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ORIGINAL PAPER

Impact of peroxydisulphate on disintegration and sedimentation properties of municipal wastewater activated sludge‡

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In the study, a thermally activated sodium peroxydisulphate (PDS; $Na_2S_2O_8$) was applied in order to disintegrate wastewater activated sludge (WAS). Chemical disintegration of WAS results in organic matter and polymer transfer from the solid phase to the liquid phase. Soluble chemical oxygen demand (SCOD) is often used to characterise the disintegration efficiency of WAS flocs and microorganisms cells. The present study was conducted in order to chemically disintegrate WAS using PDS in doses of 0.2 %, 0.4 %, 0.6 %, 0.8 % and 1.0 % activated at temperatures of 50 °C, 70 °C and 90 °C for 30 min. The temperature rise induced the PDS to form free radicals, which resulted in an increase in SCOD, i.e. for the highest dose of PDS, the SCOD value attained 2140 mg dm^{-3} (almost a 15-fold increase over the WAS value). A further positive effect from using this method was a decrease in the sludge volume index (SVI) from 89.8 cm^3 g^{-1} to 30.6 cm^3 g^{-1}. On the basis of the results obtained, it may be concluded that thermally activated PDS is suitable for disintegration and has a positive impact on WAS sedimentation properties.

Keywords: peroxydisulphate, chemical disintegration, soluble oxygen demand, sludge volume index

Introduction

The disintegration pretreatment of wastewater activated sludge (WAS) using physical (e.g. thermal), chemical (e.g. acids or alkali), mechanical (e.g. hydrodynamic or acoustic cavitation), oxidation–reduction (e.g. ozone or hydrogen peroxide) or biological (using e.g. enzymes) treatment processes has been studied as a possible pretreatment for WAS disintegration, which is characterised by biomass break-up, destruction of microbial cell walls and cytoplasm release (Burgess & Pletschke, 2008; Hiraoka et al., 1984; Kennedy et al., 2007; Grübel & Suschka, 2015; Wang et al., 2006). The substances released in the liquid and colloidal states are more available for anaerobic degradation, enhance the sludge degradation rate (biodegradability) or extent, potentially increase settling capacity and/or dewaterability after digestion and can facilitate the application of anaerobic digestion (Braguglia et al., 2011; Carrère et al., 2010; Eskicioglu et al., 2006; Grübel & Suschka, 2015). Although the methods of disintegration are different, their common aim is partial or complete bacterial cell rupture, i.e. destruction of the cell wall and the re-

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sulting release of the organic cell content to the liquid phase.

Recent studies have focused on applying thermal pretreatment for sludge dewatering, with the operating temperatures most frequent ranging from 40 ℃ to 200 ℃ (Neyens et al., 2003a; Bougrier et al., 2006). Furthermore, Bougrier et al. (2008) noted a threshold temperature of 150 ℃, for sludge dewatering. The capacity for dewaterability could diminish below this temperature, but be profoundly promoted above it. The study by Liu et al. (2012) determined that, after 60 min of thermal treatment at 175 ℃, cell flocs could be effectively destroyed, increasing the availability of soluble sugars and proteins for biological degradation, while at the same time decreasing viscosity and enhancing the sludge dewatering performance. However, thermal treatment (particularly at elevated temperatures) requires higher energy consumption and has a larger unsafe substance emission potential once the temperature reaches 180 ℃ or above (Wilson & Novak, 2009), which renders this process uneconomical and unsafe. A few studies confirmed that the combined thermo-chemical treatment could act synergistically and be advantageous to lowering the temperature, thereby affording an energy-efficient and cost-effective alternative to sludge dewatering. Guan et al. (2012) stated that $CaCl_2$ solution in a mild temperature range from 50 ℃ to 90 ℃ could strengthen the bridging between calcium ions and the flocs, thus promoting the sludge dewaterability. Abelleira et al. (2012) treated secondary sewage sludge with H_2O_2 under mild operating conditions, resulting in an enormous reduction in the time-to-filter (TTF) and in dewaterability improvement. Simultaneous suphuric acid and thermal treatment was applied in order to remove water from waste sludge (Neyens et al., 2003a); as a result, the dry sludge content of the dewatered sludge increased from 22.5 % to approximately 70 %. The dewaterability of sludge could also be distinctly improved by $Ca(OH)_2$ at a temperature of 100 ℃ for 60 min (Neyens et al., 2003b).

One of the chemical reagents which has a high oxidation potential and could be used for WAS disintegration is sodium peroxydisulphate ($Na_2S_2O_8$, PDS). PDS dissociates in the liquid phase to a peroxydisulphate anion – a strong and stable oxidant with a very high redox potential of 2.01 V (Eq. 1) (Latimer, 1952). Furthermore, PDS can be catalysed to form $SO_4^{-\bullet}$ and $OH^{\bullet}$, thereby affording free radical reaction mechanisms similar to the hydroxyl radical pathways generated by Fenton's chemistry (Eq. 2) (Block et al., 2004).

$$Na_2S_2O_8 \rightarrow 2Na^+ + S_2O_8^{2-} \quad (1)$$

$$S_2O_8^{2-} + \text{catalyst} \rightarrow SO_4^{\bullet -} + (SO_4^{\bullet -} \text{ or } SO_4^{2-}) \quad (2)$$

The sulphate radicals and hydroxyl radicals are among the strongest aqueous oxidising species with a redox potential estimated to be 2.6 V and 2.7 V, respectively (Block et al., 2004).

Hydroxyl radicals can be produced from sulphate radicals according to Yuan et al. (2014) (Eq. 3).

$$H_2O + SO_4^{-\bullet} \rightarrow OH^{\bullet} + SO_4^{2-} + H^+ \quad (3)$$

These radicals are usually generated under heat-, photo-, metal-, chelated metal-, quinone-, phenols-, hydrogen peroxide-, alkaline-catalysed conditions (Huang et al., 2002; Block et al., 2004; Fang et al., 2013; Siegrist et al., 2011; Ahmad et al., 2013).

The major by-products of persulphate oxidation with a pK_a value higher than 1.92 are sulphate ions (SO_4^{2-}) (Liang & Guo, 2012). Base activation of PDS involves increasing the pH value (> 11) by the addition of a concentrated base, often sodium hydroxide or potassium hydroxide (Siegrist et al., 2011). Other reagents such as CaO, which can react with water to produce ($Ca(OH)_2$) and create an alkaline pH, can also be used (Liang & Guo, 2012).

It was proposed that alkaline conditions could induce PDS decomposition in accordance with the base-catalysed hydrolysis of PDS to hydroperoxide anions (HO_2^-). The reduction of PDS by hydroperoxide anions can further generate sulphate and superoxide radicals (Zhou et al., 2013). PDS can be used for the oxidation of both organic (Huang et al., 2002; Liang et al., 2004; Fang et al., 2013; Liang & Guo, 2012) and inorganic (Romero et al., 2010; Zhou et al., 2013) contaminants. A few attempts have been made to use PDS to increase the dewaterablity of sludge (Zhen et al., 2013; Oncu & Balcioglu, 2013; Zhen et al., 2012a, 2012b) and enhance the digestion stage (Sun et al., 2012).

The new concept described in this paper is based on the process of WAS chemical disintegration by a thermally activated sodium peroxydisulphate. This study investigated the chemical disintegration of WAS by PDS in doses of 0.2 %, 0.4 %, 0.6 %, 0.8 % and 1.0 % (per gram of total solids; 169.5 mg, 339.0 mg, 508.5 mg, 678.0 mg, 847.5 mg) activated at temperatures of 50 ℃, 70 ℃ and 90 ℃ for 30 min. The efficiency of this method was evaluated in terms of organic matter release to the supernatant – expressed as soluble chemical oxygen demand – and improvement to the settling properties of WAS – expressed as changes in the sludge volume index.

Experimental

PDS (99.0 %), was purchased from Lach-Ner (Czech Republic). COD cuvette tests were purchased from Hach (USA).

The municipal wastewater activated sludge (WAS samples, concentration of total solids (TS) on average 9.4 g dm^{-3}) was taken from the Liberec (Czech Re-

Table 1. Characteristics of WAS sample (mean values ± standard deviations of concentration/value in supernatant were calculated from 5 repeat measurements)

Indicator	Unit	Mean value ± standard deviation
pH	–	6.9 ± 0.3
temperature	℃	15.6 ± 2.0
oxidation/reduction potential	mV	72.4 ± 6.3
conductivity	mS cm^{-1}	1.1 ± 0.2
total solids	g dm^{-3}	9.4 ± 0.3
volatile solids	g dm^{-3}	6.3 ± 0.4
NH_4^+	mg dm^{-3}	15.8 ± 3.2
PO_4^{3-}	mg dm^{-3}	34.4 ± 2.8
soluble chemical oxygen demand	mg dm^{-3}	145 ± 12
SVI	cm^3 g^{-1}	89.80 ± 2.87

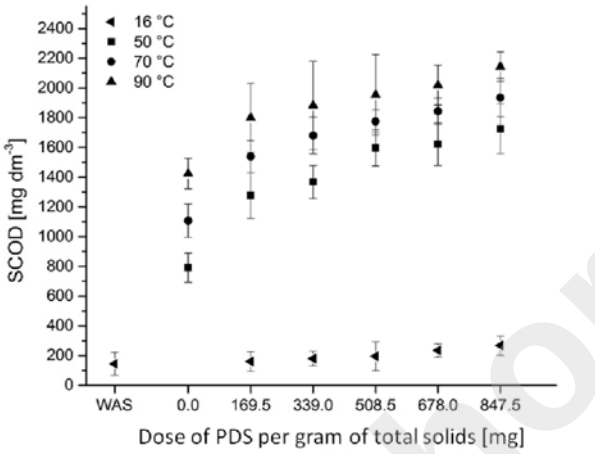

Fig. 1. Impact of PDS dose and thermally activated PDS on SCOD release (mean values ± standard deviation).

public) Waste Water Treatment Plant (WWTP), the largest WWTP in both the Liberec and the Ústí regions. The WWTP is owned by Severočeská vodárenská společnost (Teplice, Czech Republic) and operated by Severočeské vodovody a kanalizace (Teplice, Czech Republic). The treatment plants were arranged for nutrients liquidation. With the intention of the removal of phosphorous and nitrogen substances, anaerobic, anoxic and aerobic regions were specified. The plant was designed for a flow of 103882 m^3 d^{-1}. At present, the amount of treated wastewater is approximately 54806 m^3 d^{-1}. The characteristics of the WAS sample are presented in Table 1.

For chemical WAS disintegration, PDS was used. PDS was added to samples of WAS in amounts/doses of 0.2 %, 0.4 %, 0.6 %, 0.8 % and 1.0 % of volume (per gram of total solids; 169.5 mg, 339.0 mg, 508.5 mg, 678.0 mg, 847.5 mg). In addition, PDS was activated at temperatures of 50 ℃, 70 ℃ and 90 ℃ for 30 min in order to disintegrate the sludge.

All the chemical analyses were performed on samples prior to and after each phase of disintegration. Soluble chemical oxygen demand (SCOD) and sludge volume index (SVI) were determined following the standard methods for the examination of water and wastewater procedures 5220D and 2710D, respectively (Rice et al., 2012). The content of total solids (TS) was also determined. For colorimetric determinations, a spectrophotometer HACH DR 6000 UV/VIS (Hach Lange) was used.

The investigations presented here were repeated 5 times and (mean values, $n = 5$) the arithmetical average and standard deviation were calculated. The standard deviation was determined according to the estimator of the highest credibility in STATISTICA 6.0 (StatSoft Polska, Poland).

A scanning electron microscope (SEM) Vega XMU (Tescan, Czech Republic) was used to acquire confirmation of destruction of WAS particle size during chemical disintegration.

Results and discussion

The presence of exopolymers (EPS; sugar composites, amino acids and uronic acids) contributes to the agglomeration of bacteria in WAS flocs. The adsorptive properties of EPS have been well documented, especially in respect of the biosorption of hazardous organic pollutants and COD onto aerobic and anaerobic biomass in biological wastewater treatments (Esparza-Soto & Westerhoff, 2003; Cloete & Oosthuizen, 2001; Guellil et al., 2001). Disintegration of the WAS flocs caused the destruction of EPS as well as bacteria-scattering and partial or complete bacterial cell destruction/lysis. This led to the release of intracellular organic matter and the enzymes present in the cells cytosol as well as the destruction of EPS and increased dissolved organic matter concentrations in the supernatant. In addition, the effectiveness of these changes (release of intracellular and exocellular organic matter) can be measured as an increase in SCOD.

Thirty minutes of chemical WAS flocs disintegration resulted in a SCOD increase in the filtrate. This SCOD was due to VS by the sludge pretreatment; the marked increase in SCOD concentration indicates a significant increase in the amount of soluble organic matter in WAS. In addition, under thermally acti-

Table 2. Impact of thermally activated PDS on SVI

Dose of PDS per gram of total solids [mg]	Changes in SVI in sedimentation time [$cm^3\ g^{-1}$]								
	2 min	4 min	6 min	8 min	10 min	15 min	20 min	25 min	30 min
Raw wastewater activated sludge									
0.0	98.31 ± 3.21	98.05 ± 3.01	97.95 ± 2.91	97.80 ± 2.95	95.70 ± 3.13	93.70 ± 2.82	91.68 ± 3.11	90.74 ± 2.81	89.80 ± 2.87
Activation temperature 50 °C									
0.0	97.80 ± 3.01	95.70 ± 2.13	93.70 ± 2.42	91.68 ± 2.21	83.12 ± 3.53	78.17 ± 3.19	74.13 ± 2.94	71.05 ± 4.46	69.52 ± 3.87
169.5	91.20 ± 2.11	90.32 ± 2.44	88.62 ± 1.63	86.54 ± 1.92	74.31 ± 3.23	69.25 ± 3.85	66.51 ± 3.16	63.41 ± 4.12	60.07 ± 4.29
339.0	85.71 ± 1.90	82.90 ± 1.24	81.06 ± 2.30	77.83 ± 2.04	64.82 ± 2.50	59.24 ± 3.24	56.44 ± 2.47	52.32 ± 4.02	49.57 ± 3.80
508.5	76.32 ± 1.48	72.08 ± 1.58	68.64 ± 5.15	66.11 ± 0.83	57.08 ± 2.86	51.82 ± 1.82	48.03 ± 1.14	46.71 ± 2.61	45.91 ± 2.35
678.0	71.12 ± 2.32	68.16 ± 2.06	66.52 ± 0.84	64.73 ± 0.94	53.74 ± 2.17	49.06 ± 1.34	46.07 ± 2.98	44.24 ± 1.93	43.40 ± 1.69
847.5	69.34 ± 2.19	62.14 ± 1.55	60.97 ± 0.72	59.91 ± 1.19	48.25 ± 1.62	44.93 ± 0.90	42.96 ± 0.86	40.93 ± 1.59	44.21 ± 1.12
Activation temperature 70 °C									
0.0	92.21 ± 4.13	88.83 ± 2.28	85.81 ± 3.43	83.71 ± 3.42	78.31 ± 2.27	74.15 ± 2.10	71.11 ± 2.85	68.52 ± 3.96	66.92 ± 4.19
169.5	85.81 ± 4.11	82.31 ± 1.20	79.70 ± 3.61	77.12 ± 3.01	69.70 ± 2.46	66.52 ± 3.05	61.92 ± 2.83	59.06 ± 3.63	55.84 ± 3.45
339.0	84.23 ± 4.01	78.21 ± 3.88	73.53 ± 3.59	69.62 ± 2.01	61.44 ± 1.77	56.41 ± 2.24	52.20 ± 2.11	48.81 ± 3.41	46.51 ± 3.12
508.5	69.81 ± 5.11	65.72 ± 2.31	62.05 ± 2.15	58.12 ± 1.81	51.61 ± 2.13	46.82 ± 1.65	41.45 ± 1.40	41.83 ± 3.19	41.12 ± 3.13
678.0	63.12 ± 1.98	60.23 ± 4.28	58.36 ± 1.99	57.22 ± 2.23	51.51 ± 1.47	47.45 ± 1.13	42.82 ± 1.28	39.32 ± 2.48	37.51 ± 3.03
847.5	61.92 ± 1.57	58.04 ± 1.17	55.52 ± 1.03	54.815 ± 2.11	48.82 ± 1.45	45.16 ± 0.90	41.39 ± 0.65	38.36 ± 3.47	38.44 ± 3.14
Activation temperature 90 °C									
0.0	86.71 ± 4.43	81.81 ± 3.83	77.91 ± 3.11	75.71 ± 3.19	73.51 ± 2.17	70.81 ± 3.17	68.22 ± 2.85	66.03 ± 3.76	64.23 ± 4.42
169.5	80.56 ± 4.14	74.44 ± 4.20	70.82 ± 6.25	67.81 ± 3.20	65.14 ± 3.49	61.62 ± 5.21	57.24 ± 3.13	54.63 ± 3.65	51.54 ± 3.91
339.0	82.74 ± 1.90	73.52 ± 1.23	66.04 ± 2.30	62.05 ± 2.08	58.12 ± 2.51	52.84 ± 3.34	44.00 ± 2.47	41.69 ± 4.05	39.70 ± 3.89
508.5	63.42 ± 1.48	59.42 ± 1.53	55.03 ± 1.15	50.21 ± 0.83	46.23 ± 2.86	41.83 ± 1.85	34.81 ± 1.74	33.22 ± 4.26	32.29 ± 2.45
678.0	55.05 ± 2.66	52.47 ± 3.48	50.21 ± 2.12	49.71 ± 1.82	45.31 ± 2.18	40.21 ± 1.68	34.12 ± 1.41	32.56 ± 3.22	31.71 ± 3.18
847.5	54.63 ± 1.92	53.25 ± 1.60	50.17 ± 1.34	49.65 ± 2.23	44.27 ± 1.41	39.17 ± 1.16	33.05 ± 1.08	32.06 ± 2.56	30.62 ± 3.03

vated conditions (i.e. temperatures of 50 ℃, 70 ℃ and 90 ℃), there is considerable evidence that the PDS can be converted to powerful oxidants denoted as sulphate and hydroxyl free radicals which can penetrate the cell more readily. As a result, intracellular organic material is released to the liquid phase and increases the SCOD value. The thermally activated PDS at temperatures of 50 ℃, 70 and 90 ℃ caused a higher SCOD increase in the supernatant. In addition, an increased PDS dose at the same time enhanced the SCOD value.

During the research, the impact of PDS on the sedimentation properties of WAS was also determined. Table 2 presents changes in the SVI value with increased doses of PDS added to samples and after thermal activation of PDS.

Table 1 shows that the great decrease in the SVI value was achieved for the highest dose of PDS and the highest activation temperature. The SVI value decreased from 89.80 $cm^3\ g^{-1}$ to 30.62 $cm^3\ g^{-1}$. Changes in the SVI value caused a definite improvement in the sludge sedimentation properties. Low values of the sludge volume index, approximately 100 $cm^3\ g^{-1}$, indicate the very good sedimentation properties of sludge. SVIs reported in the literature ranged from 4 $cm^3\ g^{-1}$ (Kjellerup et al., 2001) to 700 $cm^3\ g^{-1}$ (Sezgin, 1982; Jenkins et al., 1993). Sludges are typically classified into three groups in terms of their settleability, based on the SVI (Jenkins et al., 1993; Gray, 2004): SVI < 50–70 $cm^3\ g^{-1}$: very good settleability; 50–70 $cm^3\ g^{-1}$ < SVI < 120–150 $cm^3\ g^{-1}$: good settleability; 120–150 $cm^3\ g^{-1}$ < SVI: poor settleability.

It was observed that increased temperatures (without oxidant addition) to 50 ℃, 70 ℃ and 90 ℃ caused decreases in the SVI values to 69.52 $cm^3\ g^{-1}$, 66.92 $cm^3\ g^{-1}$ and 64.23 $cm^3\ g^{-1}$, respectively. However, the addition of PDS and its activation at temperatures of 50 ℃, 70 ℃ and 90 ℃ led to higher sedimentation/compaction efficiency. In comparison with the thermal disintegration used separately, it was observed that SVI decreased by a further 36.5 %, 42.5 % and 52.3 % respectively. Moreover, Peeters et al. (2009) showed that sludge with a low SVI dewatered better than sludge with a high SVI. In addition, according to Sanin et al. (2011), one way to assess the potential for dewatering of biological sludge by centrifugation is to run the SVI test: a sludge that does not settle well will also have poor centrifugation properties. Kjellerup et al. (2001) demonstrated this dependence of the sludge dewaterability on the SVI by measuring the solids dryness of the filter cake at a large industrial WWTP: the dryness dropped markedly from approximately 60 % to 30 % of dry solids when the SVI increased from 5 $cm^3\ g^{-1}$ to 30 $cm^3\ g^{-1}$.

It should be noted that heating of WAS to obtain the required temperatures (in order to activate PDS) is possibly the biggest issue in respect of introduction of the method tested here to a WWTP. From the results presented here, it may be concluded that an increase in temperature to 50 ℃ can already act as a good sulphate radical initiator, hence a combination of PDS and 50 ℃ can contribute significantly to WAS disintegration and settleability improvement. Attaining this temperature threshold can be achieved by using recovered heat from the combustion of biogas generated during the digestion of WAS. Steam, hot air injection or electrical heating techniques can also be applicable to WAS heating. WAS disintegration with elevated temperatures has been studied by many authors (Stasta et al., 2006; Houdková et al., 2008; Wett et al., 2010). Additional tests are needed to determine whether thermal activation of PDS is a cost-effective technique for wastewater treatment plants.

In addition, sulphate contamination (in the sludge directed to the anaerobic digestion process) can be another problem in the PDS use for chemical disintegration of WAS. Many studies have found that small concentrations of sulphate anions do not inhibit methanogenic fermentation (Cabirol et al., 2003; Yin et al., 2015; Sun et al., 2012; Wei et al., 2007). Moreover, Sun et al. (2012) found that PDS disintegration had a positive influence on biogas yield. Our recent study showed the biogas yield from the fermentation of sludge treated by the heat-activated PDS to be higher than in a sample of raw WAS.

The experiments performed on WAS confirmed the good influence of PDS on the sedimentation properties of sludge. Future studies will investigate the impact of PDS on fermented sludge, which has a higher concentration of total solids. Moreover, the enhanced sedimentation properties of such sludges would reduce the amount of sludge requiring further management.

The higher sedimentation and compaction efficiency for various PDS doses at temperature of 70 ℃ is shown on Fig. 2.

To gain an insight into the destructive mechanisms of thermally activated PDS, SEM analysis of the sludge treated under different conditions was performed.The SEM images revealed cleavage of the sludge particles on the colloidal scale, which induced the release of EPS-bound water, thereby enhancing sedimentation properties (Peeters et al., 2009). Large differences can be observed in the sludge appearance prior to and after treatment. The particle size became much smaller after disintegration by thermally activated PDS at 50 ℃, 70 ℃ and 90 ℃. In addition, the WAS flocs appeared to be much less regular after treatment, with many pores. Fig. 3 presents an example of the changes in particle size of WAS after PDS addition and thermal activation at 70 ℃.

Similar results were obtained by Zhen et al. (2012b), who found the combined thermal (under mild temperature 25–80 ℃) oxidation process to be efficient in enhancing the dewaterability of sludge. They concluded that, when the temperature was increased to 25 and 80 ℃, the flocs of WAS were drastically changed and that this pretreatment resulted in the disruption

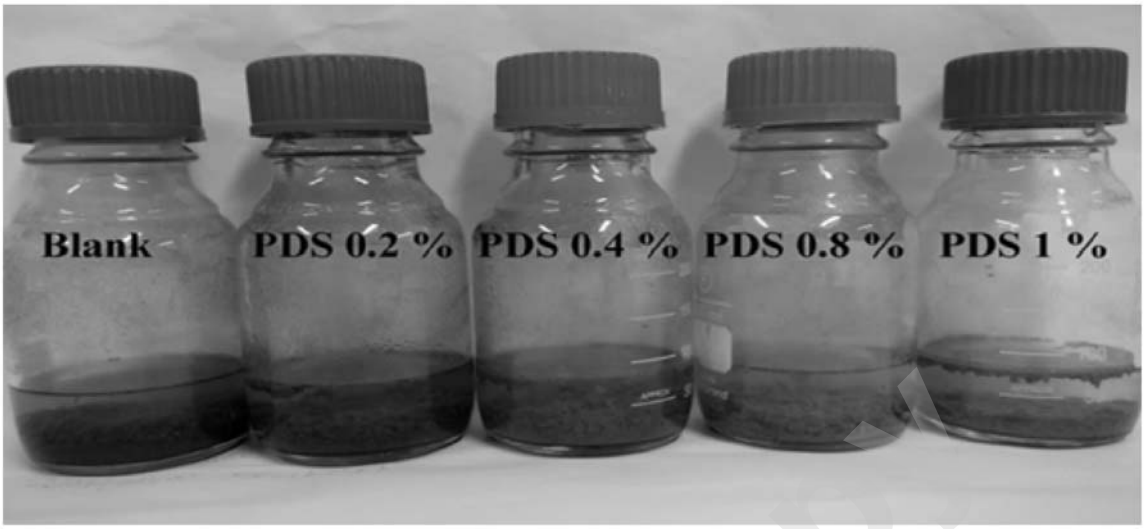

Fig. 2. Image of reactors with various dosages of PDS after thermal activation at 70 °C.

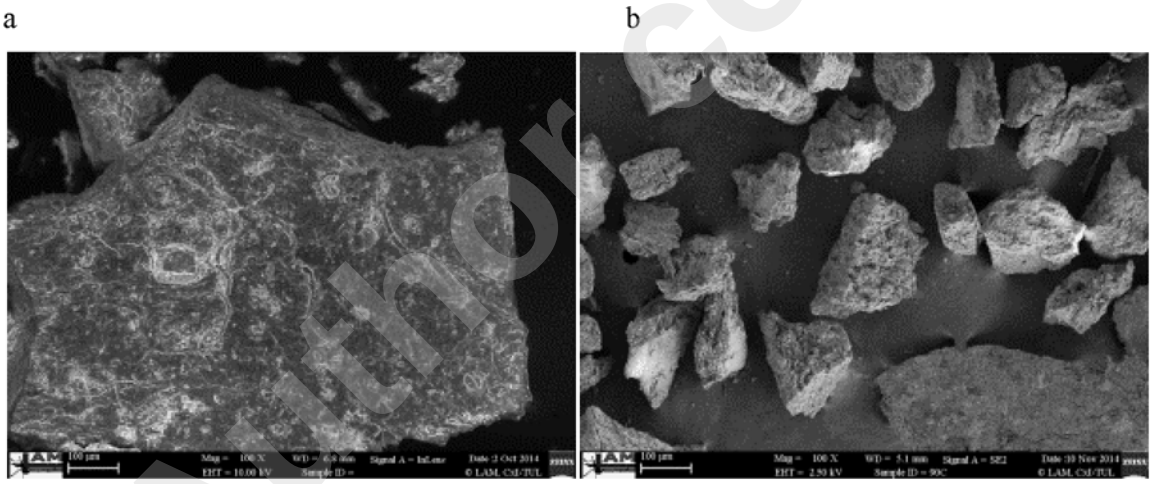

Fig. 3. Scanning electron microscope observations of particle of WAS; raw WAS (a) and WAS + PDS + 70 °C (b).

of sludge flocs by degrading EPS, hence enhancing the sedimentation properties of WAS.

Conclusions

The experiments clearly demonstrated chemical disintegration to be a suitable method for destroying flocs and microorganisms in WAS. This study examined the addition of PDS to WAS in order to improve the disintegration and sedimentation properties. The most important conclusions are: (i) the chemical pretreatment of WAS by PDS destroys the structure of the sludge flocs and damages the cells of the microorganisms. As an outcome of WAS disintegration, organic substances were transferred from the solid particles into the liquid phase (expressed as SCOD). SCOD increased from 145 mg dm^{-3} to 268 mg dm^{-3} in direct proportion to the PDS dose; (ii) thermally activated PDS caused the organic matter release to increase several-fold. SCOD values increased to 1725 mg dm^{-3}, 1935 mg dm^{-3} and 2143 mg dm^{-3} for the highest dose of PDS after thermal activation at 50 °C, 70 °C and 90 °C, respectively; (iii) it was observed that raising the temperatures (without oxidant addition) to 50 °C, 70 °C and 90 °C caused decreases in SVI values to 69.52 cm^3 g^{-1}, 66.2 cm^3 g^{-1} and 64.23 cm^3 g^{-1}, respectively. However, the addition of PDS activated at temperatures of 50 °C, 70 °C and 90 °C improved the efficiency of the sedimentation properties. In comparison with thermal disintegration used separately, it was observed that SVI decreased by a further 36.5 %, 42.5 % and 52.3 %, respectively. Disintegration of WAS by thermally activated PDS caused a decrease in WAS particle size, which was confirmed by SEM observations.

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