第5講義
表面利用

活性炭の応用

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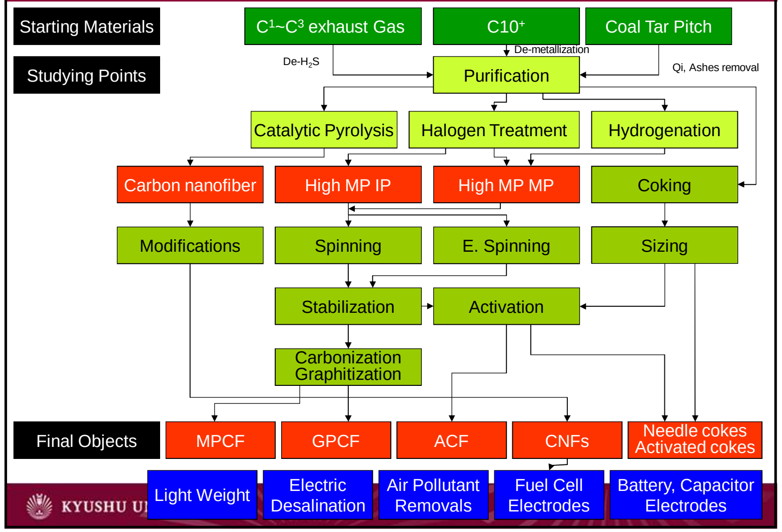
More concrete connections with raw materials to FCs



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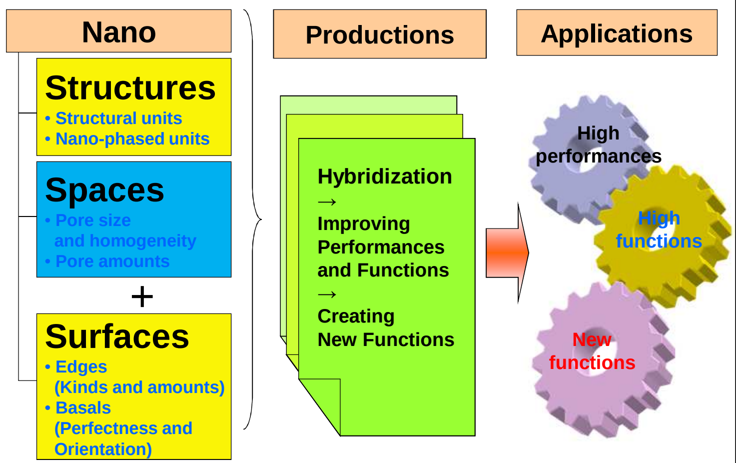
Understanding carbon structures:Carbon nano-world



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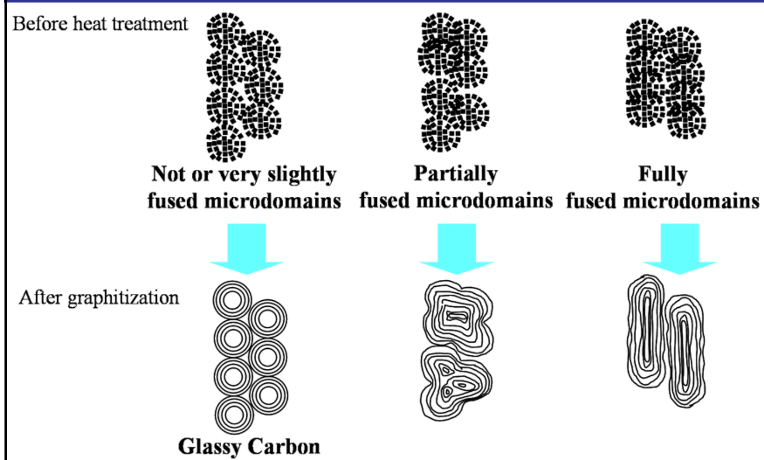
Basic structure and structural control of carbon



IAMS, Kyushu University "Axial nano-scale microstructure in the graphitized fiber inherited from liquid crystal mesophase pitch"
Carbon, 34, 83–88 (1996) S. H. Yoon, Y. Korai, K.Yokogawa, S. Fukuyama, M. Yoshimura, I. Mochida

Nanoscopic Structure of PAN Based CF

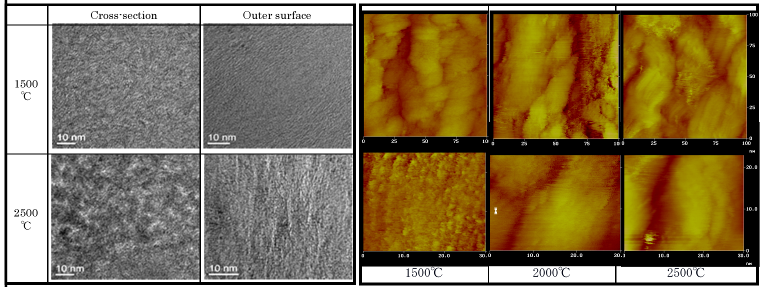
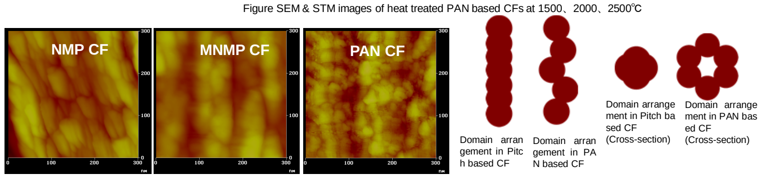


Figure SEM & STM images of heat treated PAN based CFs at 1500、2000、2500℃



"Structural comparison of mesophase and PAN based carbon fibers"
S.H. Hong, S. H. Yoon, I. Mochida *Carbon2006*、(2006、7) England

Key Materials for Energy and Environmental Devices

Carbon is an Indispensable Material for Energy related Devices

Best Structure for Best Performance
↕
Best Selection

Best Selection

Scientific Cycle

- Structural Understanding
- Structure Preparation
- Working Mechanism
  - Molecular Level
  - Electrochemical
  - Catalytic / Kinetics
  - Molecular / Heat Transfer

Carbon

Preparation of Activated Carbons

Selection of Precursor

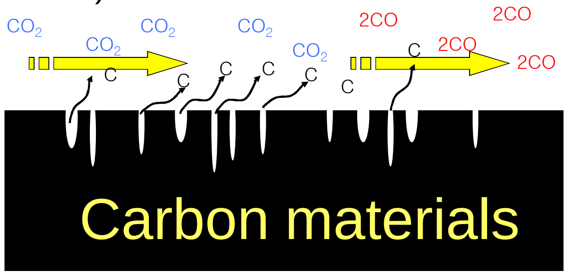
- *Pore Framework / Density*
- *Properties of Pore Wall, Composition / Graphitic Extent*
- *Reactivity at Activation*
- *Non-graphitizable precursors like polymer, biomass and isotropic coke for usual AC or ACF*
- *Graphitizable precursors like anisotropic cokes or mesophase pitch for EDLC electrode materials*

Activation Procedures

- *CO_2, H_2O*
- *Alkali Hydroxides / Carbonates; More Research*
- *Selective Catalytic Gasification ; Catalyst Control*

❖Very Large Surface Area > 3000 m^2/g
❖Adequate Pore And Wall

Activation (Making small pores in the carbon materials)



Activation reagents

- Air, CO2, Steam
- KOH (NaOH), ZnCl2

KOH Activation

$2KOH \rightarrow K_2O + H_2O$ (Dehydration)

$C + H_2O \rightarrow H_2 + CO$ (Water gas reaction)

$CO + H_2O \rightarrow H_2 + CO_2$ (Water gas shift reaction)

$K_2O + CO_2 \rightarrow K_2CO_3$

Catalytic progress Under K2O

Carbon is mainly consumed as a form of K_2CO_3

$K_2O + H_2 \rightarrow 2K + H_2O$

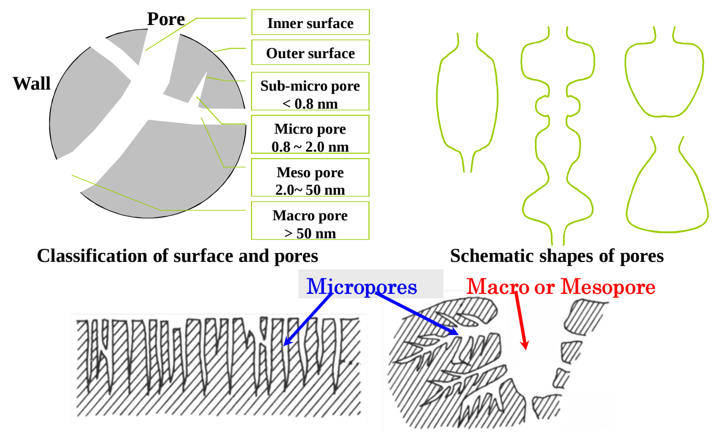
$K_2O + C \rightarrow 2K + CO$

K compounds are reduces as a metal

- Higher surface area compared to the steam activation
- Almost no productions of CO and CO_2
- K metal intercalation: higher diffusivity than steam molecule

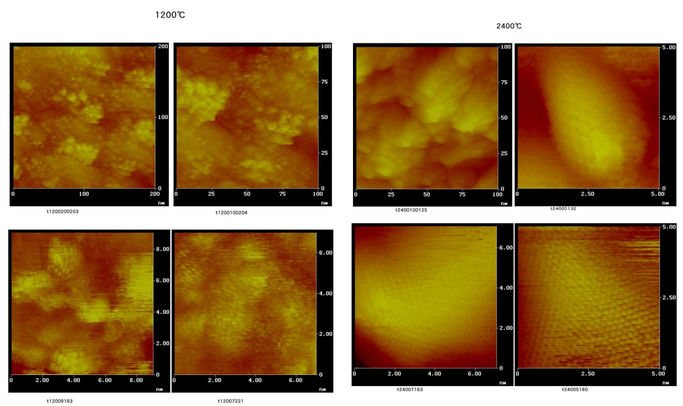
KOH	K_2O	K_2CO_3	K
MP：380℃ BP：1324℃	MP：490℃（350℃, KO and K）	MP：891℃	MP：64℃ BP：774℃

Activated carbon : Images of pores



Schematic pore images of activated carbon fiber and activated carbon

Glassy carbons from Novolak resin



Structure of Activated Carbon

Surface Area, Pore：*Depth & Volume*

Surface Structure

Surface Chemistry

Based and Edge Plane, Substituents

Hetero Atoms in Hexagon

Carbon Structure of Wall

Micro, Nano, Macro Structure of Carbon Wall

-Graphitization Extent

-Domain Structure

Density, Reactivity (Activated Surface)

→ *Precursor : Structure and Reactivity*

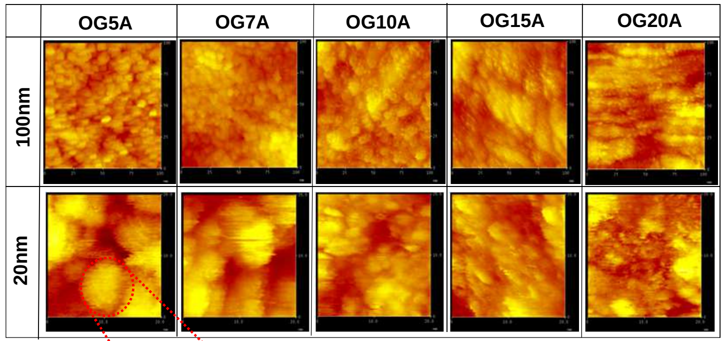
Some properties of pitch based ACFs

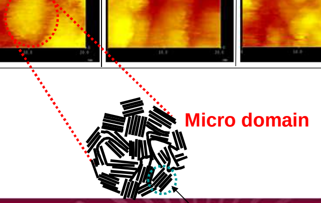
♦ Pitch-based activated carbon fibers (ACFs) used in this study.
OG5A, OG7A, OG10A, OG15A and OG20A were provided by Osaka Gas Co.

Some physical properties of ACFs given by Osaka Gas Co.

	OG5A	OG7A	OG10A	OG15A	OG20A
Specific surface area (m^2/g)	480	850	1300	1725	2000
Average pore size (nm)	1.4	1.6	1.8	1.9	2.2
Average pore volume (cm^3/g)	–	0.38	0.54	0.81	1.13

STM images of ACFs

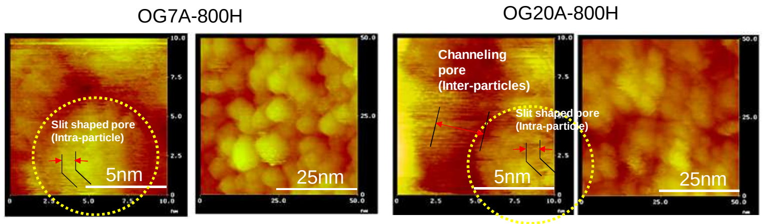




♦ACFs consist of structural units of micro domain with diameter of around 5nm.

STM images of ACFs

In order to remove oxygen containing functional groups for removing the heterogeneous effect of STM, OG7A and OG20A were heat-treated at 800°C in a hydrogen atmosphere (H_2 / He =1/4).



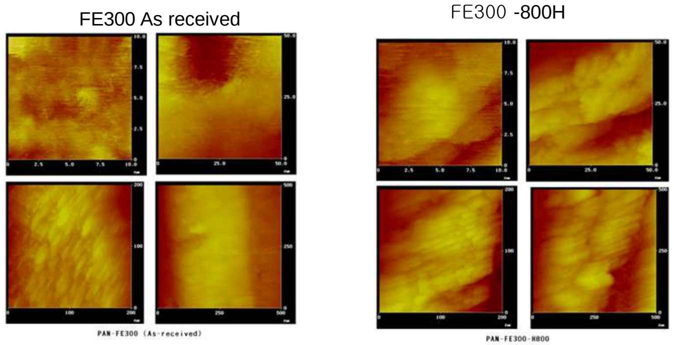
♣Vacant spaces between the two domains of OG20A are larger than that of OG7A.

♣Domain size of OG20A is a little smaller than that of OG7A.

♣Slit type pores were observed in domains of OG7A and OG20A.

♣It can be presumed that almost pores larger than 2nm nucleated by the inter-particle mechanism.

STM images of PAN Based ACFs



♣ FE300 is also composed of micro-domain structures.
♣ Basic structural units of PAN based ACF are more difficult to observe because of high heterogeneous effects of surface heterogeneous atoms and many defects.

Activation of carbons

Precursor of ACF has been composed of nano-structural primary units

Structural factor should be considered for the better understanding of activated carbons and their applications.

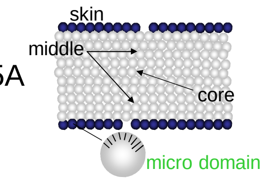
Size and arrangement of BSU
Etching and diffusion of oxidative agent against BSU

Pores from intraparticles (Slit shaped? Micropores less than 2 nm)
Pores from interparticles (Channeled shaped having wider pore size distributions (0.2 ~ 50 nm)

Mechanism of the pore formations in ACFs

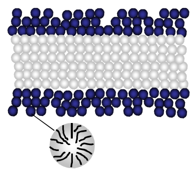
A model for cross section of ACFs

OG5A



♣Only skin is activated, homogeneous narrow pore exist on the surface of domains.
♣The activation does not reach to the middle and core parts.
↓
OG5A has smallest surface area but homogeneous pores.

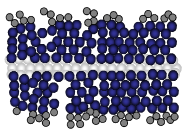
OG7A



♣Skin and middle parts are activated.
♣Pores in domain become wider and longer than that of OG5A because of the pores formed by inter-domain mechanism.
♣Pores are formed by the intra-domain and inter-domain mechanisms.
↓
Heterogeneous pores exist.

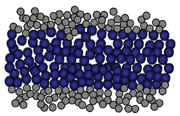
Mechanism of the pore formation in ACFs

OG10A



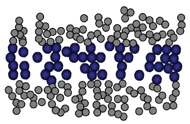
♣The activation proceeds to near the core.
♣Domains in the surface become smaller by activation.
↓
OG10A have various size pores.

OG15A



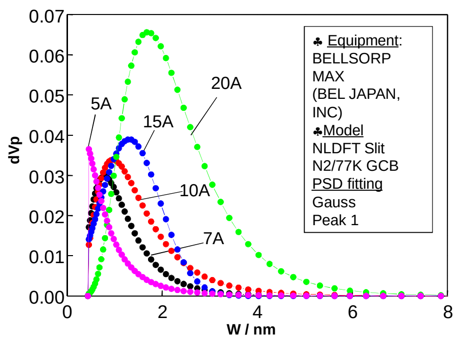
♣Whole parts of skin, middle and core are activated.
♣The pores from the intra-domain become larger by activation.
♣Owing to some domains burning, fiber diameter becomes smaller than OG5A, 7A and 10A.
↓
More homogeneous pore distribution than that of OG10A

OG20A



♣Over 70% of domains are burned-off.
♣Pore portions from interparticle nucleation becomes larger
↓
♣OG20A diameter become smaller than OG15A.
OG20A have largest surface area but heterogeneous pores.

Pore distribution using NLDFT method



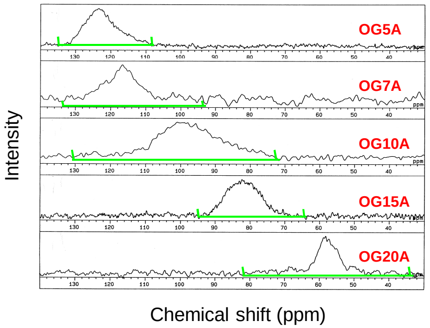
♣**Advantage of DFT method**
Determination of a pore size distribution in the wide range of pore size from micro pore to meso pore.

♣Disadvantage
This method needs
>measurement in low pressure
>assumption of pore structure
>homogeneity of adsorbent surface

	OG5A	OG7A	OG10A	OG15A	OG20A
pore size at peaks (nm)	Less than 0.44	0.72	0.98	1.28	1.86
Pore distribution range in this DFT profile (nm)	~2.8	~3.0	~5.0	~3.0	0.44~7.0

♦The pore size at peak increased in the order of OG5A<7A < 10A < 15A < 20A.

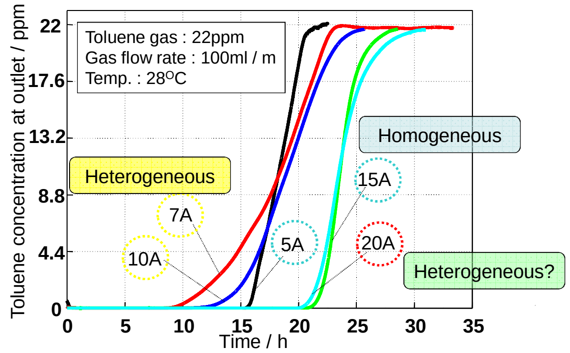
^{129}Xe-NMR spectrum of ACFs



	pore size at peak (nm)	Pore distribution width (nm)
OG5A	0.84	0.81~0.98
OG7A	0.89	0.79~1.15
OG10A	1.04	0.82~1.50
OG15A	1.23	1.15~1.55
OG20A	1.49	1.32~3.84

- ♦ Chemical shift at highest intensity, corresponding to pore size, increased in the order of OG5A<7A < 10A < 15A < 20A.
- ♦ 5A and 15A exhibited narrow peaks. →relatively homogeneous pore size
- ♦ Peak broadening can be seen for 7A, 10A and 20A. → heterogeneous pore size

Toluene adsorption characteristics of ACFs



♦The slopes of breakthrough curves for 5A and 15A were steeper than those of curves for 7A and 10A.

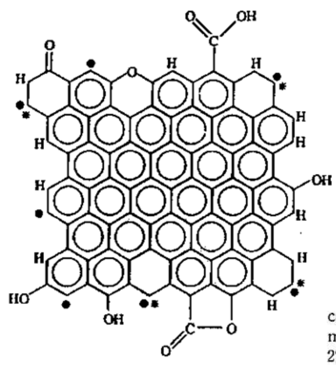
♦ACFs with homogeneous pores (5A and 15A) showed rapid toluene adsorption and larger capacity per unit area and longer breakthrough time, whereas ACFs with heterogeneous pores (7A and 10A) showed slow toluene adsorption and smaller capacity per unit area.

Some Properties of ACFs

Pitch based ACF	BET (m^2 / g)	Elemental analysis (wt %) C	H	N	O	N / C
OG5A	563	92.4	0.6	0.7	6.0	0.007
OG7A	901	93.0	0.6	0.8	5.4	0.007
OG10A	1085	95.3	0.6	0.5	3.4	0.004
OG15A	1606	95.2	0.6	0.3	3.4	0.003
OG20A	1924	94.1	0.6	0.4	4.8	0.003

PAN based ACF	BET (m^2 / g)	Elemental analysis (wt %) C	H	N	O	N / C
FE100	450	70.9	2.0	8.4	17.3	0.102
FE200	650	72.5	1.8	4.8	17.9	0.057
FE300	880	74.3	1.6	3.3	17.2	0.038
FE400	1020	76.8	1.6	2.3	19.4	0.026

Surface Oxygen Functional Groups of ACF



This structure is representative of an activated carbon with a crystallite width of 15 Å and an elemental analysis (by weight) of 87.5% C, 11.3% O, 1.2% H, ● represents an unpaired σ electron ; ●* represents an "in-plane σ pair" with * being a localized π electron, (Radovie)

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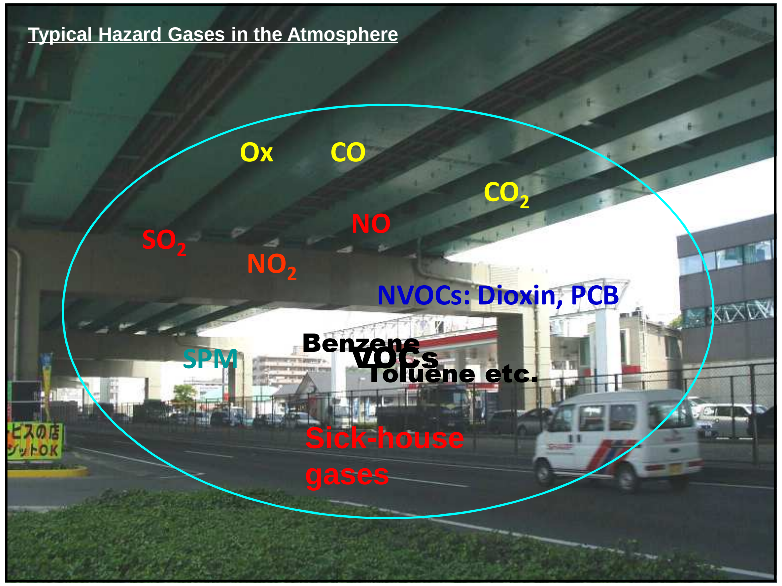
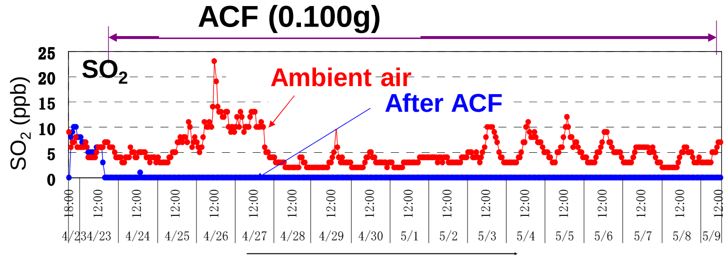
Removal of SOx and NOx Using ACFs



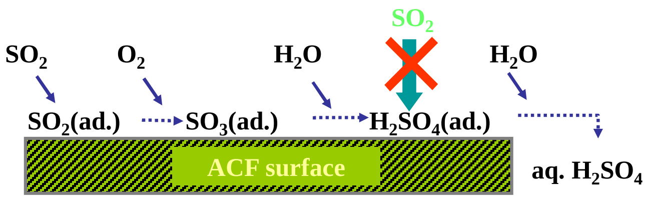
ACF

SO_2 → SO_4^{2-}



Data from Dr. Shimohara
Of Fukuoka H & E Institute

DeSOx mechanism using ACF



SO_2+ad.→SO_2ad.

SO_2ad.+1/2O_2→SO_3ad.

SO_3ad.+H_2O→H_2SO_4ad.

H_2SO_4ad.+H_2O→aq.H_2SO_4

Active Sites on Carbon Surface

Oxygen functional group — Acidic nature: Oxidative; Basic nature: Reductive

Free valence

Benzyne bonds on edge — Oxygen activation

Hetero-atoms in edge — Zigzag or Armchair

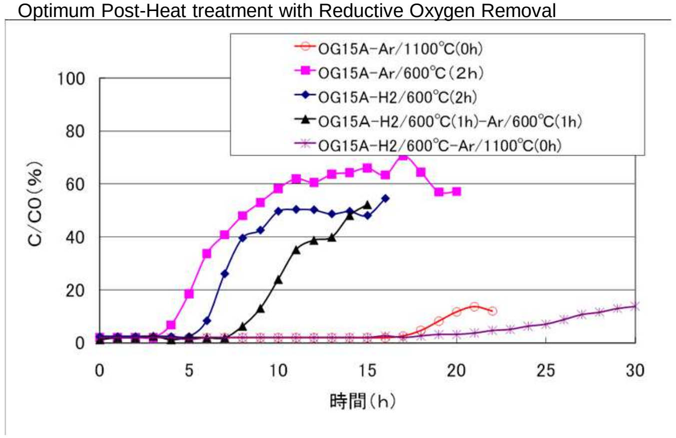
Hexagon stacking height

Hydrophilic/Hydrophobic

Small surface energy

How to control ?

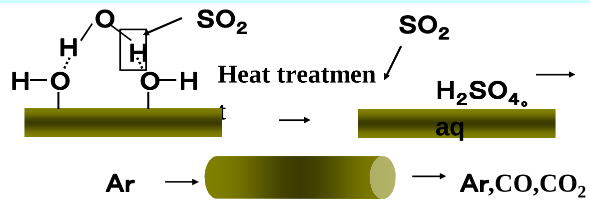
Breakthrough of SO_2 Removal



SO_2 1000ppm,O_2 5vol.% H_2O 10vol.%,N_2 balance, Flow rate: 100ml/min, Temperature: 50℃

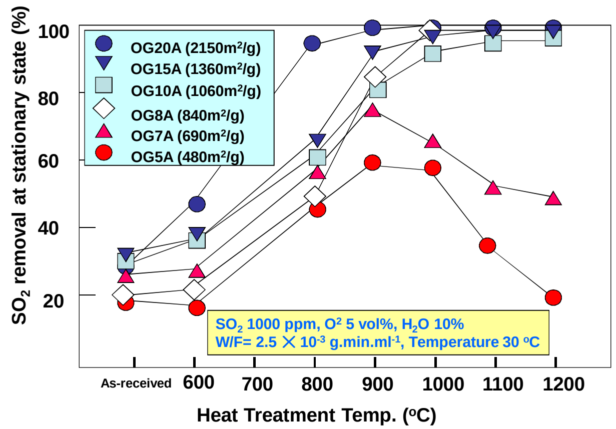
Effect of Heat Treatment

Removal of Functional Groups
with Least Changes of Carbon Structure
Surface Area and Hexagon Stacking



Carbon	C	H	N	O	Ash(%)
	(wt.%, dry)				
OG-15A	93.5	0.6	0.5	5.5	0.0
OG15A-H1100(0min)	97.0	0.4	0.5	2.2	0.0
OG-15A-H1100(1h)	96.8	0.2	0.3	2.7	0.1

The Effect of Heat Treatment for SO_2 Removal



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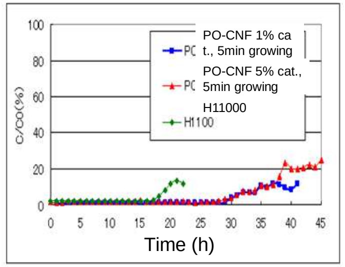
DeSOx by ACF and CNF-ACF Composite

DeSOx Properties of ACF and ACF-CNF



DeSOx condition: SO_2 1000ppm, O_2 5vol%, H_2O 10vol%, N_2 balance、Total flow rate: 100 ml/min Reaction Temperature: 50 ℃

PDU for SOx Removal by ACF

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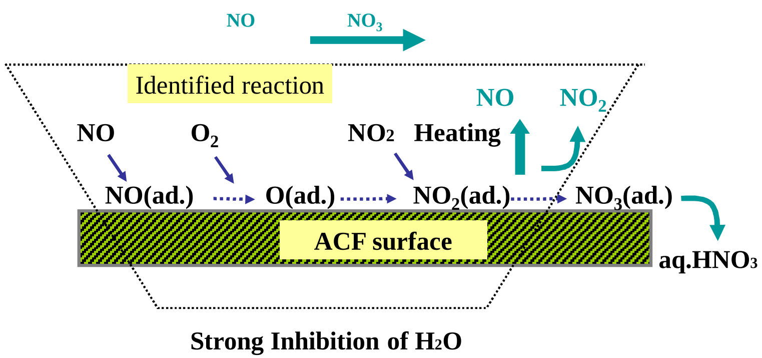
NO & NO_2 Oxidation over ACF



The oxidation of NO_2 always produces NO And NO_3^- through the disproportionation.

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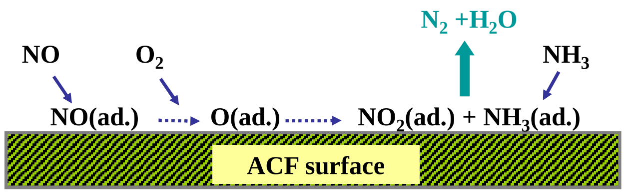
The Mechanism of NO Reductive Removal



The mechanism of NO removal consists of adsorption and oxidation of NO into NO_2 which is reduced with NH_3

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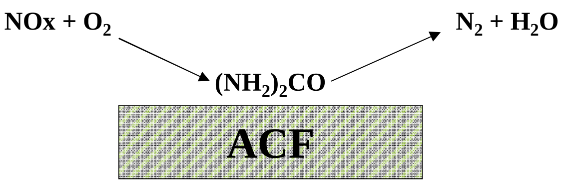
NOx Reduction at Room Temperature



- NOx oxidation
- Urea Activation
- NOx in Environment
- Roles of ACF : More variety of ACF

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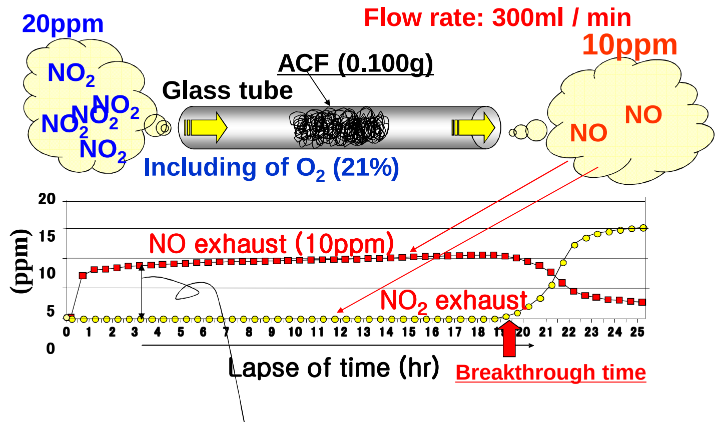
Adsorption of NO_2 in ACF



Half amount of adsorbed NO_2 is exhausted as NO.

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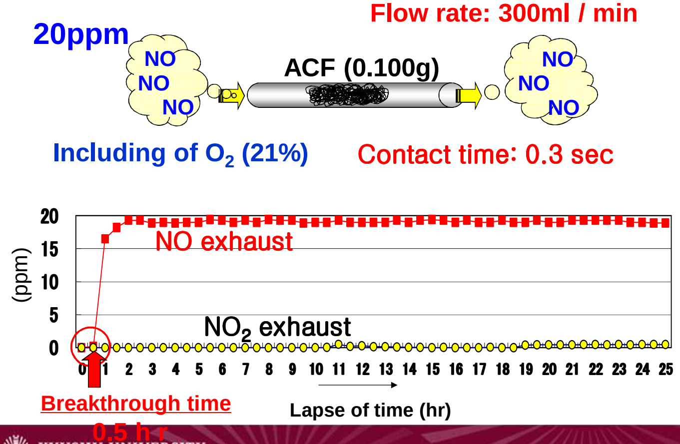
Scavenging activities against NO



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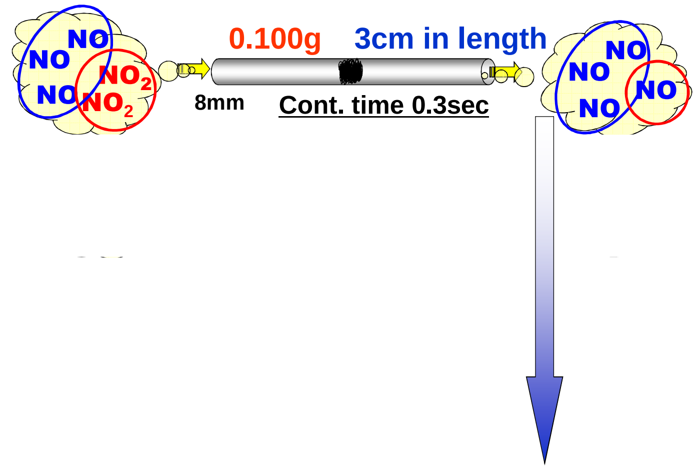
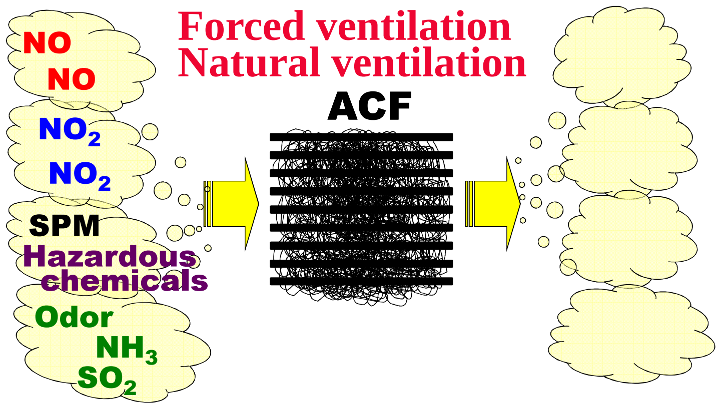
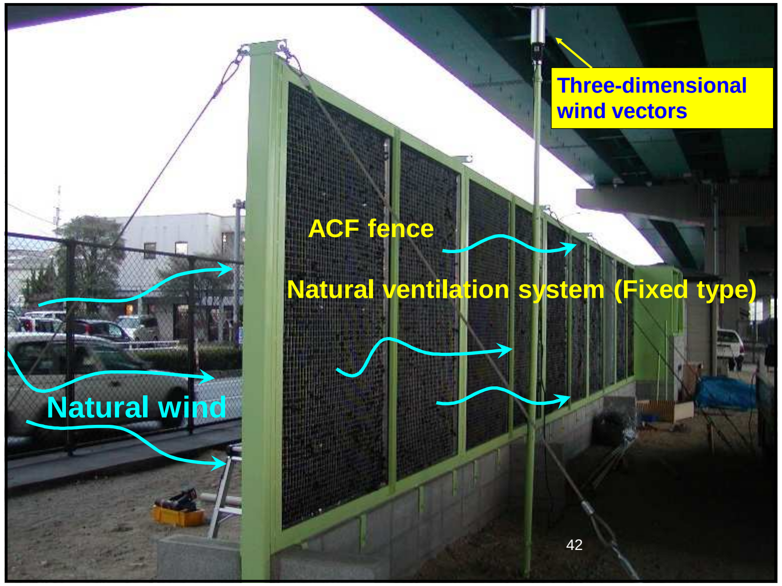
The characteristics of NOx purification



Characterization of ACF purification

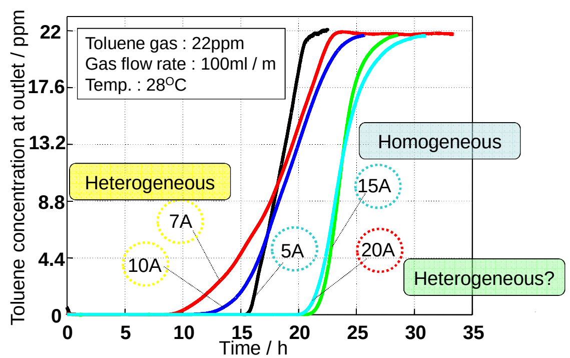


Room temperature, ozonizer is no need, no light irradiation, compact design



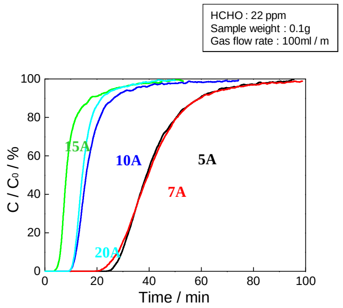
Removal of Sick-house gases using ACF/MnO_2

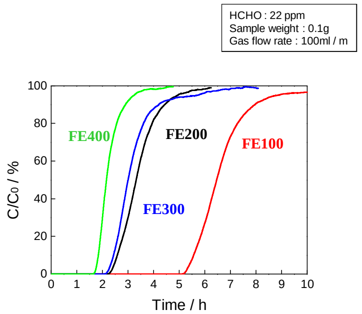
Toluene adsorption characteristics of ACFs



- ♦The slopes of breakthrough curves for 5A and 15A were steeper than those of curves for 7A and 10A.
- ♦ACFs with homogeneous pores (5A and 15A) showed rapid toluene adsorption and larger capacity per unit area and longer breakthrough time, whereas ACFs with heterogeneous pores (7A and 10A) showed slow toluene adsorption and smaller capacity per unit area.

HCHO adsorption characteristics of ACFs



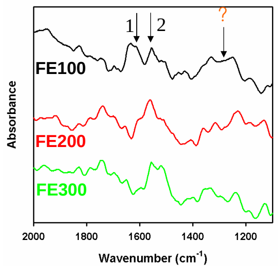


Break through time

◆Pitch-based ACF : 15A < 20A < 10A < 7A < 5A

◆PAN-base ACF : FE400 < FE300 < FE200 < FE100

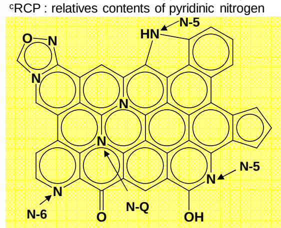
Micro ATR-FTIR spectrum of FE series



1) *Pyridinic nitrogen band*
2) *Internal standard*

	Micro-ATR FTIR analysis			EA and XPS analysis		
	I.S[a]	Pyridinic N[b]	RCP[c]	N / C ratio	Pyridinic N[b]	RCP[c]
FE100	279	134	0.48	0.118	0.737	0.087
FE200	276	108	0.39	0.066	1.050	0.069
FE300	332	70	0.21	0.044	0.734	0.032

[a]I.S : Internal standard (1560cm⁻¹)
[b]Wavenumber related with pyridinic nitrogen : 1610~1600cm⁻¹
[c]RCP : relatives contents of pyridinic nitrogen



Pyridinic N (N-6)
Pyrrolic or Pyridinic N (N-5)
Quaternary N (N-Q)

Micro-ATR FTIR analysis

Relative amount of pyridinic nitrogen functional groups for PAN based ACFs by micro-ATR FTIR analysis

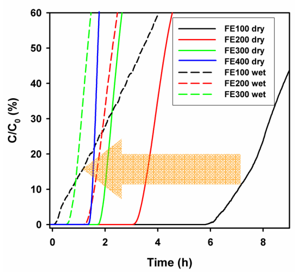
Sample	Internal Standard	Pyridinic N [a]	Internal Standard /Pyridinic N
FE100	279	134	0.48
FE200	276	108	0.39
FE300	332	70	0.21
FE400	330	64	0.19

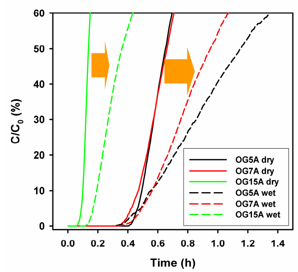
[a] Wavenumber related with pyridinic nitrogen: 1610 ~ 1600 cm⁻¹

Breakthrough curves of formaldehyde adsorption

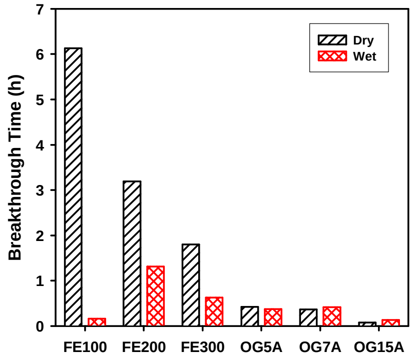
WATER Competitive adsorption decreases the adsorption amount of HCHO

Dry condition (solid line) and wet condition (dashed line) for the different kinds of a) FE series and b) OG series





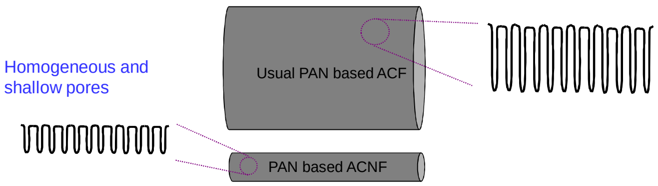
Comparison of formaldehyde adsorption in different ACFs between dry and wet condition



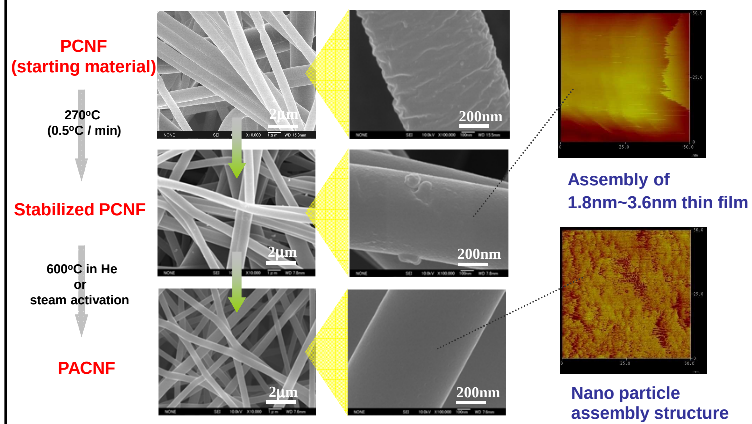
Selection of Precursor for ACF

♦Electrospun PAN based nanofiber (100% PAN)
Diameter: 800 nm, Nanotechnics (Korea)

100 times surface area compared to usual PAN fiber
→Can be expected very shallow and homogenous pores。

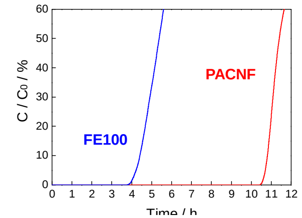


SEM images of PCNF, stabilized PCNF and PACNF

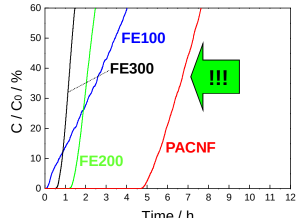


HCHO adsorption characteristics of PACNF in humidified atmosphere

RH	BET	Elemental analysis (wt%)					N / O	Microporous
	(m2 / g)	C	H	*N*	*Odiff*	ash		ratio (%)
90%	375	68.06	1.19	*18.02*	*11.41*	1.32	1.80	94.7%



Experimental
HCHO : 11 ppm
Sample weight : 0.05g
Gas flow rate : 100ml / ml
Humidity of condition : 0%

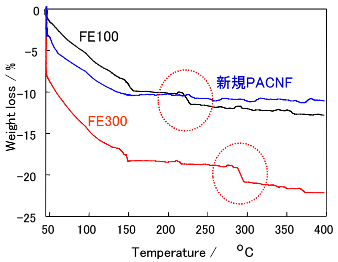


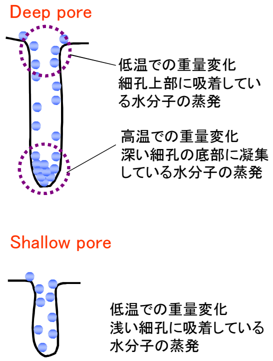
Experimental
HCHO : 11 ppm
Sample weight : 0.05g
Gas flow rate : 100ml / ml
Humidity of condition : 50%

Under the circumstances of humidity (RH=50%), PACNF shows specific prominent adsorption characteristics for formaldehyde.

Water adsorption property

水分を飽和吸着させたサンプルを
アルゴン中で400℃まで昇温させ、
重量変化を観察





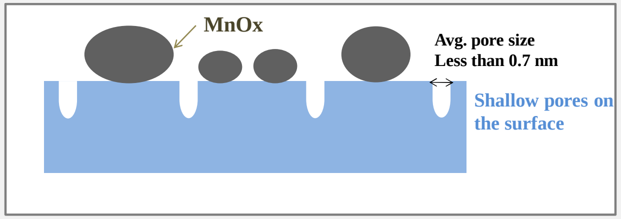
Concept of the newly designed MnOx/ACNF

① Activated nano carbon fiber

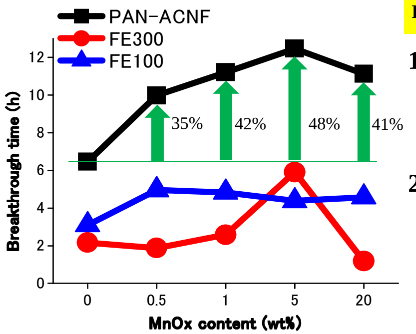
② Catalytic decomposition of HCHO by MnOx into water and carbon dioxide

➡ ③ ▪Clean removal into Water and Carbon dioxide
▪Lifetime prolonged

The conceptual model of MnOx-carbon catalyst



Influence of MnOx content on HCHO breakthrough time



Weight-0.05g base

Dry condition

1. MnOx combined with carbon support to enhance HCHO removal.
2. PAN-ACNF showing the highest synergy at 5% MnOx.

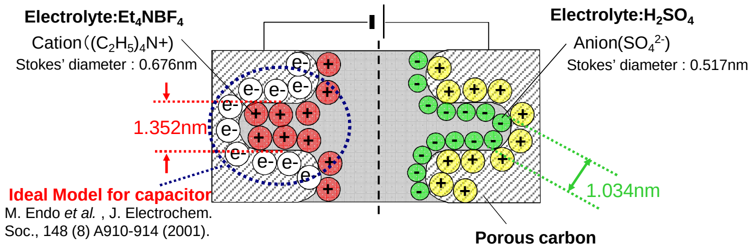
Mn_3O_4 or MnO_2 alone breakthrough in less than an hour.

➡ Deposition on carbon support improves catalytic activity of MnOx.

Conjecture of pore size using capacitance data

EDLC with organic electrolytes

EDLC with inorganic electrolytes



M. Endo *et al.* , J. Electrochem. Soc., 148 (8) A910-914 (2001).

In using Et_4NBF_4 as an electrolyte, at least pore size larger than 1.3nm is necessary to have electric double layered capacitance.

In using H_2SO_4 as an electrolyte, pore size of about 1.0nm is enough to have electric double layered capacitance.

Samples

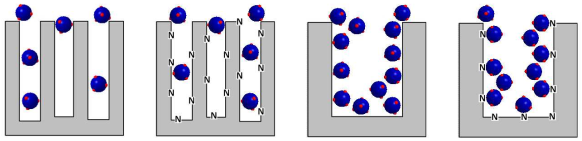
Pitch-based Activated Carbon Fibers (ACFs)

OG series : OG-5A, OG-7A, OG-10A, OG-15A, OG-20A (Osaka Gas Co., Japan)

PAN-based ACFs

FE series : FE-100, FE-200, FE-300, FE-400 (Toho TENAX Co., Japan)

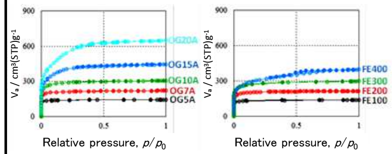
Model of micropores of OG and FE series



Aqueous and non-aqueous electrolytes with different ion sizes

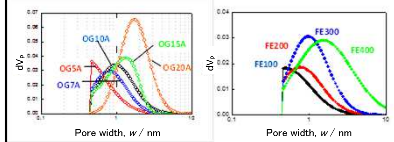
Properties of Activated Carbon

N_2 adsorption/desorption isotherms @77K



Pore size distributions

(*calculated by NL-DFT method*)



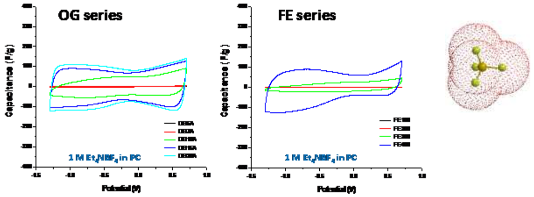
Pore structure parameters

(*calculated from t-plot method*)

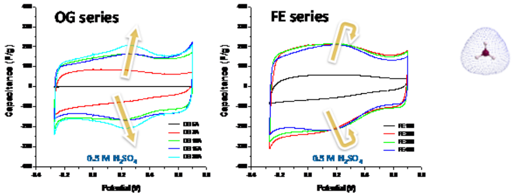
	Surface area (m^2/g)				Pore volume (cm^3/g)			Pore width (nm)	
	A_{total}	$A_{external}$	A_{micro}	A_{meso}	V_{total}	V_{micro}	V_{meso}	W_{micro}	W_{meso}
OG-5A	676.8	1.2	675.6	0	0.22	0.22	0	0.65	0.0
OG-7A	987.6	3.4	984.2	0	0.34	0.34	0	0.68	0.0
OG-10A	1211.7	5.4	1206.3	0	0.46	0.46	0	0.77	0.0
OG-15A	1488.0	13.9	1474.1	0	0.66	0.66	0	0.90	0.0
OG-20A	1817.4	15.9	1801.5	0	0.97	0.97	0	1.08	0.0
FE-100	636.9	1.2	635.7	0	0.21	0.21	0	0.67	0.0
FE-200	909.2	2.2	907.0	0	0.33	0.33	0	0.72	0.0
FE-300	1130.6	3.8	1099.7	27.1	0.45	0.43	0.02	0.78	1.82
FE-400	1187.1	5.2	931.2	250.7	0.60	0.38	0.22	0.82	1.73

Cyclic Voltammograms in Aqueous/Non-Aqueous Electrolytes

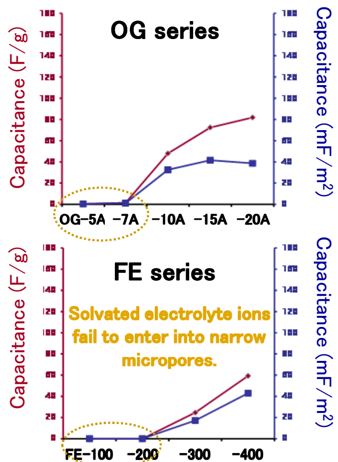
Cyclic voltammograms in 1 M Et_4NBF_4 / PC

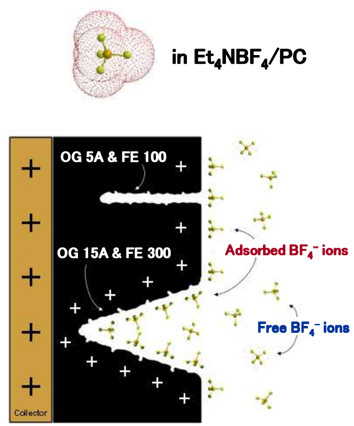


Cyclic voltammograms in 0.5 M H_2SO_4

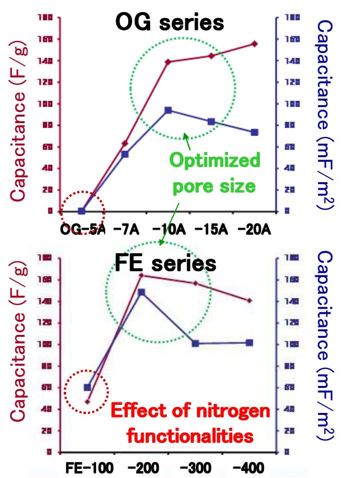


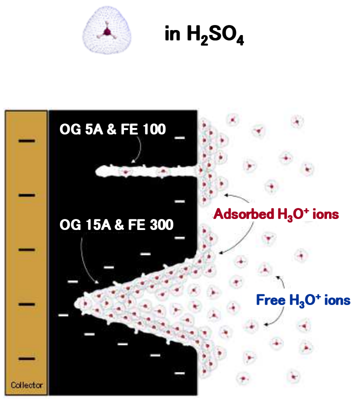
Specific Capacitances *in Non-Aqueous Electrolyte (Et_4NBF_4/PC)*





Specific Capacitances *in Aqueous Electrolyte (H_2SO_4)*





T_1 Values Measured from ^2H MAS Solid-State NMR Spectra

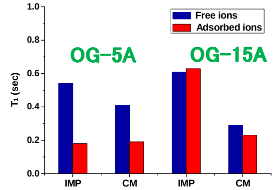
The shorter the T_1 value of relaxation time, the stronger the adsorption interaction between adsorbed electrolyte ions and carbon electrodes.

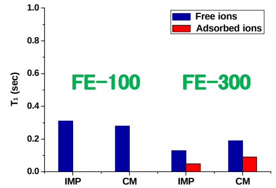
T_1 (sec) for OG series

OG-5A				OG-15A			
IMP		CM		IMP		CM	
Free	Adsorbed	Free	Adsorbed	Free	Adsorbed	Free	Adsorbed
0.54	0.18	0.41	0.19	0.61	0.63	0.29	0.23

T_1 (sec) for FE series

FE-100				FE-300			
IMP		CM		IMP		CM	
Free	Adsorbed	Free	Adsorbed	Free	Adsorbed	Free	Adsorbed
0.31	–	0.28	–	0.13	0.05	0.19	0.09





Steam vs. Chemical Activations

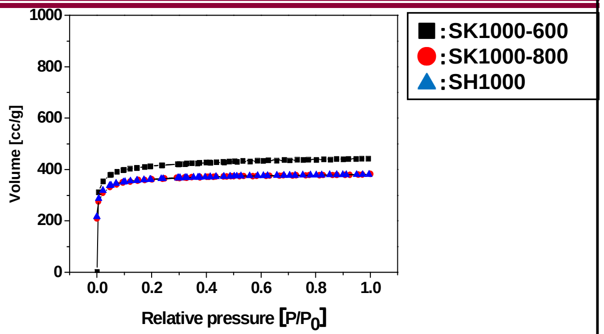
What is the difference:

- Surface area, pore size and its distribution
- Surface compositions
- Surface structure (?)
- Cost
- Waste materials

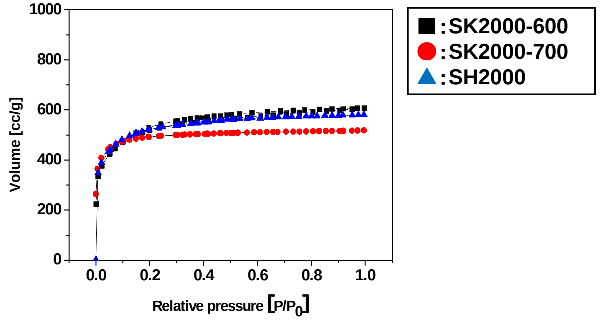
Capacitance, cost, …
How to overcome the differences?

Structure of Activated Carbon

- **Surface Area, Pore: Depth & Volume**
  - Surface Structure
  - Surface Chemistry
  - Based and Edge Plane
  - Hetero atoms in Hexagon
- **Carbon Structure of Wall**
  - Nano, Micro, Macro Structure of Carbon Wall
  - -Graphitization Extent
  - -Domain Structure
    - → Density, Reactivity (Activated Surface)
    - Precursor : Structure and Reactivity

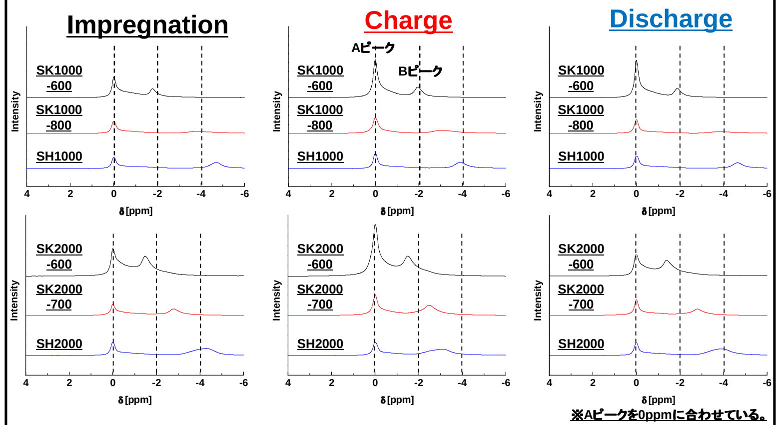


試料名	賦活温度	BET比表面積 [m²/g]	電極密度 [g/ml]	キャパシタンス		
				F/g	F/ml	F/m²
SK1000-600	600℃	1420	0.63	20.4	10.4	0.014
SK1000-800	800℃	1240	0.69	18.7	10.3	0.015
SH1000	800℃	1250	0.73	21.5	14.6	0.017



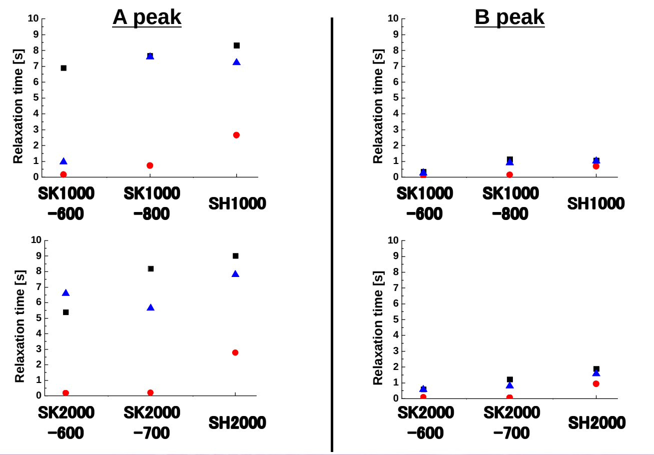
Sample	Activation temperature	BET Surface area [m²/g]	Electrode density [g/ml]	Capacitance		
				F/g	F/ml	F/m²
SK2000-600	600℃	1850	0.63	37.8	16.6	0.020
SK2000-700	700℃	1700	0.60	30.2	14.5	0.018
SH2000	850℃	1850	0.61	24.0	10.4	0.013

^{11}B Solid NMR Spectra at each state



A Peak : Free electrolyte, B Peak : Electrolyte adsorbed on the pore wall

Relaxation time（T_1）on each state

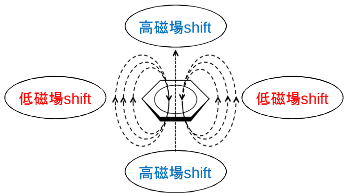


■:IMP ●:Charge ▲:Discharge

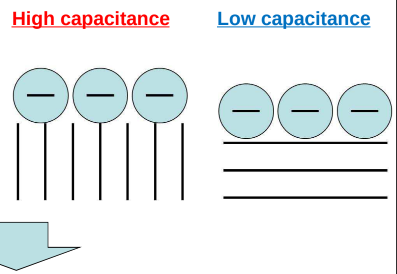
Discussion

Effect of wall surface

Contribution to chemical shift (Ring current effect)



Contribution to capacitance



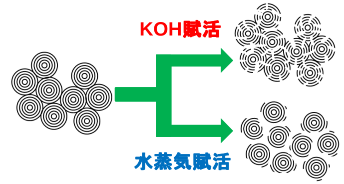
Adsorption on the edge →Lower magnetic field shift, Higher capacitance
→SK2000 is conjectured to have more edges on the pore walls
Adsorption on the basal planes → Higher magnetic field shift, lower capacitance
→SH2000 might have less edges on the pore walls

Discussion

Difference of chemical and steam activations

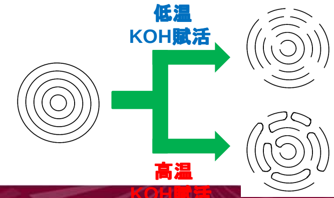
Methodological effect

KOH: Edge surface
Steam: Basal surface



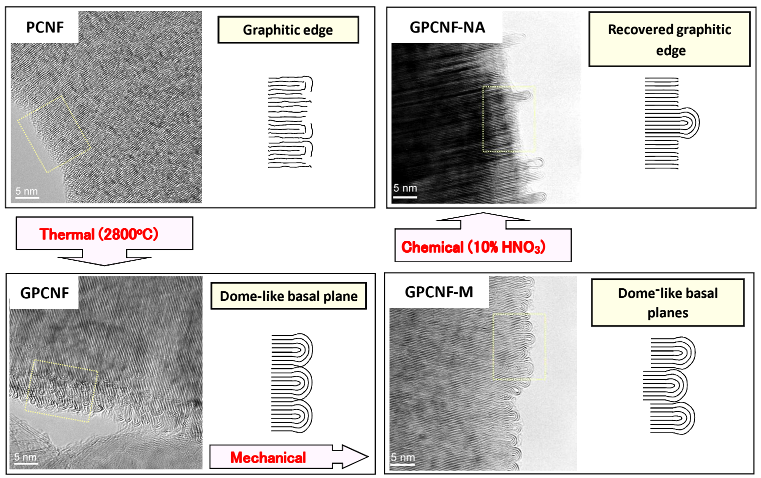
Conditional effect

Less than 600oC:
Most pore walls have edge containing surfaces



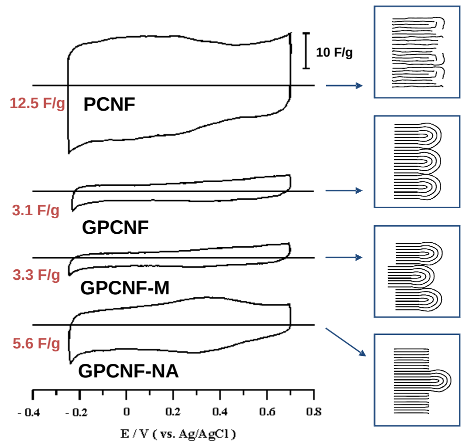
Surface modified PCNFs

Langmuir 2006, 22(22), 9086.



Capacitances of PCNF series

Langmuir 2006, 22(22), 9086.



PCNFs having edge surfaces Showed 3-5 times Higher capacitancs

Conclusion

- **Carbon is a key material for energy and environmental devices.**
- **Full understanding of carbon structure is necessary for useful applications**

- **Korea has a lot of sources for carbon materials.**
- **No manpower and skill for carbon manufacturing.**

University:

- ❖ **Changing the consciousness**
- ❖ **Creation and leading of projects**
- ❖ **Manpower cultivation**