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Original research article

Oxidative treatment of waste activated sludge by different activated persulfate systems for enhancing sludge dewaterability



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ABSTRACT

The enhancement in dewaterability of waste activated sludge (WAS) by oxidative treatment using thermally- and alkali-activated persulfates (i.e., peroxymonosulfate (PMS) and peroxydisulfate (PDS)) was studied with two indices representing dewaterability change, i.e., centrifuged weight reduction (CWR) and standardized-capillary suction time (SCST). The tested conditions include 50 °C/PMS, 50 °C/PDS, 80 °C/PMS, and 80 °C/PDS as thermally-activated persulfate systems and NaOH/PMS, NaOH/PDS, KOH/PMS, and KOH/PDS as alkali-activated persulfate systems. The oxidation by activated persulfates caused the disintegration of bacterial cells and extracellular polymeric substance (EPS) of WAS, affecting the sludge dewaterability. The highest dewaterability was found at the KOH/PDS treatment in CWR and at the 80 °C/PDS treatment in SCST. The EPSs were stratified as soluble, loosely-bound (LB) and tightly-bound (TB) fractions, and contents of protein and polysaccharide in each fraction were measured to characterize the EPS matrix before and after treatments. The statistical analysis of the relationship between EPS character and dewaterability indicated that the protein content in LB-EPS was the dominant negative factor for the dewaterability represented by SCST, whereas the polysaccharide content in soluble-EPS was identified as the dominant positive factor for the dewaterability by CWR.

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1. Introduction

Over the last few decades, the amount of municipal and industrial wastewater has tremendously increased worldwide along with rapid industrialization and urbanization. As a cost-effective method, the activated sludge process has been widely used to treat various types of wastewaters. However, massive production of waste activated sludge (WAS) has become another big challenge for environmental engineers to resolve. In response to this challenge, investigators have been conducting extensive research on the volume reduction and reuse of WAS [1–4].

WAS consists of microorganisms with various organic/inorganic substances and has a high water content (over 90%) [5]. In order to reduce the volume of WAS in wastewater treatment plants (WWTPs), the primarily produced WAS is mechanically dewatered

by gravity settling, centrifugation or filtration/compression using a filter (or a belt) press. However, bacterial cells and extracellular polymeric substances (EPSs) in WAS retain water, inhibiting the sludge dewatering process [6,7]. As a result, the sludge treatment cost (for dewatering and disposal) accounts for up to 60% of total operating cost of wastewater treatment processes [8].

A proper pretreatment to disintegrate cells and EPSs in WAS is beneficial to enhance the dewaterability of WAS and subsequently reduce the sludge treatment cost in WWTPs. Several methods for WAS disintegration have been investigated, e.g., chemical oxidation, sonication, alkali-treatment, and their combined applications [9–12]. In particular, chemical oxidation is effective in disrupting cell membranes and destructing the polymeric structures of EPSs [6,13–18].

Recently, persulfates (i.e., peroxymonosulfate (PMS) and peroxydisulfate (PDS)) are gaining attention as new chemical oxidants for environmental applications including wastewater treatment, soil and groundwater remediation, sludge pretreatment, etc. [14–21]. Persulfates themselves are strong oxidants that favor two-electron oxidation ($E^0(S_2O_8^{2-}/SO_4^{2-}) = 1.96 V_{SHE}$; $E^0(HSO_5^-/SO_4^{2-}) = 1.75 V_{SHE}$) [22,23]. Persulfates can also be converted into

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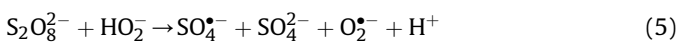
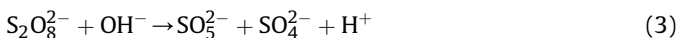
sulfate radical anion ($\text{SO}_4^{\bullet-}$) by different ways such as thermal activation, alkali activation, transition metal catalysis, and photocatalysis [24–28]. $\text{SO}_4^{\bullet-}$ has a high redox potential for one-electron transfer ($E^0(\text{SO}_4^{\bullet-}/\text{SO}_4^{2-}) = 2.43 \text{ V}_{\text{SHE}}$) [29], and is capable of rapidly oxidizing a wide spectrum of organic compounds [30].

Several studies have reported the application of activated persulfate systems to WAS dewatering. Zhen et al. have demonstrated that the Fe(II)-catalyzed PDS oxidation increased the dewaterability of WAS and the system performance could be further improved by supplying heat or electricity [14–17]. PDS activated by zero valent iron also has been shown to enhance the sludge dewaterability [18]. However, with aforementioned studies only limited to iron-activated PDS systems, little is known about the feasibility of thermal- and alkali-activated persulfate systems for WAS dewatering even if these systems could be good available alternatives to the use of iron.

It is well known that the thermal activation of PDS yields 2 M equivalents of $\text{SO}_4^{\bullet-}$ (Eq. 1) [31]. In a mechanism analogous to the PDS activation, PMS should produce $\text{SO}_4^{\bullet-}$ and hydroxyl radical ($\bullet\text{OH}$) by thermal activation (Eq. 2). However, it is not clearly documented whether PMS produces reactive radical species by thermal activation. Obviously, elevating temperature will also accelerate the direct non-radical reactions of PDS and PMS with organic substrates.



Under strong alkaline conditions, the decomposition of PDS is accelerated generating $\bullet\text{OH}$ (Eqs. 3–6) [25]. PMS also has been suggested to undergo the alkali-activation to form $\bullet\text{OH}$ as an intermediate [32]. However, its detailed mechanism is not clearly elucidated.



The objectives of this study are: i) to comparatively assess the potential of thermally- and alkali-activated persulfate systems for enhancing the dewaterability of WAS, and ii) to identify the relationship between EPS character and WAS dewaterability.

2. Materials and methods

2.1. WAS sample and reagents

WAS was sampled at the end of secondary clarifier from the Gul-Hwa wastewater treatment plant located in Ulsan, South Korea. Sampled WAS was thickened by gravity settling for 24 h in the laboratory and stored at 4 °C prior to use. The thickened WAS sample had $14.1 \pm 0.6 \text{ g L}^{-1}$ of volatile suspended solids (VSS) with $\text{pH } 6.7 \pm 0.2$.

Chemicals used in this study include potassium hydroxide, sodium hydroxide (Daejung Chemical), sodium peroxydisulfate, potassium peroxymonosulfate, potassium iodide, sodium bicarbonate, phenol, sulfuric acid, sodium citrate, copper(II) sulfate, sodium hydroxide, folin & ciocalteu's phenol reagent, sodium phosphate dibasic, sodium

phosphate monobasic, potassium chloride, sodium chloride, albumin from bovine serum, and D-(+)-glucose (Sigma–Aldrich). All chemicals were of reagent grade and used without further purification. All solutions were prepared in deionized water ($> 18 \text{ M}\Omega \text{ cm}$) produced from a Milli-Q ultrapure water-purification system (Millipore). Stock solutions of persulfates (250 mM for PMS and PDS, respectively) were prepared prior to experiments.

2.2. Treatment of WAS by activated persulfate systems

Persulfate treatment with thermal- and alkali-activation was conducted in a 100 mL solution containing WAS; a 10 mL of 250 mM persulfate stock solution was added into 90 mL WAS sample (25 mM). For thermal activation, the solution temperature was elevated to 50 or 80 °C in 3 min, and maintained constant ($\pm 2 \text{ }^\circ\text{C}$) during the entire reaction time of 1 h. The pH of WAS decreased from 6.7 to 2.8 and 5.4 after treatments by PMS and PDS, respectively.

Alkali-activation was conducted using 2.5 M NaOH or KOH at 25 °C. Pre-weighed powder of NaOH or KOH (10 g NaOH or 14 g KOH) was directly added into a 100 mL solution containing persulfate and WAS, and the solution was stirred for 1 h for reaction. The pH was maintained at approximately 13.2–13.7 during the treatment.

2.3. Analyses

2.3.1. Dewaterability

Two indices (i.e., centrifuged weight reduction (CWR) and standardized-capillary suction time (SCST)) were used to evaluate the change of WAS dewaterability by different treatments. CWR indicates the weight reduction percentage of the centrifuged WAS before and after the treatment. To measure the centrifuged weight (CW) of WAS, 40 mL of the WAS sample was centrifuged at 3000 g for 30 min, then the supernatant was gently discarded and the remaining sludge sediment was weighed. The CW values for raw and treated WAS samples (CW_{raw} and $\text{CW}_{\text{treated}}$, respectively) were analyzed, and the CWR values were calculated by Eq. 7. The positive CWR value indicates the enhancement of WAS dewaterability.

$$\text{CWR (\%)} = \left(1 - \frac{\text{CW}_{\text{treated}}}{\text{CW}_{\text{raw}}}\right) \times 100 \quad (7)$$

SCST was used as another index for the dewaterability change, which represents the filterability using a filter press type dewatering device. Capillary suction time (CST) for WAS was measured by the Type 304B capillary suction timer (Triton) equipped with a 10 mm inner diameter funnel. SCST values were calculated by Eq. 8 using the CST values for raw and treated WAS samples. The SCST value higher than unity indicates the enhancement of dewaterability.

$$\text{SCST} = \frac{\text{CST}_{\text{raw}}}{\text{CST}_{\text{treated}}} \quad (8)$$

2.3.2. Stratification and analysis of EPS

The protocol of EPS extraction was based on the method suggested by Yu et al. [33] with a slight modification. The procedure is briefly described below. The WAS sample (30 mL) was taken and centrifuged at 2000 g for 15 min. The supernatant was gently poured into a separate bottle (soluble EPS). The remaining sediment was re-suspended to the original volume with a prepared buffer solution ($\text{Na}_2\text{PO}_4:\text{NaHPO}_4:\text{NaCl}:\text{KCl} = 2:4:9:1$ in molar ratios), and the suspension was centrifuged at 5000 g for 15 min. The

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