Cite this article

Zeng P, Li J, Cheng J, Song Y and Wei J (2019)
Optimisation of conditions of phosphorus release from pharmaceutical waste sludge.
Journal of Environmental Engineering and Science **14**(1): 13–23,
<https://doi.org/10.1680/jenes.18.00035>

Research Article

Paper 1800035
Received 23/07/2018; Accepted 14/11/2018
Published online 14/12/2018

Keywords: health & safety/pollution/
waste management & disposal

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Optimisation of conditions of phosphorus release from pharmaceutical waste sludge

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With the short supply of phosphorus (P) resources all over the world, more and more phosphorus-rich excess sludge is produced. In this study, a pharmaceutical surplus activated sludge was subjected to alkali and ultrasonic treatment to promote the release of phosphorus. The optimisation of phosphorus release from pharmaceutical surplus activated sludge by alkali and ultrasonic treatment was investigated. The response surface methodology was adopted to optimise the conditions for phosphorus and organic matter release. A quadratic model was established to describe phosphorus and organic matter release. The variance was analysed by using the Design-Expert software, and the optimum operation conditions were decided on. The results indicated that the quadratic model could well fit the relationship between the release of phosphate phosphorus ($\text{PO}_4^{3-}\text{-P}$) and chemical oxygen demand (COD) and the influencing factors. The order of influence on the release of phosphate phosphorus and COD was as follows: pH > total solids > ultrasonic power > time. The maximum release of phosphate phosphorus was 15.3%.

Introduction

Phosphorus (P) is an essential nutrient for living organisms. A large amount of phosphate fertiliser needs to be added to the soil for agricultural development to support the growing population (Reijnders, 2014; Van Vuuren *et al.*, 2010). Phosphate is a non-renewable resource in the world, since almost all phosphorus used in fertilisers is processed from phosphate rocks (Egle *et al.*, 2016) and phosphate rocks are disappearing quickly. The existing phosphate rocks are estimated to last only 50–100 years (Wang, 2010). Thus, it is necessary and urgent to find new phosphate resources and develop efficient technology for phosphorus recovery.

Sewage sludge is an important secondary phosphorus recovery source (Saktaywin *et al.*, 2005). Most of the phosphate in wastewater is transferred into the sludge during the treatment of wastewater and a large amount of sludge has been produced every year (Dong *et al.*, 2013; Yang *et al.*, 2015). With the improvement of water-treatment technology, the phosphorus content in sludge will become higher and higher. However, the direct agricultural use of sewage sludge as fertiliser has been increasingly restricted due to soil and food safety reasons (Ucaroglu and Talinli, 2012). Compared with landfill or agricultural use, incineration was a much more widely used method for sewage sludge treatment at present (Lundin *et al.*, 2004). Therefore, the recovery of phosphorus from sewage sludge has great significance for the sustainable supply of phosphorus.

The key step for phosphorus recovery from sludge is phosphorus release from sewage sludge. Then, phosphate can be precipitated from the aqueous phase as Struvite or calcium phosphate (Jaffer *et al.*, 2002; Parsons and Smith, 2008). The authors' previous study found that phosphorus could be released from sludge by alkali and ultrasonic treatment. However, compared with sludge from municipal wastewater-treatment plants, less phosphorus can be released from the sludge of a pharmaceutical sewage treatment plant treating phosphorus-antibiotic-containing wastewater. The authors hope to find the optimal conditions such as total solids (TS), pH, ultrasonic power and time for phosphorus release from pharmaceutical sludge to increase phosphorus release.

The response surface methodology (RSM) was adopted to optimise the conditions for phosphorus release in this study. RSM consists of a group of mathematical and statistical techniques which are used in the development of an adequate functional relationship between a response of interest and a number of associated conditions variables (Tong and Chen, 2009). In general, such a relationship is unknown but can be approximated by a low-degree polynomial model. RSM can display the relationship by the graph, so that the optimal conditions can be chosen by intuitive observation. RSM has been widely used in many fields such as biometrics (Mead and Pike, 1975), the chemical industry (Hill and Hunter, 1966) and environmental science (Ahmadi *et al.*, 2005; Zhai *et al.*, 2017).

Table 1. Characteristics of the surplus activated sludge

Parameter	Average value
pH	7.96
TS: g/g	0.91
VS: g/g	0.51
TC: mg/g	299.50
TP: mg/g	9.02
TN: mg/g	22.61

In this study, RSM was applied to optimise the conditions that affect the release of phosphate phosphorus ($\text{PO}_4^{3-}\text{-P}$) and organic matters from pharmaceutical wastewater.

Materials and methods

Source of sewage sludge

The surplus activated sludge was collected from one pharmaceutical wastewater-treatment plant whose influent contained fosfomycin. Fosfomycin is a kind of antibiotic with phosphorus in its structure. The collected condensed sludge was air-dried for 10 d at ambient temperature, ground in a hammer mill and screened with a 0.45 mm mesh and then stored at -20°C for later experiments. The characteristics of the surplus activated sludge before treatment are shown in Table 1.

Experimental instruments

The ultrasonic instrument was composed of a reactor, an ultrasonic producer, a water-recycling cooling system and a magnetic stirrer, as shown in Figure 1. A mixture of sludge and water were put into the 300 ml reactor, and the reactor stayed in a 1000 ml beaker with cooling water. The frequency and the time of ultrasonic-chemical treatment were 20–25 kHz and 30 min, respectively. The ultrasonic-chemical pretreatment was performed with an ultrasonicator (Scientz-IID, Ningbo Xinzhi Ultrasonic

Electronic Equipment Company, China). The operation condition was set as follows: intermediate impulse ratio of 1:1 and sound energy density of 2 W/ml. During the pretreatment, the prepared sample was mixed with a magnetic stirrer. The tip diameter of the ultrasonic detector was 6 mm, and the tip was put under a 1 cm mixed liquor. The temperature was set at 20°C by the heater and cooling water-recycling system.

Batch experiment

The dried sludge was put into the reactor. All the samples were treated in triplicate. Deionised water (150 ml) was added into the first part as blank. Added to the second part was 150 ml deionised water, with the pH value adjusted using saturation sodium hydroxide (NaOH) solution. The third part was subjected to ultrasonic treatment after the pH value had been adjusted to the set value. The sludge was left for 20 h after ultrasonic instrument (intermediate impulse ratio of 1:1). The solid and liquid components were separated by using a centrifuge machine (Thermo Fisher Scientific, USA) for 20 min at 10 000 revolutions/min.

Response surface methodology

In order to find the optimal conditions for phosphorus and organic matter release as indicated by chemical oxygen demand (COD) release from pharmaceutical waste activated sludge by ultrasonic and alkaline pretreatment, RSM was introduced and designed by the software Design-Expert v8.0 (Stat-Ease Inc., Minneapolis, USA). Based on these experiments, four factors (TS, pH, ultrasonic time, ultrasonic power) were set as independent variables and named X_1 – X_4 , respectively. Five differently coded levels (-2 , -1 , 0 , $+1$, $+2$) with TS from 4.5 to 18.5 g, pH value from 10 to 14, ultrasonic time from 15 to 75 min, ultrasonic power from 175 to 475 W were adopted for each factor using Box–Behnken design analysis (Table 2), and the specific experimental arrangement is listed in Table 3. Concentrations of COD and phosphate in solutions were set as responses 1 and 2. For predicting the optimal point, a function was fitted to correlate the relationship between independent variables and responses.

Analytical methods

The total carbon (TC) of the sludge was detected by a total organic carbon (TOC)/total nitrogen TN analyser (Multin N/C 2100, Jena, Germany). The supernatant of the sludge was detected by a TOC machine (TOC-L, CPN, CN200, Shimadzu, Japan). Total phosphorus (TP), pH, phosphate phosphorus, TN, total COD and soluble COD were determined according to a standard

Figure 1. Schematic diagram of ultrasonic treatment device.
1, sound-proof box; 2, ultrasonic detector; 3, beaker; 4, beaker;
5, cooling water; 6, cooler; 7, magnetic stirrer; 8, operator;
9, constant-temperature circulator

Table 2. Codes and levels of RSM design factors

Factor	Code	Value of factors				
		-2	-1	0	1	2
TS: g	X_1	4.5	8	11.5	15	18.5
pH	X_2	10	11	12	13	14
Ultrasonic time: min	X_3	15	30	45	60	75
Ultrasonic power: W	X_4	175	250	325	400	475

Table 3. The experimental design and results of RSM

Series number	Value				RSM	
	X_1	X_2	X_3	X_4	Phosphate phosphorus release: $\mu\text{g/g}$	COD release: mg/g
1	0	-2	0	0	2.75	11.41
2	-1	1	1	1	214.36	131.80
3	1	-1	1	-1	4.74	37.91
4	-1	-1	-1	1	6.41	25.27
5	-1	1	-1	-1	160.66	97.85
6	0	0	0	0	32.57	51.90
7	0	0	0	0	32.57	51.90
8	1	-1	-1	-1	3.37	35.15
9	0	2	0	0	3521.74	895.88
10	1	1	-1	1	474.60	139.11
11	-1	-1	1	-1	7.27	23.40
12	2	0	0	0	32.16	46.79
13	-2	0	0	0	46.69	42.98
14	0	0	0	-2	24.42	42.61
15	1	1	1	-1	493.90	158.44
16	1	1	-1	-1	448.74	141.54
17	0	0	0	0	32.57	51.90
18	1	-1	1	1	5.78	39.30
19	-1	-1	-1	-1	5.35	23.04
20	0	0	-2	0	14.99	35.13
21	-1	1	-1	1	187.60	95.14
22	0	0	0	0	32.57	51.90
23	1	1	1	1	516.43	185.88
24	0	0	0	0	32.57	51.90
25	1	-1	-1	1	4.35	33.38
26	0	0	0	0	32.57	51.90
27	0	0	2	0	42.75	60.45
28	-1	1	1	-1	209.34	131.23
29	-1	-1	1	1	7.82	14.41
30	0	0	0	2	35.60	50.72

method (Apha *et al.*, 1998). TS and volatile solids (VS) were determined according to the method of the American Public Health Association (Apha *et al.*, 1998). TS0, VS0 and concentration of phosphorus (CP) 0 referred to the parameters of sludge without pretreatment, while total solid sludge (TSS) and volatile solid sludge (VSS) are the parameters total suspended solids and volatile suspended solids after pretreatment, respectively (Liu *et al.*, 2008). The soluble ratios of TS and VS (STS and SVS, respectively) were then calculated as follows

$$1. \quad \text{STS} = (\text{TS0} - \text{TSS})/\text{TS0} \times 100 \%$$

$$2. \quad \text{SVS} = (\text{VS0} - \text{VSS})/\text{VS0} \times 100 \%$$

Results

Model establishment and saliency analysis

The Design-Expert software was used to perform linear fitting of data in tables (Tong and Chen, 2009; Wan *et al.*, 2017), and two polynomial regression equations for phosphate phosphorus release and COD release with TS (X_1), pH (X_2), ultrasonic time (X_3) and ultrasonic power (X_4) were established, as shown in Equations 3 and 4. The results are shown in Tables 3 and 4.

$$Y_{\text{PO}_4^{3-}\text{-P release}} = 32.57 + 46.84X_1 + 404.11X_2 + 9.34X_3 + 4.43X_4 + 73.14X_1X_2 + 0.69X_1X_3 + 1.05X_1X_4 + 9.77X_2X_3 + 4.80X_2X_4 - 1.61X_3X_4 - 47.16X_1^2 + 383.55X_2^2 - 49.80X_3^2 - 49.01X_4^2$$

$$Y_{\text{COD release}} = 51.90 + 9.59X_1 + 109.34X_2 + 7.09X_3 + 1.85X_4 + 6.06X_1X_2 + 1.18X_1X_3 + 1.71X_1X_4 + 8.09X_2X_3 + 2.26X_2X_4 + 0.08X_3X_4 - 12.66X_1^2 + 89.53X_2^2 - 11.93X_3^2 - 12.21X_4^2$$

A fitted three-dimensional response surface plot was generated by the above statistical model, and the corresponding predicted contour lines of phosphate phosphorus and COD release could be observed in the contour diagrams (Figures 2–7).

Interaction between pH and TS

The interaction between pH and TS on the response surface and contour map of phosphate phosphorus and COD release was

Table 4. Analysis of variance for regression model

Variation source	Phosphate phosphorus					COD				
	Quadratic sum	Degrees of freedom	Mean square	F	P	Quadratic sum	Degrees of freedom	Mean square	F	P
Model	8 818 600	14	629 900	2.9488 36	0.0230	5.54×10^5	14	39 592.03	3.41	0.0122
X_1	52 645.39	1	52 645.39	0.2464 56	0.6268	2205.81	1	2205.81	0.19	0.6689
X_2	3 919 239	1	3 919 239	18.347660	0.0007	2.87×10^5	1	2.87×10^5	24.75	0.0002
X_3	2092.32	1	2092.32	0.0097 95	0.9225	1206.95	1	1206.95	0.1	0.7514
X_4	471.0868	1	471.0868	0.0022 05	0.9632	81.82	1	81.82	7.06×10^{-3}	0.9342
X_1X_2	85 601.74	1	85 601.74	0.4007 39	0.5362	587.62	1	587.62	0.051	0.8249
X_1X_3	7.5730 78	1	7.5730 78	3.55×10^{-5}	0.9953	22.41	1	22.41	1.93×10^{-3}	0.9655
X_1X_4	17.7149	1	17.7149	8.29×10^{-5}	0.9929	46.95	1	46.95	4.05×10^{-3}	0.9501
X_2X_3	1526.599	1	1526.599	0.0071 47	0.9337	1046.8	1	1046.8	0.09	0.7679
X_2X_4	368.061	1	368.061	0.0017 23	0.9674	81.52	1	81.52	7.03×10^{-3}	0.9343
X_3X_4	41.31932	1	41.31932	0.0001 93	0.9891	10.12	1	10.12	8.73×10^{-4}	0.9768
X_1^2	60 991.75	1	60 991.75	0.2855 29	0.6009	4395.09	1	4395.09	0.38	0.5473
X_2^2	4 035 017	1	4 035 017	18.889680	0.0006	2.20×10^5	1	2.20×10^5	18.96	0.0006
X_3^2	68 010.93	1	68 010.93	0.3183 89	0.5809	3904.54	1	3904.54	0.34	0.5703
X_4^2	67 234.46	1	67 234.46	0.3147 54	0.5831	4090.6	1	4090.6	0.35	0.5614
Residue	3 204 145	15	213 609.7			1.74×10^5	15	11 595.25		
Lack of fit	3 204 145	10	320 414.5			1.74×10^5	10	17 392.87		
Error	0	5	0			0	5	0		
Sum	1202×10^7	29				7.3×10^5		29		
			$R = 0.8564$					$R = 0.8725$		

studied, as shown in Figure 2. The release of phosphate phosphorus increased with the increase in pH and TS. When the TS used in the experiment ranged from 8 to 15 g, the amount of phosphate phosphorus release at pH from 11 to 12 was very small. With the increase in pH from 12 to 13, the release of phosphate phosphorus increased gradually. When the pH is between 11 and 13, the amount of phosphate phosphorus released increased with the increase in TS, but the increase was very small. When the TS changed from 13 to 15 g, the amount of phosphate phosphorus released was higher. The effect of pH and TS interaction on COD release was particularly similar to that of phosphate phosphorus release. However, the perfect interaction between pH and TS would be reflected by elliptical contours (Muralidhar *et al.*, 2001). In this case, the uncompleted elliptical contours were detected in contour lines of TS and pH, which indicated the independence of the three parameters.

Interaction between TS and ultrasonic time

Figure 3 shows the effect of interaction between TS and ultrasonic time on the response surface and contour map of phosphate phosphorus and COD release. It is found that the release of phosphate phosphorus increased with the increase in TS. When TS ranged from 10.3 to 15 g, the release of phosphate phosphorus increased with the increase in ultrasonic time, with the highest release of phosphate phosphorus achieved at 48 min. A further increase in ultrasonic time caused the phosphate phosphorus release to decrease slightly, indicating that the ultrasonic time of 48 min was enough. The release of COD increased with the increase in TS and ultrasonic time in a certain range. It can be seen from the variation trend of the

contour lines shown in Figure 3(d) that the elliptical contours represent a perfect interaction between TS and ultrasonic time. Thus, a perfect correlation between TS and ultrasonic time is detected. In addition, the COD release amount was of the maximum value when TS was 12–14 g and ultrasonic time was 48–54 min.

Interaction between TS and ultrasonic power

Shown in Figure 4 is the effect of interaction between TS and ultrasonic power on the response surface and contour map of phosphate phosphorus and COD release. It can be seen that the release of phosphate phosphorus increased with the increase in TS. The release phosphate phosphorus initially increased with the increase in ultrasonic power, with the highest released amount of phosphate phosphorus reached at an ultrasonic power of 310–350 W. A further increase in ultrasonic power caused the phosphate phosphorus release to decrease slightly, indicating that the an ultrasonic power of about 325 W was enough. Similar to the interaction between TS and ultrasonic time, elliptical contours indicated a perfect interaction between TS and ultrasonic power. The effect of TS and ultrasonic power on COD release was particularly similar to that of phosphate phosphorus release. The release of COD and phosphate phosphorus attained the maximum value at TS of 12–14 g and ultrasonic power of 310–350 W.

Interaction between pH and ultrasonic time

The effect of the interaction between pH and ultrasonic time on phosphate phosphorus release is illustrated in Figure 5. The pH had a significant effect on phosphate phosphorus release. The

Figure 2. The effect of interaction between pH and TS on response surface and contour map of phosphate phosphorus and COD release. (a) The effect of interaction between pH and TS on phosphate phosphorus release. (b) Contour map of phosphate phosphorus release affected by pH and TS. (c) The effect of interaction between pH and TS on COD release. (d) Contour map of COD release affected by pH and TS

amount of phosphate phosphorus released increased significantly when the pH was between 11.5 and 13. In addition, the uncompleted elliptical contours indicated the independence of ultrasonic time and pH. Ultrasonic time did not have much influence on phosphate phosphorus release. The effect of pH and ultrasonic time on COD release was just particularly similar to that of phosphate phosphorus release.

Interaction between pH and ultrasonic power

The effect of interaction between pH and ultrasonic power on the response surface and contour map of phosphate phosphorus release is shown in Figure 6. It can be seen that pH had a significant influence on the release of phosphate phosphorus. The release of phosphate phosphorus increased significantly when the

pH was 11.5–13, while ultrasonic power did not have much influence on phosphate phosphorus release. The effect of pH and ultrasonic power on COD release was also particularly similar to that of phosphate phosphorus release.

Interaction between ultrasonic time and ultrasonic power

The effect of the interaction between ultrasonic time and ultrasonic power on phosphate phosphorus release was also studied. As shown in Figure 7, both ultrasonic time and ultrasonic power do not have much influence on the release of phosphate phosphorus. With the increase in ultrasonic time and ultrasonic power, the release of both phosphate phosphorus and COD increased first and then decreased. The amount of COD and phosphate phosphorus release increased to the maximum value at

Figure 3. The effect of interaction between TS and ultrasonic time on response surface and contour map of phosphate phosphorus and COD release. (a) The effect of interaction between ultrasonic time and TS on phosphate phosphorus release. (b) Contour map of phosphate phosphorus release affected by ultrasonic time and TS. (c) The effect of interaction between ultrasonic time and TS on COD release. (d) Contour map of COD release affected by ultrasonic time and TS

an ultrasonic time of 42–50 min and an ultrasonic power of 310–350 W.

Discussion

Compared with sludge from a municipal wastewater-treatment plant, less phosphorus can be released from the sludge of a pharmaceutical sewage treatment plant treating phosphorus-antibiotic-containing wastewater (Liu *et al.*, 2014). After ultrasonic and alkaline pretreatment, the amounts of phosphate phosphorus released by the two kinds of municipal sludge were 12.60 and 2.24 mg/g, respectively, under the conditions of a pH of 12, ultrasonic time of 30 min and ultrasonic energy of 2 W/ml. However, the phosphate phosphorus released by the pharmaceutical sludge was 0.05 mg/g. For the pharmaceutical sludge, the amount of phosphate phosphorus released was

0.05 mg/g, while its TP was 8.92 mg/g. For the Qinhe sludge, one kind of municipal sludge, the amount of phosphate phosphorus released was 12.60 mg/g, while its TP was 13.07 mg/g. It seemed that the operating conditions needed to be improved.

In this study, optimal conditions were determined to improve the release of phosphate phosphorus and organic matter. The RSM model was applied in this study. *P* is the standard for checking the regression model. When $P < 0.01$, the regression equation is highly significant. When $0.01 < P < 0.05$, the regression equation is significant. When $P > 0.5$, the regression equation is not significant. As shown in Table 4, the regression model in Equation 3 for phosphate phosphorus release obtained a P of $0.01 < P < 0.05$, indicating that the regression model in Equation 3 was significant. In addition, the regression model in Equation 3 had an

Figure 4. The effect of interaction between TS and ultrasonic power on response surface and contour map of phosphate phosphorus and COD release. (a) The effect of interaction between ultrasonic power and TS on phosphate phosphorus release. (b) Contour map of phosphate phosphorus release affected by ultrasonic power and TS. (c) The effect of interaction between ultrasonic power and TS on COD release. (d) Contour map of COD release affected by ultrasonic power and TS

R of 0.8564, showing that there was a high correlation between the measured values and the predicted values. Therefore, the regression model in Equation 3 can be used to predict the release of phosphate phosphorus. It can also be seen from Table 4 that TS, ultrasonic time and ultrasonic power had no significant influence on phosphate phosphorus release, but pH had a significant influence on phosphate phosphorus release. The interactions of the four factors were not significant except for X_1X_2 , indicating that the interaction of TS and pH was the most significant interaction for phosphate phosphorus release.

The value of F suggested the different effects on phosphate phosphorus and COD release: a higher F meant a more significant effect, while a lower F meant a less significant effect. Thus, in this study, the significance of the four factors on phosphate

phosphorus and COD release was as follows: $\text{pH} > \text{TS} > \text{ultrasonic time} > \text{ultrasonic power}$. pH had the most obvious effect on phosphate phosphorus and COD release, suggesting that alkaline treatment was the primary factor in detaching phosphate phosphorus and organic matter.

Compared with the phosphate phosphorus release ratio before and after optimisation, the phosphate phosphorus release ratio of pharmaceutical sludge increased from 0.55 to 15.3%. It was lower than the phosphate phosphorus release ratio of Qinhe sludge, which was 47.7% (Liu *et al.*, 2014). Considering the influent of the pharmaceutical wastewater-treatment plant, fosfomycin, containing organic phosphorus, was fed to the activated sludge as one component (Qiu *et al.*, 2011). Thus, part of TP came from fosfomycin. The triangular structure of

Figure 5. The effect of interaction between pH and ultrasonic time on response surface and contour map of phosphate phosphorus and COD release. (a) The effect of interaction between ultrasonic time and pH on phosphate phosphorus release. (b) Contour map of phosphate phosphorus release affected by ultrasonic time and pH. (c) The effect of interaction between ultrasonic time and pH on COD release. (d) Contour map of COD release affected by ultrasonic time and pH

fosfomycin possessed high bond energy, which caused the difficulty of breaking the structure and degrading it completely. The low release ratio of phosphate phosphorus by ultrasonic and alkaline treatment indicated that a stronger pretreatment is desired in the future to release phosphate phosphorus from sludge.

Conclusion

In this study, a quadratic model was applied to simulate the phosphate phosphorus and COD release from pharmaceutical activated sludge treated by alkaline and ultrasonic treatment. The quadratic model could well fit the relationship between the release of phosphate phosphorus and COD and the influencing factors. The effect of pH, TS, ultrasonic power and ultrasonic time on phosphate phosphorus and COD release followed the order $\text{pH} > \text{TS} > \text{ultrasonic power} > \text{ultrasonic time}$. The maximum release of phosphate phosphorus was 15.3% under the optimal conditions of

pH of 13, TS of 15 g, ultrasonic time of 48 min and ultrasonic power of 332.6 W.

Acknowledgements

This research was funded by the National Major Scientific and Technological Projects for Water Pollution Control and Management (2017ZX07402003), the Chinese Research Academy of Environmental Sciences Central Public Welfare Scientific Research Project (2016YSKY-027, 2016YSKY-005), the Scientific Research Foundation for the Returned Overseas Chinese Scholars and the Ministry of Education study on phosphorus recovery from surplus activated sludge.

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Figure 6. The effect of interaction between pH and ultrasonic power on response surface and contour map of phosphate phosphorus and COD release. (a) The effect of interaction between ultrasonic power and pH on phosphate phosphorus release. (b) Contour map of phosphate phosphorus release affected by ultrasonic power and pH. (c) The effect of interaction between ultrasonic power and pH on COD release. (d) Contour map of COD release affected by ultrasonic power and pH

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Figure 7. The effect of interaction between ultrasonic time and ultrasonic power on response surface and contour map of phosphate phosphorus and COD release. (a) The effect of interaction between ultrasonic power and ultrasonic time on phosphate phosphorus release. (b) Contour map of phosphate phosphorus release affected by ultrasonic power and ultrasonic time. (c) The effect of interaction between ultrasonic power and ultrasonic time on COD release. (d) Contour map of COD release affected by ultrasonic power and ultrasonic time

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