Response surface methodology for the optimization of sludge solubilization by ultrasonic pre-treatment

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Abstract. The present study examines the optimization of the ultrasonic pre-treatment conditions with response surface experimental design in terms of sludge disintegration efficiency (solubilisation of organic components). Ultrasonic pre-treatment for the maximum solubilization with residual sludge enhanced the SCOD release. Optimization of the ultrasonic pre-treatment was conducted through a Box-Behnken design (three variables, a total of 17 experiments) to determine the effects of three independent variables (power, residence time and TS) on COD solubilization of sludge. The optimal COD was obtained at 17349.4mg/L, when the power was 534.67W, the time was 10.77, and TS was 2%, while the SE of this condition was 28792J/kg TS.

1. Introduction
The sludge production is huge in municipal wastewater treatment plant (WWTP). The water industry is facing unprecedented economic and environmental constraints because of large amounts of sewage sludge produced. The disposal of sewage sludge is one of the expensive items and complicated problem in a WWTP[1]. Since most of the sludge is composed of microbial cells, cell wall block the organic matter in sludge, the disposal is also one of the most important. The sludge pre-treatment technology is necessary for promoting organic matter release, improve the utilization efficiency of sludge[2].

To accelerate the hydrolysis and enhance subsequent methane productivity, a variety of sludge pre-treatment options, such as heat treatment, alkali treatment, ozone oxidation treatment, ultrasonic, mechanical processing, microwave radiation, and a variety of joint pre-treatment technology have been developed at laboratory or pilot level[3].

Pre-treatment of sludge by ultrasonic technology, observe the effect of ultrasonic time and ultrasonic frequency of sludge pre-treatment effect. Prolonging the time of ultrasonic action or increasing the frequency of ultrasonic wave are helpful to release the organic matter, nitrogen and phosphorus in the sludge[4].

In this paper, optimization of the ultrasonic pre-treatment conditions with response surface experimental design in terms of sludge disintegration efficiency (solubilisation of organic components) was reported

2. Materials and methods
2.1. Sludge samples
Optimization of the ultrasonic pre-treatment for the maximum solubilization was performed with residual sludge taken from a waste water treatment plant (WWTP) in Beijing, China. The TS of the sludge was 12.1%. The SCOD of sludge was 3805.7 mg COD/L before the pre-treatment process.

2.2. Pretreatment assays
The ultrasonic processor (FS-600N, 20 kHz, 600W) was used for the experiments. The ultrasonic pre-treatment was carried out in a 0.6 L glass reactor, in which 100g thickened sludge was introduced. The ultrasonic energy was applied to the sludge using a probe which submerged in the sludge.

2.3. Analytical methods
The total solids (TS) content of the wastewater was determined as the residue after drying a sludge sample at 105°C to constant weight. These components were determined according to the standard methods (APHA, 2006). The chemical oxygen demand (COD) was determined using standard COD test tubes (HACH DR200, DR2800). The analyses were performed on the supernatant (obtained by centrifugation at 5,000 rpm and subsequent filtration through 0.22μm microfiber filter paper).

2.3.1. Data analysis
Optimization of the ultrasonic pre-treatment was conducted through a Box-Behnken design (three variables, a total of 17 experiments) to determine the effects of three independent variables (Power, residence time and initial TS) on COD solubilization of sludge.

3. Results and discussion

3.1. The summary of these experimental conditions determined
Ultrasoundication is a well-established mechanical technology for sludge disintegration. It is very important to determine the reasonable experimental parameters for the performance of ultrasonic pre-treatment.

In the literatures, sludge solubilization efficiency relies on specific energy (SE) input \( SE = \frac{P \times t}{(m \times TS)} \) J /kg TS\(^{[4]}\). For complete disaggregation of sludge flocs around 80 kJ/L is required, while for the damage and death of the released free cells, between 1000 and 16,000 kJ/kg TS is usually reported in literature\(^{[5]}\).

The more insoluble organics into soluble form in the sludge, the more SE increased, while the greater the energy consumption.

The three parameters affect the SE were power(P), time(T) and sludge concentration(TS). To obtain optimal parameters for the solubilization of COD, response surface methodology (RSM) is introduced. RSM is a statistical technique that can be used to selected responses and determine optimal conditions. In this experiment, the power was from 60-540 W, and the time was from 0.5-11.5 min, and the TS was from 1-11%. The total number of observations required was 17. All experimental points were tested in three times.

| No. | Power(W) | Time(min) | TS (%) | SE (J/Kg) |
|-----|----------|-----------|--------|-----------|
| 1   | 300      | 6         | 6      | 9000      |
| 2   | 540      | 6         | 1      | 16200     |
| 3   | 300      | 11.5      | 11     | 17250     |
| 4   | 300      | 6         | 6      | 9000      |
| 5   | 300      | 11.5      | 1      | 17250     |
| 6   | 300      | 6         | 6      | 9000      |
| 7   | 300      | 6         | 6      | 9000      |
| 8   | 60       | 0.5       | 6      | 150       |
| 9   | 300      | 0.5       | 1      | 750       |
| 10  | 60       | 6         | 1      | 1800      |
3.2. Optimization of chemical oxygen demand solubilization by response surface methodology

The common influence of the three parameters of comprehensive power, time and TS was analyzed by Design-Expert software, and the maximum SCOD value was 17349.4 mg/L, while the SE of this condition was 28792 J/kg TS. The condition was shown in Table 2. The results were consistent with the literature. For instance, Feng et al. [5] investigated the effect of ultrasound the physical-chemical characteristics of sludge and confirmed that further increase in the applied SE to 26,000 kJ/kg TS contributes more to the destruction of sludge flocs and the transformation of insoluble organics into soluble form [2].

Table 2. The conditions of Maximum SCOD obtained

| Power (W) | Time (min) | TS (%) | SCODmax (mg/L) |
|----------|------------|--------|----------------|
| 534.67   | 10.77      | 2.00   | 17349.4        |

Figure 1. The conditions of SCOD simulated by RSM

4. Conclusions

Ultrasonic pre-treatment for the maximum solubilization with residual sludge enhanced the SCOD release. The optimal COD was obtained at 17349.4 mg/L, when the power was 534.67 W, the time was 10.77, and TS was 2%, while the SE of this condition was 28792 J/kg TS.

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