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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 extracelluar 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 extracelluar 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

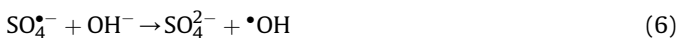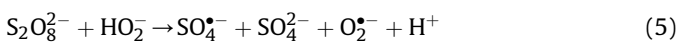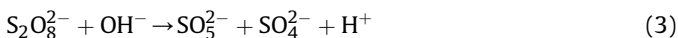* Corresponding author.
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sulfate radical anion ($SO_4^{\bullet-}$) by different ways such as thermal activation, alkali activation, transition metal catalysis, and photocatalysis [24–28]. $SO_4^{\bullet-}$ has a high redox potential for one-electron transfer (E^0 ($SO_4^{\bullet-}/SO_4^{2-}$) = 2.43 V_{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 $SO_4^{\bullet-}$ (Eq. 1) [31]. In a mechanism analogous to the PDS activation, PMS should produce $SO_4^{\bullet-}$ and hydroxyl radical ($^{\bullet}OH$) 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.

$$S_2O_8^{2-} \xrightarrow{\Delta} 2SO_4^{\bullet-} \tag{1}$$

$$HSO_5^- \xrightarrow{\Delta} {}^{\bullet}OH + SO_4^{\bullet-} \tag{2}$$

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

$$S_2O_8^{2-} + OH^- \rightarrow SO_5^{2-} + SO_4^{2-} + H^+ \tag{3}$$

$$SO_5^{2-} + OH^- \rightarrow HO_2^- + SO_4^{2-} \tag{4}$$

$$S_2O_8^{2-} + HO_2^- \rightarrow SO_4^{\bullet-} + SO_4^{2-} + O_2^{\bullet-} + H^+ \tag{5}$$

$$SO_4^{\bullet-} + OH^- \rightarrow SO_4^{2-} + {}^{\bullet}OH \tag{6}$$

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 ± 0.6 g L^{-1} of volatile suspended solids (VSS) with pH 6.7 ± 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 MΩ 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 (± 2 °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 $CW_{treated}$, respectively) were analyzed, and the CWR values were calculated by Eq. 7. The positive CWR value indicates the enhancement of WAS dewaterability.

$$CWR (\%) = \left(1 - \frac{CW_{treated}}{CW_{raw}}\right) \times 100 \tag{7}$$

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.

$$SCST = \frac{CST_{raw}}{CST_{treated}} \tag{8}$$

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 (Na_2PO_4:$NaHPO_4$:NaCl:KCl = 2:4:9:1 in molar ratios), and the suspension was centrifuged at 5000 *g* for 15 min. The

supernatant was separated as loosely-bound EPS (LB-EPS). Again, the remaining sediment was re-suspended to the original volume with the buffer solution. The suspension was sonicated by an ultrasonic homogenizer (STH-750S, Sonictopia) at 20 kHz, 480 W for 10 min, and then centrifuged at 20,000 g for 20 min. The supernatant was separated as tightly bound EPS (TB-EPS). All the procedures for EPS extraction were conducted at around 4 °C to minimize the denaturing of samples.

EPS samples were filtered by a 0.45 μm hydrophilic polytetrafluoroethylene (PTFE) membrane filter (HP045AN, Advantec MFS) before analysis. The filtrates were used to measure concentrations of protein and polysaccharide. To quantify the protein content in each EPS fraction, Lowry method was used [34]. Polysaccharide in each EPS fraction was analyzed by phenol-sulfuric acid method using D-(+)-glucose as a standard [35].

2.3.3. Statistical analysis

Pearson's correlation analysis was conducted to investigate the linear relationship between EPS contents and dewaterability change indices. Pearson's correlation coefficients (R_p) were presented for different correlations of two variables. The R_p value ranges from -1 (perfect negative linear relationship) to $+1$ (perfect positive linear relationship), and the 0 value signifies no relationship between the two variables. To verify the reliability of R_p values, the confidence interval (p-value) was also calculated, e.g., it is considered that the R_p value is statistically sound with 95% of confidence interval when p-value is smaller than 0.05. The software Sigmaplot 12.0 (Systat Software) was used for the analysis.

2.3.4. VSS and persulfate

VSS was measured by following the guidelines of the Standard Methods [36]. The residual concentration of persulfate after the reaction was measured by iodometry [37].

3. Results and discussion

3.1. Decomposition of persulfates in WAS

Using the iodometric analysis, concentrations of residual persulfates were measured after 1 h reaction under different conditions with and without thermal- or alkali-activation. In general, PMS was decomposed to a greater extent than PDS. At room temperature, 22% of initial PMS was decomposed by the reaction with the WAS components, whereas PDS was decomposed by only 3%. When the temperature was elevated to 50 °C, the decomposition efficiencies of PMS and PDS were 65 and 10%, respectively. At 80 °C, the decomposition efficiencies of PMS and PDS increased up to 80 and 83%, respectively.

With the addition of bases for alkali-activation, PMS was completely decomposed in 1 h. However, PDS was decomposed by 89% with NaOH and by 97% with KOH. The higher decomposition efficiency of PDS with KOH than NaOH appears to result from the higher molar enthalpy of solution of KOH (-57.6 kJ mol^{-1}) than that of NaOH (-44.5 kJ mol^{-1}) [38]. Indeed, the temperature of WAS samples increased to 50.7 °C upon the addition of KOH, whereas the addition of NaOH increased the temperature to 42.6 °C. The WAS samples containing bases were slowly cooled down during the treatment, and the average temperatures were 37.3 and 33.6 °C for KOH and NaOH-treated samples, respectively.

3.2. Effect of persulfate treatment on WAS dewaterability

The change of WAS dewaterability by persulfate treatment was evaluated by CWR and SCST (Fig. 1a and b). The WAS dewaterability represented by CWR increased under all treatment conditions (Fig. 1a). The alkali-activated persulfate systems generally showed greater CWR values (66–76%) than the thermally-activated systems (29–68%). For thermally-activated persulfate systems, PMS showed higher CWR values than PDS. For alkali-activation, PDS exhibited a better performance than PMS even though PMS was completely decomposed during the treatment. This observation implies that the alkali-catalyzed decomposition of PMS does not significantly produce reactive radical species to disintegrate WAS components; a non-radical decomposition mechanism could be dominant. Meanwhile, alkali-treatments without persulfate exhibited 50–54% CWR values, indicating that the alkaline lysis of cells accounts for an important portion of CWR by alkali-activated persulfates systems.

In terms of SCST, the dewaterability decreased in most of treatment conditions (i.e., the SCST values are lower than unity) except for the thermally-activated PDS system at 80 °C (Fig. 1b). The PDS system at 80 °C dominates the production of $SO_4^{\bullet-}$ and the oxidation by $SO_4^{\bullet-}$ is mainly responsible for the increase of SCST. It appears that the $SO_4^{\bullet-}$ non-selectively oxidizes the WAS components including cell structures, EPSs, and intracellular substances released from damaged cells, rendering the WAS medium less viscous.

In contrast, the thermally-activated PMS system at 80 °C did not increase SCST even though the amount of PMS consumed was similar to that of PDS at 80 °C (refer to the Section 1). This result may imply that the thermally-activated PMS system relies on the direct non-radical reactions of PMS (likely selective reactions) rather than the reactions of reactive radical intermediates such as $SO_4^{\bullet-}$ and $^{\bullet}OH$ (non-selective reactions).

Meanwhile, a notable observation is that all the alkali-treatments (with and without persulfates) drastically suppressed the SCST value. The alkaline lysis of cells may release small colloidal EPSs that render the WAS medium more viscous, subsequently decreasing the SCST value. However, such small colloidal substances are readily removed by centrifugation (remaining in the supernatant solution), leading to high CWR values.

3.3. Effect of persulfate treatment on EPS fractions

EPSs in raw and treated WAS samples were fractionated as soluble EPS, LB-EPS, and TB-EPS, and protein and polysaccharide in each EPS fraction were quantified (Figs. 2 and 3).

In almost all the treated samples, concentrations of protein and polysaccharide in soluble EPS and LB-EPS increased compared to untreated controls (Fig. 2a and b for protein, Fig. 3a and b for polysaccharide). However, concentrations of protein and polysaccharide in TB-EPS decreased after treatment in most of cases (except four conditions in Fig. 2c and one condition in Fig. 3c). These observations collectively indicate that such treatments disrupt and disintegrate the sludge flocs, shifting the fraction of EPS from TB- to soluble or LB-EPS. The increase of TB-EPS found in a few treatment cases may suggest that a certain fraction of EPS is strongly-bound to cells or trapped in some rigid matrix (not even released by sonication for the TB-EPS analysis), and is shifted to the TB-EPS fraction after thermal or alkali-treatments.

Alkali-activated persulfate treatments tend to increase soluble and LB-EPS more effectively than thermally-activated treatments, which may be explained by the oxidative degradation of EPS. Chemical oxidation does not only dissolve EPS but also degrade EPS. As discussed earlier, the WAS disintegration by alkali-activated persulfate systems relies on the alkaline cell lysis rather than oxidation, which minimizes the loss of released EPSs by oxidative degradation.
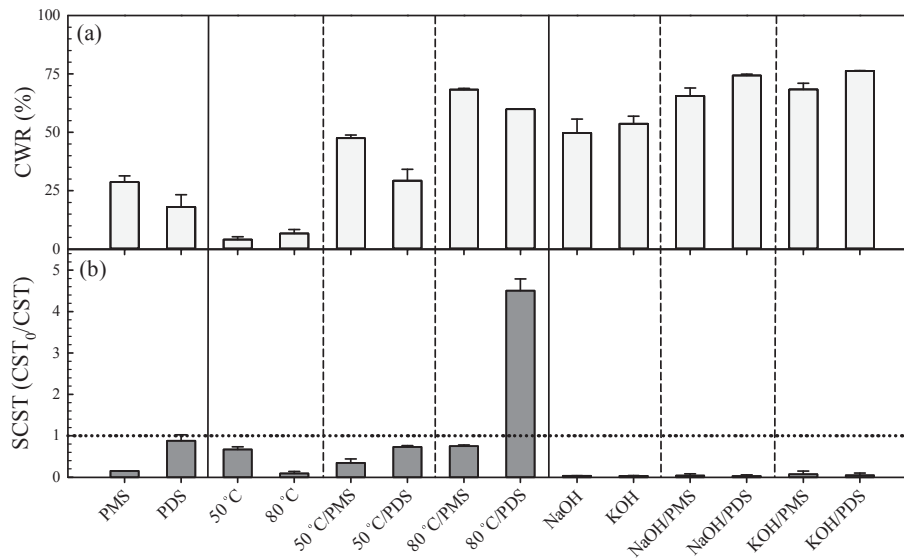
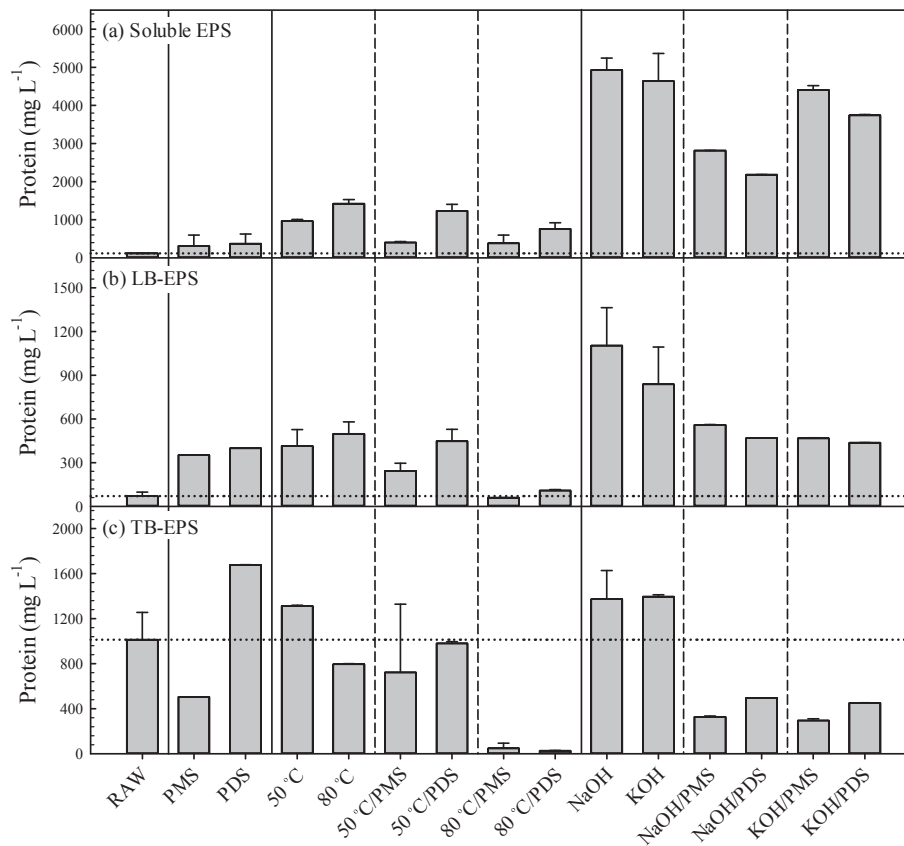**Fig. 1.** Dewaterability change in WAS by different treatments.

Fig. 2. Protein contents in different EPS fractions of WAS before and after treatment.

3.4. Pearson's correlation analysis

Pearson's correlation analysis was conducted by correlating EPS contents (Figs. 2 and 3) with dewaterability change indices (Fig. 1). The correlations between six variables of EPS content (i.e., protein and polysaccharide contents in soluble, LB-, and TB-EPS fractions) and two variables of dewaterability change index (i.e., SCST and CWR) were analyzed for three groups of treatment conditions (i.e.,

PMS, PDS, and PMS + PDS) ($6 \times 2 \times 3 = 36$ cases total). The R_p and the p-value were presented for each case as shown in Table 1.

Strong negative correlations ($R_p < -0.9$) were found between the protein content in LB-EPS and SCST in Group 1 and 2. This observation is consistent with a previous finding that protein in LB-EPS is a critical factor that lowers the dewaterability represented by CST [7]. However, no correlation between the protein content in LB-EPS and SCST was found when the data for Group 1 and 2 were used
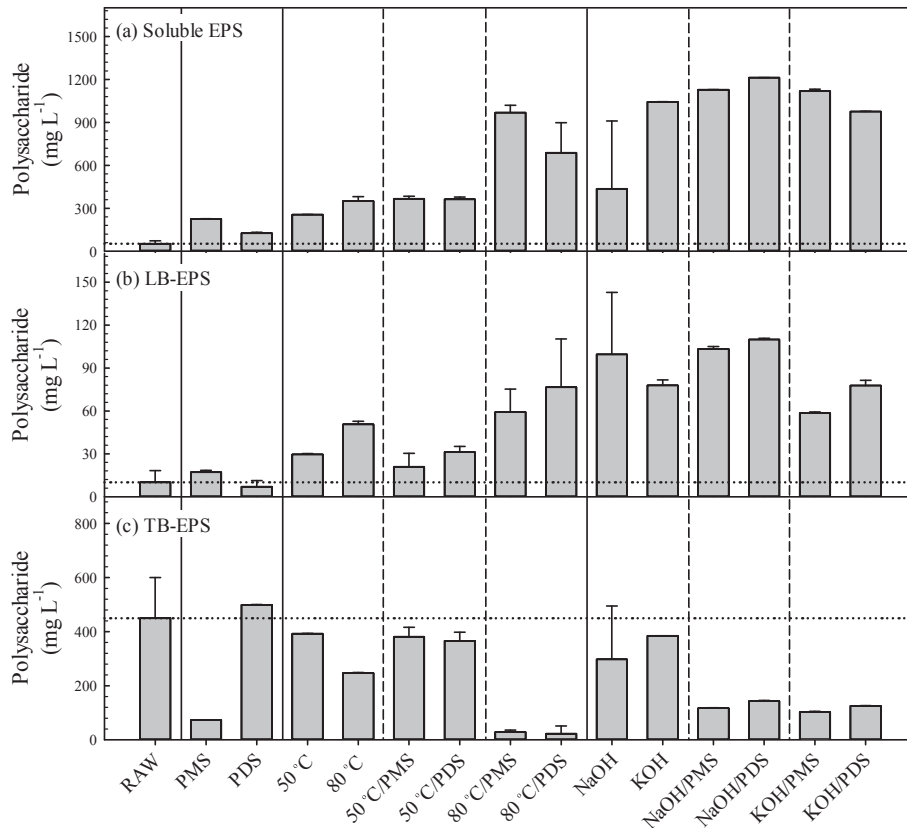
Fig. 3. Polysaccharide contents in different EPS fractions of WAS before and after treatment.

Table 1
Pearson's correlation analysis[a] between EPS contents and dewaterability change indices.

		Group 1 (PMS)[b]		Group 2 (PDS)[c]		Group 3 (PMS + PDS)[d]	
		SCST	CWR	SCST	CWR	SCST	CWR
Protein	Soluble EPS	−0.619 (0.265)	0.592 (0.293)	−0.534 (0.354)	0.76 (0.136)	−0.395 (0.145)	0.52 (0.0471)
	LB-EPS	**−0.958 (0.0104)**	0.35 (0.563)	**−0.988 (0.00154)**	0.289 (0.638)	−0.468 (0.0787)	0.193 (0.491)
	TB-EPS	−0.408 (0.495)	−0.682 (0.204)	−0.4156 (0.44)	−0.829 (0.083)	−0.286 (0.301)	−0.599 (0.0184)
Polysaccharide	Soluble EPS	−0.0399 (0.949)	**0.945 (0.0153)**	−0.186 (0.764)	**0.964 (0.0083)**	−0.145 (0.605)	0.883 (0.0000131)
	LB-EPS	−0.19 (0.76)	0.769 (0.128)	0.0202 (0.974)	**0.949 (0.0137)**	−0.629 (0.824)	0.739 (0.00164)
	TB-EPS	−0.0907 (0.885)	−0.237 (0.702)	−0.42 (0.481)	−0.885 (0.046)	−0.2 (0.474)	−0.665 (0.00681)

[a] Pearson's correlation coefficients (R_p) are presented with p-values (numbers in parentheses). Strong correlations ($|R_p| > 0.9$) are indicated in bold.
[b] Group 1: Data for treatments using PMS (including thermally- and alkali-activated systems) were used.
[c] Group 2: Data for treatments using PDS were used.
[d] Group 3: All the data in Group 1 and 2 were used.

together (Group 3); the slopes of the two regression lines for Group 1 and 2 are quite different (Fig. 4a) so that the integrated data of the two groups do not provide a good correlation. This observation indicates that the EPS properties in the treated WAS samples are different depending on the type of persulfate employed, influencing SCST differently.

On the other hand, strong positive correlations ($R_p > 0.9$) were found between the polysaccharide content in soluble EPS and CWR in Group 1 and 2, and also between the polysaccharide content in LB-EPS and CWR in Group 2. It is generally acceptable that samples with much soluble EPS (and even some of LB-EPS) exhibit high CWR values because these EPS can be readily removed by centrifugation. However, it is not clearly understood why only the polysaccharide content (not the protein content) in soluble or LB-EPS exhibits a good correlation with CWR, which warrants further study. In the plot of the polysaccharide contents in soluble EPS

versus CWR values (Fig. 4b), the data points of Group 1 and 2 are located closely, and thereby the integrated data of the two groups (Group 3) exhibit a relatively good correlation ($R_p = 0.883$, refer to the value in Table 1).

4. Conclusions

The dewaterability of WAS was greatly enhanced by oxidative treatments using thermally- and alkali-activated persulfates. The change of dewaterability was quantified by two indices representing weight reduction and SCST. The WAS dewaterability denoted by CWR increased in all the treated samples. In particular, samples treated by alkali-activated persulfates showed greater CWR values than those treated by thermally-activated persulfates. The dewaterability denoted by SCST increased only by the thermally-activated PDS system at 80 °C. The SCST values in all
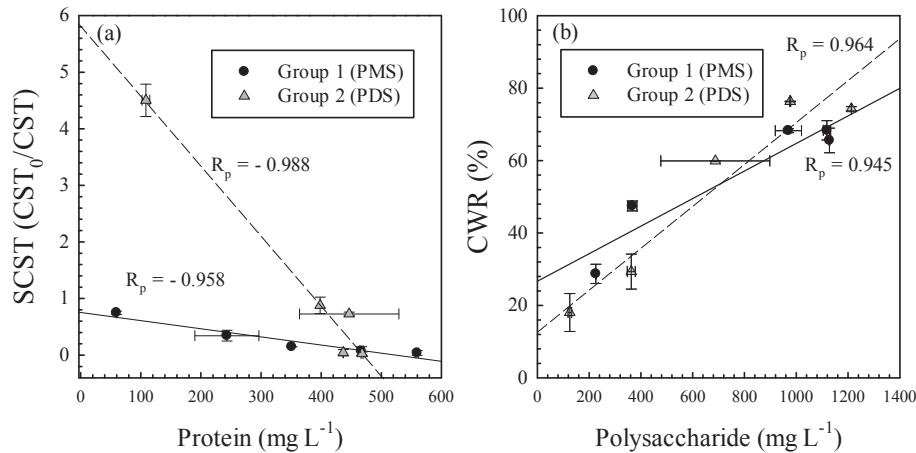
Fig. 4. Linear plots between EPS contents and dewaterability change indices with high correlation coefficients.

other treatment conditions were lower than unity. Non-selective oxidation of WAS components by reactive radical intermediates such as $SO_4^{\bullet-}$ appears to be more effective in increasing SCST compared to direct oxidation by persulfates and alkaline lysis that selectively disintegrate WAS components.

The EPSs in WAS samples were stratified as soluble, LB-, and TB-EPS fractions, and protein and polysaccharide contents in each fraction were quantified. The amount of soluble and LB-EPS increased in most of the treated samples, whereas that of TB-EPS decreased in many conditions, indicating that the fraction of EPS shifts from TB- to soluble or LB-EPS by those treatments. The Pearson's correlation analysis showed that the protein content in LB-EPS and the polysaccharide content in soluble-EPS were the dominant negative factor for SCST and the dominant positive factor for CWR, respectively.

Acknowledgement

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