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Evaluation of thermal hydrolysis efficiency of mechanically dewatered sewage sludge via rheological measurement



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ABSTRACT

In this study, laboratory tests of both low temperature $(60-90 \degree C)$ and high temperature $(120-180 \degree C)$ thermal hydrolysis (LTHP and HTHP) were performed on mechanically dewatered high-solid sludges (at total solid of 14.2 wt% and 18.2 wt%) to evaluate the extent of organic solubilization through rheological measurements. The effects of treatment temperature and duration on organic solubilization and viscoelastic behavior of the sludge were comprehensively investigated. The results indicated that the organic solubilization contents including soluble chemical oxygen demand, soluble protein, and soluble polysaccharides increased logarithmically with the treatment time. Protein solubilized considerably faster than polysaccharides during thermal hydrolysis. The rheological curves exhibited the Payne effect in the amplitude sweep oscillation test. The elastic modulus in linear viscoelastic regime decreased logarithmically with treatment time. The viscoelastic behavior of sludge was well modeled by the Kaye -Bernstein-Kearsly-Zapas (KBKZ) model with paralleled Maxwell elements to describe the frequency dependence of elastic modulus and viscous modulus. With respect to the relaxation spectrum, the relaxation modulus first decreased with relaxation time and then increased. The relaxation modulus in each Maxwell element decreased with the treatment temperature and duration. Furthermore, in the HTHP, the influence of treatment temperature on enhancing organic solubilization and decreasing viscoelasticity exceeded the influence of treatment duration. In contrast, the treatment duration played a more important role than temperature in the LTHP. The content of organic matters was linearly related and logarithmically related to the elastic modulus in the LTHP and in the HTHP, respectively. The rheology analyses demonstrated that viscoelastic properties could be used as indicators to estimate the extent of organic matter solubilization in thermal hydrolysis process. The developed viscoelastic model provided insights for future research in numerically simulating the fluid dynamics of sludge.

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1. Introduction

In wastewater treatment plants, excess sludge disposal processes, such as dewatering and anaerobic digestion, are utilized to reduce the volume and mass of sludge and stabilize and recover energy. In order to enhance the main treatment processes, a variety of pretreatments that partially solubilize the sludge are commonly used (Carrère et al., 2010), and among these processes, thermal hydrolysis is proposed as a preferable pretreatment to improve dewaterability (Neyens and Baeyens, 2003) and anaerobic degradability (Carrère et al., 2010) of sludge. Additionally, thermal

* Corresponding author. E-mail address: zhuoli2013@tongji.edu.cn (Z. Li). hydrolysis can improve the fluidity of the sludge to facilitate its subsequent handling in operations involving pumping and transportation in the sludge treatment process (Urrea et al., 2015).

In the structure of sludge, it is generally assumed that three types of water exist, namely free water, bound water, and in-cell water (Neyens and Baeyens, 2003). Extracellular polymeric substances (EPS) occur as highly hydrated capsules surrounding the cell wall and are released as slime polymers in the solution. They are of considerable importance in maintaining floc structure (e.g., bridging by hydrophobic interactions) and an entanglement network of sludge (Neyens and Baeyens, 2003). Thermal treatment destroys the structural integrity of sludge, lyzes cell walls of aggregate microbes, and releases EPS, bound water, and in-cell water. Macromolecular components are reduced to lower molecular weight derivatives or released as soluble monomeric



Nomenclature		$\omega \ au$	frequency (rad/s) shear stress (Pa)
LTHP, _L	low temperature thermal hydrolysis process	$ au_1$	the first critical shear stress in creep test, yield stress
НТНР, _{<i>н</i>}	high temperature thermal hydrolysis process		(Pa)
rSCOD	released soluble chemical oxygen demand	$ au_2$	the second critical shear stress in creep test (Pa)
rSPr	released soluble protein	t	time
rPs	released soluble polysaccharide	Т	temperature
0	parameters of untreated sludge	i, j	number, <i>i</i> , <i>j</i> = 1, 2, 3, …
G'	storage modulus (Pa)	Ν	total Maxwell elements number
G''	loss modulus (Pa)	λ_i	relaxation time of Maxwell element (s)
γ	shear stain (rad)	gi	relaxation modulus of Maxwell element (Pa)
γ_*	complex shear strain (%)	R	experimental result range
γ_0	oscillation shear strain amplitude (rad)		

substances (Wilson and Novak, 2009). Thus, thermal hydrolysis treatment can increase the soluble organic content of sludge.

Treatments involving higher temperature and longer duration are beneficial to solubilize increased amounts of soluble chemical oxygen demand (SCOD), soluble proteins (SPr), and soluble polysaccharides (SPs) (Nazari et al., 2017; Xue et al., 2015). A higher hydrolysis temperature in the range of 130 °C to 220 °C is particularly effective in solubilizing organic matter as it increases the SCOD fraction by approximately ten times that of the nonhydrolyzed counterparts (Wilson and Novak, 2009). In addition, the organic compounds did not react in the same way during thermal hydrolysis process (Neyens et al., 2004). For example, the solubilization of proteins was significantly higher than polysaccharides (Nazari et al., 2017). While the kinetics of the solubilization of organic compounds over a wide range of temperature and time still need to be systematically analyzed.

In order to evaluate the effect of thermal hydrolysis, quantitative changes in physical or chemical parameters can be used to define the efficiency of thermal hydrolysis process (Pilli et al., 2015). Chemical parameters reflecting soluble organic content including SCOD, SPr, and SPs are commonly used. Studies on thermal hydrolysis have been conducted for over 50 years. However, there is no generalized method to evaluate the efficiency of the pretreatment process (Pilli et al., 2015).

In addition to soluble organic content, rheological behavior is an important macroscopic physical property that can reflect the microscopic structure of sludge. A few studies reported that thermal hydrolysis influences organic solubilization as well as the rheological behavior of sludge. The thought based on the physical properties combined with chemical properties of sludge is thus anticipated to perfect the evaluation method of thermal hydrolysis. Rheological studies to-date have concentrated on thermally treated sludge under a temperature ranging from 60 °C to 100 °C, and the results revealed that thermal hydrolysis reduced apparent viscosity (Farno et al., 2014; Ruiz-Hernando et al., 2015), yield stress (Farno et al., 2014), and weakened thixotropic behavior (Ruiz-Hernando et al., 2015). An extant study (Ruffino et al., 2015) indicated that the influence of thermal hydrolysis appeared especially at a low shear rates regime. With respect to the sludge treated under higher temperature ranging from 120 °C to 200 °C, the viscosity decreased more significantly (Feng et al., 2014, 2015; Urrea et al., 2015). Recently, Farno et al. examined the change in COD with the variation in rheological properties and found a linear relationship between SCOD and infinite viscosity of digested sludge (3.5 wt%) (Farno et al., 2015) and waste activated sludge (Farno et al., 2016a) treated under 50-80 °C within 60 min. The above discussion indicated that the rheological characterization of sludge had the potential to serve as a new tool to evaluate the efficiency of thermal hydrolysis in sludge treatment.

Treatment temperature and duration are the two main variables that affect thermal hydrolysis. Typically, treatments applied at temperatures above 100 °C are considered as high temperature thermal hydrolysis processes (HTHP). These processes are more commonly used for dewatering and anaerobic digestion pretreatment and were commercialized by CAMBI and BIOTHELYS (Pilli et al., 2015). Treatments under 100 °C are considered as low temperature thermal hydrolysis process (LTHP) and possess advantages as they overcome high-energy requirements and form refractory compounds by Maillard reactions (over 200 °C) in HTHP (Gavala et al., 2003). In order to achieve efficient solubilization, the required treatment duration varied significantly between HTHP and LTHP processes. The solubilization could reach a stable state within 30-60 min with respect to HTHP (Carrère et al., 2010) but ranged from hours to days with respect to LTHP (Climent et al., 2007; Xue et al., 2015). Previous studies mainly examined the effects of thermal treatment conditions on rheological properties of sludge in the context of the LTHP within a short treatment duration less than 60 min. There is a lack of systematical evaluations on the rheological properties of the same type of sludge treated using both HTHP and LTHP process for sufficient time to reach a stable solubilization.

Additionally, most of the above-mentioned rheological studies used unconditioned excess sludges. However, conditioned mechanically dewatered sludge with high-solid (total solid (TS) more than 10 wt%) is another common product in waste water treatment plants. The dewatering process reduces the volume of sludge, increases the energy content of the sludge for incineration, and decreases the amount of leachate production in landfills (Lo et al., 2001). In addition to incineration and landfill, the technique of advanced anaerobic digestion has advantages in managing the high-solid mechanically dewatered sludge by energy recovery to reduce the reactor volume and increase the organic loading (Zhang et al., 2016). Thermal hydrolysis is a necessary pretreatment step for advanced anaerobic digestion in achieving better fluidity. Chemical conditioning is often applied to improve the mechanical dewaterability of the waste sludge by flocculating sludge particles with flocculants. Ferric chloride, cationic derivatives of polyacrylamide (PAM), and lime are used as conditioners in the wastewater treatment plants (Lo et al., 2001). This critical process strengthens the floc structure and forms network structures of sludge (Stickland et al., 2008; Yen et al., 2002). As a result, highsolid mechanically dewatered sludge with a conditioned process is a highly complex fluid and exhibits different flow behavior with strong viscoelasticity that is evidently different from unconditioned

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