Application of Nano Zero Valent Iron Coated Granular Activated Carbon for Pathogen Inactivation in Septic Tank Effluent

Prof. Chongrak Polprasert
Dr. Thammarat Koottatep
Mr. Krailak Fakkaew
Ms. Witchuda Tassanasuwan
Introduction

- Septic systems treat and dispose of household black water onsite.
- Economical for rural areas
- Effluent contains high pathogens and organic matters

Commercial Septic Tank

Drainage System
ZVI and NZVI Technologies

- Treated metals in groundwater by permeable reactive barriers (PRB)
- Removed contaminants; heavy metal and dyes from wastewater
- Literatures investigated the pathogens inactivation
NZVI possible to regeneration by heating air method

\[
\begin{align*}
\text{Fe}_2\text{O}_3 + 3\text{H}_2\text{S} & \rightarrow \text{Fe}_2\text{S}_3 + 3\text{H}_2\text{O} \\
2\text{Fe}_2\text{S}_3 + 3\text{O}_2 & \rightarrow 2\text{Fe}_2\text{O}_3 + 3\text{S}_2
\end{align*}
\]

- NZVI has excellently adsorption capacity.
- Heating is economical regeneration method of Fe$_2$S$_3$.
- Regeneration process converts Fe$_2$S$_3$ to Fe$_2$O$_3$
Objectives

1. To determine the treatment performance of NZVI/GAC for *Escherichia coli* (*E. coli*) inactivation

2. To study breakthrough point of NZVI/GAC column.

3. To evaluate the potential regeneration method of the used NZVI/GAC.
Methodology

PHASE I
Synthesis and Characterize of NZVI coated GAC

PHASE II
Determination of Optimum Conditions of NZVI Coated GAC Column

PHASE III
Determination of NZVI Coated on GAC Regeneration Method
Synthesis and Characterize of NZVI/GAC

\[
\begin{align*}
2\text{Fe}^{2+} + \text{BH}_4^- + 2\text{H}_2\text{O} & \rightarrow 2\text{Fe}^0 + \text{BO}_2^- + 2\text{H}_2 + 4\text{H}^+ \\
\text{Fe}^{2+}_{(aq)}\text{or(s)}/\text{GAC} + 2\text{BH}_4^-_{(aq)} + 6\text{H}_2\text{O}_{(l)}_{(aq)} & \rightarrow \text{Fe}^0_{(s)} + \text{Fe}^0_{(s)}/\text{GAC} + 2\text{B(OH)}_3(aq) + 7\text{H}_2(g)
\end{align*}
\]

Characterization method of NZVI

- Scanning Electron Microscopy (SEM)
- Scanning Electron Microscopy - Energy Dispersive (SEM-EDS)
- Specific Brunauer-Emmett-Teller (BET) Surface Area Analysis
- Inductively Coupled Plasma (ICP)
**PHASE II**

**Determination of Optimum Conditions of NZVI Coated GAC Column**

**Optimization NZVI/GAC in Batch Experiments**

- **Determination of optimum condition of NZVI coated GAC**
- **Optimization HRTs in batch scale varied: 0.5, 1, 2, 3 and 4 hours**
- **Determination relationship between NZVI and removed pathogen (E.coli)**
- **By plate count method**

Batch Mixing
Optimization NZVI/GAC in Continuous Experiments

Determination of Optimum Conditions of NZVI Coated GAC Column

Operation modes (Up-flow)

Operated in continuous experiment with HRT varied: 0.5, 1, 2 and 3 hours

Determination relationship between NZVI and removed pathogen (E.coli)

By plate count method
Breakthrough study

Determination of Optimum Conditions of NZVI Coated GAC Column

Operation modes (Up-flow)

Breakthrough study

Determination relationship between NZVI and removed pathogen (*E.coli*)

By plate count method
Methodology

PHASE II

Determination of Optimum Conditions of NZVI Coated GAC Column

**NZVI/GAC column**
- NZVI/GAC pack as column cover by PVC pipe ø 4 cm.
- Height 25 cm.
- Cover by sieve#200, ø 4 cm. for protect media loss

---

**Granular activated carbon (GAC) properties**
- **Commercial grades** made from coal
- **Size of GAC** in largest size (0.8-2 mm.)
- Iodine number is 968 mg/g
- pH 7.2
- Density 0.9 gm/cm³

---

Determination of Optimum Conditions of NZVI Coated GAC Column

PHASE II

Methodology

Toilet

Septic tank

NZVI/GAC column

Effluent discharge to environment

PHASE II

Granular activated carbon (GAC) properties

- **Commercial grades** made from coal
- **Size of GAC** in largest size (0.8-2 mm.)
- Iodine number is 968 mg/g
- pH 7.2
- Density 0.9 gm/cm³
Feasibility of NZVI/GAC regeneration methods

Used NZVI/GAC

Cleaning with deionized water

Heating by using oven (50°C, 12 h)

Add reducing agent (0.22 M NaBH₄)

Regeneration feasibility (Analyze: Fe, Fe₂O₃)

Determined pathogens inactivation efficiency
Results and discussion

NZVI/GAC characteristics

ICP analysis of synthesized NZVI/GAC

<table>
<thead>
<tr>
<th>Samples</th>
<th>% Iron Elements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Synthesized NZVI/GAC</td>
<td>0.9%wt</td>
</tr>
</tbody>
</table>

Specific BET surface analysis of initial GAC and synthesized NZVI/GAC

<table>
<thead>
<tr>
<th>Samples</th>
<th>Results (m²g⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial GAC</td>
<td>836.05</td>
</tr>
<tr>
<td>Synthesized NZVI/GAC</td>
<td>549.45</td>
</tr>
</tbody>
</table>

SEM/EDS analysis of synthesized NZVI/GAC

Initial GAC

Synthesized NZVI coated on GAC

Samples %

Iron Elements

Table:

<table>
<thead>
<tr>
<th>Samples</th>
<th>% Iron Oxide (Fe₂O₃) on Surface Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>Synthesized NZVI/GAC</td>
<td>11.91%</td>
</tr>
</tbody>
</table>
Results and discussion

- Particles sizes of Fe(0) on surface of GAC was less than 100 nm.
- Morphology of Fe(0) on surface of GAC was displayed to flat and sphere shape of NZVI crystal growth texture.
**Results and discussion**

**Optimization NZVI/GAC in Batch Experiments**

**E. coli inactivation using NZVI/GAC and GAC for treating synthetic wastewater and septic tank effluent**

- *E. coli* concentrations in the actual septic tank effluent and synthetic wastewater samples with using NZVI/GAC were reduced about 5 and 8 logs, respectively, after the operating time of 1 h.
- Pure GAC could not reduce or inactivate the *E. coli* at any operating times.
Results and discussion

Optimization NZVI/GAC in Continuous Experiments

*E. coli* inactivation using NZVI/GAC columns with the synthetic wastewater at various HRTs

- *E.coli* inactivation to 8 logs reduction operated at the HRT of 3 h and 2 h for the operating times of 3-9 h and 2-6 h, respectively.
- *E. coli* inactivation operated at low HRTs of 0.5 and 1 h was found to be approximately 1 log reduction.
Results and discussion

Breakthrough study of NZVI/GAC column

Breakthrough time of NZVI/GAC columns on *E. coli* inactivation in the septic tank effluent at various HRTs

- The breakthrough points of the NZVI/GAC column operated at the HRTs of 3, 6 and 12 h were found to be 1, 4 and 7 days, respectively, which increased with increasing HRTs.
Feasibility of NZVI/GAC regeneration

NZVI Color Variation

1. Synthesized NZVI/GAC

2. Used NZVI/GAC

3. Regenerated NZVI/GAC by heating (at 50°C, 12 h)

4. Regenerated NZVI/GAC by adding the reducing agent

Specific BET surface analysis of NZVI/GAC and Regenerated NZVI/GAC

<table>
<thead>
<tr>
<th>Samples</th>
<th>Results (m²g⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial GAC</td>
<td>836.05</td>
</tr>
<tr>
<td>Synthesized NZVI/GAC</td>
<td>549.45</td>
</tr>
<tr>
<td>Regenerated NZVI/GAC</td>
<td>652.94</td>
</tr>
</tbody>
</table>
Results and discussion

**E. coli removal efficiency of regenerated NZVI/GAC**

<table>
<thead>
<tr>
<th>Regeneration Method</th>
<th>E. coli Inactivation (Log reduction)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heating (50°C, 12h)</td>
<td>0</td>
</tr>
<tr>
<td>Heating with adding the reducing agent (NaBH₄)</td>
<td>6</td>
</tr>
</tbody>
</table>
Conclusions

- The synthesized NZVI/GAC could be used for pathogen inactivation in the septic tank effluent.

- Batch experiments conducted with actual septic tank effluent and synthetic wastewater samples showed that NZVI/GAC could reduce *E. coli* about 5 and 8 logs, respectively, after the operating time of 1 h.

- At the HRTs of 2 and 3 h, the continuous NZVI/GAC columns could reduce *E.coli* about 8 logs for the operating times of 6 and 9 h, respectively.

- At HRT of 12 h, the *E.coli* breakthrough time occurred after 7 days of operation. The NZVI/GAC could be regenerated and able to inactivate *E.coli* about 6 logs.
Acknowledgement

The project Sustainable Decentralized Wastewater Management in Developing Countries, financially supported by the Bill & Melinda Gates Foundation
Thank You