## Potentiostatic Anodization for Resource Recovery and Purification in Water

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## 01 Iron Oxide Nanotubes for Phosphate Recovery in Water.

# Photocatalytic Metal Membrane with Self-organized Reactive TiO<sub>2</sub> Nanotubes.



## Anodization

Useful method for modifying the surface structure to obtain nanoporous array.

The surface of valve metal is instantaneously covered with a native oxide film when these metals are exposed to oxygen

containing environment.

## **Nanotube Formation**



- High surface area.
- Short solid-state diffusion path for catalysis and energy application.
- Fabrications are simple and cost effective.



## Iron Oxide Nanotubes for Phosphate Recovery in Water



## **Phosphorus**





Dees Lijmbach (Chris Thornton) "Phosphate removl and phosphate recovery:

towards sustainable development" COPPERAS-November 2000

Others: Lithium Ion Battery, Developer, Ceramics, Cosmetics, Cement, Artificial teeth *etc*.

Phosphorus is an essential component for food production and industrial growth.

- The global supply of this non-renewable resource is limited.
- Most researches were focused on the removal not recovery.
- Recovered phosphate can be reused at various demands.





## **Schematic diagram of Iron Oxide Nanotubes**





### An anodization apparatus

### A lab-scale column for continuous adsorption





### **Device for desorption**



## **FE-SEM** images of INTs





### AFM 2D and 3D images



Environmental Materials Laboratory (EML)

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3D

**2D** 

### **XRD** pattern



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### **Kinetic Model Analyses for Adsorption**





- (a) Pseudo first-order model
- (b) Pseudo second-order model
- (c) Elovich model



## **Desorption and Reusability**



(a) Desorption efficiency of phosphateadsorbed INTs depending on various concentrations of NaOH.

(b) Repeated adsorption and desorption for reusability.



## X-ray Photoelectron Spectroscopy (XPS) of INTs





# Photocatalytic Metal Membrane with Self-Organized Reactive TiO<sub>2</sub> Nanotubes.



## TiO<sub>2</sub>

- Water and wastewater treatment systems using TiO<sub>2</sub> photocatalytic reaction have been widely developed.
  - High oxidation-reduction potential for refractory organics decomposition.
  - Reduction of excess sludge.
  - Low cost & Non-toxic.



- Requires post-TiO<sub>2</sub> particles recovery process.
- Causes engineering difficulties in automatic operation.





 Although several immobilization methods were tried to improve the treatment performance, there are not satisfied aspects due to  $TiO_2$  particle exfoliation and activity decrease in long-term operation.





## **Characteristics of proposed technology**



Ti membrane anodization Nanostructured photocatalytic TiO<sub>2</sub> membrane

- Enhancement of adsorption
- Long retention time for photocatalytic reaction



Minimize membrane fouling & Maximize degradation efficiency of contaminants





### **FE-SEM** images



pH vs. pF curve in various electrolyte



### L 150 mm D 15 mm



### Before anodization



5.00um

#### FCTIC 20.0kV 12.0mm x10.0k SE(M)

After anodization





## Energy Dispersive X-ray spectroscopy (EDX)



Pure Ti membrane

Oxygen content 9 wt%

TiO<sub>2</sub> membrane embedded with nano tube

Oxygen content 41 wt%



# X-ray diffraction (XRD) patterns



- (a) Anodization and annealing
- (b) Untreated Ti membrane

### **TEM** analysis



(a) cross section

(b) a part of nano tube



(c) Crystal fringe distance

#### Crystal fringe distance

: 3.51 Å corresponding to spacing of (101) of the anatase phase  $TiO_2$ 





## Photocatalytic activities

## **Organics removal**



### Rate constants :

 $TiO_2$  membrane with UV >  $TiO_2$  membrane without UV > without anodization with UV .



## Permeation flux ratio $(J/J_0)$ of anodized TiO<sub>2</sub> metal membrane



Permeation flux ratio with UV-on showed about 8 times higher value than that of UV-off at 2 hrs filtration.



Environmental Materials

## Conclusion

- The use of nanomaterials in water and wastewater treatment has attracted a growing amount of attention due to the excellent electrical, magnetic, and catalytic properties of nanomaterials.
- In phosphate recovery, INTs are useful to recover phosphates in wastewater because additional collection of adsorbents is unnecessary and industrial byproducts can be used as raw materials to prepare INTs.



efficiency compared to the conventional methods.



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