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Improvement in settleability and dewaterability of waste activated sludge by solar photocatalytic treatment in Ag/TiO$_2$-coated glass tubular reactor

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Abstract

In this study, photocatalysis was used to improve the dewaterability and settleability of waste activated sludge (WAS) by a solar photocatalytic reactor with transparent Ag/TiO₂ film as photocatalyst. Specific resistance of filtration (SRF) and sludge volume index (SVI) were used to evaluate WAS dewaterability and settleability, respectively, and the mechanism of photocatalysis was interpreted from the changes of pellets, loosely/tightly bound extracellular polymeric substances (LB-EPS/TB-EPS), proteins (PN) and polysaccharides (PS) in WAS. Results showed that the SRF and SVI values decreased by 86.0% and 80.0%, respectively after photocatalysis treatment for 18 h. The changes of LB-EPS/TB-EPS and morphology of WAS indicated that WAS was degraded in a stepwise and mild manner, in which the sludge pellets were possibly converted into TB-EPS and then LB-EPS. Simultaneously, LB-EPS were degraded into carbon dioxide and water by Ag/TiO₂ photocatalysis.

Key words: Ag/TiO₂; solar photocatalysis; waste activated sludge; dewaterability; settleability

1. Introduction

Large quantities of wastewater is treated successfully by activated sludge technology. Meanwhile, a lot of waste activated sludge (WAS) is produced in this process. Reducing the volume and water content of the WAS is still a major concern (Yuan et al., 2011). The commonly used chemical treatment methods hardly decrease water content below 80%, and the volume of dewatered sludge obviously increases
with the addition of inorganic conditioners (Chen et al., 2001). In order to dispose
WAS economically and efficiently, dewatering and settling processes are essential to
reduce the sludge volume, which is still a bottleneck for sludge treatment (Guan et al.,
2012).

The dewatering and settling characteristics of WAS are different based on
wastewater sources and treatment processes. Furthermore, detailed factors that
influence sludge dewaterability and settleability are not yet well understood (Yu et al.,
2008). Extracellular polymeric substances (EPS) concentration of WAS, proteins and
polysaccharides content of EPS are reported to play a predominant role in sludge
dewaterability and settleability (Chen et al., 2001; Li & Yang, 2007). Moreover, the
layered theory of EPS is proposed to explicate the mechanisms of EPS impact on
WAS dewaterability and settleability. EPS can be divided into loosely bound
extracellular polymeric substances (LB-EPS), tightly bound EPS (TB-EPS) and pellet
(Li & Yang, 2007; Yu et al. 2008).

Based on the theory above, many kinds of methods have been developed to
improve the sludge dewaterability and settleability, including acid or alkaline
treatment (Devlin et al., 2011; Thapa et al., 2009), metal ions (Fe$^{3+}$, Ca$^{2+}$) addition
(Liu & Horn, 2012), electro-chemical treatment (Yuan et al., 2010), thermal treatment
(More et al., 2012), sonication (Feng et al., 2009; Saha et al., 2011), microwave (Tang
et al., 2010), explosive explosion shockwave (Chen & Yang, 2012), pressurised
electro-osmotic (Citeau et al., 2012) and biological treatment (More et al. 2010).

Although these technologies exhibit some enhancement effect on sludge
dewaterability and settleability, some problems limit their application. On the other
hand, photocatalytic technology may be proposed to improve sludge dewaterability
and settleability by degrading EPS in WAS. Moreover, it is eco-friendly compared to
acid or alkaline treatment, metal ions addition and electro-chemical treatment, and
low-energy consumption compared to thermal treatment, sonication, microwave,
explosive explosion shockwave, and pressurised electro-osmotic treatment. It is also a
low-cost method compared to other treatment methods because it uses sunlight.

TiO$_2$ photocatalytic technology, an advanced oxidation process (AOP) utilizing free
radicals as a primary oxidant, has been successfully applied in wastewater treatment
(Gaya & Abdullah, 2008). Many organics can be degraded due to its non-selective
oxidation capability. It is supposed that the pore water and interstitial water in WAS
could be released with the degradation of WAS. In addition, the odour, turbidity and
organics content in WAS can be decreased during the process. Two kinds of
photocatalytic reactors (suspended- and supported-type) can be used in the treatment of
WAS. Although having higher photocatalytic activity, the suspended-type TiO$_2$
photocatalysis has higher cost of post-treatment (Mozia, 2010). Due to lower
interaction efficiency with contaminants and higher recombination rate of
electron–hole, the supported-type TiO$_2$ photocatalysis has lower photocatalytic
activity (van Grieken et al., 2009). Therefore, it is promising to synthesize some novel
and effective immobilized photocatalysts. Ag exhibits an efficient plasmon resonance
effect under sunlight and plays an important role of electron–hole separation produced
by TiO$_2$ (Ma et al., 2012). Thus, several modified TiO$_2$ photocatalysts with Ag-doped
have been developed (Ji et al., 2011). However, the effect of photocatalysis on WAS dewaterability and settleability has not been reported up to now. The objective of this research is to synthesize a novel Ag/TiO$_2$ immobilized as photocatalyst which can be used under sunlight irradiation to improve WAS dewaterability and settleability. The changes of LB-EPS, TB-EPS, proteins (PN) and polysaccharides (PS) in WAS have been investigated during the photocatalytic process in addition to their effects on specific resistance of filtration (SRF) and sludge volume index (SVI) values. In addition, the variation of morphology was compared between the WAS samples before and after photocatalysis by scanning Electron Microscopy (SEM). The related mechanisms, especially the role of EPS in WAS dewaterability and settleability are also discussed.

2. Methods

2.1. WAS and catalyst

The WAS was collected from a domestic wastewater treatment plant in Shimodate, Ibaraki, Japan. The sludge sample was immediately transferred to the lab and stored in a plastic container at 4$^\circ$ for use. The initial characteristics of WAS were as follows (g/l except pH): pH 6.7, total solid (TS) 4.0, volatile suspended solid (VSS) 3.6, total and soluble chemical oxygen demand (TCOD and SCOD) 8.7 and 0.31, respectively. The catalysts used in this study were TiO$_2$ (control, main characteristics, bought from which company, country) and Ag/TiO$_2$ film coated on the inner wall of glass-tube, respectively. The catalyst of Ag/TiO$_2$ was synthesized using a modified impregnation–precipitation–photoreduction method (Ma et al., 2012). Briefly, the
TiO$_2$-film coated glass tubes were impregnated by immersing into AgNO$_3$ (which company, country?) solution (0.5 mol/l) for 20 min with UV light irradiation; then the glass tubes were calcined (300$^\circ$C, 1 h) in a vacuum oven (Hasuc, Shanghai, China); and finally, cooled to room temperature under UV-light. The phase composition and the degree of crystallinity in TiO$_2$ and Ag/TiO$_2$ were determined by X-ray diffraction (XRD). The XRD patterns of the as-prepared samples (2θ ranges from 10$^\circ$ to 90$^\circ$) were recorded at room temperature with scanning speed of 10$^\circ$ min$^{-1}$ using Cu K$\alpha$ radiation ($\lambda$ = 0.154 Å) from a 40 kV X-ray source (Bruker D8 Advance).

2.2. Experimental set-up

All the experiments were carried out in a solar photocatalytic reactor with support catalyst (shown in Fig.1), which contained 10 glass tubes (18 cm in length, 1 cm in diameter) with TiO$_2$ or Ag/TiO$_2$ film coated on inside wall. The working volume of the reactor was 700 ml, and the WAS flowed in the reactor at 100 ml/min by a peristaltic pump (??Which company, country). In this study, sunlight was used as light source, and the mean UV-light intensity (290 nm – 390 nm) in sunlight was recorded from 9:00 to 15:00 (shown in Fig.2(a)). The control experiments were conducted by using the same reactor under the same light conditions but without catalyst on the inner wall of the glass tubes.

2.3. Experimental procedure

The WAS was treated in the solar photocatalytic reactor for 48 h (6 h photocatalysis for one day under the sunlight, then the reactor was covered with a black cloth avoiding sunshine) during September 2012. Sampling was done right after
photocatalysis every day for subsequent analysis. Specific resistance of filtration (SRF) and sludge volume index (SVI) were checked to assess the dewaterability and settleability of the WAS before and after photocatalysis. LB-EPS, TB-EPS, PN and PS concentrations of the sludge samples were also measured to disclose the mechanisms related with the changes of sludge dewaterability and settleability.

2.4. EPS extraction

LB-EPS and TB-EPS were extracted from WAS by a modified heat extraction method (Li & Yang, 2007). The WAS was first centrifuged (MX-301, TMY) at 4000 g for 5 min in a 15-ml centrifuge tube to dewater, then the sludge sediment in the centrifuge tube was re-suspended with NaCl solution (0.05%, 70°C) to the original volume. Without any delay, the sludge suspension was sheared by a vortex mixer (VORTEX-GENIE G-560, scientific industries, INC.) for 2 min, followed by centrifugation at 4000 g for 10 min. The organic matter in the supernatant was the LB-EPS of WAS. For the TB-EPS, the WAS was firstly centrifuged at 4000 g for 5 min in a 15-ml centrifuge tube, then the liquid phase was discarded and the solid phase was re-suspended with 0.05% NaCl solution to its original volume of 15 ml. Secondly, the WAS suspension was heated to 60°C (confirm this temperature, 70?) for 30 min in an incubator (WFO-700, EYL4). Finally, the re-suspension was centrifuged (MX-301, TMY) at 4000 g for 15 min and the supernatant was collected for TB-EPS analysis. The residue in the centrifuge cube was again re-suspended by adding NaCl solution (0.05%, 70°C) to the original volume of 15 ml. This fraction was the pellet.

2.5. Indices and analytical methods
The photocatalytic activity of the TiO$_2$ and Ag/TiO$_2$ was assessed by the degradation rate of methyl orange (company name, country). The changes of WAS dewaterability and settleability were assessed based on the specific resistance of filtration (SRF) and sludge volume index (SVI), respectively before and after treatment. The SRF was measured using a slightly modified method of Li & Yang (2007). The SRF(cm/g) of the sludge was calculated by

$$SRF = 2 \cdot b \cdot p \cdot A^2/(\mu C)$$

where $p$ (60 kPa) is the pressure applied, $A$ ($3.0 \times 10^{-4}$ m$^2$) is the filter area, $\mu$ (1.0mPa s) is the viscosity of the permeate, $C$ is the sludge concentration in mixed liquor suspended solids (MLSS,kg/m$^3$) and $b$ (s/m$^6$) is the time-to-filtration ratio, which is the slope of the curve obtained by plotting the ratio of the time of filtration to the volume of filtrate ($t/V$) versus the filtrate volume ($V$).

The SVI value of WAS was obtained by measuring the sludge volume change in a 100-ml cylinder (100ml, ARROW) after 30 min settlement together with MLSS concentration.

The morphology of untreated or photocatalysis treated WAS was obtained by scanning electronic microscope (SEM, XL30, Philips, Holland). EPS (LB-EPS and TB-EPS) concentrations were analyzed for COD, proteins (PN) and polysaccharides (PS). COD and MLSS were detected in accordance with the standard methods (Pawlowski, 1994). Phenol sulfuric method (Mecozzi, 2005) with glucose as standard and Lowry method (Dawson & Heatlie, 1984) with bovine serum albumin as standard were used to determine the concentrations of PS and PN, respectively.
3. Results and discussion

3.1. Characterization and photocatalytic capacity of TiO$_2$ and Ag/TiO$_2$

The XRD patterns of pure TiO$_2$ (1) and Ag/TiO$_2$ (2) samples are shown in supplementary Fig.S1(a). The XRD patterns indicate that anatase is the only crystalline phase of TiO$_2$ in the pure TiO$_2$ sample. The presence of Ag diffraction lines (peaks marked by arrowhead in supplementary Fig.S1(a)) was clearly detected at approximately 22.55° (2θ) for the Ag/TiO$_2$ sample.

The photocatalytic activity of TiO$_2$ and Ag/TiO$_2$ was assessed by the degradation of methyl orange (MO, 20 mg/l) under the sunlight for 2 h from 11:00 to 13:00. The UV-intensity (290 nm – 390 nm) is shown in Fig.2(b) and the degradation rate of MO is shown in supplementary Fig.S1(b). It was found that Ag/TiO$_2$ exhibited much higher MO degradation rate (99.0%) than pure TiO$_2$ (35.2%), and the MO degradation could be negligible under single sunlight condition. This observation indicates that Ag/TiO$_2$ possesses better photocatalytic activity than pure TiO$_2$. The improvement is probably owing to the Ag particles deposited on the TiO$_2$ acting as electron–hole separation and interfacial charge transfer (van Grieken et al., 2009). The charge separation resulted from the formation of Schottky barriers at the Ag/TiO$_2$ interaction region is attributable to the electron transfer from the TiO$_2$ conduction band to silver particles (van Grieken et al., 2009). Due to the fact that the trapping of electrons by Ag deposits is faster than its recombination with holes (Krejčíková et al., 2012), the silver deposits on the surface can accelerate the transfer of trapped electrons in the Ag deposits to the oxygen molecules to form superoxide radicals in solution and then
improve the photocatalytic activity (Pulido Melián et al., 2012). Because of having higher photocatalytic activity than TiO₂, Ag/TiO₂ was chosen as photocatalyst in the following experiments.

3.2. Effect of Ag/TiO₂ photocatalytic treatment on sludge dewaterability

The effect of Ag/TiO₂ photocatalytic treatment on sludge dewaterability was evaluated by measuring SRF value (Fig.3a). It can be seen that the sludge dewaterability could be greatly enhanced after Ag/TiO₂ photocatalytic treatment for 18 h. Further prolonging the photocatalysis duration has little enhancement effect. The SRF value decreased from $2.42 \times 10^9$ cm/g (untreated WAS) to $3.4 \times 10^8$, $3.83 \times 10^8$ and $4.7 \times 10^8$ cm/g after photocatalytic treatment for 18 h, 24 h and 36 h, respectively. Therefore, in this study, 18 h was chosen as the optimal photocatalysis time, under which the SRF value could be reduced by 86.0%.

The above effect of photocatalysis on sludge dewaterability can be explained as follows. The flocs of WAS are in layered structure (Nguyen et al., 2008), in which the loose surface layer can be first damaged and dispersed into the aqueous solution and then quickly oxidized into carbon dioxide by photocatalysis within a few hours. Later the free water, pore water and some bound water are released resulting in rapid decrease in SRF value. With the degradation of the loose surface layer, the highly porous sludge flocs can become compacted spheroidal structure thus the flocs density increases, which further improve the dewaterability of WAS (Jin et al., 2003). Along with the photocatalysis process, the spheroidal structure of flocs is disrupted and EPS is released gradually. Simultaneously, the released EPS can be degraded by Ag/TiO₂
photocatalysis, which may contribute to the fluctuation of SRF values during the 24 and 48 h treatment. This observation is in some agreement with the finding of Feng et al. (2009) who treated WAS by ultrasound conditioning and pointed out that sludge dewaterability could be slightly enhanced at low specific energy dosages while significantly deteriorated at larger specific energy dosages (>4400 kJ/kg TS).

3.3. Effect of Ag/TiO₂ photocatalytic treatment on sludge settleability

The settleability, indicated by SVI, is an important factor influencing the operation of sewage treatment plant. The change of SVI values is shown in Fig.3(b). Clearly, the SVI decreased quickly from 161.4 ml/g to 86.7 ml/g and 58.7 ml/g after photocatalysis for 12 h and 18 h, and increased slightly to 69.4 ml/g at 36 h, respectively. On the other hand, the SVI value of the untreated WAS (control) fluctuated between 159.1 ml/g and 172.1 ml/g during the 48 h photocatalysis treatment. After 18 h photocatalysis treatment, the sludge settleability was comparable to that of thermophilic aerobic granular biomass (SVI = 60 ml/g) (Zitomer et al., 2007).

As it is known, the WAS has high SVI value due to some unfavorable operation conditions resulting in slowly settleable large flocs (supplementary Fig.S2(a)). However, the loose surface of flocs can be changed by Ag/TiO₂ photocatalysis as the reaction progresses. The flocs become compact granule-like structure and the surface roughness reduces, and then the frictional resistance between flocs begins to decline during the settlement process. When the flocs are severely destructed and the EPS are released gradually, the sludge density become smaller and the particles surface area
become larger resulting in increased buoyancy force, ultimately leading to a slight increase in SVI value (Jin et al., 2003). Meanwhile, the released EPS may be degraded by Ag/TiO₂ photocatalysis subsequently. That is, the concentration of EPS could have some contribution to the fluctuation of SVI values between the 24 h and 48 h photocatalysis.

3.4. Changes of EPS, PN and PS concentrations in WAS during Ag/TiO₂ photocatalysis process

3.4.1 Changes of LB-EPS, PN and PS concentrations in WAS

The changes in LB-EPS (expressed in COD), PN and PS concentrations of the WAS are shown in Fig.4. The concentrations of LB-EPS and PN in LB-EPS (LB-EPS/PN) declined from the initial 364.0 mg/l and 248.2 mg/l to 210.8 mg/l and 164.3 mg/l at 18 h, and increased to 269.1 mg/l and 221.0 mg/l at 24 h, and then decreased to 220.6 mg/l and 182.7 mg/l at 36 h, respectively. However, the concentrations of LB-EPS and LB-EPS/PN increased to 439.5 mg/l and 306.5 mg/l at 48 h, respectively. On the other hand, the concentration of PS in LB-EPS (LB-EPS/PS) decreased from the start to the end of the Ag/TiO₂ photocatalytic treatment. When the SRF and SVI reached the minimal values after 18 h photocatalysis yielding its maximum dewaterability and settlability, the LB-EPS concentration in the treated sludge was 210.8 mg/l (Figs.3 and 4), less than the result (400-500mg/l) obtained by Feng et al. (2009). Moreover, during the first 36 h duration of Ag/TiO₂ photocatalysis, LB-EPS concentration was found to have a similar change tendency with the SRF and SVI values of the sludge (Figs.3 and 4).
The above phenomena can be interpreted as that the widely dispersed LB-EPS in the aqueous phase has more chance to interact with the photocatalyst, possibly leading to its rapid degradation into carbon dioxide. With the degradation of LB-EPS, an abundant amount of bound water is released, resulting in the decrease of SRF and SVI values. The main components of LB-EPS are PN and PS, and the concentration of PN is much higher than that of PS (Yu et al., 2008), which may be the reason why PN concentration changed in a similar tendency as LB-EPS did. When the sludge flocs are destroyed further by Ag/TiO₂ photocatalysis, more EPS and cellular substances can be released into the aqueous phase, bringing about the increase in protein and polysaccharide levels. On the other hand, EPS may be released from the pellets of WAS along with its degradation. That is, the degradation and release rate of LB-EPS in WAS dominates the SVI and SRF values.

3.4.2 Changes of TB-EPS, PN and PS concentrations in WAS

During the first 12 h photocatalysis, the concentrations of TB-EPS and PN in TB-EPS (TB-EPS/PN) decreased slightly from 470.8 mg/l and 430.0 mg/l, and their concentrations varied with the same tendency as the SVI and SRF values of WAS (Fig. 4). After that the concentrations of TB-EPS and TB-EPS/PN increased significantly till the 24 h, and then decreased. This observation is inconsistent with the variation of SVI and SRF values (Figs. 3 and 4). Still the concentration of PS in TB-EPS (TB-EPS/PS) decreased gradually during the whole photocatalysis process.

The reduction of TB-EPS in the initial phase is probably due to the conversion of TB-EPS to LB-EPS by Ag/TiO₂ photocatalysis, and during this period the WAS
pellets haven’t been disrupted into TB-EPS or LB-EPS. With the photocatalysis going on, the pellets are disrupted, and then more and more LB-EPS and TB-EPS are released, leading to the increase of TB-EPS in WAS. Simultaneously, the increase of TB-EPS can supply more LB-EPS. When the increasing portion of TB-EPS is more than its decreasing counterpart, the concentration of TB-EPS will increase and vice versa. However, no direct correlation has been found between TB-EPS and the SVI or SRF value in this study. LB-EPS content appears to have a closer correlation with the sludge characteristics in settleability and dewaterability than the TB-EPS, which agrees with the finding of Yuan et al. (2011). Detailed and followed-up research is necessary.

3.5. Effect of Ag/TiO₂ photocatalytic treatment on the morphological change of WAS

The morphological change between untreated and photocatalysis treated WAS samples is obvious (supplementary Fig.S2). From the direct observation as shown in supplementary Fig.S2(a), the color of sludge was changed from the original dark-gray to the earth-yellow, and the concentration of flocs in WAS decreased after treatment by Ag/TiO₂ photocatalysis. In addition, the obvious difference in the microstructures of WAS could be observed by SEM. The surface of untreated WAS sample (supplementary Fig.S2(b)) had a relatively rough surface and layered structure, while the surface of photocatalysis treated WAS (after 18 h treatment) became level and smooth (supplementary Fig.S2 (c)). This morphological change indicates that the WAS could be degraded gradually by Ag/TiO₂ photocatalysis. When the flocs in WAS are gradually degraded from surface to inside, free water and pore water can be
released and the frictional resistance between flocs decreases, which may contribute to the decrease of SRF and SVI values. This mechanism is different from other sludge treatment methods such as explosive explosion shockwave, microwave and ultrasonic processes characterized as rapid and violent (Chen & Yang, 2012; Feng et al., 2009; Tang et al., 2010). The appearance of photocatalysis treated WAS (for 36 h) became rough, fluffy and irregularly shaped again, but not the layered structure as before (supplementary Fig.S2 (d)). This observation indicates that the sludge flocs might be broken, and the intracellular substances could be solubilized into the aqueous phase, leading to the increase of EPS. Meanwhile, some of the EPS, especially LB-EPS, can be degraded by Ag/TiO$_2$ photocatalysis (shown in Fig.4 (a)). So the physically bound water and interstitial cell water can be released into the solution during the degradation progress of EPS, enhancing sludge dewaterability and settleability.

4. Conclusions

A photocatalyst of Ag/TiO$_2$ film was synthesized and immobilized successfully, and its photocatalytic activity was significantly higher than TiO$_2$ film. The effect of Ag/TiO$_2$ photocatalysis on WAS dewaterability and settleability was dependent on photocatalytic time. The optimal photocatalytic time was 18 h with Ag/TiO$_2$ as photocatalyst, and the SRF and SVI values were reduced by 86.0% and 80.0%, respectively. The LB-EPS and PN had more positive relation with SRF and SVI during the first 36 h photocatalysis. The EPS change and SEM images of WAS indicated that WAS were degraded by Ag/TiO$_2$ photocatalysis in a stepwise and mild manner.
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Supplementary data

The supplementary data associated with this article included Fig.S1 (XRD patterns and photocatalytic activity of pure TiO\textsubscript{2} and Ag/TiO\textsubscript{2}) and Fig.S2 (morphological changes of WAS).

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Figure captions

Fig. 1. Schematic of Ag/TiO$_2$-coated glass tubular photocatalytic reactor.

Fig. 2. UV-light mean intensity used in WAS treatment (a) and used in methyl orange treatment (b).

Fig. 3. Effect of Ag/TiO$_2$ photocatalytic treatment on sludge dewaterability (a) and settleability (b).

Fig. 4. Changes of LB-EPS and TB-EPS concentrations in WAS during the Ag/TiO$_2$ photocatalysis process (a); Changes of PN and PS in LB-EPS and TB-EPS during the Ag/TiO$_2$ photocatalysis process (b).
Fig. 1. Liu et al.
Fig. 2. Liu et al.
Fig. 3(a). Liu et al.

Fig. 3(b). Liu et al.
Fig. 4(a). Liu et al.

Fig. 4(b). Liu et al.