



Characteristics of organic, nitrogen and phosphorus species released from ultrasonic treatment of waste activated sludge

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ABSTRACT

Batch ultrasonic treatments (sonication) were performed on two waste activated sludge (WAS) samples, BNR-WAS from the biological nitrogen removal unit and BNPR-WAS from the biological nitrogen and phosphorus removal unit of two Shanghai municipal WWTPs, to determine the effects of sonication time and intensity on the amount and distribution of the organic, N and P species released from the samples. The concentration profiles of COD, TOC fractions in different molecular weight (MW) ranges (<2 kDa, 2–100 kDa, and >100 kDa), TN, organic-N, NH₃-N, TP and PO₄-P were monitored during the treatment at three sonication intensity levels (0.167, 0.330 and 0.500 W/mL). Species releases increased with sonication time and/or intensity; the release rates were accelerated when the sonication intensity was above a critical level between 0.330 and 0.500 W/mL. After 1 h of treatment, 37.9%, 37.5% and 50.8% of the organic content (measured as COD) of BNR-WAS were released, while the same for BNPR-WAS were 40.9%, 55.3% and 56.9%. It also resulted in the release of 40.9%, 38.7%, and 52.1% of total nitrogen from BNR-WAS, relative to 46.2%, 61.6%, and 70.4% of the same from BNPR-WAS; most released nitrogen were organic-N (65.0% and 84.9%), followed by NH₃-N (34.7% and 14.9%) and trace amounts of nitrate and nitrite. More total phosphorus of a higher orthophosphate content was released from BNPR-WAS (>60% release after 1 h of sonication, 80% was PO₄-P) than from BNR-WAS (<50% release, 40% was PO₄-P). The differences in the releases as well as the molecular weight distribution pattern of the soluble TOC species were due to the different structure and composition of the sludge samples. Sonication is a viable sludge treatment process when it is combined with a phosphorus recovery process to remove most of the released PO₄-P so that the supernatant may be returned for further biological treatment.

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1. Introduction

Various forms of the aerobic activated sludge process are employed in more than 700 municipal wastewater treatment plants (WWTPs) in China with a combined treatment capacity of about $2534 \times 10^4 \text{ m}^3/\text{day}$ [1]. The large amounts of activated sludge generated from those WWTPs pose a significant threat to ecological environment. Proper treatment and disposal of waste activated sludge (WAS) are very costly; they would present an unaffordable burden to most WWTP owners. To minimize the production of WAS, many sludge treatment processes have been proposed, including mechanical treatment [2], ultrasonic treatment (sonication) [3–5], ozonation [6,7] and chemical treatment [8,9]; among them, sonication and ozonation are attractive because they are environmentally benign and controllable.

Ultrasound is a pressure wave that propagates through a medium with a vast amount of energy dissipation generating numerous gas and vapor bubbles which may grow, and then collapse violently at high speeds to cause acoustic cavitation [10]. Cavitation occurs more readily at a frequency of 20–40 kHz [11]. High temperature and pressure developed inside the collapsing bubbles may produce many physico-chemical effects [12]. Under sonication, large and stable activated sludge flocs are disintegrated [13] and that a portion of the insoluble organic matter may be dissolved [14]. Sonication of WAS is capable of destroying the flocs holding extracellular polymeric substances (EPS) to result in much smaller flocs due to the hydro-mechanical shear force created in the reactor [15] and also producing highly oxidative •OH in the amount that increases with treatment time and intensity [16]. Along with floc disintegration and cell lysis, many organic, nitrogen and phosphorus constituents of WAS are dissolved, hydrolyzed and released, especially when it came from a biological nutrient removal process. Recycling of the ultrasonic treated sludge to the existing biological treatment unit will therefore expect to significantly increase its organic, nitrogen and phosphorus loadings. Although many literature reports have documented releases of the

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organic constituents of WAS under sonication, few have focused on the investigation of concurrent releases of nitrogen and phosphorus species. Sonication causes different degrees of cell lysis and releases of organic and nutrient species when it is performed on WAS of different forms of activated sludge process [17,18]. Given the more stringent requirements for nutrient removal, a comprehensive understanding of nitrogen and phosphorus releases under sonication is essential to design a cost effective ultrasonic treatment system for the WWTP WAS reduction program. The objectives of this study were to (1) determine the effects of sonication time and intensity on amount and distribution of organic, nitrogen and phosphorus species released, (2) the role of WAS origin on the releases, (3) assess the potential impacts of extra organic and nutrient loadings of the sonicated WAS, and (4) propose a cost effective ultrasonic treatment system for the WWTP WAS reduction program.

2. Materials and methods

2.1. WAS samples

Two different types of WAS samples were collected from the biological treatment units of two municipal WWTPs in Shanghai, China. BNR-WAS came from a WWTP with a primary treatment followed by a biological nitrogen removal unit; BNPR-WAS came from another WWTP employing a biological nitrogen and phosphorus removal unit. The samples are representative of WAS from the two most common aerobic biological treatment processes in China for removing most organic and nutrient constituents of the wastewater to prevent eutrophication of the receiving water body. Characteristics of the WAS samples are listed in Table 1. Total COD, total nitrogen and total phosphorus of the WAS sample were measured after it was completely mixed using a magnetic stirrer; liquid phase COD, total nitrogen (TN), organic-N, $\text{NH}_3\text{-N}$, total phosphorus (TP), and ortho-P were measured for the supernatant (liquid phase) after the sample centrifugation at 4000 rpm for 20 min. All measurements were conducted within 2 h after sampling to be representative of a fresh WAS sample. Reported values are averages of the duplicates; the experimental data were within $\pm 8\%$ of the average value. Table 1 data show that the two WAS samples were indeed quite different.

2.2. Experimental methods

The key unit of the ultrasonic treatment system (Fig. 1) was a homogeneous sonicator (JYD-650L, Shanghai Zhixin Inc., China), consisted of the ultrasound generator and a probe (10 mm diameter), with an operating frequency of 20–25 kHz and a maximum

Table 1
Characteristics of the two WAS samples (unit: mg/L, except pH and VS/TS).

Parameter	BNR-WAS	BNPR-WAS
Liquid phase (supernatant)		
pH	6.82 \pm 0.06	7.50 \pm 0.15
COD	40.7 \pm 10.9	32.7 \pm 5.5
Total Nitrogen	31.08 \pm 2.07	13.70 \pm 1.67
Organic-N	1.91 \pm 0.32	6.25 \pm 1.34
$\text{NH}_3\text{-N}$	29.09 \pm 1.82	3.94 \pm 0.45
Total phosphorus	2.78 \pm 0.98	1.11 \pm 0.24
Ortho-P	2.13 \pm 0.58	0.25 \pm 0.06
Whole sample (overall)		
TS	6780 \pm 360	6730 \pm 390
VS/TS	0.61	0.69
COD	7310 \pm 550	7540 \pm 450
Total nitrogen	523 \pm 48	448 \pm 67
Total phosphorus	250 \pm 30	552 \pm 107

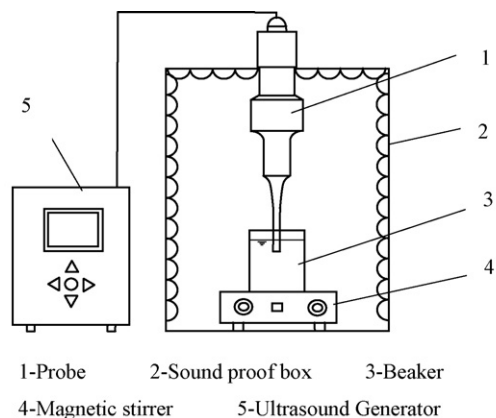


Fig. 1. Schematic diagram of the ultrasonic treatment system.

power input of 650 W. Batch treatment runs were carried out in 400 mL beakers containing 300 mL the WAS sample in each under mixing of a magnetic stirrer. The tip of the probe was 15 mm below the sample surface. During the treatment, the sonication intensity was controlled at one of the three power input levels of 0.167, 0.330 and 0.500 W/mL. The sonicated WAS sample was centrifuged at a speed of 4000 rpm for 20 min, and the concentrations of organic, nitrogen and phosphorus species of the liquid phase were measured.

2.3. Analyses

Analyses of COD, BOD_5 , TN, $\text{NH}_3\text{-N}$, $\text{NO}_2^- \text{-N}$, $\text{NO}_3^- \text{-N}$, TP, ortho-P, total solids (TS) and volatile solids (VS) were performed according to the Standard Methods [19]. Total organic carbon (TOC) was measured by a TOC analyzer (LiquiTOC, Elementar Analysensysteme Co., Germany) and pH by pH analyzer (Delta 320, Mettler Toledo Co.).

Molecular weight (MW) distribution of the TOC constituents of the liquid phase before and after sonication was determined by UF membranes (SCM model) with MW cut-offs of 2 kDa and 100 kDa. Membranes were first rinsed several times with ultra-pure water in a beaker 12 h before use. Nitrogen gas (99.999%) pressure was controlled at 0.15–0.25 MPa to promote filtration. After rinsing, filtrate of the primary filter (0.45 μm) before and after sonication was pressurized; the permeate was collected from each MW cut-off for TOC measurement. The results were expressed as percentage of the total TOC for each of its three fractions (MW <2 kDa, 2–100 kDa, and >100 kDa).

The percentage of COD released was calculated by: $(\text{COD}_i - \text{COD}_0) \times 100 / \text{COD}_s$, where COD_i and COD_0 are COD of the liquid phase after and before sonication and that COD_s is COD of the WAS sample. The release percentages of TN and TP were calculated similarly.

3. Results and discussion

3.1. Organic matter release

It was well known that sonication can disintegrate the structure of sludge flocs and solubilize some of the cellular organic substances. The changes in COD concentration of the liquid phase during sonication (COD release profiles) for the two samples at three power input levels are shown in Fig. 2a (cumulative COD release) and Fig. 2b (cumulative % COD released). As the intensity increased three folds from 0.167 to 0.500 W/mL, the 1 h cumulative % COD release increased only 34.0% for BNR-WAS and 39.1% for BNPR-WAS. The much smaller differences in COD releases between the two lower intensity levels relative to the two higher levels sug-

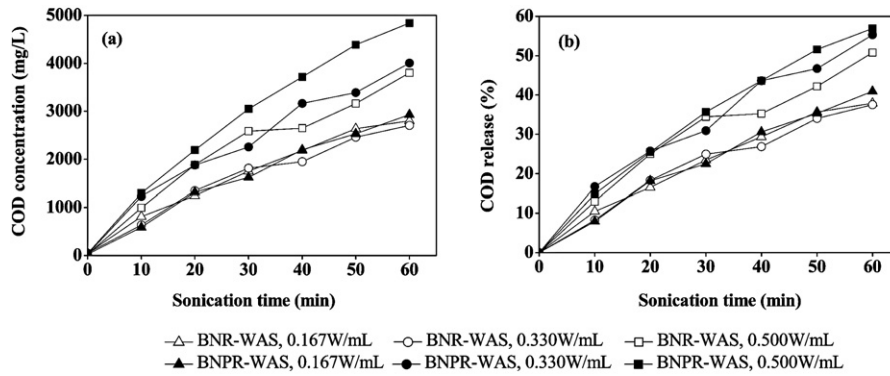


Fig. 2. COD release profiles of the liquid phase: (a) cumulative release of COD, and (b) cumulative percent COD released.

gests that the release rates were accelerated when the sonication intensity was above a critical level between 0.330 and 0.500 W/mL.

2820, 2720 and 3830 mg/L of COD (37.9%, 37.5% and 50.8% of the total WAS COD) was released from BNR-WAS after 1 h treatment at 0.167, 0.330 and 0.500 W/mL intensities, respectively. For BNPR-WAS, the comparable releases were slightly higher at 2930, 3850 and 4840 mg/L (40.9%, 55.3% and 56.9% of the total). Such results were much higher than those reported by Chu et al. [3] showing that 20% of the WAS COD was released after 2 h of sonication (20 kHz, 0.44 W/mL).

3.2. Molecular weight distribution of solubilized organic matter

To ensure the sonicated WAS can be beneficially recycled to the WWTP, the BOD₅/COD ratios of the liquid phase before and after the treatment were assessed. The higher ratios of the sonicated samples (0.33–0.37 vs. 0.13–0.14 for BNR-WAS and 0.33–0.68 vs. 0.01–0.07 for BNPR-WAS) suggest that most of the organic species released will be biodegraded in the existing biological treatment unit of the WWTP.

To further characterize the released organic species, series of filtration runs were conducted to obtain distributions of organic fractions (MW <2 kDa, 2–100 kDa, and >100 kDa) released by sonication. Table 2 shows specific amount of TOC released (mg TOC/g TS) from the two WAS samples during sonication at the three intensities, and Fig. 3 exhibits the distribution profiles of the three TOC fractions for the two sonicated WAS samples.

TOC concentrations of the liquid phase before sample sonication were 14.6 and 8.8 mg/L, respectively, for BNR-WAS and BNPR-WAS. Table 2 shows specific amount of TOC release increased with sonication time and ultrasonic intensity and that organic constituents of BNPR-WAS were more readily dissolved; 18.5, 21.3, and 36.4 mg TOC/g TS were released from BNR-WAS after 1 h of sonication at 0.167, 0.330, and 0.500 W/mL, respectively, relative to the much higher releases of 54.7, 65.0, and 114.9 mg TOC/g TS

Table 2
Specific amount of TOC released (unit: mg TOC/g TS).

Ultrasonic intensity level (W/mL)	Sonication time (min)					
	10	20	30	40	50	60
BNR-WAS						
0.167	9.2	9.5	10.9	13.9	14.2	18.5
0.330	10.9	12.0	14.0	16.0	16.8	21.3
0.500	11.1	13.1	22.4	23.7	27.7	36.4
BNPR-WAS						
0.167	10.3	24.2	23.1	35.2	38.2	54.7
0.330	13.1	22.7	29.2	50.6	56.2	65.0
0.500	22.9	38.9	60.2	85.3	100.3	114.9

from BNPR-WAS. The difference was primarily due to the fact that large organic polymers and colloidal organic matter released were excluded by the primary filter (0.45 μm) employed to prepare the liquid phase samples for TOC measurements and that such exclusion was likely to be much more for BNR-WAS than BNPR-WAS since organic constituents of the latter were more soluble after the treatment (Fig. 2). Another minor reason was the higher organic content of the latter as evidenced by its higher VS/TS (Table 1). The presumed presence of a high density of phosphorus accumulating organisms (PAOs) in BNPR-WAS might have also been the reason for its much higher total phosphorus content (552 mg/L vs. 250 mg/L).

Fractionating the TOC constituents of the liquid phase to different MW size fractions provides detailed information about their characteristics helpful for selecting effective treatment technologies [20,21]. Fig. 3 shows that organic species of MW <2 kDa were most abundant in all samples and that the distribution profile was dependent on the sample type and sonication time and intensity. For BNR-WAS (Fig. 3a–c), the <2 kDa fraction declined while the 2–100 kDa fraction increased with sonication time at the two lower intensity levels of 0.167 and 0.330 W/mL and that this trend was more notable at the higher intensity. Some of the small organics of MW <2 kDa were oxidized by the highly oxidative •OH radicals produced during sonication, while breaking down of larger organic constituents increased percentage TOC of the smallest fraction. At 0.500 W/mL level, the dominant fraction of MW <2 kDa increased further (82.3–91.8%) with sonication time.

The organic release patterns presented in Table 2 show that the net cumulative organic release was slow at both 0.167 and 0.330 W/mL because the ultrasonic energy was insufficient to cause a significant degree of cell lysis. At 0.500 W/mL, the change in the MW distribution was quite different; the fraction of organic species of 2 kDa < MW < 100 kDa increased initially and then declined, indicating that those organics were broken into smaller organics as the treatment continued at the high intensity level; such hypothesis was consistent with the profile of the smallest fraction (MW <2 kDa). The above observations coupled with the lower VS/TS of BNR-WAS suggested that most of the released organics came from the EPS content of the sample.

The distribution profiles of the three TOC fractions for BNPR-WAS were different, as illustrated in Fig. 3d–f. At 0.167 and 0.330 W/mL intensity levels, the distribution profiles of MW <2 kDa was not much changed during the treatment. At these intensity levels, the floc holding EPS were solubilized releasing organic constituents of 2–100 kDa MW which were then broken down to keep the smallest fraction (MW <2 kDa) at nearly the same level. At 0.500 W/mL intensity level, the smallest fraction declined from 94.6% to 53.2% while the largest fraction (MW > 100 kDa) increased from 2.7% to 44.5% during the 1 h treatment. This different pattern of TOC fraction distribution profiles compared to BNR-WAS sample

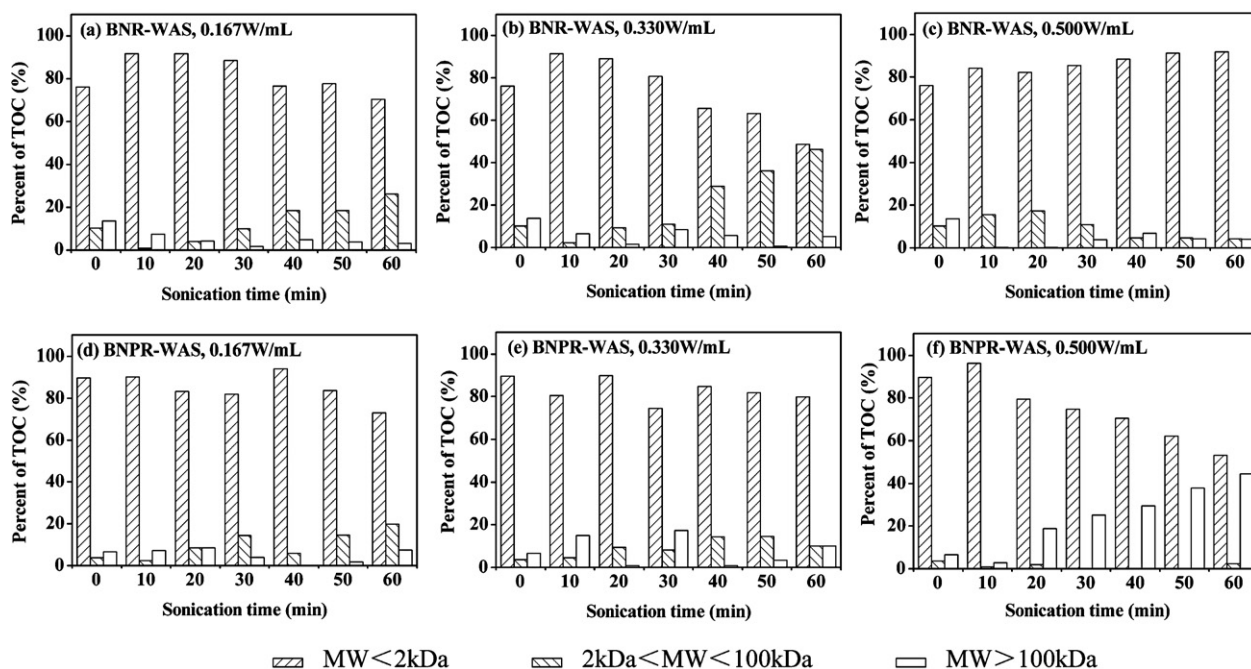


Fig. 3. Distribution profiles of the three TOC MW fractions of the liquid phase.

(Fig. 3c) could be explained by the following reasons. As the specific TOC release from BNPR-WAS (114.9 mg TOC/g TS) was more than three folds of that from BNR-WAS (36.4 mg TOC/g TS) after 1 h treatment at 0.500 W/mL intensity, much more net amounts of soluble TOC with MW < 2 kDa were released from BNPR-WAS sample than those from BNR-WAS sample. Therefore, with the sonication at high intensity the generated $\cdot\text{OH}$ radicals would be trapped more easily for organic matters with MW < 2 kDa during BNPR-WAS treatment, hence resulting in the percentage of MW < 2 kDa decline and MW > 100 kDa increase. On the other hand, it is deduced that floc disintegration occurred significantly and the fragments of cells increased along with large release of the cellular organic constituents at 0.500 W/mL intensity level, as shown in Table 2.

The above observations are consistent with the hypothesis that sonication at an intensity above the critical level would cause a high degree of cell destruction [3,22]. The critical intensity for this study was likely to be within 0.330–0.500 W/mL for the two WAS samples recognizing their different characteristics would affect the sonication performance, such as the dissolution of inorganic constituents.

Generally the low MW organics are more biodegradable [23]. Based on BOD_5/COD data of the liquid phase and the fractional distribution profiles (Fig. 3), the organic species of the sonicated WAS would mostly be biodegraded in the front end biological treatment unit.

3.3. Nitrogen release

Along with the release of the organic compounds, many nitrogen species were also released during sonication of the WAS samples. The cumulative release profiles of TN of the liquid phase are shown in Fig. 4a and the cumulative percentage of TN release profiles are presented in Fig. 4b. Similar to the COD release patterns, the cumulative TN release increased gradually and that BNPR-WAS released more TN to the liquid phase than BNR-WAS. After 1 h of treatment, liquid phase TN concentrations of all sonicated samples were in the range of 200–300 mg/L relative to the initial TN of <31 mg/L. The percentages of the TN release reached 40.9%, 38.7%, 52.1% for BNR-WAS and 46.2%, 61.6%, 70.4% for BNPR-WAS at 0.167, 0.330 and 0.500 W/mL, respectively. Organic-N accounted for most of the TN

released. The ratio of organic-N to TN of the liquid phase increased to 65.0% and 84.9%, respectively for BNR-WAS and BNPR-WAS after 1 h of treatment, while the ratio of $\text{NH}_3\text{-N}$ to TN declined to 34.7% and 14.9% (Fig. 4c and 3d). Under the contribution of oxidative effect of $\cdot\text{OH}$, the concentration of nitrate and nitrite ($\text{NO}_x\text{-N}$) in the supernatant slightly increased with sonication time and intensity. However, the amounts of $\text{NO}_x\text{-N}$ in the supernatants for both BNR-WAS and BNPR-WAS after 1 h sonication treatment were less than 1.40 mg N/L, which could be completely neglected when compared with organic-N and $\text{NH}_3\text{-N}$ amounts.

It has been stated that extra nitrogen loading would have no adverse effect on the biological nitrogen removal performance if the resulting COD/TKN is over 9 [24]. In this study, the sonication resulted in a rapid increase in COD/TKN of the liquid phase (from 1.4 to 1.6 before the treatment) although the rate of increase slowed down after 30 min. After 1 h of treatment, the COD/TKN ratios were 10, 12 and 14 at 0.167, 0.330 and 0.500 W/mL, respectively, for BNR-WAS and 13, 13 and 16 for BNPR-WAS. Therefore, with the necessary modifications, the existing biological treatment unit can accommodate the extra organic and nitrogen loadings of the recycled sonicated WAS. Since most domestic wastewaters have a rather low C/N ratio in southern part of China, the recycling of sonicated WAS may in fact enhance the biological treatment performance since it will result in a more favorable C/N ratio for the influent. Optimization studies should be performed if sonication treatment of WAS is considered to reduce the sludge production of the WWTP.

3.4. Phosphorus release

The cumulative release profiles of TP of the liquid phase are shown in Fig. 5a and cumulative percent TP release profiles are presented in Fig. 5b. Based on the average of five replicate measurements, the TP contents were 3.4–3.9% for BNR-WAS and 7.5–9.0% for BNPR-WAS. For the former, there were no significant differences for TP of the liquid phase (95.6–122.7 mg/L) after 1 h treatment at the three intensities due perhaps to its relatively small phosphorus content. For the latter, much higher and different TP concentrations of the liquid phase of 371.3, 402.3 and 448.0 mg/L, corresponding

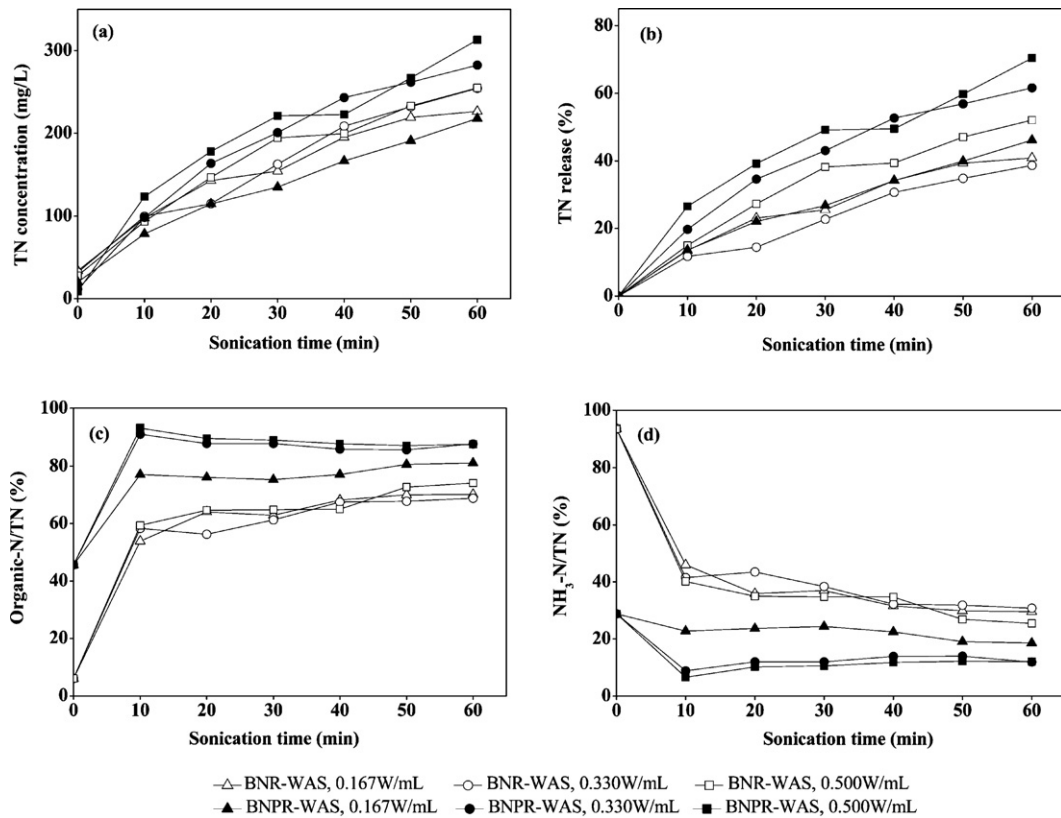


Fig. 4. Nitrogen release profiles of the liquid phase: (a) cumulative release of TN, (b) cumulative percent TN released, (c) organic-N/TN, and (d) NH₃-N/TN.

to 61.4%, 78.1% and 88.2% of TP release (Fig. 5b), were observed after 1 h of treatment at 0.167, 0.330 and 0.500 W/mL, respectively. Such results were consistent with literature reports of phosphorus release by thermal heating [25,26]. Fig. 5c illustrates that the specific amount TP released (TP released per TS) increased only slightly for BNR-WAS during the treatment, while it increased significantly for BNPR-WAS reaching 55.0, 59.7 and 66.6 mg/g TS after 1 h sonication treatment at 0.167, 0.330 and 0.500 W/mL, respectively. Ortho-P was the predominant form (over 80%) of TP released

from BNPR-WAS due most likely to its high density of PAOs; far less ortho-P was found in TP released from BNR-WAS (about 40%) because of its much lower total phosphorus content (Table 1).

Unlike nitrogen removal which can be accomplished by the nitrification–denitrification process, biological phosphorus removal of a wastewater depends on the PAOs function. Direct recycle of the sonicated WAS would significantly increase the phosphorus loading and result in a high phosphorus concentration of the effluent which might exceed the effluent discharge standard.

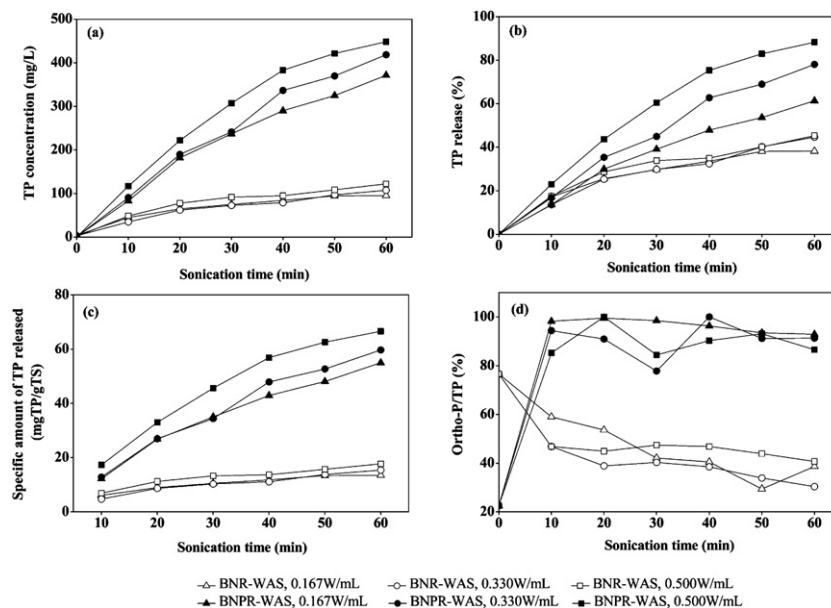


Fig. 5. Phosphorus release profiles of the liquid phase: (a) cumulative release of TP, (b) cumulative percent TP released, (c) specific amount of TP released, and (d) ortho-P/TP.

Such an adverse effect has been reported for the SBR system producing an effluent with 6.0 mg/L of TP, or three to four folds of its normal effluent TP concentration, after the sonicated sludge was included in the feed [27]. Therefore, the extra loading of ortho-P released from sonication of a high TP WAS should be substantially reduced before the ultrasound treated WAS can be recycled to the biological treatment unit. Studies of phosphate recovery by precipitation or crystallization should be performed to design a cost effective sludge reduction process incorporating both sonication and phosphate recovery units in WWTP with biological nitrogen and phosphorus removal system.

4. Conclusions

Series of batch ultrasonic treatment (sonication) experiments were performed on two WAS samples, BNR-WAS from a biological nitrogen removal unit and BNPR-WAS from another biological nitrogen and phosphorus removal unit, to determine the effects of sonication time and intensity on the amount and distribution of solubilized organic matter, nitrogen and phosphorus. The following conclusions are presented based on the results and discussions.

- (1) From the release amounts and percentages of COD, nitrogen and phosphorus, sonication time is more important to the release of cell lysis compared with sonication intensity. However, the release rates could be accelerated when the sonication intensity was above a critical level between 0.330 and 0.500 W/mL. Sonication at an intensity below the critical level, the releases came primarily from dissolution of many EPS constituents; and above it, some cellular constituents were also dissolved.
- (2) After 1 h of treatment at 0.167, 0.330 and 0.500 W/mL intensities, 37.9%, 37.5% and 50.8%, respectively, of the organic content (measured as COD) of BNR-WAS were released, while the same for BNPR-WAS were 40.9%, 55.3% and 56.9%. It also resulted in the release of 40.9%, 38.7%, and 52.1% of TN from BNR-WAS, relative to 46.2%, 61.6%, and 70.4% from BNPR-WAS. Organic-N accounted for the most (65.0% and 84.9%), followed by NH₃-N (34.7% and 14.9%) and negligible amounts of nitrate and nitrite for BNR-WAS and BNPR-WAS.
- (3) The high C/N ratio of the sonicated WAS suggests that most of the released organic and nitrogen species will be removed in the front end biological treatment unit of the WWTP through nitrification–denitrification after the necessary system modifications to accommodate the extra organic and nitrogen loadings associated with the recycling of sonicated WAS. The recycle may enhance the performance of biological treatment of some influents with a low C/N ratio.
- (4) More TP of a higher orthophosphate content was released from BNR-WAS (>60% release after 1 h of sonication, 80% was PO₄-P) than from BNPR-WAS (<50% release, 40% was PO₄-P). The differences in the releases as well as the molecular weight distribution pattern of the soluble TOC species were due to the different structure and composition of the sludge samples tested in this study.
- (5) Sonication is a viable sludge treatment process when it is combined with a phosphorus recovery process to remove most of the released PO₄-P so that the supernatant may be returned for biological treatment in fulfillment of sludge reduction in WWTP.

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