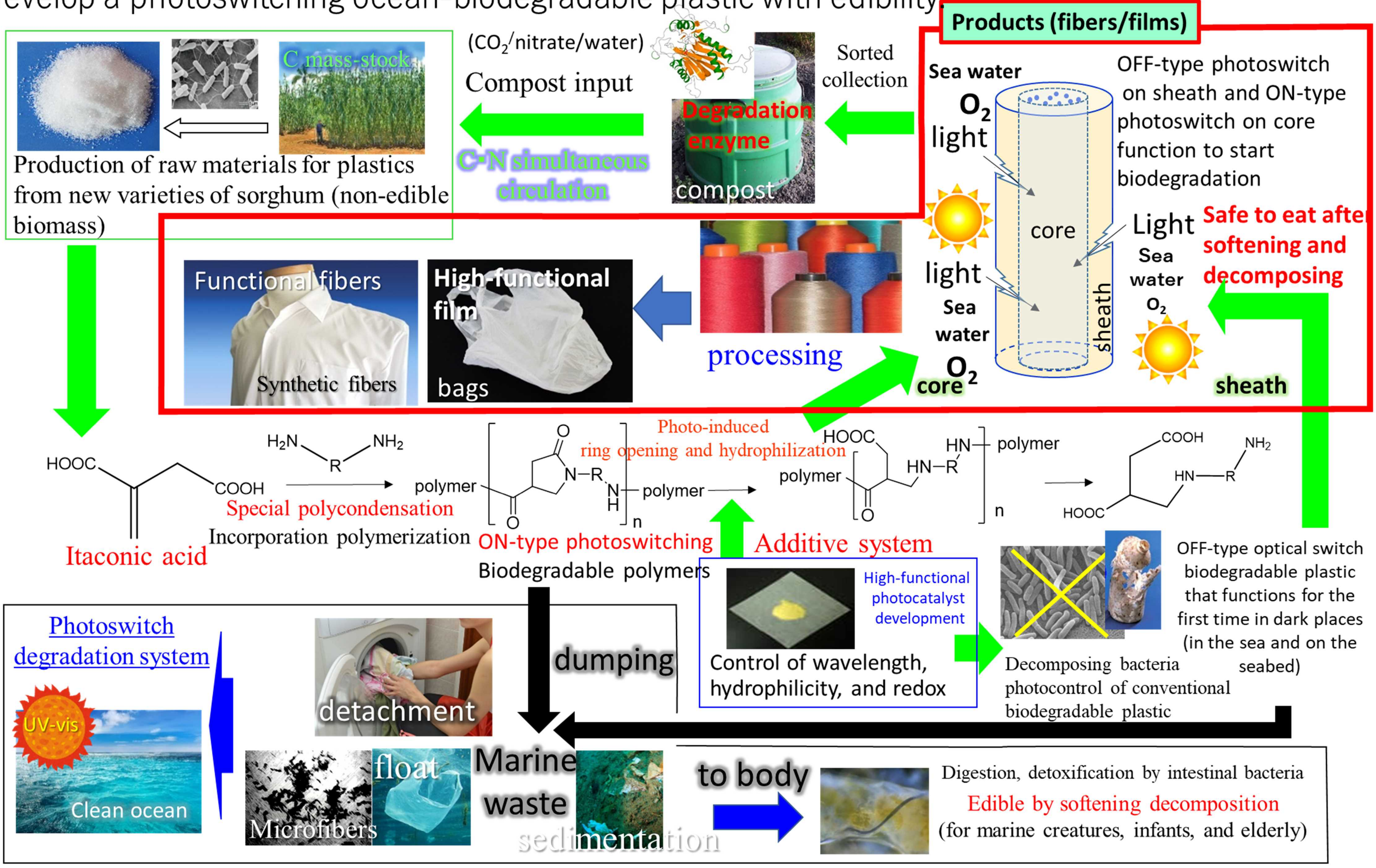
番号： A-17-1E

PJ： Development of Photo-switching Ocean-degradable Plastics with Edibility

Organization: Japan Advanced Institute of Science and Technology, Kobe University, Nagoya University, Kagoshima University, Tokyo University of Science, Tokyo University of Agriculture and Technology, National Institute of Advanced Industrial Science and Technology (AIST) ,Osaka Research Institute of Industrial Science and Technology (ORIST).

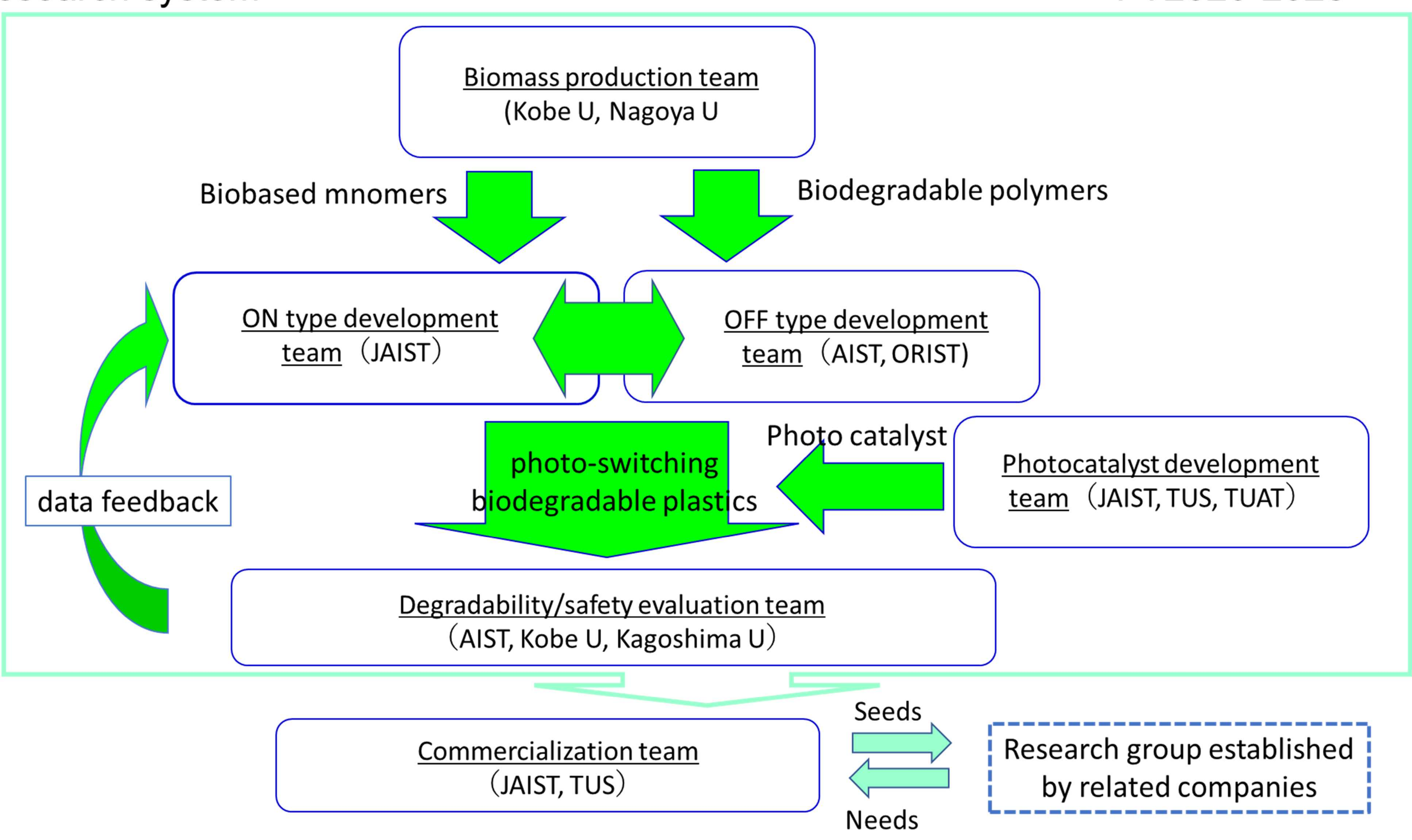
Contact: Atsuyoshi Nakayama（a.nakayama@aist.go.jp）(PM)

【Final goal】Using itaconic acid produced from a new cultivar of sorghum and a biodegradable polymer, a newly developed high-performance photocatalyst is composited to develop a photoswitching ocean-biodegradable plastic with edibility.



Research system

FY2020-2023



No. A-17-2E

PJ : Development of Photo-Switching Ocean-Degradable Plastics With Edibility

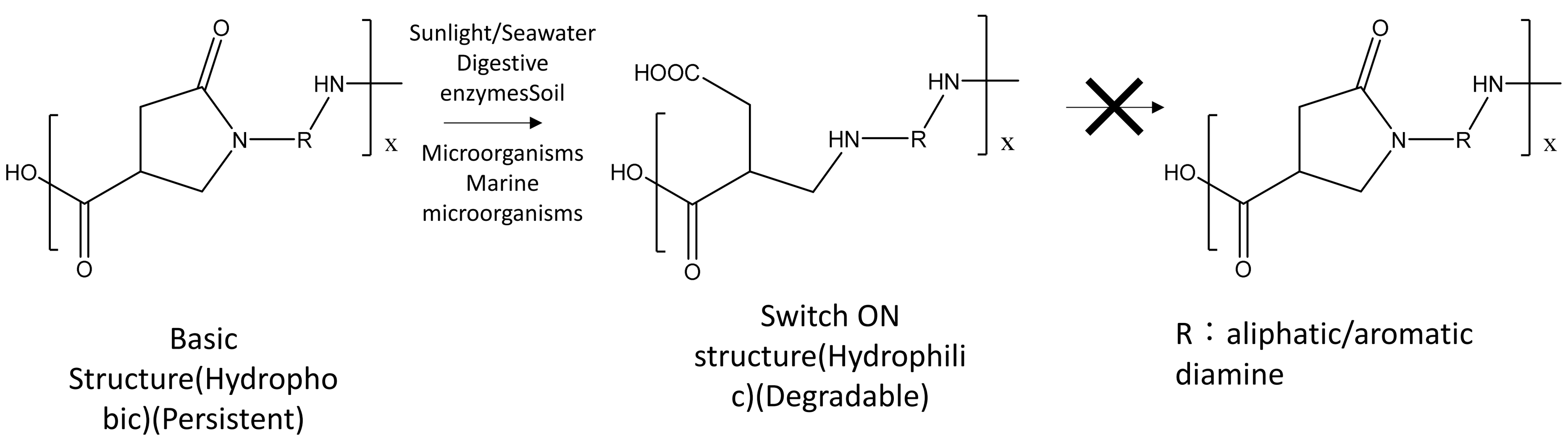
Theme: Development of Photo-Switching Degradable Plastics

Organization: Japan Advanced Institute of Science and Technology

Contact: Dr. K. Takada (takada@jaist.ac.jp) / Prof. M. Yamaguchi (m_yama@jaist.ac.jp) / Prof. T. Taniike(taniike@jaist.ac.jp)

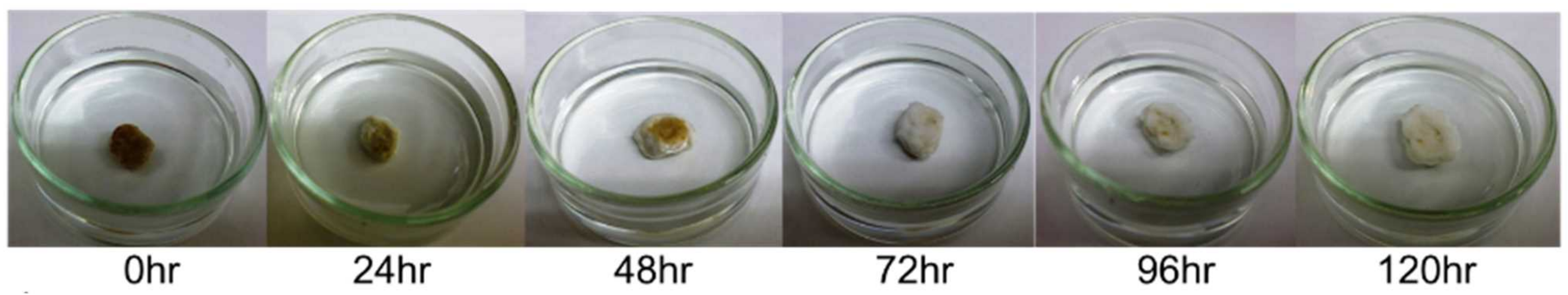
1. Synthesis of Polyamide

Functions of ON-type Bio-Nylon

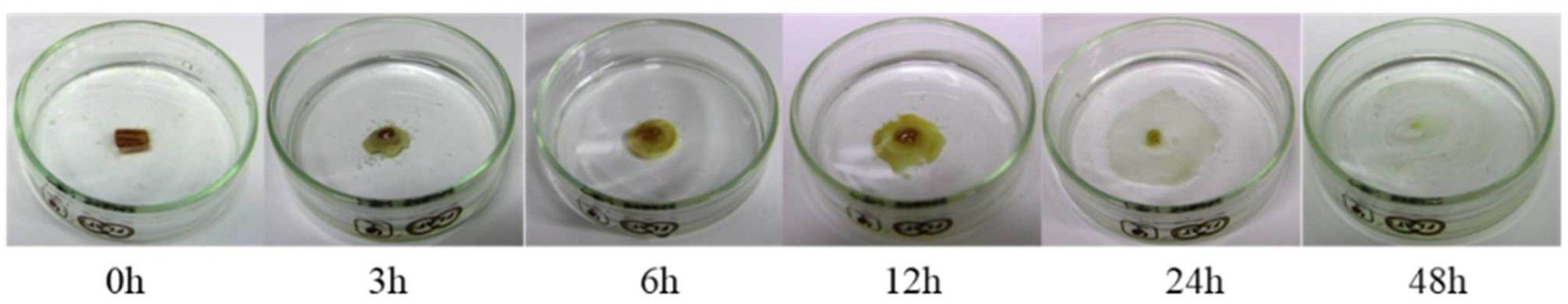


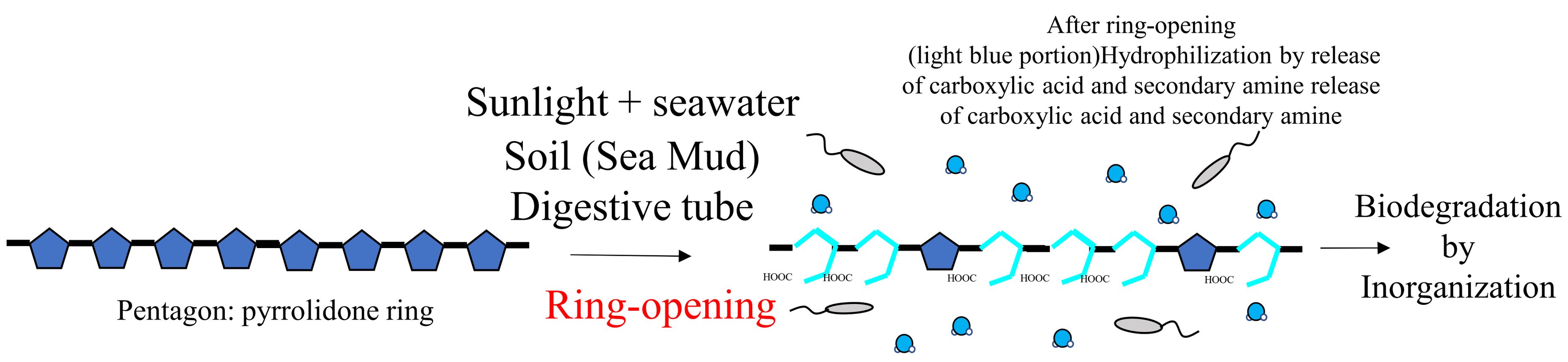
Disintegration by stimulation of light and water (carbonyl excitation, reactive oxygen species, OH, etc.)

R: nonamethylenediamine



R: *m*-xylenediaminediamine

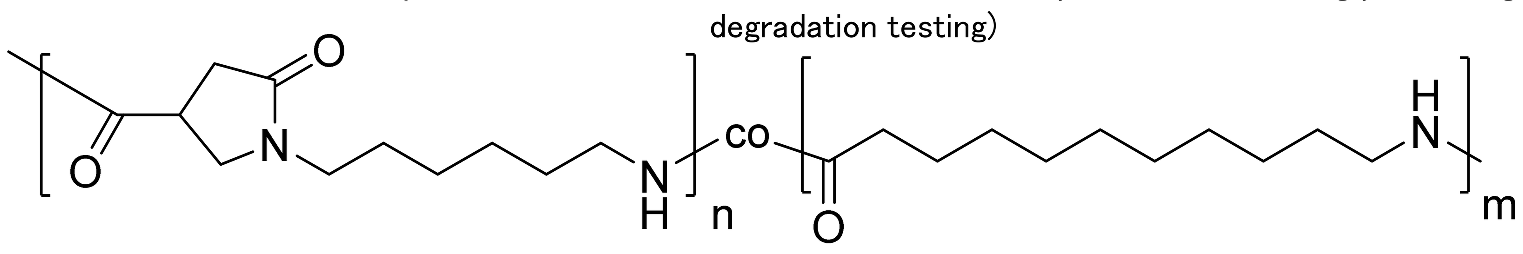




If the ring-opening ratio exceeds a certain value, it becomes hydrophilic and collapses at once.Water approaches the molecule and the bacteria accesses it.

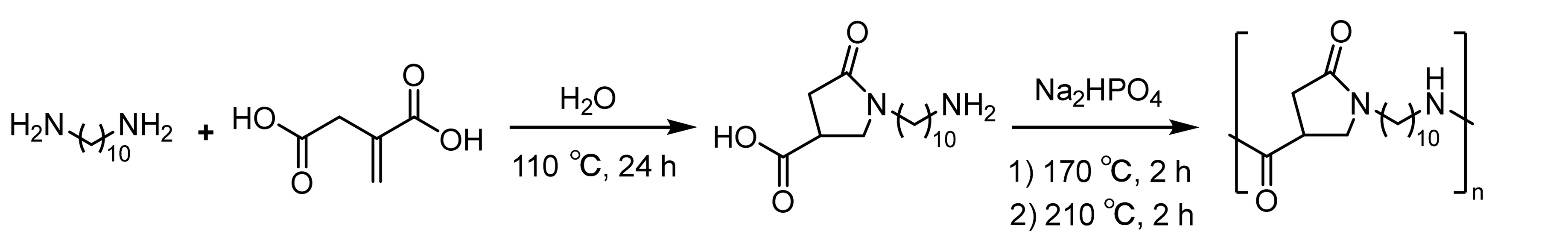
✓ Found conditions under which 11-aminoundecanoic acid can be introduced and polymerized

(10L bench scale synthesis conditions were also established → provided for molding processing and degradation testing)



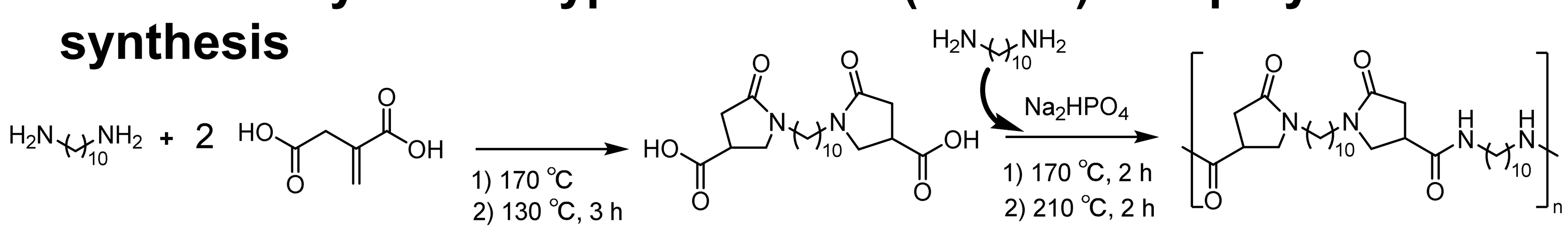
Pyrrolidone rings incorporated into other types of polymers to give them degradability

1. Amino acid type monomer (10i-1) and polyamide synthesis



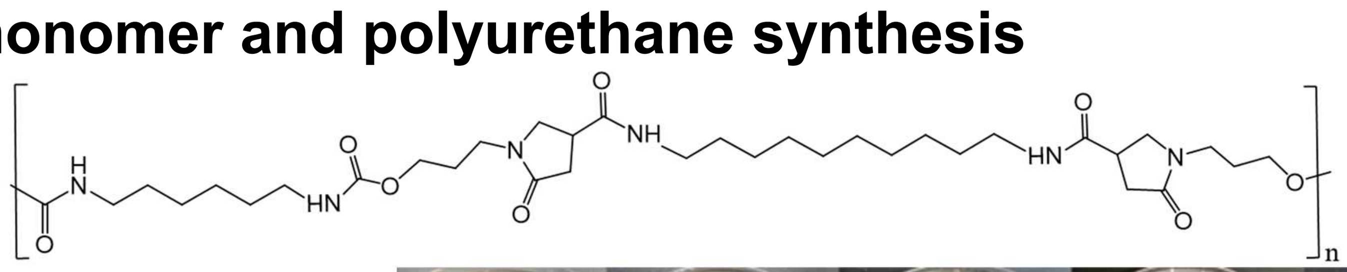
Scheme 1. Synthesis of 1-(10-aminodecyl)-5-oxopyrrolidine-3-carboxylic acid (10i-1) and polyamide10i-m0 (PA10i-m0).

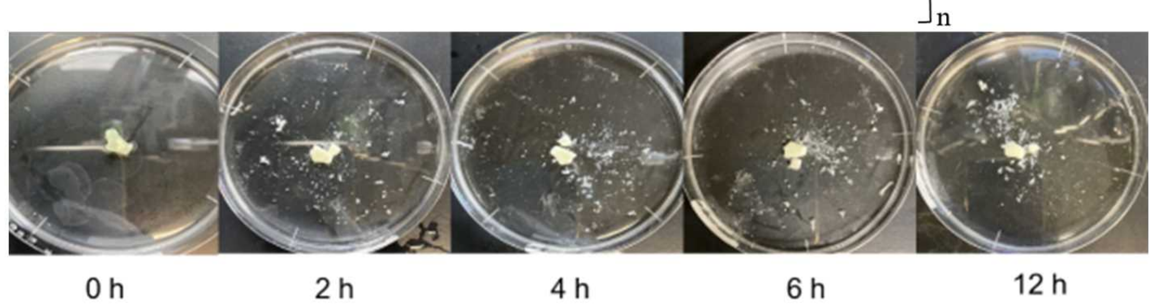
2. Dicarboxylic acid type monomer (10i-1.5) and polyamide synthesis



Scheme 2. Synthesis of 1,1'-(decane-1,10-diyl)bis(5-oxopyrrolidine-3-carboxylic acid) (10i-1.5) and PA10i-m100.

3. Diol monomer and polyurethane synthesis

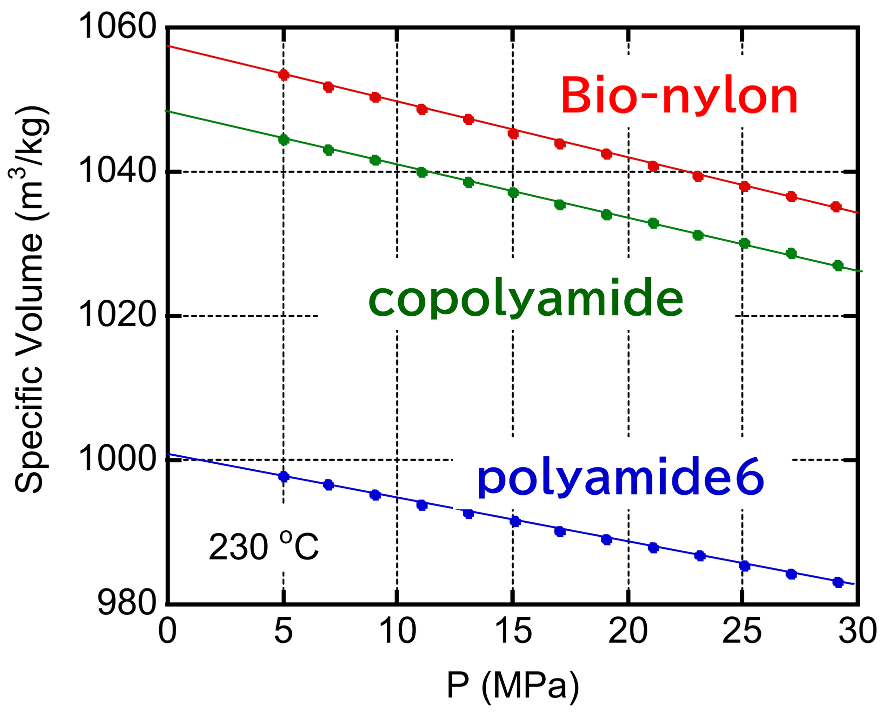




2. Processability

Nylon 6i-11-50% (Bio-nylon)

Pressure-Volume relation



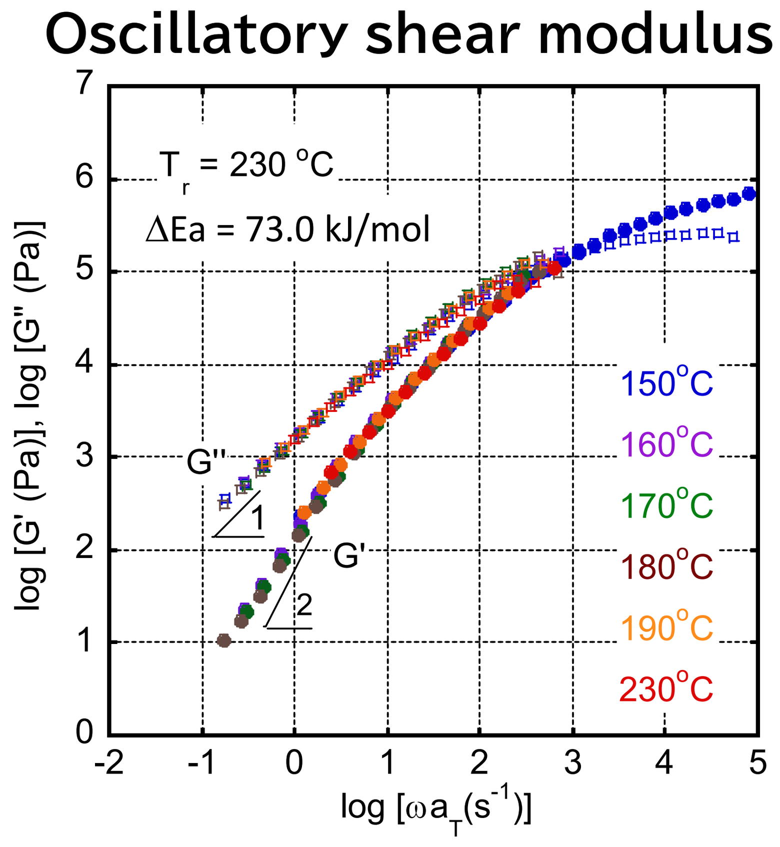
Bio-nylon
Melt density 946 kg/m^3
Bulk modulus 1.40 GPa

copolyamide＊
Melt density 954 kg/m^3
Bulk modulus 1.47 GPa

PA6
Melt density 999 kg/m^3
Bulk modulus 1.66 GPa

＊copolyamide	PA6	55 mol
	PA66	13 mol
	PA610	32 mol

Oscillatory shear modulus



Viscoelastic properties of bio-nylon are similar to those of conventional nylons.

Entanglement molecular weight

M_e = 2700　PA6 M_e = 2490
PA66 M_e = 2000

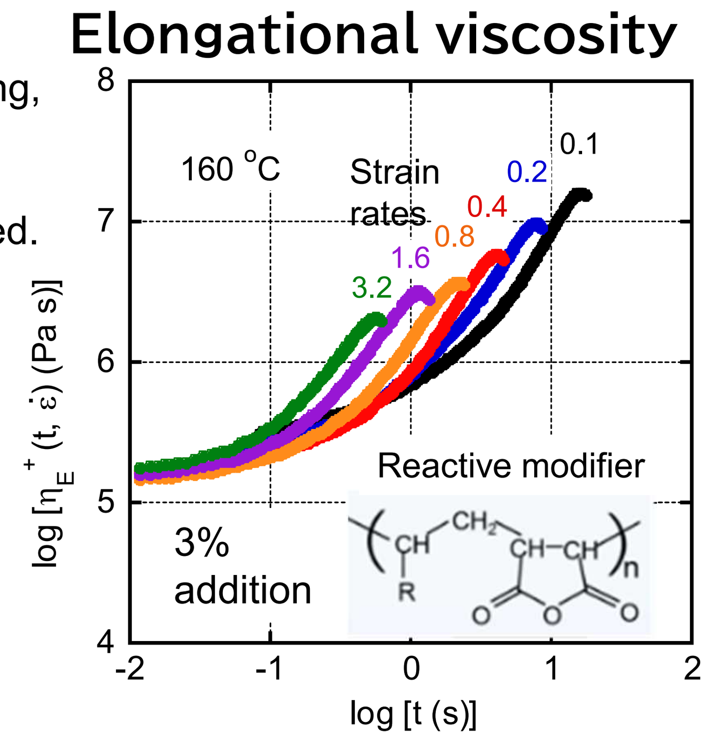
Processability

Spinning
・Melt spinning is available (diameter 15-50 μm)
・It is possible to obtain a sheath-core fiber.

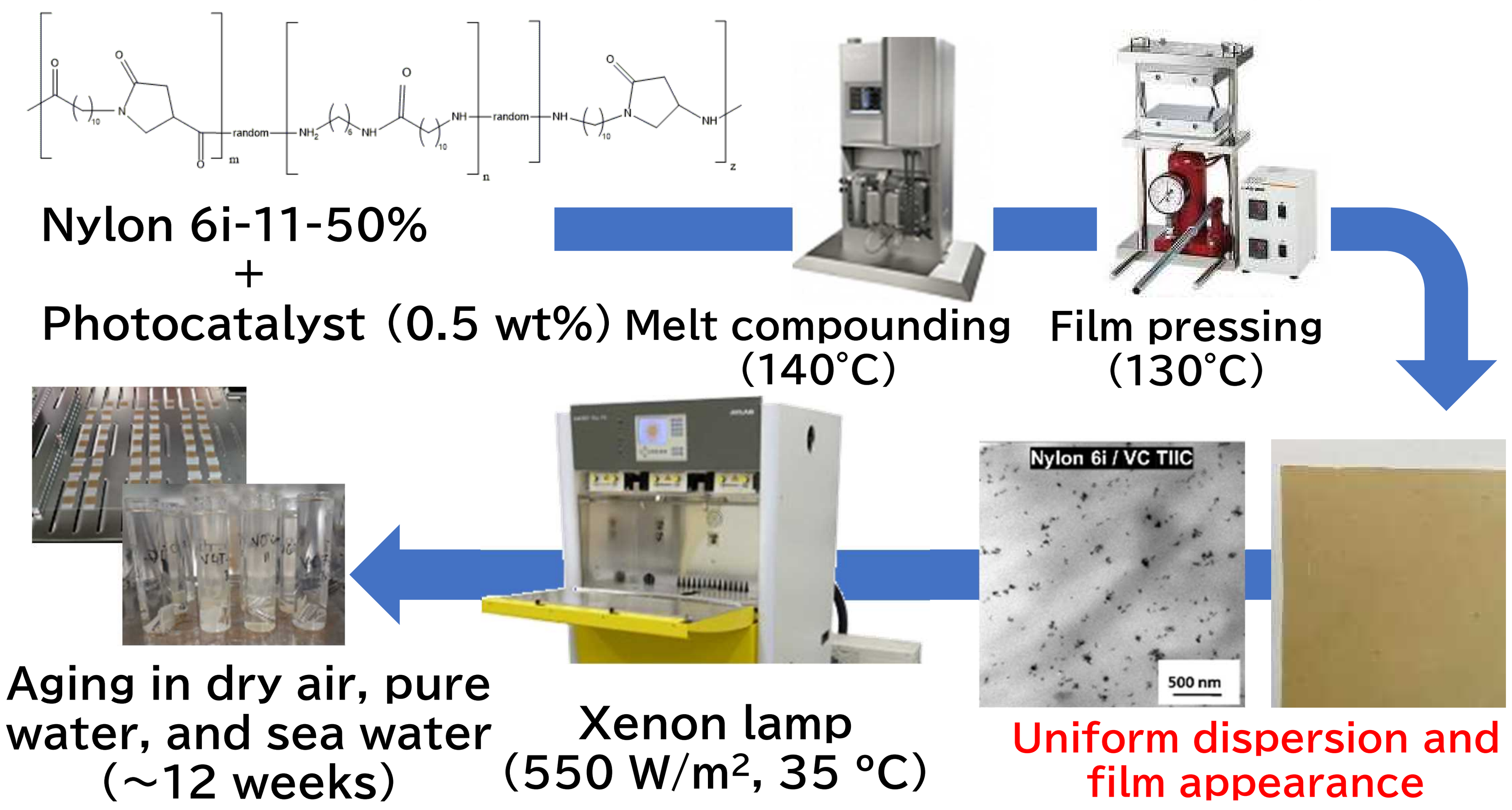
Film, foaming, and blow-molding
・A small addition of reactive modifier provides strain-hardening in elongational viscosity, leading to good processability.

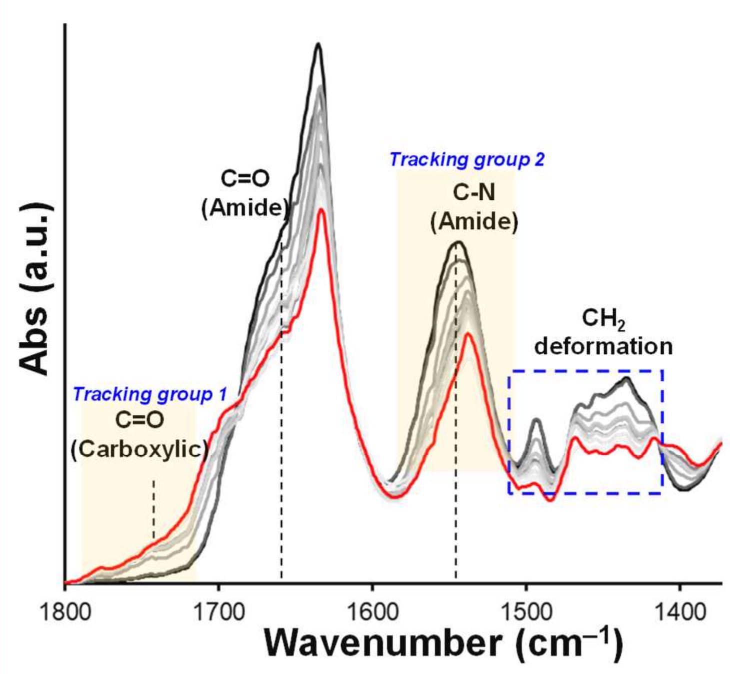
Elongational viscosity

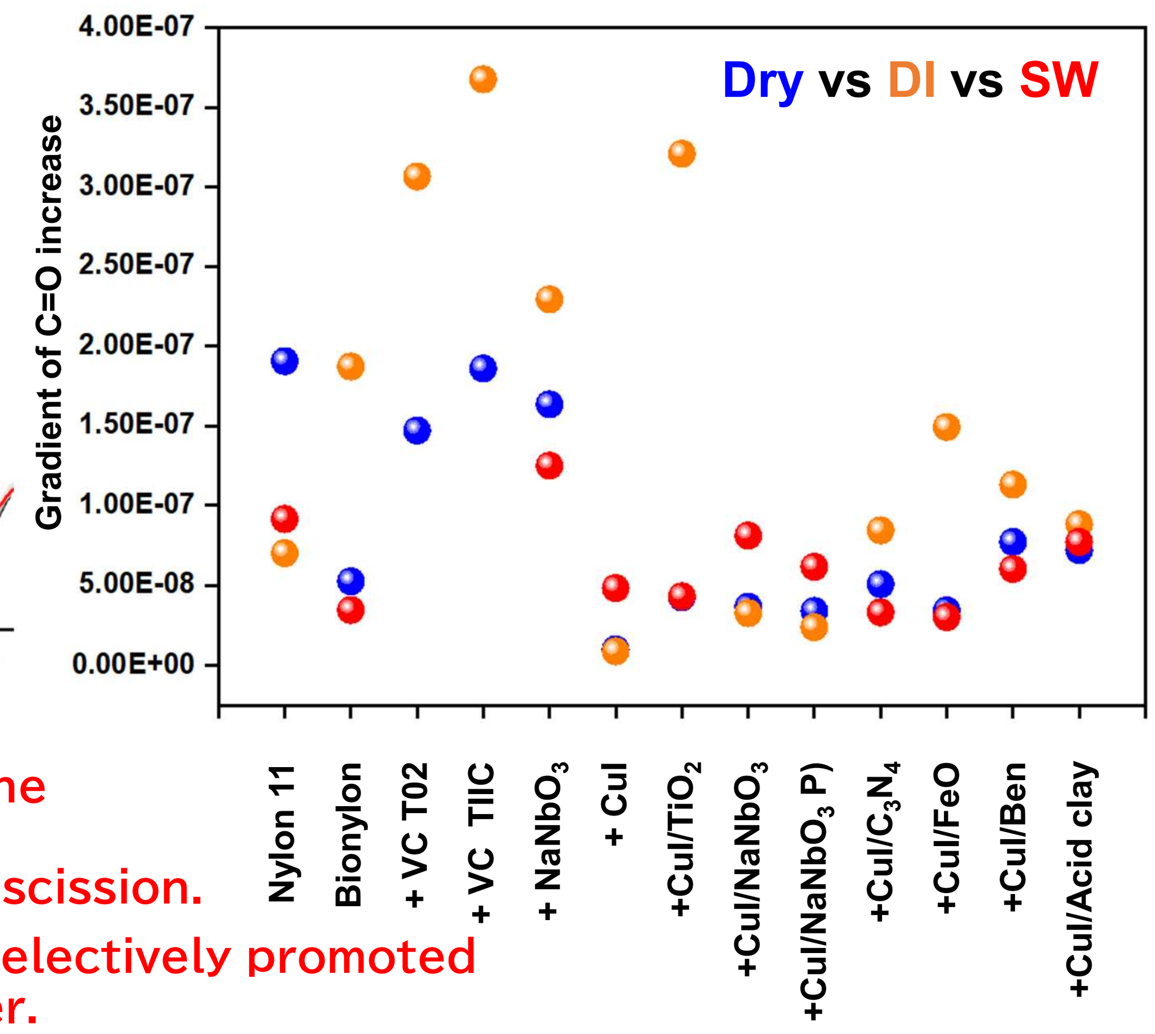
Strain hardening, i.e., viscosity increase with time, is detected.



3. Photodegradation







- Bio-nylon degraded via the oxidation of CH_2 next to pyrrolidone N and amide scission.
- The pyrrolidone groups selectively promoted degradation in pure water.
- The TiO_2 addition accelerated the photodegradation by 2-3 times.
- The addition of CuI largely suppressed the degradation, while the degradability in water could be recovered by TiO_2.

【Challenges】Further acceleration, particularly in saline water

No. A-17-3E
PJ : Development of Photo-switching Ocean-degradable Plastics with Edibility
Theme: Biodegradation and Safety Assessment

Organization: Kobe University

Contact: C. Ogino (ochiaki@port.kobe-u.ac.jp), H. Okamura (okamurah@maritime.kobe-u.ac.jp), T. Shintani (shintani@person.kobe-u.ac.jp)

Marine degradability of photo-switched biodegradable plastics

1. Biodegradability of ON type sample (Nylon6i11(33), <2 mm) in water, irradiated with ultraviolet lamp, was evaluated in natural seawater using a BOD test (Joint research with AIST).

- The absorbance of water increased depending on the light irradiation time, and it seemed organic matter eluted from the plastic (Fig. 1).
- No change was observed in the infrared spectra of the sample surface exposed to UV light in water for 8 hours.
- Approximately 4% of the irradiated plastic samples in natural seawater was biodegraded after one month, but no significant difference was observed in the biodegradation rate between samples with/without UV irradiation (Table 1).

2. OFF-type samples (PHBH, PBSA, PCL, and CA-L) showed disintegration of more than 50% after 2 months in field seawater, and PCL in particular had about 90% disintegration within 1 month (Fig. 2, Joint research with AIST and ORIST).

3. Other test plastics were immersed in field seawater to evaluate their disintegration properties (Joint research with AIST and ORIST).

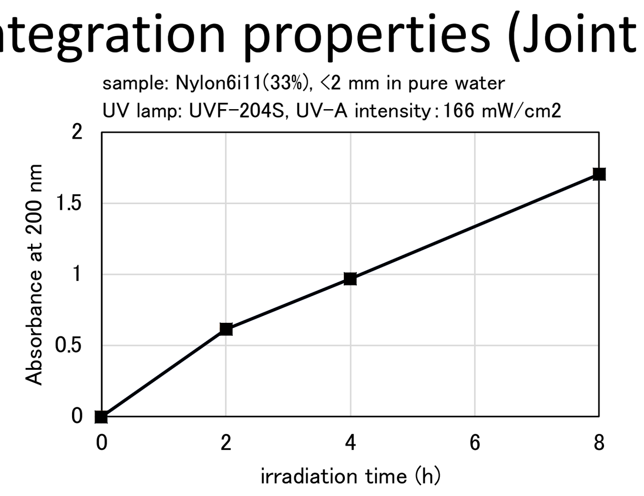


Fig.1 Change in absorbance with irradiation time

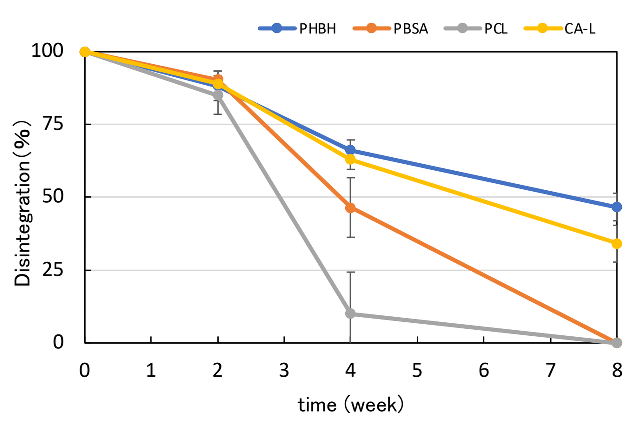


Fig.2 Disintegration of test plastics in natural seawater at ship mooring pond of Kobe Univ.

Table 1 Biodagradation of pre-irradiated plastic in natural seawater*

Test specimen	UV lamp	UV-A Intensity (mW/cm^2)	irradiation time (h)	Biodegradation rate (%) after 1 month
Cellurose	no	0	0	67 ± 3.1
Nylon6i11(33), <2 mm	no	0	0	0.93 ± 1.4
Nylon6i11(33), <2 mm	HL400BH	50	8	3.9 ± 3.0
Nylon6i11(33), <2 mm	UVF-204S	166	8	4.0 ± 3.0

*BOD test with NP strengthned seawater using OxiTop (2023.11.27-12.27)

Environmental risk of plastic decomposition products

1. The estimated no-effect concentration (PNEC) of water-soluble degradation products derived from ON-type resins was calculated for marine and freshwater organisms (Table 2).

- Closed ring dicarboxylic acid type 1.5mer: 370 μg/l
- Closed ring amino acid monomer: 3,800 μg/l
- Open ring amino acid monomer: 4,400 μg/l

The degradation products are considered to be ecotoxic if they remain in the aquatic environment at concentrations exceeding the above PNEC.

2. No acute toxicity of ON type samples (particulate Nylon6i11(50), Nylon6i11(50)+$NaNbO_3$) to freshwater crustaceans (*Daphnia magna*) and freshwater fish (zebrafish) was observed (Fig. 3). However, some *D.magna* died due to particles adhering to their bodies.

3. No acute toxicity of OFF type samples (particulate PCL, PCL+P25, TiO_2, gC_3N_4, heat-treated gC_3N_4) to *Daphnia magna* and zebrafish was observed.

Table 2 Acute toxicity of degradation products from bionylon on aquatic species (EC_{50}, LC_{50} in ∝g/l, initial pH adjusted)

test organisms	Closed ring: Dicarboxilic type 1.5 dimer	Closed ring: Amino acid type monomer*	Open ring: amino acid type monomer*
Marine luminescent bacteria	> 1,000	>10,000	>10,000
Marine microalgae	> 1,000	7,200	7,100
Brine shrimp	> 1,000	>10,000	>10,000
Marine rotifer	> 1,000	>10,000	>10,000
Freshwater microalgae	> 1,000	3,800	4,400
Freshwater crustacean	820	>10,000	7,600
Freshwater rotifer	370	>10,000	6,300

*including salt

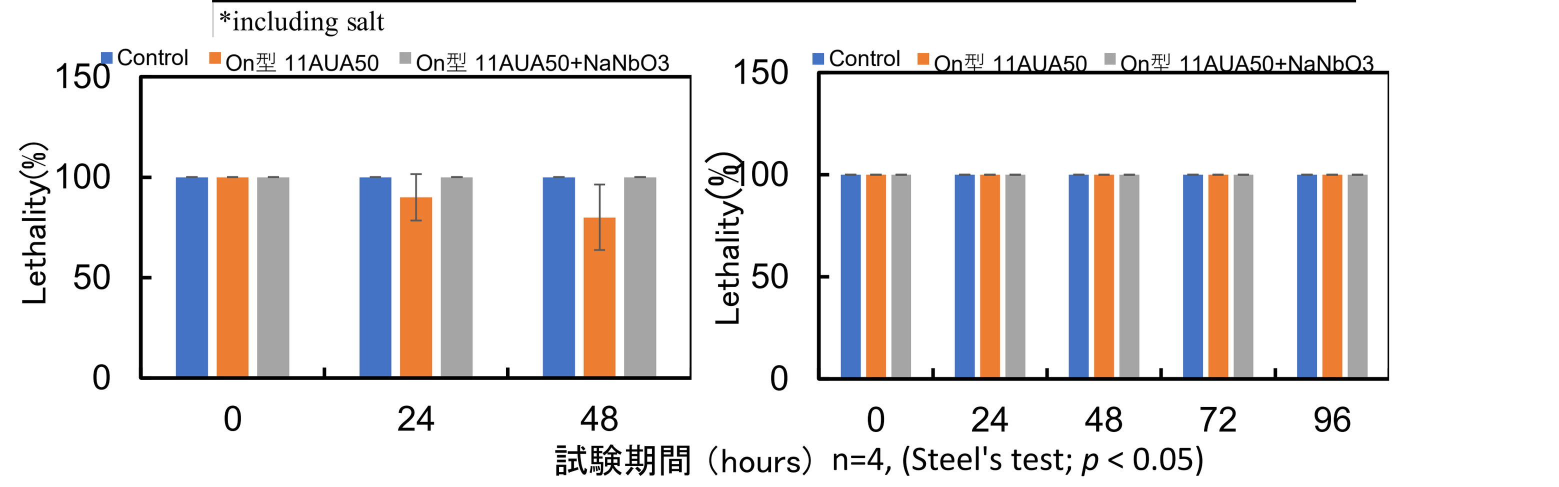
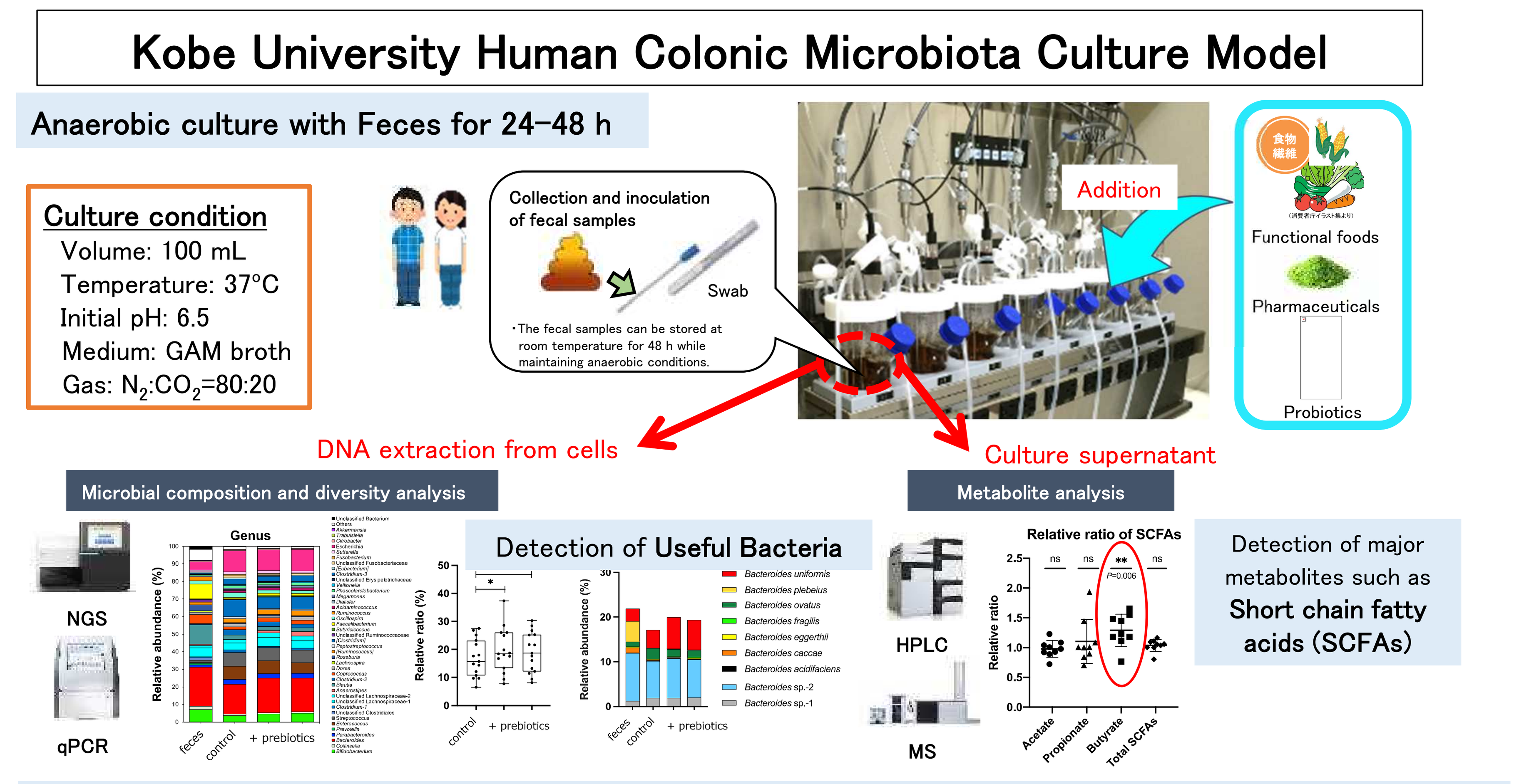


Fig. 3 Acute toxicity of ON-type particulate plastics to *Daphnia magna* (left) and zebrafish (right)

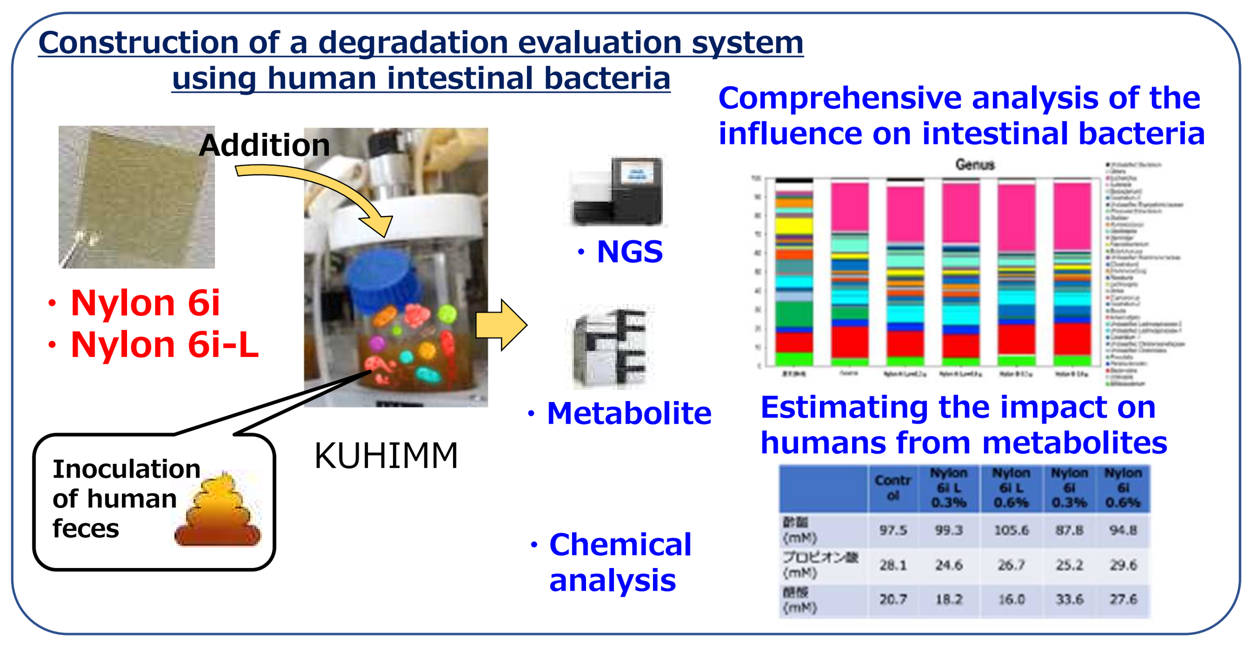
Degradability and safety evaluation in simulated intestinal environment

1. We have previously developed the "Kobe University Human Colonic Microflora Culture Model" (KUHIMM), which can successfully reproduce and culture the human colon microflora.



Evaluate the **effects of functional ingredients, drugs, and probiotics** easily.

2. We used KUHIMM to study the effect of Nylon6i addition on the human colon microbiota.



By adding Nylon 6i, -L

- No major impact on intestinal flora and its diversity
- Metabolites compared to non-added There is no major change in concentration

→ The effect of human intake would be small.

【Chemical analysis】

A) Results of total carbon content (TOC) analysis of culture supernatant without bacteria

	Before (mg/L)	After (mg/L)	Differences
Control (非添加)	16,000	13,500	2,500
Nylon-6i-L (0.3%添加)	16,500	15,000	1,500
Nylon-6i-L (0.6%添加)	19,500	17,000	2,500
Nylon-6i (0.3%添加)	15,500	12,500	3,000
Nylon-6i (0.6%添加)	16,500	14,179	2,321

Carbon before and after cultivation would be constant

→ Possibility of microbial utilization of dissolved components is low

B) Suspended solids (SS) analysis (→ amount of Nylon decomposed not dissolved)

Bacterial cells after culture + Nylon 6i Dry Solid − Dry bacterial weight (control) ≒ SS (Amount of decomposition)

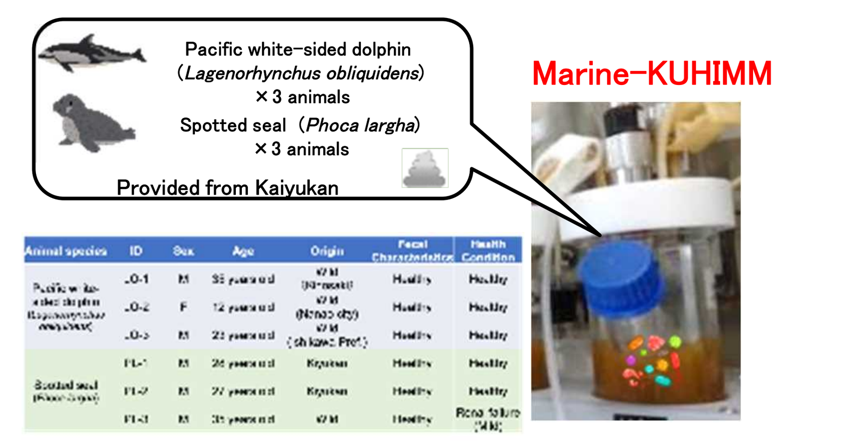
Although the initial concentration was 6.0 g/L, the residual SS was 10.0 g/L.

→ The possibility of weight loss due to decomposition is low. It is necessary to reconsider the analysis method in the future.

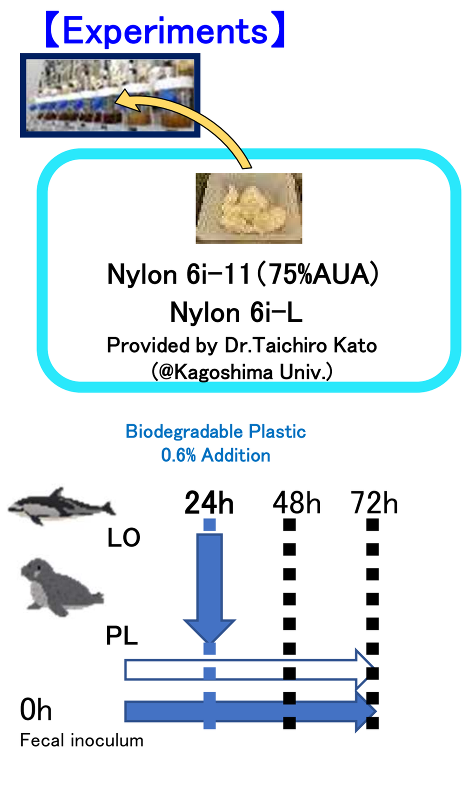
There is little interaction between human intestinal bacteria and Nylon 6i/Nylon 6i-L.

3. We constructed a model of marine mammals (Marine-KUHIMM) and tested the biodegradable plastic with a Marine-KUHIMM.

- Based on the conditions of KUHIMM, culture conditions (preparation of marine mammal feces, inoculum volume, reducing agent, medium composition, etc.) were examined and Marine-KUHIMM was being developed.
- The following condition was used to conduct the Nylon 6i-11 addition test.

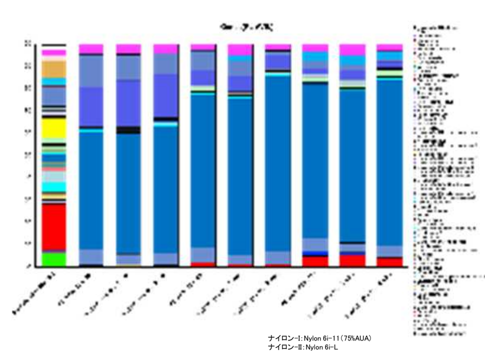


【Experiments】



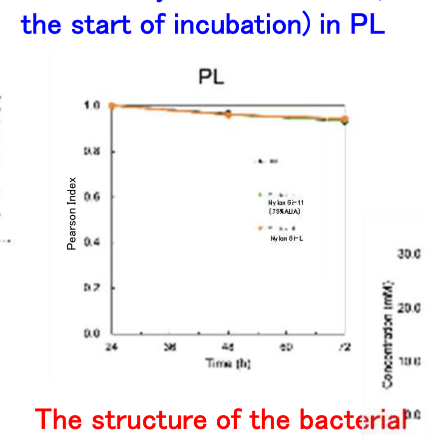
【Results】

Change in the structure of the bacterial flora in PL



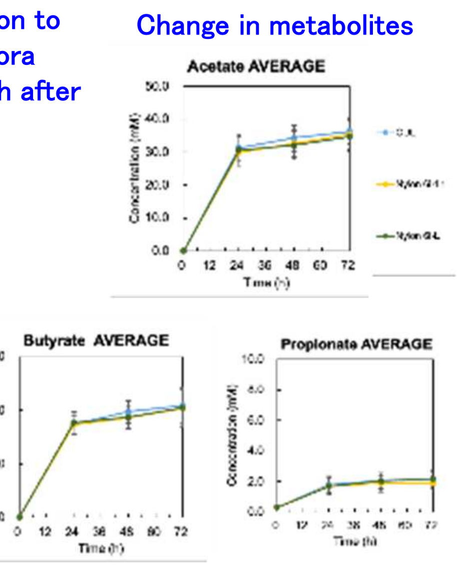
In the LO culture, reproducibility appeared to be low at 24 h, with an increase in E. coli genus (data not shown), while reproducibility of the PL flora was observed to be relatively high (see figure above).
Also, some bacterial symbioses, which are characteristically detected in marine mammals, were observed.

Change in similarity after addition to the structure of the bacterial flora immediately before addition (24h after the start of incubation) in PL



The structure of the bacterial flora with the addition of Nylon was very similar to that without Nylon.
Nylon addition had little effect on the structure of the bacterial flora.

Change in metabolites



No difference was observed in the concentration of short-chain fatty acids, the major metabolite (Welch's t-test (vs CUL)).

No. A-17-4E

PJ : Development of Photo-switching Ocean-degradable Plastics with Edibility

Theme: Enzymatic Degradation and Recycling Approach for iNylon

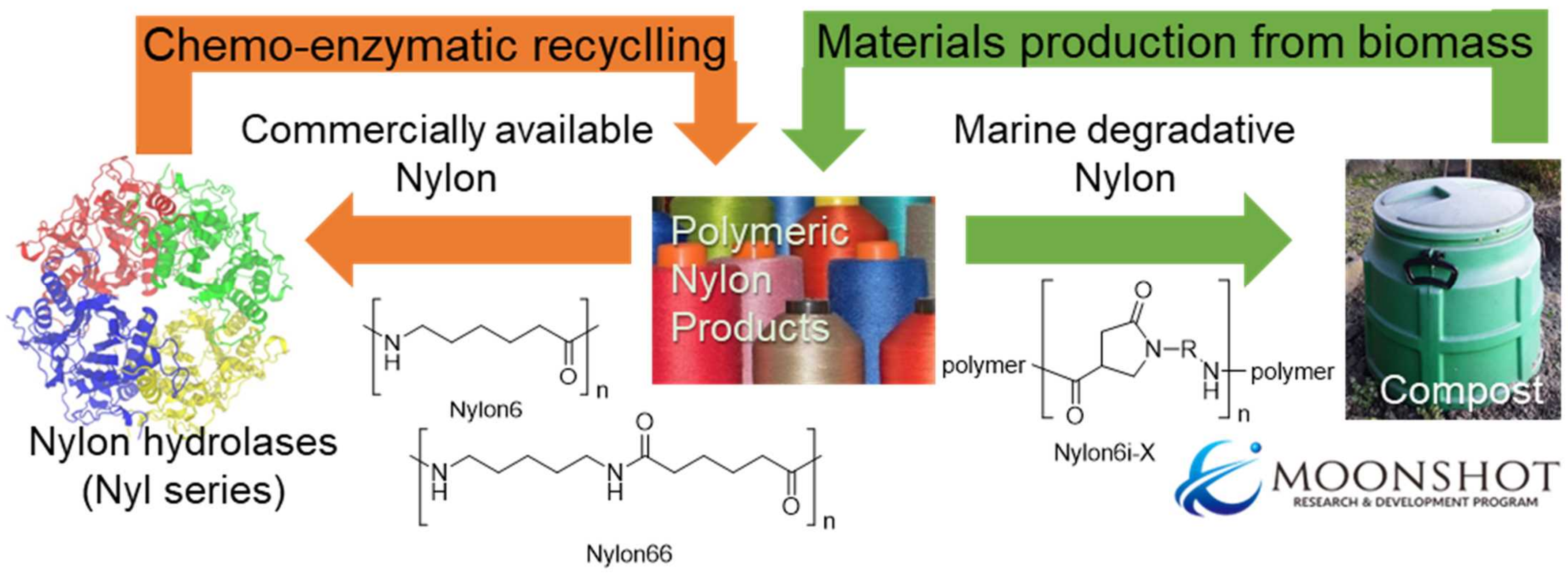
Organization: Kagoshima University

Contact: Dai-ichiro Kato (k0035454@kadai.jp)

Initiatives in Kagoshima University : Clarify the biodegradation pathway of iNylon in the ocean environment.

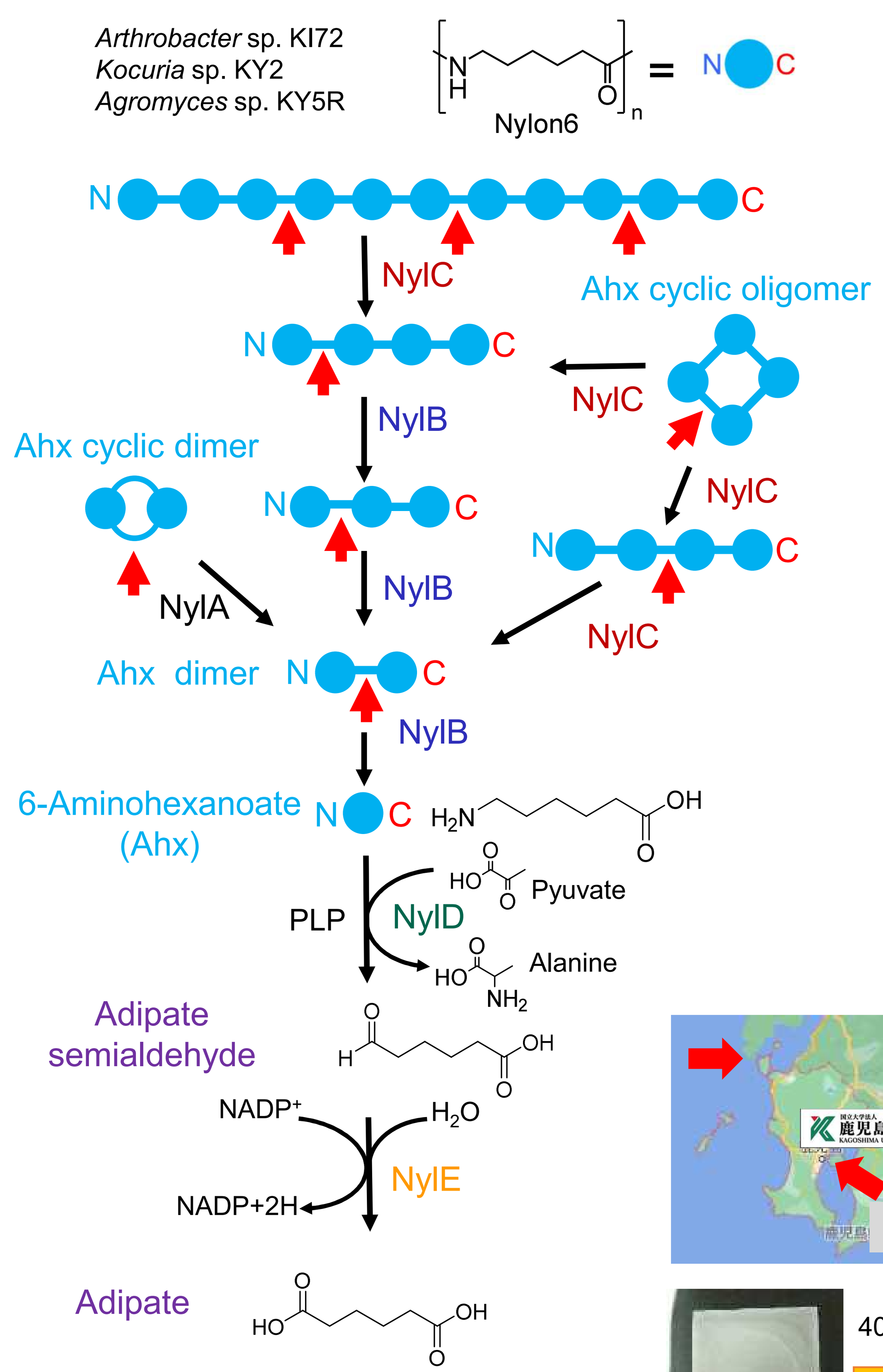
D.KatoG Enzym.Org.Lab

Contribution to this PJ: Providing basic scientific data for exploring biodegradable structures of photoswitchable ocean degradative nylon, and developing the methods of enzymatic nylon recycling

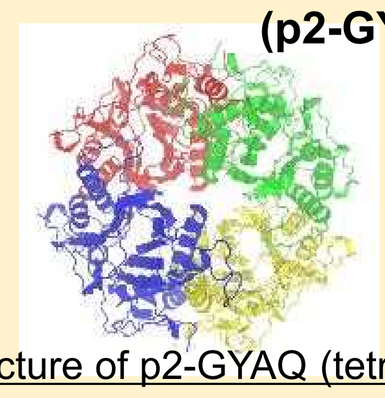


- 1st Enzymatic degradation of iNylon using nylon-hydrolysing enzymes (Nyl series)
- 2nd Screening new nylon-degradative bacteria/enzymes
- 3rd Screening marine bacteria degrading monomer unit
- 4th Elucidation of photo-solubilization mechanism of iNylon
- 5th Development into Nylon recycling demonstration study

Nylon degrading microorganisms and degradation pathway



NylA : cyclic dimer hydrolase
NylB : exo-type hydrolase
NylC : endo-type hydrolase (p2-GYAQ)



Structure of p2-GYAQ (tetramer)

Unique enzymes that only we have in the world

Complete monomerization of commercially available nylon

Treatment	Conversion ratio to monomers (%) Nylon6	Nylon66
1. No treatment	1	2
2. Homogeneous dispersion	10	35
3. Chemical fragmentation	74	86
4. Combined methods 2 and 3	97	quant.

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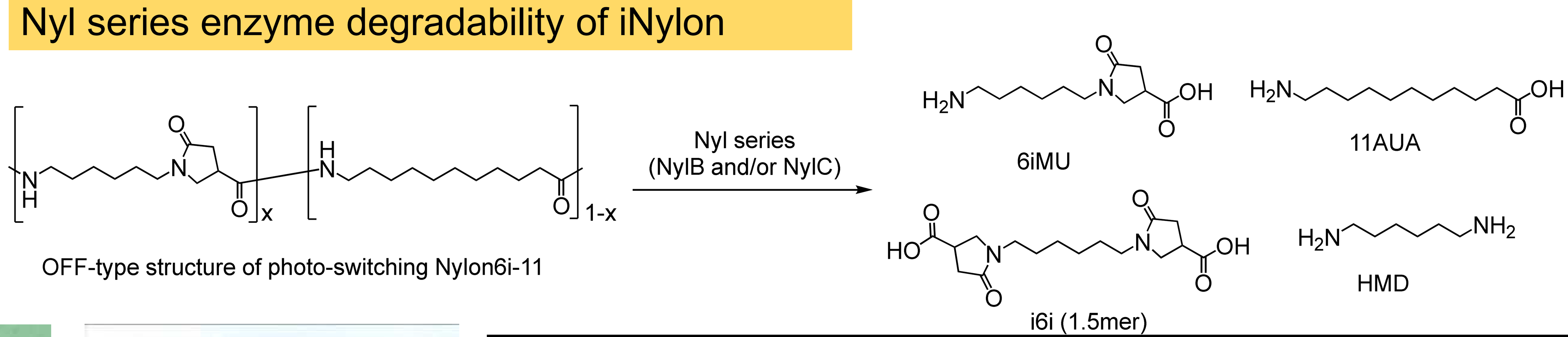
Nyl series enzyme degradability of iNylon

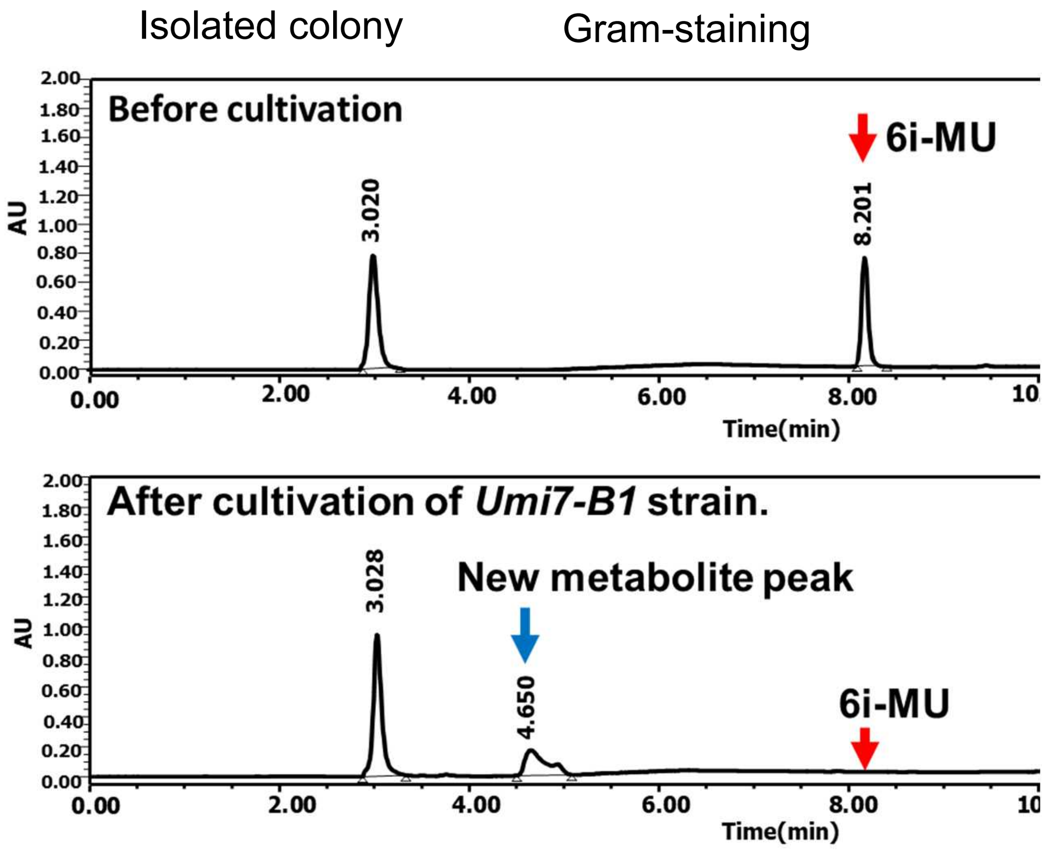


OFF-type structure of photo-switching Nylon6i-11

Photo-switching Nylon		x	M_w	Pretreat.	Monomerization (%)
H-Nylon6i		1	67,000	− / +	50 / −
Nylon6i-11(50%)		0.5	61,100	− / +	3 / 64
Nylon6i-11(75%)		0.25	120,800	− / +	0.1 / 26

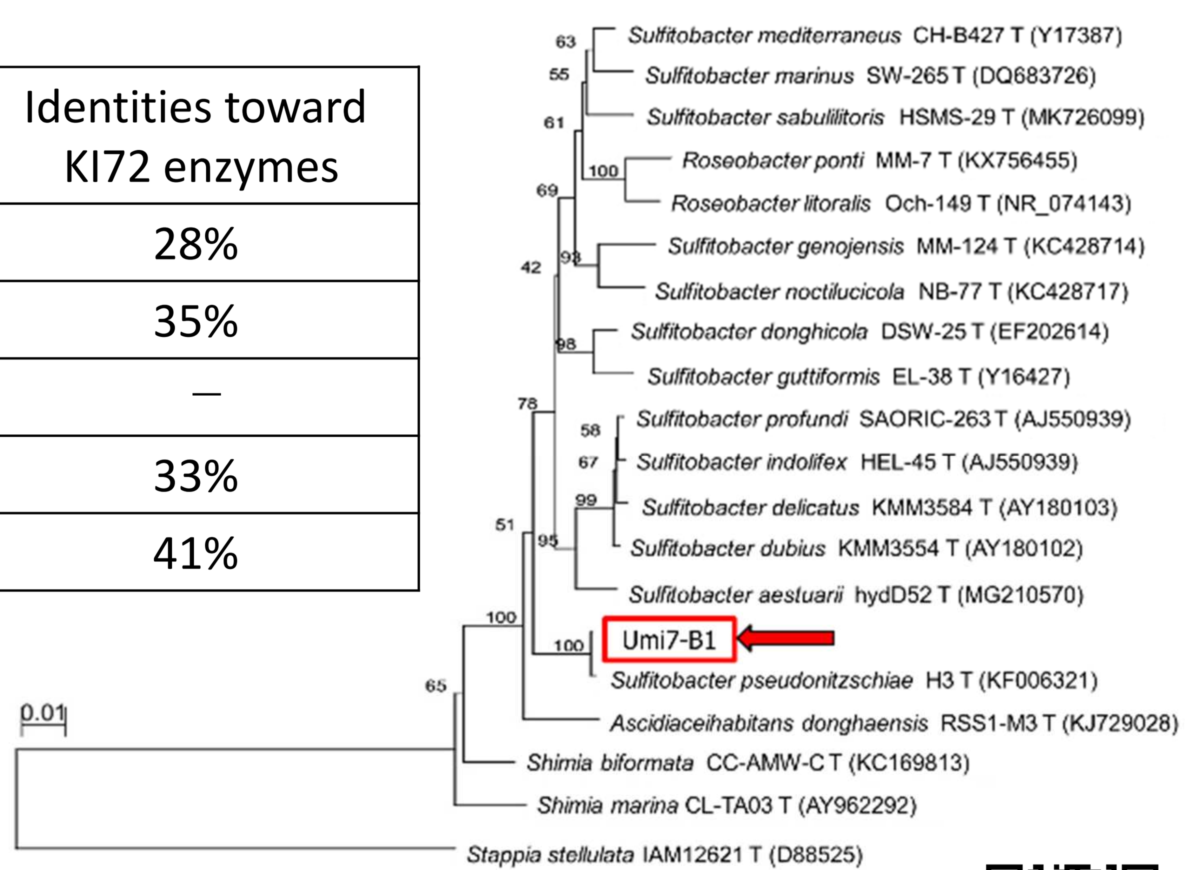
The biodegradability by the Nyl series enzymes was confirmed.

Isolation of iNylon degradation bacterium



Sulfitobacter gulosus Umi7-B1

protein	Identities toward KI72 enzymes
NylA	28%
NylB	35%
NylC	−
NylD	33%
NylE	41%

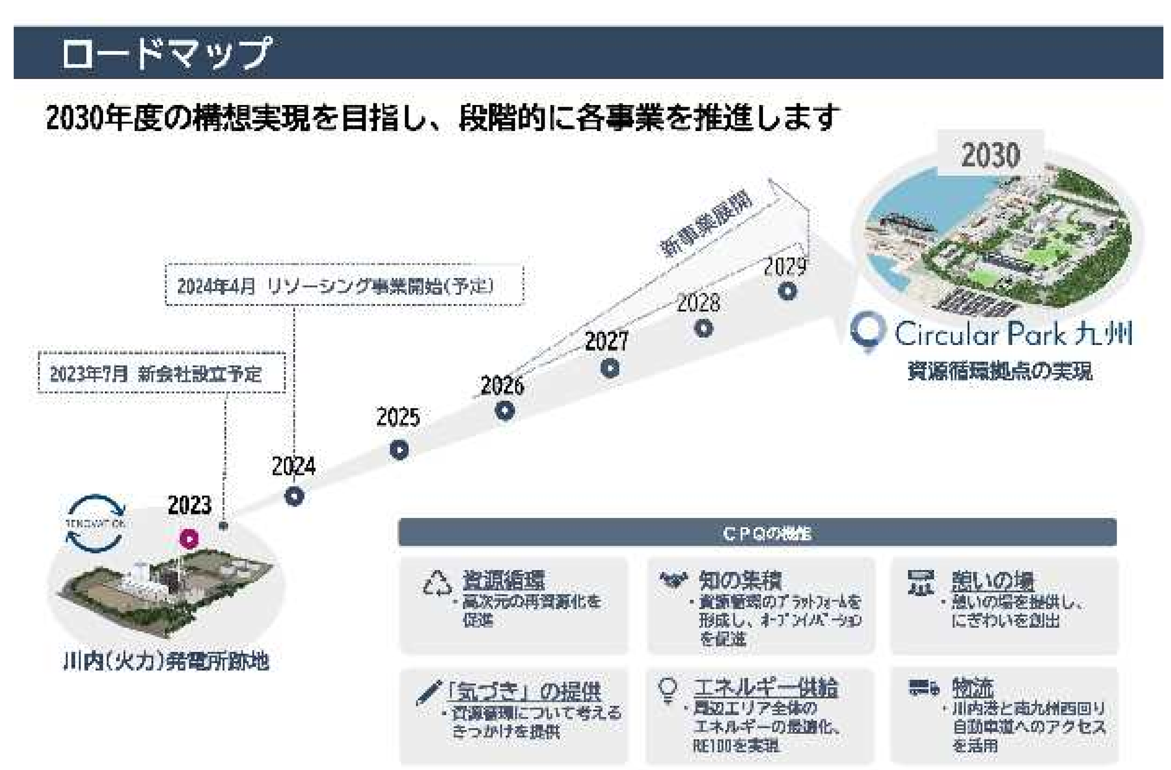


A new marine bacterium degrading 6iMU was successfully isolated.

JP2022-034081

Nylon recycling demonstration study ～Circular Park Kyushu (CPQ)～

Recycling campus using the former site of the Kawauchi Thermal Power Plant in Satsumasendai, Kagoshima



2023.04.06_press release：https://www.kyuden.co.jp/press_h230406-1.html

2024/2/28, 29 The 1st CPQ Summit will be held in Tokyo.

Startup of Nylon chemical recycling from Kagoshima!

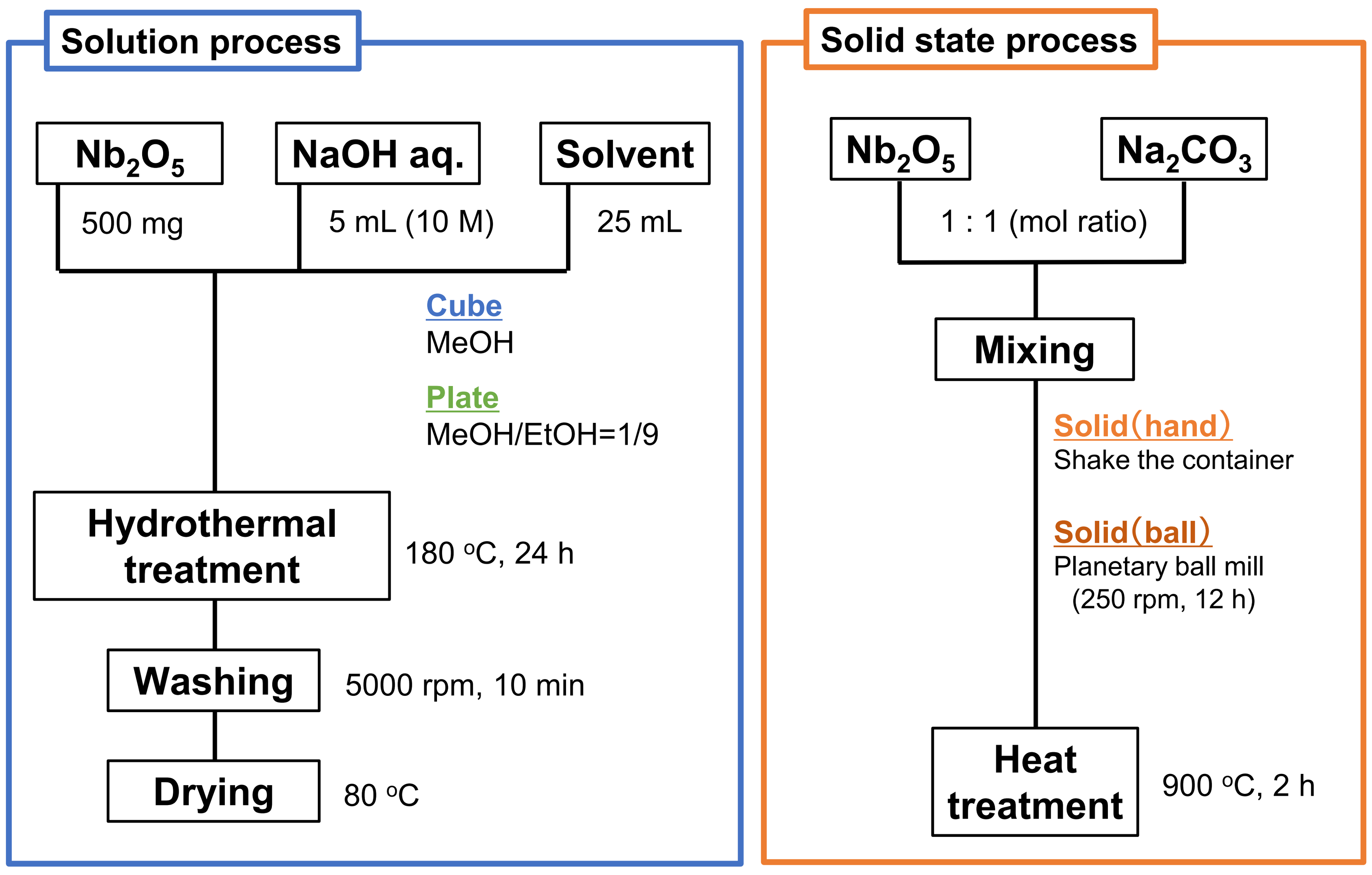
No. A-17-5E

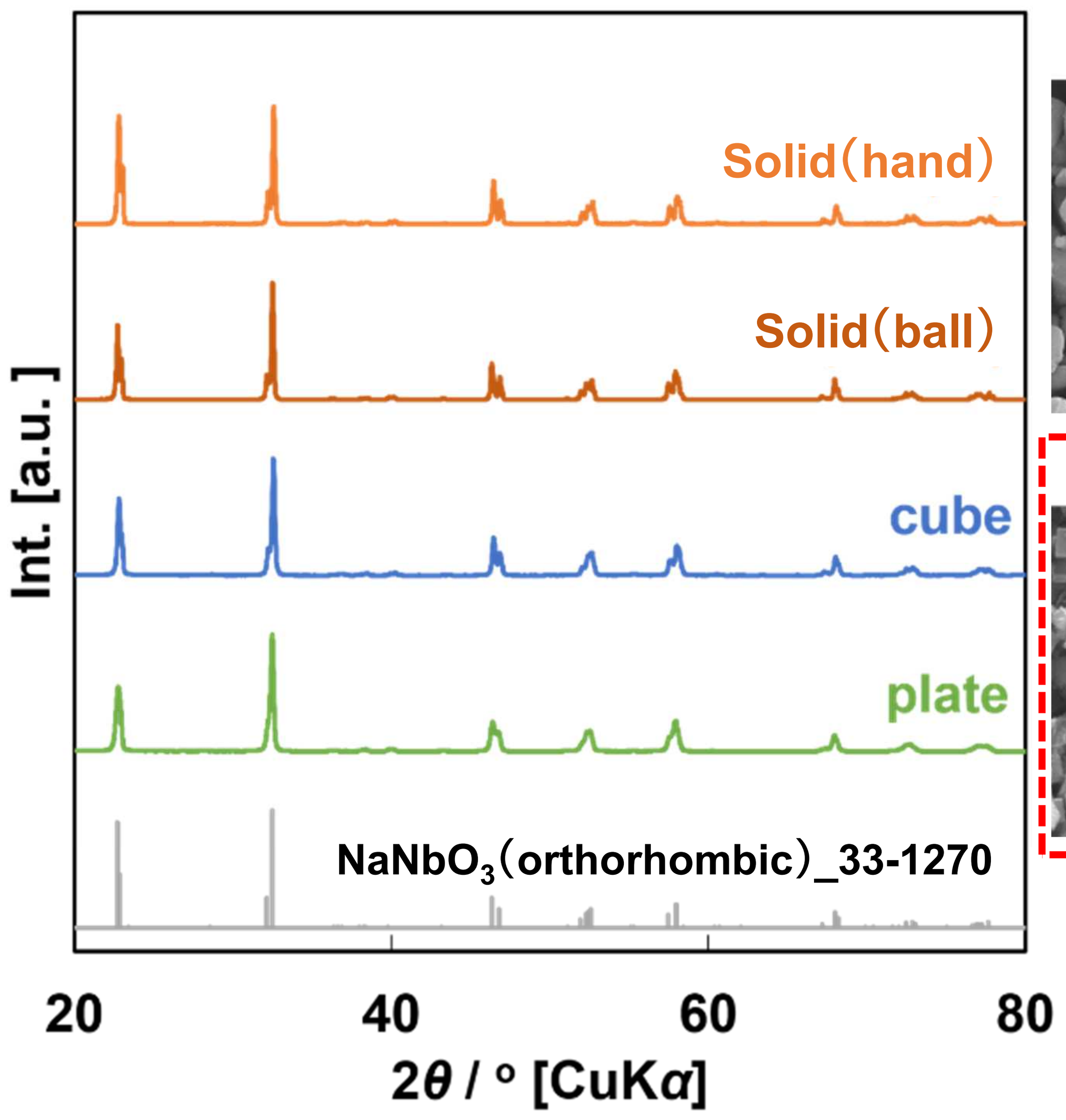
PJ : Development of Photo-switching Ocean-degradable Plastics with Edibility

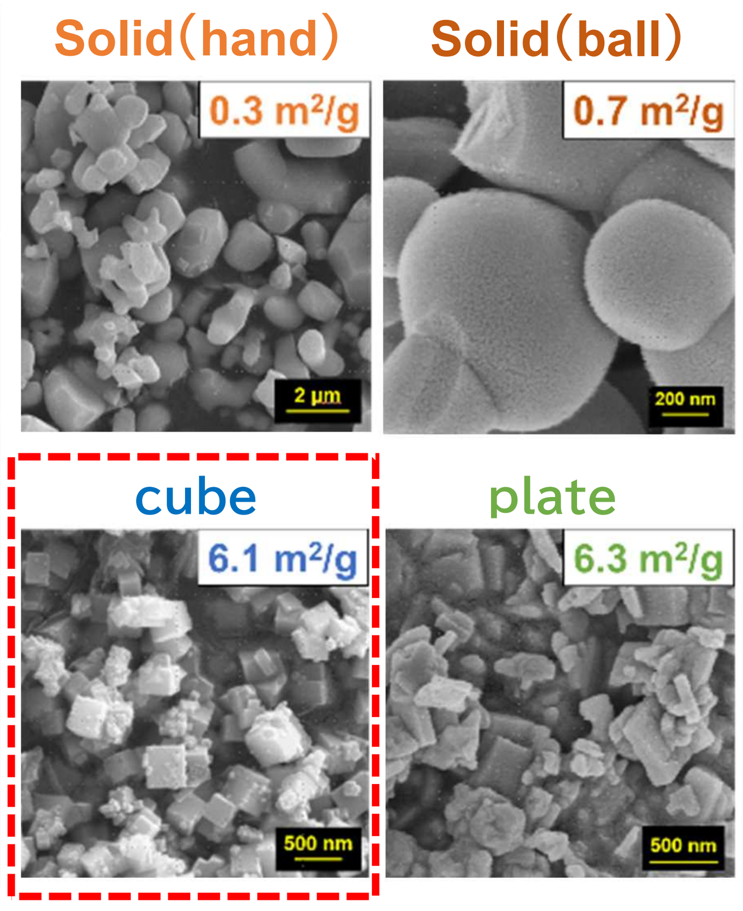
Theme: Nanocomposites with Photo-catalytic Switch
Development of ON-type Photocatalyts

Organization: Tokyo University of Science
Contact: Ken-ichi Katsumata (k.katsumata@rs.tus.ac.jp)

1. Synthesis of ON-type photocatalyst



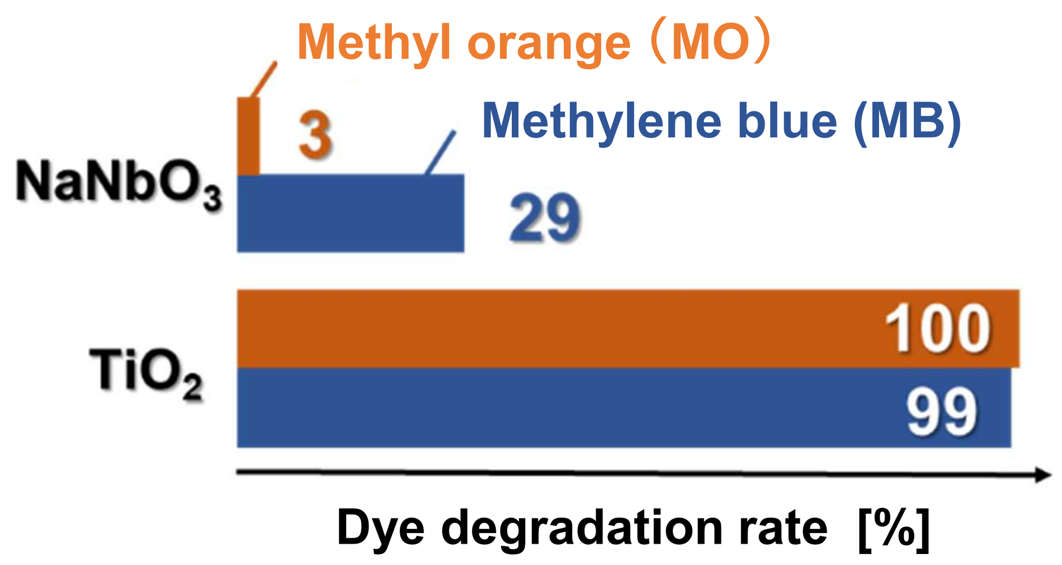


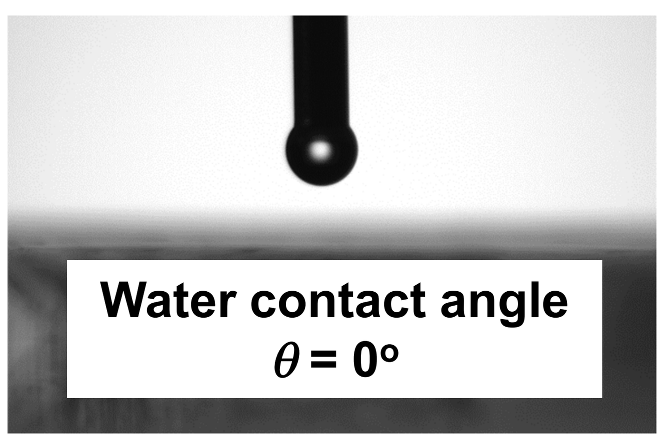


The same $NaNbO_3$ can be prepared.

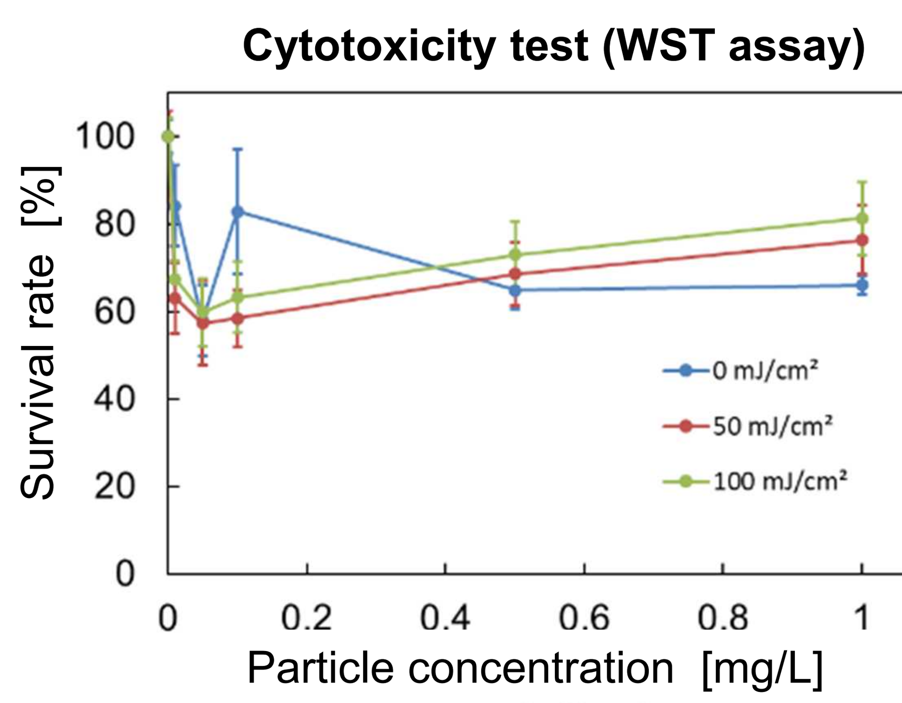
Characterization
- Photocatalytic activity
  → Photodegradation of dye
  → Photoinduced hydrophilicity
- Cytotoxicity test
  → WST assay
- Antimicrobial performance
  → *Escherichia coli* (*E. coli*)

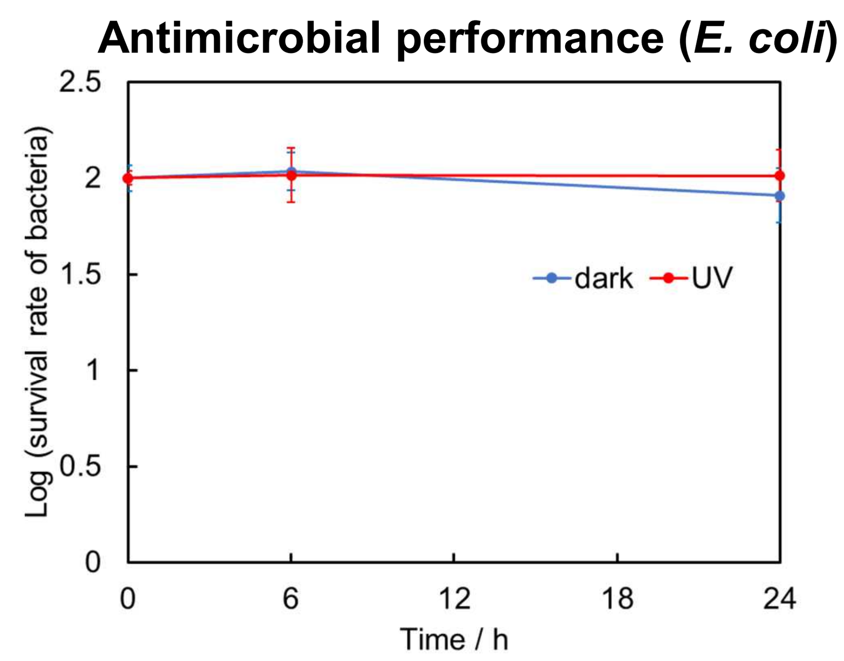
2. Photocatalytic activity





$NaNbO_3$ has lower degradation activity than TiO_2.
$NaNbO_3$ exhibits photoinduced superhydrophilicity.





$NaNbO_3$ has little or no cytotoxic under light irradiation.

※Ack. Prof. C. Ogino (Kobe-U)

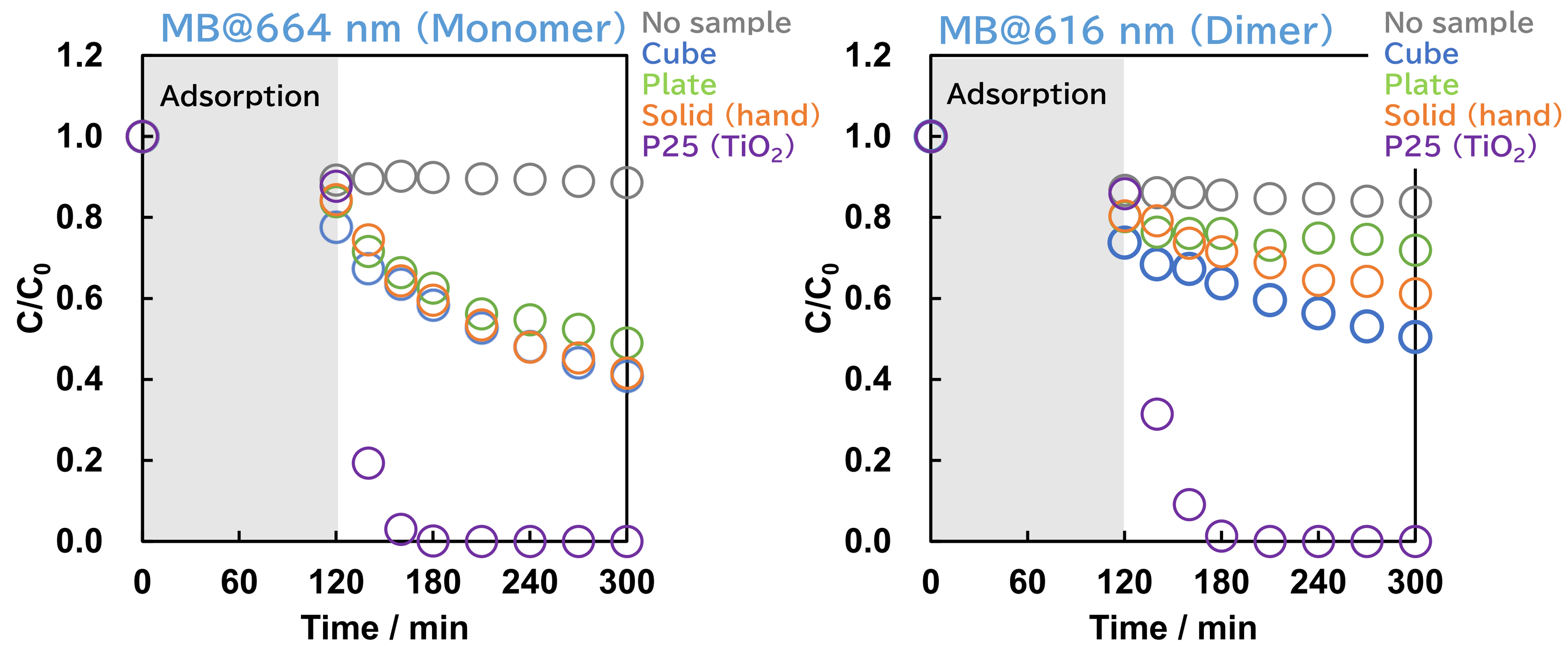
$NaNbO_3$ has little or no antimicrobial performance under light irradiation.

※Ack. Prof. K. Nakata and Dr. S. Usuki (TUAT)

3. Mechanism of ON-type photocatalysis

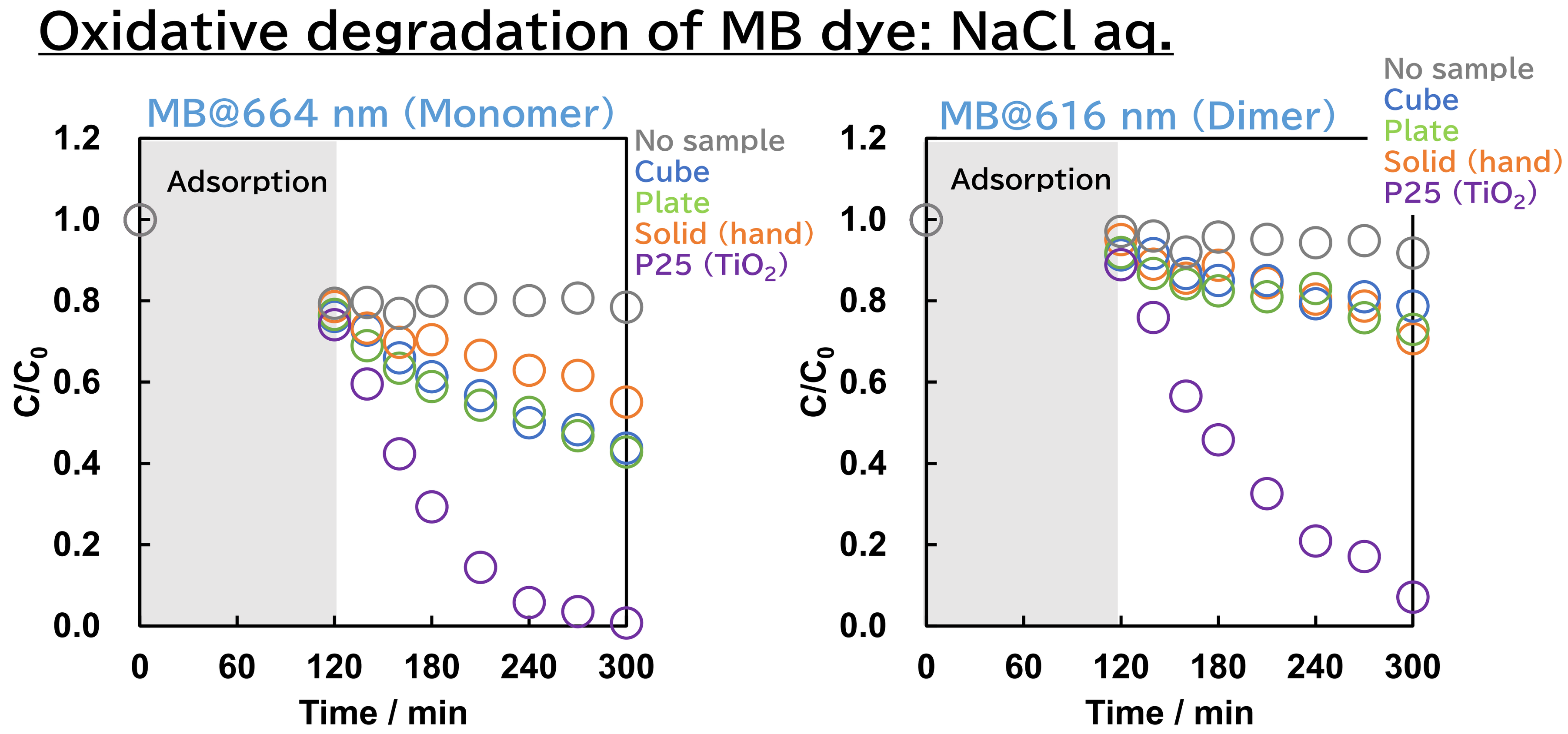
Oxidative degradation of MB dye: Water

Amount of catalyst: 50 mg Concentration of dye: 10 mmol/L
Light source: UV-B lamp



- MB was adsorbed by Solid (hand), Cube, and Plate, and degraded by photoirradiation.
- Solid (hand) and Cube degraded MB dimers, while Plate hardly degraded MB dimers.

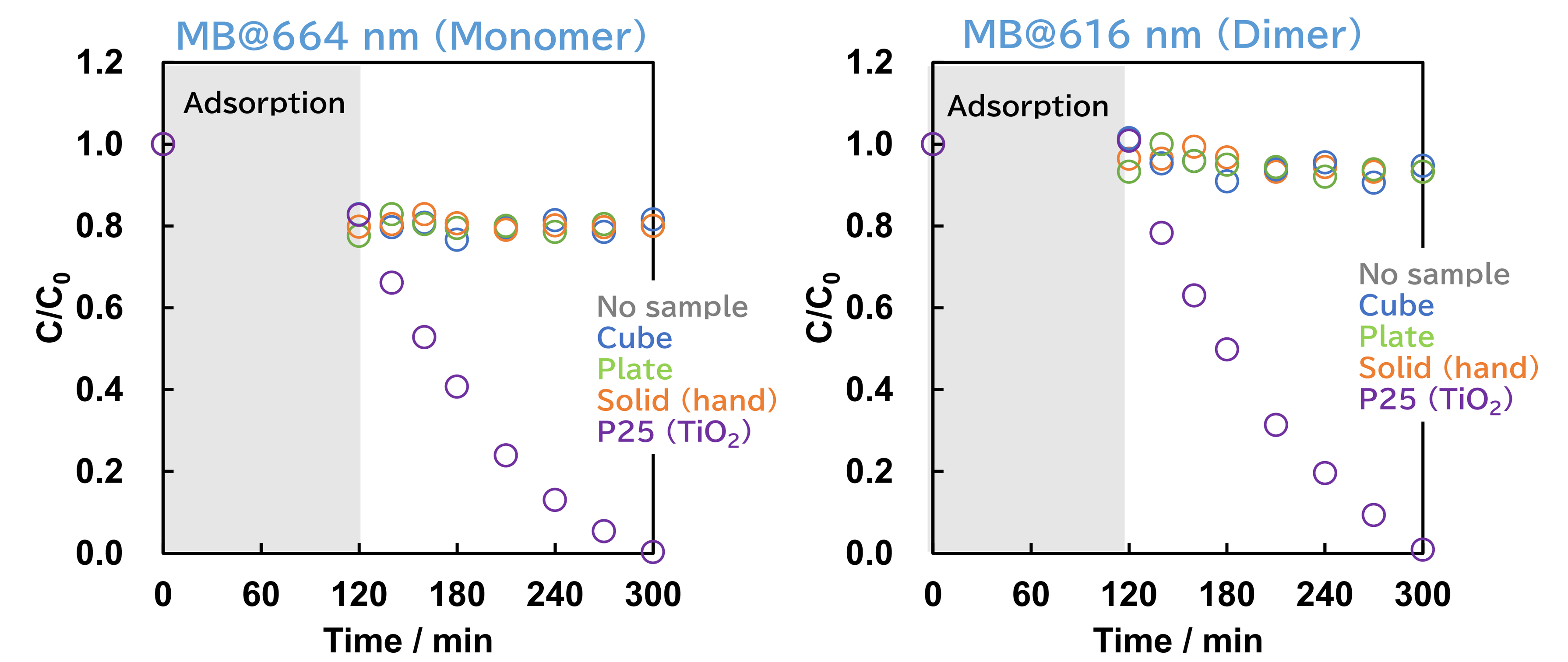
Oxidative degradation of MB dye: NaCl aq.



- Solid (hand)phase, Cube, and Plate adsorbed MB and degraded by photoirradiation.
- Solid (hand), Cube and Plate degraded MB dimer.

With the addition of NaCl,
- The adsorption and degradation of MB monomer were not significantly affected.

Oxidative degradation of MB dye: Artificial seawater



- Solid (hand), Cube, and Plate adsorbed MB but showed no degradation upon photoirradiation.
- P25 (TiO_2) showed MB degradation in artificial seawater.

4. Summary and Acknowledgment

We investigated the synthesis process of $NaNbO_3$ photocatalyst, which exhibits low degradation activity and light-induced hydrophilicity, and were able to synthesize $NaNbO_3$ with controlled particle size and morphology. The photocatalyst was found to have lower oxidative degradation activity, highly hydrophilic, and almost no cytotoxic and antibacterial properties compared to titanium dioxide. It is considered to be effective as an assist material for plastic degradation by microorganisms.

The researches were supported by Grant-in-Aid from moon-shot project (JPNP18016) of New Energy and Industrial Technology Development Organization (NEDO), Japan.

No. A-17-6E

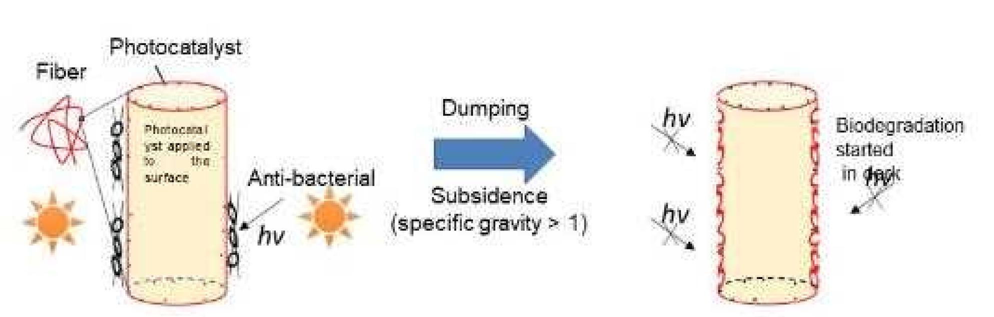
PJ : Development of Photo-Switching Ocean-Degradable Plastics with Edibility

Theme: Design of OFF-type composites with photocatalysts

Organization: TUAT, TUS, AIST, ORIST
Contact: Kazuya Nakata (nakata@go.tuat.ac.jp)

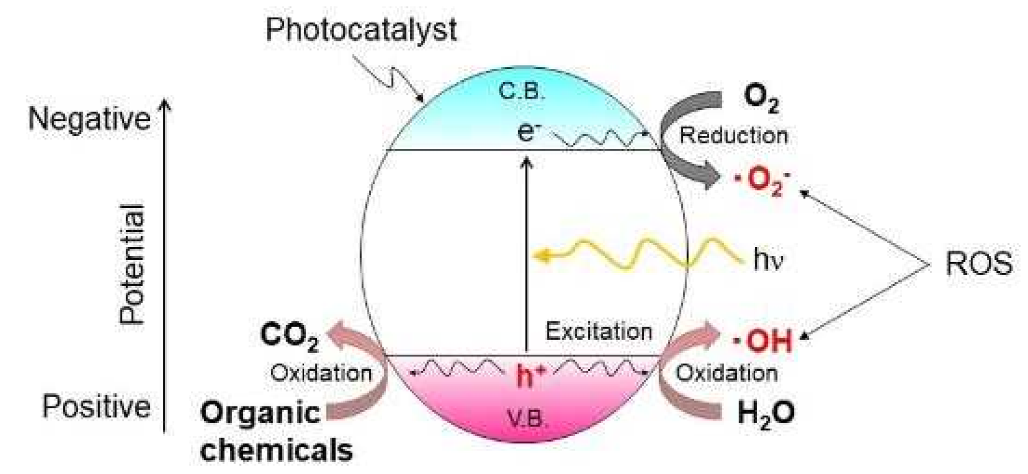
Introduction

Bio-degradable plastics with OFF-type photo switch system



What kind of photocatalyst is required?

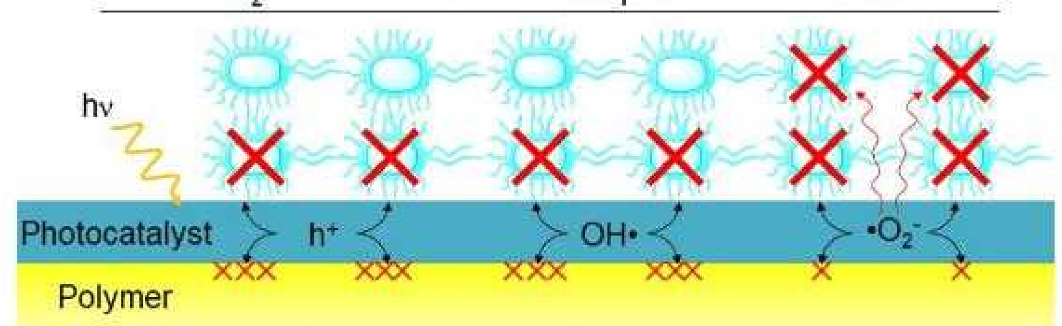
(1) Under visible light, (2) without decomposing polymer, (3) sterilizable photocatalyst



Three types of h^+, OH•, and O_2^-• affect for sterilization and polymer decomposition.

Comparison of ROS

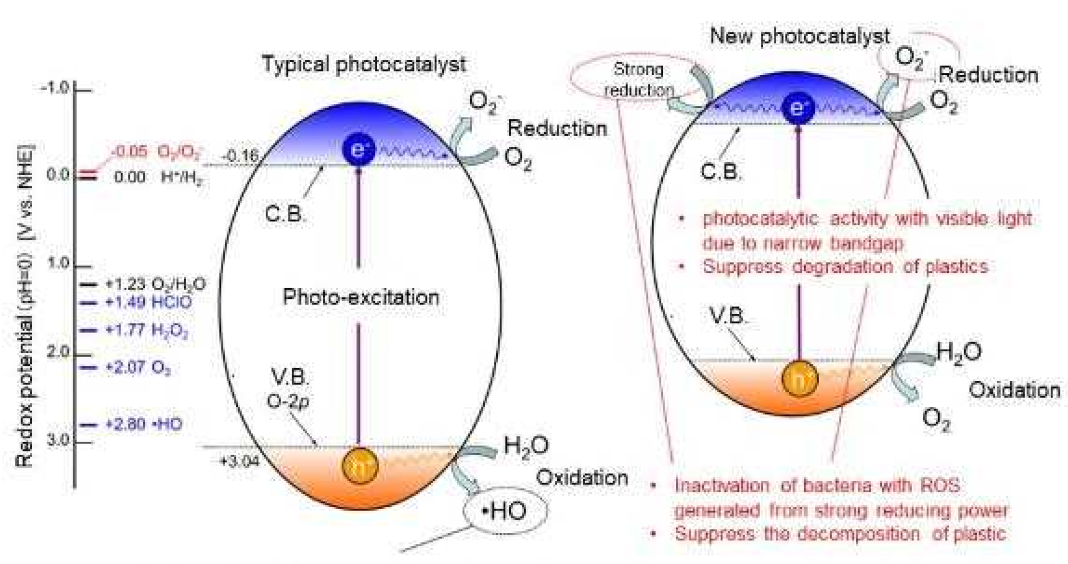
ROS	Life time	Diffusion length	Redox potential (vs. NHE)
h^+	<1 ns	In photocatalyst	Depends on photocatalyst
OH•	70 ns	20 nm	+2.8 V
•O_2^-	5 s	100 μm	+0.16 V



• Sterilization by •O_2^- is preferable

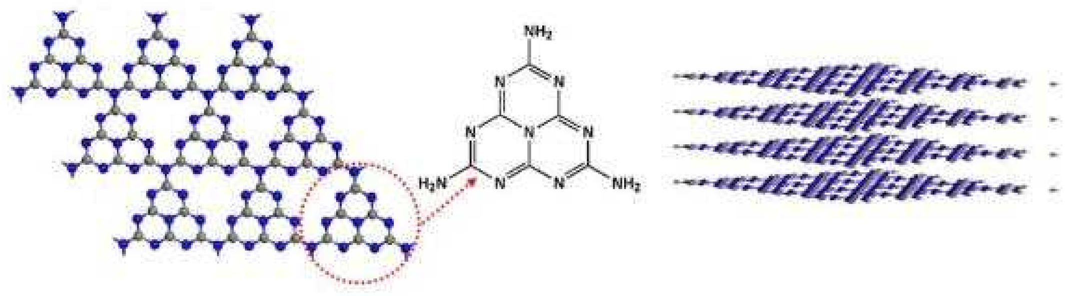
Development of antibacterial photocatalyst

Photocatalyst that can be sterilized under visible light without decomposing polymer



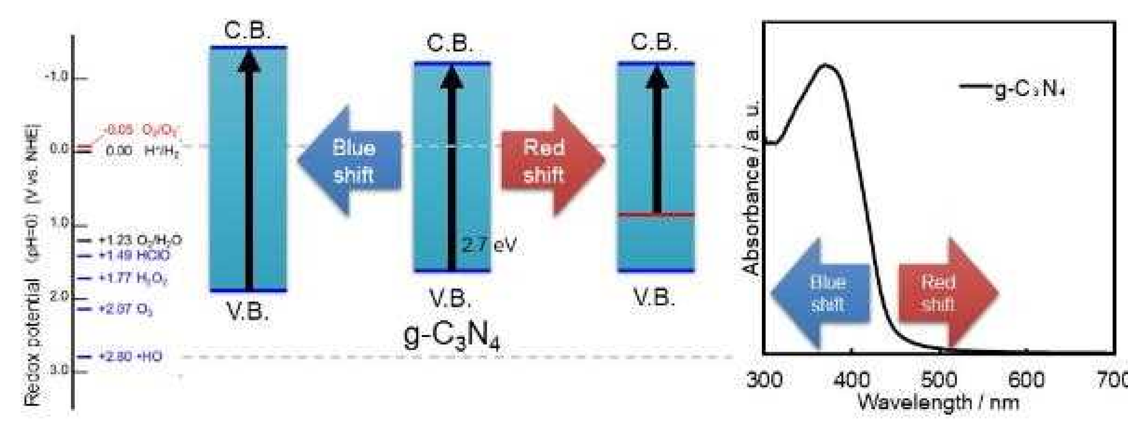
Most organic substances including polymer and bacteria can decomposed by the strong oxidizing power of reactive oxygen species.

g-C_3N_4 photocatalyst



- Visible-light-responsive photocatalyst composed of carbon and nitrogen
- Metal-free and low toxicity
- Layered structure stacking 2D sheets
- 2D sheet can be peeled off by various treatments
- It is possible to dope elements, allowing control of the electronic structure
- Easy to generate $O_2^{\bullet -}$ than OH radicals

Improvement of g-C_3N_4

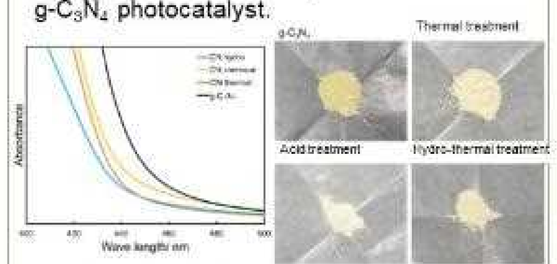


	Blue shift	Red shift
Adv.	High activation by improving oxidization ability (less amount of sample necessary)	Works in environments where short wavelength light is not present (Can be used in a wide range of environments)
Disadv.	Inactivated in the absence of short wavelength light	Low activation due to decreased oxidation ability
Method	Delamination by thermal oxidation treatment	Doping

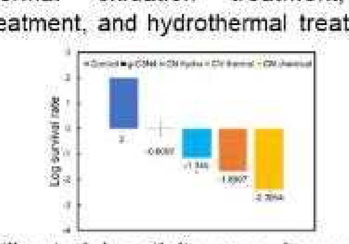
Development of blue-shift type photocatalyst

Effect of treatments

Various treatments were performed on the g-C_3N_4 photocatalyst.

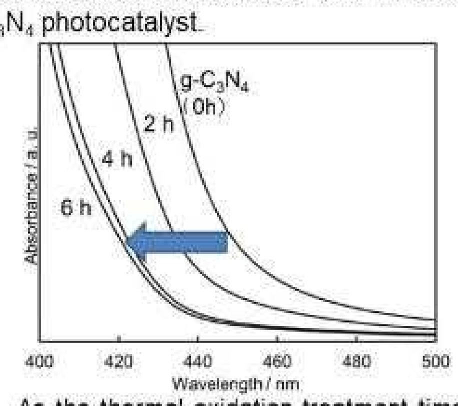


- All of g-C_3N_4 were blue-shifted by thermal oxidation treatment, acid treatment, and hydrothermal treatment.



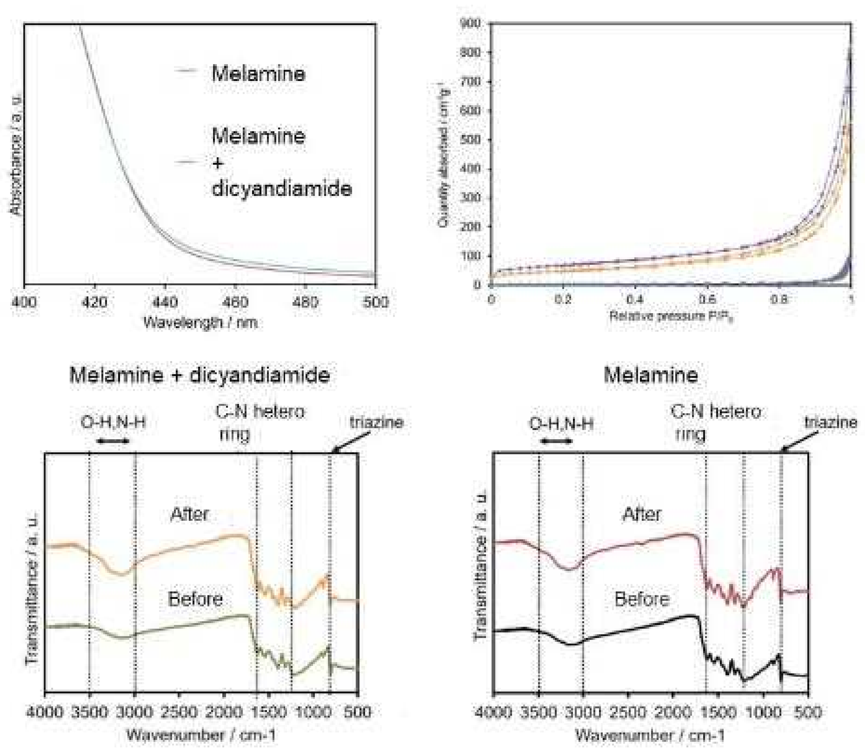
- Antibacterial activity was improved by thermal oxidation treatment, acid treatment, and hydrothermal treatment of g-C_3N_4.

We investigated the optimization of the thermal oxidation treatment time of the g-C_3N_4 photocatalyst.

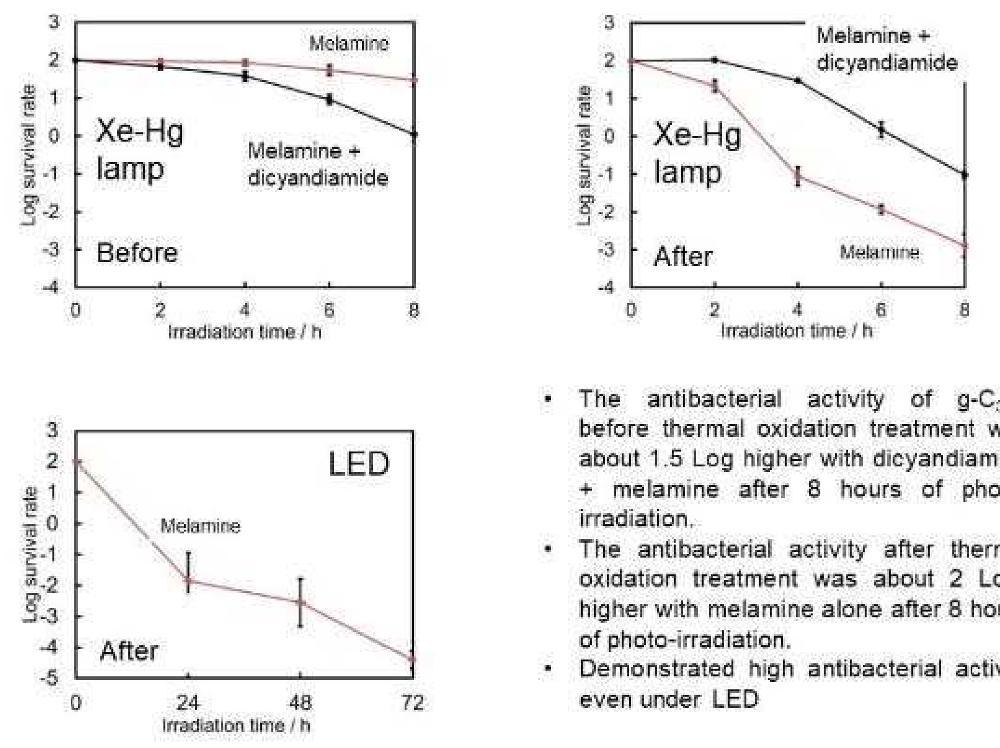


- As the thermal oxidation treatment time became longer, the absorption edge was blue-shifted.
- Since the sample having a long thermal oxidation treatment time is lighter in color than the sample having a short thermal oxidation treatment time, it is expected that it will be easier to apply to white fibers in the future.

Charcterization



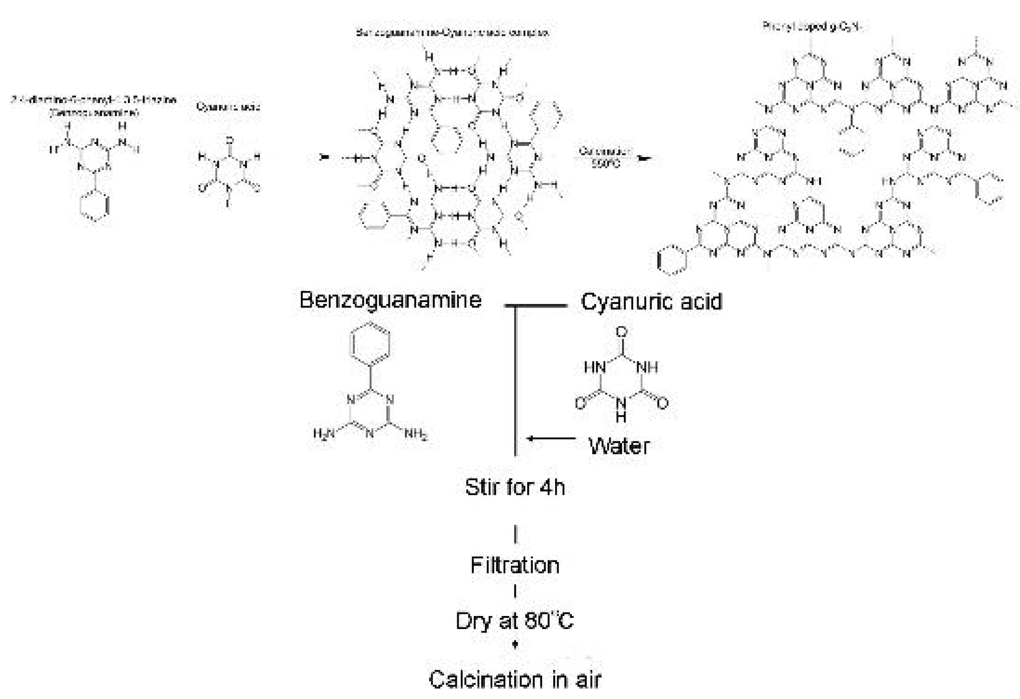
Antibacterial activity



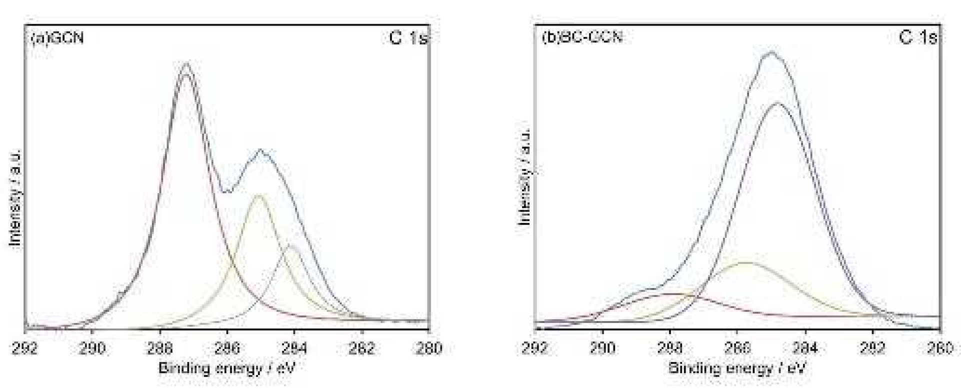
- The antibacterial activity of g-C_3N_4 before thermal oxidation treatment was about 1.5 Log higher with dicyandiamide + melamine after 8 hours of photo-irradiation.
- The antibacterial activity after thermal oxidation treatment was about 2 Logs higher with melamine alone after 8 hours of photo-irradiation.
- Demonstrated high antibacterial activity even under LED

Development of red-shift type photocatalyst

Synthesis of BC-GCN

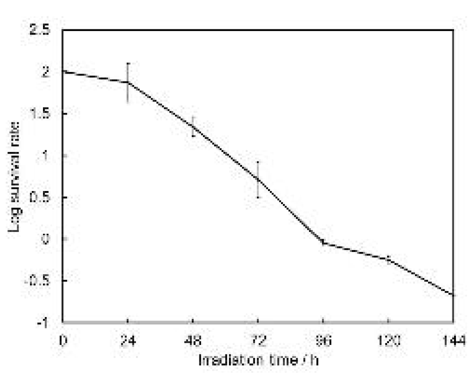


XPS

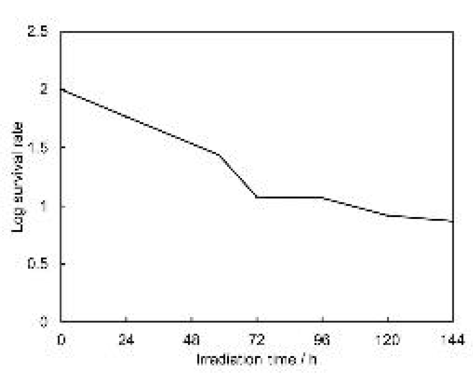


Sample	C(atm %)	N(atm %)	O(atm %)
GCN	37.75	45.08	17.17
BC-GCN	61.54	26.79	11.67

Antibacterial activity



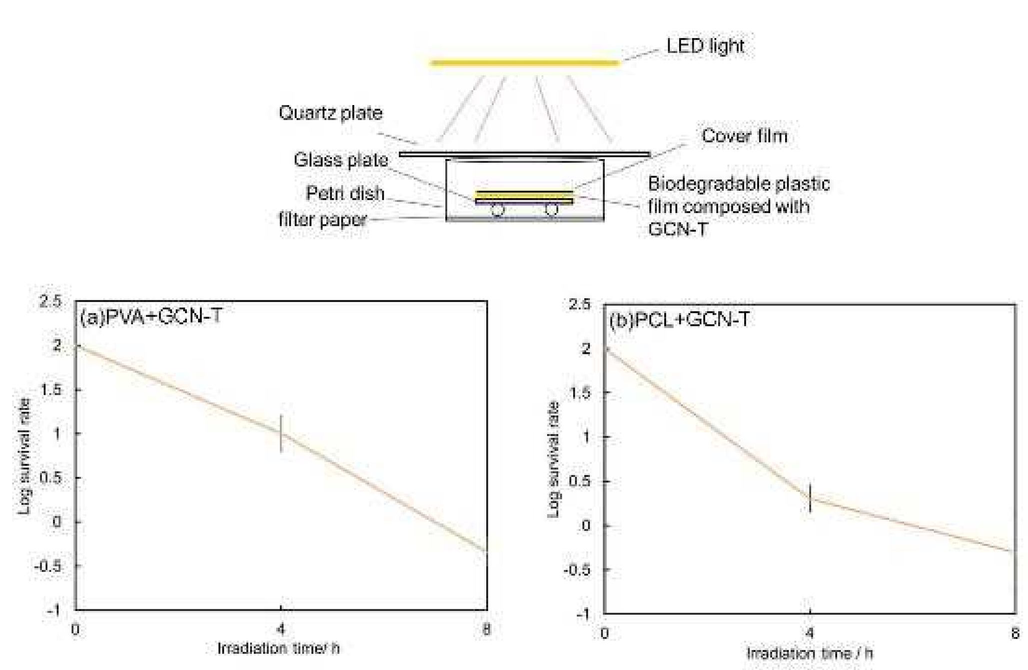
Test conditions: E. Coli.
in physiological saline solution
λ>420 nm
1000 W/m²



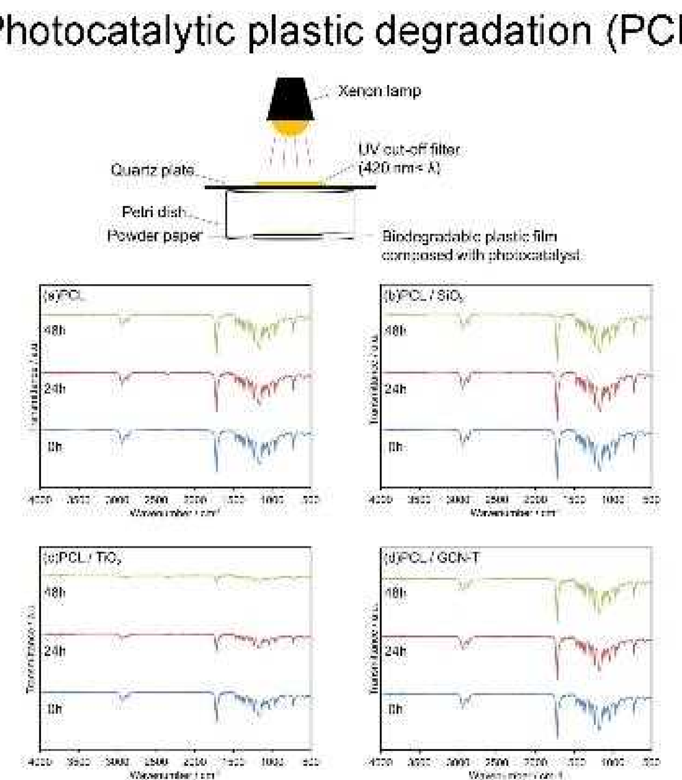
Test conditions: E. Coli.
in physiological saline solution
λ>650 nm
1000 W/m²

Development of red-shift type photocatalyst

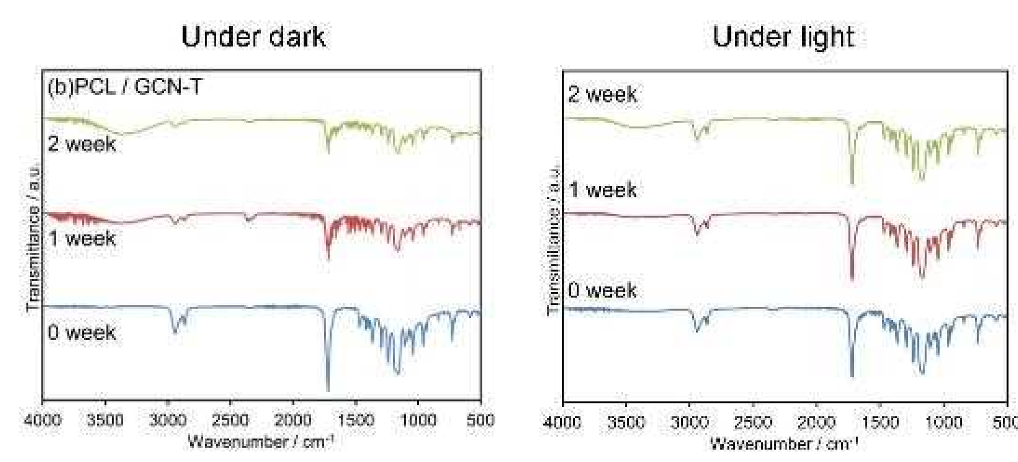
Antimicrobial performance of plastic/photocatalyst composite films



Photocatalytic plastic degradation (PCL)



OFF-typed photo-switching function

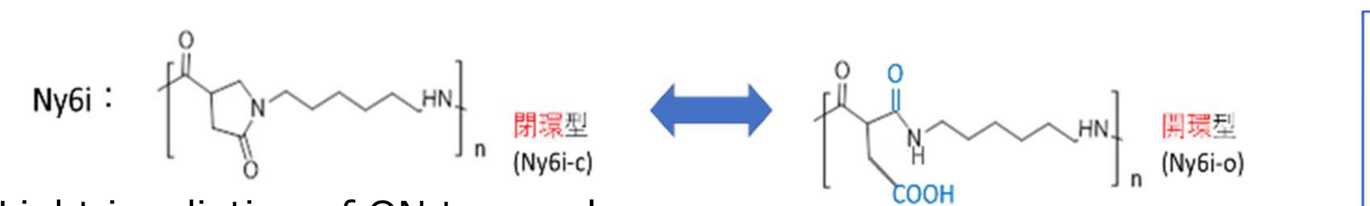


- In seawater (Sagami Bay, after autoclave treatment)
- PCL-degrading bacteria: Alcanivorax xenomutans (NBRC 108843 (JC109))
- Light source: white LED (light intensity: 6000 lx).

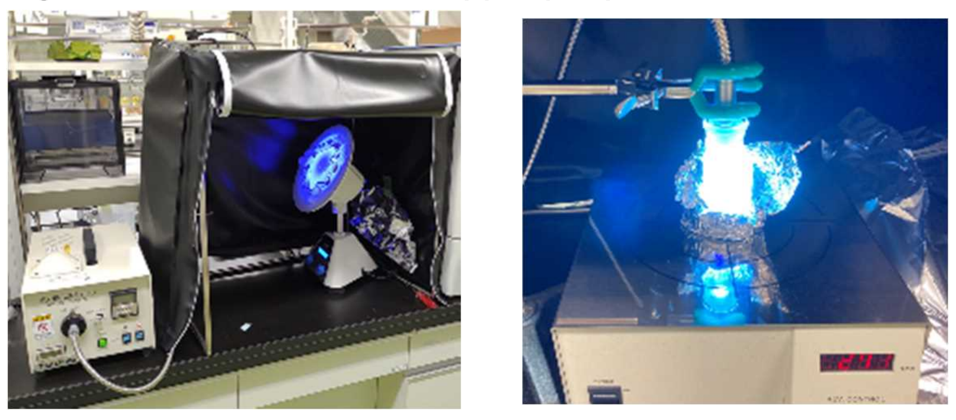
No. A-17-7E
PJ : Development of Photo-switching Ocean-degradable Plastics with Edibility
Theme: Switch performance evaluation and safety evaluation

Organization: National Institute of Advanced Industrial Science and Technology (AIST)
Contact: Nakayama Atsuyoshi (a.nakayama@aist.go.jp)

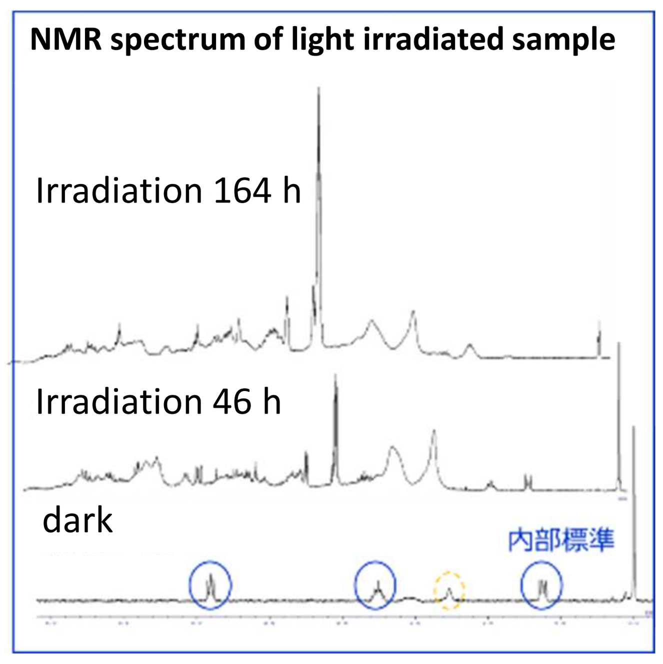
Photo switch operability and biodegradability…ON type



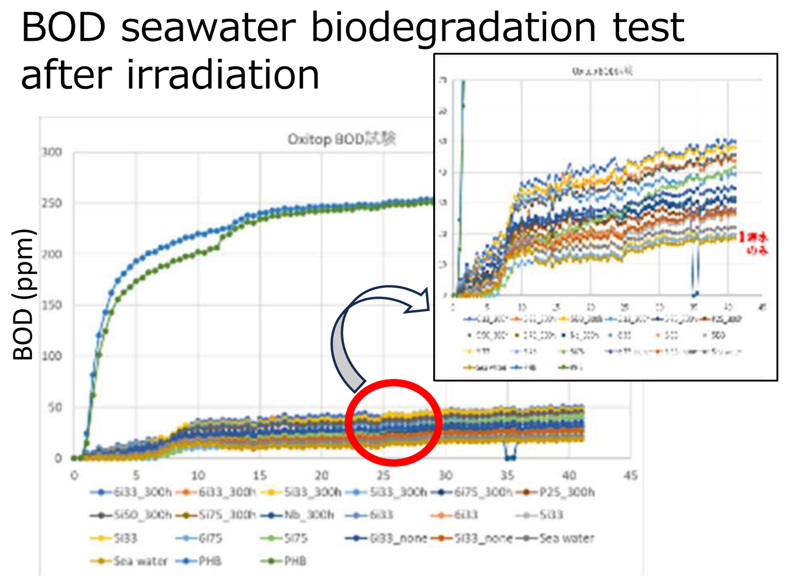
Light irradiation of ON type polymer

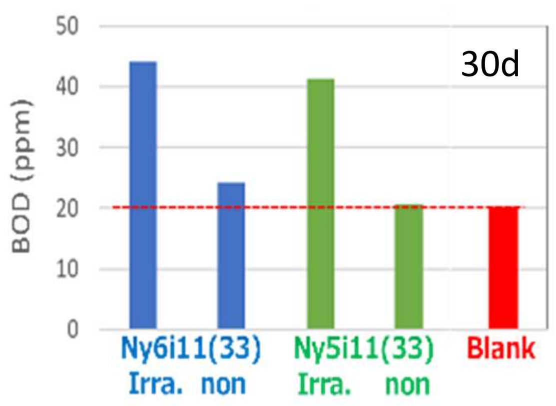


Water-soluble components are generated and increased by light irradiation (by NMR)

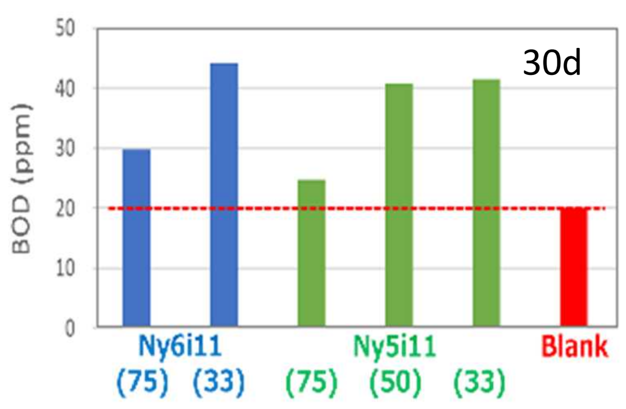


BOD seawater biodegradation test after irradiation



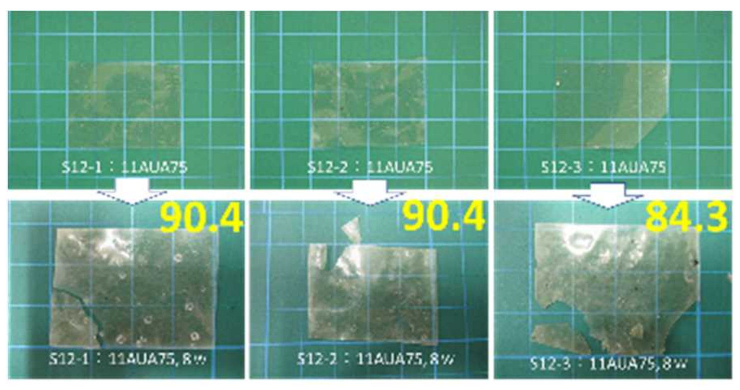


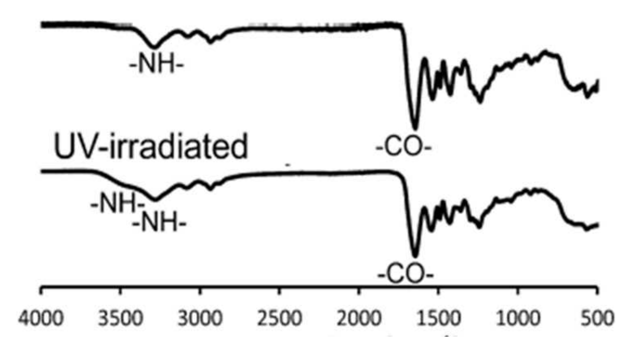
In the light-irradiated samples, biodegradation of water-soluble components progressed for both Ny6i11(33) and Ny5i11(33).



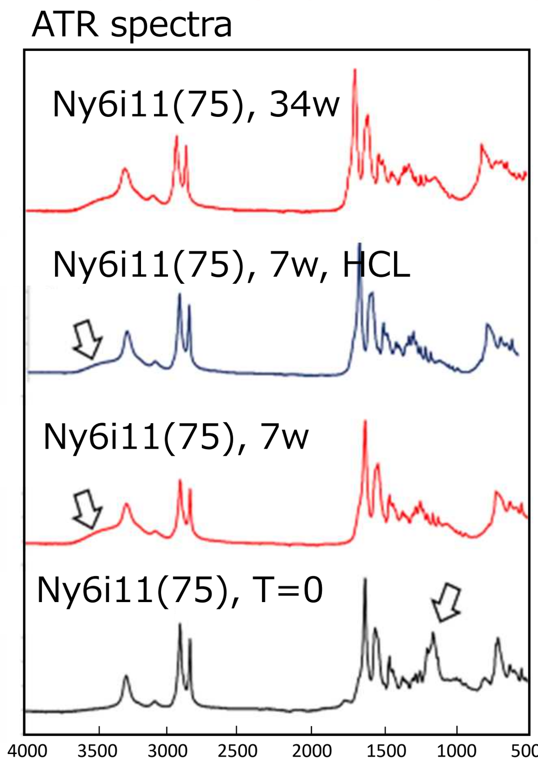
When the second monomer component (Ny11) increases, the BOD value decreases and the progress of biodegradation becomes slower.

sea immersion test of Ny6i11 series

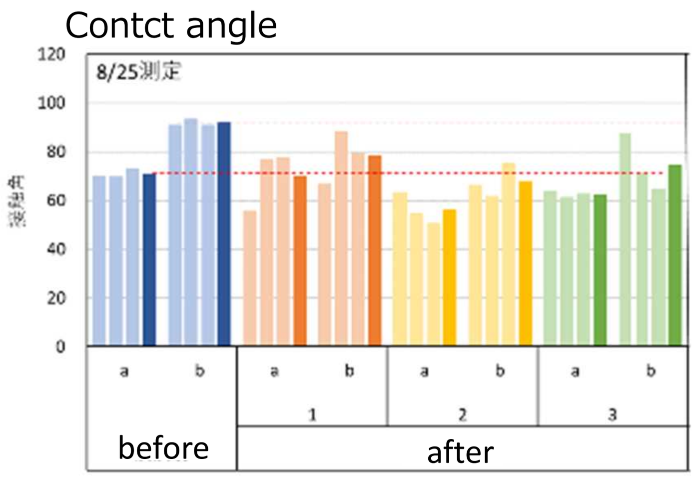




ATR spectra



Contct angle

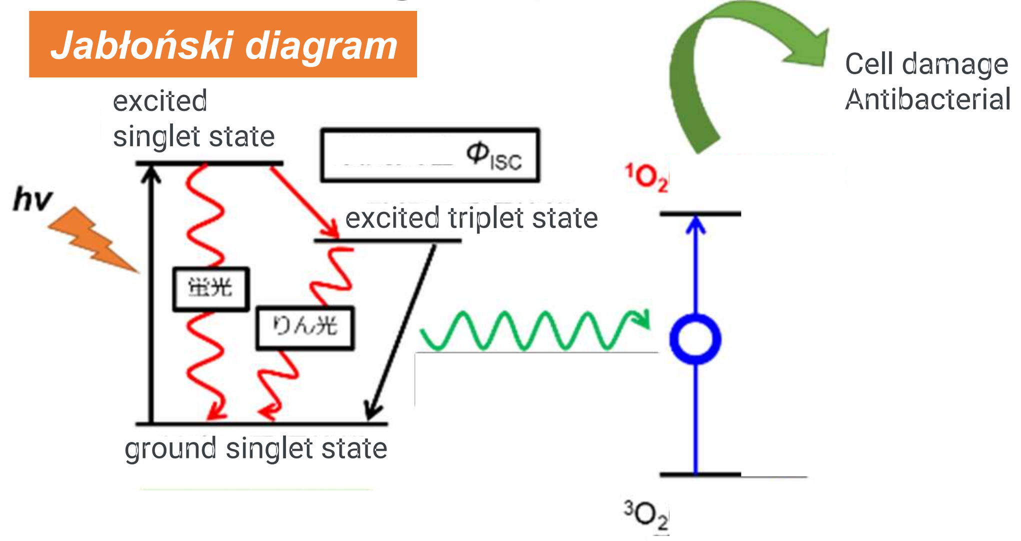


Due to the long-term immersion, hydrolysis of the resin surface progressed, and it was confirmed that the surface became hydrophilic. The ATR patterns were consistent between laboratory light irradiation and actual sea immersion.

Pigment-based OFF type resin

Utilization of organic photosensitizers

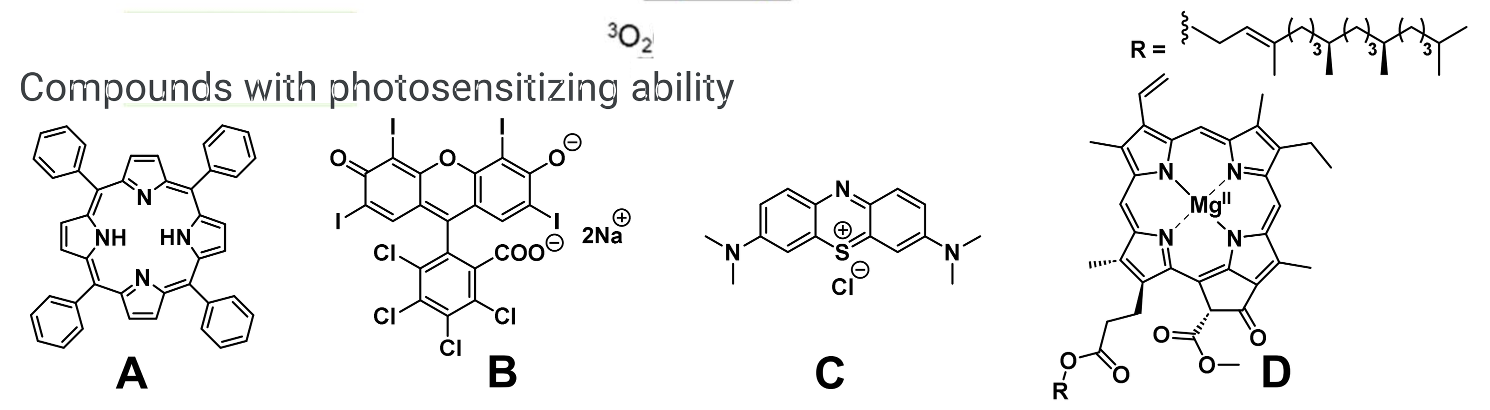
Jabłoński diagram



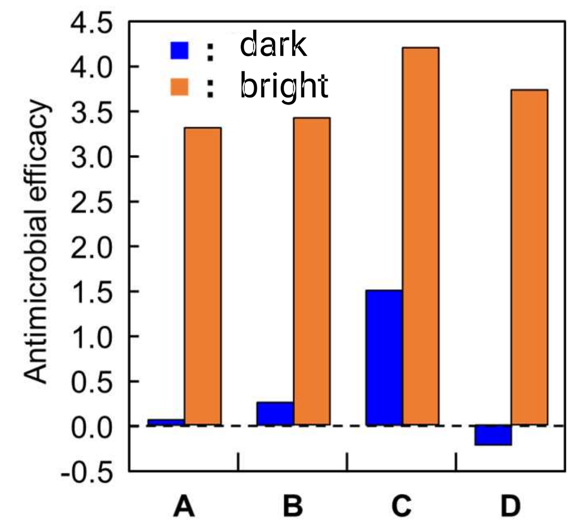
Utilizes singlet oxygen, which is not a radical but has strong oxidizing power.

Because it has a very short lifespan, it can effectively exert its antibacterial power on the material surface.

Compounds with photosensitizing ability

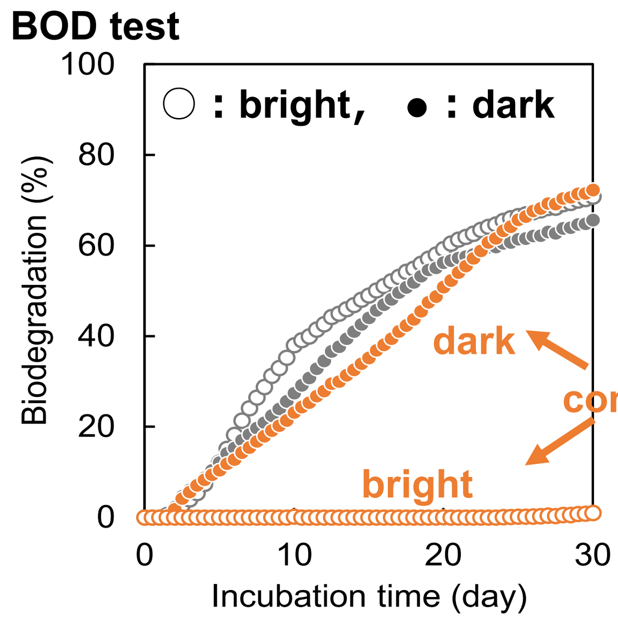


Photoantibacterial test



Antibacterial activity value exceeds 2 under light irradiation conditions
⇒ Exhibits photo-antibacterial properties

BOD test



Seawater biodegradability (BOD test) is significantly suppressed in resins containing Substance A, which has strong photo-antibacterial properties.

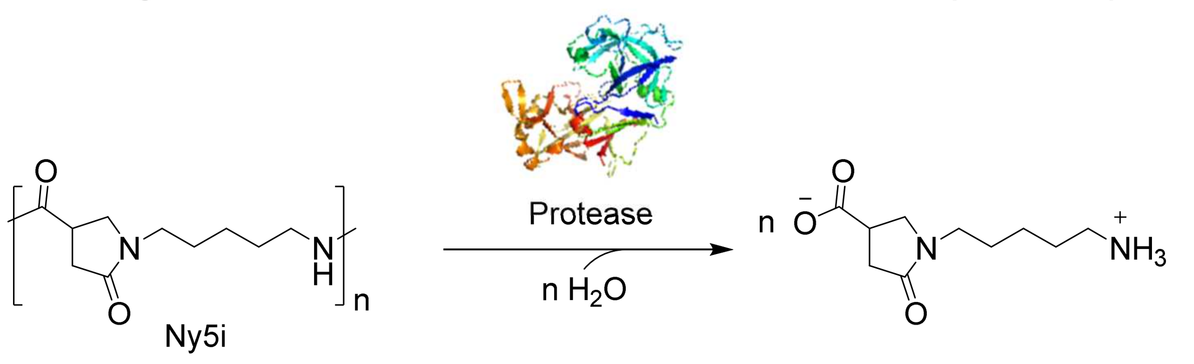
Bacteria count after test(cfu/mL)	Irrad. ($\times 10^3$)	Control ($\times 10^3$)
Blank	915	10
Polymer	7,725	6,417
+compd. A	50	2,045

- The number of bacteria in the test seawater containing compound A and under light irradiation conditions was significantly reduced.
- ⇒ Sterilized by active oxygen

Photo-antibacterial activity contributes to suppression of decomposition

Enzymatic degradation evaluation of polymers

An evaluation method for the enzymatic degradation of synthetic polymers possessing amide bonds was developed. Proteases, which digest proteins and peptides in living organisms, were chosen for hydrolysis of the peptide bonds within the targeted polymer. The generated monomers could be detected by NMR spectroscopy.



quantified by NMR

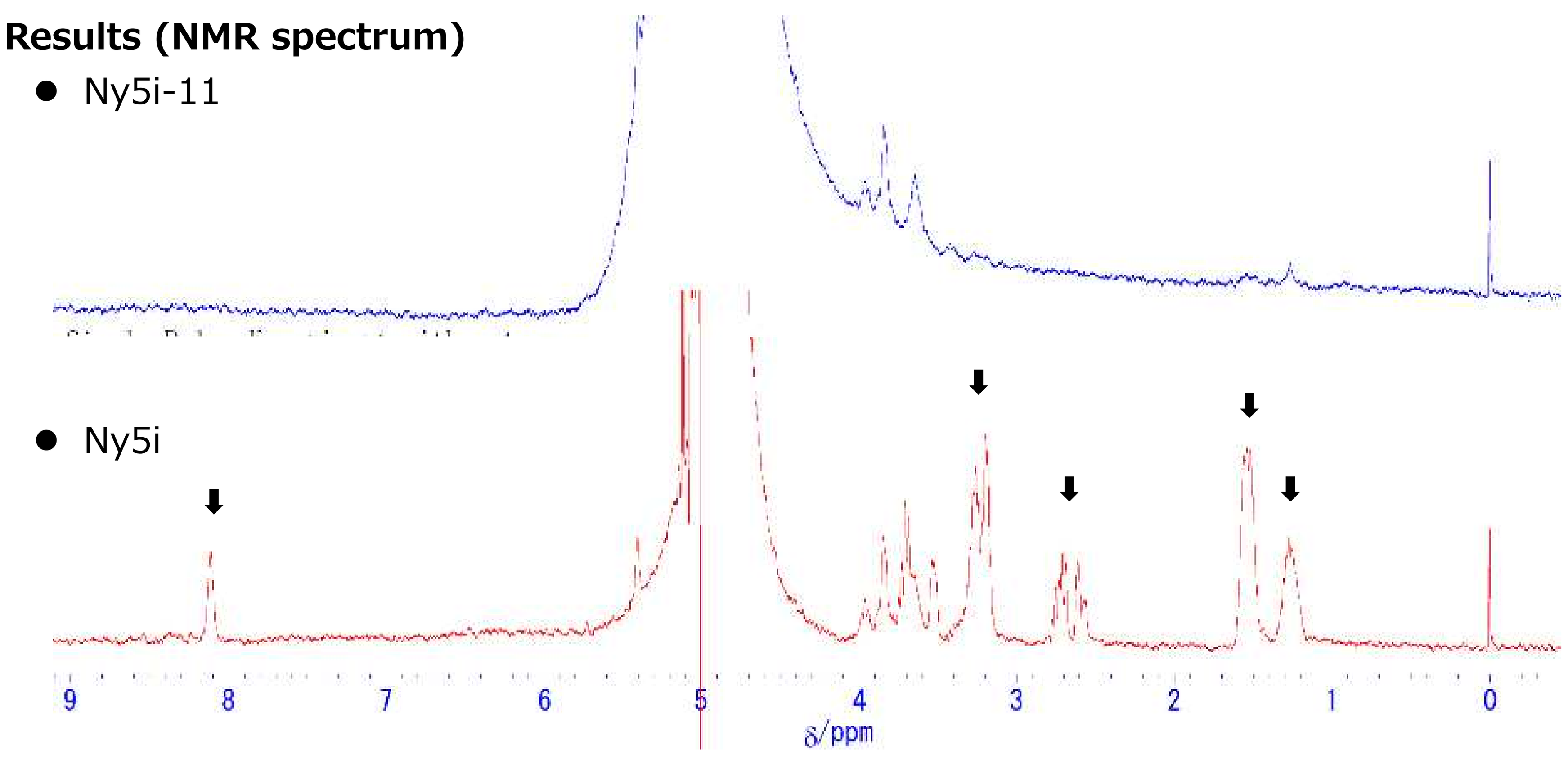
Tested proteases

1. Pepsin (aspartic protease), optimum pH1-3, cleave the bond neighboring acidic or aromatic amino acids
2. Papain (cysteine protease), optimum pH7-8, cleave the bond neighboring basic or Glycine or Leucine
3. Trypsin (serine protease), optimum pH7-8, cleave the bond neighboring basic amino acids
4. Chymotrypsin (serine protease), optimum pH8-9, cleave the bond neighboring aromatic amino acids

Method

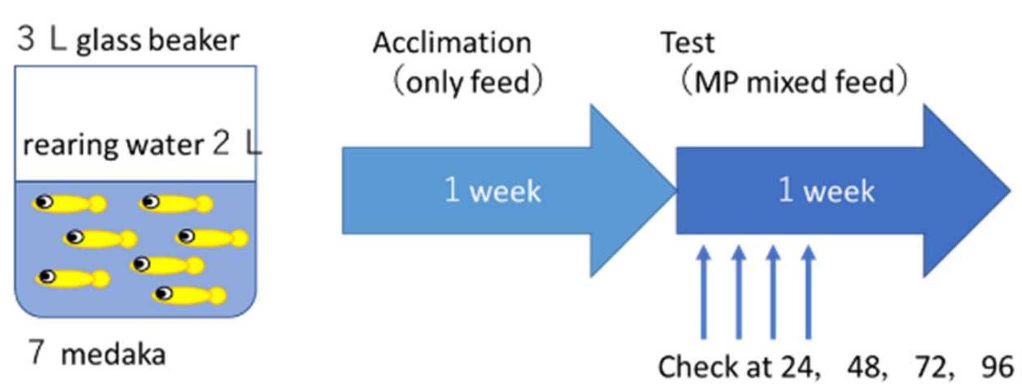
A protease and the polymer (Ny5i or Ny5i-11) were mixed in a buffer with the optimum pH. After the reaction, the solid was filtered off, and the resultant solution was analyzed by NMR spectroscopy to detect soluble monomers.

Results (NMR spectrum)



When Ny5i-11 was tested as a substrate, any peaks other than the enzyme were not observed.
When Ny5i was tested as a substrate, characteristic peaks were detected, which are being assigned.

Oral ingestion/acute toxicity study by medaka



Appropriate feeding rate : 2-3% of body weight (ca. 250mg) ≒ 7.3mg/medaka/day → 360mg/week

360 mg of feed mixed with 180 mg of MP →MP : 3.7mg/medaka/day

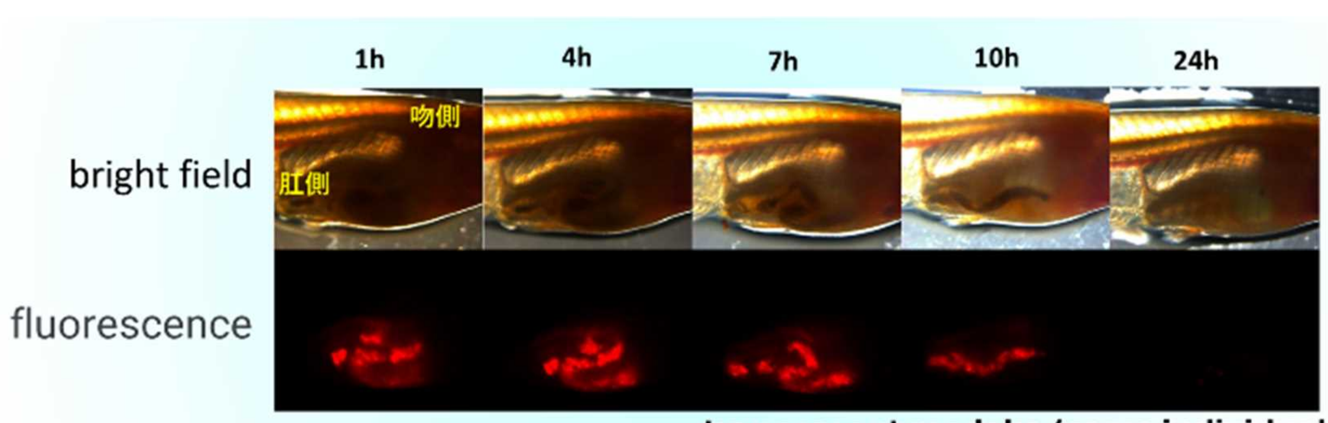
ON type

Kind of MP	acute toxicity
Ny6	No
Ny6-L	No
Ny6i(0.5%TiO_2)	No
Ny6i(1%TiO_2)	No
Ny6i(1.5-mer)	No
Ny6i 75%	No
Ny6i 11 50%	No
Ny6i 11 50% CuI $NaNbO_3$	No
Ny6i11-33	No
Ny5i11-33	No
Ny5i11-50	No
Ny5i11-75	No

OFF type

Kind of MP	acute toxicity
PS	No
PCL	No
PCL 5% Anatase	No
PCL 5% P25	No
PCL 5% gC_3N_4	No
PCL 5% heat treatment gC_3N_4	No

<Monitoring until the plastic powder is discharged after consumption>



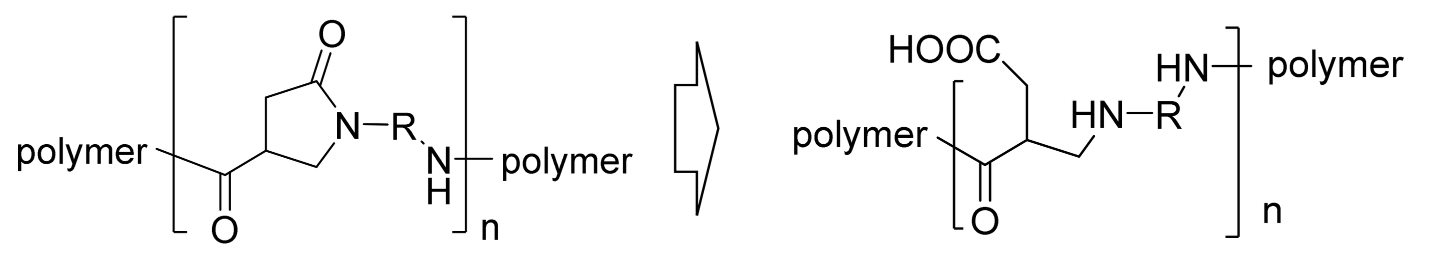
transparent medaka/same individual

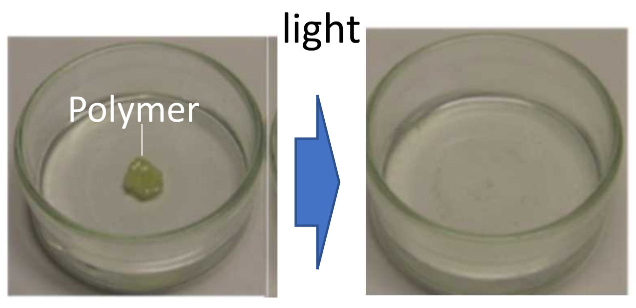
➡Excreted within 24 hours after oral vaccination

No. A-17-8E
PJ:Development of Photo-Switching Ocean-Degradable Plastics With Edibility
Theme: Accelerated Degradability Evaluation of Photo-Switching Biodegradable Plastics

Organization: Osaka Research Institute of Industrial Science and Technology（ORIST）
Contact: Akihiko Masui（aki@orist.jp）

ON type photo switching biodegradable plastic



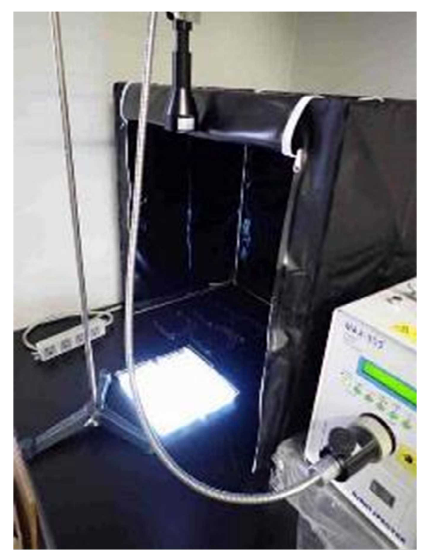


→When the photo switch is turned on, polymer becomes low molecular weight (solubilized) .
(under mercury lamp irradiation)

To evaluate the long-term degradability of samples, realistic and accelerated switching sample is required.
Obtain knowledge for long-term evaluation of biodegradable plastics by using accelerated switching sample.

ON type accelerated switching sample

- Polymer：bionylon(Ny5i11-33)
- Photocatalyst：Two inorganic types, One organic type
- Accelerated switching samples ：Ny5i11-33 + photocatalyst
- Light irradiation conditions ：
  Xenon lamp irradiation (8,000 lx, 0.45mW/cm^2) 60h/120h
  No Xenon lamp irradiation (dark condition) 60h/120h

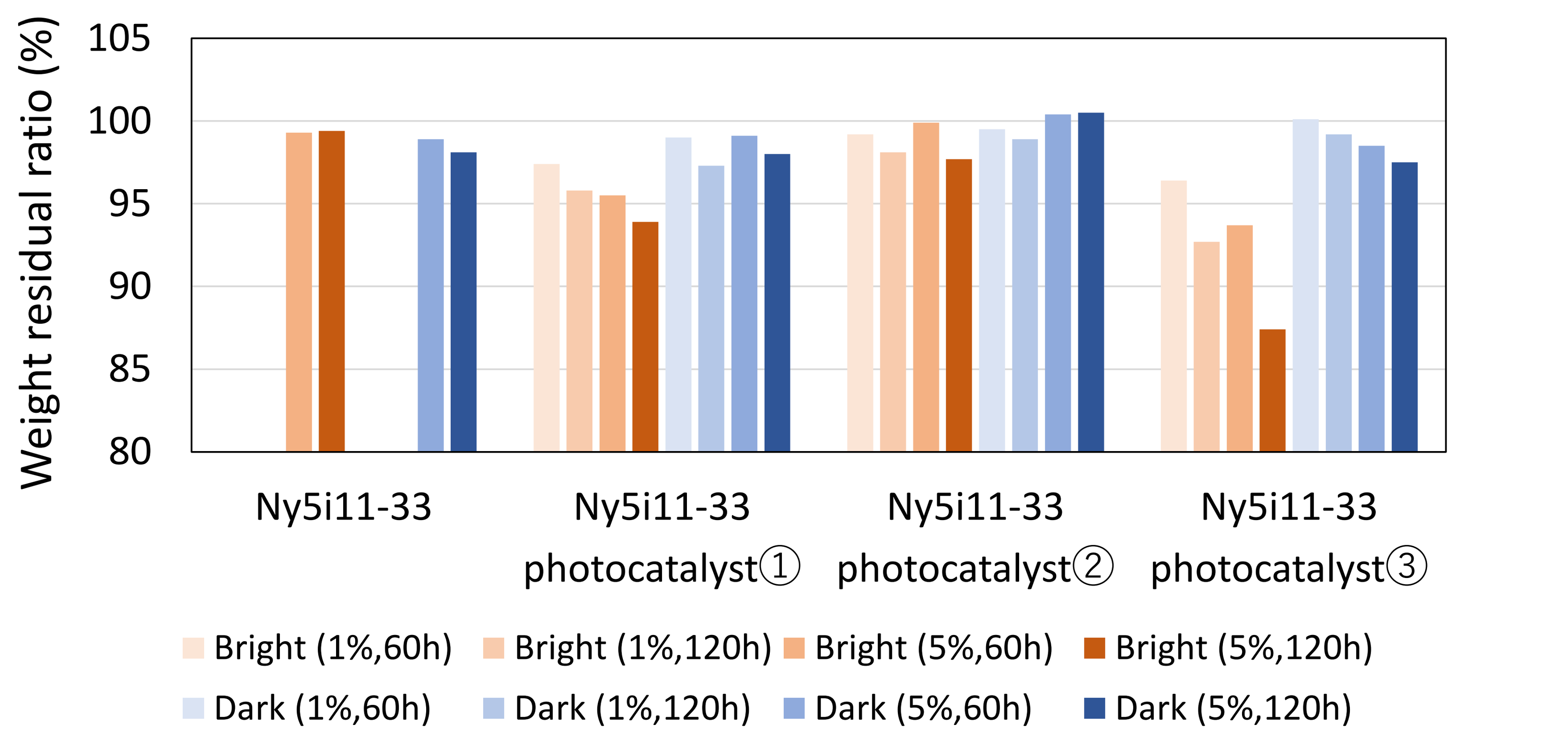


Xenon lamp irradiated sample
(immersed in artificial seawater)

Laboratory degradability test

Weight residual ratio after Xenon lamp irradiation (%)

	Bright condition				Dark condition			
	Photocatalyst 1% Irradiation 60h	1% 120h	5% 60h	5% 120h	1% 60h	1% 120h	5% 60h	5% 120h
Ny5i11-33			99.3	99.4			98.9	98.1
Ny5i11-33 photocatalyst①	97.4	95.8	95.5	93.9	99.0	97.3	99.1	98.0
Ny5i11-33 photocatalyst②	99.2	98.1	99.9	97.7	99.5	98.9	100.4	100.5
Ny5i11-33 photocatalyst③	96.4	92.7	93.7	87.4	100.1	99.2	98.5	97.5



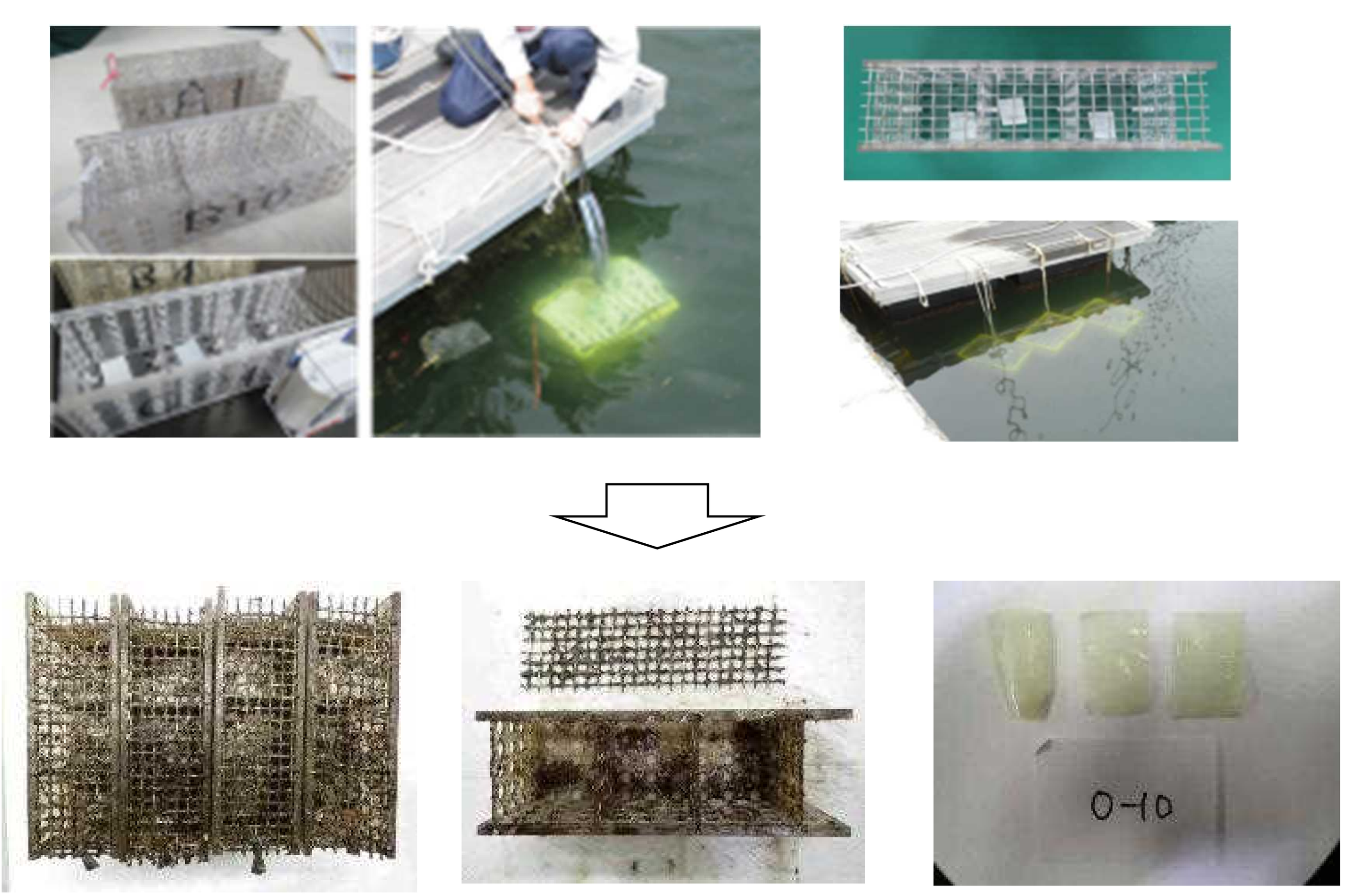
Ny (bionylon) only films do not decompose under Xenon lamp irradiation (8,000 lx, 0.45ｍW/cm^2).
Weight residual ratio decreased with the addition of photocatalyst.

Marine immersion test

ON type accelerated switching sample

Bionylon + photocatalyst 5%
↓
Cast mixing and filming
↙ ↘
No irradiation | Xenon lamp irradiation
↓ | ↓
Marine immersion (4w) | Marine immersion (4w)

Marine immersion test



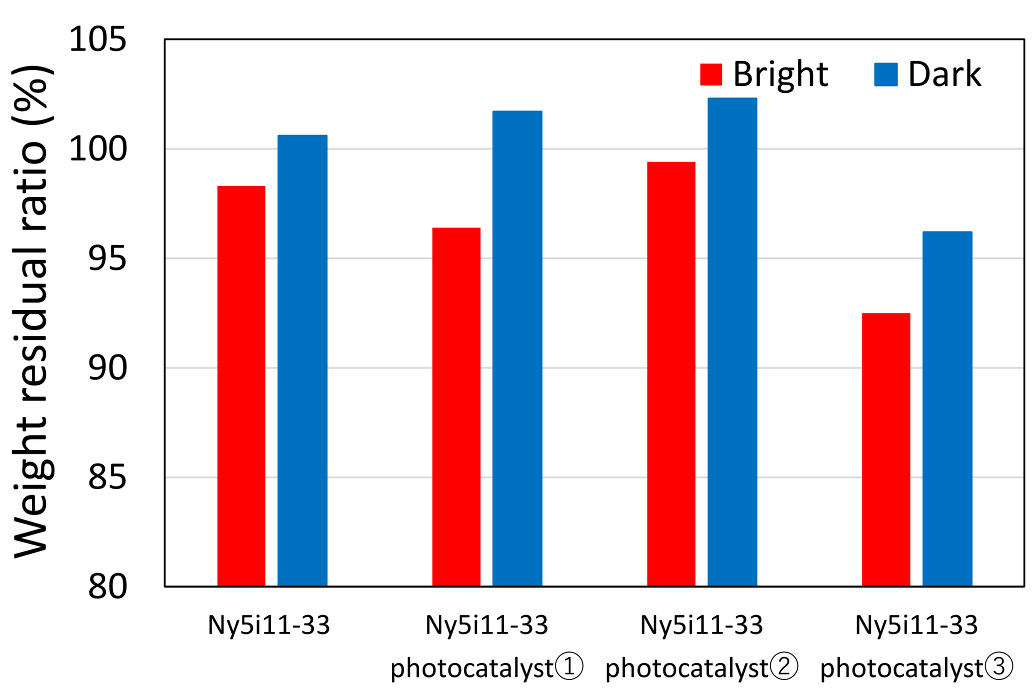
Weight residual ratio after marine immersion

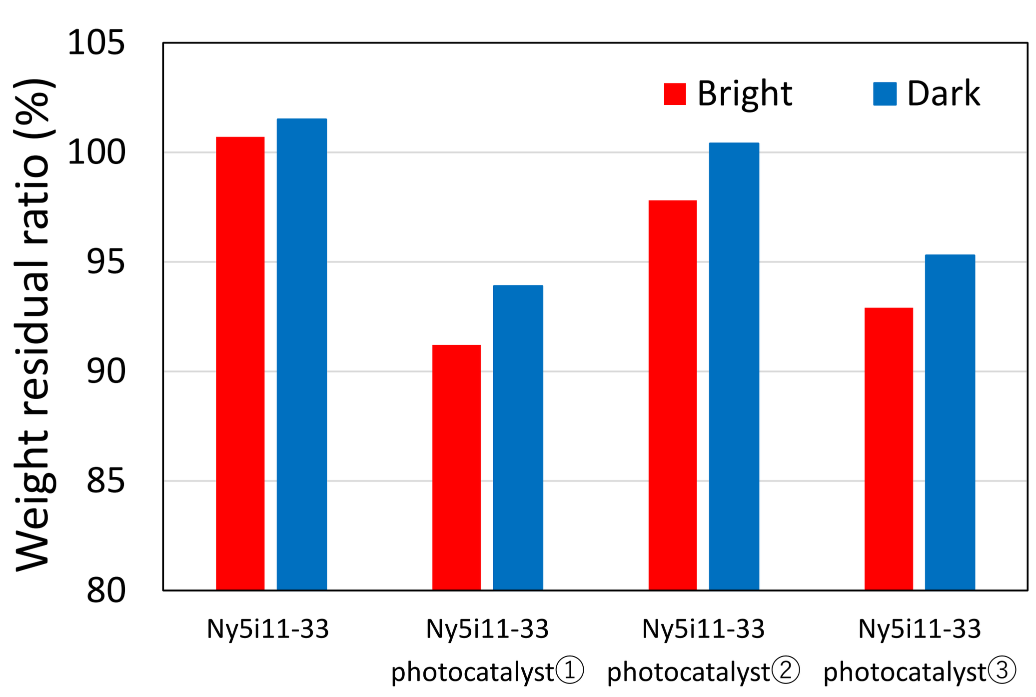
No irradiation +
Marine immersion (4w)

	Weight residual ratio (%)	
	Bright	Dark
Ny5i11-33	98.3	100.6
Ny5i11-33 photocatalyst①	96.4	101.7
Ny5i11-33 photocatalyst②	99.4	102.3
Ny5i11-33 photocatalyst③	92.5	96.2

Xenon irradiation (0.45ｍW/cm^2,120 h) +
Marine irradiation (4w)

	Weight residual ratio (%)	
	Bright	Dark
Ny5i11-33	100.7	101.5
Ny5i11-33 photocatalyst①	91.2	93.9
Ny5i11-33 photocatalyst②	97.8	100.4
Ny5i11-33 photocatalyst③	92.9	95.3





Ny (bionylon) only films, with or without prior Xenon lamp irradiation had no difference in weight residual ratio after marine immersion.
Photocatalyst composited films showed a decreasing effect on weight residual ratio by Xenon lamp irradiation.