ROLE OF EXTRACELLULAR POLYMERIC SUBSTANCES IN DEWATERABILITY OF UNTREATED, SONICATED AND DIGESTED WASTE ACTIVATED SLUDGE

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ABSTRACT

Dewatering of waste activated sludge is a complex process because of the influences of different factors. Previous studies have shown that extracellular polymeric substances (EPS) are the major constituents of waste activated sludge. Results of previous works on determining the role of EPS on dewaterability of WAS are limited, different and sometimes seem controversial. In this study, protein and carbohydrate parts of EPS were measured in untreated, sonicated and digested waste activated sludge and the relationship between these parameters to dewaterability of sludge was found. In untreated sludge samples, a decrease of dewaterability (increase of specific capillary suction time) was observed with the decrease of protein and carbohydrate parts of EPS. On the other hand, sonication and digestion caused an increase in the amounts of protein and carbohydrate parts of EPS, but a decrease in dewaterability of sludge samples. It is also shown that sonication, in general, made the dewatering of sludge more difficult even after biological digestion. Sonication, however, caused 26.5%, 18.6% and 3.8% dewaterability improvement at sonication intensities of 18.4, 73.6 and 165.6 W/cm², respectively.

Key words: Capillary Suction Time; Dewaterability; Digestion; Extracellular polymeric substances; Sonication

INTRODUCTION

Waste activated sludge (WAS) which is an inevitable drawback of activated sludge process consists of a large amount of water. WAS is typically pumped from secondary settling tank with a content of 99.2% water (Metcalf and Eddy, 2003). Therefore, the dewatering is considered as an effective method of their volume reduction (Scholz *et al.*, 2005). Dewatering of WAS, however, is not always an easy process. In fact, sludge dewatering is one of the most expensive and least understood processes in wastewater treatment (Liu *et al.*, 2003; Feng *et al.*, 2009).

The lack of understanding and knowledge about sludge dewatering is largely due to the complex

nature of sludge (Liu *et al.*, 2003). Solid part of biological sludge is composed of microbial cells and extra-cellular polymeric substances (EPS) that crosslink cells together (Comte *et al.*, 2007). The exact function of the EPS matrix is still uncertain but some of the confirmed functions are; adhesion to surfaces, aggregation of bacterial cells in flocs and formation of a protective barrier that provides resistance to harmful affects such as biocides (Liu *et al.*, 2003). Trying to achieve better understanding of sludge, this paper intended to evaluate the effect of such biological parameters as type and concentration of extracellular polymeric substances on the waste activated sludge dewaterability.

Studies on ultrasound application for the pretreatment of sludge have increased in number

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during the last decade (Akin, 2008). The aim of sonication treatment in this field is to solubilize and/or to reduce the size of organic compounds to overcome rate-limiting step of hydrolysis in biological digestion and to make sludge more easily biodegradable (Bougrier *et al.*, 2006).

The impact of sonication on the dewaterability of WAS has been also investigated in the past, but in most of the studies, a comparison between dewaterability data of sonicated and un-sonicated samples were made right after sonication process, while sonication is used to accelerate the digestion processes (Mason *et al.*, 2001). The present study, therefore, compares the dewaterability data of unsonicated samples with those of sonicated after digestion process is done.

MATERIALS AND METHODS

Sludge samples

Gravity-thickened waste activated sludge was collected from the municipal wastewater treatment plant of Ekbatan (West of Tehran). Table 1 presents the sludge samples main characteristics, in which the amounts of protein carbohydrate (EPS_p) and carbohydrate (EPS_c) parts are also given. This plant has a capacity of 100,000 people equivalent and the activated sludge is the main method of treatment within this plant.

Table 1: Waste activated sludge samples main characteristics

Sludge sample	а	b
TS concentration (g/L)	6.05	5.7
VS content (%TS)	76.4	74.9
Soluble COD (mg/L)	365	395
EPS_p (g eqBSA/L)	223	199
EPS_c (g eqGluc/L)	88	126
$CST_{10}(s)$	124	171
$SCST_{10}$ (s.L/g)	20.5	30

CST: Capillary suction time SCST: Spesific CST TS: Total solids VS: Organic solids BSA: Bovin serum albomine Glue: Glucose

Utrasonic treatment

The ultrasonic apparatus used was a 400W laboratory ultrasonic processor (Hielscher, Germany). It was equipped with several probes and worked with an operating frequency of 20

kHz. Batch experiments were carried out in 50 mL Croning tubes without temperature regulation (no cooling) and with the conical probe (Sonotrode H_3). Treated samples had a volume of 50 mL. "Specific supplied energy" ranged from 0 to 5,000 J/g of total solids (TS).

Analysis

In order to determine sludge composition, several measurements were made on samples according to Standard methods. Measurements of COD were done according to No. 5220 APHA standard method (APHA, 1998). For COD measurements, first soluble and particulate fractions were obtained centrifugation (Heraeus-Labofuge200, after 5000 rpm, 15 min); then, a known amount of oxidant was added to samples. After reaction completed, the excess oxidant was measured. By this analysis, it is possible to calculate the oxygen quantity necessary to degrade pollution, that is to say the COD concentration. COD was measured on the total sludge and on the supernatant. For this paper, COD measured on supernatant will be called "soluble COD" (SCOD).

Measurements of total and organic solids (TS and VS) were done on sludge according to No. 2540 APHA standard method (APHA, 1998). Samples were heated at 105 °C for 2 h; water was evaporated, leading to total solid (TS) concentration. Then, samples were heated at 550 °C for 2 h, leading to mineral matter concentration. Organic matter concentration was then deduced from TS and VS was then obtained.

EPS_P concentration was determined by measuring protein in supernatant using the Lowry method (Lowry et al., 1951). The technique quantified the peptidic bounds. After reactions with salts and Folin reagent, absorbance of samples was determined at 750 nm, using a spectrophotometer. By using different known solutions of bovine serum albumin (BSA), a calibration curve was obtained and protein concentrations were determined in BSA equivalent gram per liter. EPS_c concentration was determined by measuring carbohydrate concentration in supernatant using anthrone method (Moris, 1948). The technique quantified the carbonyl functions (C=O). After reaction with anthrone and sulphuric acid, absorbance of samples was determined at 625 nm using a spectrophotometer. By using different known solutions of glucose (Gluc), a calibration curve was obtained and carbohydrate concentrations were determined in glucose equivalent g/L.

The dewaterability was measured using capillary suction time (CST) according to standard method APHA 2710G [APHA, 1998] and was reported as CST and Specific CST (SCST). The apparatus was Triton CST meter, model 304B (Triton Electronics Ltd.).

RESULTS

Effect of EPS_p and EPS_c on sludge dewaterability Fig. 1 presents results obtained from measurement of EPS_p and EPS_c concentrations in the two different sludge samples before and after 2 months storage in different temperatures (5°C and 15°C). At the same time SCST data are measured to determine how the dewaterability changes with EPS concentration. As Fig. 1 implies, Difficulty in sludge dewatering decreased (SCST increased) with a decrease in EPS_p and EPS_c concentration in both cases. Also this experiment shows that the effect of EPS_p on sludge dewaterability is more significant than that of EPS_c (see Fig. 1). *Effect of sonication on sludge EPS concentration and dewaterability*

The effect of sonication with different intensities and energies on the release of protein is illustrated in Fig. 2.

Effect of sonication on sludge dewaterability after biological digestion

Anaerobic temperature-phased digestion process carried out on a sample of thickened (TS=2%) and sonicated (with different energies) waste activated sludge. Fig. 4 illustrates the effect of exposing WAS with ultrasound of 18.4 W/Cm intensity on dewaterability of sludge after the digestion process. The digestion process was a two step process in which the sludge first digested mesophilically at the temperature of 35°C for 10 days and then digested thermophilically at the temperature of 55°C for 7 days both anaerobically.

Dewaterability measurements (SCST) carried out in 3 steps: before any digestion, after mesophilic digestion, and after thermophilic digestion. The results of these measurements show that sonication, in general, made the dewatering of sludgemore difficult even after biological digestion (compare SCST of sonicated sludge with that of un-sonicated, see Fig. 4). Sonication, however, caused 26.5% improvement in dewaterability at sonication specific energy of 300 J/g TS. When



Fig. 1: Relationship between EPS_p, EPS_c and SCST in different sludge samples, (a) stored at 5°C; (b) stored at 15°C



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Fig. 2: Effect of sonication with different energies and intensities on Sludge protein concentration



Fig. 3: Relationship between protein concentrations in ultrasound treated sludge and SCST



Fig. 4: Effect of sonication energy on dewaterability of sludge after biological digestion

sonication intensity is set on 73.6 and 165.6 W/ Cm, these improvements were 18.6 and 3.8%, respectively, both at sonication specific energy of 300 J/gTS (data not shown here).

DISCUSSION

The result of this experiment was in contrast with some of the previous studies [Li *et al.*, 2008; Houghton *et al.*, 2000] where it was believed that EPS, due to their highly hydrated nature, have a negative effect on sludge dewaterability and in agreement with some studies in which otherwise were concluded (Haggins *et al.*, 1997) (Fig. 1). Recent studies found that the "proteins" are predominant constituents in EPS of mixed culture systems, such as activated sludge [Liu *et al.*, 2003]. Therefore in this study protein analysis is used as a determinant parameter for measuring EPS.

When sonication intensity is set on 18.3 W/ cm², protein concentration increases slowly as sonication energy increases to the point where sonication specific energy reaches 3000 J/g TS. After that point, protein concentration increases rapidly with energy. In case of 73.6 W/cm², this sudden increase happens at 1000 J/gTS. When sonication intensity is 165.6 W/cm², protein concentration increases rapidly from the first beginning (Na *et al.*, 2007).

These sudden increments could be due to the release of intracellular polymeric substances into the liquid phase as a result of breaking of microorganism cell walls under influence of highly exposed sonication.

Trying to find out the effect of sonication on dewaterability, result of another study shows a rapid increment of CST from 53 s before sonication to 68 s after sonication at low sonication densities (E_v less than 1000 KJ/L) and then sudden decrement of CST from 68 to less than 20 (at E_v between 1000 to 2000 KJ/L). At higher sonication densities, the study shows, CST decreases slightly with increasing E_v .

Fig. 3 shows how the increase in EPS_p as a result of sonication affects the dewaterability of sludge. SCST increases almost linearly with EPS_p . The increment of SCST (decrement of dewaterability) as a result of exposing sludge to ultrasound can be due to de-agglomeration of sludge flocs by ultrasound energy. Separation of sludge flocs causes the formation of small particles which will clog the filter pores and cause difficulty of dewatering.

Another researcher whose main purpose was to determine the impact of digestion process on dewaterability plotted the relationship between CST and the EPS yield graphically and found a poor relationship between these two parameters for raw sludge (Houghton *et al.*, 2000).

Overlly, polymeric substances, uncharacteristic of their highly hydrated nature, sometimes help dewatering. Ease of sludge dewatering (SCST) increased with a decrease in EPS_p and EPS_c concentration. The effect of EPS_p on sludge dewaterability is more significant than that of EPS_c . Biopolymers released by sonication not only do not help sludge dewatering, but also they have a negative effect on dewaterability. The deterioration of dewaterability had a strong correlation with the amount of soluble proteins. Low energy and low intensity sonication could help dewatering of thermophilically digested sludges.

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