



# Characterization of key organic compounds affecting sludge dewaterability during ultrasonication and acidification treatments



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## ABSTRACT

This study investigated the mechanism and effects of ultrasonic pretreatment followed by acidification on sludge dewaterability through looking at the changes of extracellular polymeric substances (EPS) content, composition and stratification. The results suggested sludge filterability was closely correlated with quantity of protein ( $R = 0.94$ ,  $p < 0.01$ ) and polysaccharide ( $R = 0.97$ ,  $p < 0.01$ ) present in loosely bound EPS rather than in soluble and tightly bound EPS. The fractions of polymers, especially tryptophan-like proteins and microbial by-product like material at molecular weight of  $10^6$ – $5 \times 10^7$  Da, were the key compounds related to sludge filterability. Ultrasonication may increase biopolymers concentrations that in turn deteriorate sludge filterability as evidenced at high ultrasonic power conditions. However, the subsequent acidification can reduce the concentrations of these organic compounds, reduce negative zeta potential, and increase floc size, thus increase sludge filterability. Combined ultrasonic-acid pretreatment was more effective than the acidification treatment alone in reducing the concentrations of macromolecular compounds that may deteriorate sludge filterability.

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## 1. Introduction

Sludge is a by-product of wastewater treatment, and its dewatering is an essential step to reduce sludge volume, transportation, disposal and storage costs (Devlin et al., 2011; Subramanian et al., 2010). Currently, in many wastewater treatment facilities, the bottleneck of sludge handling process is sludge dewatering (Neyens et al., 2004).

Ultrasonication, a mechanical process, has been applied to improve sludge dewaterability, with introduced effects of local heating, acoustic streaming, agitation and cavitation (de Sarabia et al., 2000). Li et al. (2009) reported ultrasonication could improve sludge dewaterability if the disintegration degree was controlled in the range of 2–5%. However, Wang et al. (2005a) and Ruiz-Hernando et al. (2014) reported ultrasonication may result in

deteriorated sludge dewaterability. In fact, the effects of ultrasonication on sludge dewaterability depended on the specific conditions applied, i.e. sonication time and power, sludge type and solids concentration (Lee and Liu, 2001; Wang et al., 2005a). The information about effects of ultrasonication on sludge dewaterability was limited and contradictory. A more general and/or universal set of parameters should be proposed to evaluate the ultrasonication effects.

The combination of acidification with other methods has been reported to improve sludge dewaterability than either single method. Combining Fenton oxidation with acidification showed a synergistic effect in enhancing sludge dewaterability (Zhang et al., 2015). Neyens et al. (2003) reported the combined thermal and acidification treatment (120 °C, pH = 3, 60 min) was effective in reducing residual sludge amount and improving sludge dewaterability compared to the traditional thermal treatment. However, the possibility of combined ultrasonication and acidification to improve sludge dewaterability has rarely been investigated.

Extracellular polymeric substances (EPS) was the most crucial deciding factor for sludge dewatering (Mikkelsen and Keiding, 2002). Houghton and Stephenson (2002) determined sludge dewaterability by the contents of EPS and a best sludge dewaterability

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**Abbreviations**

CST	Capillary suction time	PAC	Polyaluminium chloride
CST <sub>normalized</sub>	Normalized capillary suction time	PAM	Polyacrylamide
Da	Dalton	PN	Protein
d <sub>50</sub>	Medium particle size	PS	Polysaccharide
DOC	Dissolved organic carbon	R	Pearson's correlation coefficient
E <sub>m</sub>	Emission	RU	Raman units
EPS	Extracellular polymeric substances	rpm	Revolutions per minute
E <sub>x</sub>	Excitation	SCOD	Soluble chemical oxygen demand
g	Gravitational acceleration	S1	Region I in soluble extracellular polymeric substances
HAL	Humic acid-like substance	S2	Region II in soluble extracellular polymeric substances
HI DOC	Hydrophilic dissolved organic carbon	S3	Region III in soluble extracellular polymeric substances
HMW	High molecular weight	S4	Region IV in soluble extracellular polymeric substances
HO DOC	Hydrophobic dissolved organic carbon	S5	Region V in soluble extracellular polymeric substances
HPLC	High performance liquid chromatography	second	s
L1	Region I in loosely bound extracellular polymeric substances	SB EPS	Soluble extracellular polymeric substances
L2	Region II in loosely bound extracellular polymeric substances	SEC	Size exclusion chromatography
L3	Region III in loosely bound extracellular polymeric substances	3D EEM	Three dimensional excitation emission
L4	Region IV in loosely bound extracellular polymeric substances	T1	Region I in tightly bound extracellular polymeric substances
L5	Region V in loosely bound extracellular polymeric substances	T2	Region II in tightly bound extracellular polymeric substances
LB EPS	Loosely bound extracellular polymeric substances	T3	Region III in tightly bound extracellular polymeric substances
LC-OCD-OND	Organic carbon detection and organic nitrogen detection	T4	Region IV in tightly bound extracellular polymeric substances
LMW	Low molecular weight	T5	Region V in tightly bound extracellular polymeric substances
mAU	m absorbance unit	TB EPS	Tightly bound extracellular polymeric substances
min	Minutes	TOC	Total organic carbon
NaCl	Sodium chloride	TS	Total solids
p	Probability	VS	Volatile solids
		vs	Versus
		W	Watt

can be achieved with an optimal EPS content and composition. EPS are three-dimensional, gel-like, high molecular weight biopolymers originating from bacterial secretion, cell lysis and hydrolysis, leakage of exocellular constituents, and absorb organic matter from the surrounding wastewater (Zhang et al., 2016). EPS in sludge flocs consisted of soluble EPS (i.e. slime) and bound EPS. Bound EPS showed a dynamic double-layer-like structure, which included loosely bound EPS (LB EPS) and tightly bound EPS (TB EPS) (Li and Yang, 2007). Many studies have been carried out to investigate the role of EPS in sludge dewaterability during ultrasonication through EPS stratification. For example, Yu et al. (2008) reported ultrasonication promoted the shifts of proteins, polysaccharides and enzymes from inner sludge layers to outer layers, i.e. transformed TB EPS into SB EPS and LB EPS, thus improving aerobic digestibility. Shao et al. (2010) reported ultrasonication as pretreatment step can disintegrate sludge flocs and improve soluble organic compounds in EPS matrix, which would favour the degradation of organic matters in the subsequent anaerobic digestion. Furthermore, the dewaterability of treated sludge by ultrasonication combined anaerobic digestion process was better than anaerobic digestion alone. However, the correlations about EPS fractions and sludge dewaterability are inconsistent, with sludge dewaterability was correlated with protein in LB EPS during anaerobic digestion (Shao et al., 2010), biopolymers in EPS (Wang et al., 2006), soluble/colloidal fraction in EPS (Yin et al., 2004) or followed a polynomial equation (Feng et al., 2009). Therefore, the exact role of EPS during sludge ultrasonication and the combined

ultrasonication and acidification processes needs to be established. Moreover, previous studies mainly focused on characterizing overall dissolved organic compounds in EPS matrix, without prior fractionation based on different molecular weight. This may limit understanding about effects of EPS composition on sludge dewaterability during ultrasonication and the combined ultrasonication and acidification, as sludge dewaterability was largely affected by the molecular weight of related compounds (Niu et al., 2013). Therefore, further fractionation of organic compounds in EPS matrix based on different molecular weights is necessary.

The objectives of this study were to investigate the efficiency of combined ultrasonic-acid pretreatment to improve sludge dewaterability. Ultrasonic-acid, ultrasonic only and ultrasonic-alkaline conditions were compared and evaluated. Fraction and characterization of organic compounds in EPS matrix under different conditions were studied. Meanwhile, the correlation (positive or negative) between the key factors and sludge dewaterability was established through mathematical modelling. This study provides insights into the mechanism, merits and demerits of combined ultrasonic-acid pretreatment on sludge dewatering process.

## 2. Materials and methods

### 2.1. Sludge source

The sludge was collected from Ulu Pandan Water Reclamation Plant (Singapore). The sludge sample (pH 5.7–6.0), containing

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