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Extraction of heavy metal from sewage sludge using ultrasound-assisted nitric acid

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ABSTRACT

The effects of nitric acid concentration and sonication time on the removal efficiencies of heavy metals from sludge were investigated. Nitric acid concentrations were varied from 0 to 0.65 M and sonication time from 0 to 20 min. The extracted metals Cu, Zn, and Pb were determined. The results indicated that the removal efficiencies of Cu, Zn, and Pb increased with increases in nitric acid concentration and sonication time. The optimal nitric acid concentration for heavy metals extraction was 0.325 M, while a maximal sonication time of 20 min resulted in maximal heavy metals removal efficiencies. The removal efficiencies of Cu, Zn and Pb reached 9.5%, 82.2%, and 87.3%, respectively, at the optimal concentration of nitric acid assisted by ultrasound for 20 min. Additionally, the residual contents of Zn and Pb in sludge met Chinese legal standards, while Cu was not significantly removed regardless of the nitric acid concentrations and sonication time. The order of heavy metal removal efficiencies was Pb > Zn > Cu. During heavy metal extraction, contribution proportions of ultrasonication and nitric acid to the extraction were 18–22% and 78–82%, respectively. Ultrasound alone was not effective enough to remove heavy metals from sludge and the role played by nitric acid predominated over that of ultrasound. However, ultrasound was synergistic with nitric acid when these were used together to extract heavy metals from sludge. Possible mechanisms of heavy metal extraction are also discussed.

1. Introduction

Biological wastewater treatment processes produce large amounts of surplus sludge. Proper waste treatment and final disposal methods are important for reducing excess sludge. Traditional sludge disposal methods include landfilling, incineration, and agricultural use [1]. In recent years, many countries have developed a series of regulations [2] that forbid the landfilling of solid wastes with high organic matter content due to land consumption and landfilling leachate pollution. Incineration seems to be a good option for sludge disposal; however, it can cause air pollution due to emissions and thus requires expensive off-gas treatment. Moreover, incineration contributes to the greenhouse effect and leads to the production of highly contaminated and hazardous slags and fly ashes, which must be treated [3]. Therefore, the only option left for sludge disposal is land use. The disposal method appears to be an economical and promising option because sludge can supply crops with large amounts of organic matter and inorganic nutrients and improve soil structure. Compared with landfilling and incineration, land use for sludge as organic fertilizer is a more sustainable alternative. Nevertheless, a large number of heavy metals in sludge exceed Chinese legal standards (GB4284-1984) and can become a constraint for sludge application in agriculture in China [4]. Unlike organic pollutants, heavy metals are persistent environmental contaminants that cannot be destroyed [5]. Thus, removing heavy metals from sludge before composting is a necessity for achieving more sustainable sludge treatment.

Currently, there are a few clean-up techniques for removing heavy metals from sludge, including chemical extraction [6], thermal treatment [7], bioleaching treatment [8], cementation and ion exchange [9]. Chemical extraction of heavy metals has received extensive attention due to its simple operation, short extraction time and high removal efficiency. Extraction efficiency of heavy metals depends on pH, temperature, contact time, and extracting agent type. Previous studies showed that low pH, high temperature, and long contact time can improve heavy metal extraction [3]. Various inorganic acids (HNO_3, HCl and H_2SO_4) [10], organic acids (oxalic and citric acid) [3], and strong complexing agents (NTA and EDTA) [11] have been proposed as effective extracting agents. Nevertheless, high extraction efficiency requires a large number of dosages, which results in high processing costs and difficulty in pH adjustment of the sludge compost. Thus, it is necessary to reduce the dosages of extracting agents.

Previous studies have showed that ultrasound, as an assisted extraction method, can efficiently release heavy metals from sludge and shorten extraction time [12–14]. Thus, ultrasound may be

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Table 1
Heavy metal content of sewage sludge compared to Chinese legal standards for heavy metals in sludges for agricultural use (GB4284-1984).

Heavy metal	Value (mg/kg)	Legal standards (mg/kg)
Cu	286 ± 3	≤250
Zn	1258 ± 13	≤500
Pb	751 ± 6	≤300
Cd	2 ± 0.1	≤5
Cr	99 ± 1.4	≤600

regarded as an efficient tool to enhance extraction efficiency of heavy metals together with the use of extracting agents. This study utilized ultrasonic energy to assist nitric acid extraction for heavy metals from sewage sludge in order to improve the ability to extract heavy metals with nitric acid and reduce the dosage of nitric acid required. The advantages of heavy metal extraction by nitric acid over extraction by other inorganic acids include the fact that elemental nitrogen in nitric acid can act as a nutrient source for fertilizers. Additionally, the mechanisms by which ultrasound facilitates heavy metal extraction are also discussed.

2. Experimental

2.1. Materials

Samples of sewage sludge were collected from Datansha Municipal Wastewater Treatment Plant in Guangzhou, China, which utilizes an anaerobic–anoxic–oxic process at a flow rate of 550,000 tons daily. The collected sludge was immediately transferred to the lab and stored in a plastic box at 4 °C prior to use. Heavy metal contents of the sludge samples are presented in Table 1.

2.2. Experimental set-up

The ultrasonic apparatus used in this study has been described in detail in our previous work [15]. It was equipped with an MS73 titanium cylindrical probe with a diameter of 20 mm and a length of 135 mm. It operated at a frequency of 20 kHz and supplied an adjustable power of 300–2000 W. During treatment, the probe was immersed into the sludge at a depth of 10 mm, the power output was 500 W and the sonication time was varied between 0 and 20 min.

3. Methods

3.1. Experimental procedure

Sludge samples of 500 ml were prepared in 1000 ml beakers and a series of concentration of nitric acid varying between 0 and 0.65 M was added and mixed well prior to sonication. Ultrasound was used to assist nitric acid in extracting heavy metals from sludge by varying sonication time from 0 to 20 min. The treated sludge samples were immediately centrifuged in polypropylene centrifuge tubes at 5000 rpm for 30 min at 25 °C (Universal 32 R centrifuge, Hettich, Germany). The supernatant was removed, acidified to pH 1 and stored at 4 °C for analysis of soluble heavy metals. The total heavy metal contents of sewage sludge were also determined by digestion with concentrated nitric and perchloric acid, as described by Walker et al. [16]. The removal efficiency of heavy metal was defined as the heavy metal content of the supernatant divided by the total metal content of the sewage sludge. The pH and oxidation reduction potential (ORP) of sludge samples were measured before and after sonication.

The heating effect during ultrasonication is inevitable and must be taken into account in the design of treatment processes [17]. As reported in the literature [3], a rise in sludge temperature can cause significant heavy metal release. In our work, the change in sludge temperature during ultrasonication was observed and identified to be insignificant [15]. Therefore, no efforts were made to control sludge temperature. Each treatment was performed in triplicate, and average values and standard deviations were obtained.

3.2. Analytical methods

Heavy metals were determined according to standard methods [18] by flame atomic absorption spectrometry (Z-2000, Hitachi).

4. Results and discussion

4.1. Effect of sonication time

Among the five kinds of heavy metals evaluated, Cu, Zn, and Pb exceeded Chinese legal values (Table 1). Thus, only these three heavy metals were investigated. Fig. 1 presents the soluble heavy metal contents of the sludge extracted by 0.065 M nitric acid assisted by ultrasound. The heavy metal contents in the supernatant increased with increasing sonication time whether or not the sludge was extracted by 0.065 M nitric acid. For untreated sludge, the increases were low at less than 5 min and became more rapid with increases in sonication time. The soluble Cu, Zn, and Pb contents increased from 0.4018, 0, 5.125 mg/kg initially to 6.633, 25.63, 12.48 mg/kg, respectively, at 20 min. For the sludge extracted by 0.065 M nitric acid, the soluble Cu, Zn, and Pb contents increased from 0.4018, 445.2, 412.9 mg/kg initially to 10.86, 511.6, 511.6 mg/kg, respectively, at 20 min. Thus, extended ultrasound exposure facilitated the release of heavy metals from sludge. Nevertheless, the extraction efficiency was low for only 20 min of sonication time, while the addition of nitric acid significantly improved the extraction efficiency of heavy metals, except for Cu (Table 2). It is clear that ultrasound alone is not an effective method for extracting heavy metals from sludge. Of course, the extension of sonication time may facilitate to extract more heavy metals from sludge, but it brought about negative effects such as higher energy consumption and stronger break-up of sludge floc structures that caused more difficulty in dewaterability [19].

The extraction efficiency was improved when ultrasound was used to assist nitric acid in extracting heavy metals. When 20 min ultrasound was applied to the sludge extracted by 0.065 M nitric

Table 2
Effect of sonication time on extraction efficiency of heavy metals from the sludge extracted by nitric acid and the changes in sludge pH and ORP.

Sonication time (min)	The initial sludge					The sludge extracted by 0.065 M nitric acid assisted by ultrasound				
	pH	ORP (mV)	Cu (%)	Zn (%)	Pb (%)	pH	ORP (mV)	Cu (%)	Zn (%)	Pb (%)
0	6.65	27.5	0.1	0	0.8	1.57	330.5	0.1	35.4	55.0
1	6.65	27.0	0.1	0	0.7	1.56	328.0	0.2	35.4	59.2
2	6.64	27.5	0.5	0	0.7	1.53	329.0	0.1	36.1	62.7
5	6.65	28.0	0.5	0.2	0.9	1.51	329.0	0.3	38.3	63.1
10	6.63	28.5	1.2	1.2	1.1	1.49	330.5	1.2	40.5	64.8
20	6.62	30.5	2.3	2.0	1.7	1.51	331.5	3.8	40.7	68.2

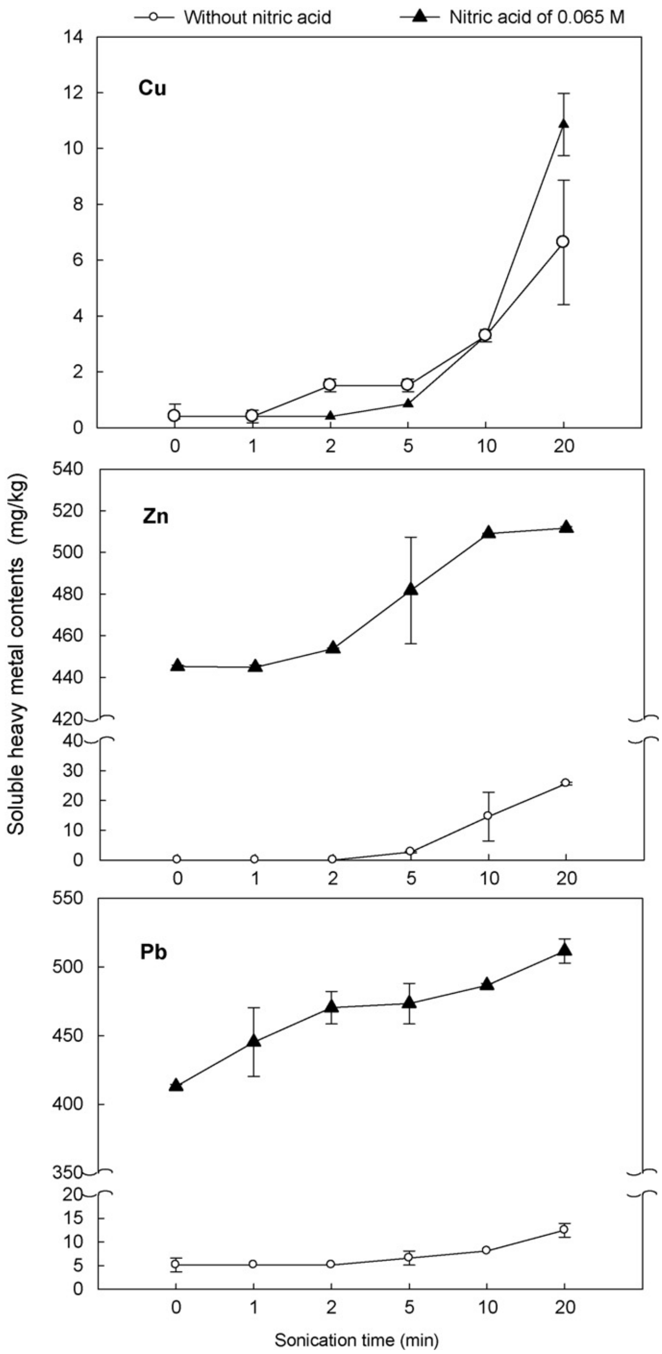

Fig. 1. Effect of sonication time on heavy metal extraction.

acid, the soluble Zn and Pb contents increased by 66.43 mg/kg and 98.65 mg/kg, respectively, with 5.3% and 13.2% increases in extraction efficiencies, where these increases were higher than those generated by ultrasound alone. Thus, ultrasound showed synergy with nitric acid in extracting heavy metals from sludge. Nevertheless, the increase caused by this synergistic effect was very low compared to that generated by nitric acid alone. As was shown in Fig. 1, the soluble Zn and Pb contents in the sludge extracted by 0.065 M nitric acid were 445.2 mg/kg and 412.9 mg/kg, respectively, far more than those in the sludge extracted without nitric acid. It is obvious that the role of nitric acid predominated over that of ultrasound during heavy metal extraction.

On the other hand, both nitric acid and ultrasound seldom worked for Cu extraction, where the extraction efficiency was only 3.8% at 0.065 M nitric acid assisted by ultrasound for 20 min (Table 2). Jenkins et al. [20] reached the same conclusion, namely that Cu was difficult to extract, with extraction efficiencies of less than 10%. Kazi et al. [12] obtained 4.7% removal efficiency for Cu extraction when 0.11 M acetic acid was assisted by 30 min exposure to ultrasound to extract sewage sludge. Although nitric acid was not selective and all the heavy metals were simultaneously extracted from sludge, extraction efficiencies of heavy metals were different under the same conditions. The difficulty in nitric acid extraction of Cu was attributable to its insoluble forms existing in sludge and the stronger binding of Cu to sludge biomass [21].

In the present work, the extraction efficiencies of heavy metals were lower than that reported in the literature [6,20]. This was because only a contact time of 20 min between nitric acid and sludge was very short. Veeken and Hamerlers [3] concluded that extraction efficiencies of Cu and Zn by citric acid reached 60% and 100%, respectively, but that 11 days of extraction time was needed. This would be difficult for large-scale application of acid extraction to remove heavy metals from sludge.

4.2. *Effect of nitric acid concentration*

According to the results in Fig. 1, the maximal removal efficiencies of heavy metals were obtained at the longest testing time, 20 min. Thus, for investigation of the effect of nitric acid concentration, this time was chosen to obtain maximal extraction efficiency. Fig. 2 presents the soluble heavy metal contents of the sludge extracted by nitric acid assisted by ultrasound. The soluble heavy metal contents increased with increasing nitric acid concentration whether or not ultrasound was applied. For the sludge extracted by nitric acid alone, the soluble Cu, Zn, and Pb contents increased from 0.4018, 145.2, 16.99 mg/kg at 0.0065 M to 21.22, 830.6, 537.9 mg/kg at 0.325 M, respectively. For the sludge extracted by nitric acid assisted by ultrasound, the corresponding contents increased from 7.861, 111.6, 81.59 mg/kg to 27.73, 1034, 655.9 mg/kg, respectively. These results illustrate that the synergistic effect of ultrasonication was greater at higher nitric acid concentrations. However, further increases in nitric acid concentration led to only a slight increase in the extraction of heavy metals. Thus, the optimal concentration of nitric acid to extract heavy metals was 0.325 M. Of course, further increases in nitric acid concentration may extract more heavy metals from sludge. However, the increases in processing costs and the difficulty of pH readjustment for sludge composting had to be considered.

Of the heavy metals tested, Pb was most easily extracted and Cu was most difficult to extract (Table 3). The extraction efficiency of Pb reached 68.1% at 0.065 M nitric acid assisted by 20 min of ultrasound exposure, while the Cu extraction efficiency was still only 10%, even when the nitric acid concentration reached 0.65 M and the sonication time was 20 min. Thus, it is not feasible to extract Cu from sludge using nitric acid and ultrasound.

4.3. *Optimal conditions analysis*

It was necessary to determine the optimal extraction conditions and contribution proportions for heavy metal removal to save costs. Table 4 presents the analysis of optimal conditions for heavy metal extraction from sludge. The optimal sonication time and nitric acid concentration were 20 min and 0.325 M, respectively, according to the extraction efficiency of heavy metals shown in Tables 2 and 3. The extraction efficiencies of Cu, Zn, and Pb reached 9.5%, 82.2%, and 87.3%, and the residues in sludge were 258.8, 223.9, and 95.38 mg/kg, respectively, under the optimal conditions. The extraction efficiencies for Zn and Pb were high enough to reduce the contents and meet Chinese legal standards for agricultural use.

Table 3
Effect of nitric acid concentration and 20 min of ultrasound on the extraction efficiency of heavy metals from the sludge and the changes in sludge pH and ORP.

Concentration of nitric acid (mol/l)	The initial sludge					The sludge conditioned by nitric acid assisted by ultrasound of 20 min				
	pH	ORP (mV)	Cu (%)	Zn (%)	Pb (%)	pH	ORP (mV)	Cu (%)	Zn (%)	Pb (%)
0	6.73	35.0	0.1	0.0	0.7	6.67	34.5	2.3	2.0	1.7
0.0065	5.93	79.5	0.1	11.5	2.3	5.89	79.5	2.7	8.9	10.9
0.0325	2.79	275.0	0.1	20.0	8.1	2.24	295.0	3.2	27.9	20.8
0.0650	1.97	324.5	0.1	35.4	55.0	1.60	332.0	3.8	40.7	68.1
0.3250	1.16	374.5	7.4	66.0	71.6	0.75	387.5	9.5	82.2	87.3
0.6500	0.90	389.0	8.2	67.7	73.0	0.46	402.5	10.3	84.4	89.0

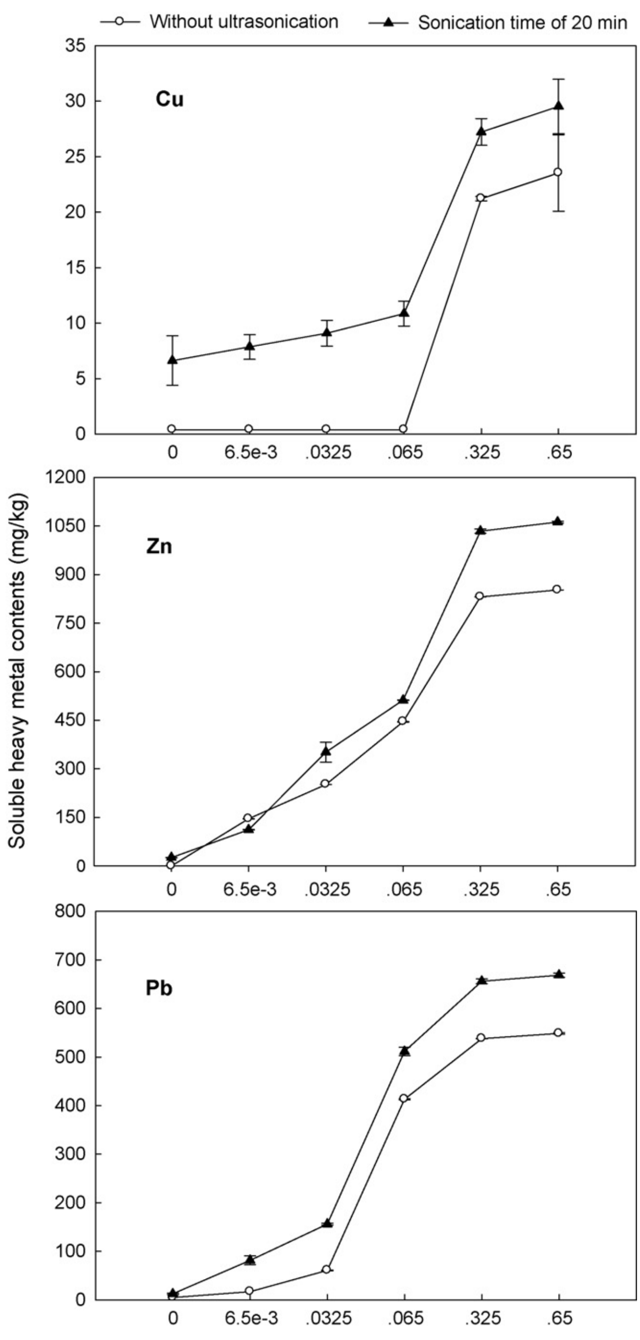

Fig. 2. Effect of nitric acid concentration on heavy metal extraction assisted by ultrasound.

However, the optimal conditions did not extract a sufficient amount of Cu to meet legal standards in large-scale applications.

Additionally, contribution proportions of ultrasonication and nitric acid to the extraction were calculated according to the extraction efficiency of heavy metals at the optimal conditions. The contribution proportion of ultrasonication was calculated as the difference between the extraction efficiency of heavy metals at both optimal conditions and at the single optimal sonication time divided by the extraction efficiency of heavy metals at both optimal conditions, while the contribution proportion of nitric acid was calculated as the extraction efficiency of heavy metals at the single optimal nitric acid concentration condition divided by the removal efficiency of heavy metals at both optimal conditions. The contribution proportions of ultrasonication and nitric acid to the extraction were 18–22% and 78–82%, respectively, for extraction of the three heavy metals. Thus, the extraction effect generated by nitric acid predominated. Ultrasound, as an assisting tool, further improved the heavy metal extraction by nitric acid. Kazi et al. [12] and Arain et al. [13] recently utilized this synergistic effect to significantly reduce extraction time for effective extraction of heavy metals. Ultrasonication was expected to be a substitute for the development of sequential extraction procedures proposed by BCR protocol (the community Bureau of Reference, now the European Union "Measurement and Testing Programme").

4.4. Mechanisms analysis

Previous studies have shown that the extraction of heavy metals from sludge typically consists of three steps: a primary extraction process, solid–liquid separation, and cleaning and recycling of the extracting solution [3]. In the first step of the extraction process, heavy metals are transferred from the solid phase to the aqueous phase. In the second step, the aqueous phase is separated from the sludge by a solid–liquid separation process. In the third step, the heavy metals are removed from the extracting solution to recover the economic value of the extracting agent and prevent environmental impact associated with discharge of extracting liquid. Three steps are necessary regardless of the chemical extraction methods adopted to remove heavy metals. The first step is the most important because it is crucial to determine the extraction efficiency, thus affecting the feasibility of the extraction methods. In the present work, the method of nitric acid extraction assisted by ultrasound

Table 4
Optimal conditions analysis for heavy metal extraction by nitric acid assisted by ultrasound.

Heavy metal	Cu	Zn	Pb
Optimal duration of ultrasonication (min)	20	20	20
Optimal nitric acid concentration (mol/l)	0.325	0.325	0.325
Extracted heavy metal content (mg/kg)	27	1033	656
Extraction efficiency (%)	9.5	82.2	87.3
Contribution proportion of ultrasonication (%)	22.1	19.7	18.0
Contribution proportion of nitric acid (%)	77.9	80.3	82.0

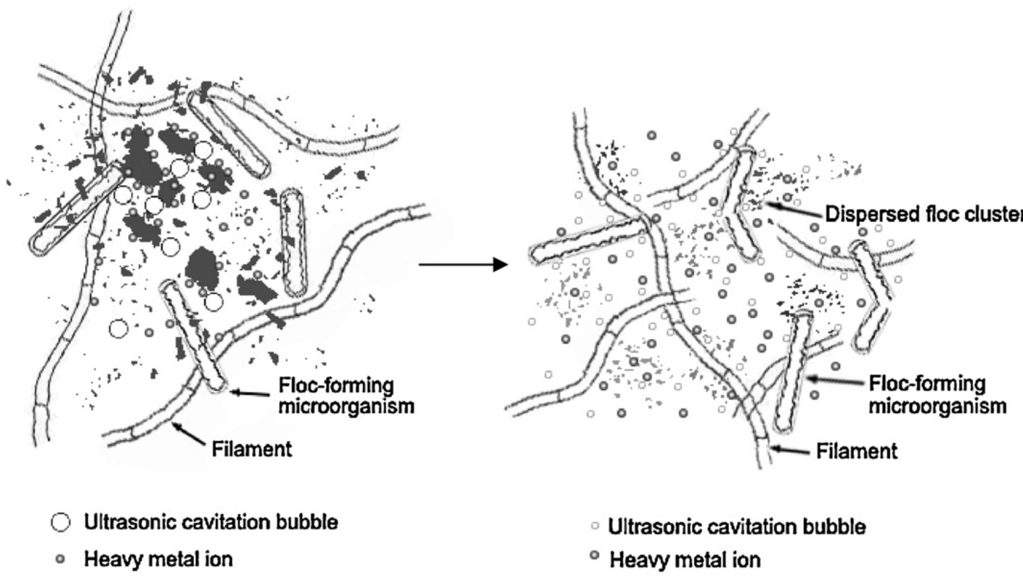

Fig. 3. Mechanism of heavy metal release from sludge flocs by ultrasound.

was used to investigate the extraction efficiency of heavy metals from sludge.

It was clear that both acidification and acoustic cavitation occurred during the extraction and that these were important factors affecting heavy metal extraction. In acidification, higher nitric acid concentrations made the heavy metals more soluble, and the extraction was significant in the concentration range of 0.065–0.65 M, except for Cu extraction (Table 3). This was attributable to the fact that the acidification of nitric acid decreased sludge pH and improved the ability to dissolve heavy metals. Veeken and Hamelers [3] and Marchioretto et al. [22] reached the same conclusion. Additionally, high concentrations of nitric acid also caused the disintegration of sludge floc structures and cell walls. This released the heavy metals attached to the surface of sludge floc structures and cell walls into solution, thus improving the process of dissolving heavy metals. In contrast, acidification at low pH was the main factor in improving the extraction efficiency of heavy metals.

In acoustic cavitation, the process of releasing heavy metals by ultrasound is illustrated in Fig. 3. Ultrasonic cavitation effects are brought about by a large number of cavitation bubbles produced by low-frequency ultrasonication of liquid [23]. The collapse and implosion of these cavitation bubbles instantaneously generates local high temperature, pressure, and shear stress, which cause damage to adjacent substances. Many studies have demonstrated that low-frequency ultrasonication disintegrates sludge floc structures and cell walls and releases intracellular substances into the liquid [15,17,24]. This changes the characteristics of soluble and particulate matters and releases the heavy metals attached to the surface of the sludge particles and flocs into solution, thus improving the dissolving capacity of heavy metals. The ultrasonic cavitation effect further strengthens heavy metal extraction by disintegrating sludge floc structures and cell walls. Thus, ultrasonication generated a good synergy for the improvement of heavy metal extraction by nitric acid. Nevertheless, the effect did not change the sludge pH and ORP (Table 2). The results indicated that the phenomena of acidification and oxidation did not occur during ultrasonication. The improvement of heavy metal extraction by ultrasound was a result of acoustic cavitation, though ultrasound simultaneously caused acoustic cavitation, agitation, and local heating.

Between the two processes, acidification was predominant and acoustic cavitation was an important and necessary assisting method in improving the ability to dissolve heavy metals according to the extraction efficiencies in Tables 2 and 3.

5. Conclusions

The extraction efficiencies of heavy metals increased with increasing sonication time and nitric acid concentration. The maximal sonication time tested was 20 min and generated maximal extraction efficiencies of heavy metals. However, ultrasound alone was not effective enough to extract most of the heavy metals from the sludge. The extraction efficiency was improved when ultrasound was used to assist nitric acid in extracting heavy metals. Ultrasound generated synergy with nitric acid treatment, and the synergistic effect became greater with increasing concentrations of nitric acid. The optimal nitric acid concentration for extraction of heavy metals was 0.325 M. Zn and Pb could be effectively extracted at this optimal concentration when assisted by ultrasound for 20 min, and their residual contents in sludge met legal standards. However, the extraction efficiency of Cu was still rather low regardless of the nitric acid concentrations and sonication time. The order of heavy metal removal efficiencies was Pb > Zn > Cu. During heavy metal extraction, contribution proportions of ultrasonication were 18–22%, while contribution proportions of nitric acid reached 78–82%. Although the role of extraction by nitric acid predominated over that by ultrasound, ultrasound is a necessary assisting method for the improvement of heavy metal extraction.

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