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Andrei VEKSH, Azhar UDDIN, Eiji SASAOKA, Yoshiei KATO

Journal of Chemical Engineering of Japan, Vol. 45, No. 6, pp. 387-394, 2012.

### Prediction of glass structure by using multiple regression analysis

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#### Summary

Multiple regression analyses were applied to the prediction of glass structures, such as O 1s binding energy and fraction of four-fold coordinated boron atoms,  $N_4$ . In the case of linear combination of the content of glass constituents, an acceptable prediction accuracy was obtained for O 1s binding energy, and as for  $N_4$ , however, a poor agreement was observed between the prediction and measurement. After introducing quadratic and cubic interaction terms into the regression formula, a drastic improvement was achieved in the prediction of  $N_4$ . Some regression coefficients were dependent on basicity of each glass constituent, suggesting the feasibility of prediction for the glasses containing novel constituents whose regression parameters have never been determined.



Fig. 1. Results of multiple regression analyses for the fraction of four-fold coordinated boron atoms,  $N_4$  by assuming (a) linear, (b) quadratic and (c) cubic interaction models.

Regression expression used is given in equation (1).

$$\begin{cases} y = \sum_{i} a_{i}x_{i} + \sum_{i < j} a_{ij}x_{i}x_{j} + \sum_{i < j < k} a_{ijk}x_{i}x_{j}x_{k} + a_{other}x_{other} \\ x_{other} = 1 - \sum_{i} x_{i} \end{cases}$$
(1)

where y is the dependent variable, that is, the predicted values of O1s binding energy or  $N_4$ ,  $x_i$  is the *i*-th independent variable, that is, content of *i*-th component, and  $a_i$  is the regression coefficient for  $x_i$ . The second and third terms are introduced to reproduce the interactions between the independent variables, and the quadratic  $x_i x_j$  and cubic  $x_i x_j x_k$  interactions are treated as independent variables. The last term represents the contribution of minor components.

#### Key word

Glass, Structure, Database, Regression analysis, XPS, NMR

#### Affiliation

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#### ■Printing

Journal of the Ceramic Society of Japan, Vol. 120(3), pp. 98-103, 2012.3.

## Effect of TiO<sub>2</sub> Addition on the Distribution of Phosphorus Associated with Phase Separation of Borosilicate Glasses

OHTSUKI Yuki<sup>1)</sup>, SAKIDA Shinichi<sup>2)</sup>, BENINO Yasuhiko<sup>3)</sup>, NANBA Tokuro<sup>4)</sup>

#### Summary Summary

Behavior of phosphorus associated with phase separation and the effect of TiO<sub>2</sub> addition on the phosphorus distribution were investigated in Na<sub>2</sub>O-B<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> ternary glass system. After heat treatment, phase separation by spinodal decomposition was commonly observed. In the phase separation of TiO<sub>2</sub>-free glasses, phosphorus was preferentially distributed into B<sub>2</sub>O<sub>3</sub>-rich phase, and after TiO<sub>2</sub> addition, phosphorus distribution into SiO<sub>2</sub>-rich phase was also observed. With increasing TiO<sub>2</sub> content, the amount of phosphorus distributed into SiO<sub>2</sub>-rich phases increased up to 90%. <sup>31</sup>P MAS NMR indicated that phosphorus in the glasses was mainly present in negatively-charged Q<sup>2</sup> units with terminal oxygen atoms, and in the TiO<sub>2</sub>-containing glasses, Q<sup>1</sup> or Q<sup>0</sup> units containing P-O-Ti bond were confirmed. Despite the formation of negatively-charged Q<sup>1</sup> or Q<sup>0</sup> units, phosphorus was distributed into SiO<sub>2</sub>-rich phase, which was due to the distribution of Ti<sup>4+</sup> ions with higher coordination numbers acting as network modifiers.

Table 1. Analytical compositions of glass phases after phase separation.



#### Key word

Borosilicate glass, Phase separation, Phosphorus, Glass structure

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#### Printing

Processing, Properties, and Applications of Glass and Optical Materials: Ceramic Transactions, Vol. 231, pp. 145-149, 2012.8.

## Functional and Structural Characterizations of Fresnoite Glass-Ceramics Oriented with UST Technique

BENINO Yasuhiko<sup>1)</sup>, ENDO Atsuko<sup>2)</sup>, SAKIDA Shinichi<sup>3)</sup>, NANBA Tokuro<sup>4)</sup>

#### ■ Summary ■

Transparent glass ceramics with highly oriented surface crystalline layers of fresnoite  $Ba_2TiSi_2O_8$  were prepared by heat treatment of precursor glasses in BaO-TiO<sub>2</sub>-SiO<sub>2</sub> system. The orientation along c-axis of fresnoite crystals normal to the glass surface was evaluated by the surface XRD patterns, and it was remarkably enhanced by ultrasonic surface treatment (UST) before the crystallization using fresnoite powder suspension in water. The effect of UST disappeared after heat treatment in the glass transition region, and the mechanism of UST was discussed on the basis of the persistence at elevated temperatures.



Fig. 1. Surface XRD patterns of full crystallized (after HT-2) glass ceramics with various temperatures T1 of preceding heat treatment HT-1 for 1h.

Fig. 3. Change in orientation of surface crystals represented by Lotgering factors, LF characterized by degradation and regeneration of surface crystallization during long term heat treatment HT-1 up to 600 min.

#### Key word

Glass ceramics, Surface crystallization, Ultrasonic surface treatment, Fresnoite

#### Affiliation

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#### Printing

Processing, Properties, and Applications of Glass and Optical Materials: Ceramic Transactions, Vol. 231, pp. 161-165, 2012.8.



Fig. 2. Change in orientation of surface crystals represented by Lotgering factors, LF for surface XRD patterns in Fig. 1.



# Fabrication and characterization of $Er^{3+}$ -doped tellurite glass Waveguides By $Ag^{+}$ - $Na^{+}$ Ion-exchange Method Using a Dry Electromigration Process

SAKIDA Shinichi<sup>1)</sup>, KIMURA Kiyoe<sup>2)</sup>, BENINO Yasuhiko<sup>3)</sup>, NANBA Tokuro<sup>4)</sup>

#### Summary Summary

Planar waveguides were prepared on  $12Na_2O-10NbO_{2.5}-25WO3-53TeO_2-1Er_2O_3$  ([NbWEr]) substrate glass by Ag<sup>+</sup>-Na<sup>+</sup> ion-exchange using a dry electromigration process. The optical properties of the waveguides were characterized and compared with those of [NbWEr] glass waveguides prepared by Ag<sup>+</sup>-Na<sup>+</sup> ion-exchange by thermal diffusion. After ion-exchange by both a dry electromigration process and thermal diffusion, the transmittance of the glasses slightly decreased from that of the [NbWFr] substrate glass but frosting was not observed on these ion-exchanged glasses. The depth of the waveguide fabricated at 300°C by a dry electromigration process was about 8  $\mu$ m and deeper than that at 380°C by thermal diffusion. On the [NbWEr] glass ion-exchanged at 300°C by a dry electromigration process, the stains seen in the [NbWEr] glass ion-exchanged at 380°C by thermal diffusion were hardly observed. Therefore, the dry electromigration process is promising due to the reduction of the process temperature and fewer stains.



Fig. 1. Transmission spectra of the substrate glass (Non I-E) and glasses ion-exchanged at 290-300°C tor 5 h using a dry electromigration process and at 380°C for 5 h by thermal diffusion for [NbWEr].

Fig. 3. Photographs of [NbWEr] substrate glass (left) and [NbWEr] glass ion-exchanged at 300°C for 5 h using a dry electromigration process (right).



Fig. 2. Relation between the fraction of  $Ag^+/(Ag^++Na^+)$  and the depth of [NbWEr] waveguides fabricated by  $Ag^+-Na^+$  ion-exchange at 290 and 300°C using a dry electromigration process by EDS.



#### Key word

Planar waveguide, Ion exchange, Dry electromigration process, Thermal diffusion process, Tellurite glass

#### Affiliation

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#### Printing

Processing, Properties, and Applications of Glass and Optical Materials: Ceramic Transactions, Vol. 231, pp. 201-207, 2012.8.

## Effect of additives on the distribution of phosphorus associated with phase separation of borosilicate glasses

BENINO Yasuhiko<sup>1)</sup>, OHTSUKI Yuki<sup>2)</sup>, SAKIDA Shinichi<sup>3)</sup>, NANBA Tokuro<sup>4)</sup>

#### Summary

Sodium borosilicate glasses containing phosphorus were prepared, and the effect of  $Al_2O_3$  and  $TiO_2$  addition on the distribution behavior of phosphorus atoms associated with the phase separation was investigated. After heat treatment, a phase separation by the spinodal decomposition was commonly confirmed by SEM observation. In the additive-free sample, phosphorus was preferentially distributed into  $B_2O_3$ -rich phase after the phase separation, and after adding  $Al_2O_3$  or  $TiO_2$ , the amount of phosphorus distributed into  $SiO_2$ -rich phase increased. It was also suggested that  $TiO_2$  had a stronger influence on the phosphorus distribution into  $SiO_2$ -rich phase than  $Al_2O_3$ . <sup>31</sup>P MAS-NMR revealed that most of phosphorus atoms in the additive-free glass were present in  $PO_4$  units consisting of two terminal oxide ions, and the terminal oxide ions decreased and increased after the additions of  $Al_2O_3$  and  $TiO_2$ , respectively. It was suggested that this structural change brought about a change in the distribution of phosphorus.



Fig. 1. SEM photos of the glass surfaces heated at  $600^{\circ}$ C for 16 h.



#### Key word

Phase separation, Borosilicate glass, Phosphorus distribution, Glass structure

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#### Printing

Journal of the Ceramic Society of Japan, Vol. 120(11), pp. 490-493, 2012.11.

### Surface crystallization of stoichiometric glass with $Bi_2ZnB_2O_7$ crystal using ultrasonic surface treatment followed by heat treatment

KANENISHI Keita<sup>1)</sup>, SAKIDA Shinichi<sup>2)</sup>, BENINO Yasuhiko<sup>3)</sup>, NANBA Tokuro<sup>4)</sup>

#### Summary Summary

Surface crystallized glass-ceramics with nonlinear optical Bi<sub>2</sub>ZnB<sub>2</sub>O<sub>7</sub> (BZB) crystals were prepared by ultrasonic surface treatment (UST) followed by crystallization heat treatment for stoichiometric glass with a composition of 33.3Bi<sub>2</sub>O<sub>3</sub>-33.3ZnO-33.3B<sub>2</sub>O<sub>3</sub>. According to the surface XRD measurements and microscopic observations, UST technique was found to be quite effective for lower temperature precipitation of BZB, indicating the ability of crystallization control of the target phase, such as phase selectivity, small size of precipitates, high orientation and their morphology. After the optimization of the process condition parameters for improving transparency and suppressing deformation of the sample, the effective optical nonlinearity of the transparent surface crystallized glass-ceramics was estimated as 0.7 pm/V.



Fig. 1. Surface XRD patterns of surface crystallized glass-ceramics after heat treatment at 420-440°C for 12 h together with powder pattern of synthesized BZB.





Fig. 2. Surface XRD patterns of surface crystallized glass-ceramics after UST and heat treatment at 400-420°C for 12 h together with powder pattern of synthesized BZB.

Fig. 3. Optical observations of (a) the glass-ceramic sample appearance and (b) the surface crystallization by polarization microscope with a sensitive tint plate. The sample was obtained by UST and long-term heat treatment at 410°C for 96 h.

#### Key word

Surface crystallization, Bismuth zinc borate glass, Glass-ceramics, Ultrasonic surface treatment, Optical nonlinearity

#### Affiliation

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#### Printing

Journal of the Ceramic Society of Japan, Vol. 120(11), pp. 509-512, 2012.11.

### Effect of $Nb_2O_5$ addition to $SnO-P_2O_5$ glass

FUKUI Satoshi<sup>1)</sup>, SAKIDA Shinichi<sup>2)</sup>, BENINO Yasuhiko<sup>3)</sup>, NANBA Tokuro<sup>4)</sup>

#### Summary

 $SnO-P_2O_5$  glasses have high refractive index and low glass transition temperature but have poor water durability. To improve water durability,  $NbO_{2.5}$  was added to  $SnO-P_2O_5$  glasses, preparing  $SnO-NbO_{2.5}-P_2O_5$  glass. It was found that addition of only 4 mol%  $NbO_{2.5}$  was enough to achieve significant improvement of water durability, and at the same time, no degradation in thermal and optical properties was observed.



#### Key word

SnO-P2O5 glass, Nb2O5, Glass transition temperature, Refractive index, Water durability

#### Affiliation

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#### Printing

Journal of the Ceramic Society of Japan, Vol. 120(11), pp. 509-512, 2012.11.

### ガラスの相分離を利用した有用元素の回収

Recovery of Valuable Elements by Using Phase Separation of Glass

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#### ■ 概 要 ■

鉄鋼スラグや都市ゴミの高温溶融スラグは、SiO<sub>2</sub>、CaO、Al<sub>2</sub>O<sub>3</sub>などの成分を多く含み、性状が石や砂 に似ていることから,砂利や砂の代替として路盤材やセメント原料などとして再利用されている.しか し、需要には限りがあるため、最終処分されるスラグの量も少なくなく、新たな用途の開拓が求められ ている. 無機系廃棄物(再資源化物を含む)の多くは、もともとガラス質であったり、ガラスに近い化 学組成を有していることから、ガラス原料などへのリサイクルが期待される.しかし、鉄をはじめとす る遷移金属元素により無機系廃棄物は着色しており、そのままでは無色透明なガラスを作製することが できない.着色原因である遷移金属元素を除去することができれば,廃棄物は無色透明なガラス原料と して再利用できると考えた. 遷移金属元素を除去する方法として, ガラスの相分離が有効ではないかと 考えた. ガラスの相分離自体は古くから知られた現象であり、工業的にも多孔質ガラスやバイコールガ ラスの作製に広く用いられている.我々は,ガラスの相分離を無機系廃棄物に含まれる遷移金属元素な どの着色成分の抽出にも適用できると考えた. 高炉水砕スラグや都市ゴミ溶融スラグを用いて実験を行 ったところ, SiO2含有量の高い無色透明なガラス固化体を得ることができた.また、リンやチタンがシ リカリッチ相に選択的に取り込まれることを見いだし、ガラスの相分離をシリカ以外の有価元素の抽出 法として利用できると考えた、図1に、当研究室で考えているガラスの相分離を用いた有価元素の回収 プロセスの概要を示す.本稿では、当研究室が行ってきたプロセス開発と問題点の克服に向けた取り組 みについて紹介した.



#### ■ 掲載先 ■

セラミックス, Vol. 47(2), pp. 105-108, 2012.2.

査読:なし,言語:日本語

#### Pr<sub>2</sub>NiO<sub>4</sub>系酸化物の中温固体酸化物燃料電池カソード特性

Electrode properties of doped Pr<sub>2</sub>NiO<sub>4</sub>-based oxide cathode for intermediate-temperature SOFCs

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#### ■ 概 要

K<sub>2</sub>NiF<sub>4</sub>型構造を有する Pr<sub>2</sub>NiO<sub>4</sub> 系酸化物(Pr<sub>2</sub>Ni<sub>0.9</sub>Cu<sub>0.1</sub>O<sub>4</sub>, Pr<sub>2</sub>Ni<sub>0.75</sub>Cu<sub>0.25</sub>O<sub>4</sub>, Pr<sub>1.91</sub>Ni<sub>0.71</sub>Cu<sub>0.24</sub>Ga<sub>0.05</sub>O<sub>4</sub>, or Pr<sub>2</sub>Ni<sub>0.9</sub>Co<sub>0.1</sub>O<sub>4</sub>)の中温固体酸化物燃料電池カソード特性が調査された。電気伝導度測定および単セル (電解質:サマリウムドープドセリア(SDC)、アノード:Ni と SDC のサーメット、カソード:Pr<sub>2</sub>NiO<sub>4</sub> 系酸化物試料)における発電特性評価の結果、Pr<sub>2</sub>NiO<sub>4</sub> 系酸化物は優れたカソード特性を示し、Ni サイトへの遷移金属ドープがカソード特性の向上に効果があることが分かった。



 $\frac{1000/T (1/K)}{Fig. 1 Temperature dependence of the electrical conductivity for Pr_2NiO_4 (circles), Pr_2Ni_{0.9}Cu_{0.1}O_4 (riangles), Pr_2Ni_{0.75}Cu_{0.25}O_4 (squares), (Pr_{1.91}Ni_{0.71}Cu_{0.24}Ga_{0.05}O_4 (diamonds), and Pr_2Ni_{0.9}Co_{0.1}O_4 (inverted triangles).$ 



Fig. 2 Cell performance (power density vs. current density) at 700°C for  $Pr_2NiO_4$  (circles),  $Pr_2Ni_{0.9}Cu_{0.1}O_4$  (triangles),  $Pr_2Ni_{0.75}Cu_{0.25}O_4$  (squares),  $Pr_{1.91}Ni_{0.71}Cu_{0.24}Ga_{0.05}O_4$  (open diamonds),  $Pr_2Ni_{0.9}Co_{0.1}O_4$  (inverted triangles), and LSCF (solid diamond).

■キーワード■

中温固体酸化物燃料電池,カソード特性, Pr<sub>2</sub>NiO<sub>4</sub>系酸化物

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J. Ceram. Soc. Japan, 120, 534-538 (2012)

査読:あり,言語:英語

赤外吸収スペクトルのピーク分離による層状複水酸化物の層間イオンの定量解析法

Determination of carbonate ion contents in layered double hydroxides by FTIR spectrometry

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#### ■ 概 要 ■

層状複水酸化物(LDH)のフーリエ変換赤外吸収(FTIR)スペクトルに対し、カーブフィッティング 法によるピーク分離を行うことで、 $CO_3^{2-}$ 含有量の定量解析法を検討した.FTIR 法で算出した  $CO_3^{2-}$ 含 有率は組成分析で決定した値とよく一致しており、従来困難であった  $CO_3^{2-}$ の含有量を直接定量できる 簡便法であることが示された.





Fig. 1 Deconvolution of the FTIR spectrum of the starting carbonate-LDH in the region 1100-1900 cm<sup>-1</sup>.

Back side cover article

Fig. 2 Relationship between the  $\text{CO}_3^{2-}$  contents (%) of carboxylate-LDHs and the molar ratio of  $2(\text{CO}_3^{2-})/\text{Al} \times 100$  (%). The circle ( $\circ$ ), triangle ( $\blacktriangle$ ), and square ( $\Box$ ) symbols correspond to formate-, acetate-, and propionate-LDH, respectively.

■キーワード■
層状複水酸化物、イオン交換、赤外吸収スペクトル、カーブフィッティング法、ピーク分離
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■ 掲載先 ■
Anal. Methods, 4, 3925-3927, (2012).

査読:あり,言語:英語

# Morphological Diversity and Nanofiber Networks of Poly(p-oxybenzoyl) generated by Phase Separation during Copolymerization

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#### ■ Summary ■

Poly(p-oxybenzoyl) (POB) precipitates prepared by reaction-induced phase separation during copolymerization exhibit wide variety of morphologies such as fibril, needle, slab, spindle, and sphere. The morphology is significantly influenced by the copolymerization conditions of structure of trifunctional comonomers, copolymerization ratio, solvent, concentration, and temperature of the polymerization. Among them, POB nanofiber networks like a texture of nonwoven fabrics are obtained only by the polymerization of 4-acetoxybenzoic acid with 3,5-diacetoxybenzoic acid (DABA) in aromatic solvent at a concentration of 1.0-2.0% at the content of DABA in feed of 10-20 mol % at  $320 \, \Gamma^{\circ}$ C. The network is comprised of fibrillar crystals connected each other at spherical nodal points. The diameters of the fibers are 90-129 nm. This network is highly crystalline and the molecules are aligned along the long direction of the fibers. Various parameters characterized the networks can be controlled by the polymerization concentration.



**Figure.** Feature of as-prepared POB network and SEM micrographs of POB network prepared from ABA and DABA at a concentration of (a) 1.0%, (b, c) 1.5%, and (d) 2.0%. Polymerizations were carried out in DBT at  $\chi_f$  of 15 mol %, and 320°C for 6 h.

#### Key word

polyesters; fibers; morphology; phase behavior; crystallization

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#### Printing

Journal of Applied Polymer Science

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Refreeing: Full paper, Language: English

# Morphosynthesis of poly(ether ketone) by reaction-induced crystallization during polymerization

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#### ■ Summary ■

Morphology control of poly(ether ketone) (PEK) was examined by using the crystallization during the nucleophilic aromatic substitution reaction of potassium salt of 4-fluoro-4'-hydroxybenzophenone. Polymerizations were carried out at 290 °C. The PEK was obtained as precipitates and its morphology was highly influenced by the polymerization condition such as the solvent, the concentration and the polymerization time. High crystalline spindle-like crystals were obtained by the polymerization in diphenyl sulfone (DPS) at a concentration of 5.0% for 2 h with the yield of 86%. The average length and width were 1.4 µm and 300 nm respectively, and the maximum thickness was 130 nm. The surface was not smooth and it was hilly. The spindle-like crystal was likely consisted of multilayered lamellae comprised of the microcrystallites. The molecules were oriented perpendicular to the lamella. The polymerization in DPS at a higher concentration of 10.0% afforded the networks of nanofibres, of which the diameter was 100–250 nm. The obtained PEK precipitates possessed excellent thermal properties.



#### Key word

Poly(ether ketone); Morphology; Reaction-induced crystallization; Spindle; Fibre

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#### Printing

European Polymer Journal

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Refreeing: Full paper, Language: English

# Morphosynthesis of poly[4-(1,4-phenylene)oxyphthalimide] and copolymers prepared by reaction-induced crystallization during polymerization

Takashi Sawai<sup>1)</sup>, Kanji Wakabayashi<sup>1)</sup>, Shinichi Yamazaki<sup>2)</sup>, Tetsuya Uchida<sup>3)</sup>, Kunio Kimura<sup>4)</sup>

#### ■ Summary ■

Morphosynthesis of poly[4-(1,4-phenylene)oxyphthalimide] (POPI) and poly[4-(1,4-phenylene)oxyphthalimide-co-4-phthalimide] (POPI-PPI) was examined by using the crystallization during the polymerization. The POPI fibrillar crystals were obtained as precipitates with the formation of spherical aggregates of plate-like crystals. Some of the POPI fibrillar crystals were longer than 15  $\mu$ m. They possessed high crystallinity and the molecules aligned perpendicular to the long direction of the fibers. On the other hand, one-dimensional structures of POPI-PPI such as ribbon, cone, rod, and fiber were obtained as precipitates by the copolymerization. The copolymer molecules might align along the long direction of the polymerization of the spleteness of the poly(ether-imide) could be controlled by not only the polymerization condition but also with the aid of copolymerization.



**Figure.** Morphologies of POPI precipitates prepared in (a) run no. 1, (b) run no. 2, (c) run no. 3, (d) run no. 4, (e) run no. 5, (f) run no. 6, and (g) run no. 7.



**Figure.** Morphologies of (a) PPI, (b) POPI-PPI-10, (c) POPI-PPI-30, (d) POPI-PPI-50, (e) POPI-PPI-70, and (f) POPI-PPI-90.

#### Key word

crystallization; fibers; high performance polymers; morphology; polyimides

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#### Printing

Journal of Polymer Science Part B-Polymer Physics "Cover Article", Vol. 50, No. 18, pp. 1293-1303, 2012. DOI: 10.1002/polb.23122

Refreeing: Full paper, Language: English

フルクトースからの 5-ヒドロキシメチルフルフラールの抽出に関する簡易モ デル: 高温高圧条件下における水/油二相系の利用 Simplified Model for Extraction of 5-Hydroxymethylfulfural from Fructose: Use of Water/Oil Biphasic System

Simplified Model for Extraction of 5-Hydroxymethylfulfural from Fructose: Use of Water/Oil Biphasic System under High Temperature and Pressure Conditions

T. Shimanouchi<sup>1)</sup>, Y. Kataoka<sup>2)</sup>, M. Yasukawa<sup>3)</sup>, T. Ono<sup>4)</sup>, Y. Kimura<sup>5)</sup>

#### Summary

5-Hydroxymethylfulfural (HMF), which is a useful component, was obtained from the dehydration of fructose treated by the high temperature and pressure condition (180 oC and 10 MPa). To avoid the decomposition of HMF in the bulk aqueous phase, we dehydrated fructose in the water / metyl isobuthyl ketone (MIBK) biphasic system. The HMF could be successfully extracted in the organic MIBK phase without the further decomposition of HMF. We presented the simplified model taking into consideration of the slug flow with wall film to get HMF with the maximal yield in MIBK phase. It was likely that the slug flow contributed to the increase in surface-to-volume for the mass transfer of HMF.



Fig. Experimental instruments for HMF production using flow system



Fig. Time-course of fructose and HMF in (a) batch system and (b) flow system. Reaction conditions are: (a)  $[HCl]_0 = 0.10 \text{ mol/l}$ ; (b)  $[HCl]_0 = 0.0250 \text{ mol/l}$ .  $V_{aq}/V_{org} = 0.8$ . Reaction temperature was 180 °C.

#### Key word

- 5-Hydroxymethylfulfural, Extraction, Mass transfer, Slug flow
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#### Printing

Solvent Extraction Research and Development Japan, in press.

ラット門脈による非消化性二糖類, ココナッツ由来β-1,4-マンノビオース, の 吸収

Absorption of the Indigestible Disaccharide,  $\beta$ -1,4-Mmanobiose, from Coconut by the Rat Portal Vein

H. Kanatani<sup>1)</sup>, Y. Kimura<sup>2)</sup>, M. Asanoma<sup>1)</sup>, A. Nakamura<sup>1)</sup>, M. Hirotsuka<sup>1)</sup>, S. Adachi<sup>3)</sup>

#### Summary

The intestinal absorption of  $\beta$ -1,4-mannobiose by rats was investgated. Mannobiose was detected in the portal vein plasma by matrix-assisted laser desorption ionization / time of flight mass spectrometry after its administration to rats. The presence of mannobiose in the rat plasma was confirmed by an experiment using b-mannosidase. These results indicate that mannobiose was directly absorbed through the intestines even without being hydrolyzed.

Fig. (Right) MALDI-TOF-MS analysis of the Ret Portal Vein Plasma. A. 90 min after administering mannobiose, B. before administration. C: MS / MS analysis of rat plasma after administering mannobiose.

Table 1. Concentrations of Mannobiose and Mannose in the Plasmaof Rats after the Treatment with  $\beta$ -Mannosidase

|            | Concentration [mM] |                   |            |
|------------|--------------------|-------------------|------------|
|            | Before             | After             | Difference |
| Mannobiose | $0.079 \pm 0.010$  | $0.062\pm0.008$   | -0.017     |
| Mannose    | $0.770 \pm 0.079$  | $0.804 \pm 0.087$ | 0.034      |



| Key | word |  |
|-----|------|--|
|-----|------|--|

 $\beta$ -1,4-mannobiose, intestinal absorption, rat, in vivo

Affiliation

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Printing

Bioscience, Biotechnology, Biochemistry, 76, 575-577 (2012)

電場形成法によるジャイアントベシクルの成長挙動:膨潤過程と変形に対す るタンパク質の効果

Growth Behavior of Giant Vesicles Using The Electroformation Method: Effect of Proteins on Swelling and Deformation

T. Shimanouchi<sup>1)</sup>, H. Umakoshi<sup>2)</sup>, R. Kuboi<sup>3)</sup>

#### Summary

The growth of giant vesicles (GVs) can be considered as a consecutive process of swelling / detachment / deformation, which is a response of lipid membranes on solid surfaces to the solvent and environmental factors such as temperature and ionic strength. The electroformation method allows to visualize the responses to such factors. The additive effect of the protein on the growth of GVs, composed of zwitterionic phospholipids, was herein investigated using the electroformation method. Proteins denatured by a pH-shift (to be in the *Molten Globule* state) perturbed the lipid membranes, resulting in the acceleration of GV growth. The GVs detached from the electrode showed deformation close to a stomatocyte. It was revealed that common factor for the response of lipid membranes was the variation of the apparent area elastic modulus associated with the interaction between proteins and lipid membranes. The present finding affords better understanding about the response of lipid membranes on solid surfaces under a variety of environment factors.



Apparent area elastic modulus  $K_a$  [N/m]

Fig. 1 Relationship between  $k_{\rm Gr}$  and apparent area elastic modulus  $K_{\rm a}$ . Dotted curve is a fitting curve with equation (3) assuming  $K_{\rm a}^{-1} \propto L$ . The concentration of lipids was about 13µM in each experiment.



Fig. 2 Morphology of GVs detached from Pt electrode. (i) sphere, (ii) stomatocyte, (iii) discocyte, (iv) pear, (v) dumbbell, (vi) tubular membranes, (vii) rod, (viii) necklace. Those GVs were observed after 2 hours later of the application of AC electric field.

Key word

Giant Vesicle, Electroformation, Protein, Deformation, Membrane perturbation

Affiliation

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Printing

Journal of Colloid and Interface Science, in press.

ドーパミンによるアミロイドβ線維の可溶化に対するリポソーム膜の効果 Effect of liposome membranes on disaggregation of amyloid β fibrils by dopamine

H. T. Vu<sup>1</sup>, T. Shimanouchi<sup>2</sup>, D. Matsumoto<sup>3</sup>, T. Matsumoto<sup>3</sup>, H. Yagi<sup>4</sup>, Y. Goto<sup>4</sup>, H. Umakoshi<sup>5</sup>, R. Kuboi<sup>6</sup>

#### Summary

The inhibition of fibril formation of amyloid  $\beta$  (A $\beta$ ) and the disaggregation of A $\beta$  fibrils are the promising approaches for a medical treatment of Alzheimer's disease (AD) therapy. In this study, we investigated the effects of liposomes on dopamine-induced disaggregation of  $A\beta$  fibrils by using the variety of liposomes. The used liposomes were normal liposomes, raft-forming liposomes, charged liposomes and oxidized liposomes. Those liposomes could accelerate the disaggregation rate of fibrils. From the comparison of normal and charged liposomes, a certain contribution of dopamine via an electrostatic interaction to the disaggregation was confirmed. From raft-forming and oxidized liposomes, we revealed a significant contribution of bound water to liposomes, which could assist the formation of the quinine-form of dopamine by a removal of its proton. It is, therefore, concluded that the membrane surface of liposomes is considered to be an adequate environment for the dopamine-induced disaggregation of fibrils.



Fig. (a) Time-course of ThT fluorescence intensity in the absence and the presence of DMPC/SA (10:4) liposome. Solid curves were well-fitted curves with Eq. (1). Effect of liposome on apparent disaggregation rate constant for (b)  $A\beta(1-40)$ . Fibrils were disaggregated by 100  $\mu$ M DA in the absence and presence of 1 mM liposomes as follows: (1) POPC/SM (7:3); (2) POPC/CH/SM (1:1:1); (3) POPC/SM/CH/GM1 (3:3:3:1); (4) POPC; (5) POPC/CH (7:3); (6) DMPC; (7) DMPC/SA (10:4); (8) DMPC/SAPCox (6:4); (9) DMPG/SAPCox (6:4); (10) POPA; (11) DOTAP; (12) POPC/DOTAP (7:3).

#### Key word

Giant Vesicle, Electroformation, Protein, Deformation, Membrane perturbation

Affiliation

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#### Printing

Biochemical Engineering Journal, in press.

リポソーム膜上における  $A\beta$ プロトフィブリルの成長挙動と膜攪乱効果 Growth Behavior of  $A\beta$  Protofibrils on Liposome Membranes and their membrane perturbation effect

T. Shimanouchi<sup>1)</sup>, K. Nishiyama<sup>2)</sup>, A. Hiroiwa<sup>2)</sup>, H.T. Vu<sup>3)</sup>, N. Kitaura<sup>2)</sup>, H. Umakoshi<sup>4)</sup>, R. Kuboi<sup>5)</sup>

#### Summary

The protofibrils of amyloid fibrils have recently drawn an attention due to their cytotoxicity although the physicochemical properties of the protofibrils as an *intermediate* of fibrillation process have still remained unclear. We investigated the growth behavior of the protofibrils of amyloid  $\beta$  protein with 40 amino acid residues in the presence of various liposomes and the membrane perturbation (calcein leakage). The growth behavior of protofibrils was apparently correlated with the protofibrils-induced calcein leakage. From the binding experiment using a hydrophobic fluorescence probe and the quartz crystal microbalance method combined with the planar lipid membrane, it is suggested that the apparent correlation between the growth behavior of protofibrils and their membrane perturbation resulted from the physicochemical properties of protofibrils such as hydrophobicity and the stability of hydrogen bonds. The protofibrils were found to show the highest hydrophobicity and the highest instability of hydrogen bonds of the proteins used in this study (monomer, protofibrils, matured fibrils, and other three typical proteins). It is anticipated that such properties of protofibrils were advantageous both for the binding of protofibrils with monomer (growth behavior) and for the binding of lipid bilayer membranes (membrane perturbation).

![](_page_26_Figure_6.jpeg)

Fig. 1 TIRFM and TEM images of suspension after the maturation of protofibrillar  $A\beta(1-40)$ . Protofibrils were matured during (a,b) 0h and (c,d) 20 h in the presence of neither liposomes nor Triton X-100. (e,f) 20 h in the presence of DOPC liposome. (g,h) 20 h in the presence of TritonX-100. The bar in TIRFM and in TEM represents 10  $\mu$ m and 200 nm in length, respectively.

#### Key word

Protofibril, Amyloid, Liposome, Hydrophobicity, Hydrogen bonds

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- Printing

Biochemical Engineering Journal, in press.

負電荷リポソームによる球状アミロイドβ凝集体の形成 Formation of spherulitic amyloid β aggregate by anionic liposomes

T. Shimanouchi<sup>1)</sup>, N. Shimauchi<sup>2)</sup>, R. Ohnishi<sup>2)</sup>, N. Kitaura<sup>2)</sup>, H. Yagi<sup>3)</sup>, Y. Goto<sup>3)</sup>, H. Umakoshi<sup>4)</sup>, R. Kuboi<sup>5)</sup>

#### Summary

Alzheimer's disease is the most common form of senile dementia. This neurodegenerative disorder is characterized by an amyloid deposition in senile plaques, composed primarily of fibrils of an aggregated peptide, amyloid  $\beta$  (A $\beta$ ). The modeling of a senile plaque formation on a model neuronal membrane under the physiological condition is an attractive issue. In this study, we used anionic liposomes to model the senile plaque formation by A<sub>β</sub>. The growth behavior of amyloid A $\beta$  fibrils was directly observed, revealing that the induction of the spherulitic  $A\beta$  aggregates could result from the growth of seeds in the presence of anionic liposomes. The seeds of  $A\beta$  fibrils strongly interacted with negatively charged liposome and the subsequent association of the seeds were induced to form the seed cluster with many growth ends, which is advantageous for the formation of spherulitic  $A\beta$ aggregates. Therefore, anionic liposomes mediated not only fibril growth but also the aggregation process. These results imply that anionic liposome membranes would affect the aggregate form of  $A\beta$  fibrils. The modeling of senile plaque reported here is considered to have great potential for study on the amyloidosis.

![](_page_27_Figure_6.jpeg)

Fig. Possible scheme on formation of spherulitic fibrillar aggregate and SEM images of (a) seeds of amyloid fibrils and (b) mixture of seeds/DMPG liposomes (80min later). (c) TEM image of non-liposome region and (d) liposome region of grew fibrils from seeds/DMPG liposomes (180min later) and its AFM image (insert in d). TIRFM images of the time-course of amyloid fibril formation of A $\beta$  fibrils in (e-h) the presence and (i-l) the absence of seeds. Bars: (white)10 µm (black) 200 nm.

#### Key word

Protein aggregation, Amyloid, Liposome, Alzheimer's disease

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Printing

Biochem. Biophys. Res. Comm., 416, 165-171 (2012)

エンドソーム pH 環境におけるホスホリパーゼ C 介在型膜融合 Membrane fusion mediated by phospholipase C under endosomal pH conditions

T. Shimanouchi<sup>1)</sup>, H. Kawasaki<sup>2)</sup>, M. Fuse<sup>2)</sup>, H. Umakoshi<sup>3)</sup>, R. Kuboi<sup>4)</sup>

#### Summary

Phospholipase C (PLC) is considered to be one of key enzymes for the design of drug delivery system using the endocytosis route, because PLC can catalyze the membrane fusion between cell membranes and phospholipid vehicles (liposomes). Membrane fusion by PLC was then studied under various pHs to model the endosomal environment. The used liposomes were composed of 1,2-dioleoyl-sn-glycero-3- phosphocholine (DOPC), 1,2-dipalmitoyl-sn-glycero-3-phosphocholine

(DPPC), and cholesterol (Ch). The membrane fusion was dominated by the enzymatic reaction at pH 6–7.5. In contrast, the membrane perturbation effect due to the conformational change of PLC could induce the membrane fusion at around pH 4. The maximal value of membrane fusion was observed at around pH 5 for three liposomes in the order of DOPC < DOPC/DPPC (1:1) < DOPC/DPPC/Ch (1:1:1). From the experiments with the hydrophobic fluorescence probe and the circular dichroic method, it was revealed that PLC took a molten-globule state, with a large fluctuation and an enzymatic activity, at 4-5. The DAG-rich around pН domain enzymatically produced by PLC played role for the field for the membrane perturbation enough to lead to the membrane fusion. The present finding would be helpful to understand the behavior of membrane fusion under the late endosomal pH condition in cell system.

![](_page_28_Figure_7.jpeg)

Fig. pH-dependency of (a) the fusion ration of DOPC/DPPC/Ch (1:1:1) in the absence of o-Phe. (Circle) Cobalt/calcein system and (triangle) ANTS/DPX system. (b) Produced DAG (enzymatic activity of PLC) on DOPC/DPPC liposomes in the absence and the presence of o-Phenanthololine (o-Phe).

#### Key word

Membrane fusion, Phospholipase C, Diacylglycerol, Membrane perturbation

Affiliation

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Printing

Colloids and Surfaces B, in press

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リポソーム膜上におけるアミロイド線維の二次核化 Secondary Nucleation of Amyloid Fibrils on Liposome Membranes

T. Shimanouchi<sup>1)</sup>, N. Kitaura<sup>2)</sup>, R. Ohnishi<sup>2)</sup>, H. Umakoshi<sup>3)</sup>, R. Kuboi<sup>4)</sup>

#### Summary

The amyloid fibrils of amyloid  $\beta$  protein  $(A\beta)$  from Alzheimer's disease are likely to show the cytotoxicity, depending on their morphology. The relationship between the nucleation kinetics of the A $\beta$  fibrils and their morphology has been investigated. From the perspective of a crystallization technique assuming primary /secondary nucleation steps and an elongation step, the secondary nucleation rate, В  $[\# m^{-3}s^{-1}],$ was experimentally and coarsely determined by using total internal reflection fluorescence microscopy combined with thioflavin T. In an aqueous solution, linear and rigid fibrils were formed with a relatively smaller B value (( $2.83\pm0.55$ ) x  $10^5 \text{ # m}^{-3}\text{s}^{-1}$ ), whereas spherulitic amyloid assemblies were formed in the presence of negatively charged liposome including oxidized lipids, with a larger B value ((7.65 $\pm$ 0.47) x 10<sup>5</sup> # m<sup>-3</sup>s<sup>-1</sup>). Those findings should lead to a better understanding of the mechanism for the formation of fibrils and senile plaques in Alzheimer's disease.

![](_page_29_Figure_6.jpeg)

Fig. (A) Direct observation of growth behavior of a linear and rigid fibril. (B) Time-course of changes in fibril length. (C) Relationship between the elongation rate of fibrils and their initial length.  $A\beta(1-40)$  monomer: 50  $\mu$ M, ThT: 10  $\mu$ M. White bar represents 10  $\mu$ m.

| Liposome                        |                                      | Habits of amyloid                 | Major possible<br>mechanism of | Secondary nucleation<br>ratee                        |  |
|---------------------------------|--------------------------------------|-----------------------------------|--------------------------------|--|--|
| Lipid composition₽              | Surface state                        | fibrils*, secondary nucleation\$, |                                | B [# m <sup>-3</sup> s <sup>-1</sup> ]+ <sup>3</sup> |  |
| No liposome₽                    | -47                                  | Type I₽                           | -47                            | $(2.83 \pm 0.55) \times 10^{5}$                      |  |
| DMPC+2                          | Zwitterionic <sup>2</sup>            | Type III₽                         | (v)₽                           | $(1.35 \pm 0.23) \times 10^{5}$                      |  |
| DMPC/SAPCox +<br>(70/30 mol%)+  | Zwitterionic/+<br>Oxidized+          | Type III₽                         | (ii),(iv),(v)↔                 | $(1.63 \pm 0.21) \times 10^{5}$                      |  |
| DMPG+2                          | Negatively<br>charged∻               | Type III₽                         | (v)~                           | $(4.03 \pm 0.68) \times 10^4$                        |  |
| DMPG/SAPCox+'<br>(70/30 mol%)+' | Negatively<br>charged/५<br>Oxidized+ | Type II₽                          | (ii)-(iv)+²                    | $(7.63 \pm 1.2) \times 10^{4_{4^{2}}}$               |  |
| DMPA+2                          | Anionic₽                             | Type III₽                         | (v)+                           | (1.58±0.33)×105                                      |  |
| DMPA/SAPCox+<br>(70/30 mol%)+   | Anionic/↓<br>oxidized↓               | Type II₽                          | (ii)-(iv)+²                    | $(7.65 \pm 0.47) \times 10^{5}$                      |  |
| *Type I: straight and ri        | gid fibrils, Type                    | II: spherulite, Type              | e III is likely to include     | a lateral association.                               |  |

\$ Major mechanisms on secondary nucleation are: (i) breaking, (ii) forking, (iii) diffusible, (iv) branching, (v) thickening. The *B* value were estimated under the experimental conditions such as  $A\beta(1-40)$  monomer: 5 µM, liposome: 250 µM, 37°C, pH 7.4, ThT : 10 µM.<sup>4</sup>

#### Key word

Membrane fusion, Phospholipase C, Diacylglycerol, Membrane perturbation

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- Printing
- AIChE Journal, 57, 3625-3632 (2012)

#### Membranome を基盤とする Bio-Inspired 膜へのアプローチ An Approach for "Bio-Inspired Membrane" Based on Membranome

馬越大<sup>1)</sup>, 島内寿徳<sup>2)</sup>, 菅 恵嗣<sup>3)</sup>

#### ■ 概 要 ■

「Bio-Inspired 膜」という視点を切 り口にして「生体膜」と「人工膜」が 関連する 膜学研究全体を, 分子・分子 集合体からデバイスに至る異なる階 層に視点を置きつつ俯瞰し, 今後の膜 学研究の方向性について考察する事 を目的としている.本稿の後半部分で は、特に、Membranome 戦略に基づく 研究成果の一例を概説した. 第一に, 人工膜・生体膜を起点とする Bio-Inspired 膜へのアプローチを紹介 した. 次に, Membranome を基盤とす る Bio-Inspired 膜へのアプローチを紹 介した.「人工膜」と「生体膜」の構 造・機能とその誘導原理について, 改 めて俯瞰的に見る事が重要となって きたと思われる.これまでそれぞれの 領域で大変精力的に研究が進められ てきたが,その一方で専門化され過 ぎ,両者が相互に発展するための共通 のプラットフォームが必要になって きた感が否めない.両者で長年蓄積さ れてきた知見を,中間的な「分子~分 子集合体」レベルにおいて,知の体系 化を図ることができれば、自然と共通 のプラットフォームが形成されるも のと考えられる.

![](_page_30_Figure_6.jpeg)

|  |  |  | キー | ・ワー | F |  |
|--|--|--|----|-----|---|--|
|--|--|--|----|-----|---|--|

bio-inspired membrane / artificial membrane / biomembrane / membranome / liposome / self-assembly

■ 所 属

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- 掲載先 ■
- 膜, vol. 37(6), 264-269 (2012)

査読:あり,言語:日本語

used.

(b)

relaxation

dipole

人工細胞膜上におけるアミロイド形成 Amyloid Fibril Formation on Artificial Biomembranes

島内寿徳<sup>1)</sup>, 北浦 奈知<sup>2)</sup>, 馬越 大<sup>3)</sup>, 久保井 亮一<sup>4)</sup>

#### ■ 概 要

人工細胞膜として、ポリ電解質の交互積層化層や脂質膜などが利用されてきた.水和による極性 の変化や界面近傍の水和部位周辺環境の運動性と荷電状態により、界面特性が異なることが明らか になった.この特性は、界面と Aβとの相互作用の違いとして現れ、結果として、αβ転移、一次核 形成や核同士の凝集などの誘導のされやすさと関連してくる事が示唆された. このような機構が老 人斑様の球状アミロイド形成の有力な要因であると考えられる.

![](_page_31_Figure_6.jpeg)

Fig. (a) Schematic illustration of a quartz crystal microbalance system combined with a liposome immobilization technique and a surface modification technique. (b) Frequency change of QCM electrodes after addition of Aβ (10 µM in IL-QCM and 7.5 µM in QCM with various modified surfaces). Data for OTS, PEI and PVS were taken from ref. 6. (c) Ladder of local hydrophobicity of various kinds of proteins, including monomeric AB, its fibrils and the seeds of fibrils.

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■キーワード■
```

bio-inspired membrane, artificial membrane, biomembrane, membranome, liposome, self-assembly

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#### 掲載先

表面科学, 33, 40-46 (2012)

査読:あり,言語:日本語

#### リポソーム固定化技術を用いたメンブレノミクス研究 Membranomics Research Using Liposome Immobilization Techniques

島内寿徳1)

#### ■ 概 要 ■

近年,脂質二重層からなる閉鎖系小胞(リポソーム)を生体膜モデルとして用いて,無細胞タンパク質合成系のコンパートメント(区画)化による人工細胞の研究が展開されている.また,リポソーム膜界面での無細胞タンパク質合成の制御,ペプチド断片の酵素様活性の発現,タンパク質の構造形成反応の促進効果など,従来のコンパートメント効果では十分に説明できない現象も報告されている.このような事例の蓄積から,生体膜,それを模倣した二分子膜分子集合体(ベシクル,リポソームなど),ならびに,複数膜から成るシステムは,(1)通常の条件では安定であるが,(2)潜在的な機能を保持し,(3)ストレス条件下においてダイナミックに潜在機能の発現を誘導する,と考えられる.筆者らのグループは,これを「メンブレノーム」と定義している.これらの機能の誘導には引き金があるはずである.それを明らかにする事が,上記の問題に応える事になるだろう.このような背景の下,筆者らは、リポソーム固定化技術を活用した相互作用解析ツールを開発し、メンブレノミクス研究を展開してきた.本稿ではその成果の一端を紹介している.

![](_page_32_Figure_6.jpeg)

(a) ILC によるタンパク質の溶出速度の遅れと疎水性変化の関係. (b) ILS により検出した拡散電流とタンパク 質の疎水性の関係. (c)IL-QCM によるペプチドの吸着過程のモニタリング. (d) ITO 上に構築したリポソーム アレイ.

| ■キーワード■  |       |        |
|--|-------|--------|
| Liposome immobilization, Membranome, Membrane chip |       |        |
| ■ 所属 ■   |       |        |
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|  | 杏読・あり | 言語・日本語 |

## Adsorption and desorption behavior of benzene on activated carbon from different precursors in dry and humid conditions

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#### ■Summary

Activated carbons (ACs) prepared by  $CO_2$  activation of five different precursors and commercial coconut shell AC are used for benzene adsorption (5 ppmv) from air at relative humidities (RHs) of 0%, 50 % and 70%. Benzene adsorption capacities of the ACs prepared in the laboratory are similar at RH 0%, but they differ significantly in the presence of moisture. The negative influence of moisture on benzene uptake is greater in a case of the ACs with large amounts of surface polar sites. The average micropore sizes of the ACs up to 0.8 nm are beneficial for benzene adsorption at RH 70%, while the average micropore sizes above 0.8 nm a beneficial for benzene adsorption at RH 50%.

![](_page_33_Figure_6.jpeg)

breakthrough curves: a) RH 0% and b) RH

#### ■Key word■

Activated Carbon, CO<sub>2</sub> Activation, Benzene Adsorption, Humidity, Temperature Programmed Desorption

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### 卒業論文

#### セラミックス材料学研究室(指導教員:難波徳郎・紅野安彦・崎田真一)

- 1. ホウ酸塩系ガラスの構造予測に関する研究
- 2. MgO-Al<sub>2</sub>O<sub>3</sub>系 Cr フリー耐火物への非酸化物添加効果
- 3. 湿式ボールミル法による蛍光体からの希土類元素の回収
- 4. ガラスの相分離を用いた Bi 系新規蛍光体の作製
- 5. 分子軌道計算による結晶およびガラス中の鉛の電子状態解析
- 6. X線および中性子回折を用いた重金属ホウ酸塩ガラスの構造解析

#### 無機機能材料化学研究室(指導教員:三宅通博・亀島欣一・西本俊介)

- 7. フェライト系可視光応答型光触媒の合成とその水質浄化特性
- 8. Ti2O3 のメカノ触媒特性
- 9. 異種金属ドープ Pr-Ni 系層状ペロブスカイトの SOFC カソード電極特性
- 10. 廃 Ni/BaTi1-xSnxO3 触媒を用いたバイオガスリフォーミング
- 11. PDDA/C 複合体を用いた新規触媒の作製
- 12. ハイドロタルサイトへのフタル酸のインターカレーションとデインターカレーション
- 13. La と Sm を共ドープした CeO2 の合成と SOFC 用電解質性能の評価
- 14. 環境水域のCs+, Sr2+新規除去材料の開発(2012年9月卒業)

#### 有機機能材料学研究室(指導教員:高口豊·田嶋智之)

- 15. 2.9 位もしくは 2.10 位に置換基を持つヘキサチアペンタセンの合成
- 16. フラーレンへのボリル基導入反応の開発
- 17. 単層カーボンナノチューブ/フラロデンドロン/SnO2 融合マテリアルの合成
- 18. 単層カーボンナノチューブ/フラロデンドロン超分子複合体の光触媒能向上
- 19. ジセレナナフタレンの合成とカドミウム硫黄クラスター分子との反応
- 20. チオール修飾グラフェンナノリボンの合成と性質
- 21. グラフェンナノリボンを用いたセシウムイオンの回収(2012年9月卒業)

#### 環境高分子材料学研究室(指導教員:木村邦生·山崎慎一)

- 22. 重合相変化を利用したポリアミドイミドの定序性重縮合法の開発
- 23. トポロジーの異なる高分子ブレンドの流動誘起結晶化から探る高分子結晶化にお ける結び目絡み合いの役割
- 24. ホウ酸触媒重縮合を用いた非等モル条件下での芳香族ポリエステルの調製
- 25. ポリエチレンの再資源化を目指した長鎖セグメント化ナイロンの調製
- 26. 分子量が大きく異なるポリエチレンブレンド系における相分離構造

#### (2012年3月および9月認定の卒業論文)

#### 環境プロセス工学研究室(指導教員:木村幸敬・小野努)

- 27. マイクロ流路内乳化と溶媒拡散法を利用したエマルション微細化技術の構築
- 28. デキストラン誘導体を用いた金属イオン架橋型ヒドロゲルの調製
- 29. マイクロ湿式紡糸プロセスで調製される高分子ファイバーの形状に与える
- 30. 溶媒の影響
- 31. マイクロ流路内でのソープフリー重合による高分子微粒子連続調製法の開発
- 32. 亜臨界水処理による木質バイオマスの主要成分の改変と残渣の特性評価
- 33. 高分子界面活性剤被覆酵素の調製及びイオン液体反応系における活性評価

#### 環境反応工学研究室(指導教員:加藤嘉英・アズハウッディン)

- 34. Fischer Tropsch 合成用コバルト系触媒の開発
- 35. 農業系廃棄物からのバイオチャーの特性に及ぼす調整条件の影響
- 36. 凝固精製によるシリコンスラッジの太陽電池用原料への再生
- 37. 石炭ガス化ガス中の水銀除去剤の開発
- 38. 冷却ガス吹き込みによるノズル先端周囲の冷却効果
- 39. ケミカルエッチングによる太陽電池セル有用元素のリサイクルプロセスの開発
- 40. RH 真空脱ガス装置における混合特性及び反応速度向上方法
- 41. 過熱水蒸気による RDF の脱塩素化プロセスの開発(2012年9月卒業)

#### (2012年3月および9月認定の卒業論文)

### 修士論文

#### セラミックス材料学研究室(指導教員:難波徳郎・紅野安彦・崎田真一)

- 1. ホウケイ酸塩ガラスの相分離におけるリンの分配挙動に及ぼす添加成分の影響
- 2. 鉄鋼スラグからのリンの回収プロセスの開発
- 3. 配向したフレスノイト結晶化ガラスの微細構造観察に基づく結晶成長メカニズム
- 4. SnO-P<sub>2</sub>O<sub>5</sub>系鉛代替ガラスへの Nb2O5 添加効果
- 5. ハロゲン含有ビスマスホウ酸塩ガラスの構造解析
- ガラスの構造情報のデータベース化および重回帰分析を利用したガラス構造の予 測(2012年9月修了)

#### 無機機能材料化学研究室(指導教員:三宅通博・亀島欣一・西本俊介)

- 7. 水熱法を用いた超撥水酸化チタン界面の作製
- 8. Ca-Nb 系層状ペロブスカイトと色素との複合化による可視光応答型光触媒の開発 とその評価
- 9. 金属塩層状複水酸化物複合体のエタノール改質触媒性能
- 10. 層状ペロブスカイト化合物 Pr2NiO4 への遷移金属ドープによるカソード電極特性 評価
- 11. 酸化チタン/モンモリロナイト複合体の作製と有機物分解特性の評価
- 12. 可視光照射下における WO3 光触媒とオゾンを用いた水処理

#### 有機機能材料学研究室(指導教員:高口豊·田嶋智之)

- 13. (Me4N)4[Cd10S4(SePh)16]の合成とX線結晶構造解析
- 14. Cd10S5Se11 クラスターをコアに持つ新規デンドリマーの合成と性質
- 2,3,9,10-位にデンドリマー型置換基を持つペンタセン誘導体とフラーレン類との Diels-Alder 反応
- 16. エッジに化学修飾を施したグラフェンナノリボンの合成と性質
- 17. C60 の 1,4-付加体からなるフラロデンドロンの合成と光物性
- 18. 高歪みペンタセンを指向した新規 6,13-ジヒドロペンタセン誘導体の合成と性質

#### 環境高分子材料学研究室(指導教員:木村邦生·山崎慎一)

- 19. 高分子の結晶化に及ぼす末端パーフルオロアルキル基の影響
- 20. 環状高分子の結晶化から探る高分子の結晶成長に及ぼすトポロジカルな拘束と結び目絡み合いの効果
- 21. 重合相変化を利用したポリイミダゾピロロンの調製と高次構造制御
- 22. エステル交換反応を介した絡み合い解消がポリ乳酸の流動誘起結晶化に及ぼす影響
- 23. 重合相変化を利用したポリアミドのケモセレクティブ重縮合と中空微粒子調製への展開

(2012年3月および9月認定の修士論文)

#### 環境プロセス工学研究室(指導教員:木村幸敬・小野努)

- 24. 界面活性 TEMPO 誘導体を用いた制御ラジカル重合によるポリスチレンカプセル 調製
- 25. ポリコハク酸イミド誘導体を用いた新規生分解性マイクロカプセル調製法の開発
- 26. 二重管型マイクロノズルを用いた湿式ナノファイバー調製法の開発
- 27. 加圧減圧法を利用した中空マイクロカプセル調製プロセスの制御因子に関する検 討
- 28. 亜臨界水乳化法と Layer-by-Layer 法によるナノカプセルの調製
- 29. 制御ラジカル重合を用いた高分子界面活性剤の合成と不均一系重合
- 30. ポリアスパラギン酸誘導体を用いた複合コアセルベーション法の開発
- 31. ポリスチレン付着ペプチドを利用した微粒子表面への生物機能付与技術の開発
- 32. Preparation of biodegradable foam with walnut shells pretreated by subcritical water(亜臨 界水で前処理した山核桃殻を含む生分解性発泡体の調製)

#### 環境反応工学研究室(指導教員:加藤嘉英・アズハウッディン)

- 33. NH3 を用いた CO2 メタネーション触媒の基礎検討
- 34. 石炭燃焼排ガス中の水銀を対象とした硫化鉄・活性炭系水銀除去剤の開発
- 35. 揮発性有機化合物の完全酸化に適した酸化銅系触媒の開発