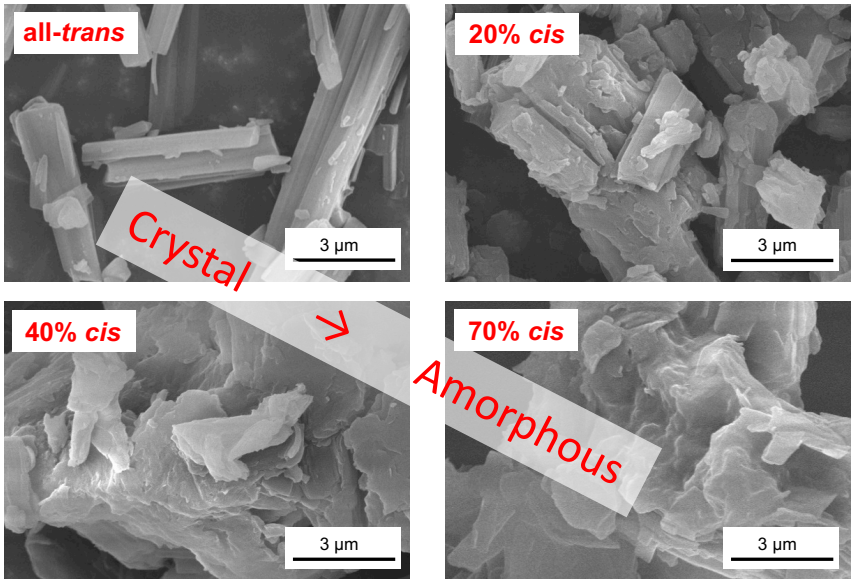
## 【Solubility in methanol】

all-*trans* 20% *cis* 40% *cis* 70% *cis*



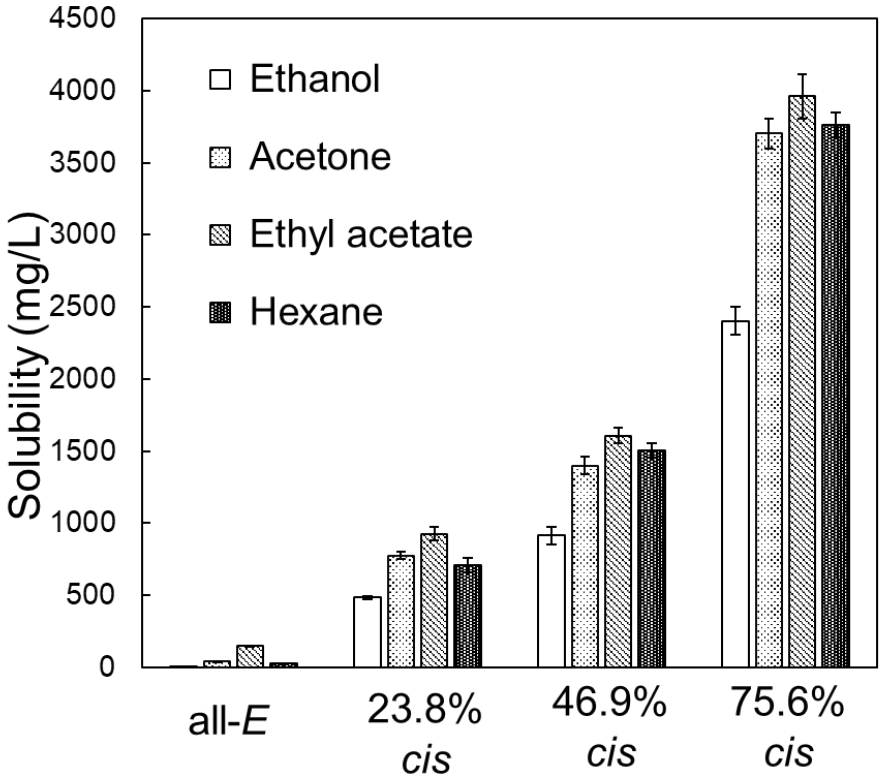
0.1 25.8 179.3 635.1

Solubility (mg/L)




# Effect of *cis*-isomers content

## 【Solubility in various solvent】




Solubility of lycopene in some organic solvents greatly increases with high ratio of *cis*-isomers

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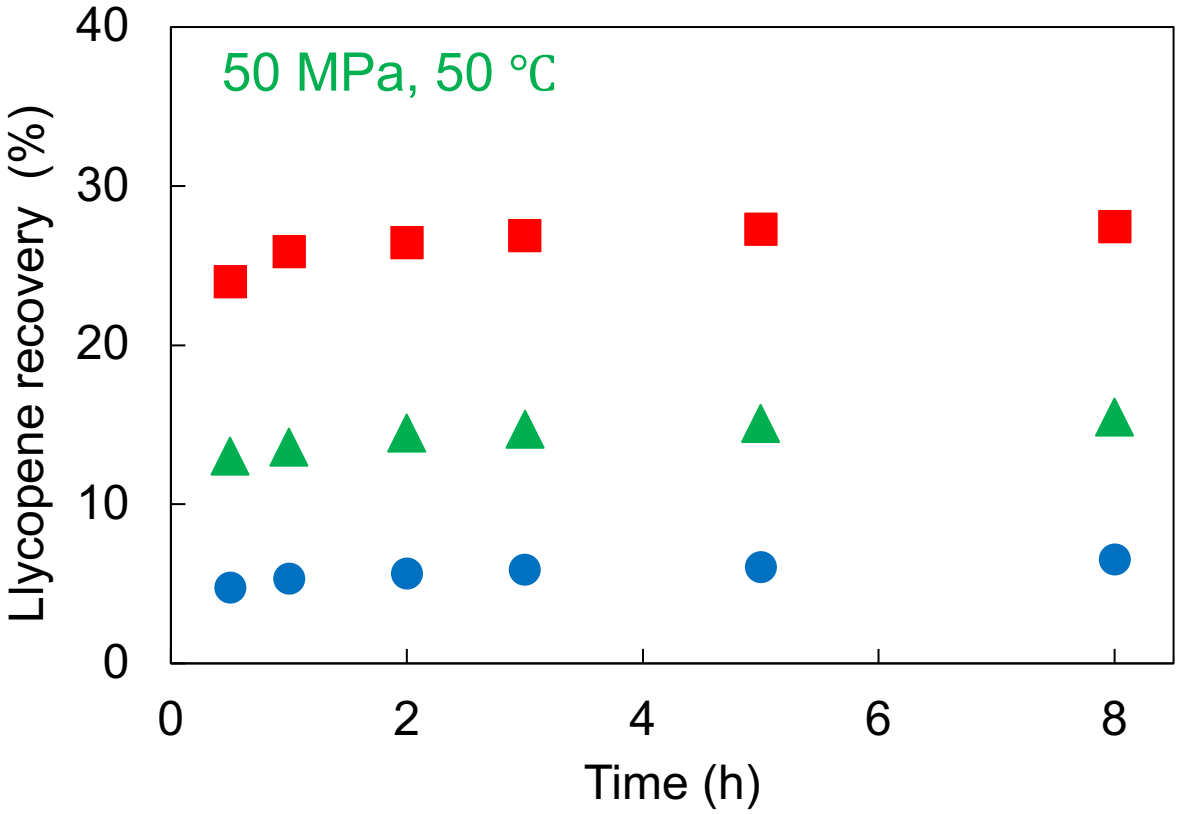
# Supercritical $CO_2$ extraction from tomato

## ■ Change in lycopene recovery




→ 75% *cis* tomato

→ 30% *cis* tomato

→ 10% *cis* tomato

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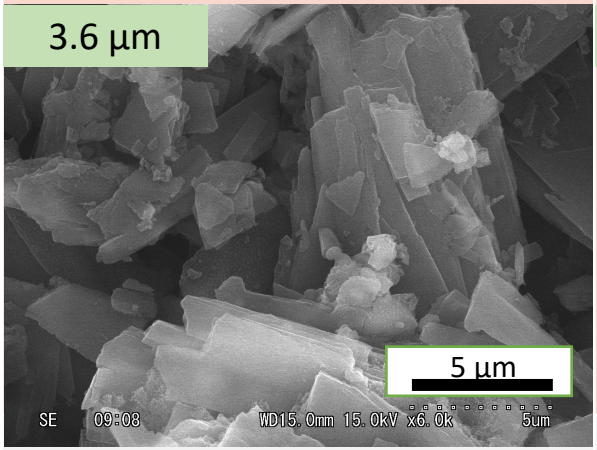
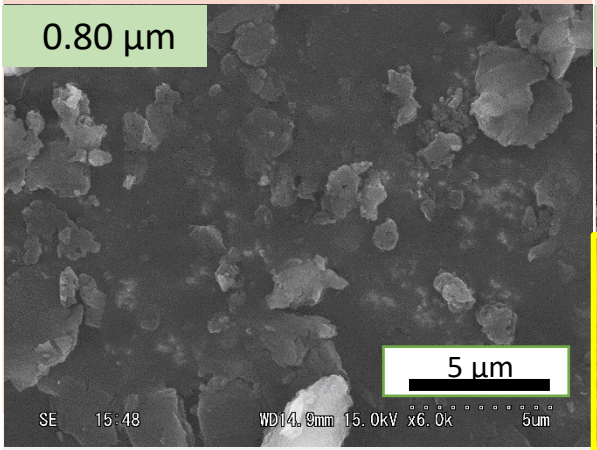
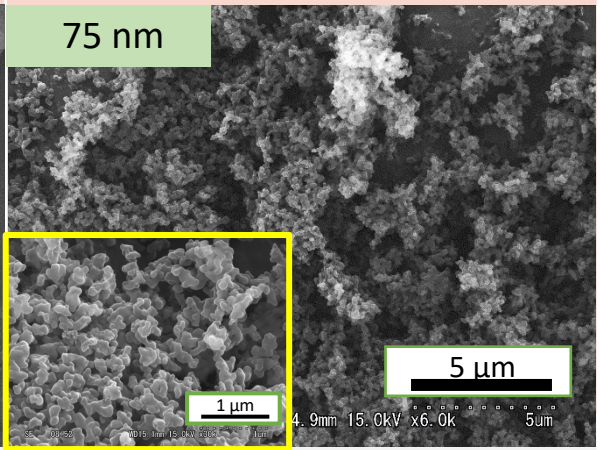
# Micronization of lycopene by SAS process

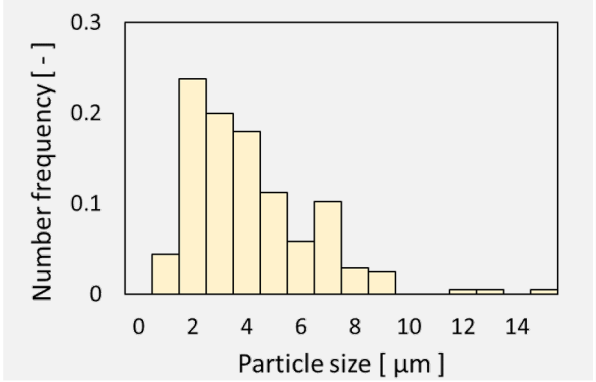
— Solution enhanced dispersion by supercritical fluid (SEDS) —

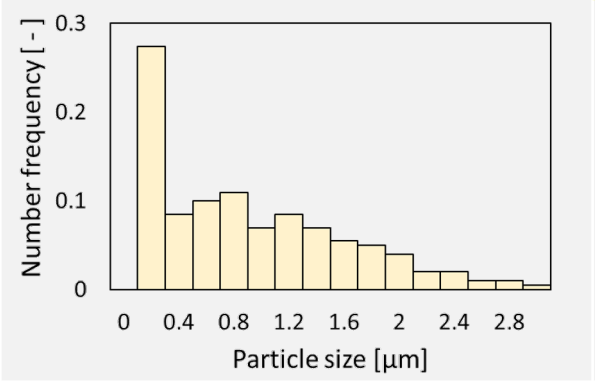
| all-*trans* | 63.5% *cis* | 97.8% *cis* |
|---|---|---|

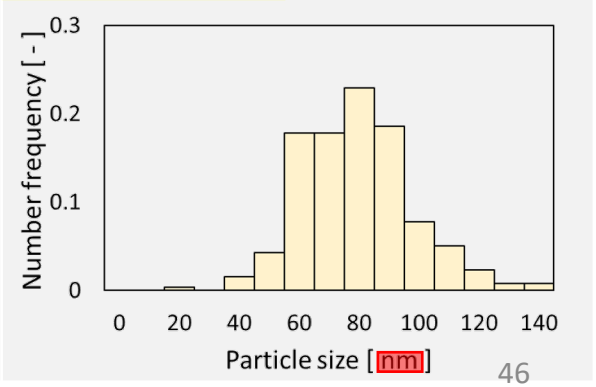


# Isomerization

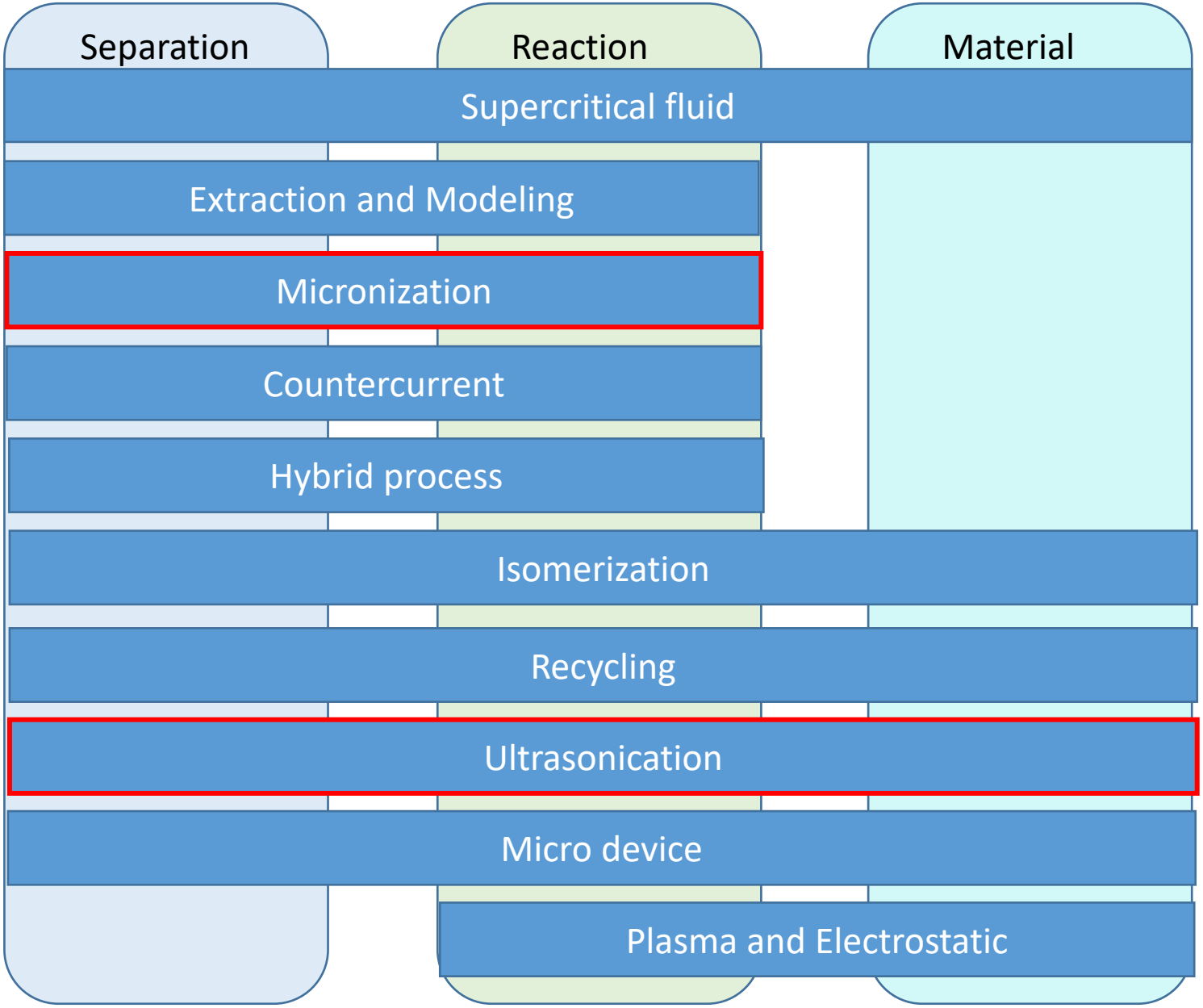
自然を、おいしく、楽しく。
KAGOME

2018年5月14日

<ニュースリリース>

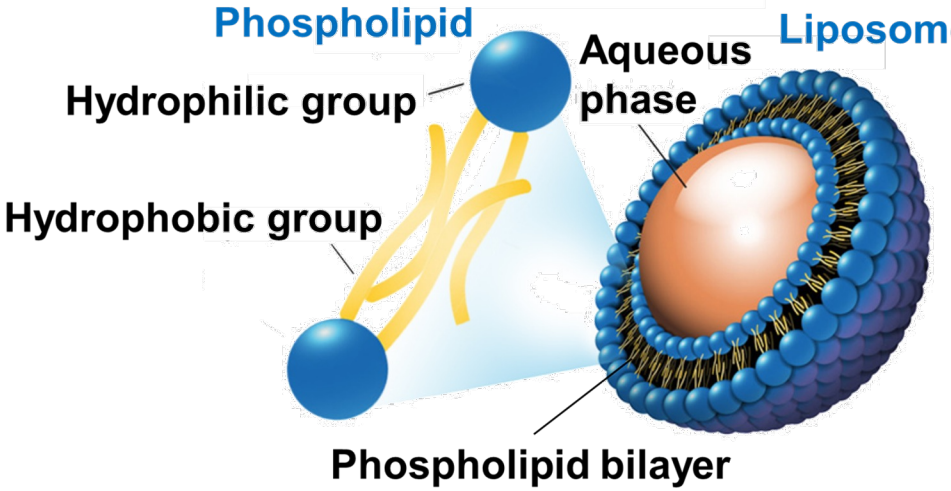
**～カゴメ・名古屋大学　共同研究～**
**トマトに含まれるリコピンの構造変化（＊1）（トランス体からシス体）を促進する新事実を発見**
**トマトをにんにくやたまねぎ、油と一緒に加熱することで、おいしさだけでなく、**
**リコピンが体内に吸収されやすくなることが期待できる**

カゴメ株式会社（社長：寺田直行、本社：愛知県名古屋市）は、名古屋大学大学院工学研究科（愛知県名古屋市）後藤元信教授との共同研究で、**トマトに含まれるリコピンは、にんにくやたまねぎ、油と一緒に加熱することで、体内に吸収されやすい構造への変化（トランス体からシス体）が促進されること、またその促進成分の1つが、にんにくやたまねぎを調理することで生成される香り成分「ジアリルジスルフィド(＊2)」であることを明らかにしました。**なお、本研究内容は化学工学会第83年会（2018年3月13日(火)～15日(木)、関西大学）において発表しました。


Separation
Reaction
Material
Supercritical fluid
Extraction and Modeling
Micronization
Countercurrent
Hybrid process
Isomerization
Recycling
Ultrasonication
Micro device
Plasma and Electrostatic

# Ultrasonic - Liposome -

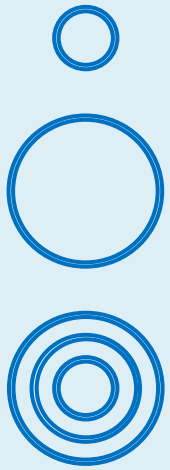
**Ultrasonic-assisted preparation of liposome using supercritical CO2**




## Liposome

| | |
|---|---|
| Component | Phospholipids |
| Shape | Spherical |
| Structure | Lipid bilayer<br>Aqueous compartment |

- Spherical vesicles composed of one or more phospholipid bilayers surrounding discrete aqueous compartments.
- Used as drug carriers in the pharmaceutical, cosmetic and food industry.



**Small unilamellar vesicle**
**SUV (<100 nm)**

**Large unilamellar vesicle**
**LUV (100-1000 nm)**

**Multilamellar vesicle**
**MLV**

## Application

- Biomembrane
- Drug carrier (DDS)



http://www.integratedhealth.com/supplements/liposomes.html. [Accessed: 1-Mar-2018].

# Ultrasonic - Liposome -

## Ultrasonic assisted supercritical fluid method

Supercritical $CO_2$ method

Water containing inclusion substance $W_1$ phase

SC-CO2 phase containing inclusion substance SC-CO2 phase

Ultrasonication

1st Emulsification

$W_1$/Oemulsion

Ultrasonication

2nd Emulsification

$W_1/O/W_2$emulsion

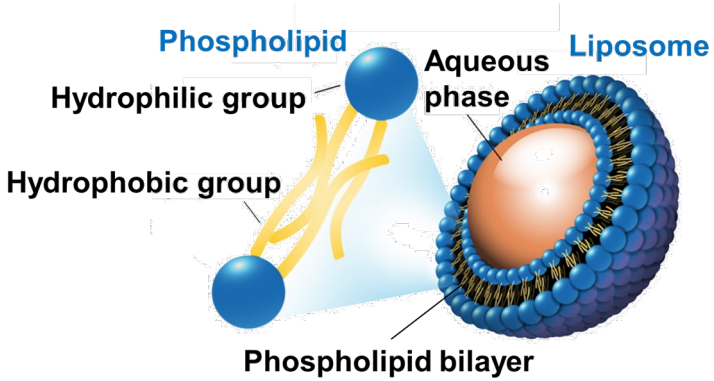
High pressure

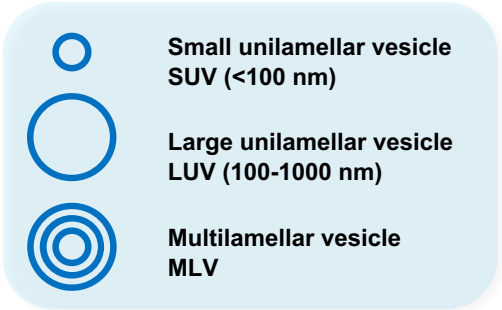
Only pressure reduction

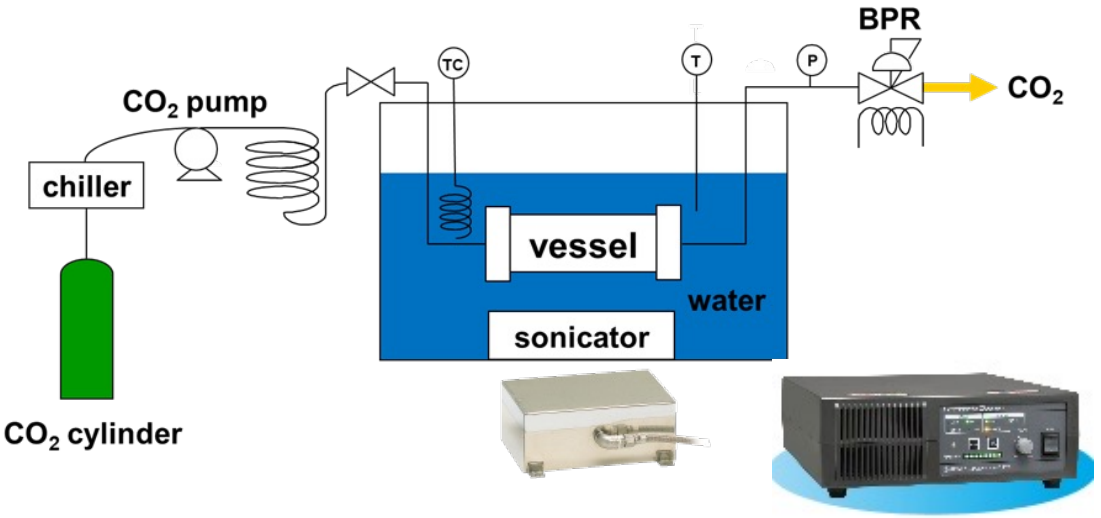
CO2 removal

Liposome

No residue of organic solvent



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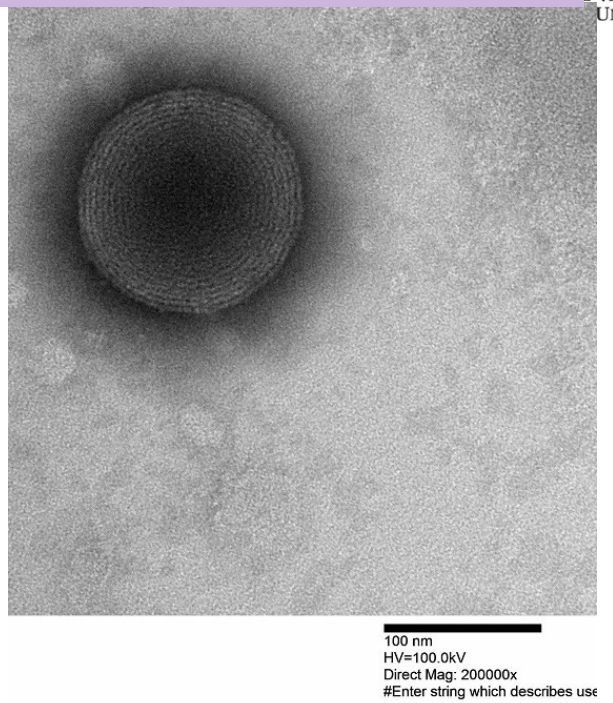
# Liposome

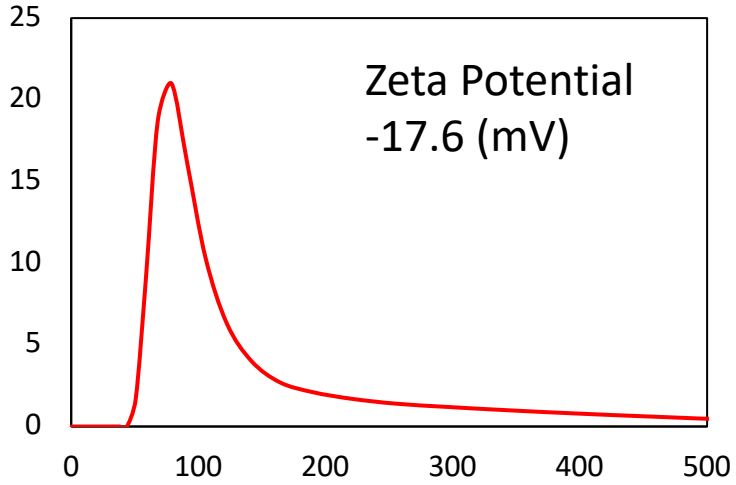
Mixture

Phosphatidylcholine (PC)

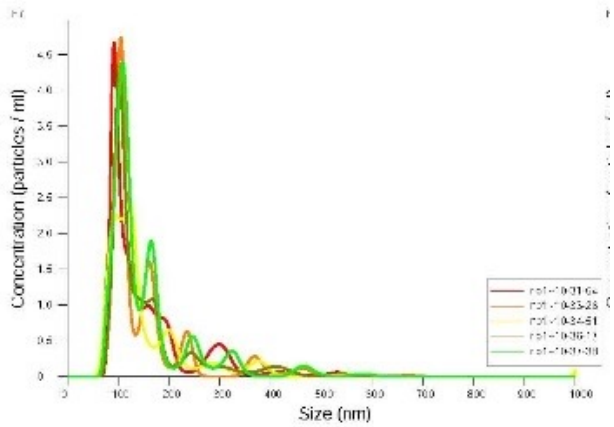
Sphingomyelin (SM)


Dynamic Light Scattering (DLS)




Nanosight




FTLA Concentration / Size graph for Experiment:
np1 2018-08-24 10:31:37

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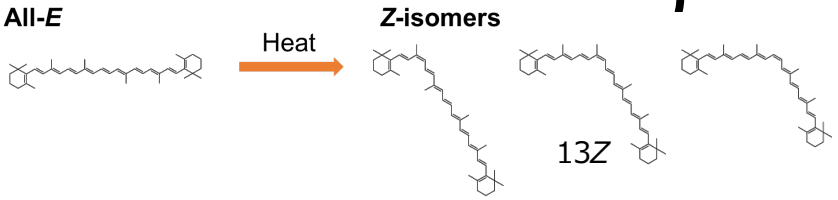
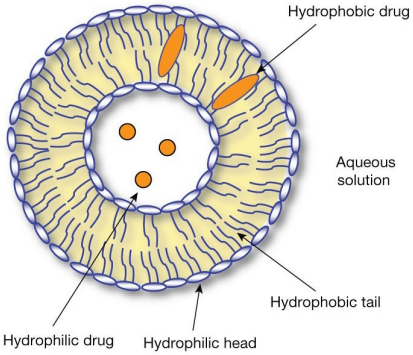
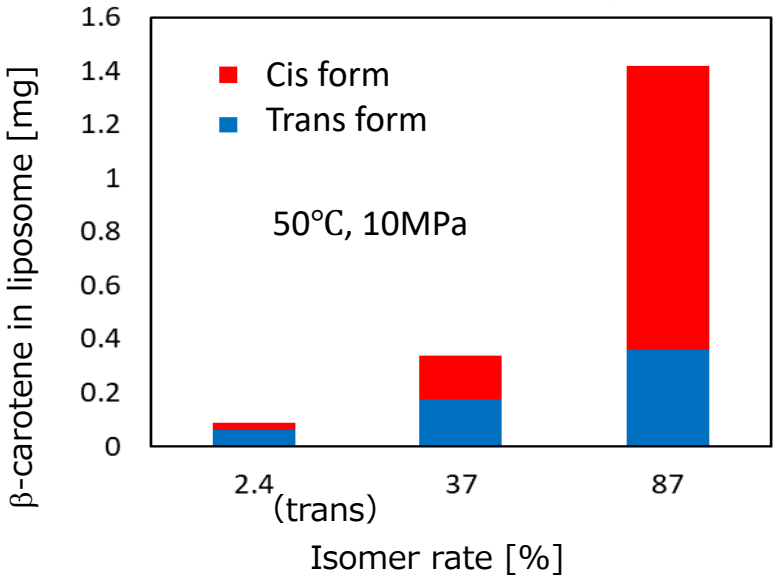
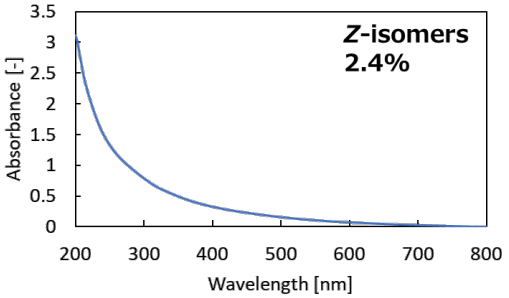
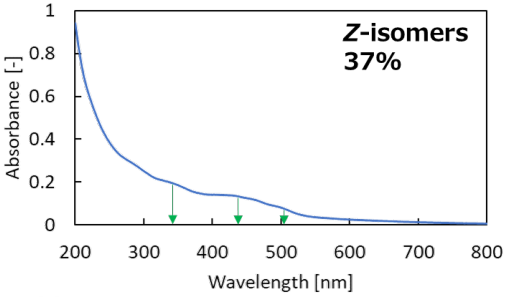
# Effect of cis-isomer ratio on β-carotene content

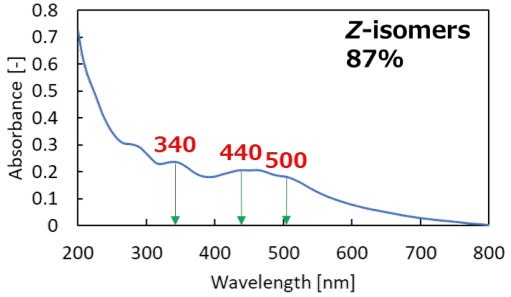


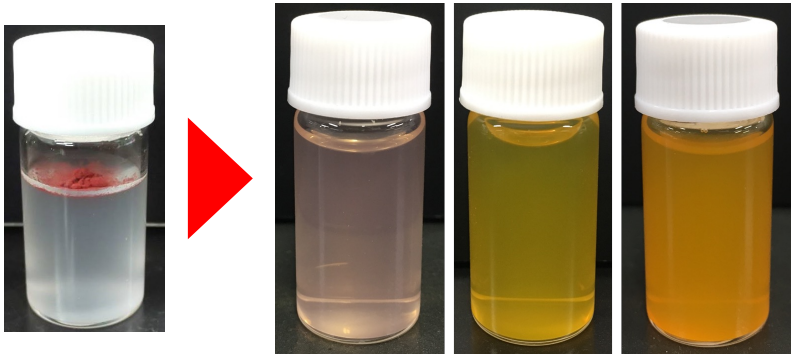



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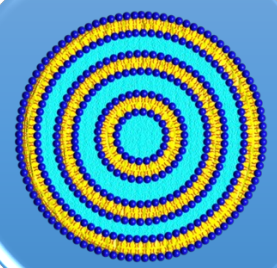
Cosmetics

First Beauty Laboratory SHALOM

株式会社 東洋発酵 TOYO HAKKO Co.,Ltd.

# Application of liposome



ビーンスターク BeanStalk 赤ちゃんのすこやかな笑顔のために

Baby formula (milk)

Medicine

JUNTENDO UNIVERSITY TOKYO 1838

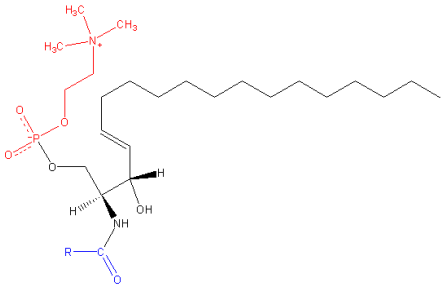
REVERENTIA VITAE KANAZAWA MEDICAL UNIVERSITY

# Ultrasonic - Liposome -

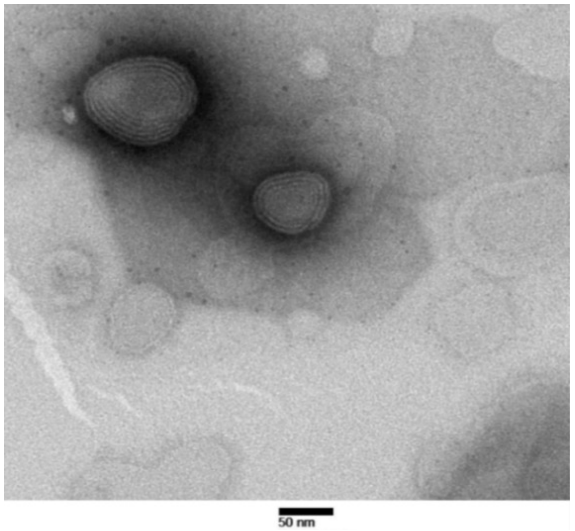
NAGOYA UNIVERSITY

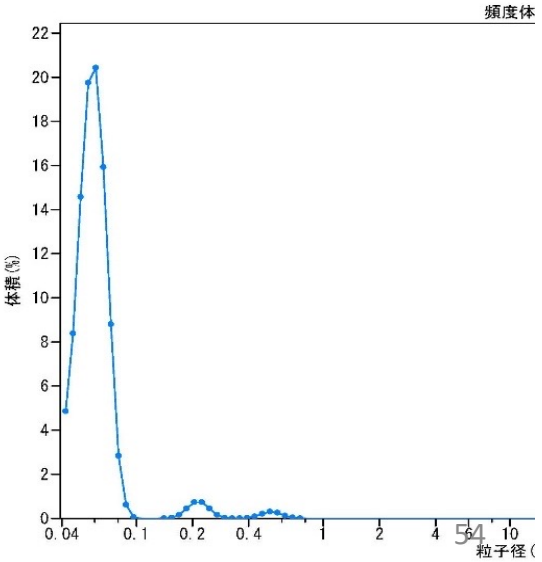
## Bench plant installed in cosmetic company

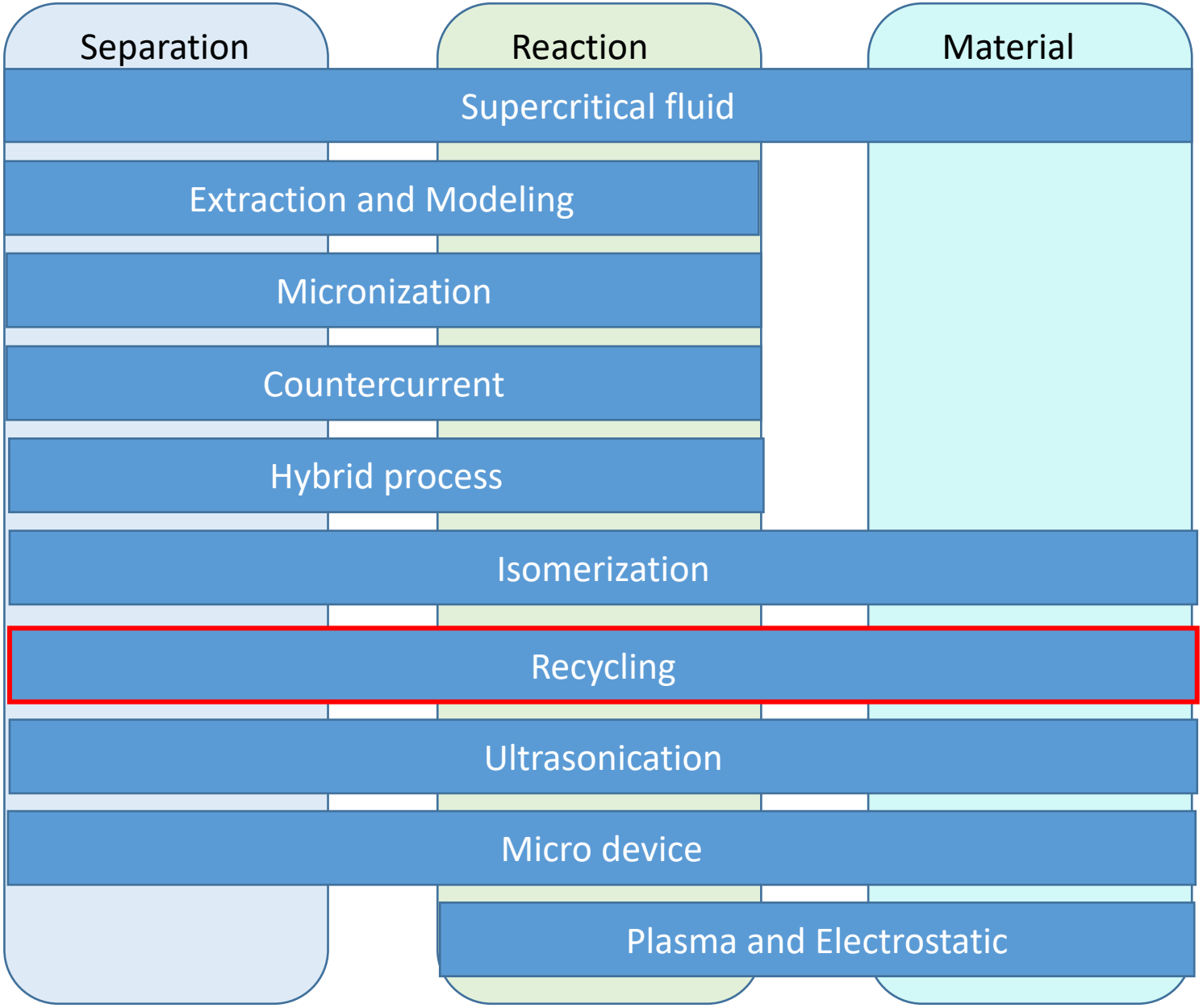
**Sphingomyelin**



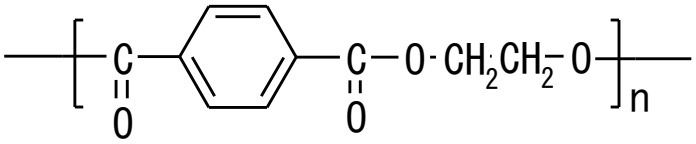
First Beauty Laboratory SHALOM



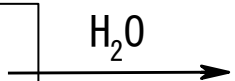

Separation
Reaction
Material
Supercritical fluid
Extraction and Modeling
Micronization
Countercurrent
Hybrid process
Isomerization
Recycling
Ultrasonication
Micro device
Plasma and Electrostatic

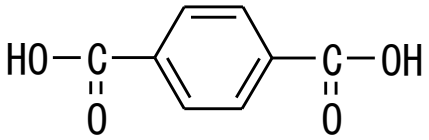
# Depolymerization of plastics using supercritical fluids



PET

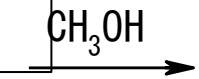
Condensation polymerization polymers
- Ester bond
- Ether bond
- Acid-amide bond

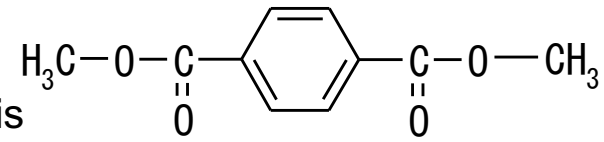


Terephthalic acid

Ethylene glycol



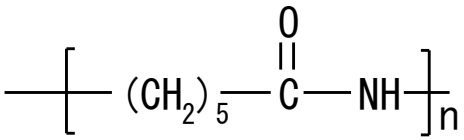



Dimethyl terephthalate

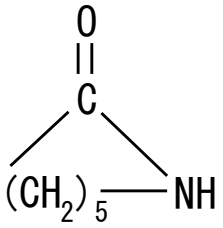


SC-water
SC-alcohol

Solvolysis
- Hydrolysis
- Alcoholysis



Nylon 6

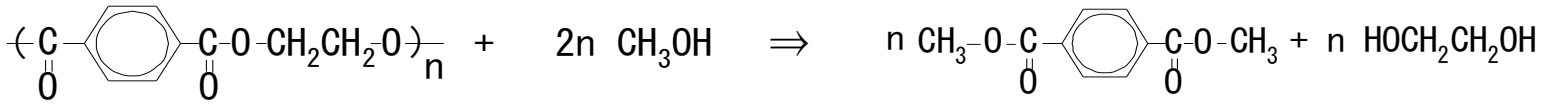



ε-caprolactam

Chemical recycling of waste plastics

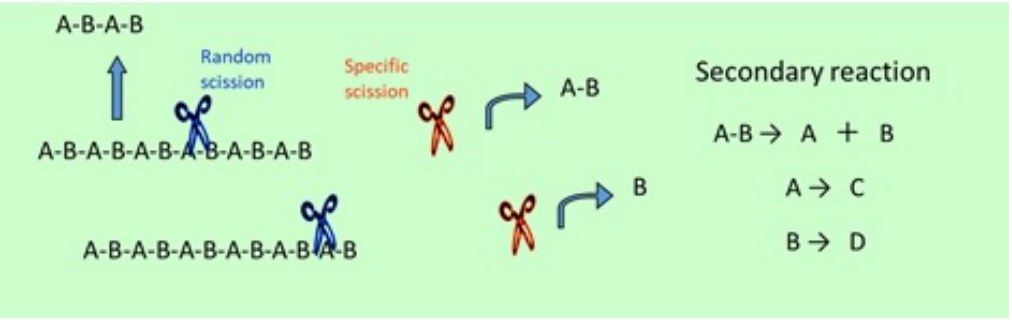
# Reaction - Depolymerization -

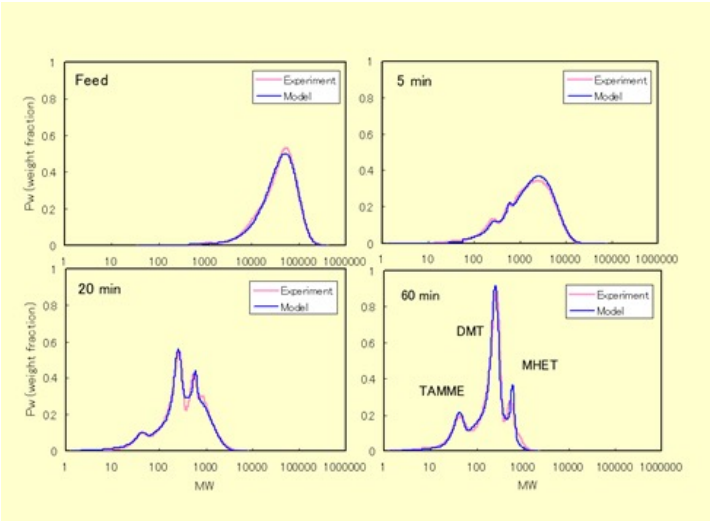
## Depolymerization of PET using supercritical methanol

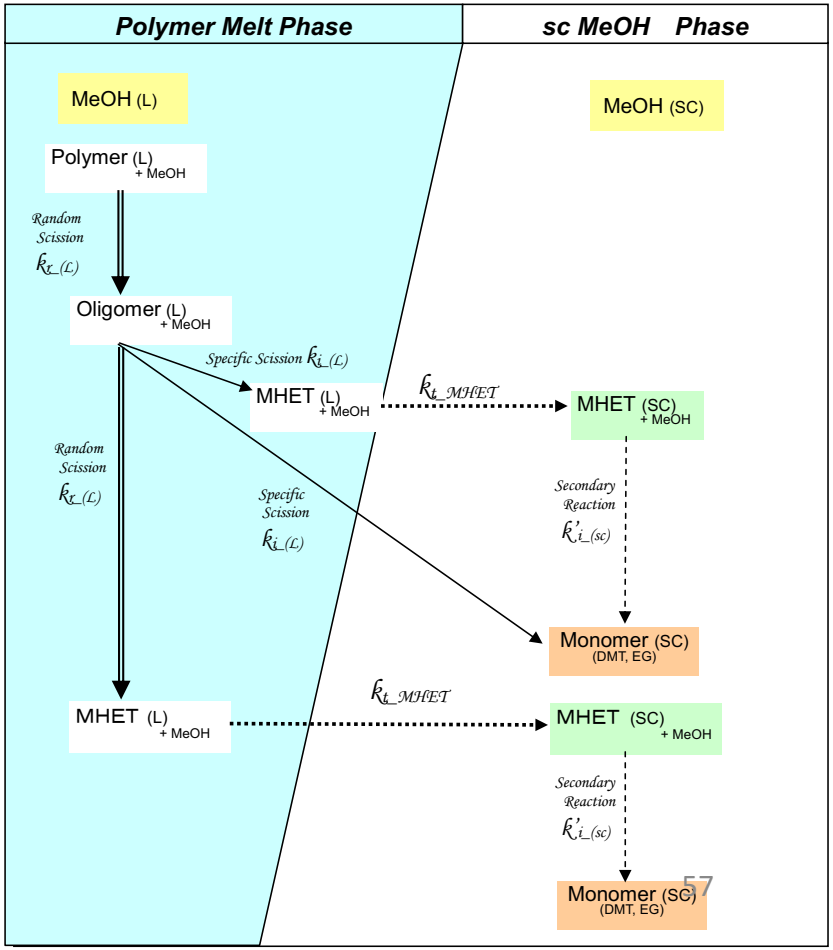


PET Methanol DMT EG

Population balance model (Distribution kinetics)





M. Goto H. Koyamoto A. Kodama T. Hirose, S. Nagaoka, B. J. McCoy, AIChE J., 48, 1, 136-144, 2002
M. Genta, T. Iwaya, M. Sasaki, M. Goto, T. Hirose, Ind. Eng. Chem. Res., 44, 11, 3894–3900, 2005
M. Genta, M. Goto, M. Sasaki, J. Supercritical Fluids, 52, 3, 266-275, 2010

# Reaction - Depolymerization -

NAGOYA UNIVERSITY

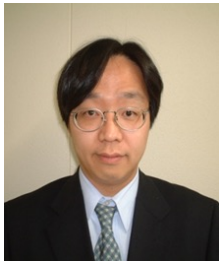
## Depolymerization of plastics using supercritical fluids

Batch test
1998–1999

Laboratory test
2000–2001

Bench test
2001–2002

Pilot test
2003–2005

Dr. Minoru Genta

( A part of construction cost was supported from Ministry of Economy, Trade and Industry, Japan . )

Road to commercialization

MITSUBISHI HEAVY INDUSTRIES, LTD.

# Reaction - Depolymerization -

NAGOYA UNIVERSITY

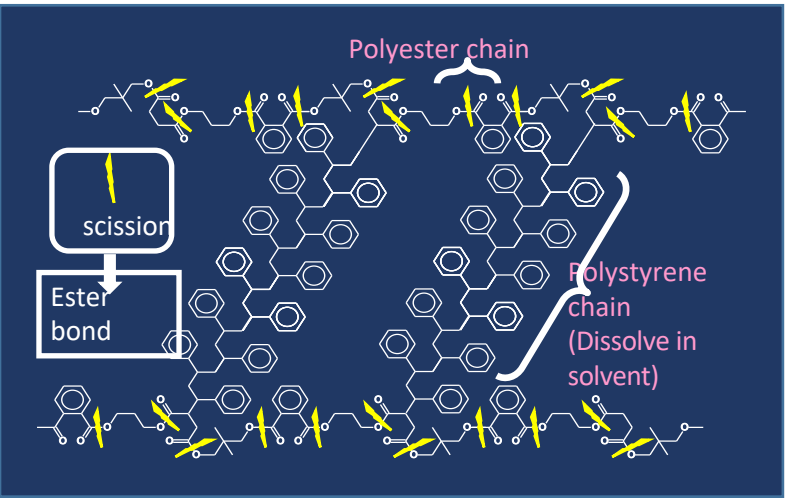
## Fiber reinforced plastics (FRP)

Hitachi Chemical
Working On Wonders

Dr. K. Shibata

Depolymerization in subcritical alcohol to recover fibers

Benzyl alcohol/$K_3PO_4$




200L bench plant
(Hitachi Chem.))

Ambient pressure -> High pressure

J Mater Sci (2008) 43:2452–2456
DOI 10.1007/s10853-007-2017-8

NOVEL ROUTES OF ADVANCED MATERIALS PROCESSING AND APPLICATIONS

**Recycling of fiber reinforced plastics using depolymerization by solvothermal reaction with catalyst**

Tomoko Iwaya · Shinpei Tokuno · Mitsuru Sasaki · Motonobu Goto · Katsuji Shibata

# Reaction - Depolymerization -

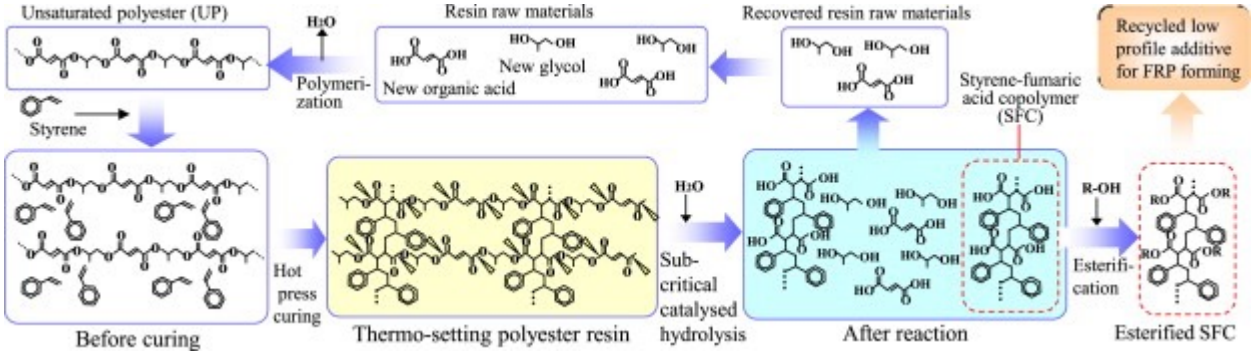
NAGOYA UNIVERSITY

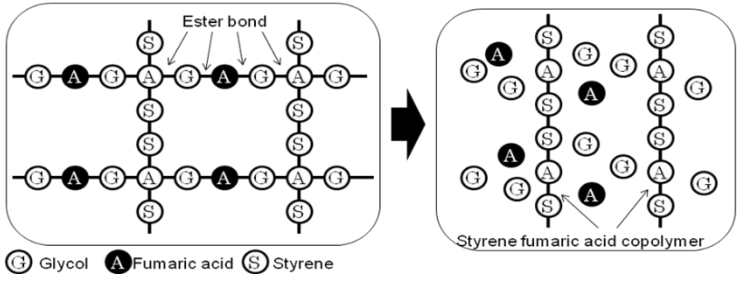
## FRP (fiber reinforced plastics)

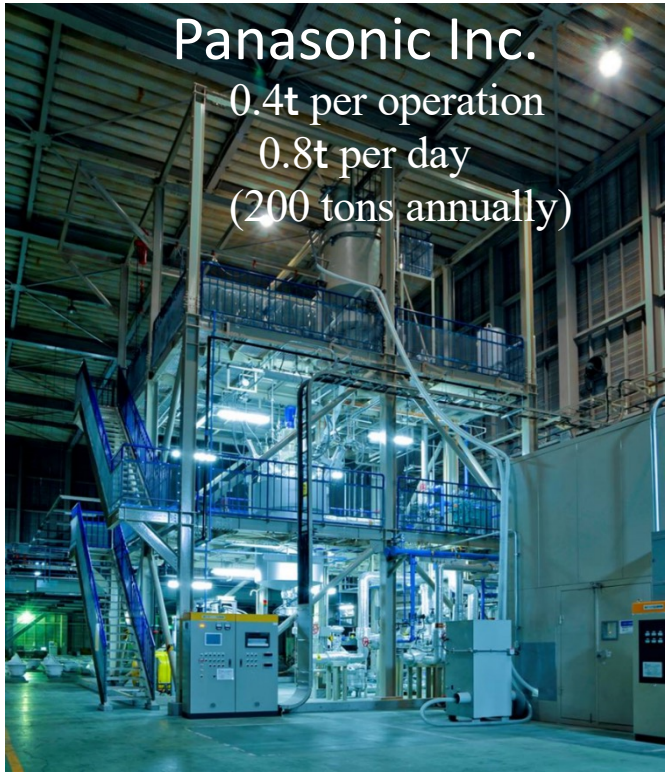
Panasonic

Dr. T. Nakagawa

### Depolymerization in subcritical water for upgrade recycling




Polymer Degradation and Stability 115 (2015) 16–23

Contents lists available at ScienceDirect

Polymer Degradation and Stability

journal homepage: www.elsevier.com/locate/polydegstab

## Recycling thermosetting polyester resin into functional polymer using subcritical water

Takaharu Nakagawa [a, b, *], Motonobu Goto [b]

[a] Eco Solutions Company, Panasonic Corporation, 1048 Kadoma, Osaka 571-8686, Japan
[b] Department of Chemical Engineering, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

# Reaction - Depolymerization -

## 総説論文

J. of Supercritical Fluids 47 (2009) 500–507

Contents lists available at ScienceDirect

The Journal of Supercritical Fluids

journal homepage: www.elsevier.com/locate/supflu

Review

### Chemical recycling of plastics using sub- and supercritical fluids

Motonobu Goto*

*Bioelectrics Research Center, Kumamoto University, Kumamoto 860-8555, Japan*

Green Chemistry

Dynamic Article Links

Cite this: *Green Chem.*, 2011, **13**, 1380

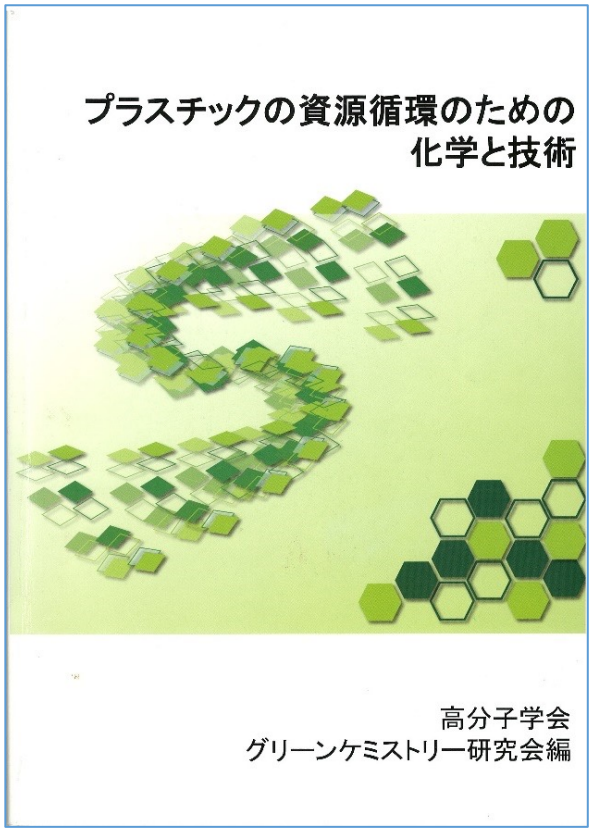
www.rsc.org/greenchem

TUTORIAL REVIEW

### Green materials synthesis with supercritical water†

**Tadafumi Adschiri,*[a,b] Youn-Woo Lee,*[a,c] Motonobu Goto*[d] and Seiichi Takami[b]**

***Received 10th February 2011, Accepted 20th April 2011***
**DOI: 10.1039/c1gc15158d**



# Reaction - Depolymerization -

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## Article Influence

**Recycling**

Renew. Sust. Energy Rev.
**Plastic Waste as Fuel**
Ngadi, 2015
498

Waste Management
**Recycling Carbon Fibers**
Pinho, 2011
565

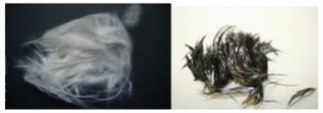
Green Chemistry
**Circular Economy**
Clark, 2016
155

JSF
**Polymer Chemical Recycling**
Goto, 2009
(Rev)
205

Green Chemistry
**Green Materials**
Adschiri, 2011
210

Waste Management
**Mechanical & Chemical Methods**
van Geem, 2017
536

**SCW, scAlcohols**
**PET, PE, FRP**
**Commercial Development**



**Cites**

(Rev) = Review Paper

M. Goto, Chemical recycling of plastics using sub- and supercritical fluids, Journal of Supercritical Fluids 47 (2009) 500-507. https://doi.org/10.1016/j.supflu.2008.10.011

28



By Prof. Richard Smith

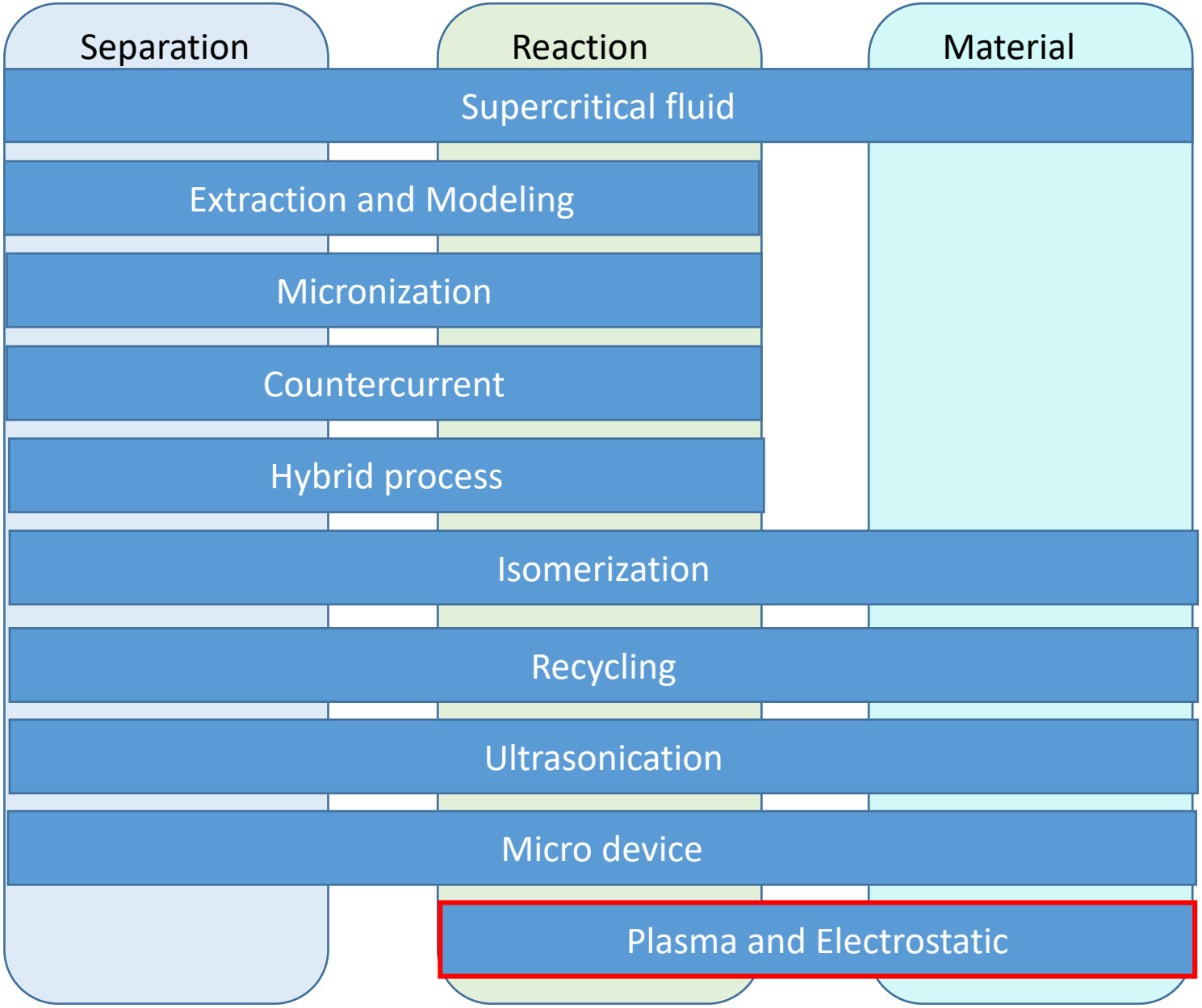
# Event in my researcher life on SCF

Prof. T. Hirose

1988 Nagoya University名大→ Kumamoto University熊大
1988 California University, Davis（15 months)

1993 NATO Advanced Study Institute on Supercritical Fluids（Turkey）
1996 JSPS Research for the Future Program未来開拓学術研究推進事業 【1996-2000】荒井康彦
1997 MESC Scientific Research on Priority Areas重点領域研究【1997-2000】超臨界
2003 21 century COE Program (Kumamoto Univ)【2003-2007】
2006 8th International Symposium on Supercritical Fluids (Kyoto) Organizer
2008 Global COE Program (Kumamoto Univ) 【2008-2012】
2009 MEXT Scientific Research on Innovative Areas新学術領域研究【2009-2013】プラズマ

2012 Kumamoto University 熊大→ Nagoya University名大
2013 Workshop on Supercritical fluids and Energy in Brazil
2013 Super Critical Technology Centre Co. Ltd. 超臨界技術センター㈱ 設立 名大発ベンチャー
2017 Supergreen (10th International Conference on Supercritical Fluids) Nagoya


Separation
Reaction
Material
Supercritical fluid
Extraction and Modeling
Micronization
Countercurrent
Hybrid process
Isomerization
Recycling
Ultrasonication
Micro device
Plasma and Electrostatic

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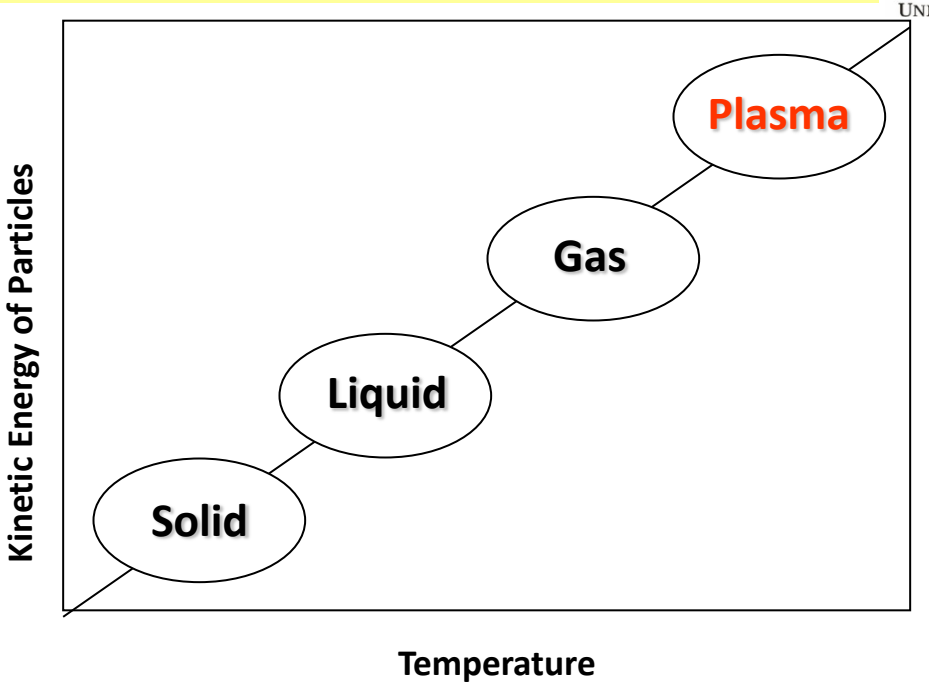
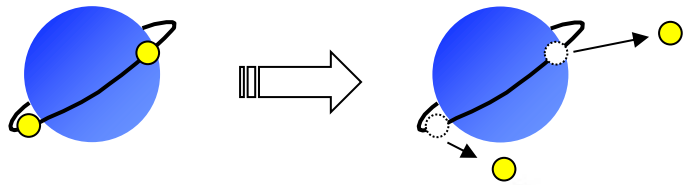
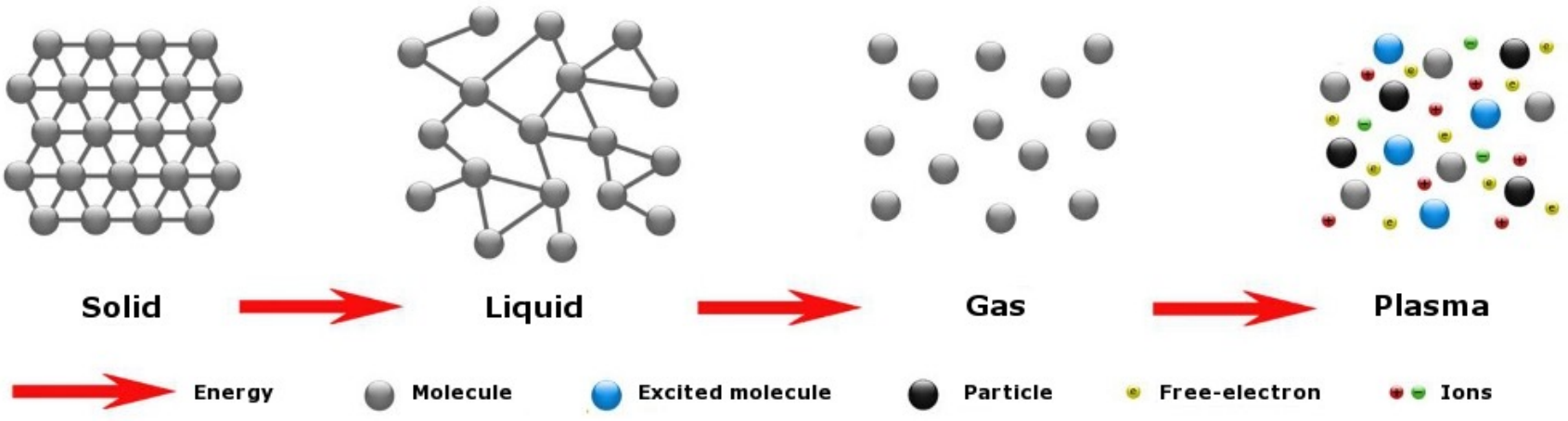
## *Plasma*

Plasma is an ionized gas.

"Ionized" refers to the presence of one or more free electrons, which are not bound to an atom or molecule.

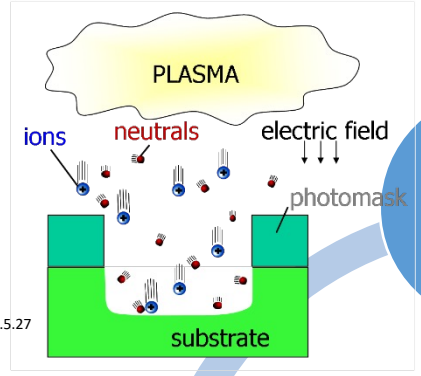


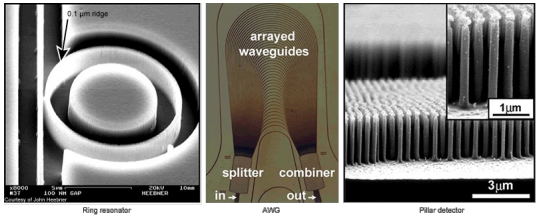



Illuminant, Reactivity, Electrically conductive
Fast particle, High temperature

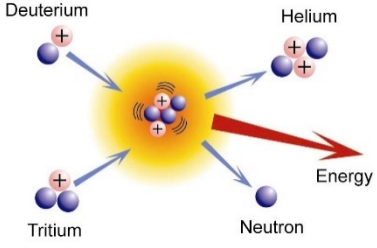
# Plasma

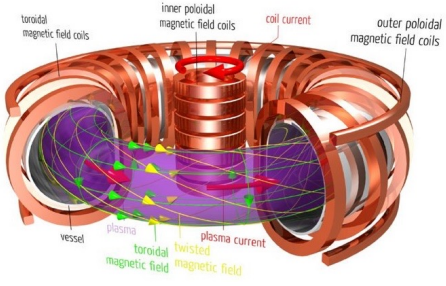


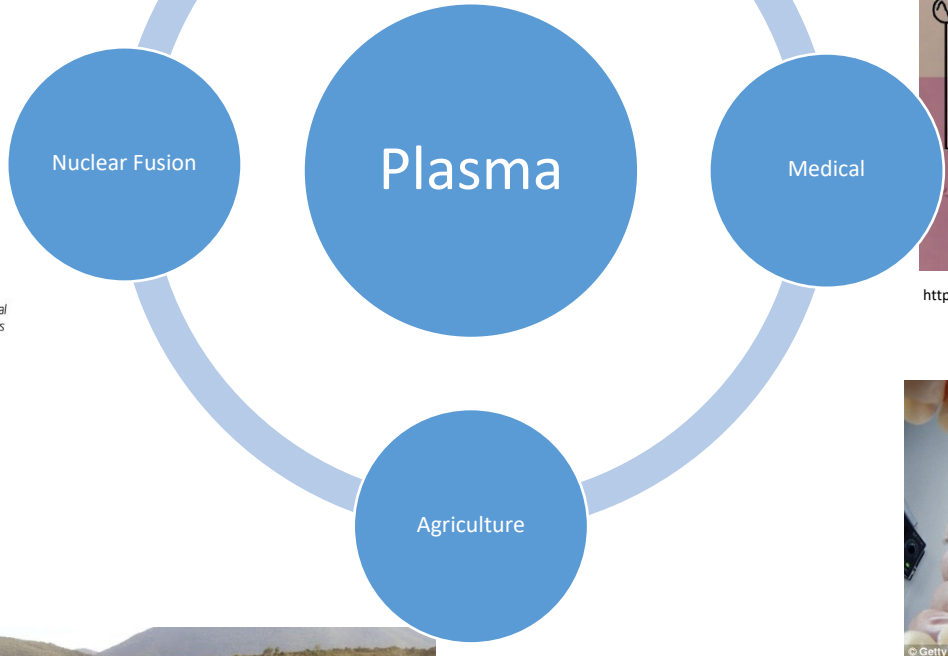

https://scorec.rpi.edu/research_plasmaetchmodeling.php 2022.5.27

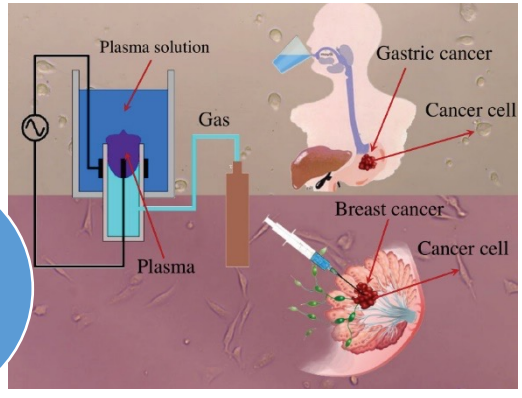



https://sterilisatievereniging.nl/wp-content/uploads/2015/08/2013-03-presentatie-Jan-van-Dijk.pdf 2022.5.27




https://onlinelibrary.wiley.com/doi/abs/10.1002/ppap.201670036 2022.5.27

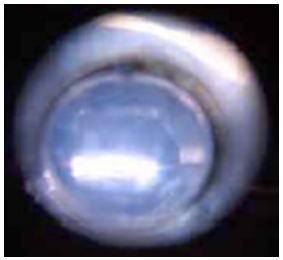

著作権の都合により
画像を削除しました。

プラズマ水と植物の生育の関係を
示した図

NAGOYA UNIVERSITY

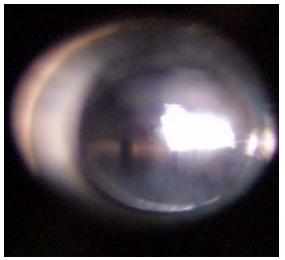
# Discharge plasma in supercritical fluid

***Supercritical $CO_2$***



T = 313K
P = 8.0MPa

***Sub-critical $H_2O$***



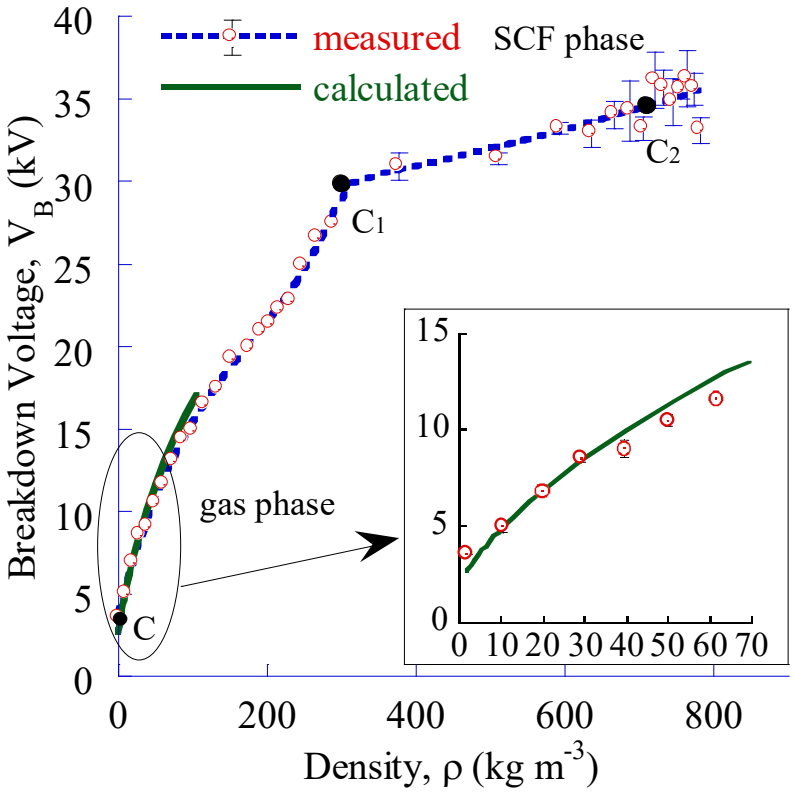
T = 563K
P = 10.0MPa

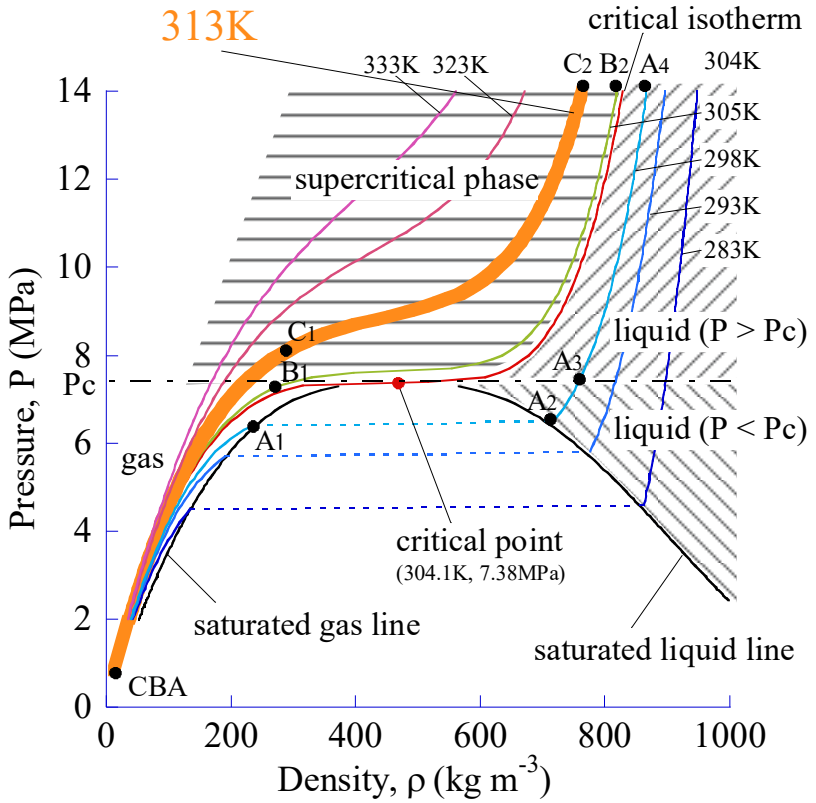
Collaboration with Prof. H. Akiyama group at Kumamoto University since 2003 as 21st COE & GCOE.

Pulse power + Supercritical fluid

NAGOYA UNIVERSITY

# Characterization of Breakdown Voltage Profile and Solvent Density




In supercritical phase, the increasing rate of the breakdown voltage to density becomes small far from liquid phase. In comparison with density curve of $CO_2$, it is observed that the increasing rate of breakdown voltages become smaller as the phase approaches to saturated line.

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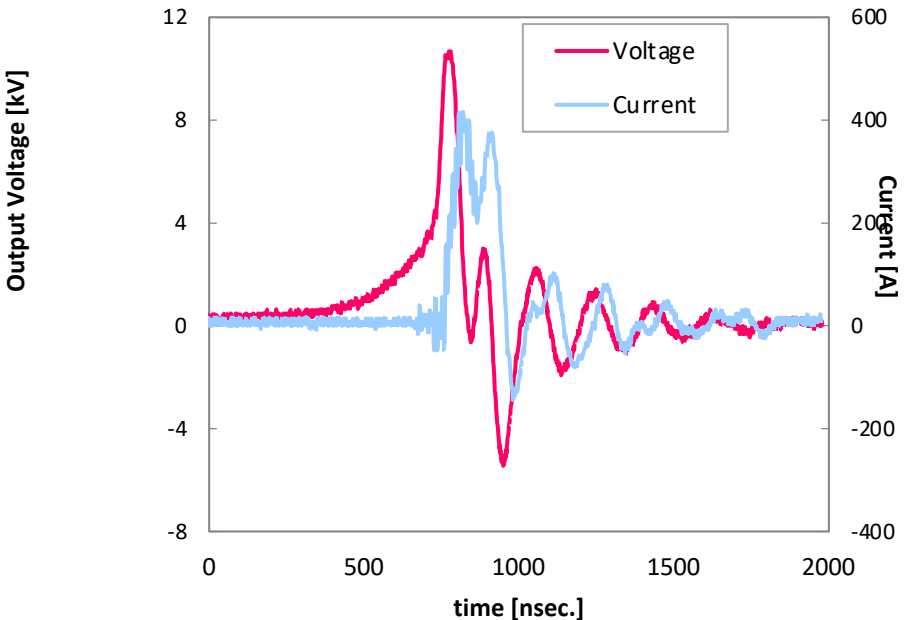
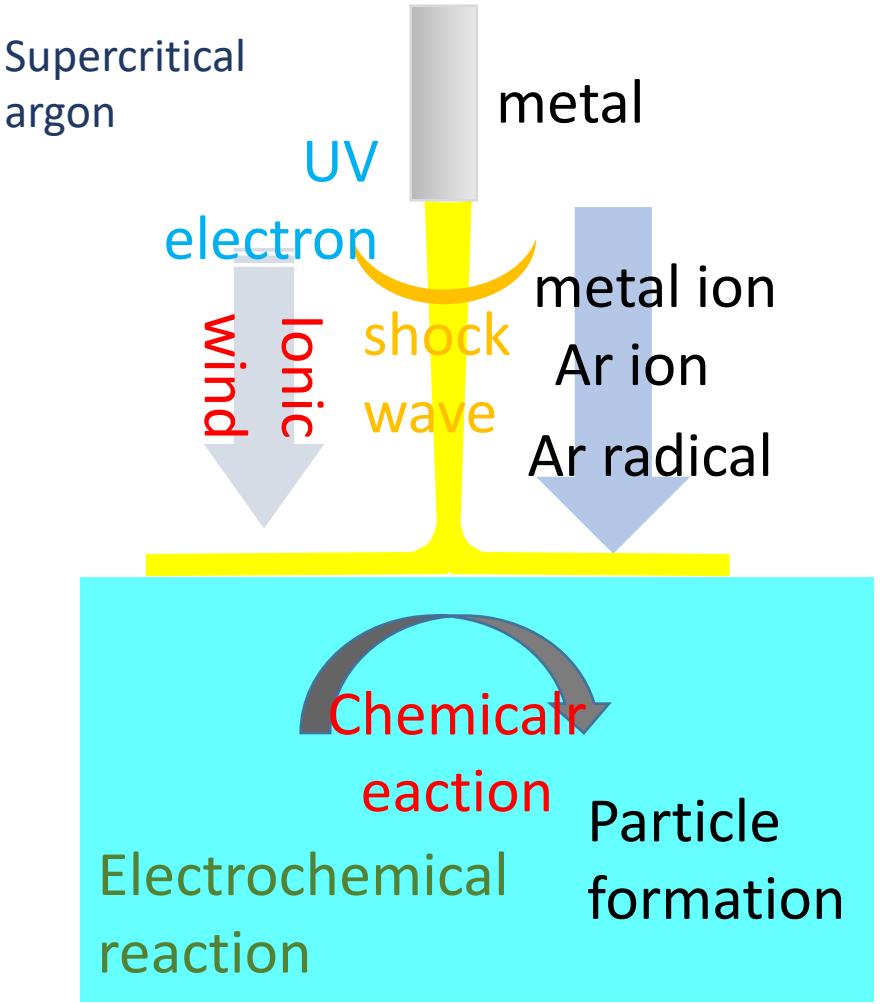
## Discharge plasma at gas/liquid interface

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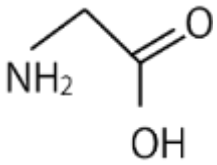
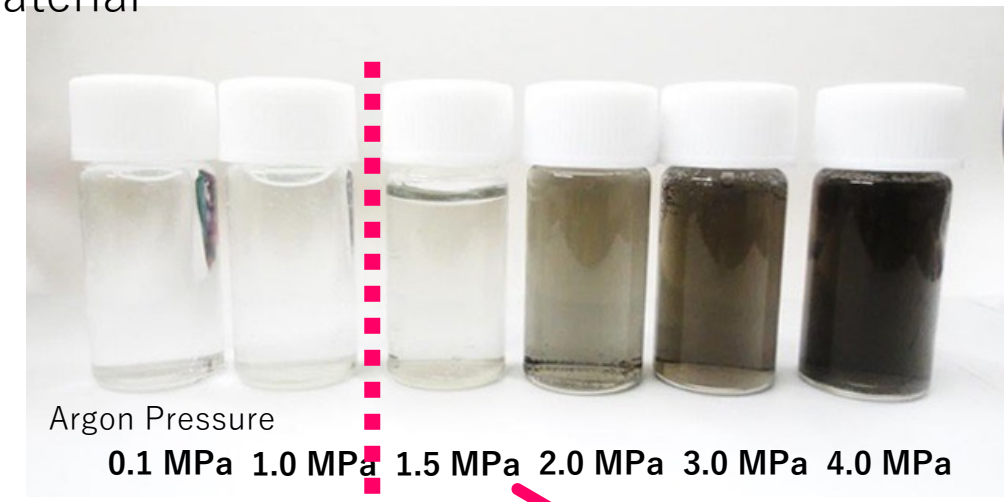
# Pressure dependence of carbon nanoparticle generation

Starting material

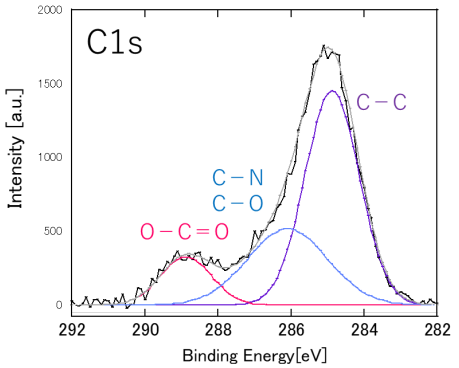



glycine

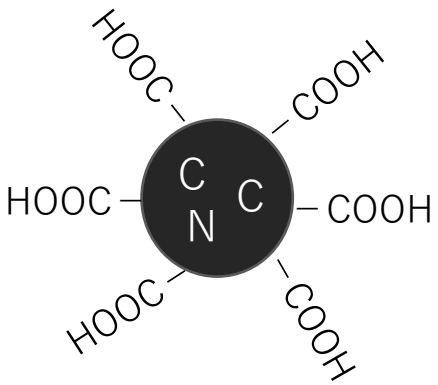



Carbon particles are generated by discharge over 1.5 MPa

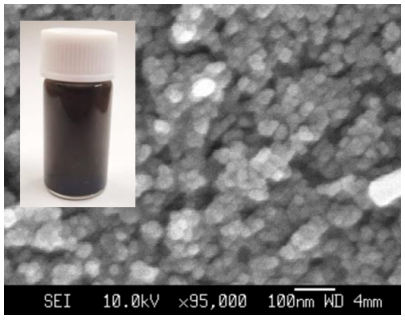
XPS



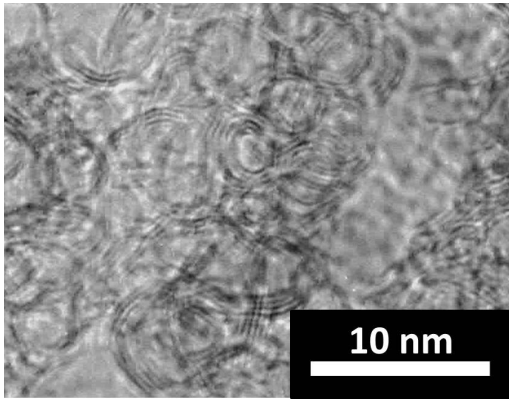

Carbon particle surface is modified with carboxyl group (**–COOH**)



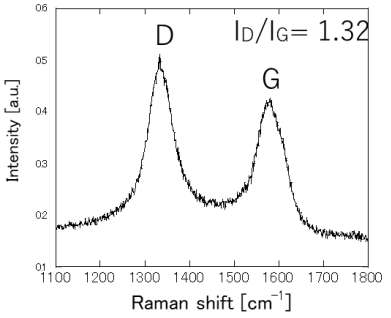

**4.0 MPa (6 month)**




TEM images (4.0 MPa)

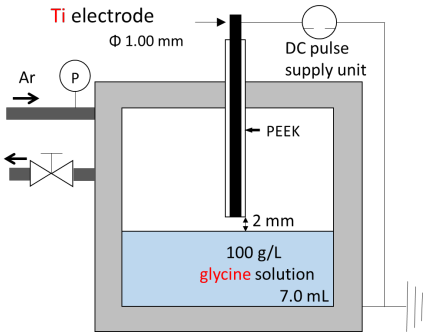


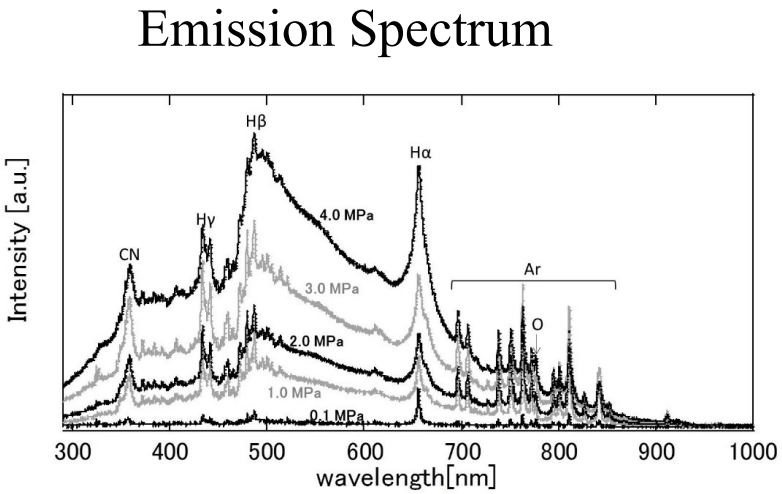

Raman

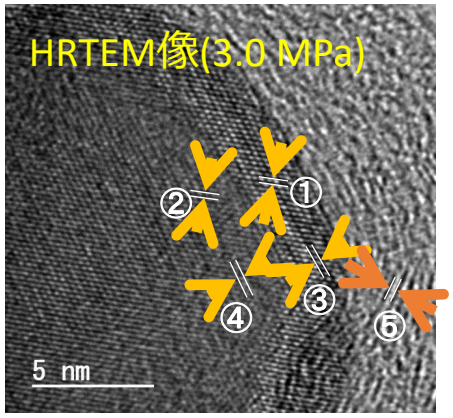


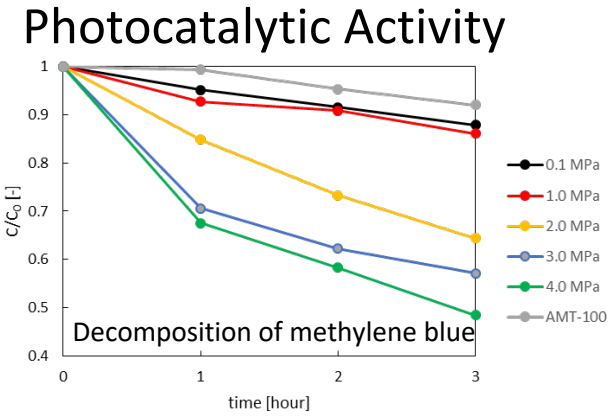
# Plasma

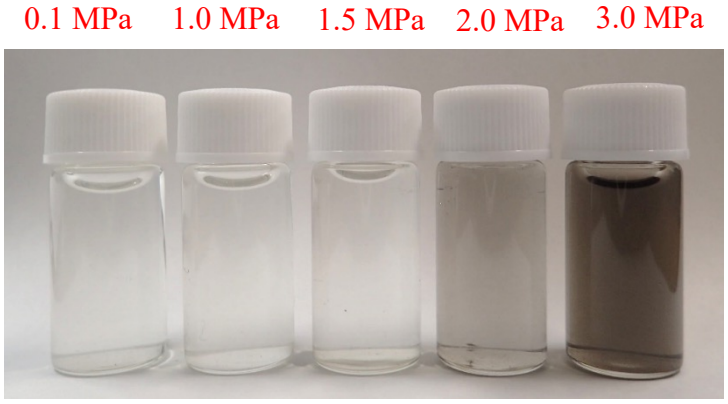
## Titania / carbon composite particle

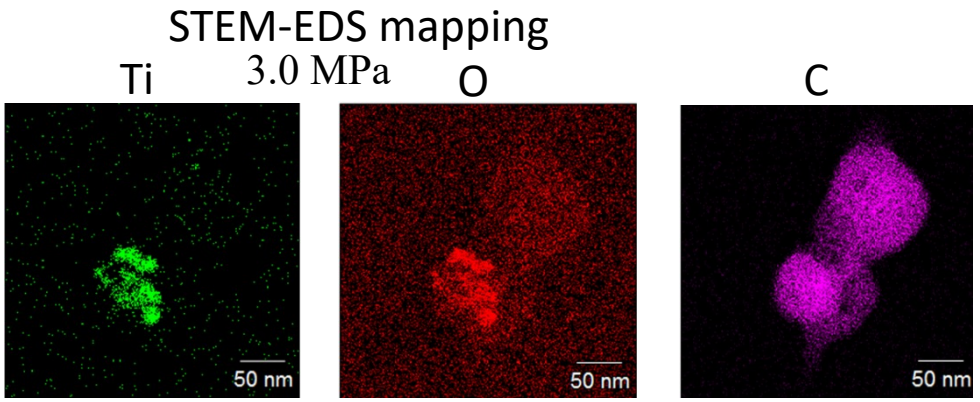


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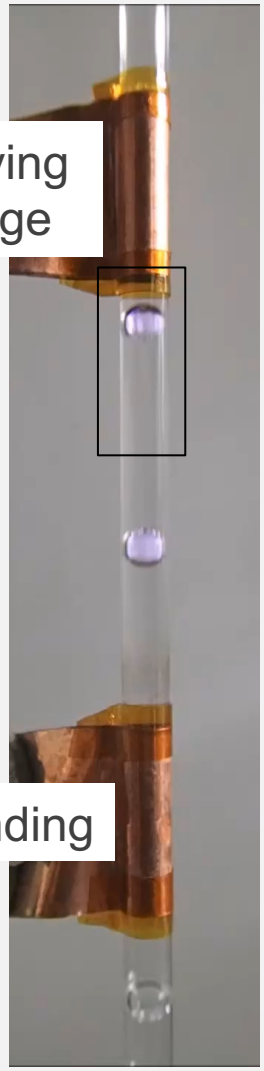
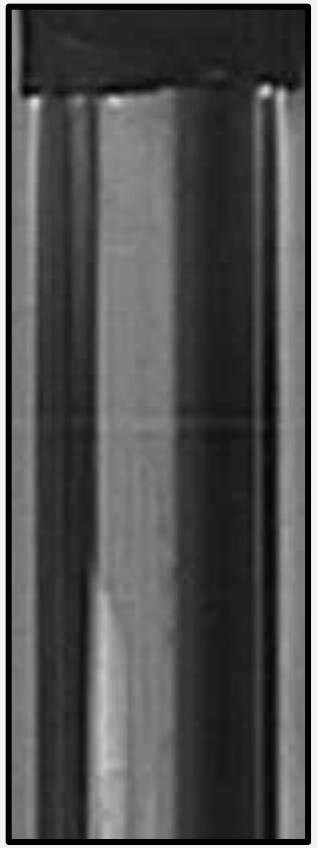
# Plasma at G/L Slug Flow in Capillary Tube



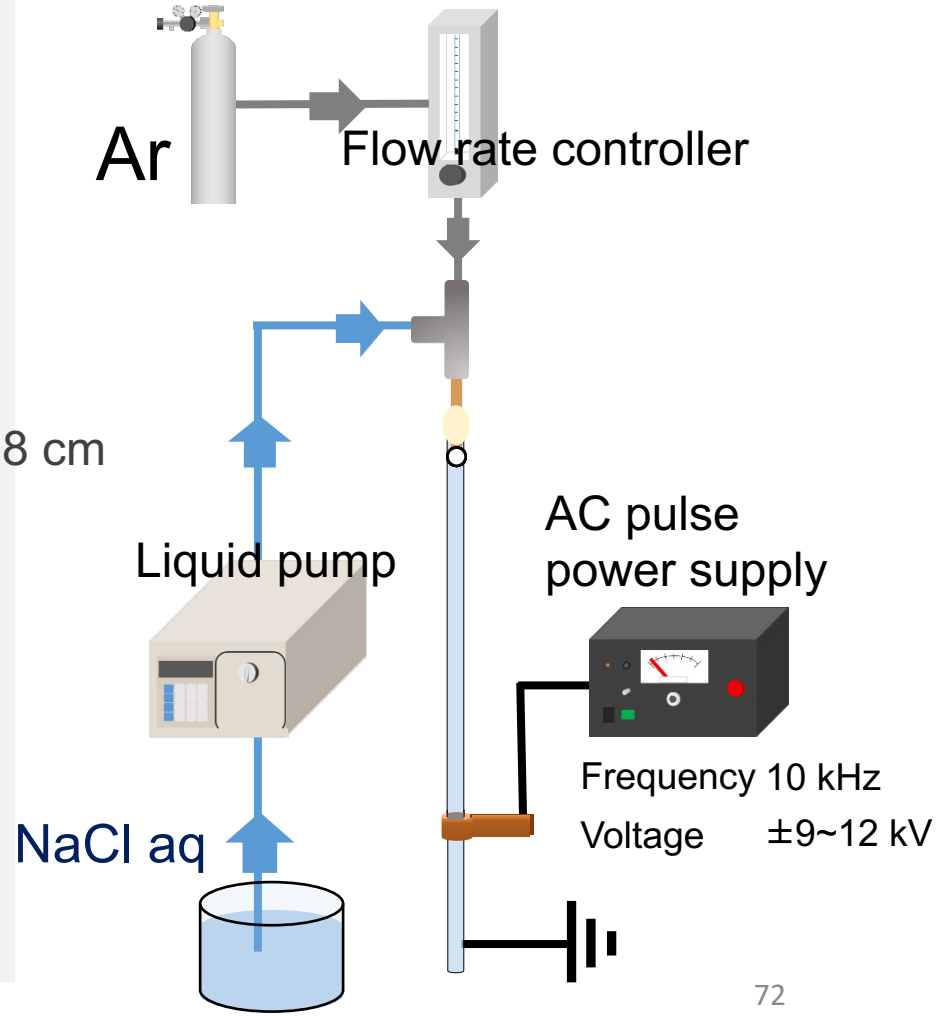

350 pulse/ frame

taken by high speed camera




real time $\times \frac{1}{30}$

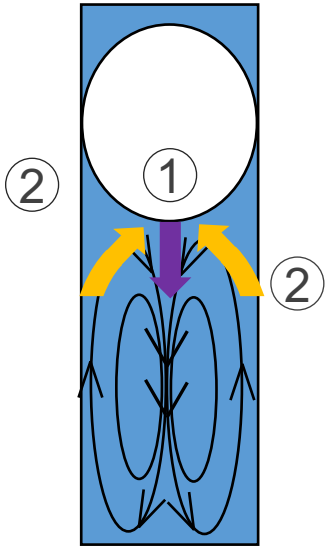
10 pulse/ frame



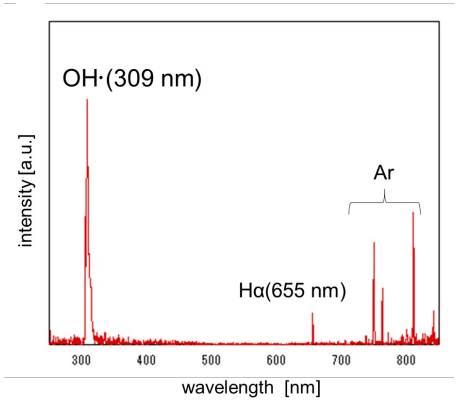
# Plasma

## Internal liquid circulation

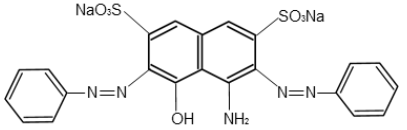
$2I^- \longrightarrow I_2 + 2e^-$




## Emission spectroscopy

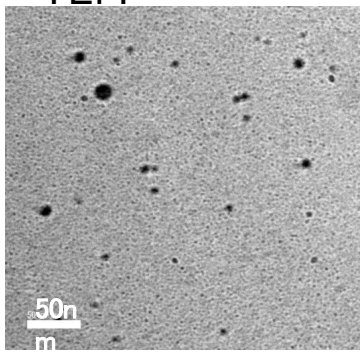



## Decomposition of naphthol blue black

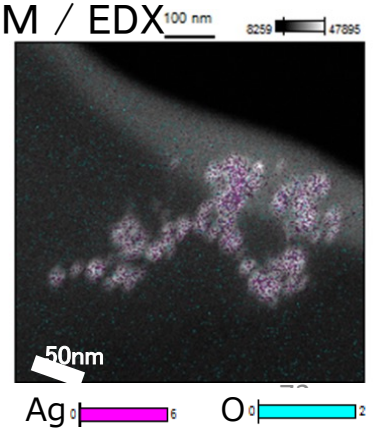



## Ag nanoparticle

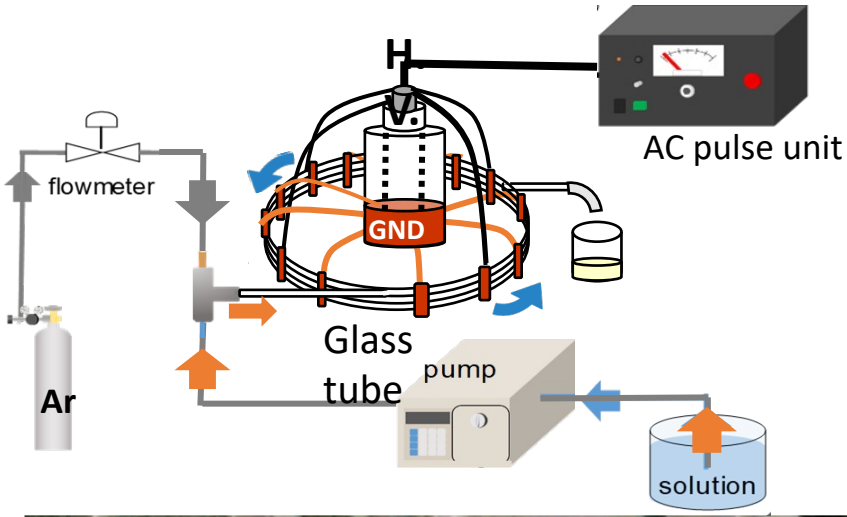
TEM

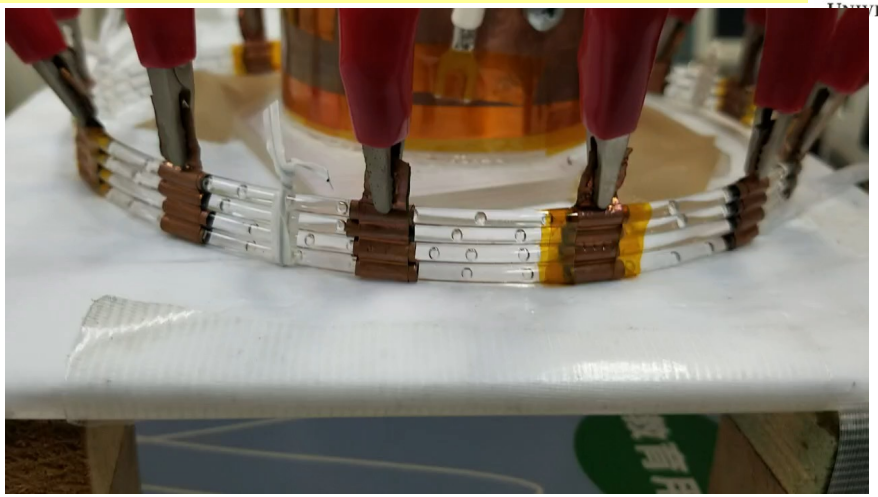


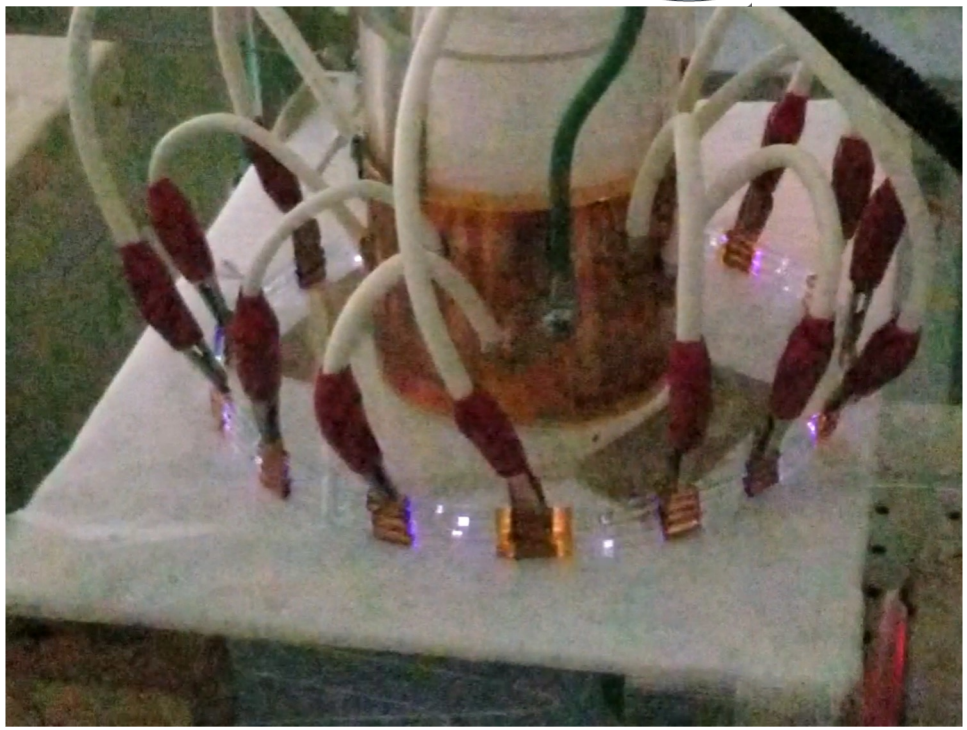

STEM / EDX



# Plasma



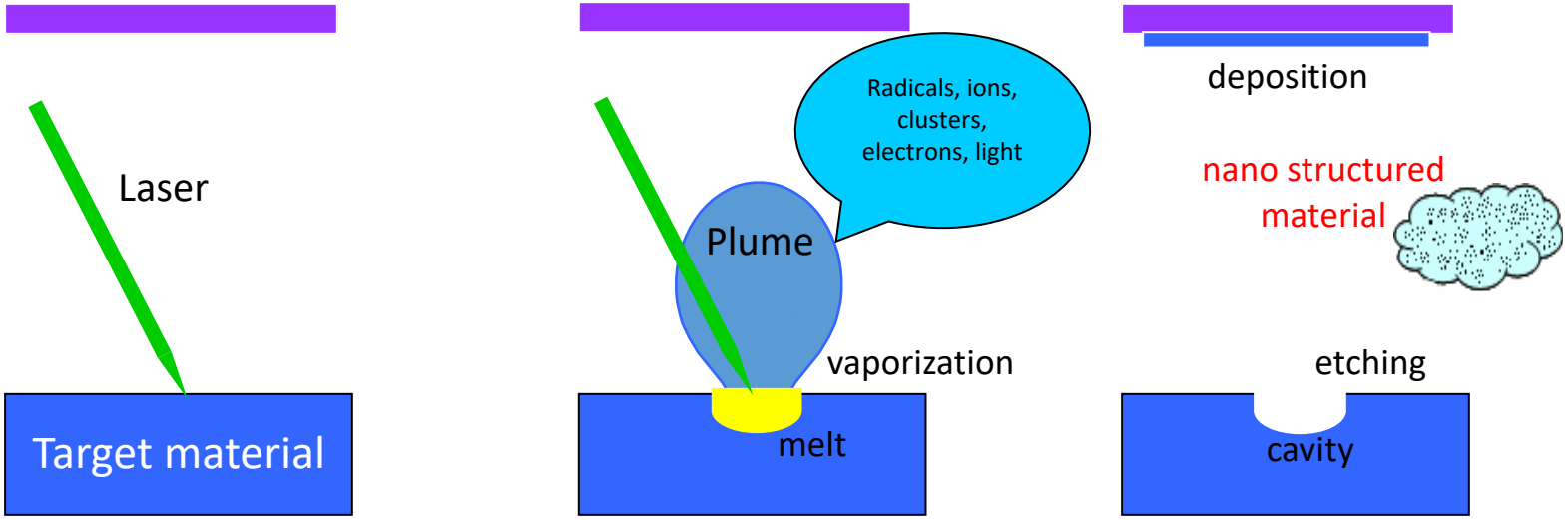



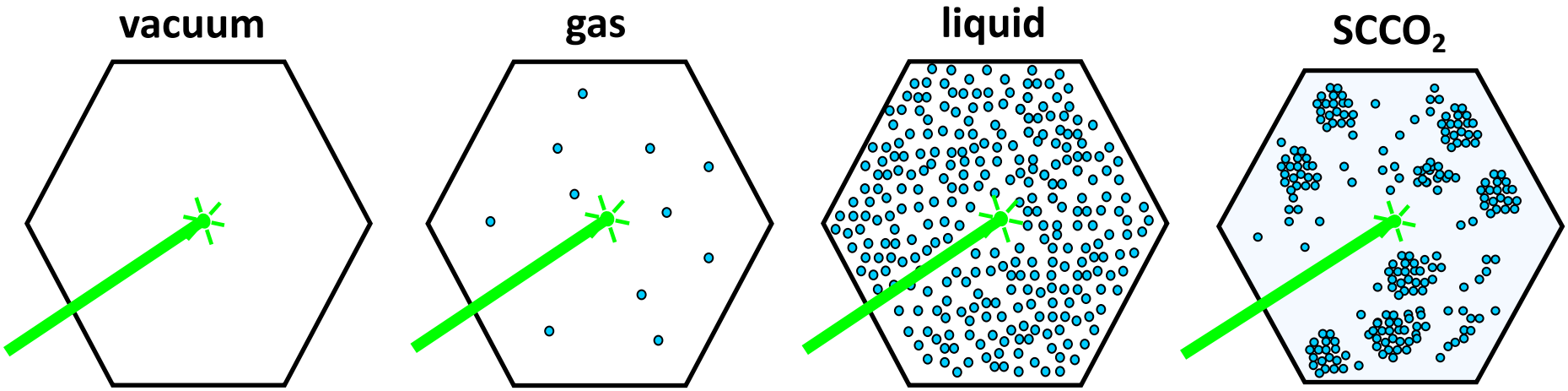


Slug flow plasma in glass column

Flow reactor using circular glass tube with plasma

# Plasma -Pulsed laser ablation-



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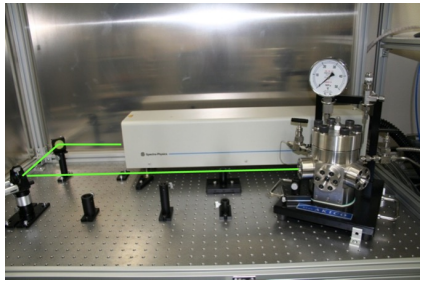
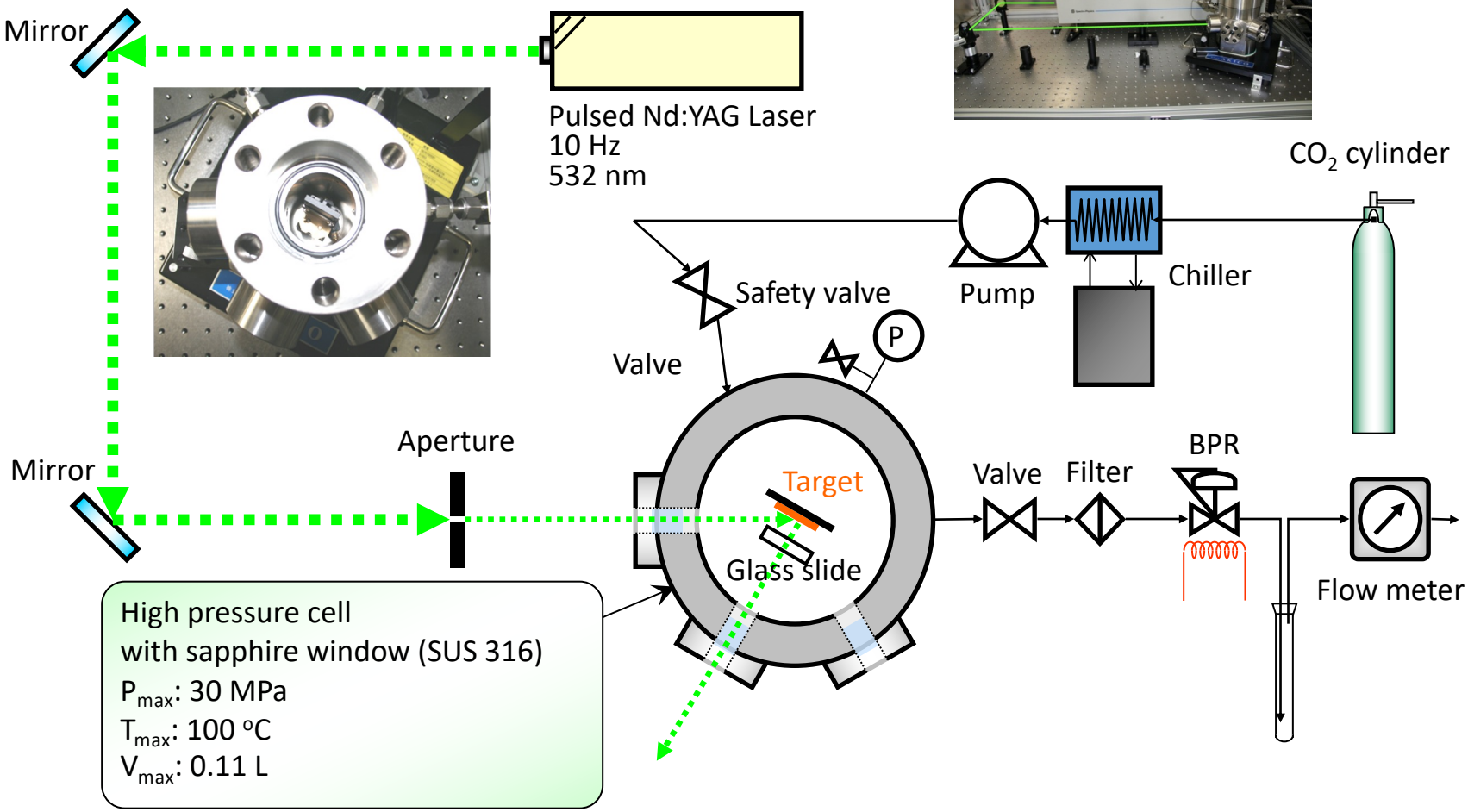
## Laser ablation in supercritical $CO_2$

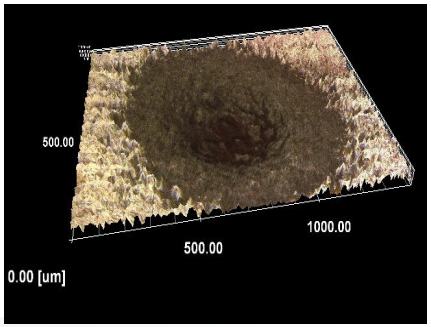


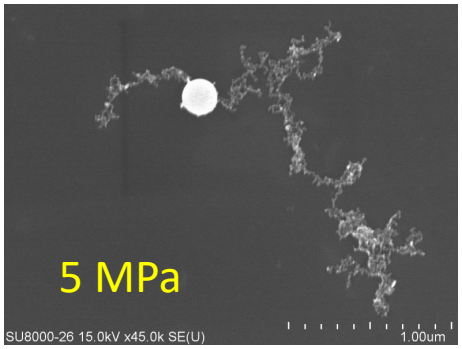


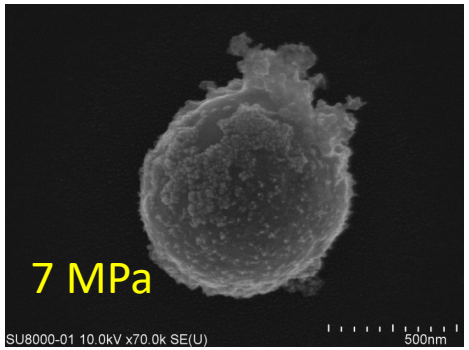

**Experimental Apparatus**

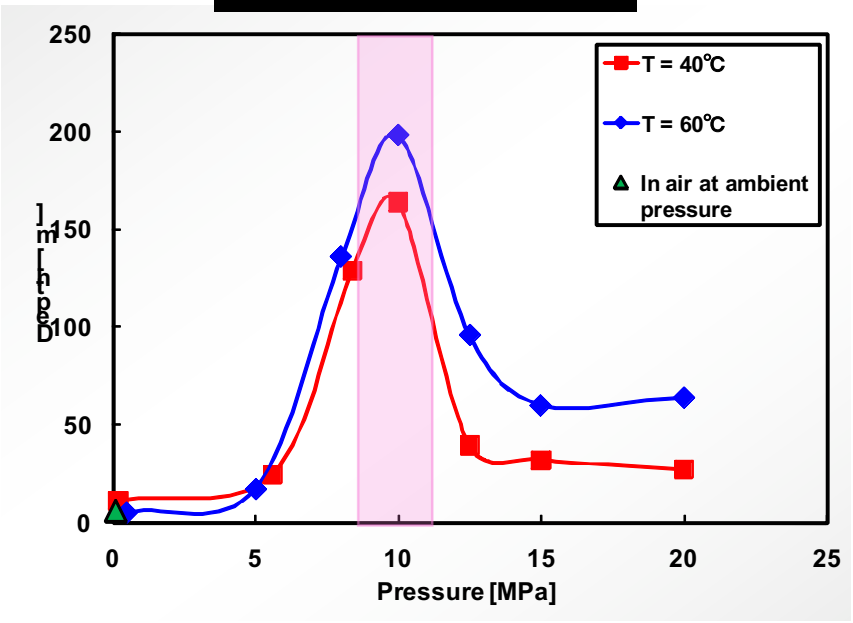
# Plasma -Pulsed laser ablation-

## Effect of pressure



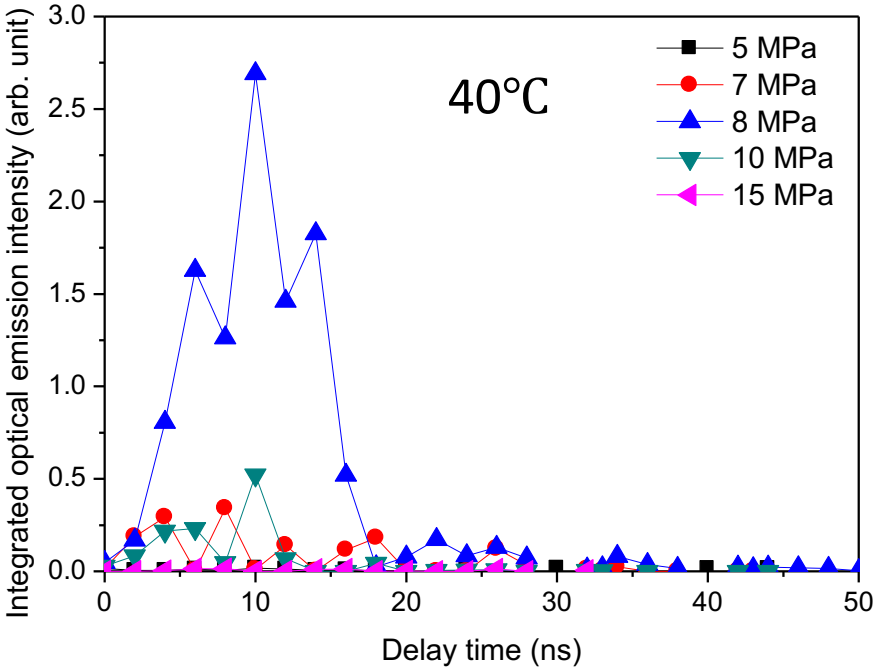




Depth of irradiated gold plate increased as increasing temperature.

## Optical Emission Intensity



# Plasma -Pulsed laser ablation-

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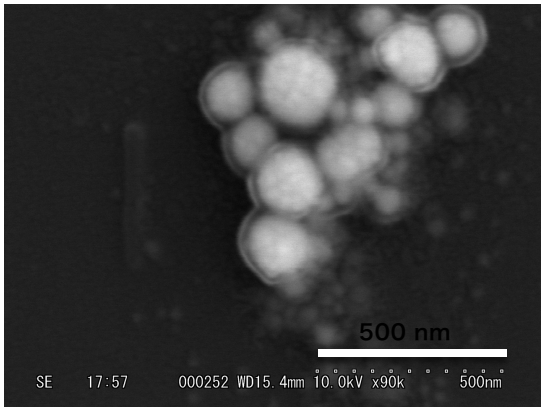
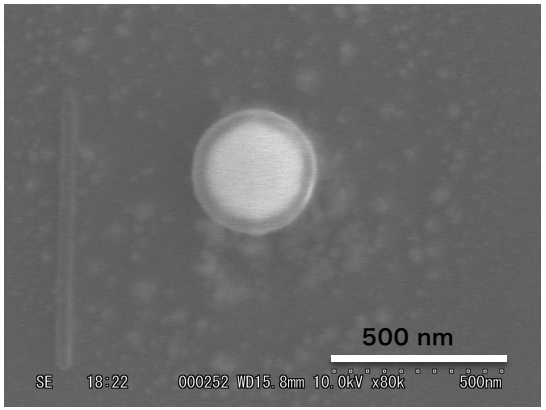
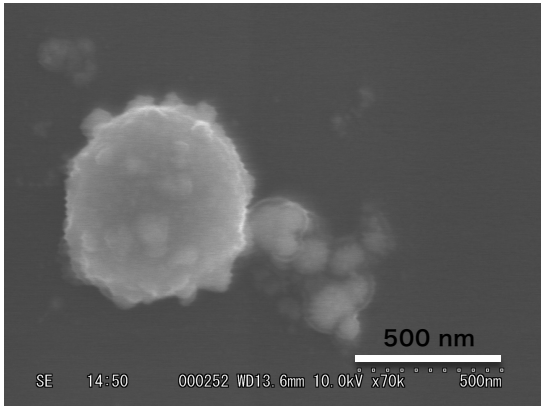
## In liquid $CO_2$

SEM

Ag

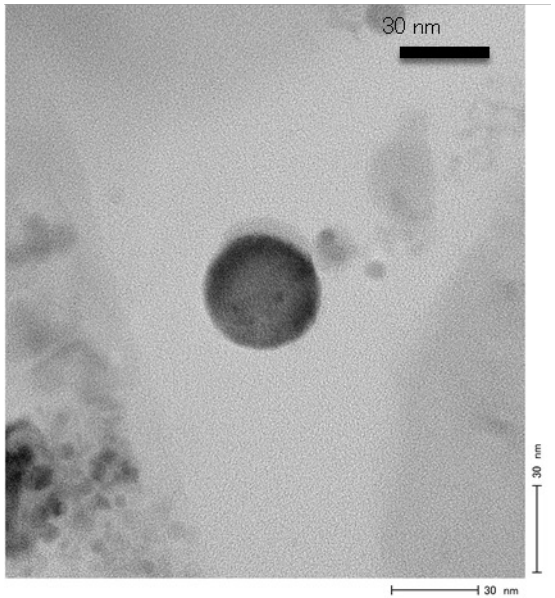



Density

Ni

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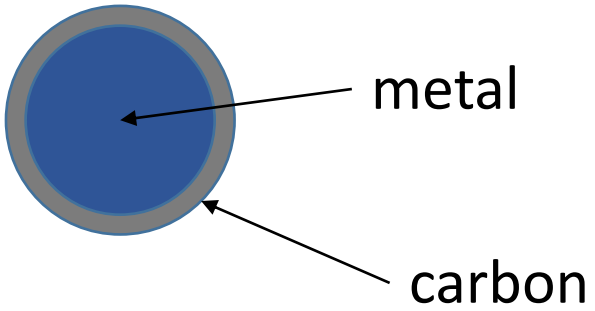


Core-shell structure

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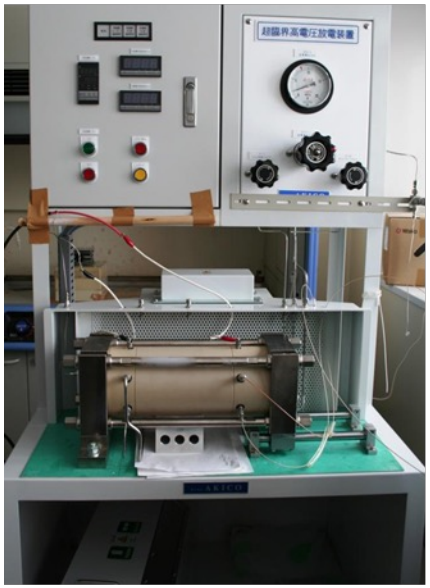
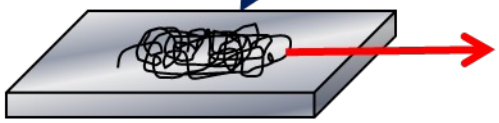
# Fiber Fabrication by Electrospinning under Pressurized $CO_2$



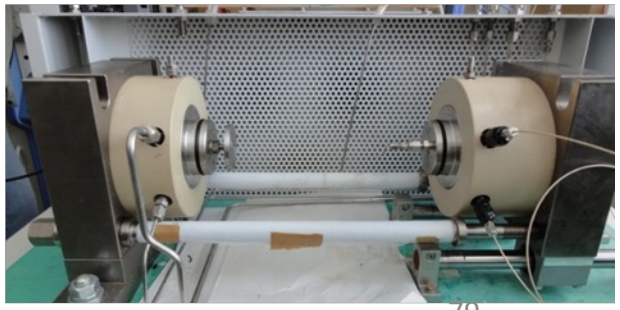



(1) Polymer solution
(2) Syringe Pump
(3) $CO_2$ cylinder
(4) High voltage source
(5) Nozzle
(6) Polymerjet/fiber
(7) Collector
(8) Spinning-Autoclave (PEEK)
(9) Heater
(10) Temperature sensor
(11) Back pressure regulator
(12) Trap
(13) Wet flow meter

Fig.1. Schematic diagram of the electrospinning system



Electrospun fiber



# Electrospinning

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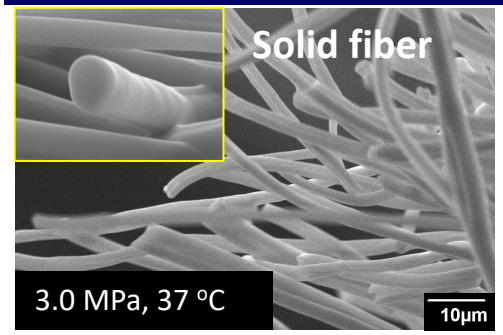
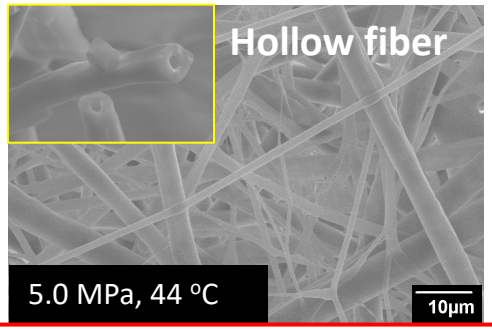
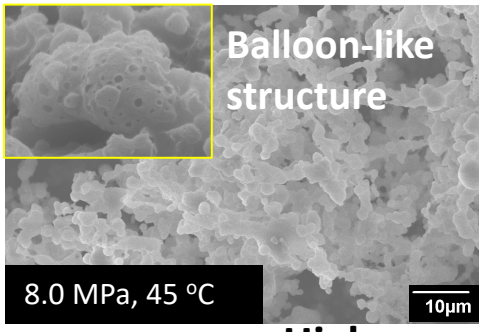
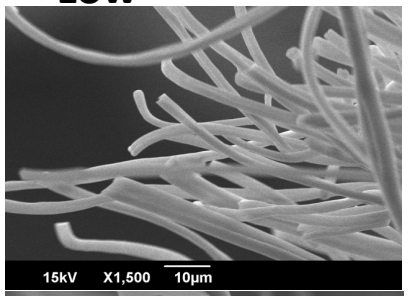
## Observation of the fiber cross-section

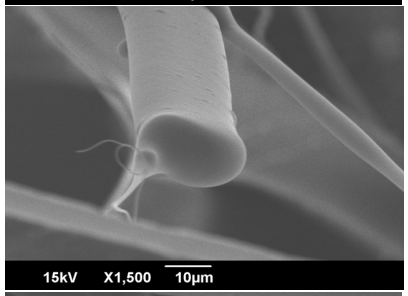


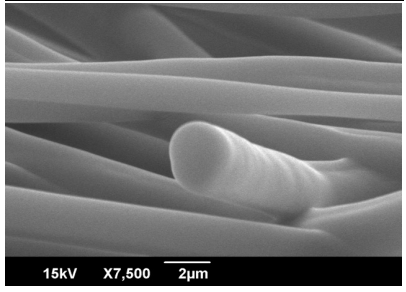



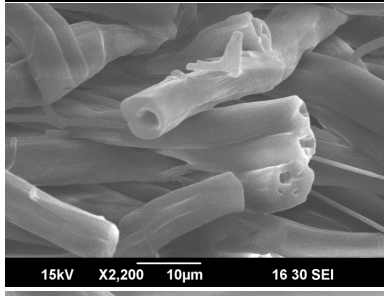

Low → **Solubility of $CO_2$ in solvent** → High

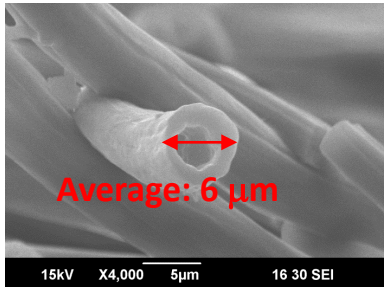


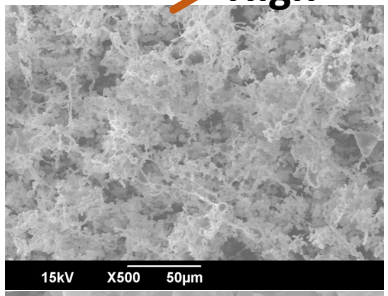



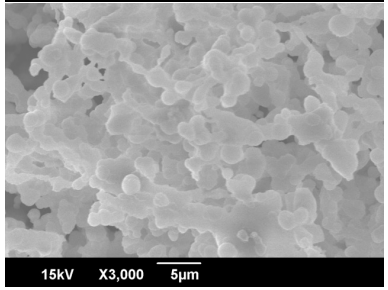



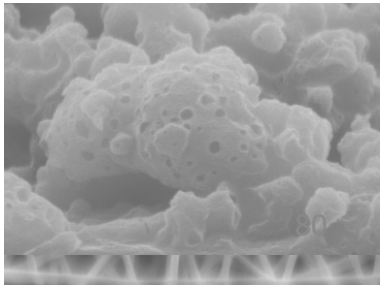




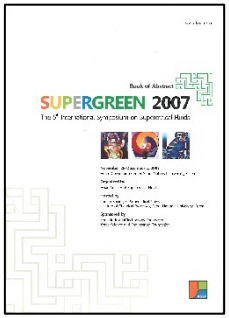



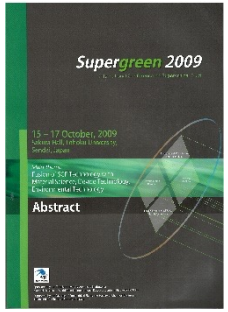
# International Activity

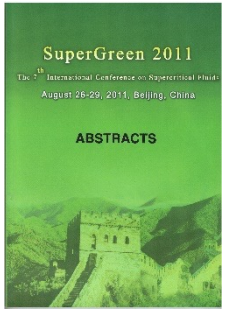
## Asian Society of Supercritical Fluids

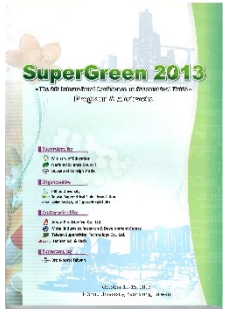
M. Goto, T. Adschiri, B. X. Han, Y. W. Lee, Yan-Ping
Ki-Pung Yoo, Ryuichi Fukuzato

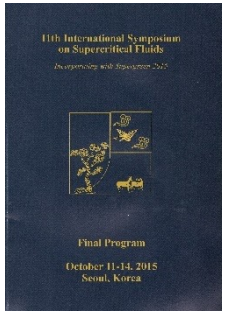
## Organizing Conference “Supergreen” in Asia

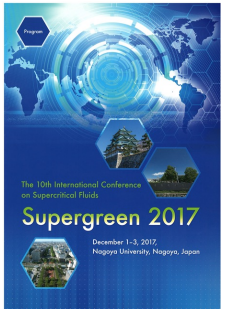


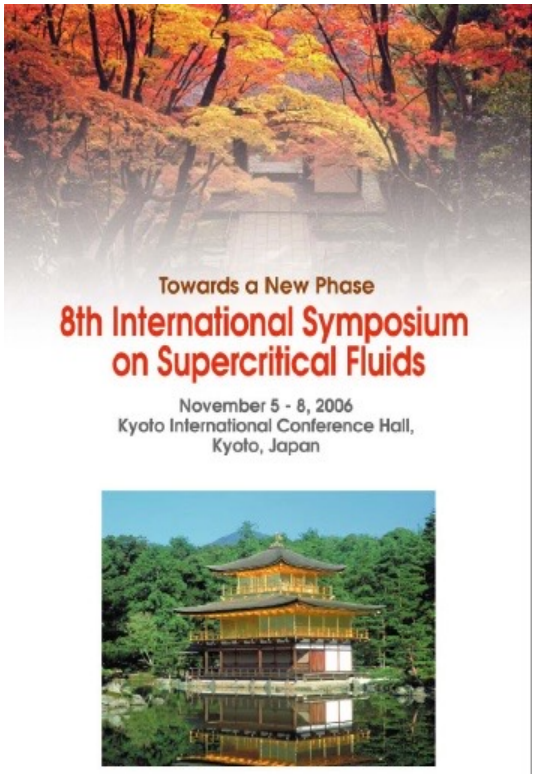




# International Conference Organization

## *ISSF2006*



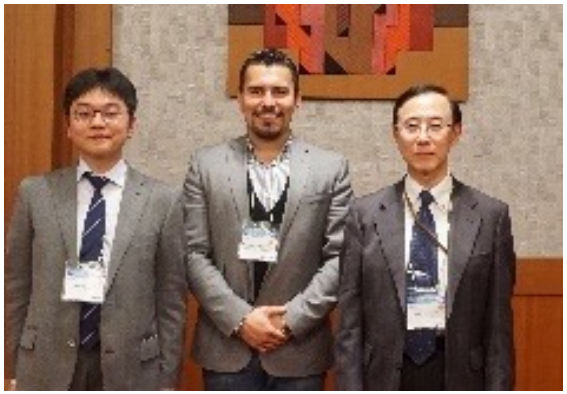
Organizing Chairs:
- Motonobu Goto
- Tadafumi Adschiri
- Buxing Han
- Youn-Woo Lee

# Supergreen 2017

M. Goto
S. Takami

NAGOYA UNIVERSITY




# Workshop on Supercritical Fluids and Energy, Brazil, 2013.12.8-11


the current status and future directions of the supercritical fluid science and technology



# International Activity

University of Valladolid, Spain
Prof. Maria Jose Cocero

PhD student: Oscar Benito and Teresa Moreno

Lecture in Spain by M. Goto
Lecture in Japan by M. Cocero

Visiting Prof.:
Dr. Juan García Serna

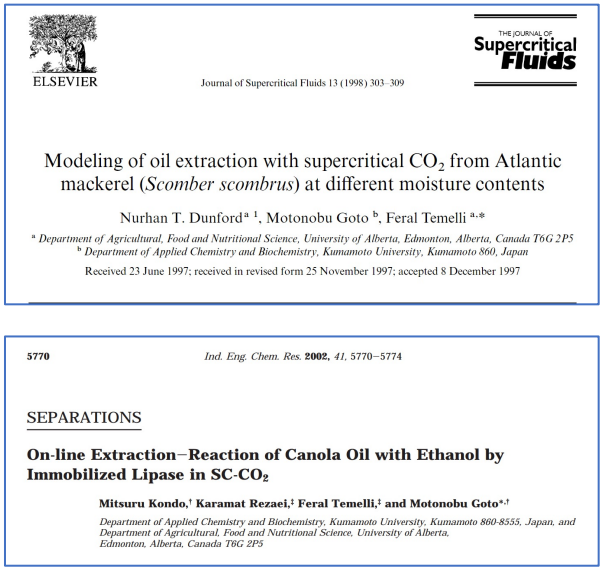

Visiting Prof.:
Dr. Angel Martin & María Dolores Bermejo Roda

# University of Alberta, Canada
# Prof. Feral Temelli

Edmonton, Canada

JSPS

Kumamoto Univ. & Kyushu Univ.



ELSEVIER

Journal of Supercritical Fluids 13 (1998) 303–309

THE JOURNAL OF Supercritical Fluids

Modeling of oil extraction with supercritical $CO_2$ from Atlantic mackerel (*Scomber scombrus*) at different moisture contents

Nurhan T. Dunford[a,1], Motonobu Goto[b], Feral Temelli[a,*]

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[b] *Department of Applied Chemistry and Biochemistry, Kumamoto University, Kumamoto 860, Japan*

Received 23 June 1997; received in revised form 25 November 1997; accepted 8 December 1997



5770 *Ind. Eng. Chem. Res.* **2002**, *41*, 5770−5774

SEPARATIONS

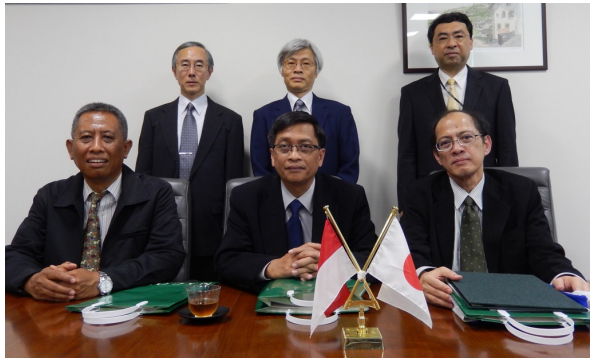
**On-line Extraction−Reaction of Canola Oil with Ethanol by Immobilized Lipase in SC-$CO_2$**

**Mitsuru Kondo,[†] Karamat Rezaei,[‡] Feral Temelli,[‡] and Motonobu Goto*[,†]**

*Department of Applied Chemistry and Biochemistry, Kumamoto University, Kumamoto 860-8555, Japan, and Department of Agricultural, Food and Nutritional Science, University of Alberta, Edmonton, Alberta, Canada T6G 2P5*

Institut Teknologi Sepuluh Nopember (Indonesia)
Dr. Siti Machmudah

Siti Machmudah



Exchange Agreement ITS & Nagoya U

Collaboration Lab at ITS

Profs of Chem. Eng.

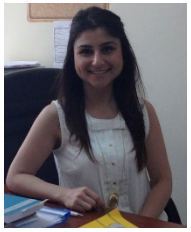
Visit UB

ITS students to Nagoya U. by Sakura Science Program

# Ege University (Turkey)

Prof. Oner Hortacsu, Bogazici Univ.

Exchange Agreement
Ege U & Kumamoto U



Prof. Asli Yuksel
Izmir Inst. Tech.

Prof. Ruhan Askin Uzel
Yasar Univ.

Ummihan Topal

Caner Uzelakcil


Satellite (Liaison)
Laboratory on SCF

Bulent Ozturk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**Yüksel, ailesinin gururu!**

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Chulalongkorn University, Thailand
Prof. Artiwan Shotipruk

Prof. A. Shotipruk

Chulalongkorn U

Former students of Chulalongkorn U

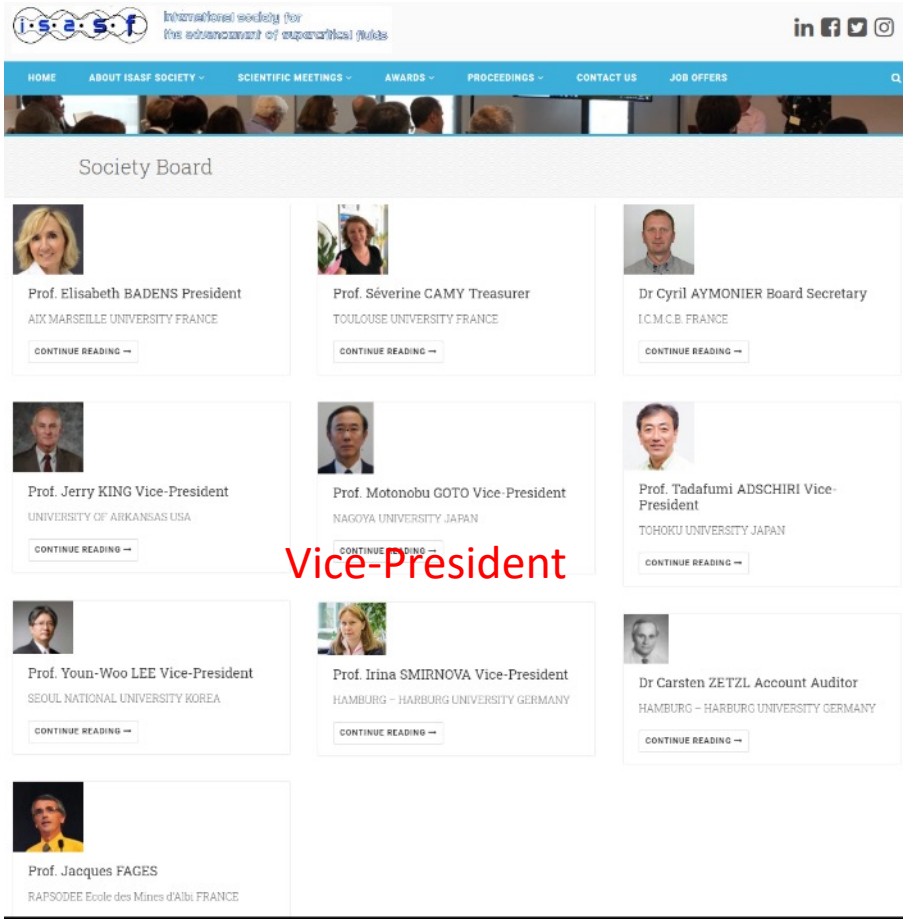
With Chula students


PhD Congress


Nagoya U office in Chulalongkorn U

Nagoya and Chula students

# International Society for Advancement of Supercritical Fluids




Vice-President

# The Journal of Supercritical Fluids

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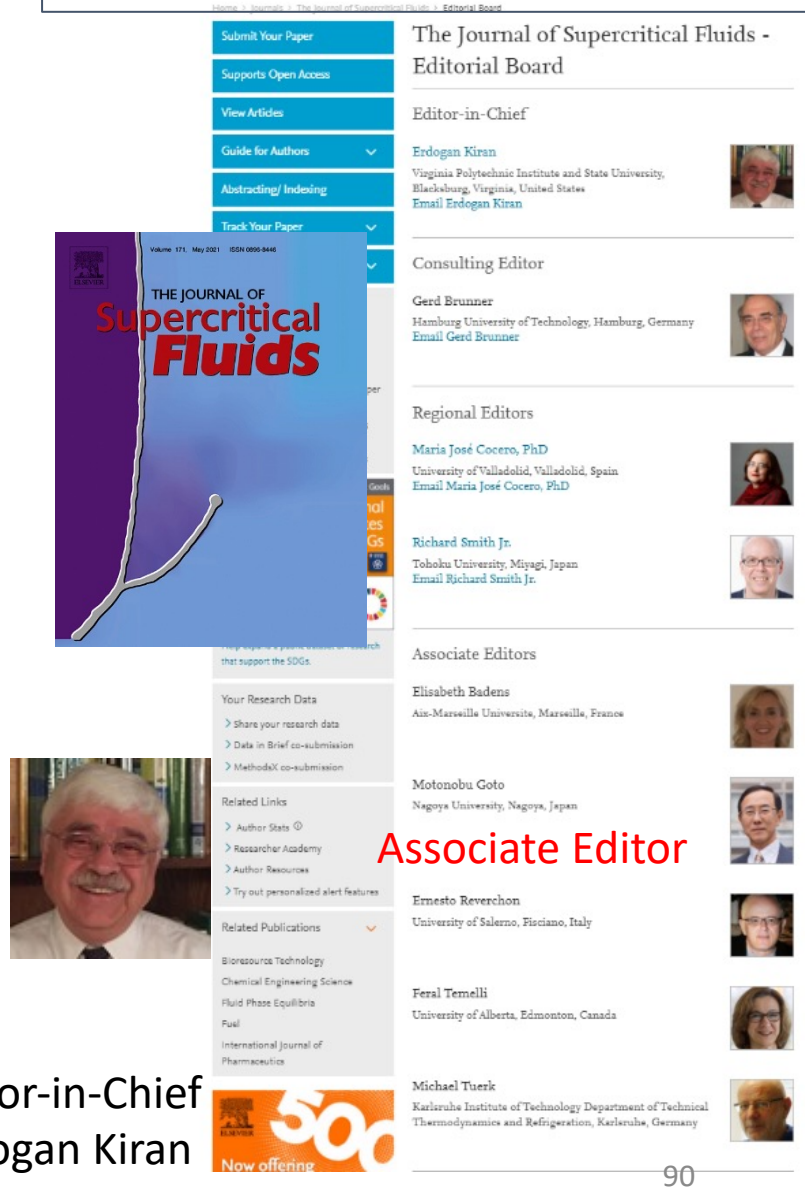


Associate Editor

Editor-in-Chief
Erdogan Kiran

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# Development of functional food materials from citrus fruit using green solvent technology

Coordinator: R. Fukuzato

オレンジブロッサム

Orange Blossom

Green orange

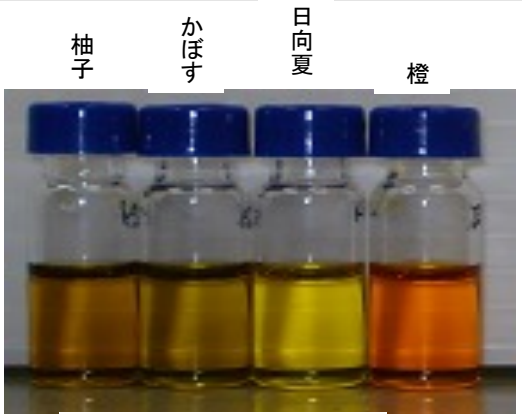
Yuzu

株式会社 アスキー

$scCO_2$
$scH_2O$

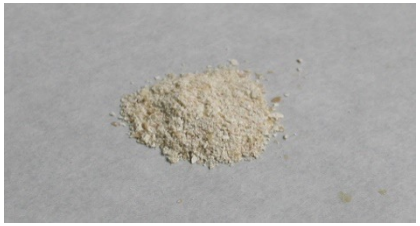
Green solvent platform

**Concentrated Terpeneless oil**




高品位柑橘果皮精油

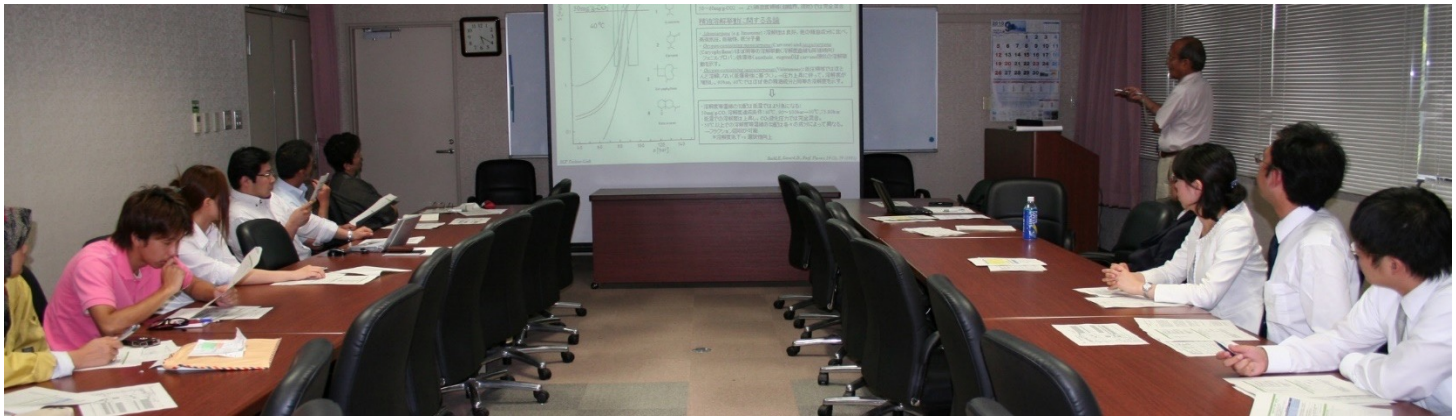
**Flavonoids**



**Pectin (functional food material)**

# Workshop on Supercritical Fluids

NAGOYA UNIVERSITY



R. Fukuzato
M. Goto

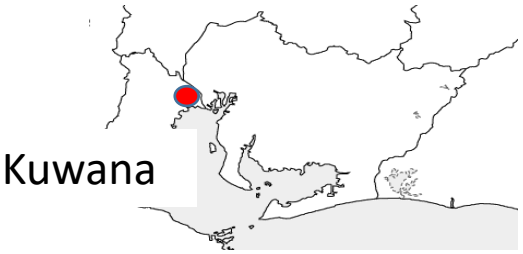
22 workshops since 2005

Lecture and Oversea plant survey
- China
- Korea
- Taiwan

# Super Critical Technology Centre

SCTC 超臨界技術センター

NAGOYA UNIVERSITY




Established in September 2013

Goto & Fukuzato

Venture plan @Kumamoto University

Venture company foundation @Nagoya University + KEC

ケー・イー・シー グループ

Focusing on extraction with $scCO_2$
Approved as Nagoya Univ. Venture

NUV Nagoya Univ. Venture


Consulting
Contract R&D and production

# Super Critical Technology Centre

SCTC 超臨界技術センター

Nagoya Univ. Venture

AGOYA NIVERSITY

## Decaffeination of green coffee beans

(30 L) pilot plant

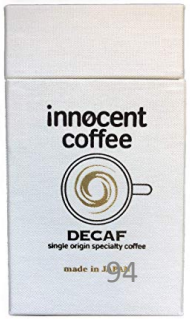
decaffeinated coffee

GREEN DECAF PROCESS
Made in Japan

**Commercial plant (2019)**



Decaf Green Tea

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福里隆一

Siti Machmudah
Wahyudiono

# これから

株式会社　SCFソリューションズ（4月１日設立）
SCF Solutions Co. Ltd.

□超臨界技術センター株式会社
Super Critical Technology Centre Co. Ltd.

□・・・

公益財団法人　――― 産業と科学をつなぐ研究所。
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Nagoya Industrial Science Research Institute