우주원자력국제협력기반조성사업 한·영 공동연구

나노구조의 하이브리드 복합체를 이용한 고도 방사성 폐기물처리

Advanced Radioactive Waste Treatment using Nanostructured Hybrid Composites

2018. 10. 24 (수)

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목차

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연구배경

■ 우주원자력국제협력기반조성사업 한·영 공동연구 (2015-2018)

- **공동연구 수행자(기관)간 연구범위 및 역할분담 내용 (South Korea-UK)**


University of Central Lancashire, UK
(£ 183,809/year)

Nanostructured Composites' Application

Radioactive Waste Treatment using Hybrid Nanostructured Composites

Hybrid Nanostructured Composites

Ligands for Fission Production and Actinides


University of Reading, UK
(£ 203,604/year)

Kyungpook National University & Chosun University
South Korea


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Professor Gary Bond (UCLan) (GB) (Project P-I)

Professor Gary Bond is Head of the Centre for Materials Science and has been a visiting professor at the CNRS (Centre National de la Recherche Scientifique) Laboratory for Catalysis and Spectroscopy in Caen (2007). His research is focused on interfacial phenomena and he has substantial experience in the analysis and characterisation of solid materials. He has particular expertise in the application of carbon substrates as adsorbents, supports and catalysts. He has secured over £2M research funding from industry and government agencies including the nuclear industry. His current portfolio of projects includes two projects which are focused on the decontamination of irradiated graphite. He has published over 40 refereed publications, contributed chapters to 3 books and is the co-inventor on one patent.

Professor Harry Eccles (UCLan) (HE) (Project Co-I)

Professor Harry Eccles is the Professor in Nuclear Materials (part-time). He is a chemist by profession, with 35 years' research and development experience in the nuclear industry. He is recognised internationally for his separation science expertise which includes ion exchange, solvent extraction and biosorption. In the early 1970s he developed a chelate ion exchange material for the recovery of uranium from sea water. On joining BNFL in the mid 1970s he was involved in the development of U and Pu purification flowsheets for the THORP PUREX process.

Since joining UCLan he has been developing new separation processes for reprocessing irradiated fuel and for treatment of waste liquors. Also developing techniques for the decontamination of irradiated graphite and investigating the mobility of fission products within cement paste and their rate of diffusion from cement paste.

He is the first recipient of the NNL's Life-Time achievement award.

Professor Laurence Harwood (University of Reading and his team) (LH) (Project Co-I)

Professor Laurence Harwood is a chemist by profession and Professor of Organic Chemistry at Reading since 1996, Regional Editor of Synlett since 2001 and Chief Scienitific Officer of TechnoPep since 2010. He has been working in the area of reprocessing since 2004 and has been a member of the PARTNEW, ACCEPT and SACSESS (current) EU consortia, MBase and PACIFIC EPSRC consortia, with total research income for nuclear associated projects exceeding £800K. The CyMe4BTP, BTBP and PTPhen ligands developed at Reading have become industry standards for selective minor actinide extraction from high-level waste (HLW). The recent immobilization of these ligands onto magnetic nanoparticles has provided a revolutionary process applicable to soil remediation and also to clean-up of low level liquid waste and sludges in storage ponds. He has published more than 140 refereed research publications, 10 patents, 9 review chapters and 4 books.

연구배경

Prof. Laurence Harwood, University of Reading

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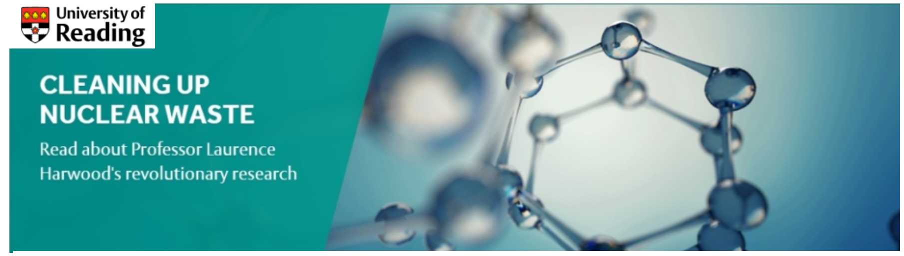


PROFESSOR LAURENCE HARWOOD: CLEANING UP NUCLEAR WASTE

Laurence Harwood, Professor of Organic Chemistry, spent decades as a pharmaceutical chemist, until a colleague asked him to get involved in a project with the nuclear industry. He applied the same structure-activity relationship studies he was using in drug discovery to helping develop molecules that could be used to clean up nuclear waste, and his research took on a whole new set of challenges.

CLEANING UP NUCLEAR WASTE

While nuclear energy leaves no carbon footprint to speak of, it does leave a legacy of hazardous waste - spent nuclear fuel. Most countries just store it with the intention eventually of burying it deep underground and this is leading to a lot of material worldwide with storage times estimated at 300,000 years. There are, however, ways to clean up and reuse most of that spent fuel.

> "If you start with 500 kg of nuclear waste, 480 kg of that is uranium and 5 kg is plutonium; these can be separated using current technology, refabricated as a mixed oxide fuel and reused. This leaves only 15 kg of waste that now only needs to be stored for 10,000 years. But the difference between 300,000 years and 10,000 years is like throwing an egg off a 30 storey building or a 1 storey building - the result is the same."

연구추진 일정

■ 한국측 연구추진 일정

Activity	Lead Organisation	Quarter No 1	2	3	4	5	6	7	8	9	10	11	12
1 Synthesis and characterization of magnetic nanocomposites with Prussian blue	KNU/ Chosun U.	Blue	Blue	Blue	Blue								
2 First meeting with UK partners to agree first year work programme	KNU/ Chosun U.		Blue										
3 Evaluation of hybrid nanocomposites' adsorption capacity for radioactive wastes	KNU/ Chosun U.		Blue	Blue	Blue								
4 Effects of background species on adsorption performance	KNU/ Chosun U.				Green	Green	Green	Green					
5 Comparative evaluation of lab-scale process performance by adsorption operating conditions	KNU/ Chosun U.					Green	Green	Green	Green	Green	Green		
6 Establishment of basic operating conditions and process design	KNU/ Chosun U.							Green	Green				
7 Performance evaluation of hybrid nanostructured composites for field application	KNU/ Chosun U.									Yellow	Yellow	Yellow	Yellow
8 Development of an optimal operating system for field application	KNU/ Chosun U.									Yellow	Yellow	Yellow	Yellow
9 Compilation of final report	KNU/ Chosun U.												Yellow
Deliverables/milestones	KNU/ Chosun U.		↑ M1 D1		↑ M2 D2	↑ D3			↑ M3 D4 D5				↑ M4 D6 D7

M1- Agreement of radionuclides to be evaluated.
M2 - Functional magnetic nanoparticles produced
M3 – Process design for radioactive liquid waste treatment using nanostructured composites produced
M4 - Optimum process system for radioactive liquid waste using functional nanostructured composites developed
D1 - First international meeting
D2 - First progress report.
D3 - Second international meeting
D4 - Second progress report.
D5 -Third international project meeting
D6 - Final progress report
D7 - Fourth international project meeting.

■ 영국측 연구추진 일정

Activity	Lead Organisation	Quarter No 1	2	3	4	5	6	7	8	9	10	11	12
1 Identify candidate substrates for NaSHCs and preparative approach	UCLan/ UoReading	Blue											
2 First meeting with Korean partners to agree first 6 months work programme	UCLan/ UoReading	Blue	Blue										
3 Selection of PDRA	UCLan/ UoReading		Green	Green	Green								
4 Preparation of and characterisation of NaSHCs for various fission products (FPs)	UCLan			Red	Red	Red	Red	Red					
5 Preparation of and characterisation of NaSHCs for actinides (A).	UoReading			Green	Green	Green	Green	Green					
6 Measurement of affinities and capacities for FPs	UCLan					Red	Red	Red	Red	Red			
7 Measurement of affinities and capacities for A	UoReading						Green	Green	Green	Green			
8 Comparison of data from UK teams.	UCLan/ UoReading			Blue	Blue	Blue	Blue	Blue	Blue	Blue			
9 Compilation of final report	UCLan/ UoReading										Blue		
Deliverables/milestones	UCLan/ UoReading	↑ M1 2 D1	↑ M3	↑ D2	↑ M4 D3	↑ M5 6	↑ D4	↑ D5	↑ M7 D6		↑ M8 M9		↑ M10 D7 D8

M1- Short list of candidate materials produced
M2- Agreement of radionuclides to be evaluated.
M3 - PDRAs identified
M4 - First candidate NaSHCs prepared.
M5 - Characterisation of NaSHCs..
M6 - Optimisation of preparative route completed
M7- Confirmation of affinities and capacities data
M8 - Comparison of data from UK partners
M9 - Draft report prepared.
M10 - Discussion on further/continued collaboration
D1 - First international meeeting
D2 - First progress report.
D3 - Second international meeting
D4 - Progress report.
D5 -Third international project meeting
D6 -Third progress report.
D7 - Final progress report
D8 - Fourth international project meeting.

연구목표

▪ 나노구조의 하이브리드 복합체를 이용한 고도 방사성 폐기물처리

1차년도 (2015)

자성 프러시안블루 나노복합체의 합성 및 방사성 핵종의 선택적 분리 흡착 메커니즘 규명

- 헥사시아노철산염을 이용한 자성을 띠는 하이브리드 나노복합체 합성
- XRD, TGA, FTIR, SEM, TEM 등에 기반한 자성 하이브리드 나노복합체의 물리화학적 특성 규명
- 합성된 하이브리드 나노복합체의 방사성 핵종에 대한 기초 흡착성능 평가
- 자성 하이브리드 나노복합체의 선택적 분리 흡착 메카니즘 규명

2차년도 (2016)

하이브리드 나노복합체를 이용한 방사성 액상 폐기물 처리 공정 설계

- 배경물질의 유무와 종류에 따른 운전 성능 비교 평가
- 흡착 운전 조건, 체류시간, 흡착제 투입량에 따른 운전 성능 비교 평가
- 자성 하이브리드 나노복합체의 흡착 거동, 경쟁 흡착, 선택성 평가
- 기초 운전조건 확립 및 반응기 설계

3차년도 (2017)

선택성을 향상시킨 리간드 합성 및 흡착 성능 평가

- 자성 하이브리드 나노복합체에 기반한 컬럼 반응기 연속운전 실험 및 공정 최적화
- 실제 조건과 유사한 환경(공통이온 및 pH 등)에서의 방사성핵종 제거 실험 및 성능평가
- 선택성을 향상시킨 리간드 합성 및 흡착 성능 평가

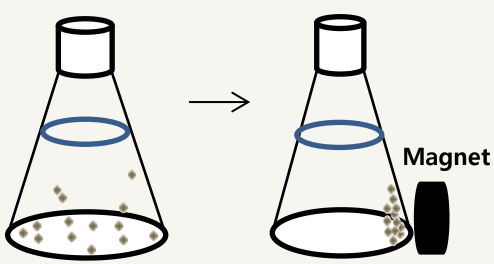
연구추진 전략

Adsorbents for radionuclides (Cs^{+}, Sr^{2+}, Co^{2+} and I^{-})

Continuous flow stirred-tank reactors (CSTRs)

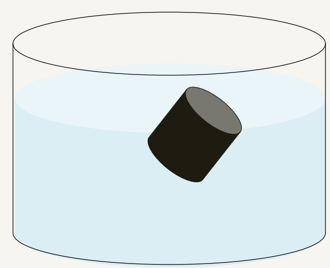
Continuous fixed-bed columns (packed-bed)

01 Powder type



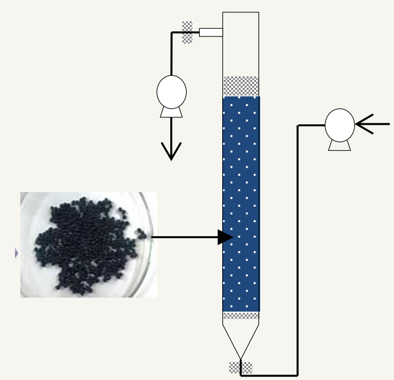
Magnetite recovery process

03 3D aerogel type



Low density

02 bead type

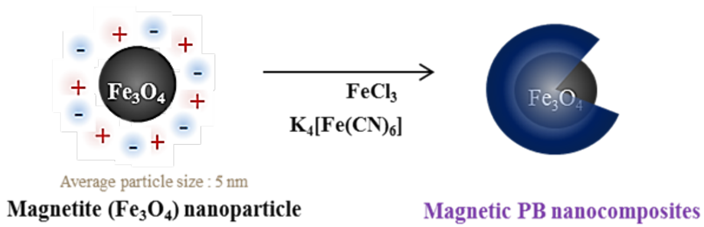


Continuous fixed-bed column

Separation Processes

연구목표 | **자성 프러시안블루 나노복합체의 합성 및 방사성 핵종의 선택적 분리 흡착 메커니즘 규명**

■ 헥사시아노철산염을 이용한 자성을 띠는 하이브리드 나노복합체 합성



Adsorbent	Average particle size (nm)	Surface area (m^2/g)	Pore size (nm)	Pore volume (cm^3/g)
Mag-PB nanocomposite	13.6	322.19	2.579	0.119

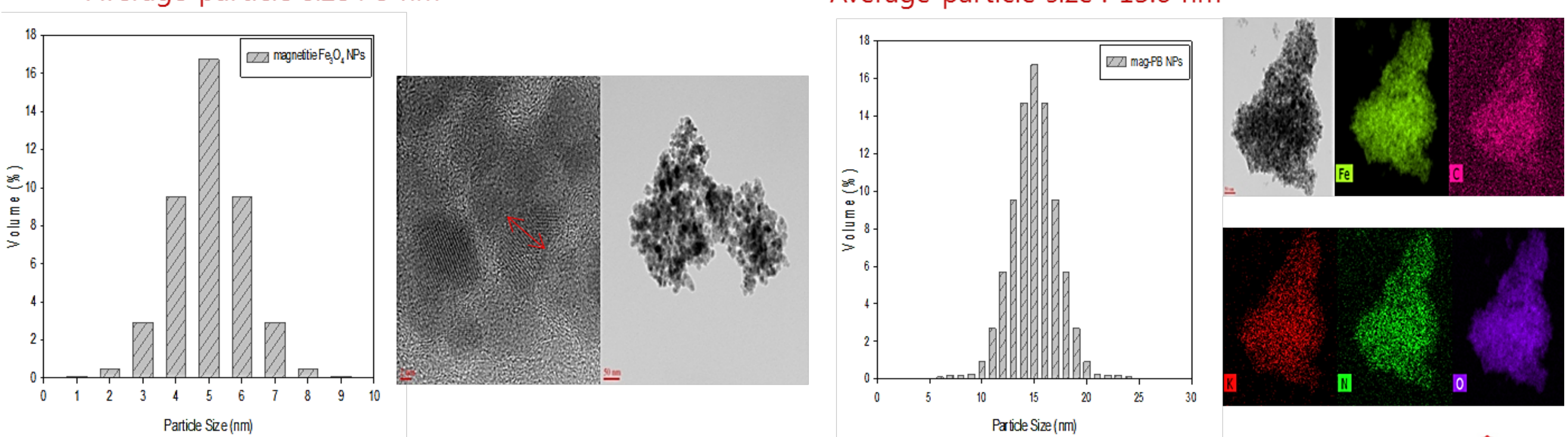
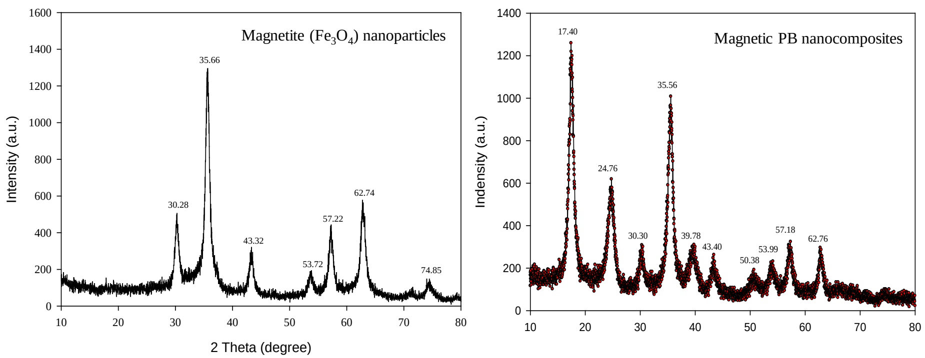


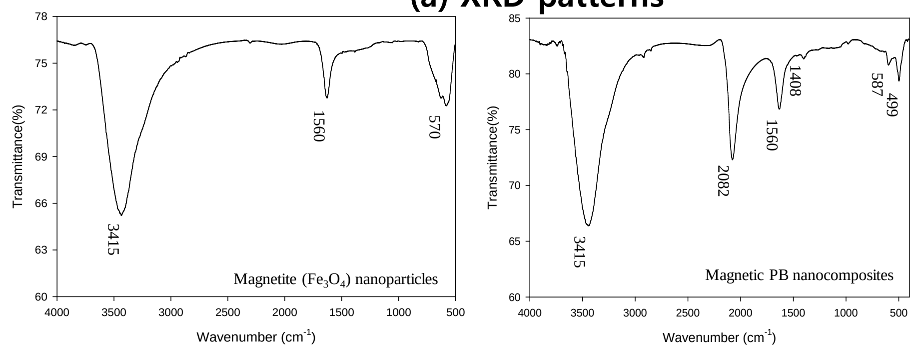
Fig. 1-1. PSA and FE-TEM images of magnetic PB nanocomposites.

1차년도 연구결과

■ 자성 하이브리드 나노복합체의 물리화학적 특성 규명



(a) XRD patterns



(b) FT-IR spectra

Fig. 1-2. XRD and FT-IR images of magnetite(Fe_3O_4) and magnetic PB nanocomposites.

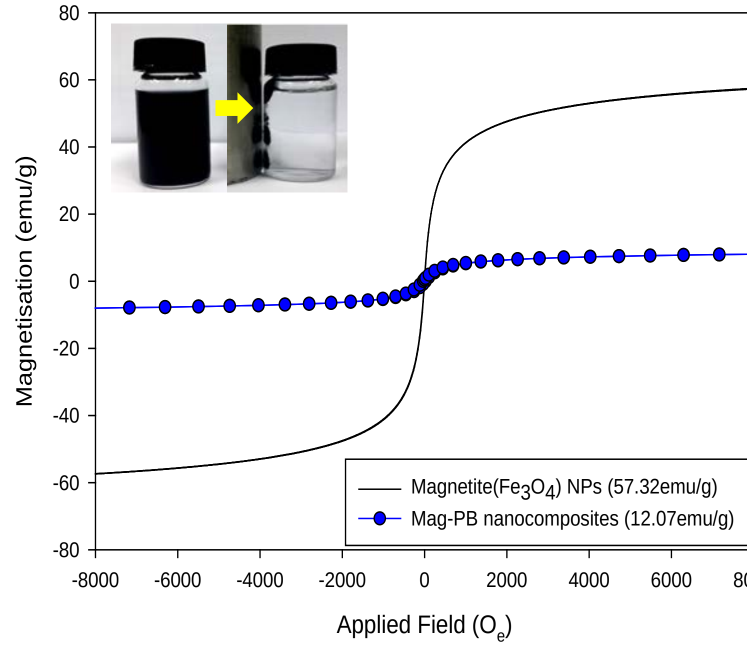


Fig. 1-3. MPMS analysis of magnetite (Fe_3O_4) NPs and magnetic PB nanocomposites.

1차년도 연구결과

■ 합성된 하이브리드 나노복합체의 방사성 핵종에 대한 기초 흡착성능 평가

▪ Adsorption kinetics

Table 1-1. Rate constants and correlation coefficients of pseudo-first-order and pseudo-second-order kinetic models at an initial Cs^+ concentration of 1 mM (initial pH = 7 and 120 rpm).

Temperature (°C)	$q_{e,exp}$ (mg/g)	First-order kinetics			Second-order kinetics		
		K_1	q_e	r^2	k_2	q_e	r^2
30	34.59	0.039	35.14	0.989	0.002	36.42	0.999
20	39.71	0.035	37.05	0.980	0.002	38.60	0.991
10	55.12	0.017	49.87	0.959	0.0003	54.41	0.981

- Rate limiting step: chemical interaction between the functional groups of adsorbent and cesium ions

▪ Adsorption isotherm models

- Equilibrium distribution of Cs^+ between the adsorbent and liquid phase

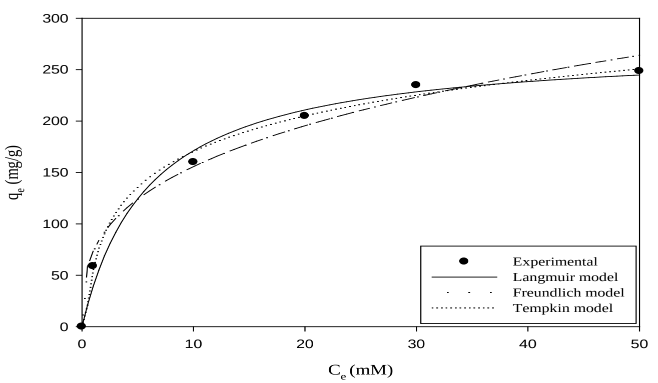


Fig. 1-4. Nonlinear isotherm models for the removal of cesium ions at 10°C.

Table 1-2. Comparison of Non-linearized Isotherm Models for the Adsorption of Cs^+ onto Magnetic PB Nanocomposites. (initial pH = 7.0, 120 rpm)

Isotherm models	Calculated isotherm parameters	χ^2	APE	r^2
10℃				
Langmuir	q_{max} = 294.39, K_L = 0.18	4.19	5.71	0.978
Freundlich	K_F = 77.39, n = 2.98	12.58	10.70	0.958
Tempkin	A = 55.82, b = 2.70	1.03	1.99	0.992

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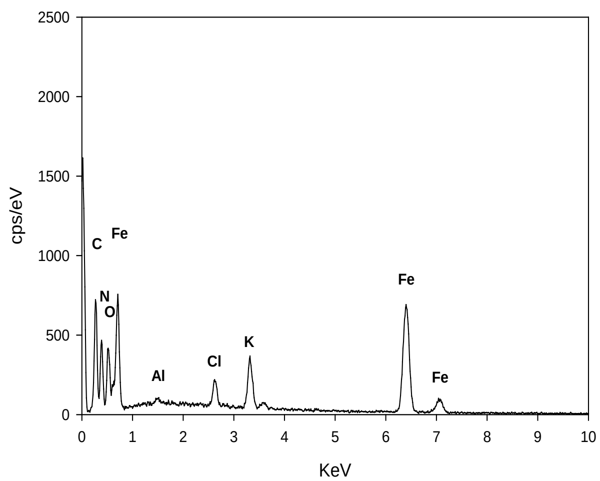
1차년도 연구결과

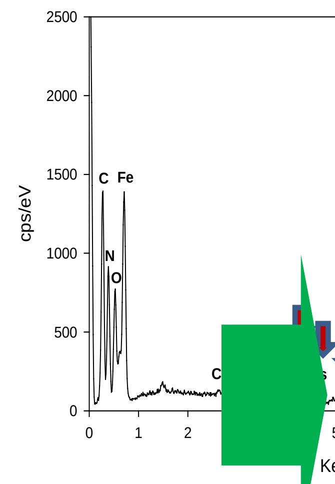
■ 자성 하이브리드 나노복합체의 선택적 분리 흡착 메카니즘 규명

Cesium ions captured by the cage of the PB lattice structure

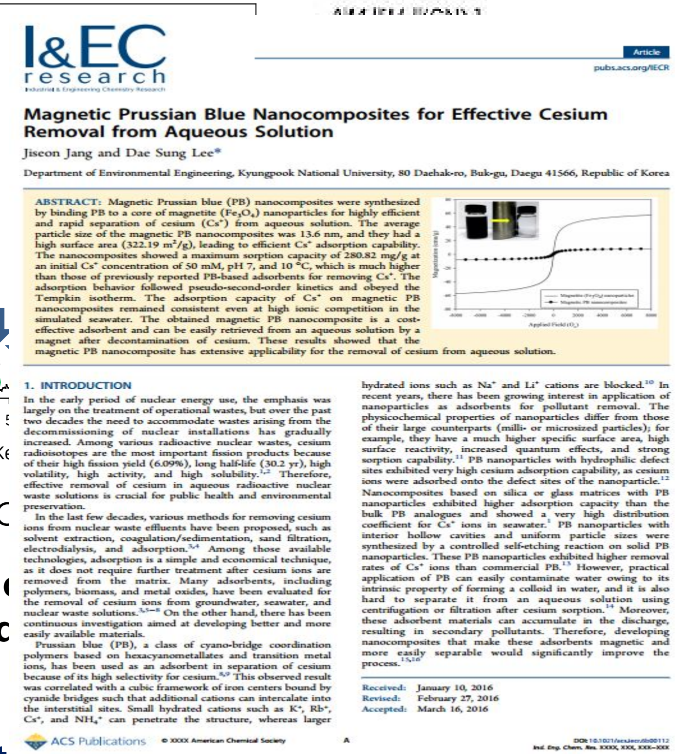
1:1:1-type (F43m)



(a) Before Cs^+ adsorption



(b) After C



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Magnetic Prussian Blue Nanocomposites for Effective Cesium Removal from Aqueous Solution

Jiseon Jang and Dae Sung Lee*

Department of Environmental Engineering, Kyungpook National University, 80 Daehak-ro, Buk-gu, Daegu 41566, Republic of Korea

ABSTRACT: Magnetic Prussian blue (PB) nanocomposites were synthesized by binding PB to a core of magnetite (Fe_3O_4) nanoparticles for highly efficient and rapid separation of cesium (Cs^+) from aqueous solution. The average particle size of the magnetic PB nanocomposites was 13.6 nm, and they had a high surface area (322.19 m^2/g), leading to efficient Cs^+ adsorption capability. The nanocomposites showed a maximum sorption capacity of 280.82 mg/g at an initial Cs^+ concentration of 50 mM, pH 7, and 10 °C, which is much higher than those of previously reported PB-based adsorbents for removing Cs^+. The adsorption behavior followed pseudo-second-order kinetics and obeyed the Tempkin isotherm. The adsorption capacity of Cs^+ on magnetic PB nanocomposites remained consistent even at high ionic competition in the simulated seawater. The obtained magnetic PB nanocomposite is a cost-effective adsorbent and can be easily retrieved from an aqueous solution by a magnet after decontamination of cesium. These results showed that the magnetic PB nanocomposite has extensive applicability for the removal of cesium from aqueous solution.

1. INTRODUCTION

In the early period of nuclear energy use, the emphasis was largely on the treatment of operational wastes, but over the past two decades the need to accommodate wastes arising from the decommissioning of nuclear installations has gradually increased. Among various radioactive nuclear wastes, cesium radioisotopes are the most important fission products because of their high fission yield (6.09%), long half-life (30.2 yr), high volatility, high activity, and high solubility.[1,2] Therefore, effective removal of cesium in aqueous radioactive nuclear waste solutions is crucial for public health and environmental preservation.

In the last few decades, various methods for removing cesium ions from nuclear waste effluents have been proposed, such as solvent extraction, coagulation/sedimentation, sand filtration, electrodialysis, and adsorption.[3,4] Among those available technologies, adsorption is a simple and economical technique, as it does not require further treatment after cesium ions are removed from the matrix. Many adsorbents, including polymers, biomass, and metal oxides, have been evaluated for the removal of cesium ions from groundwater, seawater, and nuclear waste solutions.[3,5−8] On the other hand, there has been continuous investigation aimed at developing better and more easily available materials.

Prussian blue (PB), a class of cyano-bridge coordination polymers based on hexacyanometallates and transition metal ions, has been used as an adsorbent in separation of cesium because of its high selectivity for cesium.[8,9] This observed result was correlated with a cubic framework of iron centers bound by cyanide bridges such that additional cations can intercalate into the interstitial sites. Small hydrated cations such as K^+, Rb^+, Cs^+, and NH_4^+ can penetrate the structure, whereas larger hydrated ions such as Na^+ and Li^+ cations are blocked.[10] In recent years, there has been growing interest in application of nanoparticles as adsorbents for pollutant removal. The physicochemical properties of nanoparticles differ from those of their large counterparts (milli- or microsized particles); for example, they have a much higher specific surface area, high surface reactivity, increased quantum effects, and strong sorption capability.[11] PB nanoparticles with hydrophilic defect sites exhibited very high cesium adsorption capability, as cesium ions were adsorbed onto the defect sites of the nanoparticle.[12] Nanocomposites based on silica or glass matrices with PB nanoparticles exhibited higher adsorption capacity than the bulk PB analogues and showed a very high distribution coefficient for Cs^+ ions in seawater.[1] PB nanoparticles with interior hollow cavities and uniform particle sizes were synthesized by a controlled self-etching reaction on solid PB nanoparticles. These PB nanoparticles exhibited higher removal rates of Cs^+ ions than commercial PB.[13] However, practical application of PB can easily contaminate water owing to its intrinsic property of forming a colloid in water, and it is also hard to separate it from an aqueous solution using centrifugation or filtration after cesium sorption.[14] Moreover, these adsorbent materials can accumulate in the discharge, resulting in secondary pollutants. Therefore, developing nanocomposites that make these adsorbents magnetic and more easily separable would significantly improve the process.[15,16]

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Ind. Eng. Chem. Res. XXXX, XXX, XXX−XXX

Fig. 1-5. SEM-EDX analyses of magnetic PB nan
(a) before Cs^+ adsorption and (b) after Cs^+ ac

- **Cesium ions are adsorbed into the crystal cage structure of metal hexacyanoferrate as an ion pair with a cation**

  vs

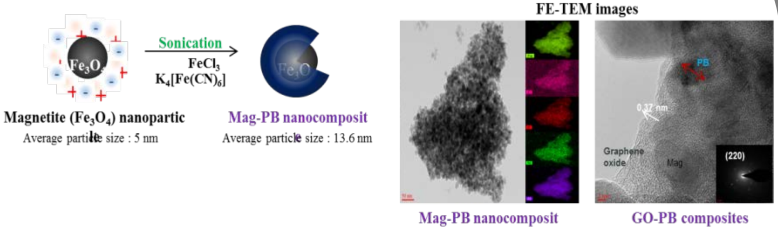
- **Cesium ions are exchanged with potassium ions**

2차년도 연구결과

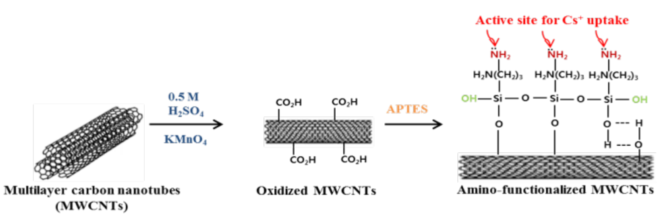
연구목표 하이브리드 나노복합체를 이용한 방사성 액상 폐기물 처리 공정 설계

Removal of Radioactive Cesium

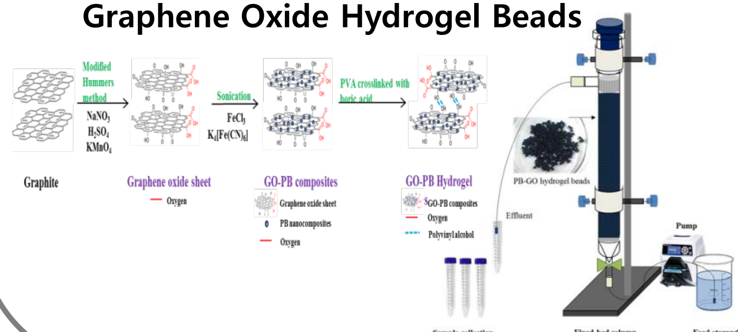
- Magnetic Prussian Blue Nanocomposites



- Amino-functionalized MWCNTs

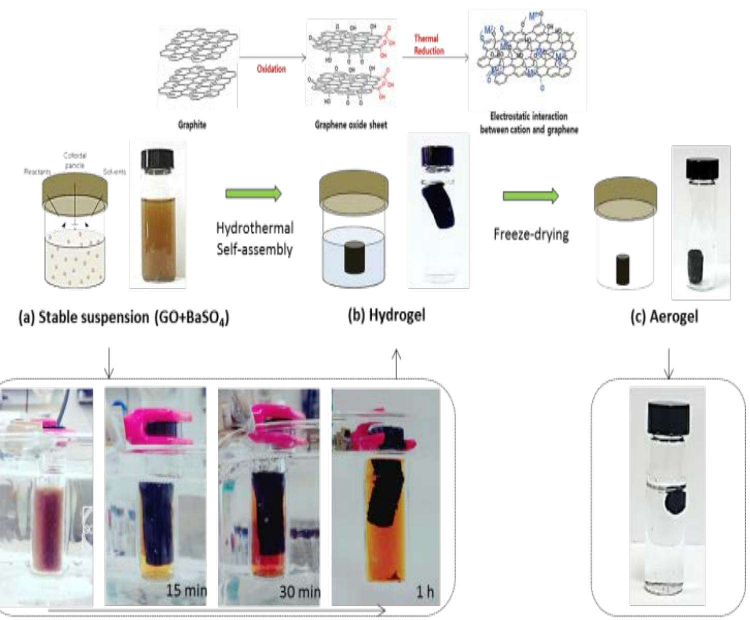


- PVA-alginate Encapsulated Prussian Blue-Graphene Oxide Hydrogel Beads



Removal of Radioactive Strontium

- 3D Barium Sulfate-Anchored Reduced Graphene Oxide Aerogel



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2차년도 연구결과

■ 기초 운전조건 확립 및 반응기 설계

- PVA-alginate encapsulated graphene oxide/PB hydrogel beads.

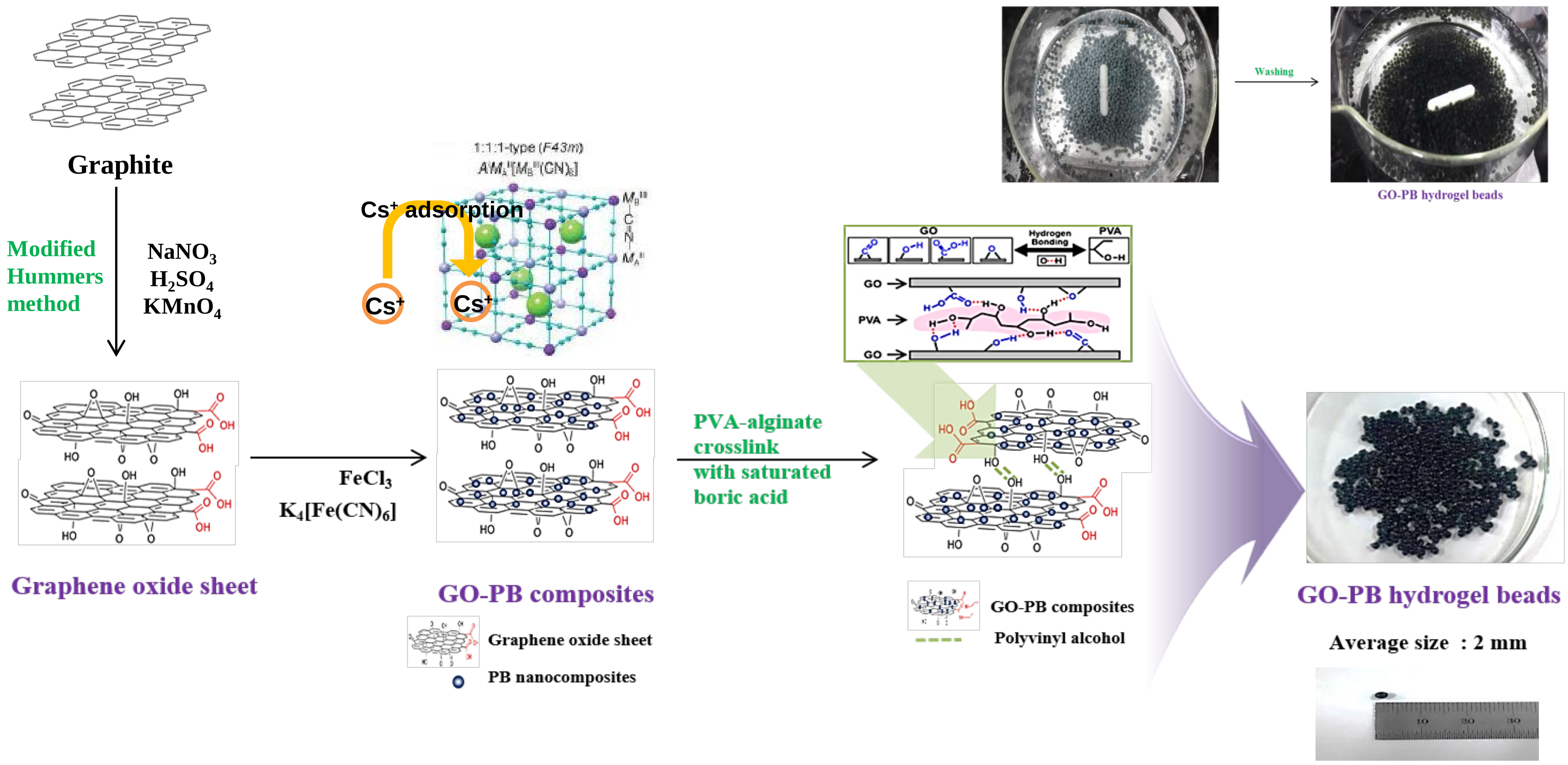
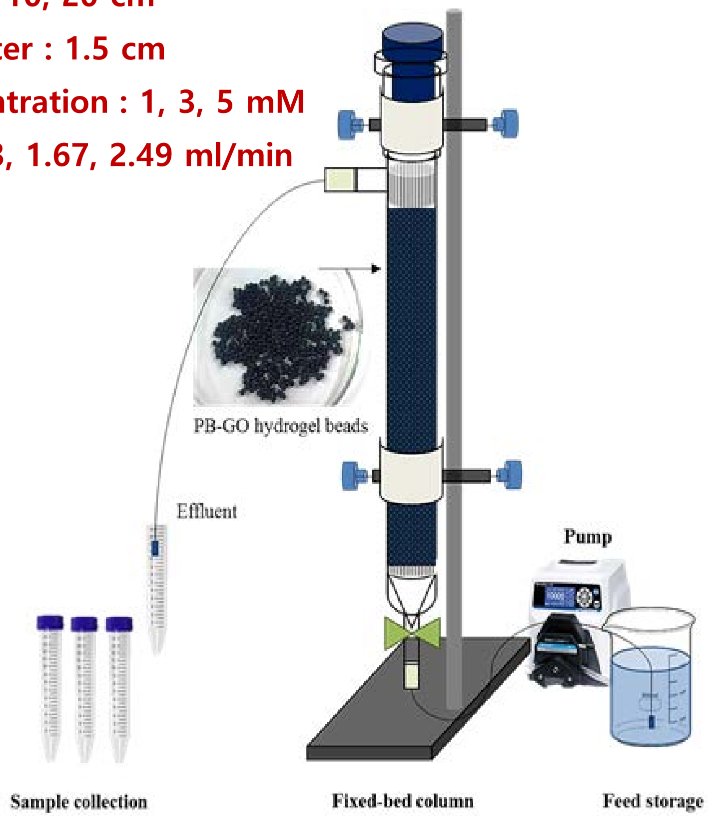


Fig. 2-3. Schematic diagram of PVA-alginate encapsulated graphene oxide/PB hydrogel beads.

2차년도 연구결과

■ 기초 운전조건 확립 및 반응기 설계

- Bed height : 5, 10, 20 cm
- Internal diameter : 1.5 cm
- Influent concentration : 1, 3, 5 mM
- Flow rate : 0.83, 1.67, 2.49 ml/min



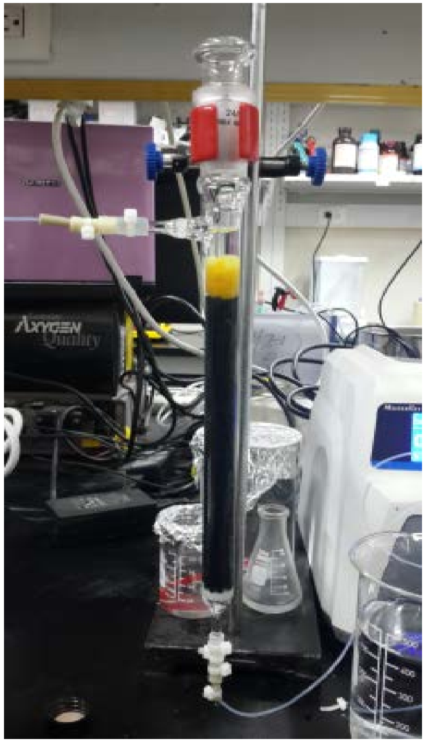
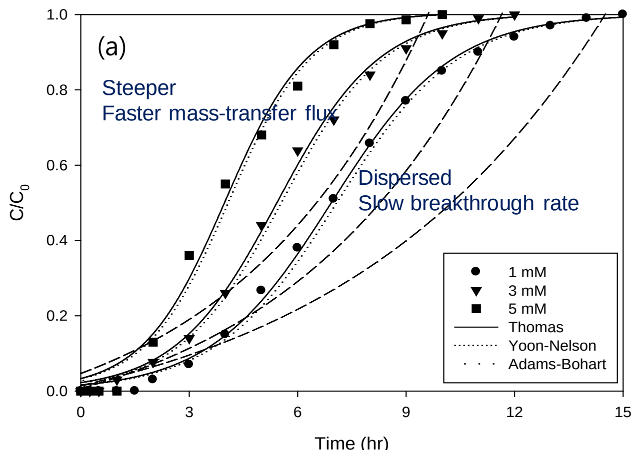


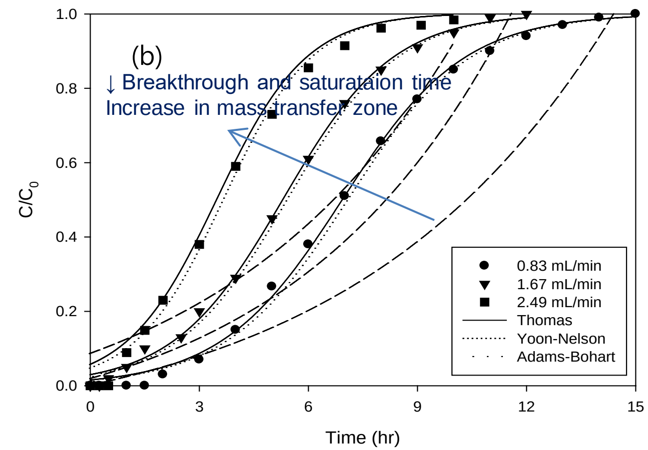
Fig. 2-4. Schematic diagram of continuous fixed-bed adsorption system.

2차년도 연구결과

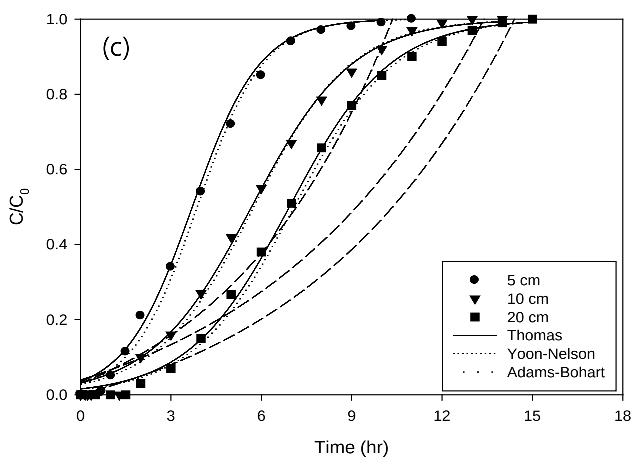
■ 기초 운전조건 확립 및 반응기 설계



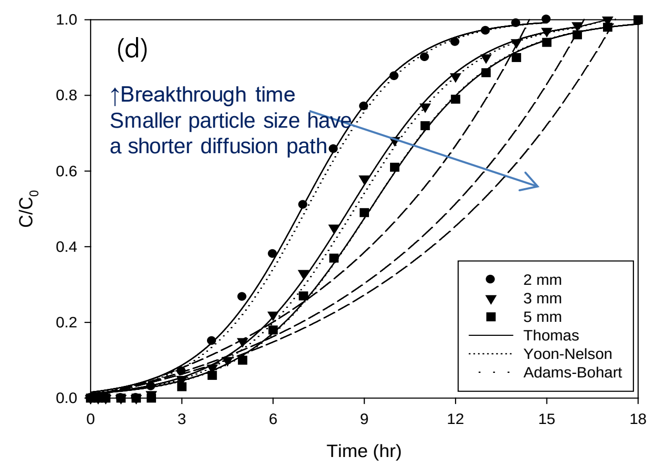
Initial C: 1 mM, Flow rate: 0.83 ml/min, Bed high: 20 cm



Initial C: 1 mM, Flow rate: 0.83 ml/min, bead size: 2mm



Initial C: 1 mM, Flow rate: 0.83 ml/min, bead size: 2mm



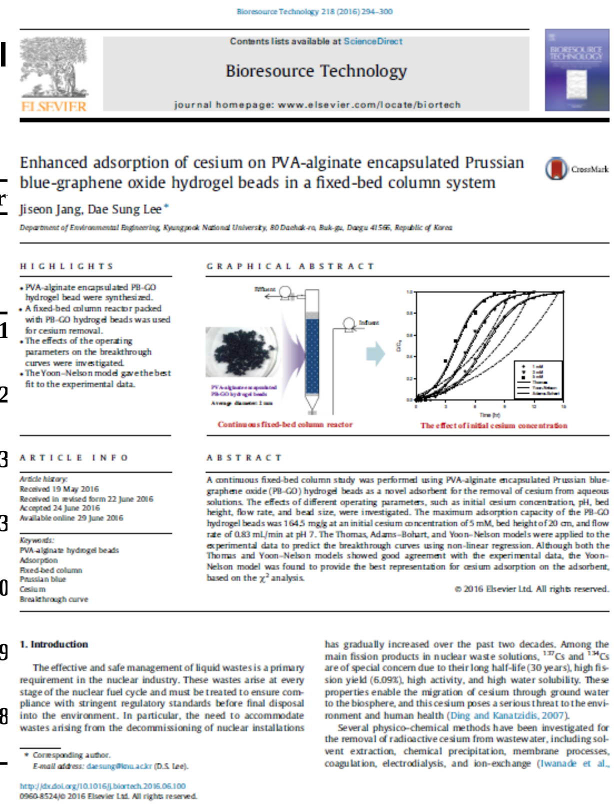
Initial C: 1 mM, Flow rate: 0.83 ml/min, Bed high: 20 cm

Fig. 2-5. The Influence of operational parameters on the cesium adsorption breakthrough curves using PB-GO hydrogel beads: (a) influent cesium concentrations, (b) flow rate, (c) bed height, and (d) adsorbent size.

■ 기초 운전조건 확립 및 반응기 설계

Table 2-1. Parameters (a) Thomas, (b) Adams-Bohart, and (c) Yoon and Nel... using nonlinear regression model.

C_0 (mM)	F (mL/min)	H (cm)	(a) Thomas model k_{TH} (L/mg/h)	q_{emax} (mg/g)	χ^2	APE	R^2	(b) Adams-Bohar... k_{AB} (L/mg/h)	N_0 (mg/L)	χ^2
1	0.83	20	0.59	165.10	0.08	24.42	0.998	0.14	[illegible]	1.61
3	0.83	20	0.43	167.22	0.14	31.23	0.997	0.12	[illegible]	1.72
5	0.83	20	0.36	169.42	0.20	45.50	0.991	0.11	19.56	1.83
1	0.83	5	0.64	159.15	0.06	14.36	0.997	0.10	15.25	1.53
1	0.83	10	0.62	161.32	0.10	15.76	0.993	0.13	16.21	1.90
1	1.67	20	0.65	158.24	0.09	21.32	0.996	0.15	15.87	2.79
1	2.49	20	0.71	156.88	0.22	25.78	0.995	0.17	13.93	1.08



Bioresource Technology 218 (2016) 294–300

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

Enhanced adsorption of cesium on PVA-alginate encapsulated Prussian blue-graphene oxide hydrogel beads in a fixed-bed column system

Jiseon Jang, Dae Sung Lee*

Department of Environmental Engineering, Kyungpook National University, 80 Daehak-ro, Buk-gu, Daegu 41566, Republic of Korea

H I G H L I G H T S

- PVA-alginate encapsulated PB-GO hydrogel bead were synthesized.
- A fixed-bed column reactor packed with PB-GO hydrogel beads was used for cesium removal.
- The effects of the operating parameters on the breakthrough curves were investigated.
- The Yoon–Nelson model gave the best fit to the experimental data.

G R A P H I C A L A B S T R A C T

Continuous fixed-bed column reactor

The effect of initial cesium concentration

A R T I C L E I N F O

Article history:
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Available online 29 June 2016

Keywords:
PVA-alginate hydrogel beads
Adsorption
Fixed-bed column
Prussian blue
Cesium
Breakthrough curve

A B S T R A C T

A continuous fixed-bed column study was performed using PVA-alginate encapsulated Prussian blue-graphene oxide (PB-GO) hydrogel beads as a novel adsorbent for the removal of cesium from aqueous solutions. The effects of different operating parameters, such as initial cesium concentration, pH, bed height, flow rate, and bead size, were investigated. The maximum adsorption capacity of the PB-GO hydrogel beads was 164.5 mg/g at an initial cesium concentration of 5 mM, bed height of 20 cm, and flow rate of 0.83 mL/min at pH 7. The Thomas, Adams–Bohart, and Yoon–Nelson models were applied to the experimental data to predict the breakthrough curves using non-linear regression. Although both the Thomas and Yoon–Nelson models showed good agreement with the experimental data, the Yoon–Nelson model was found to provide the best representation for cesium adsorption on the adsorbent, based on the χ^2 analysis.

1. Introduction

The effective and safe management of liquid wastes is a primary requirement in the nuclear industry. These wastes arise at every stage of the nuclear fuel cycle and must be treated to ensure compliance with stringent regulatory standards before final disposal into the environment. In particular, the need to accommodate wastes arising from the decommissioning of nuclear installations has gradually increased over the past two decades. Among the main fission products in nuclear waste solutions, ^{137}Cs and ^{134}Cs are of special concern due to their long half-life (30 years), high fission yield (6.09%), high activity, and high water solubility. These properties enable the migration of cesium through ground water to the biosphere, and this cesium poses a serious threat to the environment and human health (Ding and Kanatzidis, 2007).

Several physico-chemical methods have been investigated for the removal of radioactive cesium from wastewater, including solvent extraction, chemical precipitation, membrane processes, coagulation, electrodialysis, and ion-exchange (Iwanade et al.,

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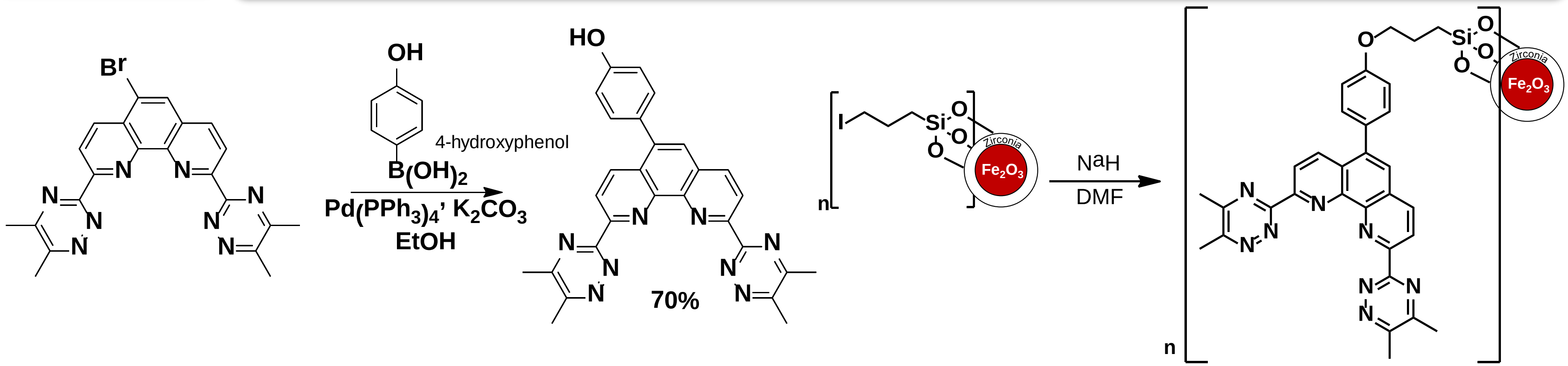
3차년도 연구결과

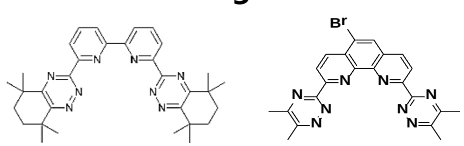
연구목표 | **선택성을 향상시킨 리간드 합성 및 흡착 성능 평가**



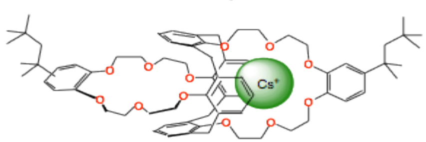
UK

Ligands

- Bis-Triazine Ligands



- Cs selective Ligands



Immoilization

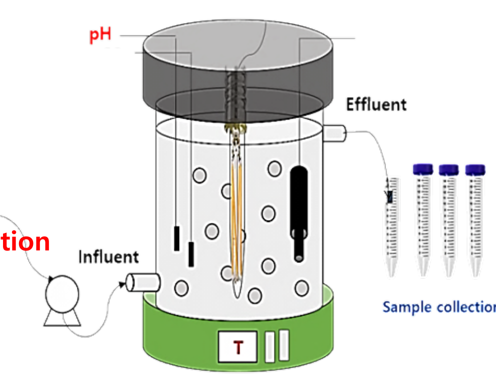
South Korea

Solid-supported materials

- Magnetic nanoparticle
- Graphene oxide sheet
- Carbon nanotube
- Clays (montmorillonite)

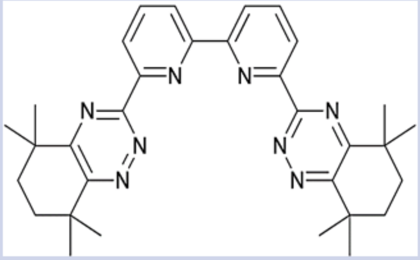
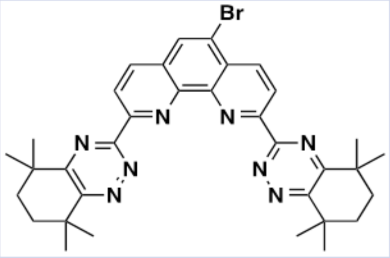
Application

Field Application



3차년도 연구결과

■ 선택성을 향상시킨 리간드 합성 및 흡착 성능 평가

CyMe$_4$-BTBP	CyMe$_4$-BTPhen
 6,6'-bis(5,5,8,8,-tetramethyl-5,6,7,8-tetrahydro-1,2,4-benzotriazin-3-yl)-2,2'-bypyridine	 5-bromo-2,9-bis(5,5,8,8-tetramethyl-5,6,7,8-tetrahydro-1,2,4-benzotriazin-3-yl)-1,10-phenanthroline
✓ Current European benchmark ligand ✓ For the development of Actinide separation process ✓ D_{Am}<10, $SF_{Am/Eu}$ = 150 ✓ Slow extraction kinetic → need to add a phase-transfer catalyst	✓ Positive effect on the extraction thermodynamics and kinetics ✓ Faster kinetics of extraction ✓ D_{Am}<1000, $SF_{Am/Eu}$ = 400 ✓ Easily immobilized to solid-supported materials

3차년도 연구결과

■ 선택성을 향상시킨 리간드 합성 및 흡착 성능 평가

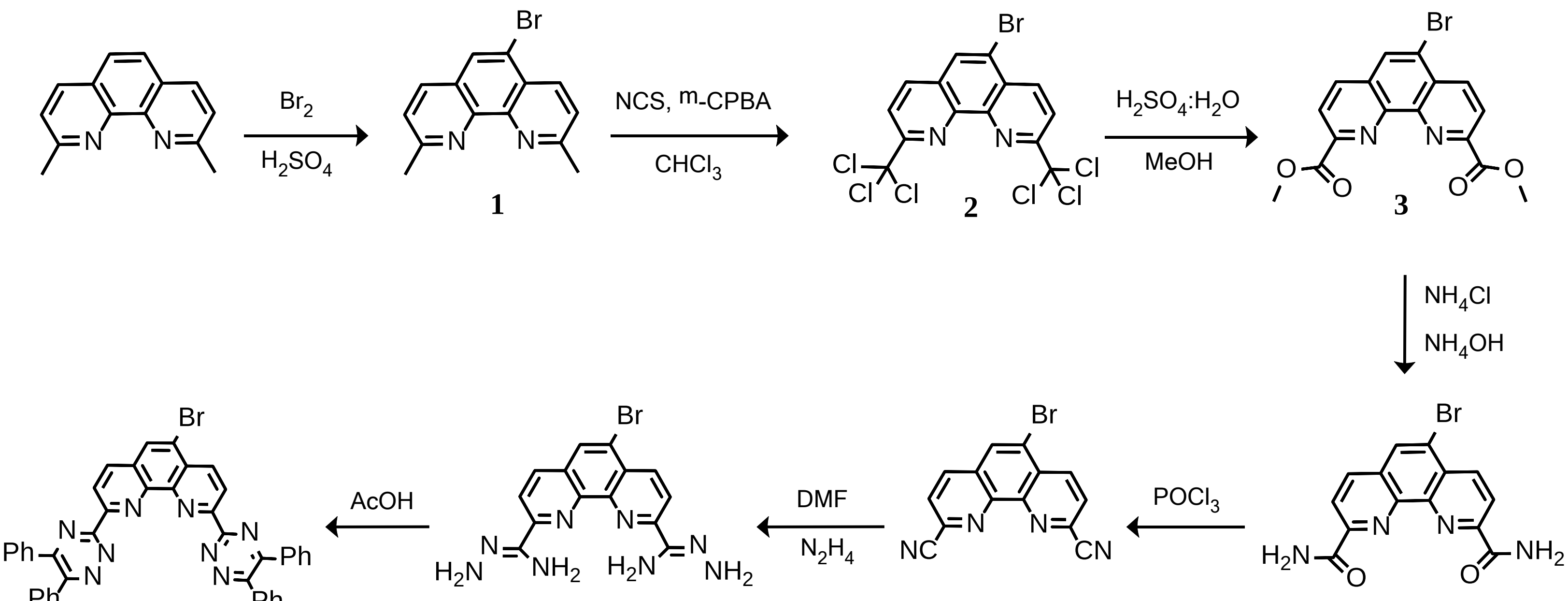
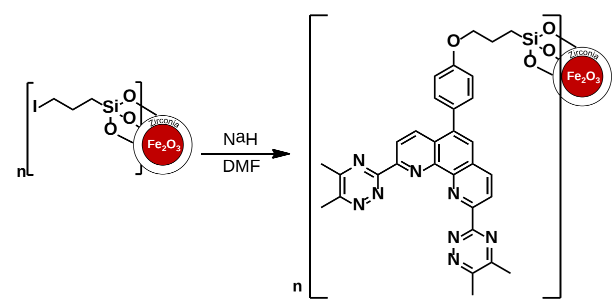


Fig. 3-8. Synthesis of 5-bromo-Ph4-BTPhen ligands

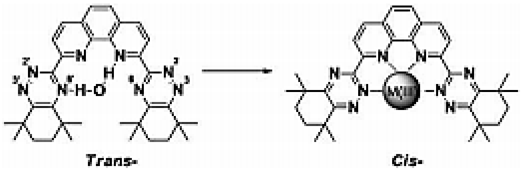
- ▸ ¹H and ¹³C NMR spectra
- ▸ Mass spectra (m/z)
- ▸ FT-IR spectra

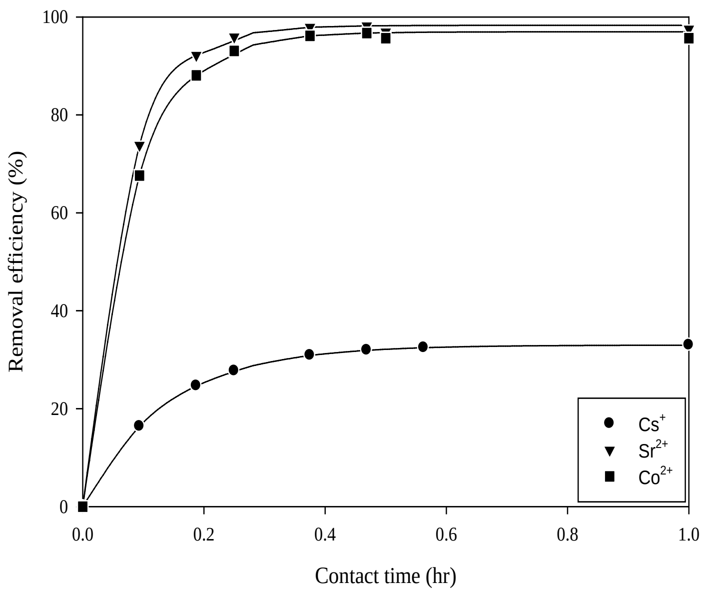
Element	Wt%
C	66.52
N	13.81
O	4.85
Br	14.83
Total:	100



3차년도 연구결과

■ 선택성을 향상시킨 리간드 합성 및 흡착 성능 평가





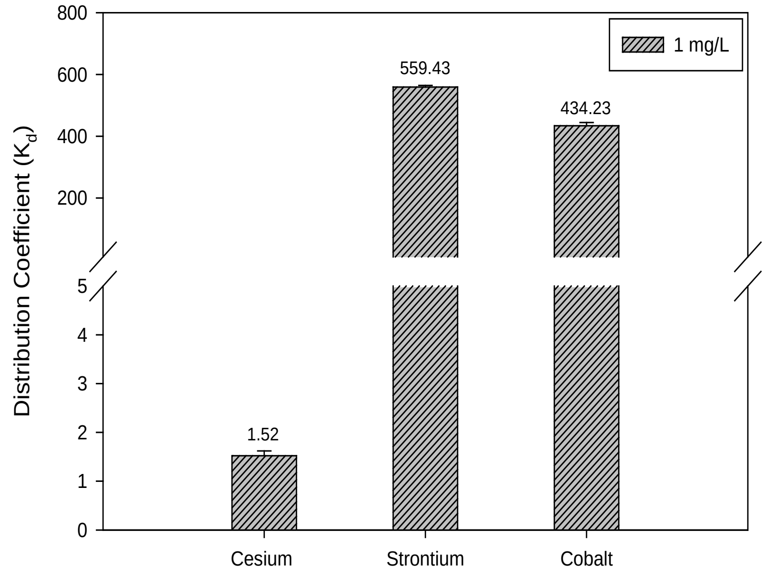


Fig. 3-9. Removal efficiency of Cs^{+}, Sr^{2+}, and Co^{2+} by 5-bromo-2,9-bis(5,6-diphenyl-1,2,4-triazin-3-yl)-1,10-phenanthroline ([5-bromo-Ph4-BTPhen] = 5 mg, $[Cs+, Sr^{2+}, Co^{2+}]_i$ = 1 mg/L, and pH_i = 7).

3차년도 연구결과

■ 실제 조건과 유사한 환경(공동이온 및 pH 등)에서의 방사성핵종 제거 실험 및 성능평가

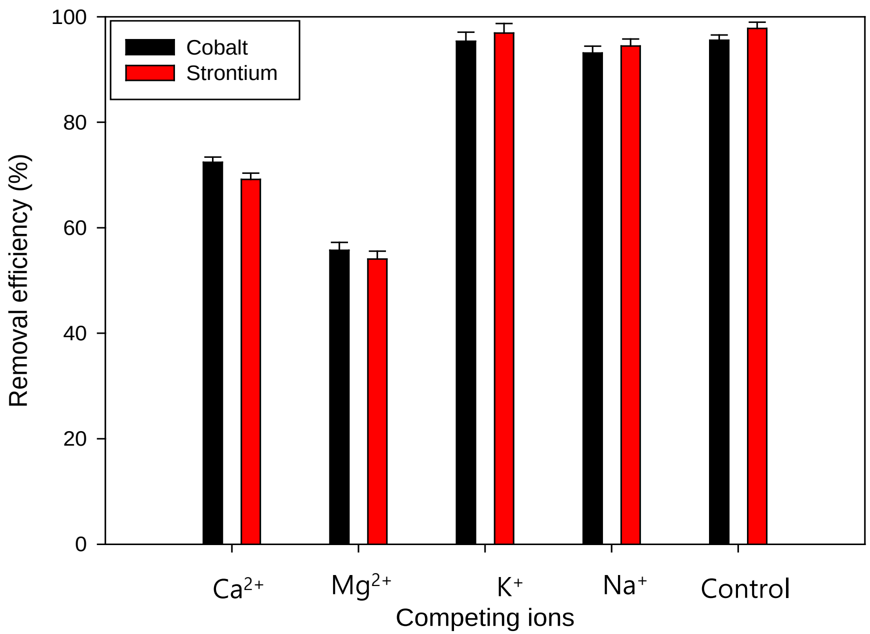


Fig. 3-10. The distribution coefficient K_d values of Cs^+, Sr^{2+}, and Co^{2+} in the stock solution including co-existing ions for 24 h at 25℃ ([5-bromo-Ph4-BTPhen] = 5 mg, $[Ca^{2+}, Mg^{2+}, K^+, Na^+]_i$ = 1 mg/L, $[Cs^+, Sr^{2+}, Co^{2+}]_i$ = 1 mg L^{-1}, and pH_i = 7).

연구성과

■ 학술활동

학술적 연구성과

국내논문		국외논문		초청강연실적	학술대회 논문 발표	
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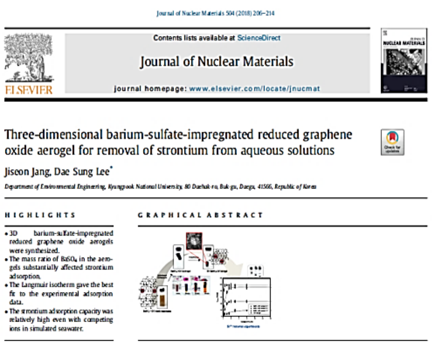
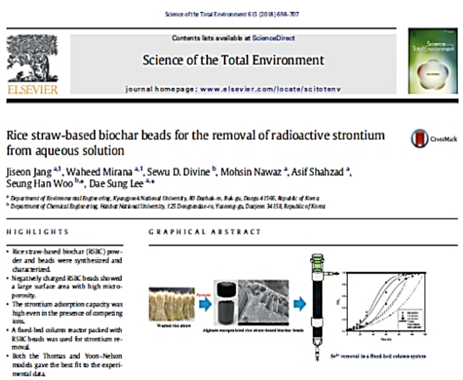

Journal of Nuclear Materials

Three-dimensional barium-sulfate-impregnated reduced graphene oxide aerogel for removal of strontium from aqueous solutions

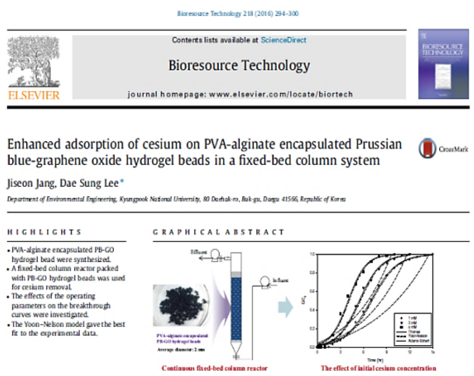
Jiseon Jang, Dae Sung Lee*

Journal of Nuclear Materials (2018)
IF 2.048 상위 4.540%


Science of the Total Environment

Rice straw-based biochar beads for the removal of radioactive strontium from aqueous solution

Jiseon Jang, Waheed Miran, Sewu D. Divine, Mohsin Nawaz, Asif Shahzad, Seung Han Woo, Dae Sung Lee


Bioresource Technology

Enhanced adsorption of cesium on PVA-alginate encapsulated Prussian blue-graphene oxide hydrogel beads in a fixed-bed column system

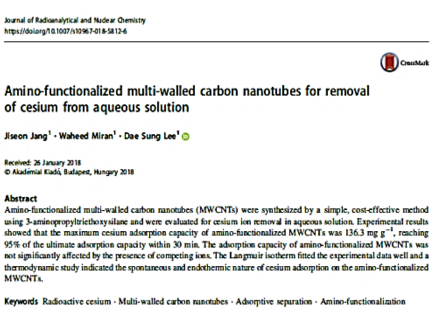
Jiseon Jang, Dae Sung Lee*


I&EC research

Magnetic Prussian Blue Nanocomposites for Effective Cesium Removal from Aqueous Solution

Jiseon Jang and Dae Sung Lee*

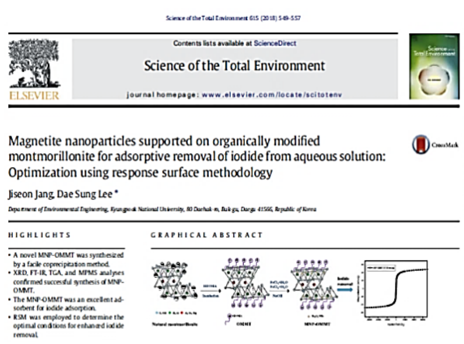
Industrial & Engineering Chemistry Research (2016)


Journal of Radioanalytical and Nuclear Chemistry

Amino-functionalized multi-walled carbon nanotubes for removal of cesium from aqueous solution

Jiseon Jang · Waheed Miran · Dae Sung Lee

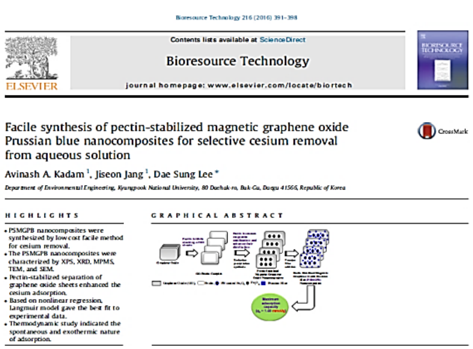
Journal of Radioanalytical and Nuclear Chemistry (2018)


Science of the Total Environment

Magnetite nanoparticles supported on organically modified montmorillonite for adsorptive removal of iodide from aqueous solution: Optimization using response surface methodology

Jiseon Jang, Dae Sung Lee*

Science of the Total Environment (2018,2018)
IF 4.9 상위 9.389%


Bioresource Technology

Facile synthesis of pectin-stabilized magnetic graphene oxide Prussian blue nanocomposites for selective cesium removal from aqueous solution

Avinash A. Kadam, Jiseon Jang, Dae Sung Lee*

Bioresource Technology (2016,2016)
IF 5.651 상위 3.571%

KNU KYUNGPOOK NATIONAL UNIVERSITY

연구성과

■ 인력양성

인력양성 및 연구시설								
학위과정지원		국내외 연수지원				참여학생수		
		장기		단기				
박사	석사	국내	국외	국내	국외	박사과정	석사과정	학사과정
16	14	0	*3	0	27	16	14	7

* 한영 인력양성사업
– 포스텍 박사과정 김규현 : University of Central Lanchashire (2016.4.1~2017.3.31)

* 자체 장기연수
– 경북대 박사과정 장지선 : University of Reading (2016.6.28~2016.9.28, 2017.3.31~2017.5.20)

연구성과

■ 특허

연도	특허명	발명자명	출원인	출원국	출원및등록번호
2015	입자크기 제어가 가능한 자성나노입자 코어 기반의 방사선 세슘 제거용 나노복합체 개발	이대성 장지선	경북대학교 산학협력단	대한민국	10-2015-0117820
2017	세슘 흡착용 히드로젤 비드 및 이의 제조 방법	이대성 장지선	경북대학교 산학협력단	대한민국	101782241
2018	스트론튬 제거를 위한 황산바륨이 함침된 에어로겔 합성 방법	이대성 장지선	경북대학교 산학협력단	대한민국	출원준비중

특허증
CERTIFICATE OF PATENT

특 허 (Patent Number): 제 10-1782241 호

출원번호 (Application Number): 제 10-2016-0002347 호
출원일 (Filing Date): 2016년 01월 08일
등록일 (Registration Date): 2017년 09월 20일

발명의 명칭 (Title of the Invention)
세슘 흡착용 히드로젤 비드 및 이의 제조 방법

특허권자 (Patentee)
경북대학교 산학협력단(176271-*******)
대구광역시 북구 대학로 80 (산격동, 경북대학교)

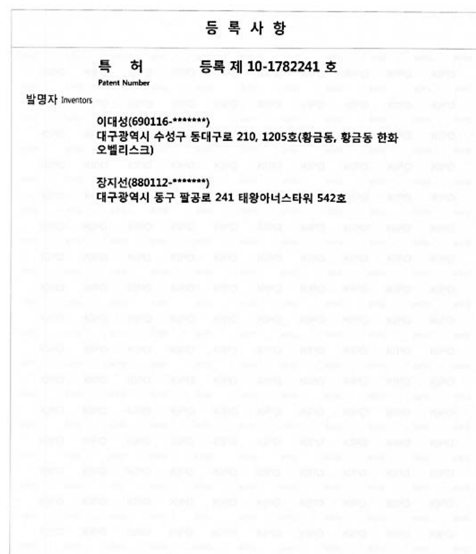
발명자 (Inventor)
등록사항란에 기재

위의 발명은 「특허법」에 따라 특허등록원부에 등록되었음을 증명합니다.
This is to certify that, in accordance with the Patent Act, a patent for the invention has been registered at the Korean Intellectual Property Office.

2017년 09월 20일

특허청장
COMMISSIONER,
KOREAN INTELLECTUAL PROPERTY OFFICE
성 윤 모

특허청
Korean Intellectual Property Office



등 록 사 항

특 허 (Patent Number): 등록 제 10-1782241 호

발명자 Inventors

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KYUNGPOOK NATIONAL UNIVERSITY

연구성과

■ 1st South Korea-UK Project Workshop (University of Reading, 2015.9.7~2015.9.11)

South Korea team's presentation

Prof. Dae Sung Lee	Advanced Radioactive Waster treatment Using Nanostructured Hybrid Composites
Prof. Sang-June Choi	D & D Research Centre in Korea
Prof. Song Soon Song	National Nuclear R & D Programs in the Republic of Korea
Dr Avinash Kadam	Pectin-Stabilized Magnetic Graphene Prussian Blue Nanocomposite for Removal of Cesium in Water
Miss Jiseon Jang	PB-based Nanocomposites and Hydrogel Beads for Removal of Radioactive Cesium in Aqueous Solution

UK team's presentation

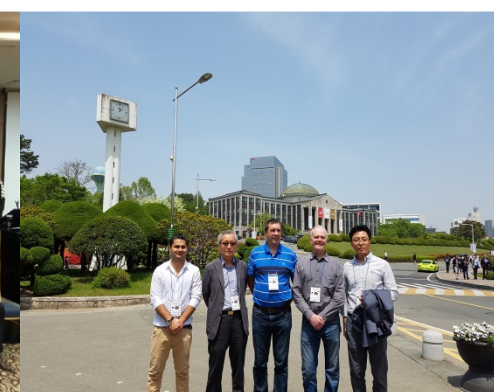
Prof. Laurence Harwood	The Development of Ligands for Separation of Minor Actinides from Lanthanides for Nuclear Fuel Reprocessing
Dr Ashfaq Afsar	Covalent Immobilization of Minor Actinide-Selective Ligands onto Magnetic Nanoparticles
Mr James Westwood	Progress Towards Actinide-Selective Ligands: Electronic Modulation of BTPhens

연구성과

■ 2nd South Korea-UK Project Workshop (경북대, 2016.4.25~2016.4.29)

South Korea team's presentation	
Prof. Dae Sung Lee	Advanced Radioactive Waster treatment Using Nanostructured Hybrid Composites
Prof. Sang-June Choi	Decommissioning Technology of Nuclear Facility in South Korea
Mr Hyunkyu Lee	Sorption of Cesium ions from Aqueous Solutions by Multi-walled Carbon Nanotubes Functionalized with Copper Ferrocyanide
Miss Jiseon Jang	Graphene Aerogel as a Highly Efficient and Recyclable Adsorbent for Removal of Radioactive Strontium

UK team's presentation	
Prof. Harry Eccles	Decommissioning of Nuclear Facilities – UK's challenges
Prof. Laurence Harwood	Covalent Immobilization of Minor Actinide-Selective Ligands
Prof. Gary Bond	Immobilization of Radionuclides



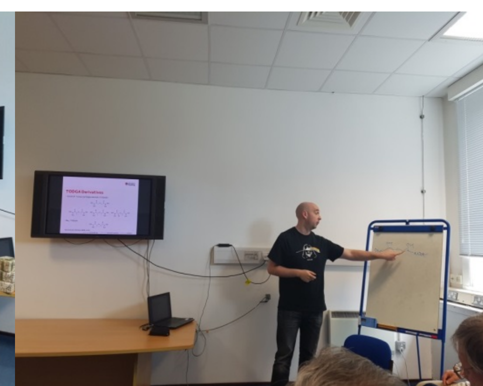
연구성과

■ 3rd South Korea-UK Project Workshop (University of Reading, 2017.5.14~2017.5.19)

South Korea team's presentation	
Miss Jiseon Jang	Biochar-based Beads and BDTP Ligands for Radionuclide Separation from Simulated Radioactive Wastewater
Miss Jungweon Choi	Copper Ferrocyanide Functionalized Core-Shell Magnetite Silica Composites for the Selective Removal of Cesium ions from Radioactive Liquid Waste
Mr Sun-il Kim	A Study on the Evaluation of the Effects of Soil Decontamination and Sedimentation Agent using Chemical Equilibrium Code

UK team's presentation	
Dr Alistair Holdsworth	Metal Phosphates for the Remediation of Decontamination Liquors: an Integrated Approach to Thorough Clean-up
Prof. Harry Eccles	Consortium Website
Dr Ashfaq Asfar	Separating Minor Actinides from Lanthanides: Solution Phase versus Immobilized Ligand – the Importance of Speciation



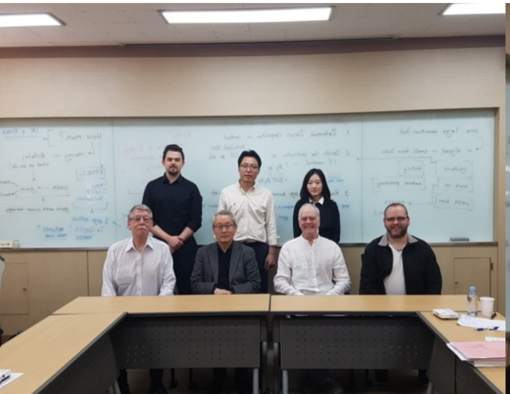


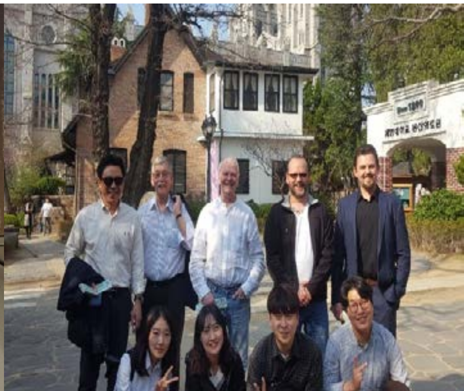

연구성과

■ 4th South Korea-UK Project Workshop (경북대, 2018.3.26~2018.3.28)

South Korea team's presentation	
Prof. Dae Sung Lee	5-bromo-2,9-bis(5,6-diphenyl-1,2,4-triazin-3-yl)-1,10-phenanthrolin for the Selective Removal of Strontium and Cobalt from Aqueous Solution
Dr Jiseon Jang	Three-dimensional Barium-sulfate-impregnated Reduced Graphene Oxide Aerogel for Removal of Strontium from Aqueous Solutions

UK team's presentation	
Dr Mark D Ogden	Functionalised Silicates for the Remediation and Immobilization of Radionuclides
Prof. Laurence Harwood	Separating Minor Actinides from Lanthanides: Solution Phase versus Immobilized Ligand - the Importance of Speciation
Prof. Harry Eccles	Nano Chemical Factories for Nuclear Waste





목차

KNU KYUNGPOOK NATIONAL UNIVERSITY

연구목표 및 내용

최종목표 | **크라운에테르 리간드를 부착한 자성 나노물질 기반의 고도 방사성폐기물 처리**

■ 크라운에테르 리간드를 부착한 자성 나노물질 합성 및 선택적 제거 메커니즘 규명

- 크라운에테르 리간드를 부착한 자성 나노물질 합성
- ^{1}H, ^{13}C NMR spectra, FT-IR, Mass spectra, XRD, TGA 이용한 특성분석
- 합성된 리간드의 방사성 핵종에 대한 기초 성능 평가
- 크라운에테르 리간드를 부착한 자성 나노물질의 기초 성능 평가

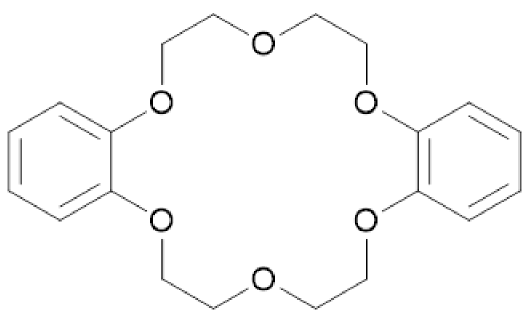
■ 크라운에테르 리간드를 부착한 자성 나노물질 기반의 액체 방사성폐기물 처리 공정 설계/운전

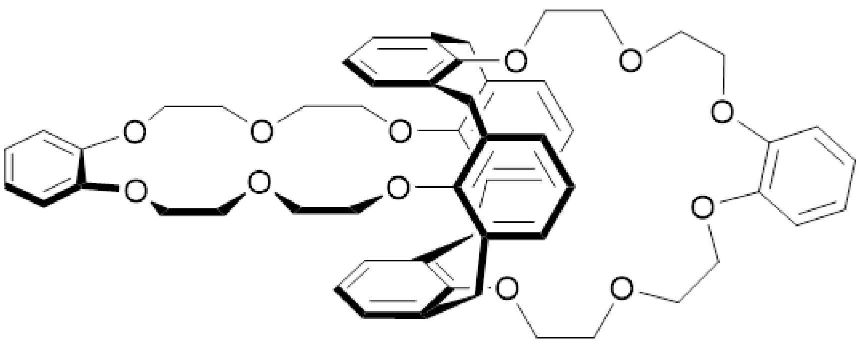
- 배경물질의 유무와 종류에 따른 운전 성능 비교 평가
- 운전 조건, 체류시간, 투입량에 따른 운전 성능 비교 평가
- 크라운에테르 리간드 거동, 경쟁, 선택성 평가
- 합성된 크라운에테르 리간드를 부착한 자성 나노물질의 안정성 평가
- 기초 운전조건 확립 및 반응기 설계/운전

연구목표 및 내용

연구목표 크라운에테르 리간드를 부착한 자성 나노물질 합성 및 방사성 핵종의 선택적 제거 메커니즘 규명

■ 크라운에테르 리간드를 부착한 자성 나노물질 합성 및 특성 분석





Crown ethers:

- Can form complexes with alkali metal cations
- Oxygen atoms donate electron density to cation
- Cavity size and the number of donor oxygen atoms determines the cation for which it is selective.
- Poor extraction in the presence of Na^+

Calix[4]crowns:

- Selectivity still dependent on crown ether size.
- Alkali metal cation also interacts with calix[4]arene π system
- Highly conformational pre-organised structure
- High selectivity towards Cs^+ over Na^+

연구목표 및 내용

연구목표 | 크라운에테르 리간드를 부착한 자성 나노물질 합성 및 방사성 핵종의 선택적 제거 메커니즘 규명

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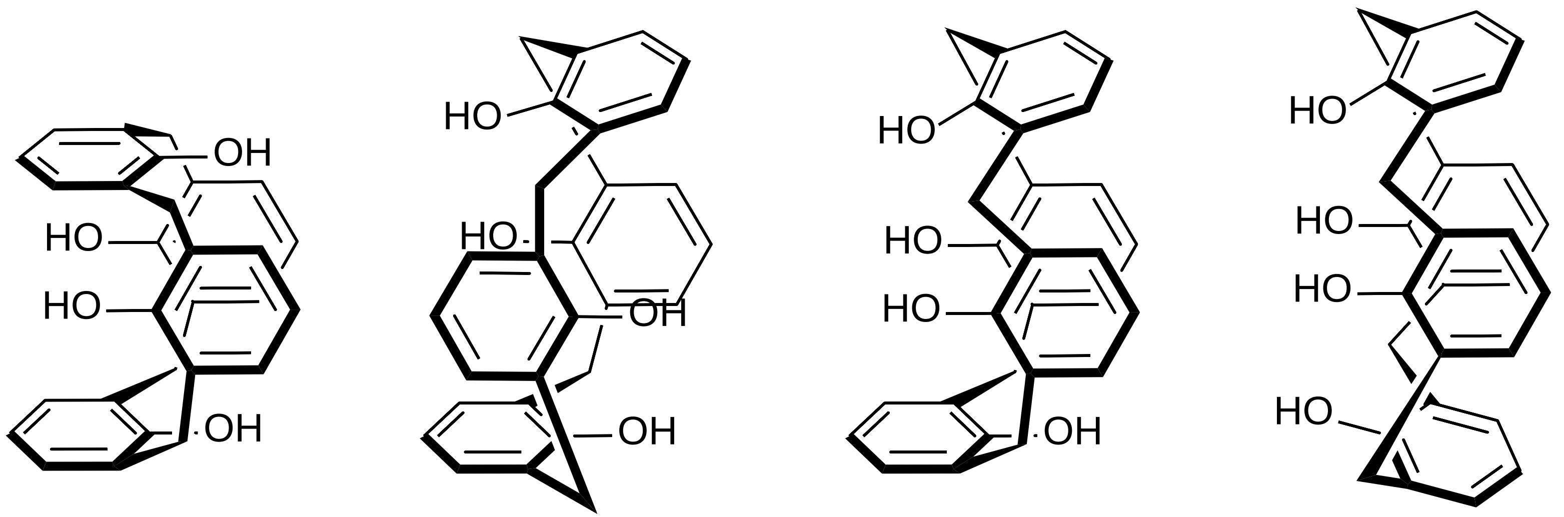
Calix[4]crown Conformations



1,3-alternate 1,2-alternate Partial Cone Cone

- Cone conformation allows to much steric bulk reducing cation interaction.
- Only calix[4]crown structures adopting the 1,3-alternate conformation show high extraction for Cs^+ due to the additional interactions from the π-system.

연구목표 및 내용

연구목표 | 크라운에테르 리간드를 부착한 자성 나노물질 합성 및 방사성 핵종의 선택적 제거 메커니즘 규명

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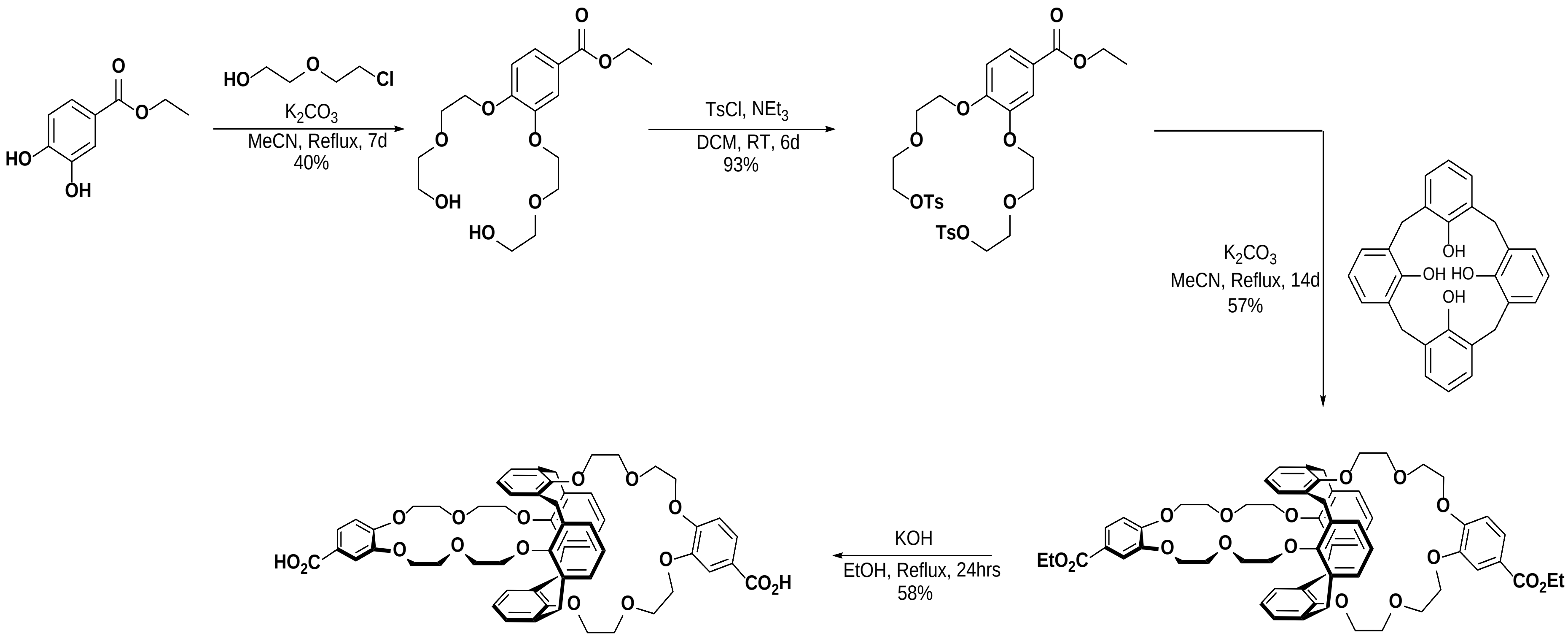
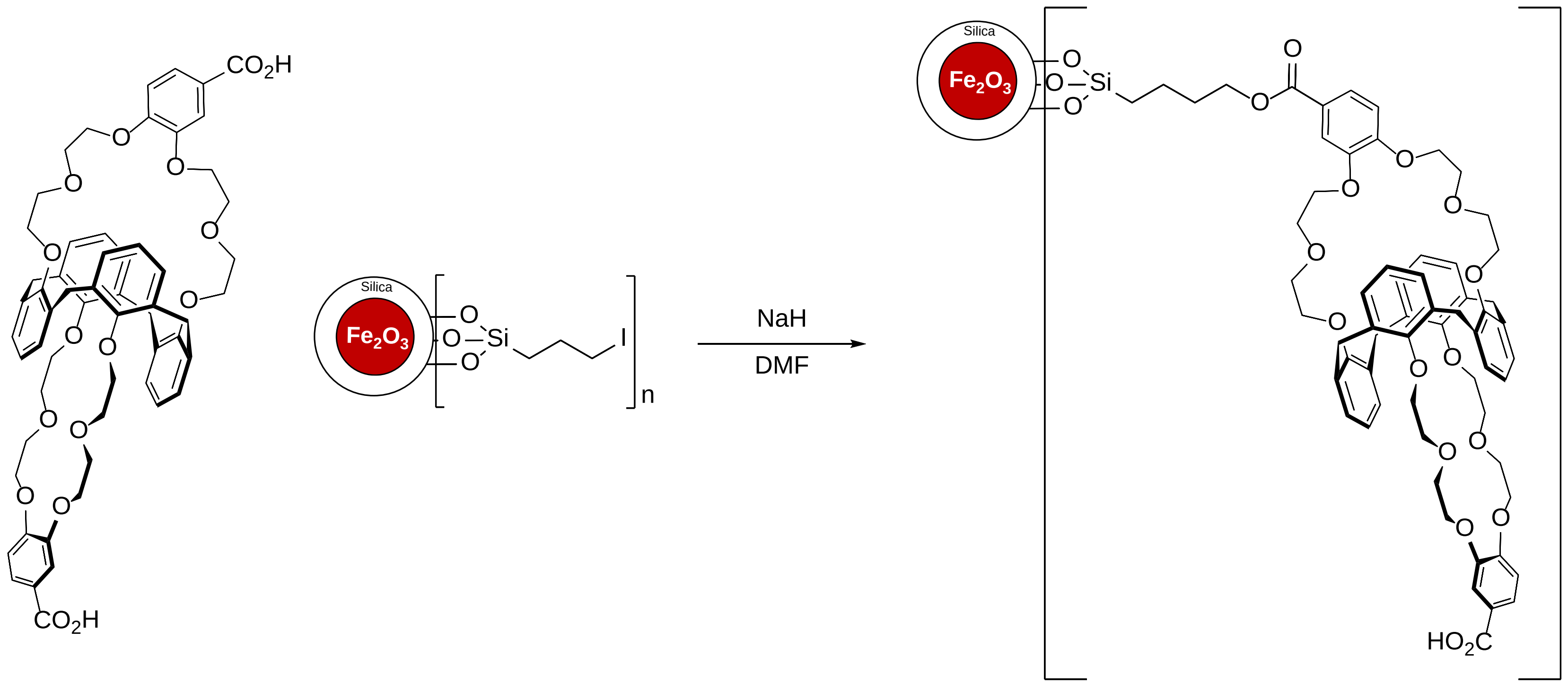


Calix[4]arene bis-(3-Carboxylbenzocrown-6)

연구목표 및 내용

연구목표 크라운에테르 리간드를 부착한 자성 나노물질 합성 및 방사성 핵종의 선택적 제거 메커니즘 규명

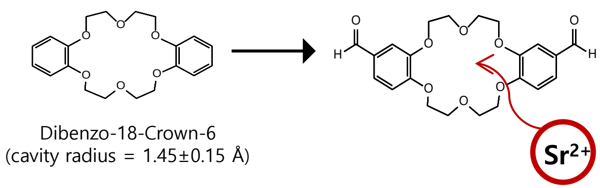
■ 크라운에테르 리간드를 부착한 자성 나노물질 합성 및 특성 분석

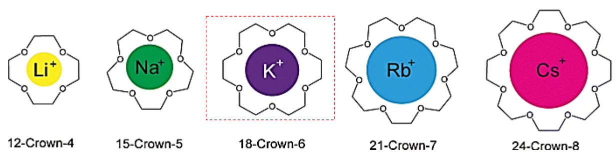


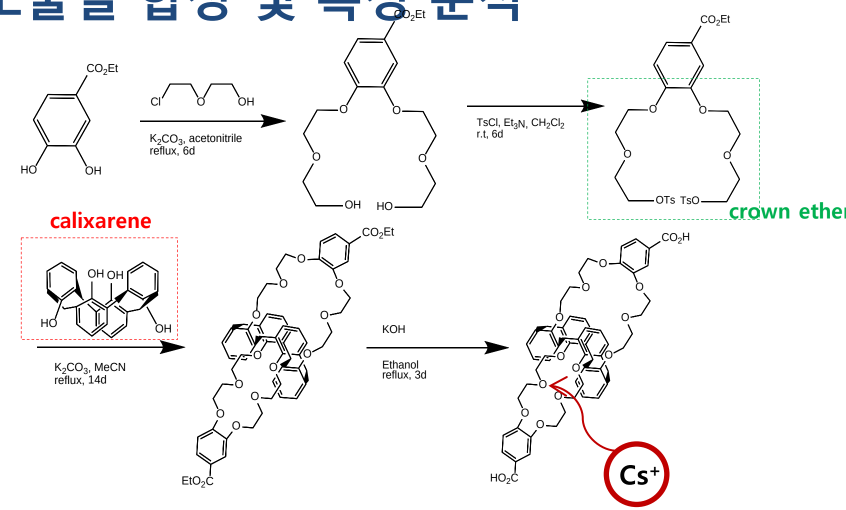
연구목표 및 내용

연구목표 | 크라운에테르 리간드를 부착한 자성 나노물질 합성 및 방사성 핵종의 선택적 제거 메커니즘 규명

■ 크라운에테르 리간드를 부착한 자성 나노물질 합성 및 특성 분석







- The ionic radius for strontium and potassium ions are similar, 1.52 Å and 1.38 Å,
- Calix-crown compounds possess a cavity that is highly complementary for Cs^+ ions rather than other alkali metal ions

- ^1H and ^{13}C NMR spectra (500MHz/Liquid)

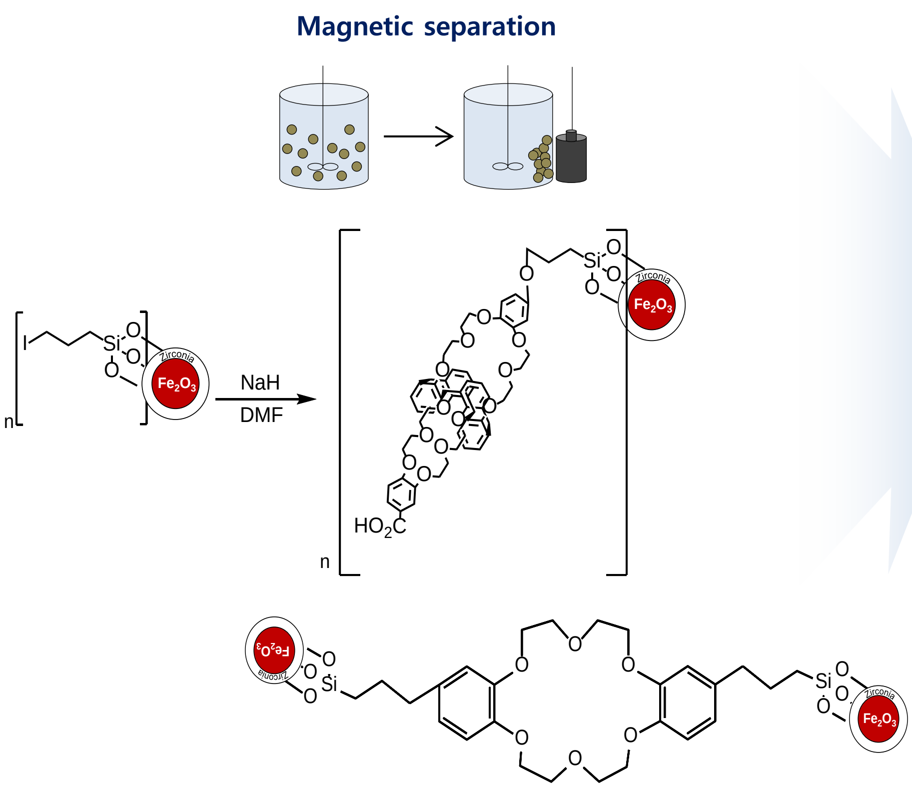
- FT-IR/NIR Spectrophotometer

- Mass spectra (m/z)

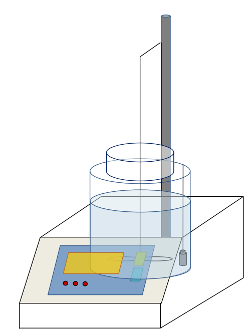
연구목표 및 내용

연구목표 크라운에테르 리간드를 부착한 자성 나노물질 합성 및 방사성 핵종의 선택적 제거 메커니즘 규명

■ 합성된 리간드의 방사성 핵종에 대한 기초 성능 평가



Batch test



Basic performance evaluation

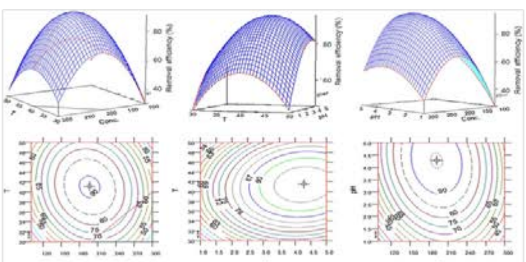
- pH & temperature
- Solvent
- Selectivity (Cs^+, Sr^{2+})
- Competing ions (Na^+, K^+, Ca^{2+}, Mg^{2+})
- Reclamation

연구목표 및 내용

연구목표 | **크라운에테르 리간드를 부착한 자성 나노물질 기반의 액체 방사성폐기물 처리 공정 설계 및 운전**

■ 실험실 규모의 반응기 설계 및 운전

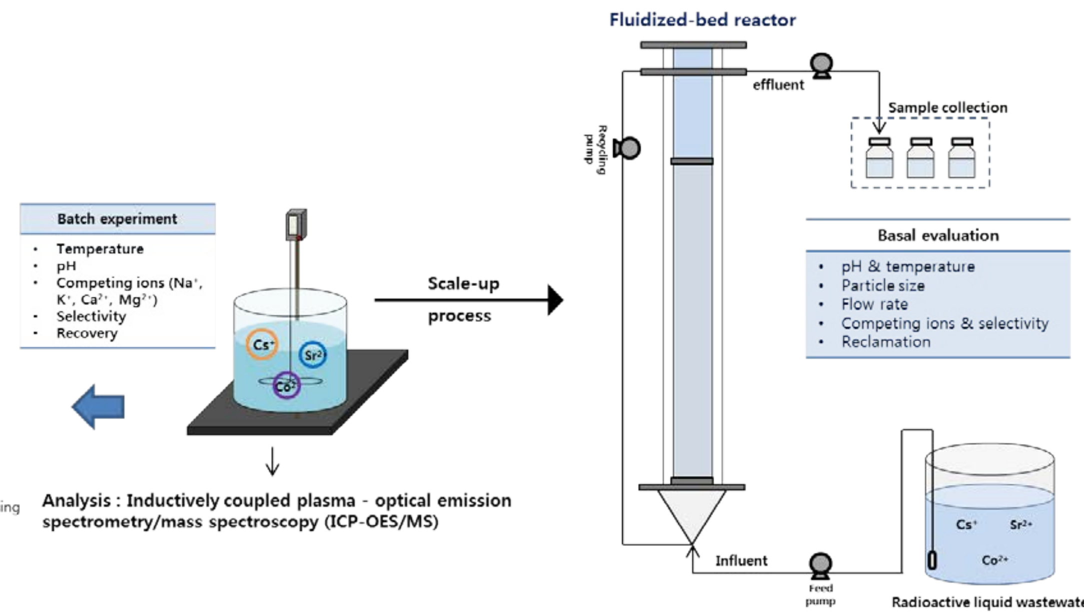
■ **Optimization** of radionuclide removal by **response surface methodology (RSM)**



Box-Behnken experimental design with three independent variables.

Trial #	Initial iodide conc. (mg/L)		Temperature (℃)		pH		Removal efficiency (%)
	X_1	Code	X_2	Code	X_3	Code	Y
1	100	−1	30	−1	3	0	32.19
2	300	1	30	−1	3	0	69.32
3	100	−1	50	1	3	0	49.48
4	300	1	50	1	3	0	32.93
5	100	−1	40	0	1	−1	26.85
6	300	1	40	0	1	−1	36.87
7	100	−1	40	0	5	1	66.71
8	300	1	40	0	5	1	53.38
9	200	0	30	−1	1	−1	44.85
10	200	0	50	1	1	−1	47.65
11	200	0	30	−1	5	1	67.32
12	200	0	50	1	5	1	81.75
13	200	0	40	0	3	0	90.75
14	200	0	40	0	3	0	90.81
15	200	0	40	0	3	0	90.79

Two-dimensional contour lines and three-dimensional response surface plots for the maximum removal efficiency using initial radionuclide concentration (X1), temperature (X2), and pH (X3).



목차

경북대 환경과학기술연구소

■ 교육부 지정 이공분야 대학중점연구소(2018-2027) (원전 제염해체 및 물산업 특성화 분야)

VISION

에너지/환경 기술 개발 및 인력양성을 통한 **국가/지역 미래성장동력 산업** 발전에 기여

경북대학교 환경과학기술연구소


원전 제염·해체 산업

- 에너지/환경 핵심 기술의 확보와 환경 개선
- 에너지/환경 산업의 비약적인 발전과 미래산업 창출
- 효율적이고 집중적인 에너지/환경 관련 연구추진 모델 제시


물산업 클러스터

추진 전략

- 선택과 집중에 따른 전략적 핵심기술개발
- 핵심역량 강화 및 인프라 구축 및 시스템화
- 학제간 연구에 따른 시너지 효과

국제 협력

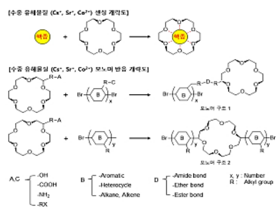

Univ. of Reading
Univ. of Oxford
Univ. of Cambridge
Qatar Environment &
Energy Research Institute
경북대학교
Caltech
Univ. of Texas Austin

연구목표 및 연구내용

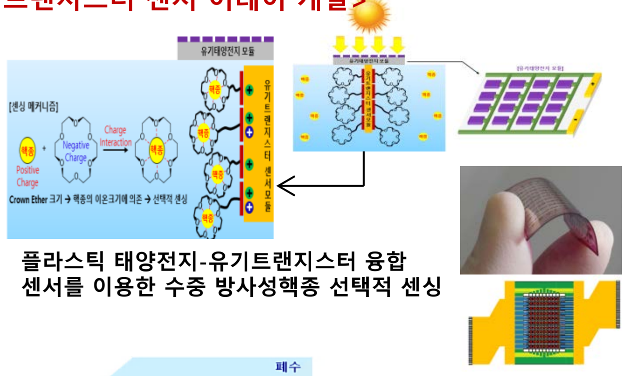
원전 제염·해체

방사성 핵종 검출센서 개발

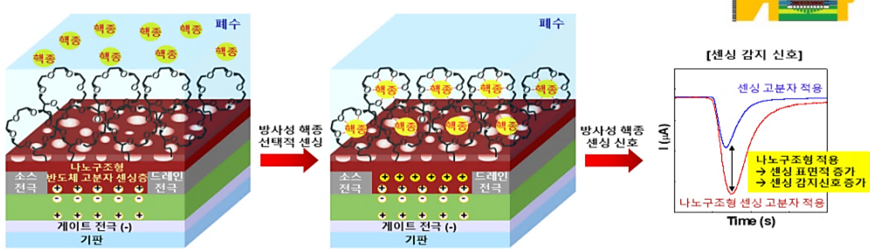
< 유기트랜지스터 센서 어레이 개발>



< 반도체 고분자 합성 >



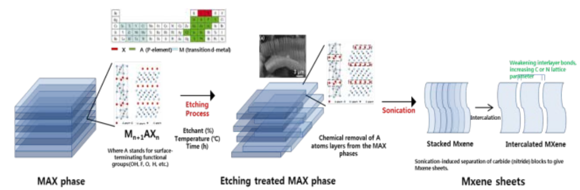
플라스틱 태양전지-유기트랜지스터 융합 센서를 이용한 수중 방사성핵종 선택적 센싱



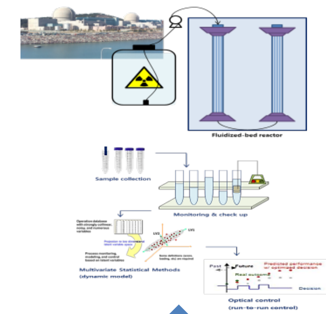
나노구조형 반도체 고분자 센싱층이 적용된 유기트랜지스터 센서 구조 및 센싱 메카니즘

방사성 핵종 제거

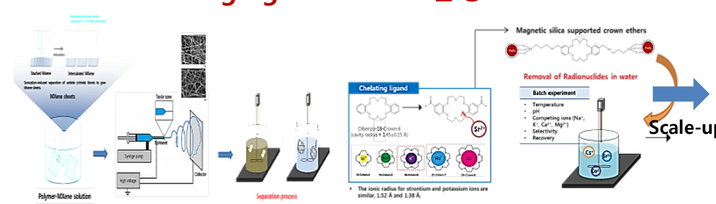
< 2D Mxene 나노복합체 합성>



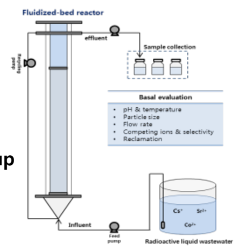
방사성핵종에 대한 선택성을 높이기 위한 2D MXene의 층간간격 조절(Etchant 농도(%), 에칭 공정 처리시간, 건조 온도, 금속이온 등)



< Chelating ligand Mxene 합성>



Polymer nanofiber와 자성 나노입자를 이용한 물속의 방사성핵종 제거 후 분리 및 제거 공정



< 유동층 반응기 설계/운전>

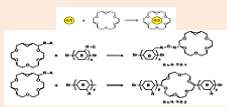
단계	연구 목표	연구 내용
1단계 (2018~ 2021)	액상 방사성 검출 센서 개발 및 방사성 핵종 제거	- 수중 방사성 핵종 검출을 위한 센서 개발을 위한 고분자 합성 및 유기트렌지스터 소자 개발 - 액상 방상성 핵종 제거를 위한 2D MXene 나노 복합 물질 합성 및 성능 평가
2단계 (2021~ 2024)	고감도 유기트렌지스터 센서 어레이 개발 및 방사성 핵종 제거 선택성 향상	- 고감도 센싱을 위한 나노구조형 고분자 반도체 트렌지스터 개발 - 방사성 핵종별 선택적 제거를 위한 chelating ligand기반의 MXene 나노복합체 개발 및 성능 평가
3단계 (2024~ 2027)	방사성 핵종 실시간 검출 및 제거를 위한 융합 센서 및 유동층 반응기 공정 최적화	- 플라스틱 태양전지 전력 기반 수중 유해물질 실시간 감지 시스템을 위한 태양전지-유기트랜지스터 융합 센서 개발 - 방사성 핵종 제거 유동층 반응기 설계, 운전, 실시간 공정 최적화 시스템 구축

단계별 추진전략 및 체계

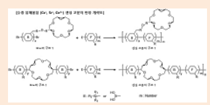
제1세부: 액체 방사성폐기물 처리용 검출 센서 및 나노구조체 기반 공정 기술 개발

1단계 (2018-2021)

Crown ether계열의 기능기가 포함된 모노머 합성



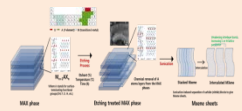
합성 센싱 고분자를 통한 방사성 핵종 검출 특성



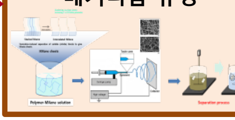
센싱 고분자 적용 유기트랜지스터 단위 소자 제작

수중 방사성 검출센서 개발

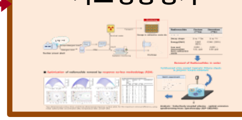
2D Mxene 나노복합체 합성



하이브리드 Mxene 나노복합체 개발을 통한 방사성핵종 제거 메커니즘 규명



하이브리드 MXene 나노복합체의 방사성핵종 제거 기초성능평가

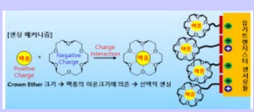


방사성 핵종 제거

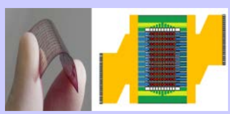
2단계 (2021-2024)

나노구조형 고분자 센싱층 적용 유기 트랜지스터 센서 제작/평가

나노구조형 고분자 센싱층 적용 유기트랜지스터 센서 제작/평가

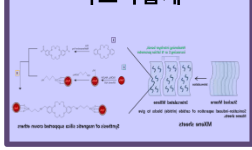


고감도 유기트랜지스터 센서 어레이 제작 및 특성 분석

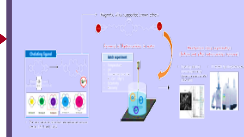


유기트렌지스터의 수중 방사성 핵종 고감도 검출

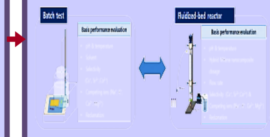
Chelating ligand 부착시킨 MXene 나노복합체



기초성능평가 및 주요 영향인자 조사



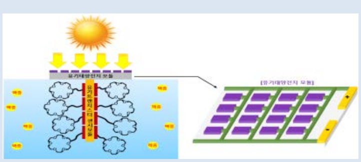
실험실 규모의 유동층 반응기 설계/운전



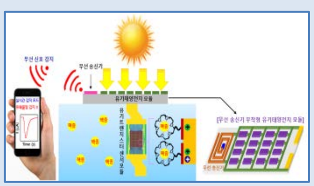
유동층 반응기 설계

3단계 (2024-2027)

감지 센서 구동용 플라스틱 태양전지 모듈 제작 및 유기트랜지스터 센서 어레이 연결 융합 센서 구조 개발

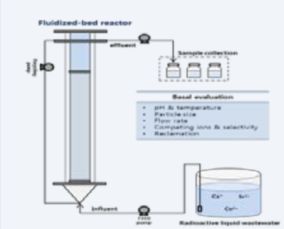


융합 센서 모듈 제작 및 수중 유해물질 실시간 감지 실험/분석



방사성핵종 검출용 융합센서 개발

유동층 반응기 장기 운전 및 운전 성능 조사



실시간 모니터링 및 최적 운전 진단 시스템 구축 및 현장 공정 적용을 위한 안정성 및 경제성 평가

실시간 공정 최적화 시스템 개발

목차

KNU
KYUNGPOOK NATIONAL UNIVERSITY

기대효과 및 활용계획

기술개발

하이브리드 나노복합체를 이용한 방사성 폐기물의 선택적 분리 및 포집 원천 기술 확보

핵심인력양성

공동연구 및 국외연수 통한 원자력시설 제염해체 분야 핵심인력양성

국제네트워크 구축

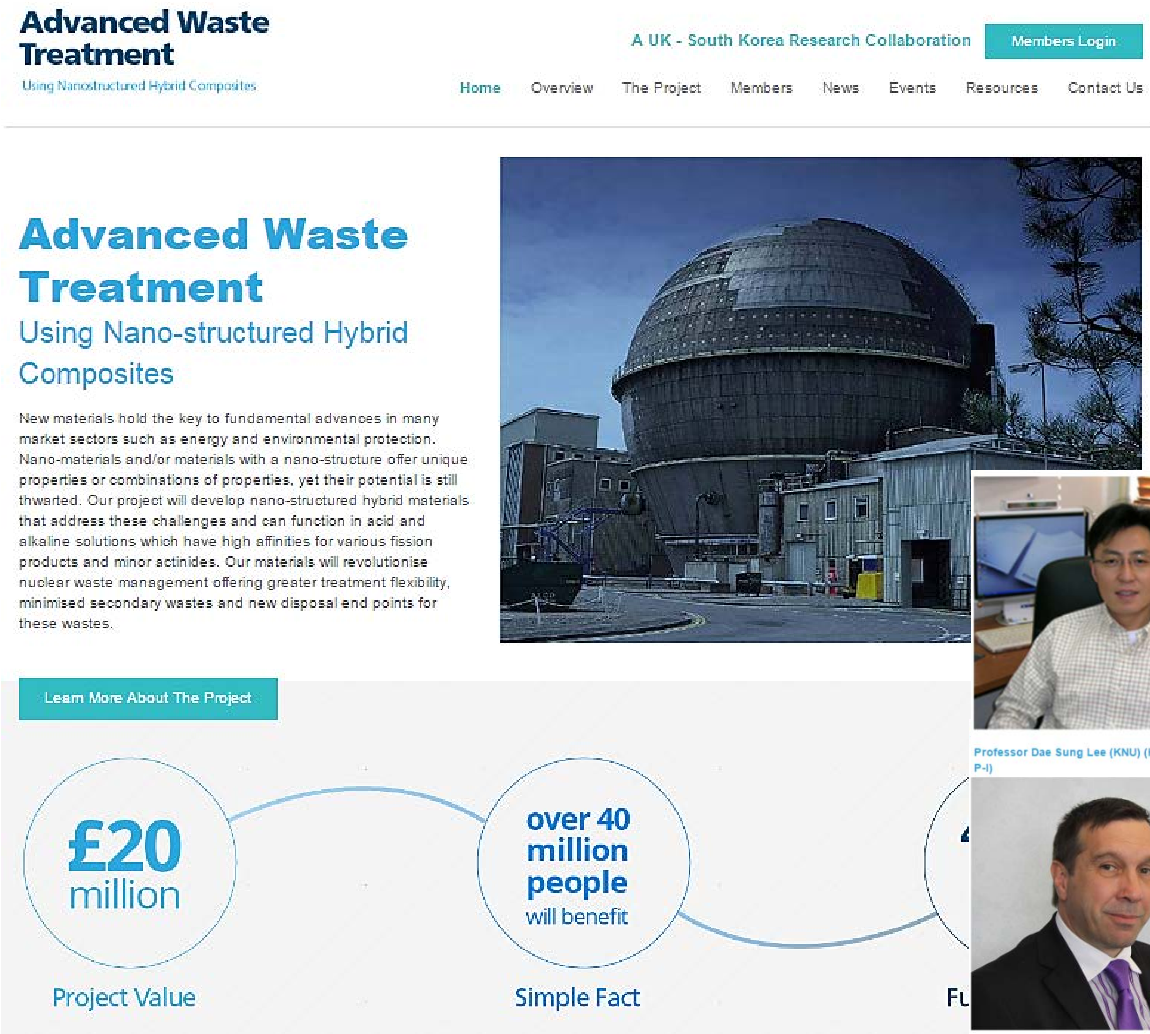
영국 연구진과의 방사성 폐기물처리 기술 국제네트워크 구축

활용 계획

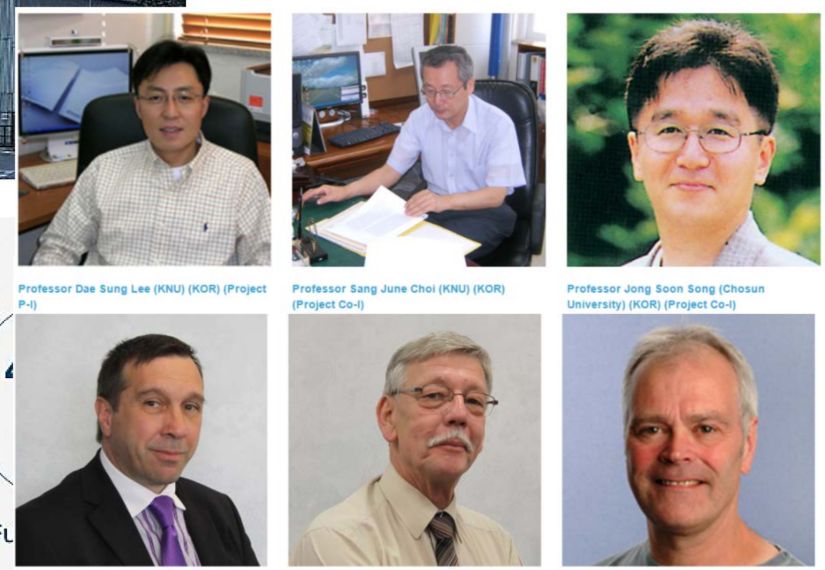
고효율, 저비용 방사성 폐기물 처리 기술 확보 및 적용

기대효과 및 활용계획

■ 한국-영국 제염해체 국제네트워크 구축



http://innovationlab.org.uk/AWT/



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Thank you!