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## Dynamic variation in physicochemical properties of activated sludge floc from different WWTPs and its influence on sludge dewaterability and settleability



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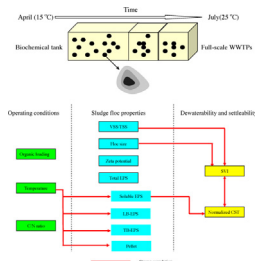
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### HIGHLIGHTS

- Dynamic change in physicochemical properties of activated sludges was investigated.
- Variation in sludge EPS properties from different WWTPs showed a similar pattern.
- Temperature had more significant influence on distribution and composition of EPS.
- Sludge dewaterability correlated well with the content of proteins in soluble EPS.
- Sludge settleability was primarily affected by floc size distribution and VSS.

### GRAPHICAL ABSTRACT



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### ABSTRACT

Biosolid–liquid separation performance plays an important role in suspended solids removal and sludge thickening and dewatering process. Since the sludge is a highly complex colloidal system, the key factors affecting sludge dewatering and settling performance were not well understood. In this study, the dynamic variation in distribution and chemical composition of extracellular polymeric substances (EPS), floc size distribution, charge property and settling and dewatering performance was investigated at four full scale WWTPs over 100 days. It was found that the change in EPS distribution and composition from different WWTPs showed a very similar pattern with operating time. The temperature rather than wastewater composition had more significant influence on content and chemical constituents of different EPS fractions. Additionally, more EPS was extracted from activated sludge at lower temperature (<15 °C), indicating that the binding strength of EPS with microbial cells was weak. According to Pearson's correlation analysis, sludge dewatering property correlated well with content of protein-like substances in soluble EPS, while there was a strong correlation between sludge settleability (measured by sludge volume index) and floc size and volatile suspended solid/total suspended solid ratio (VSS/TSR).

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## 1. Introduction

Wastewater treatment processes produce large quantities of sludge commonly containing over 90% water [1]. Biosolid–liquid separation by gravity settling and mechanical dewatering are of particular importance to the activated sludge process [2]. Poor settleability can cause operational problems and discharge of suspended solids to the effluent, while effective dewatering has a significant influence on sludge reduction.

Activated sludge is a heterogeneous colloid system and characterized by high specific surface area, irregular structure and negative charge [3]. Extracellular polymeric substances (EPS) are one of the most crucial constituents of activated sludge due to their specific chemical composition and structures. EPS were mainly consisted of high-molecular-weight polysaccharides (PS), proteins (PN), glycoproteins, nucleic acids, phospholipids, and humic acids (HA) [4]. EPS could act as glue and serve to bind cells together to form flocs. EPS is one of the key constituents of activated sludge and greatly affects physicochemical properties of sludge system. The EPS content and composition significantly influenced the bioflocculation ability, settleability, surface charge properties, floc size distribution and hydrophobicity and so on [4]. According to the binding strength with cells, the EPS could be fractionated into soluble EPS, loosely bound EPS (LB-EPS) and tightly bound EPS (TB-EPS). The soluble EPS was mainly composed by dissolved high MW biopolymers while the bound EPS primarily contained cell capsule and loose biopolymers [5]. Li and Yang found that the sludge dewatering properties were much more strongly correlated with the concentration of LB-EPS than with the concentration of TB-EPS, and excessive LB-EPS could weaken the floc structure resulting in poor bioflocculation and sludge–water separation performance [6]. Additionally, they found that the sludge flocculating behavior, settleability, compressibility and dewaterability correlated well with LB-EPS content rather than TB-EPS content under the non-steady-state operation.

Activated sludge processes are characterized by transient operating conditions. The environmental conditions change with time on short term (h) as well as on long term scales (days, months). Many factors change simultaneously and even small changes in the floc properties can affect the performance of the sludge–water separation performance. In most previous studies, the bioflocculation, settling and dewatering properties of activated sludge have been performed either in controlled laboratory or pilot scale systems where only a limited number of parameters have been varied at the same time. Therefore, the objective of this work was to: (1) monitor the dynamic variation in EPS distribution and composition of sludge floc from three WWTPs; (2) investigate the effect of operating conditions (SRT, temperature) and influent qualities on physicochemical properties of sludge floc; and (3) get insight into the relationships between EPS properties and sludge dewatering and settling performance.

## 2. Materials and methods

### 2.1. Sources of sludges

28 wastewater sludge samples were collected from sludge recycle pumping lines of the three full-scale WWTPs (four processes) in Beijing from May to October, 2013, each mainly treating domestic wastewater. The treatment process of each WWTP was presented in Table 1.

The influent qualities and operating conditions can be found in Table 2. The collected samples were transported to a laboratory within 6 h after sampling and then stored at 4 °C prior to analysis. Basic properties of all wastewater sludge were given in Table 1. The QHA and QHM received the same influent.

### 2.2. Analytical methods

#### 2.2.1. Basic properties

A laser diffraction instrument (Malvern Mastersizer 2000, Malvern, UK) was used to measure dynamic floc size. pH was measured by pH meter (pHS-3C, Shanghai, China). Zeta potential was determined with Malvern Zetasizer 2000. Other sludge parameters, including total suspended solid (TSS) and volatile suspended solids (VSS), were analyzed following standard methods. TOC (total organic carbon) was determined with a TOC analyzer (Teledyne Tekmar, USA).

#### 2.2.2. Determination of sludge dewaterability and settleability

Sludge dewaterability was measured with a portable capillary suction time (CST) instrument (CST 304B, Triton, UK) equipped with an 18 mm diameter funnel and Whatman no. 17 chromatography-grade paper. The CST values were normalized by dividing them by the initial TSS concentration and then expressed in units of seconds per liter per gram TSS. The sludge settleability was characterized by the sludge volume index (SVI), which is defined as the volume (in mL) occupied by 1 g of sludge after 30 min of settling in a 1 L cylinder.

#### 2.2.3. Extraction and analysis of EPS

EPS fractionation process of sludge samples was modified based on the procedures described by Yu et al. [7]. Firstly, raw sludge sample was settled down at 2000 g for 15 min, and the supernatant was collected as soluble EPS. The sediment was resuspended to its initial volume with phosphate buffer solution consisting of Na<sub>3</sub>PO<sub>4</sub>, NaH<sub>2</sub>PO<sub>4</sub>, NaCl and KCl. And then the suspension was transferred and centrifuged at 5000 g for 15 min, and the supernatant and sediments were collected separately. This supernatant was used to determine LB-EPS later. The collected sediment was re-suspended with the phosphate buffer solution to the original volume following by treatment with ultrasound at 20 kHz and 480 W for 10 min. The extracted solution was centrifuged at 20,000 × g for 20 min and separated as TB-EPS. Finally, the solid residues were again re-suspended with the buffer to the original volume. This fraction was the pellet. The particulates present in the supernatant, soluble EPS, LB-EPS, and TB-EPS solutions were removed with polytetrafluoroethylene membranes with a pore size of 0.45 μm prior to chemical composition analysis. Total extractable EPS for each EPS fraction was defined as the sum of protein, carbohydrate and humic substances.

All chemical analyses were carried out in duplicate using chemicals of analytical grade. The protein, carbohydrate and humic substances in extracted EPS were determined according to procedure described by Wang et al. [8]. Polysaccharide was measured using the anthrone method with a glucose standard (Sinopharm). Protein and humic substances were determined with the Lowry procedure using bovine serum albumin (BSA) (Sigma) and humic acid (Sigma) as standards respectively. In addition, the molecular weight distribution of EPS was measured with high performance size-exclusion chromatography (HPSEC). MW was measured by a Waters liquid chromatography system which was consisted of Waters 2487 Dual λ Absorbance Detector, Waters 1525 pump system. A Shodex KW 802.5 column (Shoko, Japan) was used for separation. The mobile phase, Milli-Q water buffered with 5 mM phosphate to pH 6.8, and 0.01 M NaCl, was filtered through a 0.22 μm membrane, and then degassed for 30 min by means of ultrasonication for 0.5 h before being used to the column. 600 μL sample was injected at a flow rate of 0.8 mL min<sup>-1</sup>. Polystyrene sulfonate standards (Sigma–Aldrich, USA) of molecular weights 1.8–32 kDa were used for apparent molecular weight (AMW) calibration.

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